METHOXYFENOZIDE (209)

first draft prepared by Stephen Funk, US Environmental Protection Agency, USA

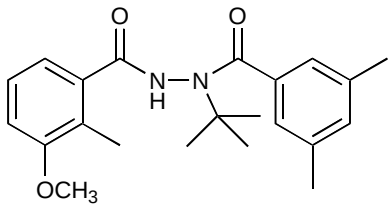
EXPLANATION

Methoxyfenozide, a substituted dibenzoylhydrazine, is an insecticide that functions by accelerating the moulting process. It acts as an ecdysone agonist or ecdysonoid, substituting for the natural insect moulting hormone, 20-hydroxyecdysone. Methoxyfenozide is active on all feeding larval stages of the target Lepidoptera.

Methoxyfenozide is a new pesticide in the Codex system. The CCPR, in 2001 (33rd Session), requested a JMPR evaluation in the year 2003. The manufacturer submitted studies on metabolism, supervised field trials, processing, farm animal feeding, analytical methods and freezer storage stability. The manufacturer and the governments of Australia and the Netherlands provided GAP information.

IDENTITY

ISO common name: methoxyfenozide
IUPAC name: *N-tert*-butyl-*N'*-(3-methoxy-*o*-toluoyl)-3,5-xylohydrazide
Chemical Abstracts name: benzoic acid, 3-methoxy-2-methyl-, 2-(3,5-dimethylbenzoyl)-2-(1,1-dimethylethyl) hydrazide
CAS No.: 161050-58-4
Synonyms/trade names: RH-2485, RH-112485
Structural formula:



Molecular formula: $C_{22}H_{28}N_2O_3$
Molecular weight: 368.47

Physical and chemical properties

Pure active ingredient

Property	Value	Reference
Minimum purity	99.8%	Meyer, 1996a
Appearance	white powder	Meyer, 1996a and 1995a
Vapour pressure	$<1.33 \times 10^{-5}$ Pa at 25, 35 and 45ºC	Meyer,1996a, Kogovsek, 1995
Henry's law constant	$<1.64 \times 10^{-4}$ Pa m^3/mol at 20ºC	Carpenter, 1996
Melting point	206 to 208ºC	Meyer, 1996a and 1995b
Partition coefficient	log P_{ow} 3.72 ± 0.04 at 24.7 ± 1.4ºC (pH 7), not dependent on pH.	Meyer, 1996a, Lin, 1994a
Water solubility	3.3 mg/l at 20 ± 0.5ºC	Meyer, 1996b
Solvent solubility butyl acetate acetone xylene methanol 2-propanol *n*-heptane 1,2-dichloroethane	at 24.7 ± 0.3ºC: 18.8 g/kg 127 g/kg 3.38 g/kg 193 g/kg 50.2 g/kg 1.87 g/kg 36.7 g/kg	Meyer, 1996a and 1995c
Relative density	0.740 g/ml at 25 ± 0.5ºC	Meyer, 1996a and 1995d

Property	Value	Reference
Hydrolysis, DT_{50} at 24.9 ± 1.6ºC	pH5, 590 ± 410 days pH 7, 1600 ± 800 days pH 9, 700 ± 230 days	Lin, 1994b
Photolysis (aqueous)	DT_{50}, 2200 days at pH 7, 25°C (irradiated or dark); DT_{50}, 77 days, pond water, 25°C	Smalley and Reynolds, 1997
Dissociation constant	does not dissociate	
Thermal stability	stable below 240ºC	Meyer, 1996a and 1995e

Technical material

Property	Value	Reference
Minimum purity	≥93%	
Melting point range	204 to 206.6ºC	Meyer, 1996a and 1995b
Stability	Stable to simulated sunlight for 3 days (33.5 ± 1.0ºC) and in contact with iron oxide for 5 days (24.7 ± 0.3ºC)	Meyer, 1996a and 1995e

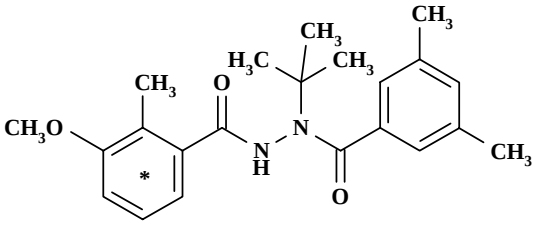
Formulations

Methoxyfenozide is available as suspension concentrate (SC) formulations, containing 240 g ai/l or 100 g ai/l, and as wettable powder (WP) formulations, containing 80% (w/w) active ingredient.

METABOLISM AND ENVIRONMENTAL FATE

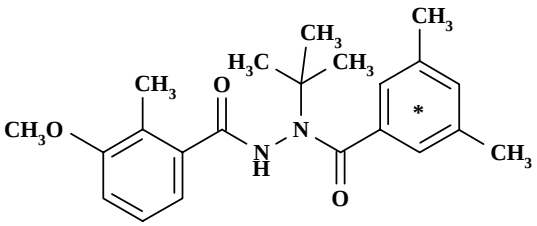
The fate and behaviour of methoxyfenozide (RH-2485 or RH-112485) in animals, plants, rotational crops and soil were investigated using the following [^{14}C] labelled test materials:

a. Methoxyphenyl ring uniformly labelled-^{14}C-methoxyfenozide (referred to below as "A-ring label")



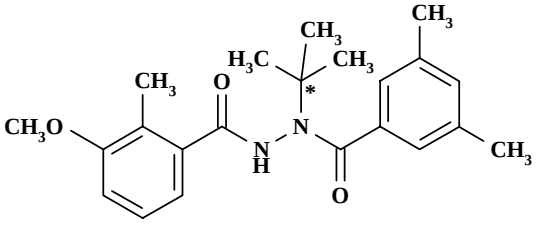
* labelling position

b. Dimethylphenyl ring uniformly labelled-^{14}C-methoxyfenozide (referred to below as "B-ring label")



* labelling position

c. *t*-Butyl ^{14}C-methoxyfenozide (referred to below as "*t*-butyl label")



* labelling position

The names and structures of metabolites and degradation products from animal and plant metabolism and environmental fate studies are given in Table 21.

Animal metabolism

The Meeting received information on the metabolism of methoxyfenozide in lactating goats and laying hens, following oral dosing with radiolabelled material.

Lactating goats

The metabolism, distribution and elimination of ^{14}C-methoxyfenozide (also known as RH-112485 or RH-2485), labelled in the A-ring, B-ring, and *t*-butyl positions (see above), were studied in lactating Toggerburg dairy goats (36–53 kg) (Wu, 1998a, report No. 34-97-47). ^{14}C-RH-methoxyfenozide was administered orally in gelatin capsules to the goats, once a day at approximately 50 ppm dietary equivalents for 7 consecutive days. Goats A, B, and C received methoxyfenozide labelled in the A-ring (radiochemical purity 98.2%), B-ring (radiochemical purity 99%) and *t*-butyl group (radiochemical purity 99%), respectively. Goat D received a lactose placebo.

Each dosing capsule contained approximately 75 mg of methoxyfenozide. Based on the dietary consumption during the dosing period, the actual dose rates were 45, 32 and 61 ppm for goats A, B and C, respectively, or 2.1, 1.8, and 1.7 mg/kg bw/day, respectively, based on the body weights on day 1 of the study. The goats were allowed *ad libitum* access to feed and water. Feed consumption of each animal was monitored. Milk, faeces and urine samples were collected twice daily (morning and afternoon); faeces and urine samples were each pooled into one daily sample. A cage rinse was performed daily. Blood samples were taken on the day prior to dosing and on days 1, 3 and 7 post-dosing. Goats were sacrificed within 24 hours after the final dose. Blood, liver, kidney, loin muscle, leg muscle and fat samples were removed at necropsy for analysis.

All samples were stored below -10ºC before analysis. Whole milk was extracted with acetonitrile and acetonitrile/water and the extract sequentially partitioned into hexane and ethyl acetate. Tissues, except fat, were extracted with methanol and chloroform and the extract partitioned between acetonitrile and hexane. Fat was extracted with hexane and the extract partitioned into acetonitrile. Unextracted radio-label (liver) was subjected to acid, base and/or enzyme (protease) hydrolysis. Procedural recoveries were performed by fortifying milk, liver and fat control samples with t-butyl- and A-ring-labeled methoxyfenozide. TRR determination in samples and extracts and post-extraction solids was by LSC or combustion LSC. Analysis was by radio-TLC and reversed-phase HPLC, with radio-chemical and UV (254 nm) detection and confirmation by LC-MS/MS and NMR (RH-141518 and other glucuronide conjugates).

The total recovery of administered dose from the A-ring, B-ring, and *t*-butyl dosed goats was 82.8%, 88.7% and 81.5%, respectively. The test compound was eliminated primarily via faeces: 76.2%, 83.7%, and 74.3% of the dosed radioactivity from the A-ring, B-ring, and *t*-butyl labels, respectively. Urine and the cage rinse accounted for 6.4% (A-ring), 5.0% (B-ring), and 7.0% (*t*-butyl), respectively, of the total dose.

The TRR levels were generally highest in the tissues of the *t*-butyl label dosed goat, with the goat dosed with B-ring label having the lowest residues among the dose levels. TRR levels were highest in the liver, followed by kidney and fat. TRR was insignificant in the muscle tissues of the B-ring label dosed goat (<0.01 mg/kg) but slightly higher in the muscle tissues of both the A-ring label dosed goat (0.013 mg/kg) and the *t*-butyl label dosed goat (0.023 mg/kg). Milk contained low residues, ranging from <0.01-0.018 mg/kg for A-ring, <0.01-0.014 mg/kg for B-ring and 0.01-037 mg/kg for *t*-butyl. Residues appeared to reach a plateau in milk within 24-36 hours after the initiation of dosing, although the t-butyl label appeared not to reach a plateau until 6 days after the initial dose. Tables 1 and 2 list the TRR distribution in milk and in the various goat tissues.

Table 1. Distribution of radioactivity in goat milk (mg/kg parent equivalents) following treatment with ^{14}C-methoxyfenozide (Wu, 1998a).

Interval		Goat A, A-ring label, (45 ppm)		Goat B, B-ring label (32 ppm)		Goat C, *t*-butyl label (61 ppm)	
		Cumulative % of dose	mg/kg	Cumulative % of dose	mg/kg	Cumulative % of dose	mg/kg
Day 1	am	<0.01	<0.010	0.01	0.010	0.01	0.020
	pm	0.02	<0.010	0.02	<0.010	0.03	0.024
Day 2	am	0.01	0.016	0.02	0.014	0.02	0.032
	pm	0.02	0.010	0.02	<0.010	0.04	0.028
Day 3	am	0.02	0.016	0.02	<0.010	0.03	0.024
	pm	0.02	<0.010	0.02	<0.010	0.04	0.019
Day 4	am	0.02	0.011	0.02	<0.010	0.03	0.019
	pm	0.02	<0.010	0.02	<0.010	0.04	0.023
Day 5	am	0.02	0.014	0.02	<0.010	0.04	0.031
	pm	0.02	<0.010	0.02	<0.010	0.04	0.031
Day 6	am	0.02	0.016	0.02	<0.010	0.04	0.037
	pm	0.02	0.012	0.02	<0.010	0.05	0.029
Day 7	am	0.02	0.018	0.02	<0.010	0.04	0.035
	pm	0.03	0.013	0.02	<0.010	0.05	0.031

Table 2. Distribution of radioactivity in goat tissues (mg/kg parent equivalents) following treatment with ^{14}C-methoxyfenozide (Wu, 1998a).

Matrix	Group 1, A-ring		Group 2, B-ring		Group 3, *t*-butyl label	
	% [1/] of dose	mg/kg	% [1/] of dose	mg/kg	% [1/] of dose	mg/kg
Blood day 1	<0.01	0.027	<0.01	0.005	<0.01	0.033
Blood day 3	<0.01	0.029	<0.01	0.006	<0.01	0.048
Blood day 7	<0.01	0.034	<0.01	0.09	<0.01	0.099
Liver	0.11	0.929	0.05	0.257	0.14	1.18
Kidney	<0.01	0.183	<0.01	0.045	<0.01	0.197
Leg muscle	<0.01	0.013	<0.01	<0.010	<0.01	0.017
Loin muscle	<0.01	0.013	<0.01	<0.010	<0.01	0.023
Omental fat	<0.01	0.051	<0.01	0.018	0.01	0.053
Faeces	76		84		74	
Urine [2/]	6.4		5.0		7.0	
Total incl. milk	83		89.		82.	

[1/] Based on sample taken, not entire tissue.
[2/] Includes cage rinse.

Day 2 (pm), day 7 (am) and day 7 (pm) milk, and all tissue samples with TRR levels greater than 0.01 mg/kg, were subjected to extraction and/or hydrolysis procedures for residue characterization and identification (Tables 3-5). The following metabolites were isolated from various matrices: RH-117236 (A-ring phenol or demethylated parent); RH-131154 (B-ring carboxylic acid); RH-152068 (B-ring mono-alcohol, B-ring mono-carboxylic acid); RH-141511 (A-ring phenol, B-ring mono-alcohol); RH-141518 (glucuronide conjugate of A-ring phenol); RH-149087 (glucuronide conjugate in A-ring); and RH-141519 (glucuronide conjugate of A-ring phenol, B-ring mono-alcohol). In addition, glucuronide conjugates of the A-ring, with an additional -OH group *ortho-* or *para-* to the glucuronide moiety, were isolated and identified (metabolite H). Methoxyfenozide was the major residue component in fat, muscle, and milk. Liver and kidney showed a large percentage of the metabolite RH-141518 (glucuronide conjugate of A-ring phenol) and trace amounts of other glucuronides. Identities and characterizations are summarized in Tables 3 and 4. A possible metabolic pathway is shown in Figure 1.

Table 3. Identification of extractable residues in goat milk collected at various time periods following treatment with ^{14}C-methoxyfenozide (Wu, 1998a).

Label		A-ring			B-ring	*t*-butyl-ring		
Metabolites		Day 2, pm	Day 7, am	Day 7, pm	Day 2, pm	Day 2, pm	Day 7, am	Day 7, pm
Methoxyfenozide	% TRR	30	15	35	31	32	11	14
	mg/kg	0.005	0.002	0.006	0.005	0.009	0.003	0.005
RH-117236	% TRR	(-)	1.1	1.8	1.8	1.5	0.96	(-)
	mg/kg	(-)	<0.001	<0.001	<0.001	<0.001	<0.001	(-)
RH-131154	% TRR	(-)	2.2	2.7	4.2	2.6	0.86	(-)
	mg/kg	(-)	<0.001	<0.001	0.001	<0.001	<0.001	(-)
RH-152068 &/or [1/] RH 141511	% TRR	21	6.1	8.8	9.5	6.1	7.7	(-)
	mg/kg	0.003	0.001	0.001	0.001	0.002	0.002	(-)
RH-149087	% TRR	(-)	(-)	(-)	(-)	2.3	6.0	4.3
	mg/kg	(-)	(-)	(-)	(-)	<0.001	0.002	0.002
RH-141518	% TRR	(-)	0.54	(-)	(-)	3.5	3.2	5.0
	mg/kg	(-)	<0.001	(-)	(-)	0.001	<0.001	0.002
RH-141519	% TRR	(-)	0.16	0.32	(-)	(-)	4.2	6.0
	mg/kg	(-)	<0.001	<0.001	(-)	(-)	0.001	0.002
Metabolite H	% TRR	(-)	2.6	1.8	2.2	1.8	0.97	(-)
	mg/kg	(-)	0.001	<0.001	<0.001	<0.001	<0.001	(-)
Lactose	% TRR	(-)	(-)	(-)	(-)	22.8	26.6	31.4
	mg/kg	(-)	(-)	(-)	(-)	0.007	0.008	0.011
Unknowns [2/]	% TRR	33	42	34	35	14	21	22
	mg/kg	0.005	0.005	0.007	0.005	0.003	0.006	0.007
PES [3/]	% TRR	16	30	15	16	14	17	16
	mg/kg	0.003	0.004	0.003	0.002	0.004	0.005	0.006
Total identified	% TRR	51	28	51	49	72	61	61
	mg/kg	0.008	0.004	0.009	0.007	0.023	0.019	0.021

(-) indicates "not observed".

[1/] Both metabolites (also designated as Met-E_2 and Met E_2) were isolated from the same retention time fraction.

[2/] Unknowns included other solvent-released metabolites. Most, about 30% TRR, were water-soluble.

[3/] PES = post-extraction solids.

Table 4. Identification of extractable residues in goat tissues following treatment with ^{14}C-methoxyfenozide (Wu, 1998a).

Label		A-Ring				B-ring			*t*-butyl-ring				
Metabolites		Liver, 0.93 mg/kg	Kidney, 0.18 mg/kg	Fat, 0.051 mg/kg	Leg muscle, 0.013 mg/kg	Liver, 0.26 mg/kg	Kidney, 0.045 mg/kg	Fat [4/], 0.018 mg/kg	Liver, 1.2 mg/kg	Kidney, 0.20 mg/kg	Fat, 0.053 mg/kg	Loin muscle, 0.023 mg/kg	Leg muscle, 0.017 mg/kg
Methoxyfen-ozide	% TRR	3.2	2.1	73	20	2.6	(-)	68	2.1	2.0	81	25	19
	mg/kg	0.030	0.004	0.037	0.003	0.007	(-)	0.012	0.024	0.004	0.043	0.006	0.003
RH-117236	% TRR	7.4	2.5	2.6	0.72	3.2	(-)	(-)	2.4	1.0	(-)	2.7	1.3
	mg/kg	0.069	0.005	0.001	<0.001	0.008	(-)	(-)	0.028	0.002	(-)	<0.001	<0.001
RH-152068 +/or [1/] RH 141511	% TRR	3.0	12	(-)	0.68	2.8	3.7	(-)	2.4	6.5	(-)	5.7	0.69
	mg/kg	0.028	0.022	(-)	<0.001	0.007	0.002	(-)	0.028	0.013	(-)	0.001	<0.001
RH-131154	% TRR	0.48	1.8	(-)	0.25	0.43	(-)	(-)	1.1	1.5	(-)	1.2	0.81
	mg/kg	0.004	0.004	(-)	<0.001	0.001	(-)	(-)	0.013	0.002	(-)	<0.001	<0.001
RH-149087	% TRR	2.1	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	20	(-)
	mg/kg	0.020	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	(-)	0.005	(-)
RH-141518	% TRR	30	42	8.1	(-)	29	32	(-)	23	25	(-)	7.3	(-)
	mg/kg	0.27	0.077	0.004	(-)	0.075	0.015	(-)	0.27	0.049	(-)	0.002	(-)
RH-141519	% TRR	7.5	14	(-)	(-)	9.5	4.9	(-)	9.8	7.2	(-)	(-)	(-)
	mg/kg	0.070	0.026	(-)	(-)	0.024	0.002	(-)	0.12	0.014	(-)	(-)	(-)
Metabolite H	% TRR	2.6	1.0	(-)	(-)	0.32	18	(-)		0.50	(-)	(-)	(-)
	mg/kg	0.024	0.002	(-)	(-)	0.001	0.008	(-)		0.001	(-)	(-)	(-)
Unknowns [2/]	% TRR	3.9	8.4	14	51	3.3	27	11	20 [5/]	34 [6/]	5.0	6.1	53
	mg/kg	0.036	0.015	0.007	0.006	0.009	0.012	0.002	0.24	0.067	0.003	0.001	0.009
Aqueous soluble	% TRR	30	11	(-)	50 [3/]	43	(-)	(-)	36	17	(-)	(-)	(-)
	mg/kg	0.28	0.021	(-)	0.006	0.11	(-)	(-)	0.43	0.034	(-)	(-)	(-)

Label		A-Ring				B-ring			*t*-butyl-ring				
Metabolites		Liver, 0.93 mg/kg	Kidney, 0.18 mg/kg	Fat, 0.051 mg/kg	Leg muscle, 0.013 mg/kg	Liver, 0.26 mg/kg	Kidney, 0.045 mg/kg	Fat [4/], 0.018 mg/kg	Liver, 1.2 mg/kg	Kidney, 0.20 mg/kg	Fat, 0.053 mg/kg	Loin muscle, 0.023 mg/kg	Leg muscle, 0.017 mg/kg
Post-extraction solids	% TRR mg/kg	1.5 0.14	3.5 0.006	1.9 0.001	27 0.003	4.1 0.011	13 0.006	20 0.004	0.17 0.002	3.8 0.007	14 0.007	32 0.007	25 0.004
Total identified	% TRR mg/kg	56 0.52	76 0.14	84 0.043	22 0.003	48 0.12	59 0.027	68 0.012	40 0.48	44 0.086	81 0.043	62 0.014	22 0.004

(-) indicates "not observed".

[1/] Both metabolites (previously designated as Met-E_2 and Met E_2) were isolated from the same retention time region and it was not possible to distinguish which one was detected without isolating them from the fractions. Hence, the value may represent either one or the sum of both metabolites.

[2/] Unknowns include other solvent-released and partly-characterized metabolites.

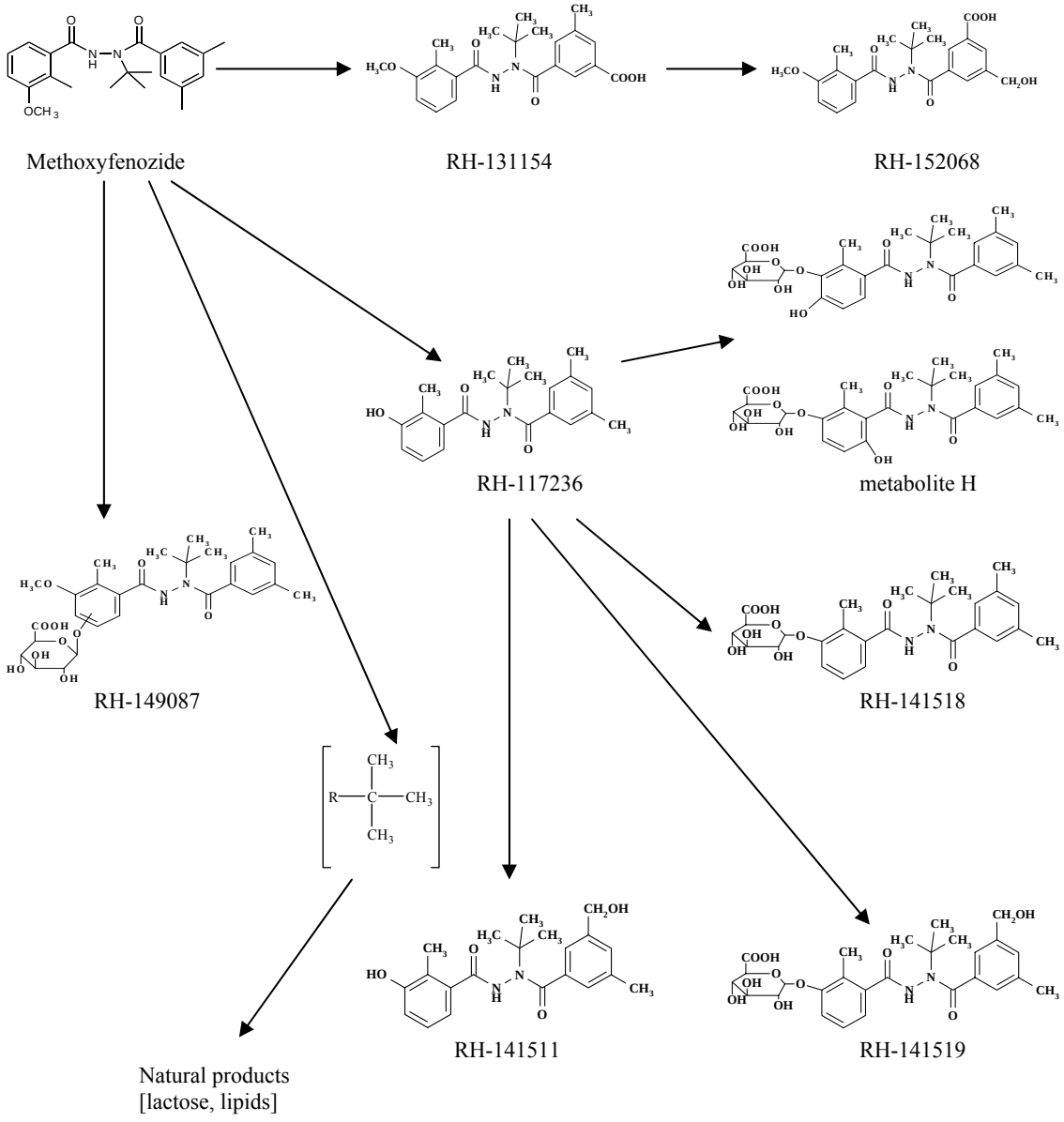
[3/] Methanol/water soluble.

[4/] Loin muscle (0.006 mg/kg): 50% TRR methanol/water soluble.

[5/] 18% TRR (0.22 mg/kg) characterized as triglycerides.

[6/] 22% TRR 9 (0.043 mg/kg) characterized as triglycerides.

Figure 1. Proposed metabolic pathway for methoxyfenozide in lactating goat.



Laying hens

The metabolism of ^{14}C-methoxyfenozide, labelled in the A-ring, B-ring, and *t*-butyl positions, was studied in laying hens (Wu, 1998b, report No. 34-97-48). The ^{14}C-methoxyfenozide was administered orally in gelatin capsules once a day at approximately 50 ppm dietary equivalents for 7 consecutive days. Based on the dietary consumption during the dosing period, the actual dose rates were 58, 60 and 68 ppm for the A-ring, B-ring, and *t*-butyl dose groups, respectively. Each hen received about 6 mg of methoxyfenozide per day.

Excreta were collected once, and eggs twice, daily. Each day, from the day prior to dosing, the morning's eggs were combined with the previous afternoon's eggs and pooled by treatment group. A cage rinse was performed daily. Tissues (kidney, liver, light muscle, dark muscle, abdominal fat, and skin) were collected after all birds were sacrificed, less than 24 hours after the final dose. The tissues were combined within each treatment group.

Eggs were homogenized and triplicate aliquots analyzed by combustion/LSC. Liver, kidney, skin and muscle samples were frozen in liquid N_2 or dry ice, homogenized and triplicate aliquots were subjected to combustion/LSC. Fat samples were frozen in liquid N_2 or dry ice, homogenized, then dissolved in hexane and triplicate aliquots were analyzed by LSC. Limits of determination for TRR were reported as 0.001-0.007 mg/kg.

Total recovery of the administered dose from the A-ring, B-ring, and *t*-butyl groups was 84, 93, and 89%, respectively. The TRR were generally highest in the eggs and tissues of *t*-butyl label dosed hens. The distribution of the TRR in tissues and eggs is shown in Table 5. Transfer of the residues into the eggs and tissues was minimal, with the majority of the dosed radioactivity passing through the hens and being recovered in the excreta. Less than 0.03% of the dosed radioactivity was recovered in the eggs and tissues from any test group.

Table 5. Total radioactive residues and the distribution of radioactivity in poultry eggs and tissues following treatment with ^{14}C-methoxyfenozide (Wu, 1998b).

Matrix/interval	A-ring labelled		B-ring labelled		*t*-butyl labelled	
	TRR % of dose	mg/kg	TRR % of dose	mg/kg	TRR % of dose	mg/kg
Eggs						
Day 1	<0.01	0.016	<0.01	0.005	<0.01	0.019
Day 2	<0.01	0.025	<0.01	0.019	<0.01	0.032
Day 3	<0.01	0.037	<0.01	0.027	<0.01	0.050
Day 4	<0.01	0.048	<0.01	0.037	<0.01	0.042
Day 5	0.01	0.056	0.01	0.053	0.01	0.079
Day 6	0.01	0.056	0.01	0.050	0.01	0.095
Day 7	0.01	0.055	0.01	0.059	0.01	0.102
Total eggs	0.03		0.03		0.03	
Blood	<0.01	0.042	<0.01	0.042	0.03	1.23
Liver	0.02	0.341	0.02	0.281	0.10	1.569
Kidney	<0.01	9.127	<0.01	0.126	0.01	0.552
Dark muscle	<0.01	0.011	<0.01	0.009	0.01	0.027
Light muscle	<0.01	0.008	<0.01	0.007	<0.01	0.014
Abdominal fat	<0.01	0.072	<0.01	0.044	<0.01	0.042
Skin	<0.01	0.052	<0.01	0.042	<0.01	0.048
Excreta	84		93		89	
Total incl eggs	84		93		89	

All tissue samples with residues greater than 0.01 mg/kg, as well as the day 7 eggs, were extracted and partitioned prior to metabolite identification and characterization by HPLC, TLC, proton NMR or LC-MS. Additional residues were extracted using enzyme hydrolysis and/or acid and base hydrolysis to release either conjugated or solid bound residues. Unknown metabolites were isolated and purified by both reversed-phase HPLC and normal phase TLC, followed by LC-MS, UV and radiometric detection. The following reference standards were used for co-chromatography: methoxyfenozide; RH-117,236 (metabolite B); RH-131,154 (metabolite C); metabolite E_1; metabolite E_2; metabolite F; RH-141,518 (metabolite G); metabolite H; RH-141,519 (metabolite I); RH-131,364; RH-131,157; RH-131,156; RH-123,790; lactose; and ^{14}C-*iso*-propanol.

Metabolites A, D, J and K were characterized based on retention time but no further attempts to identify them were reported. In the goat metabolism study, the component labelled metabolite E was identified as consisting of metabolites E_1, E_2, or both; however, in the poultry metabolism study metabolite E_2 was the only component of metabolite E identified.

Parent methoxyfenozide and the following metabolites were isolated and identified from the various matrices: RH-117236 (A-ring phenol or demethylated parent); RH-131154 (B-ring carboxylic acid); RH-141511 (A-ring phenol, B-ring mono-alcohol); and RH-141518 (glucuronide conjugate of A-ring phenol). Other metabolites were isolated but remained unidentified. Table 13 summarizes the levels of each metabolite identified in the various matrices. Parent compound was the major component in fat, muscle, and skin. In liver, kidney and eggs, in addition to the parent, the metabolite RH-141518 represented a major component of the residue. Identifications and characterizations are summarized in Table 6. The proposed metabolic pathway of methoxyfenozide in poultry is shown in Figure 2.

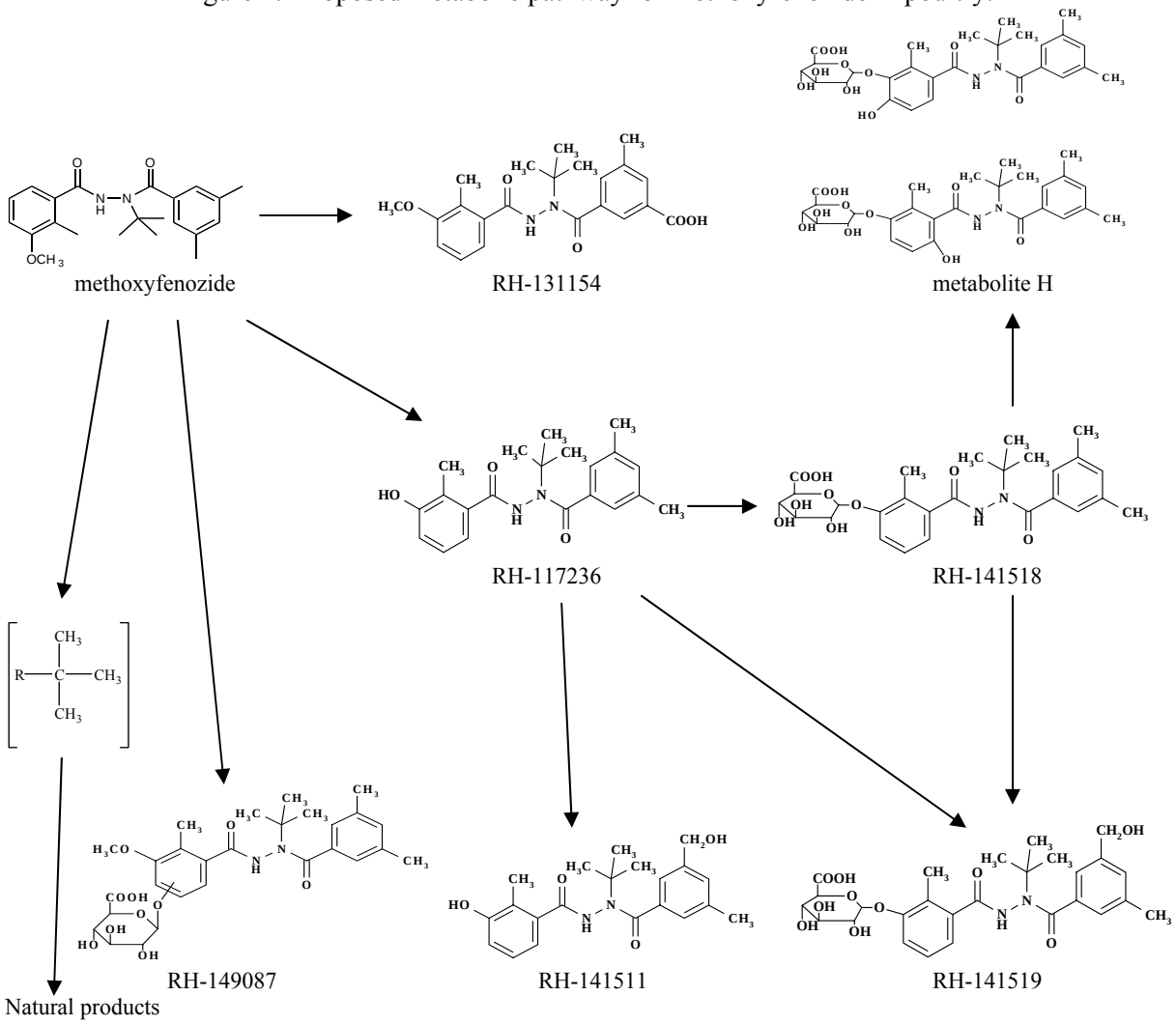
Table 6. Characterization of extractable residues in poultry eggs and tissues following treatment with ^{14}C-methoxyfenozide.

Matrix		Liver	Kidney	Fat	Skin	Dark meat	Light meat	Egg
Label: A-ring								
Methoxyfenozide	% TRR	1.8	0.60	44	23	22		2.20
	mg/kg	0.006	<0.001	0.032	0.012	0.003		0.001
RH-117236	% TRR	6.3	4.3	11	8.4	6.0		5.1
	mg/kg	0.022	0.005	0.008	0.004	0.001		0.003
RH-131154	% TRR	1.6	(-)	(-)	3.2	2.0		4.0
	mg/kg	0.005	(-)	(-)	0.002	<0.001		0.002
Metabolite E	% TRR	5.0	5.3	11	13	4.3		13
	mg/kg	0.017	0.007	0.008	0.007	<0.001		0.007
Metabolite F	% TRR	0.55	(-)	2.1	11	(-)		4.0
	mg/kg	0.002	(-)	0.001	0.006	(-)		0.002
RH-141518	% TRR	15.	36	9.7	7.8	(-)		26
	mg/kg	0.052	0.046	0.007	0.004	(-)		0.014
Metabolite H	% TRR	2.6	(-)	9.5	(-)	(-)		(-)
	mg/kg	0.009	(-)	0.007	(-)	(-)		(-)
RH-141519	% TRR	5.6	28	(-)	(-)	(-)		15
	mg/kg	0.019	0.036	(-)	(-)	(-)		0.007
Total Identified	% TRR	38	74	87	67	34		69
	mg/kg	0.13	0.09	0.063	0.035	0.004		0.038
Unknowns [1/]	% TRR	55.	3.4	9.8	23	55		17
	mg/kg	0.19	0.004	0.007	0.012	0.006		0.009
Total	% TRR	93	92	97	91	89		87
Label: B-ring								
Methoxyfenozide	% TRR	(-)	0.31	55	22	11		4.9
	mg/kg	(-)	<0.001	0.024	0.009	0.001		0.003
RH-117236	% TRR	6.2	3.2	9.3	4.1	2.3		6.8
	mg/kg	0.017	0.004	0.004	0.002	<0.001		0.004
RH-131154	% TRR	(-)	(-)	(-)	4.8	(-)		5.2
	mg/kg	(-)	(-)	(-)	0.002	(-)		0.003
Metabolite E	% TRR	4.0	3.6	11	12	1.4		16
	mg/kg	0.011	0.005	0.004	0.005	<0.001		0.008
Metabolite F	% TRR	(-)	(-)	2.4	9.1	(-)		(-)
	mg/kg	(-)	(-)	0.001	0.004	(-)		(-)
RH-141518	% TRR	19	33	3.2	6.1	(-)		28
	mg/kg	0.054	0.041	0.002	0.003	(-)		0.015
Metabolite H	% TRR	(-)	2.0	6.7	(-)	(-)		(-)
	mg/kg	(-)	0.003	0.003	(-)	(-)		(-)
RH-141519	% TRR	8.2	22	(-)	(-)	(-)		16
	mg/kg	0.023	0.028	(-)	(-)	(-)		0.009
Total Identified	% TRR	37	64	87	59	14		76
	mg/kg	0.11	0.08	0.038	0.025	0.001		0.045
Unknowns [1/]	% TRR	52	29	6.9	34	74		13
	mg/kg	0.15	0.036	0.003	0.014	0.007		0.007
Total	% TRR	89	93	94	93	88		89

Matrix		Liver	Kidney	Fat	Skin	Dark meat	Light meat	Egg
Label: *t*-butyl								
Methoxyfenozide	% TRR	0.26	(-)	40	18	8.0	2.1	2.8
	mg/kg	0.004	(-)	0.017	0.009	0.002	<0.001	0.003
RH-117236	% TRR	2.8	2.4	5.7	6.6	1.5	2.8	5.1
	mg/kg	0.044	0.013	0.002	0.003	<0.001	<0.001	0.005
RH-131154	% TRR	0.31	(-)	(-)	3.0	(-)	1.4	2.9
	mg/kg	0.005	(-)	(-)	0.001	(-)	<0.001	0.003
Metabolite E	% TRR	1.5	1.0	2.1	9.5	0.46	1.7	8.2
	mg/kg	0.023	0.006	0.001	0.004	<0.001	<0.001	0.008
Metabolite F	% TRR	(-)	(-)	(-)	3.1	8.9	6.3	0.54
	mg/kg	(-)	(-)	(-)	0.002	0.002	0.001	0.001
RH-141518	% TRR	4.2	7.6	1.8	16	31	10	30
	mg/kg	0.066	0.042	0.001	0.008	0.009	0.001	0.032
Metabolite H	% TRR	(-)	0.51	(-)	(-)	(-)	1.1	6.0
	mg/kg	(-)	0.003	(-)	(-)	(-)	<0.001	0.006
RH-141519	% TRR	3.0	5.9	(-)	2.8	10	2.2	12.
	mg/kg	0.047	0.033	(-)	0.011	0.003	<0.001	0.013
Total Identified	% TRR	12	17	49	59	60	27	68
	mg/kg	0.19	0.096	0.021	0.028	0.016	0.004	0.069
Unknowns [1/]	% TRR	85	72	41	35	21	21	23
	mg/kg	1.3	0.40	0.017	0.017	0.005	0.002	0.023
Total	% TRR	97	89	90	94	81	48	91

[1] Unknowns included other solvent-released and partly-characterized metabolites.
(-) indicates "not observed".

Figure 2. Proposed metabolic pathway for methoxyfenozide in poultry.



Plant metabolism

The Meeting received information on the metabolism of methoxyfenozide in apples, cotton, grapes and rice, following treatment with radiolabelled material.

Apples

A single apple tree (cv. Red Delicious)was treated with two applications of methoxyfenozide (Lin, 1995, report No. 34-95-02; Carpenter, 1999a, report No. 34-99-25). The radioactive test substance, a mixture of unlabelled methoxyfenozide and [methoxyphenyl-^{14}C]methoxyfenoxide was formulated in methanol/water (65:35 v/v). Each application was made at a nominal rate of 1 lb ai/acre (1.12 kg ai/ha) using methoxyfenozide labelled with ^{14}C in the A ring. The total application was 2 lb ai/acre (2.2 kg ai/ha), which was about the USA label rate. The decay profiles of methoxyfenozide on apple fruit and foliage were studied by monitoring total residues after the second application in both apple fruit and foliage, during a period of 36 and 69 days, respectively. As shown in Table 7, the total radioactive residues (TRR) in apple fruit ranged from 0.23-3.4 mg/kg, while the TRR in foliage ranged from 43-410 mg/kg. The half-lives for total residue were estimated to be 12 days on apple fruit and 23 days on foliage.

Table 7. Total radioactive residues (TRR) in apples and apple foliage samples (Lin, 1995; Carpenter, 1999a).

Samples	Days after treatment [1/]	TRR in control (dpm/g)	TRR in treated (dpm/g)	(mg/kg)
Apple fruit	0	0	27,632	1.6
	7	0	60,353	3.4
	14	0	4,062 [2/]	0.23
	36	0	4,818 [2/]	0.28
Half-life [2/]				12 ± 9 days
Apple foliage	0	0	5,956,314	340
	7	0	7,199,189	410
	14	0	1,488,883	85
	36	0	1,215,556	69
	69		757,535	43
Half-life [2/]				23 ± 8 days

[1/] Calculated with day of 2nd (i.e. final) spray defined as day 0.
[2/] Calculated with mg/kg data at day 0 and thereafter. Kinetics were assumed to be pseudo-first order.

Apples harvested at 14 days and 36 days after the second application were analyzed to determine the nature of the residues present. The apple samples were pulverized with dry ice, followed by extraction and partition. The total recovery of each analytical procedure and the distribution of TRR in each fraction were determined via liquid scintillation counting or combustion radio-assay. From apple samples harvested at 36 days after treatment, 93% (0.27 mg/kg) of total residue was readily extractable into an organic (dichloromethane) fraction, 4.2% TRR (0.012 mg/kg) remained in the aqueous fraction and 2.8% TTR (0.008 mg/kg) remained in the pomace. Table 8 summarizes the results. Since the only fraction that contained greater than 0.01 mg/kg or greater than 10% TRR was the dichloromethane fraction, only that fraction was subjected to further residue analysis.

Table 8. Summary of extraction of radioactivity from apple fruit treated with ^{14}C-methoxyfenozide (Lin, 1995; Carpenter, 1999a).

Sample	Fraction/sub-fraction	dpm or mg/kg	% TRR	Components identified
Fortified control, (399,251 dpm treated)	Acetonitrile/water	386,116 dpm	100	Methoxyfenozide
	- dichloromethane	352,946 dpm	100	
	- aqueous	0 dpm	0	
	Pomace	0 dpm	0	
	Total	-	100	
Apple, 14 DAT (0.232 mg/kg by combustion)	Acetonitrile/water	0.290 mg/kg	96.9	Methoxyfenozide, RH-131364, RH-131157, and Unknown A
	- dichloromethane	0.280 mg/kg	93.5	
	- aqueous	0.010 mg/kg	3.4	
	Pomace	0.009 mg/kg	3.1	
	Total	0.299 mg/kg	100	

Sample	Fraction/sub-fraction	dpm or mg/kg	% TRR	Components identified
Apple, 36 DAT (harvest) (0.275 mg/kg by combustion)	Acetonitrile/water	0.280 mg/kg	97.2	Methoxyfenozide, RH-131364, RH-131157, and Unknown A
	- dichloromethane	0.268 mg/kg	93.0	
	- aqueous	0.012 mg/kg	4.2	
	Pomace	0.008 mg/kg	2.8	
	Total	0.288 mg/kg	100	

DAT = days after treatment.

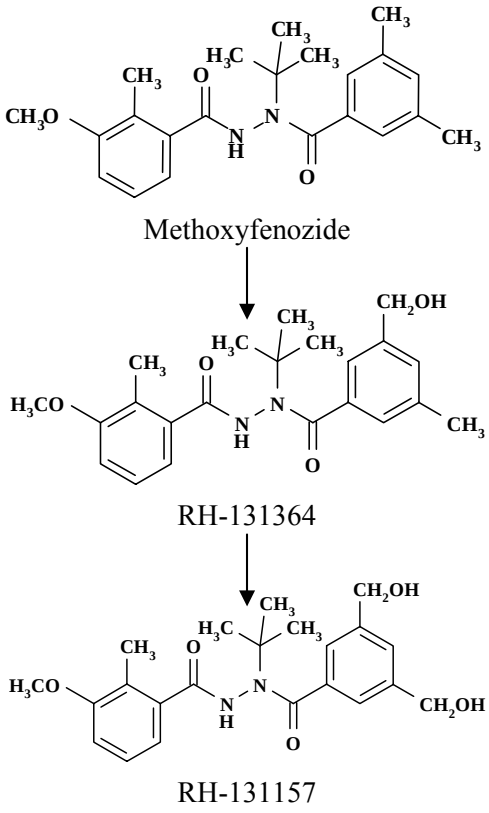
Normal-phase radio-TLC was used to identify the components detected, by matching them with available unlabelled standards. A phosphor-imager was used to determine the relative concentration of each radio-visible component. For the apple sample at harvest (36 DAT), 91% (0.26 mg/kg) of TRR was determined to be the parent compound, 1.4% TRR (0.004 mg/kg) was the B-ring mono-alcohol (RH-131364) and 0.11% TRR (0.0003 mg/kg) was B-ring di-alcohol (RH-131157). A residue of 0.42% TRR remained unidentified. Table 9 summarizes the results.

Table 9. Summary of radio-TLC analysis of organic solvent-soluble residues in apple fruit (Lin, 1995; Carpenter, 1999a).

Component	Rf	Apple, 14 DAT (average of 5 replicates)			Apple at harvest (average of 3 replicates)			Methoxyfenozide standard purity % of total
		% of dichloro-methane fraction	% TRR	mg/kg	% of dichloro-methane fraction	% TRR	mg/kg	
Methoxyfenozide	0.91	98	91	0.27	98	91	0.26	99.75
RH-131364	0.81	1.5	1.4	0.004	1.5	1.4	0.004	0.18
RH-131157	0.54	0.09	0.08	0.0003	0.12	0.11	0.0003	0
Unknown A	0.21	0.42	0.39	0.001	0.45	0.42	0.001	0
Others	NA	0.36	0.34	0.001	0.23	0.21	0.001	0.07
Total		100	94	0.280	100	93	0.27	100.00

Methoxyfenozide was not extensively metabolized in apples. A possible metabolic pathway is illustrated in Figure 3.

Figure 3. Proposed metabolic pathway for methoxyfenozide in apples.



Cotton

Methoxyfenozide, labelled with ^{14}C on the *t*-butyl, A-ring or B-ring, was applied twice to cotton plants at a rate of 0.96 lb ai/acre (1.08 kg ai/ha) per application or a total of 1.9 lb ai/acre (2.2 kg ai/ha) (Comezoglu, 1996, report No. 34-95-207). The first application was made 90 days after planting and the second application was 32 days later. Crop harvest occurred 21 days after the second application.

Samples of immature whole (above ground) cotton plants and bolls were collected from each plot immediately after the first application, immediately before and after the second application and on days 7 and 14 after the second application. Mature bolls and plant samples were harvested 21 days after the second application. Soil samples were collected for characterization and checking background radioactivity before the experiment and at harvest of the mature cotton, 21 days after the last application. Each cotton seed sample was mechanically ginned, to yield seed and lint. Grinding ginned cotton seeds generated additional lint, which was separated from seed kernels with a coarse screen. The coarsely ground seeds were then cryogenically ground to a powder. These processes yielded two seed samples: the lint generated during processing (seed/lint samples) and the processed seed part (seed/meal samples). The immature and mature plant samples were homogenized. All harvested plant and soil samples were frozen and transported frozen to the laboratory, where they were maintained frozen until analysis.

Sub-samples from seed/lint and seed/meal samples were assayed by combustion analysis, followed by liquid scintillation counting. Because seed/lint samples were derived from the original ginned cotton seed and contained a portion of the seed coat along with the lint, they were considered to contribute to the overall TRR of the whole intact seed. TRR values, therefore, for the whole seed were calculated from those obtained for seed/meal and seed/lint, based on each fraction's weight contribution to the whole seed total biomass. A summary of the TRR levels is shown in Table 10.

Table 10. Summary of total radioactive residues (TRR) levels in cotton seed [1/] (Comezoglu, 1996).

Label	Seed/meal TRR, mg/kg	Seed/lint TRR, mg/kg	Whole seed TRR (calculated), mg/kg
t-butyl	0.054	0.11	0.080
A-Ring	0.072	0.089	0.081
B-Ring	0.057	0.16	0.11

[1/] TRR levels in control samples were all <0.003 mg/kg, 21 days after treatment.

TRR levels in the plant samples are summarized in Table 11.

Table 11. Summary of total radioactive residues (TRR) in cotton plants (Comezoglu, 1996).

Matrix	*t*-butyl labelled, mg/kg	A-ring labelled, mg/kg	B-ring labelled, mg/kg
Post-application # 1, (-31 DAT)	53	87	106
Pre-application #2, 0 DAT	13	14	17
Post-application #2, 0 DAT	89	95	130
7 DAT	60	72	87
14 DAT	43	49	69
Harvest, 21 DAT	13	17	17

DAT = days after second treatment.
TRR levels in control samples were all <0.01 mg/kg.

Treated seed/meal samples were subjected to extraction with various solvents. Homogenized seed/meal samples were extracted by blending with hexane, followed by methanol and then methanol/0.1 N HCl (3:1). The extraction procedure separated the samples into non-polar (hexane) polar (methanol), aqueous-soluble (methanol/0.1 N HCl) and bound residue fractions. Partitioning the hexane fraction with acetonitrile yielded an oil-containing hexane fraction and an acetonitrile fraction. Depending on TRR levels, these fractions were fractionated further. Methanol and methanol/0.1 N HCl extracts were partitioned with chloroform. Seed/lint samples were extracted with methanol, yielding methanol-soluble and bound residue fractions. The post-extraction solids (PES) contained 3.2–10% TRR in the case of hulled kernels and 10–25% TRR in the case of lint/hulls.

Seed/meal acetonitrile extracts (produced by partition with the hexane fraction) and seed/lint methanol extracts (from methanol fraction) contained the majority of the whole-seed TRR. Amounts in acetonitrile and methanol fractions were: 16% and 44% of the whole seed TRR in samples from plants treated with the *t*-butyl label; 9.1% and 31% in samples from plants treated with the A-ring label; and 6.2% and 45% in samples from plants treated with the B-ring label, respectively. TRR levels in the chloroform fractions for all labels ranged from 2.7-6.6% of the whole seed TRR. The post-extraction solids (PES) from the seed/meal samples contributed 4.9-11% of the whole seed TRR, whereas levels in seed/lint PES ranged from 20-27%.

Bound residues in the seed/meal (A-ring) samples were subjected to cellulose enzyme digestion, followed by HCl (0.1N) hydrolysis. Cellulase enzyme digestion released 2.8% of the whole seed TRR (0.002 mg/kg). Acid hydrolysis released 5.0% of the whole seed TRR (0.004 mg/kg). The non-released TRR remaining in the PES amounted to 3.2% (0.003 mg/kg).

The PES fraction from seed/lint (A-ring) samples was digested first with 1N HCl then with 6N HCl. The released radioactivity from hydrolysis corresponded to 10% (0.008 mg/kg) of the whole seed TRR with 1N HCl hydrolysis and 6.0% (0.005 mg/kg) with the 6N HCl hydrolysis.

The fractions were analyzed by reversed-phase HPLC and/or normal-phase thin layer chromatography (TLC). The parent compound, ^{14}C-methoxyfenozide was the major ^{14}C-residue observed from TLC and/or HPLC analysis of cotton seed/meal and seed/lint extracts. ^{14}C-methoxyfenozide corresponded to 67%, 46%, and 57% of the whole seed TRR in *t*-butyl A-ring, and B-ring cotton seed samples, respectively. The TLC and HPLC analysis of methanol and hexane fractions from one immature plant also showed the presence of the parent compound (>92% of plant TRR). No further identifications were reported.

Grapes

A metabolism study was conducted on established Concord grape vines in Pennsylvania, USA (Carpenter, 1997, report No. 34-97-93). The test substance was a mixture of non-labelled, ^{13}C-*t*-butyl labelled, and ^{14}C *t*-butyl labelled methoxyfenozide. Two applications, each of approximately 1.0 lb ai/acre (1.12 kg ai/ha), were applied to a single vine at an interval of 27 days. The total application was approximately 2.0 lbs ai/acre/year (2.2 kg ai/ha/year), twice the USA label rate.

The decay profiles of methoxyfenozide on grape foliage and fruit were studied by monitoring the total residues on both. Grapes were monitored for a period of 27 days after the second application and foliage was monitored for a period of 59 days after application. The TRR in grapes ranged from 0.54-2.6 mg/kg, while that on foliage ranged from 18-240 mg/kg. Table 11 provides the TRR in grapes and foliage samples. The half-lives for the total residue were 13-21 days on grapes and 11-26 days on foliage.

Table 12 Total radioactive residues (TRR) in grapes and foliage samples (Carpenter, 1997).

Samples	Days after treatment [1/]	TRR control, (dpm/g)	TRR treated, (mg/kg)
Grape	0	0	1.9 [2/]
	7	0	2.6
	14	0	1.3
	21	0	0.54
Harvest	27	0	0.71 [3/]
Foliage	0	0	240
	10	0	100
	14	0	93
	21	0	83
Harvest	27	0	110 [4/]
Post harvest	59	0	37

[1/] Calculated with day of 2nd spray defined as day 0.
[2/] Average of two sets of combustion data.
[3/] Average from combustion of 15 samples.
[4/] Harvest day result is the average from combustion of 8 samples.

A harvested grape sample, at 27 days after the last application, was analyzed to determine the nature of the residues present. The grapes were pulverized in the presence of dry ice and a sample

was extracted and purified. The total recovery of each analytical procedure and the distribution of total radioactive residues (TRR) in each fraction were determined by liquid scintillation counting or combustion radio-assay. In the harvest grape sample, 97% (0.71 mg/kg) of the total radioactive residue was present in the organic solvent fraction and 2.1% (0.016 mg/kg) remained in the post-extraction solids (PES).

The extractable fraction was the only fraction to be further analyzed (Table 13). This fraction from the harvested grapes contained 81.% TRR as parent methoxyfenozide and two other minor components, which were identified as the glucose conjugate of RH-117236 (A-ring phenol, 3.6%) and RH-131364 (B-ring mono-alcohol, <2.3%). There were also small amounts of materials tentatively identified as the glucose conjugate of RH-131364 (2.3%), or a similar alcohol, and RH-131154 (B-ring carboxylic acid, <2.3%).

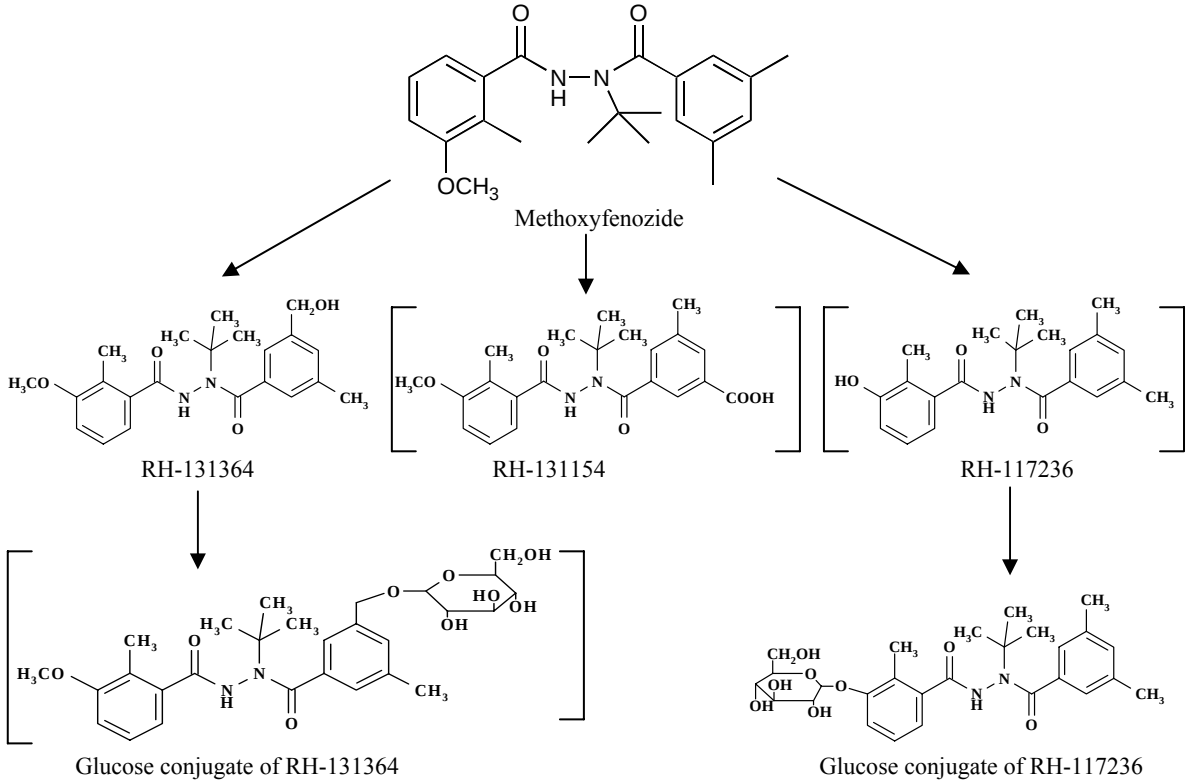
The identity of the parent compound in grape samples was confirmed by TLC and HPLC. The identification of the glucose conjugates of RH-117236 was confirmed by cleaving with glucosidase and comparing the chromatographic behaviour of the resulting material with the unlabelled standard, using normal phase TLC and HPLC. Standards of six different compounds were used to facilitate identification of the metabolites.

Table 13. Summary of identified components of residues in harvested grapes (Carpenter, 1997).

Component	% TRR	mg/kg
Methoxyfenozide (RH-2485)	81	0.60
RH-131364	<2.3	<0.017
Glucose conjugate of RH-117236	3.6	0.027
RH-131154	<2.3	<0.017
Glucose conjugate (may be RH-131364 or similar alcohol)	2.3	0.017

The proposed metabolic pathway is shown in Figure 4.

Figure 4. Proposed metabolic pathway for RH-2485 in treated grapes.



The metabolism of methoxyfenozide in grape leaves was studied (Bergin,, 1998a, Report No. 34-98-135). Samples used in this study were generated in the previously-described grape metabolism study and had been macerated and stored frozen for almost 3 years. The TRR of the grape leaf sample

harvested 27 days after the final application of methoxyfenozide, determined by combustion radioassay, was 108 mg/kg.

The grape leaf samples were extracted three times with methanol containing 1% acetic acid and the resulting extracts were combined. The methanolic extract was filtered under vacuum and partitioned between an aqueous salt solution and dichloromethane. The extract was then evaporated to a smaller volume and purified using a C-18 SepPak cartridge. Extracted residue accounted for 93 % of the TRR, while 2.6% of the TRR remained in the post-extraction solids (PES).

Thin layer chromatography was used to identify the components by comparing Rfs with corresponding available analytical standards. The standards used were based on previous results of crop metabolism studies with methoxyfenozide. Analysis of the extracts showed that the parent compound accounted for 85% of the total radioactive residue. Minor amounts of the B-ring carboxylic acid (RH-131154) and B-ring mono-alcohol (RH-131364) were also identified (<1%). The percentages of radioactivity in all remaining fractions were all much less than 10% and these fractions were not analyzed further.

Rice

The metabolism of methoxyfenozide in rice plants was studied using four confined rice plots, inside separate screenhouse enclosures in the United States (Carpenter, 1999b and 1999c, reports No. 34-98-156 and 34-99-34). Three test materials, "A-label", "B-label", and "*t*-butyl label" were used in the trials. Each test material was a mixture of ^{12}C, ^{13}C-labeled, and ^{14}C-labeled methoxyfenozide. The location of the label was as specified below:

"A-ring" (2-methyl, 3-methoxybenzoyl ring):
- ^{14}C, uniformly labelled in the 2-methyl, 3-methoxybenzoyl ring;
- ^{13}C at the carbonyl carbon adjacent to the 2-methyl, 3-methoxybenzoyl ring.

"B-ring" (3,5-dimethylbenzoyl ring):
- ^{14}C uniformly labelled in the 3,5-dimethylbenzoyl ring;
- ^{13}C in a methyl group directly attached to the 3,5-dimethylbenzoyl ring.

"*t*-butyl" (*t*-butyl group):
- ^{14}C at the tertiary carbon of the *t*-butyl group;
- ^{13}C at the tertiary carbon of the *t*-butyl group.

Rice seed was planted in the greenhouse and transplanted at the two-leaf stage to the study plots in screenhouses, nine days later. The paddy rice crops were grown according to appropriate agronomic practices. Each of the treated plots received two foliar applications of methoxyfenozide. The first treatment was applied at the pre-flagleaf (76-84 cm in height) stage, 70 days after planting. The second application was applied to the crops at the post-flowering stage (84-91 cm in height), with well developed panicles, 107 days after planting. The B-ring and *t*-butyl test materials were each applied at a rate of approximately 0.6 kg ai/ha in both applications, equivalent to 10 times the recommended USA application rate. The A-ring material was applied at rates of 0.62 kg ai/ha and 0.31 kg ai/ha in two applications as aqueous foliar sprays, at spray volumes of approximately 930 l/ha.

Immature rice plant samples, cut approximately 5 cm above soil level, were collected from each plot, immediately before and after the second application and at 14 and 31 days later. Each single sample consisted of 5 randomly selected tillers. These samples were processed with dry ice into fine powder, bagged, weighed, and stored frozen for up to 10 months, until aliquots were combusted for the determination of total radioactive residues.

Mature rice samples were harvested 62 days after the second application to the treated plots. A mature foliage sample, similar to those collected at the immature harvest, was collected at the final harvest, processed as for immature rice plant samples and stored frozen up to 10 months until samples underwent combustion for the determination of total radioactive residues.

The remaining plants in each plot were then harvested by cutting mature panicles from the straw with scissors or razor blades. The panicles were placed into bags left open to prevent moisture accumulation from grain respiration/drying. These panicle samples were then dried at room temperature and the dried panicles were mechanically husked to produce brown rice and chaff.

The mature straw was cut 20 cm above the soil level in treated crops and 7.5 cm above soil level in the controls. The straw samples were bagged, transported at ambient temperature to the laboratory, where they were kept in frozen storage until analysis. Processed panicles (brown rice and chaff) and straw samples were placed in separate, labelled bags and stored frozen for up to 10 months until analysis.

Levels of total radioactive residues (TRR) in immature and mature rice samples were determined by combusting homogenized samples, followed by liquid scintillation counting (LSC). The TRR of immature and mature rice samples was determined in order to show the distribution and decay profile of methoxyfenozide on treated rice plants. As shown in Table 14, the TRR in immature rice plants ranged from 5.8-8.5 mg/kg in the case of the A label material, 5.6-14 mg/kg in the case of the B label test material, and 4.7-13 mg/kg in the plants treated with the *t*-butyl test material. The TRRs of mature rice samples, harvested 62 days after the second application, are summarized in Table 15. Decay profiles, constructed with TRR data from post-2nd spray to harvest, showed half-lives on rice in the range of 120-430 days.

Table 14. Total radioactive residues (TRR) in immature and harvest rice plant samples (Carpenter, 1999b and 1999c).

Sampling period	DAT [1/] (days)	A-ring label, mg/kg	B-ring label, mg/kg	*t*-butyl label, mg/kg
Post-1st spray	-37	8.5	9.2	10
Pre-2nd spray	0	5.8	5.6	4.7
Post-2nd spray	0	7.2	14	13
14 DAT	14	7.5	13	10
31 DAT	31	7.3	10	11
Harvest (62 DAT)	62	6.6	10	9.4

[1/] DATs calculated with day of 2nd spray defined as Day 0.

Table 15. Total radioactive residues (TRR) in mature rice samples (Carpenter, 1999b and 1999c).

Sampling period	DAT [1/] (days)	A-ring label, mg/kg	B-ring label, mg/kg	*t*-butyl label, mg/kg
Harvest straw	62	21	44	37
Harvest grain [2/]	62	0.52	0.71	0.56

[1/] DATs calculated with day of 2nd spray defined as Day 0.
[2/] Brown rice (without husk).

Rice straw samples at harvest were analyzed to determine the nature of the residues present. The straw was comminuted and homogenized in the presence of dry ice and samples were extracted four times with a methanol/water mixture (80:20 by volume). After combining the extracts and concentrating to a smaller volume, the TRRs of the combined extract and of the post-extraction solids (PES) were measured to determine extraction recovery and ^{14}C distribution. Extraction recoveries ranged from 95-97%. The combined extract contained 88-91% of the radioactivity. The PES fraction contained 5.6-7.6 % of the TRR and was not analyzed further.

Normal phase and reversed phase TLC were used to identify and quantify the detectable residues, by matching them with suitable unlabelled standards. A phosphor-imager was used to visualize the radiolabelled components. The TLC plates were eluted with chloroform/methanol/ammonium hydroxide (90:9:1) and then divided into 7 bands. LC-MS was used to confirm the presence of the major component, methoxyfenozide. Table 16 summarizes the residue components identified in rice straw samples.

Table 16. Residues identified in rice straw samples (Carpenter, 1999b and 1999c).

Residue component	A-ring label		B-ring label		*t*-butyl label	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
RH-112485	65	13	67	29	69	25
RH-131364	0.9	0.18	1.2	0.51	1.4	0.51
RH-117236	2.7	0.56	2.9	1.29	2.8	1.07
RH-141511	2.3	0.47	2.1	0.93	2.3	0.85
RH-131154	1.6	0.34	1.2	0.55	1.3	0.50
Glucose conjugate of RH-117236	2.4	0.50	1.8	0.78	1.5	0.55
Total TRR identified	75	15	76	33	78	29

Samples of rice grain at harvest were analyzed to determine the nature of the residues present. After removal of husks, the grain was soaked in a mixture of methanol/water (80:20 v/v) and then homogenized. Seven extractions were then made: 2 with the methanol/water, followed by 2 with methanol, then 2 with acetonitrile/water/HCl (70:29:1 by volume) and finally 1 extraction with acetonitrile. All extracts were combined and concentrated to a smaller volume. The TRRs of the combined extracts and the PES were measured to determine the extraction recovery and ^{14}C distribution. The total concentration (mg/kg) of the sample analyzed was based on the sum of the concentrations of the combined extract and unextracted residue (PES). The data obtained by combustion of the grain samples could not be used, due to the variability of the results. The solvent extract contained an average of 80-88% of the ^{14}C, with 12-20% remaining in the PES.

The concentrated combined extracts of grain from rice treated with each label were diluted with water and the pH checked to be in the range 1-2. Each extract was then partitioned twice with dichloromethane and the dichloromethane layers for each label were combined. Aliquots were taken from the dichloromethane and aqueous layers for liquid scintillation counting, after each was evaporated to near dryness. Recoveries ranged from 102-108.%. The dichloromethane layers were then analyzed by TLC and LC-MS.

The concentrated aqueous fraction from partitioning was cleaned up using a C-18 Sep Pak cartridge, eluting with methanol. After clean-up, the total recovery of A-ring label was 99%, with 90% of the radioactivity in the methanol fraction. Of the B-ring label, the total recovery was 85%, with 80% of the radioactivity in the methanol fraction. Of the *t*-butyl label, recovery was 100%, with 94% of the radioactivity in the methanol fraction. The methanol eluates were concentrated to a small volume and analyzed further by TLC.

The PES fraction of the rice grain was analyzed for bound residues: by extraction with sodium acetate buffer; by successive incubation with α-amyloglucosidase and pronase enzymes; and by cellulose extraction, using sulfuric acid; which showed that radiolabelled material had been metabolically incorporated into various natural products (Table 17). All of the post-extraction solids remaining after these treatments contained less than 10% TRR and 0.05 mg/kg and no further work was done on these materials to identify the radiolabelled residues.

Table 17. Distribution of total radioactive residues in rice grain after extraction and partitioning with dichloromethane (Carpenter, 1999b and 1999c).

Distribution	A-ring label		B-ring label		*t*-butyl label	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
After initial extraction						
Extracted	80	0.42	86	0.61	88	0.50
Unextracted (PES)	20	0.10	14	0.10	12	0.069
Total	100	0.52	100	0.71	100	0.56
After partitioning						
Dichloromethane	89	0.37	90	0.55	88	0.44
Aqueous	19	0.082	16	0.098	15	0.074
Total	110	0.46	110	0.65	103	0.51
After digestion of the PES (bound residues) with:		0.10		0.10		0.069
Sodium acetate buffer	0.94	0.005	0.69	0.005	0.57	0.003
A-glucosidase	3.9	0.020	2.0	0.014	0.87	0.005
A-glucosidase -II	3.4	0.018	1.3	0.009	0.62	0.004
Pronase	2.6	0.014	2.2	0.016	1.6	0.009
Sulfuric acid (cellulose)	7.7	0.040	(-)	(-)	(-)	(-)
Total	18	0.097	6.18	0.044	3.7	0.021

(-) = not determined.

Detected components of the residue were identified using normal phase and reversed phase TLC, by matching them with suitable unlabelled standards. UV detection was used to visualize the spots of all cold standards. Quantitative analysis was by radio-TLC, a phosphor-imager being used to determine the amount of material present. LC-MS was used to confirm the presence of the major component, the parent. The parent and the metabolites in rice grain identified in the various extracts are summarized in Table 18.

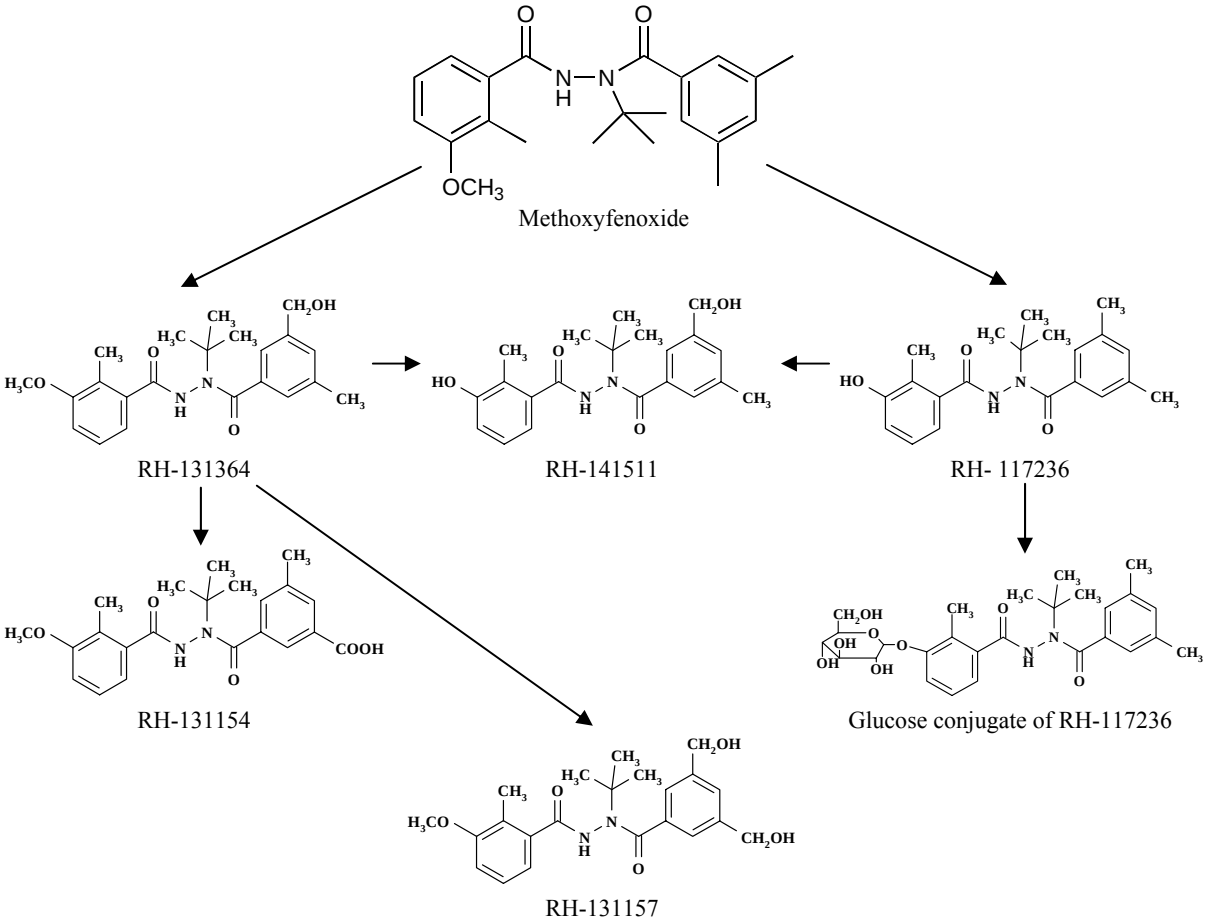
Table 18. Residues identified in rice grain samples (Carpenter, 1999b and 1999c).

Residue	A-ring label		B-ring label		*t*-butyl label	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
RH-112485 [1/]	52	0.27	59	0.42	57	0.32
RH-131364 [1/]	1.1	0.006	4.1	0.029	3.1	0.017
RH-117236 [1/]	7.5	0.039	5.8	0.041	3.2	0.018
RH-131154 [1/]	2.9	0.015	1.8	0.013	1.6	0.009
RH-131157 [1/]	0.4	0.002	0.7	0.005	0.36	0.002
Glucose conjugate of RH-117236 [2/]	2.3	0.012	2.1	0.015	1.8	0.010
RH-117236 [3/]	(-) [4/]	(-) [4/]	3.2	0.023	(-) [4/]	(-) [4/]
Total identified	66		77		67	

[1/] From the dichloromethane layer.
[2/] From the aqueous layer.
[3/] From the aqueous layer after hydrolysis (residue may have been the glucose conjugate or some other sugar conjugate).
[4/] (-) = not observed.

The proposed metabolic pathway of methoxyfenozide in rice is illustrated in Figure 5.

Figure 5. Proposed metabolic pathway for methoxyfenoxide in treated rice.



Environmental fate in soil

The Meeting received information on the behaviour and fate of methoxyfenozide in aerobic soils and in a range of rotational crops.

Degradation in aerobic soils

The aerobic soil degradation of ^{14}C-methoxyfenozide (A-ring, B-ring or *t*-butyl label) in four characterized soils was investigated using a California loam soil (Reynolds, 1996, report No. 34-96-36), an Ohio loamy sand soil (Smalley, 1997a, report No. 34-96-188), a Georgia loamy sand soil and a Texas sandy clay loam soil (Smalley, 1997b, report No.34-97-30). The soils were treated at 1 µg ai/g dry soil, equivalent to a maximum annual application rate of 0.75 a.i. kg/ha. The studies were

conducted at 25ºC and the soil moisture was maintained at 75% of field capacity. The microbial biomass was determined before and during the study. Duplicate samples of control and treated soils were collected 0-365 days post-treatment and analyzed by LSC, HPLC and TLC. Volatile organics and CO_2 were also collected during the study.

Methoxyfenozide degraded slowly in soil, with 59-75% of the dosed radioactivity remaining as the parent after 365 days. The calculated half-life values (1st order) for methoxyfenozide over the course of the study were: 570 days in California loam soil, 1100 days in Ohio loamy sand soil, 340 days in Georgia loamy sand soil and 720 days in Texas sandy clay loam soil.

The amount of radioactivity extracted from the soil decreased during the test period and accounted for 60-78% of the applied radioactivity after 365 days. The majority of the extracted radioactivity was methoxyfenozide. A major metabolite, reaching a maximum of 3.2% of the applied dose, was identified as RH-131154, a B-ring carboxylic acid. Trace levels of five other metabolites were observed, each of which did not exceed 1.2% of the total applied radioactivity. At day 365, up to 5.5% of the applied radioactivity was converted to CO_2.

The amount of radioactivity bound to the soil increased during the test period and finally approached 35%. Acid hydrolysis of the 365-day samples released up to 10% of the applied radioactivity in the post-extraction solids (PES). Most of the radioactivity released by acid hydrolysis was extractable with ethyl acetate, the majority of it (up to 8% of the applied dose) being methoxyfenozide. Another peak exhibited an identical chromatographic retention time to that of the metabolite RH-131154 and accounted for about 1% of the applied radioactivity. Other components were observed but did not exceed 0.3% of the applied dose. The majority of the bound radioactivity in the soil appeared to be incorporated into fulvic acid (up to 9% of the applied dose) and humic acid (8% of the applied dose). The humin fraction contained up to 5% of the applied radioactivity. The overall total recoveries (material balance) during the test period were 90-123%.

Confined rotational crops

A confined rotation study was conducted to determine the nature and level of residues which could be present in succeeding crops of mustard, white radish and wheat (Kim-Kang, 1998, report No. 34-98-99). In this study, the three different forms of [^{14}C] labelled methoxyfenozide were used: an A-ring label, a B-ring label, and the *t*-butyl label.

Three applications of each ^{14}C-methoxyfenozide, formulated as an emulsifiable concentrate, were made to bare ground soil at a total application rate of 2.2 kg ai/ha, equivalent to the maximum application rate in the United States. Mustard, white radish and wheat were planted in the treated soil at 30-, 90- and 365-day (nominal) intervals after the final application. Crops were harvested at an intermediate stage and at crop maturity. Soil cores were taken before application, following the last application, at the time the crops were planted and at the final harvest of the mature wheat crop.

All crop and soil samples were analyzed by combustion analysis to define the level of total radioactive residues (TRR). Crop samples were also extracted and the extracts were analyzed by HPLC in conjunction with LSC and TLC with radiometric detection. For unknown degradation products, the residues were isolated and purified by both HPLC and TLC, followed by identification using LC-MS analysis, in-line with ultraviolet (UV) and radiometric (RAM) detection.

Soil residue levels immediately after treatment were 0.78 mg/kg, 0.68 mg/kg and 0.90 mg/kg in the plots planted at 30 DAT, 90 DAT, and 365 DAT, respectively. In the plot planted at 30 DAT, soil residues were 0.39 mg/kg at the time of wheat harvest (256 DAT). In the plot planted at 90 DAT, soil residues were 0.29 mg/kg at wheat harvest (316 DAT). In the plot planted at 365 DAT, soil residues were 0.34 mg/kg at wheat harvest (621 DAT).

Soil samples collected 257 days after application (B-ring label) to the plot planted at 30 DAT were extracted and partitioned with water and acetonitrile/dichloromethane. The dichloromethane phase contained 53% TRR. Parent compound comprised 49% TRR, and 2% TRR was identified as RH-131154 (B-ring carboxylic acid). Unknowns N and R were 1.5% and 0.45%, respectively. No other soil analyses were reported.

Overall, total radioactive residue levels were <0.05-0.3 mg/kg in all crops, except for wheat forage and straw, which were 1-3 mg/kg. Residue levels were similar in immature and mature crops receiving the same treatment, except for wheat. In general, total residues found in mature and immature crop samples decreased significantly with increasing plant back time. The highest TRR residues, averaging ~3 mg/kg, were found in wheat straw samples from the plot planted at 30 DAT. Wheat forage residues averaged only approximately one-third those found in wheat straw. Wheat grain residues, at <0.05 mg/kg, were the lowest of any of the crops investigated. In general, the majority of the residue in all crops was readily extractable. Wheat straw and grain, however, contained large proportions of unextractable radioactivity, although the magnitude of the residue in the grain was low. Approximately 20-44% of the TRR in radish root was also unextractable but these levels were also low.

Analyses of plant extracts showed that extensive metabolism had occurred in all crops examined. Because of the presence of many metabolites in all samples, individual component concentrations were generally low. Only a few metabolites were observed at >10% of TRR or above 0.01 mg/kg.

In mustard leaves from the plot planted at 30 DAT, methoxyfenozide was present at up to 0.027 mg/kg (~21% of TRR) and individual metabolites were present at <0.05 mg/kg each. Two metabolites were the other major contributors to the total residue. One of them (metabolite J) was present at up to 0.037 mg/kg (33% of the TRR) and contained RH-151055 as the primary component. RH-151055 was identified as the glucose conjugate of an A-ring phenol metabolite, formed by methoxy cleavage of methoxyfenozide. The other metabolite, RH-152067, was identified as an *N*-glycosyl conjugate of a B-ring mono-alcohol metabolite and was found at up to 0.024 mg/kg (~18% of the TRR). Additionally, up to 14 other degradation products, many of which were identified as sugar conjugates, were found at <10% of the TRR in mustard leaf samples.

In radish leaves from the plot planted at 30 DAT, individual residues (up to 21 components were characterized) were also all present at <0.05 mg/kg. Methoxyfenozide was found at concentrations up to 0.013 mg/kg (~18% of the TRR). RH-152067 (*N*-glycosyl conjugate of the B-ring mono-alcohol) was detected at up to 0.035 mg/kg (~13%) while RH-151055 (glucose conjugate of the A-ring phenol) was detected at up to 0.028 mg/kg, (~12-13% of the TRR).

Radish roots contained the highest levels of non-metabolized methoxyfenozide of any crop investigated. Methoxyfenozide was the major residue present in mature radish root from the plot planted at 30 DAT, ranging from 0.022-0.033 mg/kg (up to 41% of the TRR). RH-151055, possibly together with minor amounts of 2 other metabolites, was found at up to 0.011 mg/kg (10-13% of the TRR). A total of 14 other residue components, including RH-152072 (malonylglycosyl conjugate of the A-ring phenol), were characterized in radish roots, at individual concentrations in the range <0.001-0.006 mg/kg. Enzyme and acid hydrolysis of the radish root post-extraction solids (PES) from the A-ring label treatment, accounting for ~24% of the TRR, released the bound residue. The major component of this activity was identified as RH-131364 (B-ring-monoalcohol) (0.005 mg/kg, 7% of the TRR).

Wheat forage from the plot planted at 30 DAT contained total residues in the range 0.72-1.5 mg/kg. The parent compound was a very minor component, present at less than 1% of the TRR (maximum of 0.009 mg/kg). The two major components of the extract were RH-152072 (up to 0.705 mg/kg, 48% of the TRR) and RH-151055, (up to 0.356 mg/kg, 28% of the TRR). RH-152075 and RH-152074, isomeric sugar conjugates of the A-ring phenol B-ring mono-alcohol were also found at levels up to 0.10 mg/kg (up to ~8% of the TRR) in the same samples. All other metabolites were present at less than 3% of the TRR (< 0.035 mg/kg).

Wheat straw contained the highest residues of any crop, being in the range 2-4 mg/kg in samples from the plot planted at 30 DAT. Only ~45-50% of the residue in straw was extractable. The major residue component in the extract was RH-117236, present at up to 1.4 mg/kg and accounting for up to ~37% of the TRR. RH-117236 is the A-ring phenol metabolite of methoxyfenozide and the aglycone of RH-151055. Other components of the TRR were present at lower levels. RH-151055 (glucose conjugate of the A-ring phenol) was quantified only collectively, with several other minor

components, and was found in straw samples at levels up to 0.374 mg/kg (9.54% of the TRR). RH-152072 (malonylglycosyl conjugate of the A-ring phenol) comprised up to 10% of the TRR, with a maximum level of 0.046 mg/kg. Up to 17 other minor metabolites (<10%), as well as trace levels of methoxyfenozide (up to 0.021 mg/kg) were also present in straw. Enzyme, acid and base hydrolysis resulted in release of essentially all the bound activity from the B-ring label PES. In the *t*-butyl label PES, ~11% remained unextractable, indicative of incorporation into natural products. Released activity was shown to consist of an additional quantity of RH-117236 (A-ring phenol metabolite, up to 0.243 mg/kg, ~8% of the TRR), free sugars and presumed hydrolytic degradation products such as substituted benzoic acids.

Wheat grain contained the lowest residue levels of the crops and materials tested, ~0.05 mg/kg. Only 11-23% of the residue was readily extractable from grain. The major component of the extractable grain residue was RH-117236, at a concentration of 0.006 mg/kg (~15% of the TRR). Free sugars, resulting from incorporation of the radiolabel into the carbon pool, were also found. Most of the residue in the grain (77-87% of the TRR in the 30 DAT samples) was bound in the PES. Enzyme hydrolysis of the PES released an additional 0.004 mg/kg (9%) of RH-117236, while acid hydrolysis released most of the remaining residue, which consisted of free sugars and hydrolytic degradation products of methoxyfenozide.

Residue concentrations and identifications/characterizations are summarized in Table 19.

Table 19. Metabolite distribution in rotational crop samples from planting at 30, 90 and 365 DAT after application of ^{14}C-methoxyfenozide to bare ground soil, at a total application rate of 2.2 kg ai/ha (Kim-Kang, 1998).

Metabolite [1/] or fraction	30 DAT A-ring		90 DAT A-ring		365 DAT A-ring		30 DAT B-ring		90 DAT B-ring		30 DAT *t*-butyl		90 DAT *t*-butyl	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Mustard leaf														
RH-112485	13	0.010	12	0.006	18	0.007	17	0.019	17	0.007	21	0.027	13	0.007
RH-117236	0.82	0.001												
RH-131364	1.8	0.001	2.5	0.001	3.4	0.001	2.6	0.003	2.2	0.001	2.2	0.003	2.6	0.001
Met A	2.7	0.003	2.8	0.001	5.5	0.002	1.2	0.001	5.4	0.002				
Met C											1.7	0.002		
Met D	2.8	0.002					1.3	0.001	2.5	0.001	2.1	0.003	2.8	0.002
Met E [2/]	6.2	0.005	7.0	0.003			5.3	0.006	6.0	0.002	8.7	0.011	6.6	0.004
Met F							4.0	0.004	5.5	0.002			1.6	0.001
Met G-3	3.9	0.003					3.1	0.003			3.2	0.004	1.7	0.001
RH-152072 (Met H)	6.3	0.005	8.5	0.005			7.6	0.009	2.7	0.001	5.9	0.008	8.0	0.004
RH-152071 (Met I-1)	6.1	0.004	7.0	0.003	4.5	0.002	4.0	0.004	6.4	0.003	5.4	0.006	6.4	0.003
Met I-2	4.2	0.003	6.0	0.003			4.9	0.005	2.9	0.001	2.8	0.004	2.2	0.001
Met J [3/]	21	0.017	15	0.008	30	0.012	33	0.037	17	0.006	17	0.022	16	0.009
RH-152067 (Met K)	18	0.014	17	0.008			1.4	0.002	18	0.007	18	0.024	21	0.011
RH-131157 (Met L)					12	0.005			0.58	<0.001	0.63	0.001		
HR-152073 (Met M)	1.0	0.001			4.5	0.002	2.1	0.002	0.58	<0.001	0.70	0.001		
Met N									0.79	<0.001			0.97	0.001
Met O	1.2	0.001	1.3	0.001			1.9	0.002	0.72	<0.001	2.2	0.003	1.0	0.001
Hexane-sol.	3.9	0.003	11	0.005	0	0	5.5	0.006	1.3	0.001	3.5	0.004	6.8	0.004
Bound	6.6	0.005	9.4	0.005	21	0.009	5.1	0.006	11	0.004	5.1	0.007	8.8	0.005
Total	100	0.078	100	0.049	100	0.040	100	0.11	100	0.038	100	0.13	100	0.055
Radish Leaf														
RH-112485	18	0.013	24	0.007	2.0	0.002	9.6	0.006	9.7	0.005	2.8	0.007	5.2	0.007
RH-117236	1.3	0.001							0.52	<0.001				
RH-131364	4.6	0.003	5.9	0.002	11	0.010	3.4	0.002	3.6	0.002	1.4	0.004	1.3	0.002
Met A	1.5	0.001	4.5	0.001			2.4	0.001	2.6	0.001				
Met B							2.6	0.001						
Met C	4.7	0.003	5.2	0.002	4.7	0.004	2.7	0.002	8.2	0.004	9.4	0.025	8.4	0.012

Metabolite [1] or fraction	30 DAT A-ring		90 DAT A-ring		365 DAT A-ring		30 DAT B-ring		90 DAT B-ring		30 DAT *t*-butyl		90 DAT *t*-butyl	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
Met D	4.0	0.003	2.6	0.001			3.6	0.002	3.6	0.002	6.5	0.017	5.6	0.008
Met E [4]	7.9	0.006	7.8	0.002	6.8	0.006	8.3	0.005	13	0.007	11	0.030	9.4	0.013
Met F-2											1.6	0.004	1.1	0.002
Met G-2	3.8	0.003	2.4	0.001			4.0	0.002	5.9	0.003	5.9	0.016	6.9	0.008
RH-152072 (Met H)	5.2	0.004	2.6	0.001	10	0.008	13	0.008	8.9	0.004	13	0.034	9.6	0.014
Met I [5]	7.2	0.005	7.2	0.002	6.8	0.006	2.7	0.002	6.2	0.003	11	0.028	10	0.014
Met J [3]	13	0.010	12	0.003	15	0.013	13	0.007	8.8	0.004	13	0.035	12	0.015
RH-152067 (Met K)	7.2	0.005	4.0	0.001			9.2	0.006	11	0.006	10	0.027	11	0.015
RH-131157 (Met L)	8.4	0.006	5.7	0.002	28	0.024	11	0.007	6.2	0.003				
HR-152073 (Met M)					2.2	0.002							0.68	0.001
Met N			1.8	0.001			1.0	0.001					2.1	0.003
Met P														
Hexane-sol.	3.8	0.003	0.47	<0.001	5.0	0.004	0.88	0.001	1.8	0.001	2.0	0.005	1.8	0.003
Bound	9.8	0.007	14	0.004	8.0	0.007	13	0.007	9.4	0.005	4.1	0.011	9.6	0.014
Total	100	0.073	100	0.030	100	0.036	100	0.060	100	0.050	100	0.26	100	0.14
Radish root														
RH-112485	41	0.033	35	0.012	33	0.022	27	0.011	32	0.007	34	0.033	27	0.012
RH-117236			2.1	0.001	0.99	0.001	0.95	<0.001			0.75	0.001		
RH-131364	2.3	0.002	3.3	0.001	4.5	0.003	2.0	0.001	1.8	<0.001	5.7	0.005	2.6	0.001
Met A	3.6	0.003	8.6	0.003	4.9	0.003	8.6	0.003	20	0.004	0.78	0.001	4.6	0.002
Met B							4.8	0.002						
Met C					1.2	0.001							1.3	0.001
Met D	1.8	0.001												
Met E [2]	2.2	0.002	3.4	0.001	5.0	0.003	5.9	0.002			6.2	0.006	3.5	0.002
RH-152072 (Met H)	5.3	0.004	6.1	0.002	4.0	0.003	6.4	0.003			5.0	0.005	3.5	0.002
Met I [6]			2.0	0.001	4.1	0.003	2.5	0.001			1.2	0.001	7.7	0.004
Met J [7]	13	0.011	13	0.005	9.7	0.003	5.4	0.002	2.7	0.001	4.9	0.002	3.0	0.002
RH-152067 (Met K)	4.0	0.003	4.3	0.002	3.7	0.003	5.4	0.002	2.7	0.001	4.9	0.005	3.9	0.002
RH-131157 (Met L)			1.3	<0.001	2.5	0.002	1.0	<0.001			2.0	0.002	3.0	0.002
Met N			1.4	0.001			1.4	0.001					1.3	0.001
Hexane-sol.	2.6	0.002	0.27	<0.001	0.79	0.001	1.5	0.001	0	0	4.4	0.004	4.7	0.002
Bound	24	0.019	20	0.007	26	0.070	20	0.008	44	0.010	23	0.022	27	0.012
Total	100	0.080	100	0.036	100	0.070	100	0.040	100	0.022	100	0.096	100	0.048
Wheat forage														
RH-112485	0.79	0.005	0.73	0.003	0.74	0.005	0.65	0.002	0.61	0.009	0.42	0.002		
RH-117236	1.1	0.008	0.88	0.003	0.97	0.007	2.0	0.007	0.91	0.013	0.56	0.003	7.0	0.007
Met A													4.2	0.004
RH-152074 (Met E-1)			2.8	0.010					1.7	0.025				
Met F-1	3.3	0.024	4.2	0.015	2.4	0.018	1.2	0.004	2.3	0.034	4.1	0.020		
RH-152075 (met G-1)	5.2	0.037	4.2	0.015	8.3	0.062	9.0	0.032	7.0	0.10	6.3	0.032	5.1	0.005
RH-152072 (Met H)	46	0.33	42	0.15	47	0.35	44	0.16	48	0.70	47	0.24	2.1	0.002
RH-152071 (Met I-1)	4.8	0.034	3.8	0.013	3.4	0.025	1.5	0.005	2.3	0.033	1.6	0.008		
RH-151055 (Met J or J-1)	28	0.20	26	0.090	29	0.22	32	0.11	24	0.36	25	0.13	35	0.037
RH-152078 (Met J-2)	1.3	0.009							1.7	0.025	2.0	0.010		
RH-152067 (Met K)	1.5	0.011	2.8	0.010					1.5	0.022	3.0	0.016	3.6	0.004

Metabolite [1] or fraction	30 DAT A-ring		90 DAT A-ring		365 DAT A-ring		30 DAT B-ring		90 DAT B-ring		30 DAT *t*-butyl		90 DAT *t*-butyl	
	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg
RH-131157 (Met L)			1.1	0.004									4.0	0.004
HR-152073 (Met M)	3.5	0.025	2.8	0.009	0.69	0.005	0.82	0.003	2.3	0.033	3.0	0.015	26	0.027
Met N	0.21	0.001	1.3	0.004	0.15	0.001	1.0	0.004	0.23	0.003	0.91	0.005	1.3	0.001
Met O									0.18	0.003				
Met Q									0.05	0.001				
Hexane-sol.	0.33	0.002	1.2	0.004	0.93	0.007	0.62	0.002	0.33	0.005	0.25	0.001	0.50	0.001
Bound	4.2	0.030	5.7	0.020	5.7	0.042	7.1	0.025	6.4	0.094	5.3	0.027	10	0.011
Total	100	0.72	100	0.35	100	0.74	100	0.35	100	1.5	100	0.51	100	0.10
Wheat Straw														
RH-112485	0.45	0.009	0.20	0.002			0.37	0.012	0.51	0.008	0.53	0.021	0.47	0.008
RH-117236	32	0.65	28	0.32	27	0.12	37	1.1	30	0.45	35	1.4	35	0.60
Met A			0.17	0.002										
Met C									0.24	0.004				
Met E [8]	0.25	0.005	0.78	0.009			0.96	0.030	0.80	0.012	1.6	0.062	1.6	0.29
Met F	0.57	0.011					1.5	0.048	1.4	0.021	1.01	0.040	1.0	0.18
Met G-1 [9]	1.8	0.037	1.4	0.016	3.9	0.017	1.2	0.037	1.2	0.017	1.7	0.069	2.1	0.036
RH-152072 (Met H)	0.88	0.018	0.74	0.009	9.8	0.042	0.56	0.016	1.4	0.021	1.2	0.046	1.4	0.024
Met I [10]	1.9	0.038	0.66	0.008			2.5	0.077	0.83	0.012	1.2	0.049	1.3	0.023
Met J [11]	9.5	0.19	13	0.15	20	0.087	7.5	0.24	8.6	0.13	9.4	0.37	9.2	0.16
RH-131157 (Met L)			0.18	0.002										
HR-152073 (Met M)	0.86	0.017			1.5	0.006	0.95	0.030	2.2	0.033	1.3	0.050	2.4	0.041
Met N	0.45	0.009	0.29	0.003			0.77	0.024	0.37	0.005	1.5	0.058	0.75	0.013
Met P									0.33	0.005				
Met R							0.25	0.008	0.35	0.005			1.3	0.022
Hexane-sol.	0.37	0.007	0.36	0.004	0.49	0.002	0.46	0.014	0.49	0.007	0.74	0.029	0.61	0.010
Bound	50	1.0	55	0.64	37	0.16	46	1.4	51	0.75	44	1.8	43	0.74
Total	100	2.0	100	1.2	100	0.43	100	3.1	100	1.5	100	4.0	100	1.7

[1] See Table 21 for metabolite definitions and structures.
[2] Metabolite E was shown to consist of two components, RH-152074 or RH-152075 (E-1) and RH-152069 (E-5) in 30 and 90 DAT, *t*-butyl labelled samples.
[3] Metabolite J was shown to consist of two components, RH-151055 or J-1 and RH-152068 or J-5 in 30 and 90 DAT, *t*-butyl labelled samples.
[4] Metabolite E was shown to consist of three components, RH-152074 (E-1), E-3 and RH-152070 (E-4) in 30 and 90 DAT, *t*-butyl labelled samples.
[5] Metabolite I was shown to consist of two components, RH-152071 (I-1) and I-4 in 30 DAT, *t*-butyl labelled samples.
[6] Metabolite I was shown to consist of two components, RH-152071 (I-1) and RH-131156 (I-5) in 30 and 90 DAT, *t*-butyl labelled samples.
[7] Metabolite J was shown to consist of two or three components, RH-15055 (J-1), RH-152068 (J-5) and J-6 in 30 and 90 DAT, *t*-butyl labelled samples.
[8] Metabolite E was shown to consist of two components, RH-152074 or RH-152075 (E-1) and E-2 in 30 and 90 DAT, *t*-butyl labelled samples.
[9] Metabolite G-1 was assigned as either RH-152074 or RH-152075.
[10] Metabolite I was shown to consist of two components, RH-152071 (I-1) and RH-152078 (I-3) in 30 and 90 DAT, *t*-butyl labelled samples.
[11] Metabolite J was shown to consist of several components: RH-151055 (J-1), RH-152078 (J-2), RH-141511 (J-3) and J-4 or J-7 in 30 and 90 DAT, *t*-butyl labelled samples.

A residue study was conducted to determine the residues of methoxyfenozide in rotational crops at two trial locations (Texas and California) in the USA (Sharma, 2000, report No. 34-00-07). Leaf lettuce, used as the treated crop, was planted in plots at each location, prior to subsequent planting of rotational crops. Five applications of methoxyfenozide 80W were made at 7-10 day intervals to the leaf lettuce crop, at 0.45 kg ai/ha per application and a total application of 2.25 kg ai/ha. The treated leaf lettuce crop was harvested and removed at 1-3 days after the last application. Rotational crops, representative of leafy vegetables (mustard greens), fruiting vegetables (tomato), cucurbits (cucumbers), root vegetables (turnips), cereal grains (wheat), legumes (soya beans) and bulb

vegetables (onions), were planted 6-7 days after the last application of methoxyfenozide. Crop samples were collected at normal harvest. Mustard greens, turnips, onions, cucumbers and tomatoes were analyzed for methoxyfenozide by HPLC-UV, with a limit of detection (LOD) and a limit of quantification (LOQ) of 0.006 mg/kg and 0.02 mg/kg, respectively. Wheat and soybeans were analyzed for methoxyfenozide and three other metabolites, RH-151055 (glucose conjugate of the A-ring phenol), RH-152072 (malonylglycosyl conjugate of the A-ring phenol) and RH-117236 (A-ring phenol). During analysis, RH-152072 was converted to RH-151055 and therefore the results shown below for RH-151055 included RH-152072. Analysis was performed using HPLC-MS, with an LOD and LOQ of 0.006-0.015 mg/kg and 0.02-0.05 mg/kg, respectively. See Table 20.

Table 20. Residues in rotational crops with a 7-day plant-back interval (Sharma, 2000).

Crop	Average residue level, mg/kg		
	methoxyfenozide	RH-151055 [1/]	RH-117236 [1/]
Mustard greens	0.095-0.12	NA	NA
Turnip tops	0.026-0.064	NA	NA
Turnip root	<LOQ-0.021	NA	NA
Onions	ND [2/] to 0.055	NA	NA
Tomatoes	ND	NA	NA
Wheat grain	ND	ND	ND-<LOQ
Wheat forage	0.021-0.038	0.64-1.1	0.036-0.043
Wheat hay	0.027-0.031	1.4-3.0	0.11-0.35
Wheat straw	0.023-0.057	0.32-2.1	1.1-2.2
Soya bean seeds	ND-0.02	ND-<LOQ	ND
Soya bean forage	0.09-1.2	0.58-2.5	0.040-0.079
Soya bean hay	0.049-1.1	0.061-4.1	0.11-0.21

NA = not analyzed.
[1/] Residues expressed as methoxyfenozide equivalents. See Table 21 for compound identifications.
[2/] Not detected, <LOD.

Environmental fate in water

The Meeting received information on the hydrolysis of methoxyfenozide in aqueous solution.

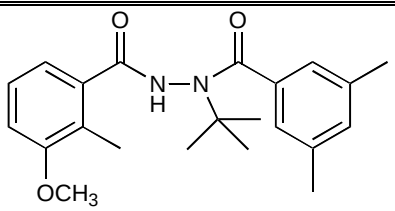
The hydrolytic stability of methoxyfenozide was evaluated in sterile buffer solutions at pH 5, 7, and 9 (Lin, 1994c, report No. 34-95-49). The concentration of combined unlabelled and ^{14}C-methoxyfenozide test material (*t*-butyl label) was 1 µg/ml. Samples were incubated in the dark at 25ºC for up to 30 days. Duplicate aliquots were taken from each buffer solution at 0, 7, 10, 14, 21 and 30 days and the methoxyfenozide was extracted by solid-phase extraction. Determinations were performed by LSC, TLC or HPLC.

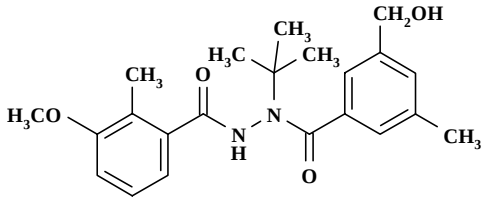
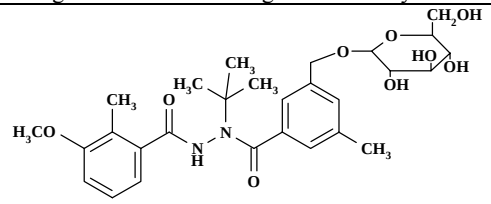
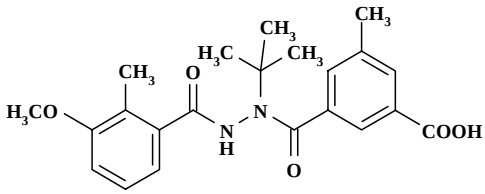
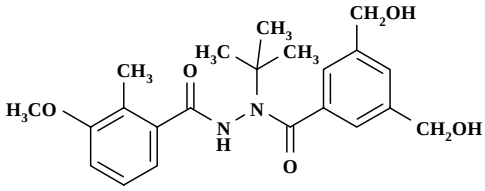
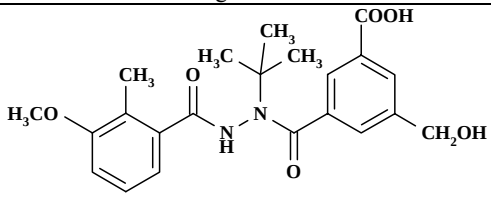
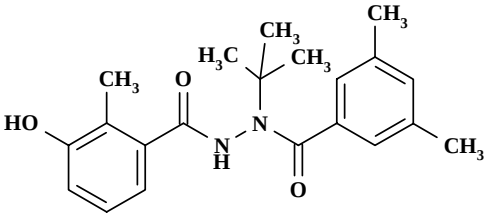
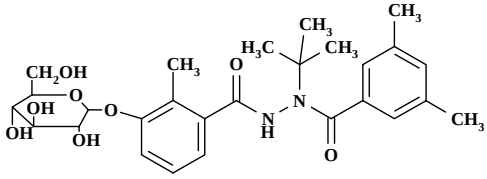
Methoxyfenozide was found to be stable to hydrolysis at pH 5, 7 and 9 throughout the 30-day testing period. In all solutions, the parent compound was the only significant component detected, averaging 96.5-99.8% of the total radioactivity. The calculated half-life values were 587 days at pH 5, 1572 days at pH 7 and 695 days at pH 9.

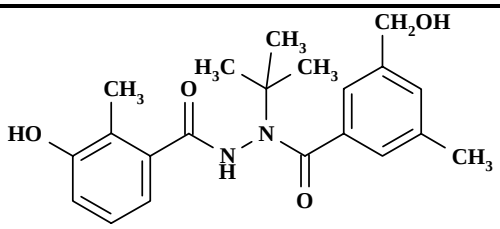
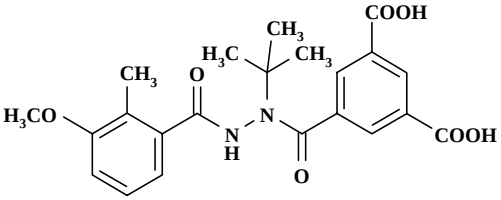
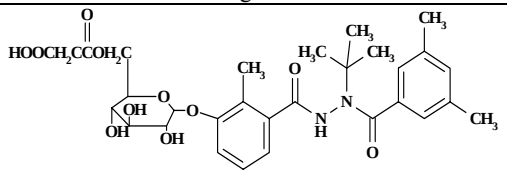
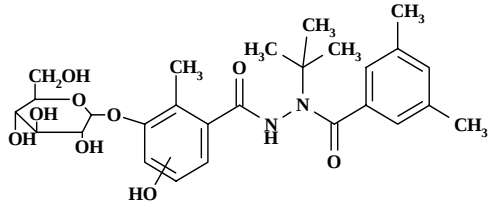
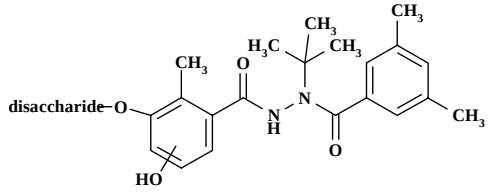
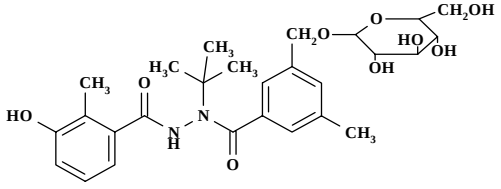
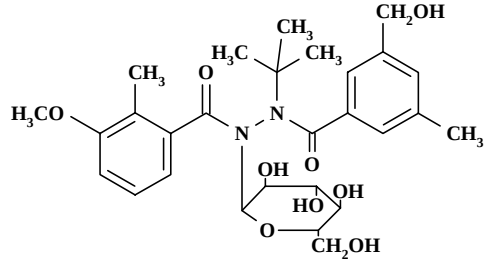
Identities of methoxyfenozide metabolism and degradation products

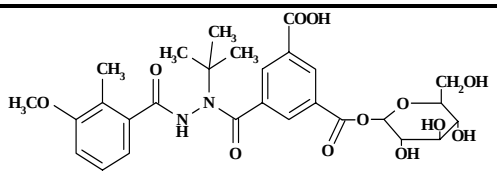
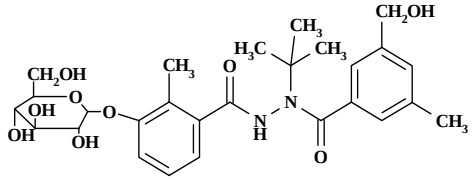
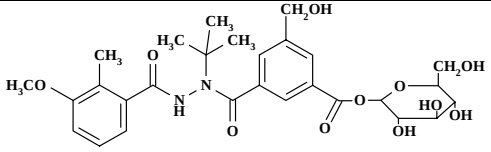
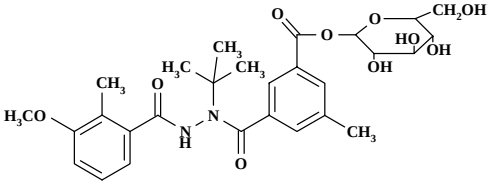
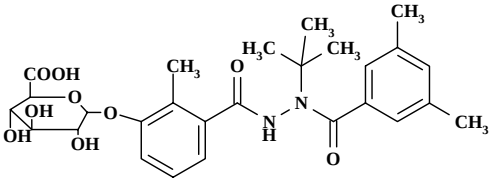
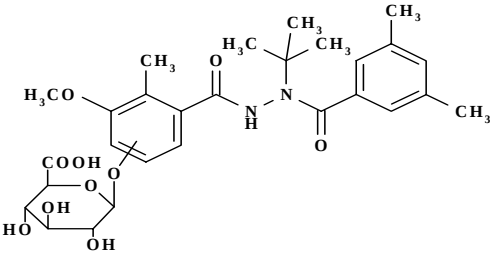
The structures and chemical names of methoxyfenozide metabolism and degradation products are summarized in Table 21.

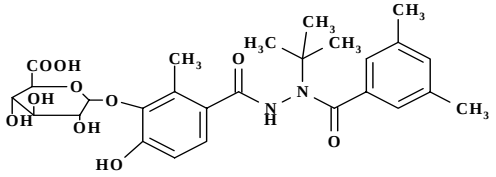
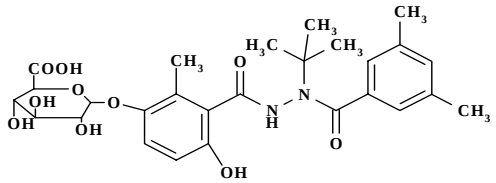
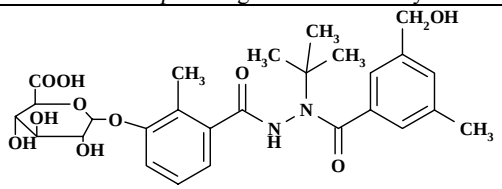
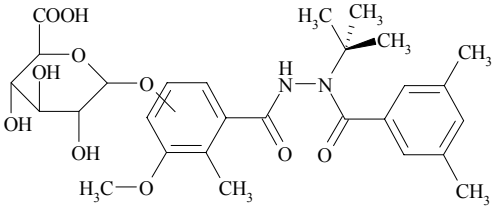
Table 21. Chemical names and structures of methoxyfenozide and its metabolites identified in plant or animal metabolism studies and in soil or rotational crops.

Manufacturer's code and chemical name	Structure and trivial name	Found in:
RH-2485 RH-112485 3,5-dimethylbenzoic acid *N*-*tert*-butyl-*N'*-(3-methoxy-2-methylbenzoyl)hydrazide	 Methoxyfenozide	Cotton, apples, grapes Goat milk, liver, kidney, leg muscle, loin muscle, fat Hen eggs, liver, dark muscle, light muscle, fat, skin with fat Rotational mustard, radish roots and tops, wheat forage and straw

Manufacturer's code and chemical name	Structure and trivial name	Found in:
RH-131364 3-hydroxymethyl-5-methyl-benzoic acid *N-tert*-butyl-*N*-(3-methoxy-2-methylbenzoyl) hydrazide	 B-ring mono-alcohol analogue of methoxyfenozide	Apples and grapes Rotational mustard, radish roots and tops, wheat straw
RH-152073 3-glycosylmethyl-5-methyl-benzoic acid *N-tert*-butyl-*N*-(3-methoxy-2-methylbenzoyl) hydrazide	 O-glycosyl conjugate of B-ring mono-hydroxy analogue of methoxyfenozide	Apples and grapes Rotational mustard, radish roots and tops, wheat straw
RH-131154 (metabolite C) 3-[*N-tert*-butyl-*N*-(3-methoxy-2-methylbenzoyl)-hydrazinocarbonyl]-5-methylbenzoic acid	 B-ring carboxylic acid	Soil, aerobic Grapes (tentative identification) Goat milk, liver, kidney, leg muscle, loin muscle Hen eggs, liver, dark muscle, light muscle, skin with fat
RH-131157 (metabolite L) 3,5-bis-hydroxymethylbenzoic acid *N-tert*-butyl-*N*-(3-methoxy-2-methylbenzoyl) hydrazide	 B-ring di-alcohol	Apples Rotational mustard, radish roots and tops, wheat forage and straw
RH-152068 (metabolite J-5) (metabolite E_1)	 B-ring mono-alcohol B-ring monocarboxylic acid	Rotational mustard, radish roots and tops Goat milk, liver, kidney, leg muscle, loin muscle
RH-117236 (metabolite B) 3,5-dimethylbenzoic acid *N-tert*-butyl-N-(3-hydroxy-2-methylbenzoyl) hydrazide	 A-ring phenol (demethylated methoxyfenozide)	Soil: aerobic Grapes (glucose conjugate) Goat milk, liver, kidney, leg muscle, loin muscle, fat Hen eggs, liver, dark muscle, light muscle, fat, skin with fat Rotational mustard, radish roots and tops, wheat forage, grain and straw
RH-151055 (metabolite J-1) 3,5-dimethylbenzoic acid *N-tert*-butyl-*N*-[3-(β-D-gluco-pyranosyloxy)-2-methyl-benzoyl] hydrazide	 glucose conjugate of A-ring phenol	Rotational mustard, radish roots and tops, wheat forage and straw

Manufacturer's code and chemical name	Structure and trivial name	Found in:
RH-141511 (metabolite J-3) (metabolite E_2) 3-hydroxymethyl-5-methyl-benzoic acid *N-tert*-butyl-*N'*-(3-hydroxy-2-methylbenzoyl) hydrazide	 A-ring phenol B-ring-monoalcohol	Rotational wheat straw Plant metabolism Goat milk, liver, kidney, leg muscle, loin muscle Hen eggs, liver, dark muscle, light muscle, fat, skin with fat
RH-131156 (metabolite I-5) 3-[*N-tert*-butyl-*N'*-(3-methoxy-2-methylbenzoyl)-hydrazino-carbonyl]-3,5-dibenzoic acid	 B-ring-diacid	Rotational radish roots
RH-152072 (metabolite H)	 malonylglycosyl conjugate of the A-ring phenol	Rotational mustard, radish roots and tops, wheat forage and straw
RH-152078 (metabolite J-2)	 glycosyl conjugate of A-ring diphenol	Rotational wheat forage and straw
RH-152071 (metabolite I-1)	 dissaccharide conjugate of the A-ring diphenol	Rotational mustard, radish roots and tops, wheat forage and straw
RH-152075 (metabolite G-1)	 glycosyl conjugate of A-ring phenol-B-ring-monoalcohol	Rotated wheat forage and straw
RH-152067 (metabolite K)	 N-glycosyl conjugate of B-ring-monoalcohol	Rotated mustard, radish roots and tops, and wheat forage

Manufacturer's code and chemical name	Structure and trivial name	Found in:
RH-152069 (metabolite E-5)	 O-ester conjugate of B-ring diacid	Rotational mustard
RH-152074 (metabolite E-1)	 glycosyl conjugate of A-ring phenol B-ring mono-alcohol	Rotational mustard, radish roots and tops, wheat forage and straw
RH-152070 (metabolite E-4)	 conjugate of B-ring-monoalcohol-B-ring-monocarboxylic	Rotational radish roots and tops
RH-152076 (metabolite I-3)	 conjugate of B-ring carboxylic acid	Rotational wheat straw
RH-141518 (RH-1518 and metabolite G) β-D-glucopyranuronic acid, 3-{[2-(1,1-dimethylethyl)-2-(3,5-dimethylbenzoyl)-hydrazino]carbonyl}-2-methylphenyl-	 Glucuronide conjugate of A-ring phenol	Goat milk, liver, kidney, loin muscle, fat Hen eggs, liver, dark muscle, light muscle, fat, skin with fat
RH-149087	 A-ring-OH-glucuronide	Animal metabolism

Manufacturer's code and chemical name	Structure and trivial name	Found in:
Metabolite H β-D-glucopyranuronic acid, 3-{[2-(1,1-dimethylethyl)-2-(3,5-dimethylbenzoyl)-hydrazino]carbonyl}-6-hydroxy-2-methylphenyl-	  Glucuronide conjugate of A-ring with additional OH group *ortho* or *para* to glucuronide moiety	Goat milk, liver and kidney Hen eggs, liver, light muscle, fat
RH-141519 (metabolite I) β-D-glucopyranuronic acid, 3-{[2-(1,1-dimethylethyl)-2-(3-hydroxymethyl-5-methylbenzoyl)-hydrazino]carbonyl}-2-methylphenyl-	 A-ring phenol glucuronide B-ring mono-alcohol	Goat milk, liver, kidney Hen eggs, liver, dark muscle, light muscle, skin with fat
Metabolite F β-D-glucopyranuronic acid, 4-{[2-(1,1-dimethylethyl)-2-(3,5-dimethylbenzoyl)-hydrazino]carbonyl}-2-methyl-3-methoxyphenyl-		Goat milk, liver, loin muscle Hen eggs, liver, dark muscle, light muscle, fat, skin with fat

RESIDUE ANALYSIS

Analytical methods

Analytical methods for residues of methoxyfenozide were developed for a wide range of substrates. In addition, methods were also developed for the principal metabolite in animal tissues, the glucuronide conjugate of the A-ring phenol (RH-1518). In general, after an extraction specific to the matrix and a standard clean-up, determination of methoxyfenozide parent compound was by HPLC, relying on UV or MS detection. For the metabolites, the determination was made by HPLC with MS detection.

Several analytical methods were used for supervised trials and processing studies. Method 00470 (Schoning and Seym, 1998, report No. 34-98-169) was developed for the determination of methoxyfenozide in plant materials. Methoxyfenozide was extracted from plant matrices using a mixture of acidic methanol/water. After filtration, an aliquot of the extract was diluted with sodium chloride solution and partitioned against dichloromethane and further cleaned-up on a ChemElut® column. For orange and mandarin peel samples, a further clean-up was performed by column chromatography on Florisil® and subsequent elution with cyclohexane/ethyl acetate. The eluate was evaporated to dryness and dissolved in acetonitrile/water. Residues were quantified by reversed phase HPLC with electrospray MS/MS detection of the *m/z* 369 $\rightarrow$ +149 transition. Table 22 lists the recoveries obtained from different matrices with this method. The overall mean recovery of methoxyfenozide from all commodities at all fortification levels was 96%, with a relative standard deviation (RSD) of 9.1%, calculated from a total of 148 individual results. The minimum recovery was 70% and the maximum was 114%.

Table 22. Summary of methoxyfenozide recovery by method 00470 (Schoning and Seym, 1998).

Crop	Matrix	Fortification mg/kg	Recovery, %				
			Individual results	Average per FL [1/]	RSD [2/]	Overall average	Overall RSD
Apple	Fruit	0.05	103, 104, 104, 108, 110	106	2.9		
		0.5	92, 97, 98, 98, 100	97	3.1	101	5.4
	Sauce	0.05	79, 82, 83	81	2.6		
		0.5	93, 94, 100	96	4.0	89	9.4
	Juice	0.05	103, 104, 108	105	2.5		
		0.5	98, 99, 100	99	1.0	102	3.7
	Pomace	0.05	74, 85, 86, 92, 93	86	8.8		
		0.5	86, 89, 92, 94, 100	92	5.8	89	7.8
	Apple, dried	0.05	78, 80, 93, 94, 105	90	12		
		0.5	78, 84, 86, 89, 90	85	5.6	88	9.6
Grape	Must	0.05	100, 110, 111	107	5.7		
		0.5	95, 97, 103	98	4.2	103	6.5
	Grapes, bunches	0.05	103, 108, 110	107	3.4		
		0.5	97, 99, 101	99	2.0	103	5.0
	Wine	0.05	106, 108, 113	109	3.3		
		0.5	98, 99, 100	99	1.0	104	5.7
	Raisin	0.05	99, 100, 100, 100, 103	100	1.5		
		0.5	93, 94, 94, 95, 96	94	1.2	97	3.5
Mandarin	Preserve	0.05	94, 99, 101	98	3.7		
		0.5	89, 92, 102	94	7.2	96	5.5
	Pulp	0.05	96, 100, 106	101	5.0		
		0.5	93, 106, 108	102	8.0	102	6.0
	Peel	0.05	91, 91, 93	92	1.3		
		0.5	81, 87, 89	86	4.9	89	4.8
Orange	Marmalade	0.05	89, 90, 95	91	3.5		
		0.5	99, 102, 102	101	1.7	96	6.0
	Pulp	0.05	89, 92, 93	91	2.3		
		0.5	94, 96, 99	96	2.6	94	3.7
	Peel	0.05	93, 95, 96	95	1.6		
		0.5	81, 86, 85	84	3.1	89	6.9
	Juice	0.05	70, 78, 82	77	8.0		
		0.5	92, 94, 94	93	1.2	85	11.7
Peach	Fruit	0.05	102, 103, 105	103	1.5		
		0.5	95, 97, 99	97	2.1	100	3.8
Pear	Fruit	0.05	105, 106, 114	108	4.6		
		0.5	98, 98, 100	99	1.2	104	6.0
Pepper	Fruit	0.05	90, 92, 94	92	2.2		
		0.5	87, 88, 92	89	3.0	91	2.9
Tomato	Fruit	0.05	93, 93, 94	93	0.6		
		0.5	93, 101, 102	99	5.0	96	4.5
	Paste	0.05	77, 78, 80	78	2.0		
		0.5	100, 104, 113	106	6.3	92	16.9
	Juice	0.05	103, 106, 108	106	2.4		
		0.5	101, 105, 107	104	2.9	105	2.5
Overall average % recovery and % RSD for all matrices at all fortification levels (n=148)						96	9.1

[1/] FL = fortification level.
[2/] RSD = relative standard deviation (%).

Method 00458 (Wu, Desai and Hofmann, 1996a, report No. TR 34-95-55) was developed for the determination of methoxyfenozide in apples and grapes. Methoxyfenozide residues were extracted by blending with acidic methanol/water solution. After extraction they were purified initially by partition with hexane, then partition with dichloromethane and finally by Florisil and silica-gel column chromatography, before HPLC analysis. Quantification was carried out by isocratic HPLC with UV detection at 240 nm. The limit of quantification (LOQ) was 0.01 mg/kg for both substrates. The average recoveries at levels from 0.01 to 1.0 mg/kg were 94 ± 11%, for 23 apple samples, and 88 ± 9.1%, for 11 grape samples (Table 23).

Table 23. Summary of recovery data for method 00458 (Wu, Desai and Hofmann, 1996a).

Matrix	Fortification mg/kg	Recovery Results				
		Average (%)	Range	Standard deviation	% RSD [1/]	n
Apple	0.01–1.0	94	79 – 130	11.1	12	23
Grape	0.01–0.25	88	69 – 95	9.1	10	11

[1] Relative standard deviation.

Various modifications of the above two methods, based mainly on changes in clean-up conditions, were developed and validated for many plant matrices.

Method 34-99-26 (Zhang and Doyle, 1999, Report No. 34-99-26) was developed to determine residues in cherries, peaches, plums, prunes, and prune juice. Methoxyfenozide residues were extracted with acidic methanol. Following hexane and methylene chloride partitioning to remove non-polar compounds, the extract was concentrated and then purified by basic alumina column chromatography (all matrices) or Envicarb (carbon) SPE (prune and prune juice only). Quantification was accomplished by HPLC with UV detection at 240 nm. The method was validated by fortification of control samples with methoxyfenozide at levels ranging from 0.02 to 1.0 mg/kg. LC-MS confirmatory analysis was performed for peaches and plums. Negative-ion LC-MS (*m/z* 367.3) was used to confirm the methoxyfenozide residues detected by LC-UV. Overall average recovery of methoxyfenozide from all matrices, determined by HPLC-UV, was 95.5%. Table 33 summarizes the recovery data for all matrices by HPLC-UV and Table 34 compares the recovery data obtained by HPLC-UV and HPLC-MS on the same peach and plum samples. The average recovery by the HPLC-MS confirmatory method, 96%, is in good agreement with the average recovery, 101%, by HPLC-UV. The LOQ for the method was 0.02 mg/kg for all matrices.

Table 24. Recovery data for methoxyfenozide in stone fruit matrices (Zhang and Doyle, 1999).

Matrix	Fortification, mg/kg	Recovery results			
		Range (%)	Average (%)	% RSD	n
Method validation (HPLC-UV)					
Cherries	0.02-0.5	84–110	94	9.5	5
Peaches	0.02-1.0	91–110	98	7.6	5
Plums	0.02-1.0	86–120	99	13	5
Prunes	0.02-1.0	91–96	94	2.2	5
Prune juice	0.02-1.0	87–100	93	7.0	5
		Overall	96	8.3	25
Confirmatory method (HPLC-MS)					
Peaches	0.02-0.5	90–100	96.	6.0	4
Plums	0.02-0.5	93–99	96	3.1	4
		Overall	96	4.2	8

Table 25. Comparison of HPLC-UV and HPLC-MS recovery data for methoxyfenozide in stone fruit matrices (Zhang and Doyle, 1999).

Matrix	Fortification, mg/kg	% Recovery by HPLC-MS	% Recovery by HPLC-UV
Peaches	0.02	100	98
Peaches	0.05	97	110
Peaches	0.2	94	95
Peaches	0.5	90	91
Plums	0.02	94	119
Plums	0.05	97	100
Plums	0.2	99	99
Plums	0.5	93	92
	n =	8	8
	Overall average =	96.0	101
	RSD % =	4.4	9.5

Methods were also developed for the determination of methoxyfenozide and the major metabolite RH-1518 in animal tissue matrices.

Method TR-34-99-11 (Wickremesinhe and Deakyne, 1999, report No 34-99-11) was for determination of methoxyfenozide in all hen matrices and the glucuronide conjugate of the A-ring phenol in egg and liver. Residues in chicken fat were extracted with hexane using a heated sonicator.

The extract was then partitioned with acidic methanol. Residues in chicken muscle were extracted with acidic methanol. Extracts from both matrices were then partitioned into dichloromethane and further purified using basic alumina column chromatography and carbon solid-phase extraction (SPE). Quantification was by HPLC-UV for fat samples and liquid chromatography-mass spectrometry (HPLC-MS) for muscle samples (and fat, if interferences were found by HPLC-UV).

Residues of methoxyfenozide and the glucuronide conjugate were extracted from liver and egg with methanol, which was cleaned up by partition with hexane. The methanolic fraction was then split into two equal portions. A dichloromethane partition, followed by basic alumina column chromatography and carbon SPE further was used to purify one portion. This fraction was analyzed by HPLC-UV or HPLC-MS for methoxyfenozide. The other fraction was concentrated and purified by C-18 and carbon SPE, then analyzed by HPLC-MS for the glucuronide conjugate. The LOQ for both analytes in all matrices was 0.01 mg/kg for both HPLC-UV and HPLC-MS. Table 26 summarizes the recoveries from poultry matrices.

Table 26. Summary of recovery data for methoxyfenozide and RH-1518 (glucuronide conjugate) in poultry (Wickremesinhe and Deakyne, 1999).

Matrix	Spiking level (mg/kg)	% Recovery				
		Average	Range	Standard deviation	% RSD	n
Methoxyfenozide						
Eggs	0.01-0.10	94	81-109	5.9	6.3	18
Fat	0.01-0.10	81	70-90	6.3	7.7	12
Liver	0.01-0.10	93	76-120	9.9	13	12
Muscle	0.01-0.10	93	73-110	10	11	16
Metabolite RH-1518						
Eggs	0.01-0.10	89	66-100	9.1	10	12
Liver	0.01-0.10	95	84-110	9.9	10	12

Several methods were presented for the purposes of MRL enforcement in both plant and animal matrices. They were modifications of the above described methods. For example, Method TR-34-98-87, a tolerance enforcement method, (Stein and Wu, 1998, report No. 34-98-87) was derived from Method 00458 (TR 34-95-55). Methoxyfenozide residues were extracted from apples, pears and apple fractions by blending with acidic methanol/water. The extract was purified by hexane and dichloromethane liquid-liquid partitions. Apple and apple pomace extracts were further cleaned up by Florisil and silica column chromatography. Analysis of pears required a C-18 SPE clean-up step but this was optional for all the other matrices. Quantification was by isocratic HPLC with UV detection. The LOQ was 0.01mg/kg for all matrices. Control samples were fortified at different levels and the average fortification recoveries are summarized in Table 27.

Table 27. Summary of recovery data for validation of the enforcement method TR 34-98-87 (Stein and Wu, 1998).

Matrix	Fortification mg/kg	Recovery results				
		Mean (%)	Range	Standard deviation	% RSD	n
Apple fruit	0.01-10.7	93	71-130	10	11	40
Pear fruit	0.01-0.5	94	57-150	17	18	46
Wet apple pomace	0.01-10.7	94	66-100	19	20	19
Apple juice	0.01-0.5	98	86-110	6.5	6.6	26

To confirm that residues determined with HPLC-UV detection were methoxyfenozide and not due to interference, a method for HPLC with tandem mass spectrometry (HPLC-MS/MS) was developed. Acetonitrile/water 54/56 (v/v) with 0.1% formic acid was used as the mobile phase and the HPLC column was a Hypersil C-18. Each sample set consisted of a control and five fortifications, ranging from 0.01 to 0.25 mg/kg. Injections were made on HPLC-MS/MS and HPLC-UV to provide data for recovery comparison. Confirmatory data showed good agreement with the HPLC-UV quantification and are summarized in Table 28.

Table 28. Comparison of results obtained by HPLC-UV and HPLC-MS/MS determination techniques (Stein and Wu, 1998).

Matrix	Fortification mg/kg	n	HPLC-UV		HPLC-MS/MS	
			% Recovery	% RSD	% Recovery	% RSD
Apple fruit	0.01-1.0	6	88.4	5.19	94.5	6.76
Wet apple pomace	0.01-0.25	5	95.3	11.0	96.1	5.35
Apple juice	0.01-2.5	5	89.0	5.51	95.2	6.73
Pear fruit	0.01-0.5	4	105.4	20.6	95.7	2.64

Other methods were validated for maize (Bender, 2000a, report No. 34-00-56) and stone fruit (Bender, 2000b, report No. 34-00-111).

Methods for the enforcement of MRLs in cattle and poultry commodities were presented. Method TR-34-98-106 (Chen, Desai, Hofmann and Kurilla, 1998, report No. 34-98-106) was intended for the determination of methoxyfenozide residues in milk, fat, muscle, kidney and liver. Additionally, residues of the glucuronide metabolite (RH-1518) were determined in bovine liver and kidney.

Methoxyfenozide residues were extracted from fat with acidic methanol, using sonication. After partitioning with hexane, the methanolic extract was concentrated and further purified by basic alumina column chromatography and carbon SPE clean-up. Residues were quantified by HPLC-UV.

Residues were extracted from milk and muscle with dichloromethane, using matrix solid-phase dispersion (MSPD), using C-18 silica. After concentration, the residues were further purified by neutral alumina column chromatography and carbon SPE clean-up. Residues of methoxyfenozide were quantified by HPLC-UV.

Residues were extracted from liver and kidney with methanol, using a tissue homogenizer and vacuum filtration. After hexane partition to remove non-polar compounds, the extract was split into two equal portions. For the glucuronide metabolite, RH-1518, one portion was concentrated and then purified on C-18 and carbon SPE columns. Methoxyfenozide in the second portion was purified with a liquid-liquid partition, basic alumina and carbon SPE column clean-ups. After clean-up, both compounds were quantified by HPLC-MS.

Average recovery for the method was determined from the manufacturer's and contract laboratory validation data and recovery determinations performed concurrently with the residue analysis. Average recoveries of methoxyfenozide were 88% from milk, 84.9% from fat, 79.4% from muscle, 105% from liver, and 104% from kidney. Average recoveries of the metabolite, RH-1518, were 91.5% from liver and 94.7% from kidney. The LOQ for methoxyfenozide in all matrices was 0.01 mg/kg, while the LOQ for the glucuronide metabolite (RH-1518), found only in liver and kidney, was 0.02 mg/kg. Limits of detection were estimated as 0.003 mg/kg for parent and 0.006 mg/kg for RH-1518. Table 29 provides a summary of the recovery data for each of the animal matrices.

Table 29. Summary of recovery data for US MRL enforcement method for bovine commodities (Chen, Desai, Hofmann and Kurilla, 1998).

Matrix	Fortification mg/kg	% Recovery				
		Mean	Range	Standard deviation	% RSD	n
Methoxyfenozide						
Milk	0.01-1.25	88	53-120	12	14	93
Fat	0.01-1.0	85	63-110	10	12	56
Muscle	0.01-1.0	79	61-120	13	16	55
Liver	0.01-1.0	100	93-120	7.0	6.7	36
Kidney	0.01-1.0	100	93-120	7.4	7.1	22
Metabolite RH-1518						
Liver	0.02 [1/] to 5.0	92	75-100	9.2	10	30
Kidney	0.02 [2/] to 1.0	95	69-110	13	13	20

[1/] Recoveries at 0.01 mg/kg fortification of 71, 80, 112 and 100% were not included.
[2/] Recoveries at 0.01 mg/kg fortification of 75 and 80% were not included.

Four samples from the goat metabolism study were subjected to analysis using this method. The ^{14}C-RH-1518 (metabolite) and ^{14}C-methoxyfenozide quantified by HPLC and by liquid

scintillation counting agreed closely with the metabolism study values. The validation results are provided in Table 30.

Table 30. Radio-validation summary results for bovine commodities (Chen, Desai, Hofmann and Kurilla, 1998).

Matrix	Methoxyfenozide expected [1/] mg/kg	Methoxyfenozide found mg/kg	RH-1518 expected [1/] mg/kg	RH-1518 found mg/kg
Fat	0.043	0.040 [2/]	NA	NA
Milk	0.009	0.006 [2/]	NA	NA
Liver	0.007	0.0066	0.075	0.078 [3/]
Kidney	0.004	0.0034 [3/]	0.049	0.02 [3/]

NA = not analyzed.
[1/] Found by ^{14}C quantification in the metabolism report.
[2/] Found by HPLC-UV quantification using this method.
[3/] ^{12}C-RH-1518 residues, found by HPLC-MS quantification using this method, were doubled to reflect ^{13}C- analyte contribution.

Mass spectrometric confirmatory methods were developed for all matrices. For fat, milk and muscle, HPLC-MS was used to confirm the methoxyfenozide residues detected by HPLC-UV. For liver and kidney, HPLC-MS/MS was used to confirm the residues of methoxyfenozide and RH-1518 detected by HPLC-MS. Agreement between results from HPLC-UV and HPLC-MS, as well as between HPLC-MS and HPLC-MS/MS, was good. Table 31 summarizes the recoveries obtained from fortified samples.

Table 31 Comparison of recovery determined by initial HPLC-UV and HPLC-MS techniques with those determined by confirmatory methods (Chen, Desai, Hofmann and Kurilla, 1998).

Matrix	Fortification mg/kg	Initial	Confirmatory	Initial	Confirmatory
		% Recovery by HPLC-UV	% Recovery by HPLC-MS	% Recovery by HPLC-MS	% Recovery by HPLC-MS/MS
Milk	0.01	78	80		
	0.05	79	76		
Fat	0.01	81	72		
	0.50	91	120		
Muscle	0.01	58	65		
	0.50	100	96		
Liver	0.01			110	100
	0.01			96	110
	0.50			110	110
Kidney	0.01			110	100
	0.01			96	110
	0.10			110	120
Overall	Average =	81.27	84	105	109
	Standard deviation =	13.99	19	6.8	6.4
	% RSD =		22	6.5	5.9
	n =	6	6	6	6

Method TR 34-00-40 (Deakyne, Desai and Hofmann, 2000c, report No. 34-00-40), was developed as an enforcement method for the determination of residues of methoxyfenozide in all chicken matrices and its metabolite, the glucuronide RH-1518, in chicken liver and egg. Residues of methoxyfenozide in chicken fat were extracted with hexane using a heated sonicator. The residues were then partitioned into acidic methanol. Residues in chicken muscle were extracted with acidic methanol. Extracts from both matrices were then partitioned into dichloromethane and further purified using basic alumina column chromatography and carbon SPE. Quantification was by HPLC using UV detection for fat samples and by HPLC-MS for muscle samples.

Residues in liver and egg matrices were extracted with methanol and initially cleaned up by partition with hexane. The methanolic fraction was split into two equal portions. A dichloromethane partition of one portion was then followed by basic alumina column chromatography and carbon SPE further clean-up. This fraction was analyzed by HPLC-UV or HPLC-MS for methoxyfenozide. The other portion was analyzed directly by HPLC-MS for RH-1518.

The method was validated (Deakyne, Desai and Hofmann, 2000c, report No. 34-00-40), using samples derived from a meat and egg magnitude of residue study (Bender, 2000c, TR 34-00-33), which supported the preliminary method validation data given in the report on poultry commodities (Wickremesinhe and Deakyne, 1999, report No. TR 34-99-11). The method LOQ for both methoxyfenozide and RH-1518 in all matrices was 0.01 mg/kg. Average recoveries of methoxyfenozide were 86 ± 8.4% from egg, 85 ± 7.7% from fat, 93 ± 10% from liver and 92 ± 9.4% from muscle. The average recoveries of RH-1518 from egg and liver were 88 ± 12% and 96 ± 12%, respectively. The recovery data are summarized in Table 32.

Table 32. Summary of recovery data for methoxyfenozide and RH-1518 from poultry matrices (Deakyne, Desai and Hofmann, 2000c).

Matrix	Fortification mg/kg	% Recovery				
		Average	Range	Standard deviation	% RSD	n
Methoxyfenozide						
Eggs	0.01-1.0	86	63-110	8.4	9.7	54
Fat	0.01-1.0	85	70-98	7.7	9.1	18
Liver	0.01-0.10	93	76-100	10	10	18
Muscle	0.01-0.10	92	73-110	9.4	10	22
Metabolite RH-1518						
Eggs	0.01 [1/]-0.50	88	61-110	12	14	50
Liver	0.01 [2/]-0.10	96	73-120	12	12	22

[1/] Recoveries at 0.01 mg/kg were 79, 55, 61, 82, 59, 98, 109, 92, 101, 88, 92, 95, 88, 83, 101 and 98%.
[2/] Recoveries at 0.01 mg/kg were 76, 86, 100, 110, 82, 116, and 100%.

Confirmatory analyses using MS/MS detection were conducted using the same extracts isolated by the primary residue methods for egg, fat, muscle, and liver samples. A comparison of the HPLC-MS/MS results with those found by HPLC-MS is shown in Table 33. Results obtained by HPLC-MS/MS compared very well with the HPLC-MS quantification of the same residues.

Table 33. Comparison of HPLC-MS and HPLC-MS/MS results for the same extracts (Deakyne, Desai and Hofmann, 2000c).

Matrix	n	Methoxyfenozide		RH-1518	
		HPLC-MS/MS µg found	HPLC-MS µg found	HPLC-MS/MS µg found	HPLC-MS µg found
Fat	6	0.11-12.	0.11-13.		
Muscle	6	0.13-1.4	0.13-1.4		
Liver	6	0.00-0.77	0.078-0.78	0.069-0.77	0.058-0.74
Eggs	6	0.056-5.7	0.062-6.1	0.034-5.2	0.042-5.6

The US FDA standardized multi-residue methods (PAM I – Pesticide Analytical Methods) were evaluated to assess if they would be adequate to detect residues of methoxyfenozide in the food supply (O'Donnell, A., 1998, report No. 34-97-126; Conrath, 2001, report No. 34-01-33).

Multi-residue Protocol A was not applicable to the analysis, as methoxyfenozide is not an *N*-methylcarbamate. Protocol B was not applicable to the analysis of methoxyfenozide, as it is neither an acid nor a phenol. In the Protocol C evaluation, methoxyfenozide showed long GC retention times and low sensitivity when determined by DB-17/NPD and DB-225/ECD systems. The DB-1/ECD combination provided the highest sensitivity, with acceptable retention times, using the higher column temperature allowed by Level II. Protocol D was not suitable, as the analyte was only detected by ECD under Protocol C and the recovery of methoxyfenozide residues was less than 30% through the elution schemes of Protocol E. Protocol E was not suitable for analysis of methoxyfenozide, as it was recovered through Florisil, which is a requirement for method validation. Protocol F was not conducted, as the recovery of methoxyfenozide residues was <30% through the elution schemes of Protocol E. Therefore it was concluded that PAM multi-residue screening methods are not suitable for the detection of methoxyfenozide residues.

Determination of the methoxyfenozide metabolites RH-117236, RH-141518, RH-151055, and RH-152072 was performed according to the US FDA Multi-Residue Method Testing Guidelines in PAM, Volume I, Appendix II (1/94). As none of the analytes was a substituted urea, testing under Protocol G was not required. RH-117236, RH-141518, RH-151055 and RH-152072 are naturally

fluorescent and all but RH-152072 produced detectable peaks when injected on the HPLC system described in Protocol A. The response generated for RH-141518 was not sufficient to warrant continuing through Protocol A. RH-117236 and RH-151055 produced enough response but neither could be recovered from the Protocol A celite/charcoal clean-up column.

Under Protocol B, although RH-117236 and RH-112485 (the methyl ether) both chromatographed acceptably under Level II conditions, the methylation of RH-117236 did not produce RH-112485. It yielded a peak with a different retention time. At the nominal temperature of 200°C (Level I), under Protocol C, RH-141518, RH-151055 and RH-152072 did not chromatograph acceptably on any of the column-detector combinations tested, as specified by the Appendix II multi-residue testing guidelines. They all chromatographed acceptably under the Level II conditions listed in DG-10. RH-117236, RH-141518, RH-151055 and RH-152072 do not elute from Florisil columns, using the methods specified in either Protocol D or Protocol E; therefore no further testing under Protocol D or Protocol E was performed. Based on the results, it was concluded that PAM multi-residue screen methods are not suitable for the detection of residues of any of the following methoxyfenozide metabolites: RH-117-236, RH-141518, RH-151055, and RH-152072.

Table 34 provides a summary of all submitted analytical methods for the determination of methoxyfenozide and its metabolites. The methods presented were used in the studies provided to the Meeting, to support the proposed MRLs for methoxyfenozide. The enforcement methods (E) are also included in the summary table. Methods for the metabolite RH-1518, the glucuronide of methoxyfenozide, are summarized in Table 35.

Table 34. Summary of residue methods for methoxyfenozide

Matrix	Method		Fortification	n	Methoxyfenozide recovery (%)				LOQ	Report No.
	No.	Type	mg/kg		Min.	Max.	Ave.	RSD	mg/kg	
Almond kernels	34-00-107 (E)	HPLC-UV	0.02-0.10	5	100	110	110	5.2	0.02	34-00-107
Almond kernels	34-00-107 (E-ILV)	HPLC-UV	0.02-0.1	4	83	90	87	2.7	0.02	34-01-06
Almond hulls	34-00-107 (E)	HPLC-UV	0.02-0.10	3	81	98	90	9.2	0.02	34-00-107
Apples	00470	HPLC-MS	0.05-0.5	10	92	110	100	5.4	0.05	34-98-169
Apples	00551	HPLC-MS	0.05-0.5	10	86	101	94	4.3	0.05	34-99-172
Apples	00458	HPLC-UV	0.01-1.0	23	79	130	94	12	0.01	34-95-55
Apples	00458/M001	HPLC-UV	0.05-5.0	15	92	100	97	3.1	0.05	34-98-173
Apples	00458 (ILV)	HPLC-UV	0.01-1.0	12	70	99	84	11	0.01	7747AgReg
Apples	34-98-87 (E)	HPLC-UV	0.01-10.7	41	71	130	93	11	0.01	34-98-87
Apples	34-98-87 (E-ILV)	HPLC-UV	0.01-1.5	4	74	93	83	13	0.01	34-98-104
Apples	00458/M001 (ILV)	HPLC-UV	0.05-0.5	10	91	72	100	9.2	0.05	34-00-01
Apple sauce	00470	HPLC-MS	0.05-0.5	6	79	100	89	9.4	0.05	34-98-169
Apple sauce	00551	HPLC-MS	0.05-0.5	6	89	98	93	5.2	0.05	34-99-172
Apple juice	00470	HPLC-MS	0.05-0.5	6	98	110	100	3.7	0.05	34-98-169
Apple juice	00551	HPLC-MS	0.05-0.5	6	94	100	97	2.6	0.05	34-99-172
Apple juice	34-98-87 (E)	HPLC-UV	0.01-0.5	26	86	110	98	6.5	6.6	34-98-87
Apple pomace	00470	HPLC-MS	0.05-0.5	10	74	100	89	7.8	0.05	34-98-169
Apple pomace	00551	HPLC-MS	0.05-0.5	10	81	97	90	7.0	0.05	34-99-172
Apple pomace	34-98-87 (E)	HPLC-UV	0.01-10.7	19	66	110	94	20	0.01	34-98-87
Apple, dried	00470	HPLC-MS	0.05-0.5	10	78	110	88	9.6	0.05	34-98-169
Apple, dried	00551	HPLC-MS	0.05-0.5	10	82	95	89	4.1	0.05	34-99-172
Avocado	00458/M002 E001	HPLC-UV	0.2-2.0	10	74	93	87	6.4	0.2	34-99-174
Bovine fat	34-98-106 (E)	HPLC-UV	0.01-1.0	56	63	110	85	11	0.01	34-98-106
Bovine kidney	34-98-106 (E)	HPLC-MS	0.01-1.0	22	93	120	100	7.1	0.01	34-98-106
Bovine liver	34-98-106 (E)	HPLC-MS	0.01-1.0	36	93	120	110	6.7	0.01	34-98-106
Bovine liver	34-98-106 (ILV)	HPLC-MS	0.01-0.05	4	97	110	110	7.8	0.01	34-98-139
Bovine muscle	34-98-106 (E)	HPLC-UV	0.01-1.0	55	61	120	79	16	0.01	34-98-106
Brassicas	34-99-74 (E)	HPLC-UV	0.02-0.2	22	78	99	88	6.6	0.02	34-99-74
Celery	34-99-74 (E)	HPLC-MS	0.02-2.0	11	72	110	95	11.	0.02	34-99-74
Celery	34-99-74 (ILV)	HPLC-MS	0.02-20	4	91	97	94	2.0	0.02	34-99-82
Cherries	34-99-26	HPLC-UV	0.02-0.5	5	84	110	93	9.5	0.02	34-99-26
Cherries	34-00-109 (E)	HPLC-UV	0.02-2.0	17	72	120	94	12	0.02	34-00-109
Maize (sweet corn) kernels	34-99-45	HPLC-UV	0.02-0.2	15	69	110	89	13	0.02	34-99-45

Matrix	Method		Fortification	n	Methoxyfenozide recovery (%)				LOQ	Report No.
	No.	Type	mg/kg		Min.	Max.	Ave.	RSD	mg/kg	
Maize (field) grain	34-00-38 (E)	HPLC-UV	0.02-1.0	33	70	94	84	8.5	0.02	34-00-38
Maize (sweet corn) grain	34-00-38 (E)	HPLC-UV	0.02-0.5	28	60	110	79	13	0.02	34-00-38
Maize (field) forage	34-00-38 (E)	HPLC-UV	0.02-15	23	74	130	96	12	0.02	34-00-38
Maize (sweet corn) forage	34-00-38 (E)	HPLC-UV	0.02-40	15	84	120	97	8.8	0.02	34-00-38
Maize fodder	34-99-54	HPLC-UV	0.02-0.50	14	69	120	92	16	0.02	34-99-54
Maize (field) fodder	34-00-38 (E)	HPLC-UV	0.04-120	23	50	110	91	13	0.04	34-00-38
Maize (sweet corn) fodder	34-00-38 (E)	HPLC-UV	0.04-75	13	85	110	95	6.6	0.04	34-00-38
Maize, processed	34-00-38 (E)	HPLC-UV	0.02-0.5	8	70	120	91	19	0.02	34-00-38
Cotton seed	34-96-88 (E)	HPLC-UV	0.01-0.5	60	59	130	91	16	0.01	34-96-88
Cotton seed	34-96-168 (E-ILV)	HPLC-UV	0.01-4.0	30	69	110	93	11	0.01	34-96-168
Cotton seed meal	34-96-88 (E)	HPLC-UV	0.01-0.25	16	61	110	93	13	0.01	34-96-88
Cotton seed hulls	34-96-88 (E)	HPLC-UV	0.01-0.25	18	74	140	100	16	0.01	34-96-88
Cotton seed oil	34-96-88 (E)	HPLC-UV	0.01-0.25	20	66	120	84	14	0.01	34-96-88
Cotton gin trash	34-96-88 (E)	HPLC-UV	0.05-20	31	55	170	78	27	0.05	34-96-88
Eggs	34-99-11	HPLC-UV	0.01-0.10	18	81	110	94	6.3	0.01	34-99-11
Eggs	34-99-11 (ILV)	HPLC-UV	0.01-0.05	4	74	96	86	12	0.01	34-00-45
Eggs	34-00-40 (E)	HPLC-UV	0.01-1.0	54	63	110	86	9.7	0.01	34-00-40
Grapes	00470	HPLC-MS	0.05-0.5	6	97	110	100	5.0	0.05	34-98-169
Grapes	00551	HPLC-MS	0.05-0.5	6	84	98	91	5.6	0.05	34-99-172
Grapes	00458	HPLC-UV	0.01-0.25	11	69	95	88	10	0.01	34-95-55
Grapes	00458/M001	HPLC-UV	0.05-0.5	15	94	100	98	2.5	0.05	34-98-173
Grapes	00458/M001 (ILV)	HPLC-UV	0.05-1.0	10	91	110	99	6.1	0.05	34-00-01
Grapes	00458 (ILV)	HPLC-UV	0.01-1.0	12	78	110	92	11	0.01	7750/AgReg
Grapes	34-99-74 (E)	HPLC-UV	0.02-1.0	20	75	100	88	8.3	0.02	34-99-74
Grape must	00470	HPLC-MS	0.05-0.5	6	95	110	100	6.5	0.05	34-98-169
Grape must	00551	HPLC-MS	0.05-0.5	6	90	100	95	5.1	0.05	34-99-172
Grape must	00458/M001	HPLC-UV	0.05-0.5	15	80	110	95	6.8	0.05	34-98-173
Grape, wine	00470	HPLC-MS	0.05-0.5	6	98	110	100	5.7	0.05	34-98-169
Grape, wine	00551	HPLC-MS	0.05-0.5	6	89	97	93	4.0	0.05	34-99-172
Grape, wine	00458/M001	HPLC-UV	0.05-0.5	15	84	100	95	5.9	0.05	34-98-173
Grape, raisins	00470	HPLC-MS	0.05-0.5	10	93	100	97	3.5	0.05	34-98-169
Grape, raisins	00551	HPLC-MS	0.05-0.5	10	75	95	88	7.6	0.05	34-99-172
Leafy vegetables	34-99-74 (E)	HPLC-UV	0.02-1.0	43	71	110	87	9.8	0.02	34-99-74
Mandarin preserve	00470	HPLC-MS	0.05-0.5	6	89	100	96	5.5	0.05	34-98-169
Mandarin pulp	00470	HPLC-MS	0.05-0.5	6	93	110	100	6.0	0.05	34-98-169
Mandarin pulp	00551	HPLC-MS	0.05-0.5	6	97	100	98	1.7	0.05	34-99-172
Mandarin peel	00470	HPLC-MS	0.05-0.5	6	81	93	89	4.8	0.05	34-98-169
Mandarin peel	00551	HPLC-MS	0.05-0.5	6	86	95	91	3.4	0.05	34-99-172
Milk	34-98-106 (E)	HPLC-UV	0.01-1.25	93	53	120	88	14	0.01	34-98-106
Orange	00458/M002	HPLC-UV	0.1-1.0	10	86	100	94	5.4	0.10	34-99-173
Orange	00458/M002 (ILV)	HPLC-UV	0.1-1.0	10	74	90	80	7.5	0.10	34-00-01
Orange marmalade	00470	HPLC-MS	0.05-0.5	6	89	100	96	6.0	0.05	34-98-169
Orange marmalade	00551	HPLC-MS	0.05-0.5	6	88	99	93	4.6	0.05	34-99-172
Orange pulp	00470	HPLC-MS	0.05-0.5	6	89	99	94	3.7	0.05	34-98-169
Orange pulp	00551	HPLC-MS	0.050.5	6	96	100	98	2.3	0.05	34-99-172
Orange pulp	00458/M002	HPLC-UV	0.05-0.5	10	79	100	94	7.2	0.05	34-99-173
Orange peel	00470	HPLC-MS	0.05-0.5	6	81	96	89	6.9	0.05	34-98-169
Orange peel	00551	HPLC-MS	0.05-0.5	6	84	94	90	4.1	0.05	34-99-172
Orange juice	00470	HPLC-MS	0.05-0.5	6	70	94	85	12	0.05	34-98-169
Orange juice	00551	HPLC-MS	0.05-0.5	6	84	96	92	5.2	0.05	34-99-172
Peaches	00470	HPLC-MS	0.05-0.5	6	95	110	100	3.8	0.05	34-98-169
Peaches	00551	HPLC-MS	0.05-0.5	6	93	97	96	2.1	0.05	34-99-172
Peaches	00458/M001	HPLC-UV	0.05-0.5	15	94	110	100	4.9	0.05	34-98-173

Matrix	Method		Fortification	n	Methoxyfenozide recovery (%)				LOQ	Report No.
	No.	Type	mg/kg		Min.	Max.	Ave.	RSD	mg/kg	
Peaches	00458 (ILV)	HPLC-UV	0.01-1.0	12	77	110	92	9	0.01	7751/AgReg
Peaches	34-99-26	HPLC-UV	0.02-1.0	5	91	110	98	7.6	0.02	34-99-26
Peaches	34-00-109 (E)	HPLC-UV	0.02-2.0	19	75	110	97	10	0.02	34-00-109
Peach preserve	00551	HPLC-MS	0.05-0.5	6	92	99	96	2.5	0.05	34-99-172
Pears	00470	HPLC-MS	0.05-0.5	6	98	110	100	6.0	0.05	34-98-169
Pears	00551	HPLC-MS	0.05-0.5	6	84	96	88	4.7	0.05	34-99-172
Pears	34-98-87 (E)	HPLC-UV	0.01-0.5	46	57	150	94	19	0.01	34-98-87
Pecan nuts	34-00-107 (E)	HPLC-UV	0.02-0.5	5	98	120	110	8.9	0.02	34-00-107
Peppers	00470	HPLC-MS	0.05-0.5	6	87	94	91	2.9	0.05	34-98-169
Peppers	00551	HPLC-MS	0.05-0.5	6	79	88	85	4.1	0.05	34-99-172
Peppers	34-99-74 (E)	HPLC-UV	0.02-0.5	20	72	100	90	12	0.02	34-99-74
Plums	34-99-26	HPLC-UV	0.02-1.0	5	86	120	99	13	0.02	34-99-26
Plums	34-00-109 (E)	HPLC-UV	0.02-2.0	13	81	120	100	11	0.02	34-00-109
Prunes	34-99-26	HPLC-UV	0.02-1.0	5	91	96	94	2.2	0.02	34-99-26
Prunes	34-00-109 (E)	HPLC-UV	0.02-1.0	6	91	99	95	2.8	0.02	34-00-109
Prune juice	34-99-26	HPLC-UV	0.02-1.0	5	87	100	93	7.0	0.02	34-99-26
Prune juice	34-00-109 (E)	HPLC-UV	0.02-1.0	5	87	100	93	6.9	0.02	34-00-109
Poultry fat	34-99-11	HPLC-UV	0.01-0.10	12	70	90	81	7.7	0.01	34-99-11
Poultry fat	34-00-40 (E)	HPLC-UV	0.01-1.0	18	70	98	85	9.1	0.01	34-00-40
Poultry liver	34-99-11	HPLC-UV	0.01-0.10	12	76	120	93	13	0.01	34-99-11
Poultry liver	34-00-40 (E)	HPLC-UV	0.01-0.10	18	76	100	93	11	0.01	34-00-40
Poultry muscle	34-99-11	HPLC-MS	0.01-0.10	16	73	110	93	11	0.01	34-99-11
Poultry muscle	34-00-40 (E)	HPLC-MS	0.01-0.10	22	73	110	92	10	0.01	34-00-40
Prune juice	34-00-109 (E)	HPLC-UV	0.02-1.0	5	87	100	93	6.9	0.02	34-00-109
Tomatoes	00470	HPLC-MS	0.05-0.5	6	93	100	96	4.5	0.05	34-98-169
Tomatoes	00551	HPLC-MS	0.05-0.5	6	93	100	96	2.5	0.05	34-99-172
Tomatoes	34-99-74 (E)	HPLC-UV	0.02-0.5	25	83	98	92	4.2	0.02	34-99-74
Tomato juice	00470	HPLC-MS	0.05-0.5	6	100	110	110	2.5	0.05	34-98-169
Tomato juice	00551	HPLC-MS	0.05-0.5	6	96	99	97	1.2	0.05	34-99-172
Tomato, canned	00551	HPLC-MS	0.05-0.5	6	97	100	99	2.2	0.05	34-99-172
Tomato paste	00470	HPLC-MS	0.05-0.5	6	77	110	92	17	0.05	34-98-169
Tomato paste	00551	HPLC-MS	0.05-0.5	10	96	110	100	2.9	0.05	34-99-172
Wheat grain	00458/M002 E001	HPLC-UV	0.05-0.5	10	89	110	95	4.9	0.05	34-99-174

(ILV) = independent laboratory validation.
(E) = enforcement method.

Table 35. Summary of residue methods for the methoxyfenozide metabolite, RH-1518.

Matrix	Method		Fortification	n	Methoxyfenozide recovery (%)				LOQ	Report No.
	No.	Type	mg/kg		Min	Max	Mean	RSD	mg/kg	
Eggs	34-99-11	HPLC-MS	0.01-0.10	12	66	100	89	10	0.01	34-99-11
Eggs	34-99-11 (ILV)	HPLC-MS	0.01-0.05	4	73	92	83	9.6	0.01	34-00-45
Eggs	34-00-40 (E)	HPLC-MS	0.01-0.50	50	61	110	88	14	0.01	34-00-40
Bovine liver	34-98-106 (E)	HPLC-MS	0.01-5.0	30	75	110	92	10	0.01	34-98-106
Bovine liver	34-98-106 (E-ILV)	HPLC-MS	0.02-0.05	5	66	94	83	15	0.02	34-98-139
Bovine kidney	34-98-106 (E)	HPLC-MS	0.01-1.0	20	69	110	95	13	0.01	34-98-106
Poultry liver	34-99-11	HPLC-MS	0.01-0.10	12	84	110	95	10	0.01	34-99-11
Poultry liver	34-00-40 (E)	HPLC-MS	0.01-0.10	22	73	120	96	12	0.01	34-00-40

Stability of pesticide residues in stored analytical samples

Residue storage stability studies were conducted in a variety of substrates, including animal tissues, soil and plant commodities. Control samples were fortified with known concentrations of methoxyfenozide and then placed in frozen storage at approximately -20 C. The fortified samples were analyzed periodically for residues of methoxyfenozide, using the same analytical method as that used for the corresponding residue field trial or processing samples. Sample extracts were not stored prior to analysis.

Apples and apple products

Control samples of apple fruit (RAC), apple juice and apple wet pomace were fortified with methoxyfenozide at a concentration of 1.04 mg/kg and the fortified samples were stored frozen at approximately -20°C pending analysis (Bender, 1998a, report No. 34-98-27). The juice and wet pomace samples were stored for intervals that corresponded approximately to the sample storage period that occurred in the apple processing study (Bender and Bergin, 1998, report No. 34-98-20). The apple fruit samples were fortified and stored for up to one year prior to analysis. All samples were analyzed at various times during storage and at the end of the storage period. Recoveries were determined from fresh fortification made on the day of the analysis.

Concentrations of methoxyfenozide were determined in all matrices by HPLC with UV detection. Apple samples were analyzed using method 34-95-55 or a variant of it (Stein *et al.*, 1997, report No. 34-96-209). The LOQ was 0.01 mg/kg for all matrices. Recoveries from stored samples were calculated by dividing the concentrations found in stored samples by the actual amount fortified prior to storage (Table 36).

Table 36. Frozen storage stability data for methoxyfenozide in apple commodities (Bender, 1998a).

Matrix	Storage period, days	Fortification, mg/kg	Apparent remainder [1/], %	Corrected remainder [2/], %	Reference
Apple fruit	0	1.04	95	100	34-98-27
	29		81	90	
	53		91	96	
	59		84	92	
	95		72	80	
	135		85	88	
	170		76	79	
	205		88	92	
	244		85	86	
	283		82	83	
	315		82	82	
	365		83	87	
Apple juice	0	1.04	98	100	34-98-27
	31		99	100	
	67		100	100	
	102		100	100	
	141		95	94	
	176		96	95	
	210		100	100	
	251		89	91	
	283		96	99	
Apple wet pomace	0	1.04	91	100	34-98-27
	48		80	91	
	84		84	95	
	119		81	92	
	157		86	94	
	192		86	93	
	226		82	90	
	270		75	80	
	302		82	88	

[1/] Values represent average of two determinations.
[2/] Residues corrected for procedural recovery.

Tomatoes

The stability of residues of methoxyfenozide in tomato fruit, under frozen storage conditions, was initially evaluated for a period of six months (Bergin, 1999a, report No. 34-99-72). A subsequent study was initiated, extending to a period of twelve months, in order to support results from residue field trials where samples had been stored frozen up to about seven months before analysis (Filchner, 2000a, report No. 34-99-200). Control samples of tomato fruit were fortified with methoxyfenozide at 1.0 mg/kg and the fortified samples were stored frozen at approximately -20°C, under the same conditions as the samples from the residue trials.

In the first storage stability study, representative samples stored for 0, 23, 51, 124, 154 and 182 days were analyzed. In the second storage stability study, samples stored for 288 and 372 days were analyzed. Samples were analyzed using analytical method TR 34-98-186 (Deakyne, 1998, Report 34-98-186), finalized as TR 34-99-74. The method had an LOQ of 0.02 mg/kg. Recoveries from stored samples were calculated by dividing the concentrations found in stored samples by the amount of fortification and were corrected for procedural recoveries, which ranged from 89% to 102%. Results are presented in Table 37.

Table 37. Frozen storage stability data for methoxyfenozide in tomato samples (Bergin, 1999a).

Matrix	Storage period, days	Fortification, mg/kg	Apparent remainder [1/], %	Corrected remainder [2/], %	Reference
Tomatoes	0	1.0	90	100	34-99-72
	23		85	95	
	51		90	100	
	96		90	100	
	124		88	98	
	154		83	90	
	182		92	100	
	288		98	96	
	372		94	93	

[1/] Values represent average of two analyses.
[2/] Residues corrected for procedural recovery.

Lettuce

Storage stability studies of methoxyfenozide in frozen head lettuce were conducted (Burnett, 2000, report No. 34-00-31; Schuck, 1999, report No. 34-99-79). Control samples of head lettuce were fortified with methoxyfenozide at a concentration of 1 mg/kg and the fortified samples were stored frozen at approximately -20°C, under the same conditions as the samples from the residue trials. In the initial study, Report No. 34-99-79, samples were stored frozen up to 193 days. As samples from the residue trials were stored up to 239 days before analysis, the second study (report No. 34-00-31) increased the storage period to 365 days, to cover storage conditions of the field trials samples.

Concentrations of methoxyfenozide were determined by HPLC using UV detection, following method TR 34-98-189, finalized as TR 34-99-74. The method had an LOQ of 0.02 mg/kg. Recoveries of methoxyfenozide from stored samples were calculated by dividing the concentrations found in the stored samples by the amount fortified prior to storage and were corrected for procedural recoveries, which ranged from 90% to 103%. Table 38 summarizes the results from both studies.

Table 38. Frozen storage stability data for methoxyfenozide in head lettuce samples (Burnett, 2000; Schuck, 1999).

Matrix	Storage period, days	Fortification, mg/kg	Apparent recovery [1/], %	Corrected recovery [2/], %	Reference
Lettuce, head	0	1.0	97	100	34-99-79
	43		80	89	
	71		89	100	
	105		95	100	
	133		98	100	
	165		99	96	
	193		97	94	
	281		98	97	34-00-31
	365		100	100	

[1/] Values represent average of two determinations.
[2/] Residues corrected for procedural recovery.

Cotton seed and cotton seed products

Cotton seed samples were fortified with methoxyfenozide at 1 mg/kg, stored frozen at approximately -20°C and analyzed at intervals of 0 day, 1 month, 3 months, 6 months, 9 months, 12 months, and 23.5 months (Szuter, 1998a, report No. 34-98-107). Similarly, cottonseed oil and gin trash samples were also fortified at 1 mg/kg with methoxyfenozide, stored frozen at approximately -20°C and analyzed at intervals of 0, 30, 90, 180, 270, and 360 days (Szuter, 1998b, report No. 34-98-40). Methoxyfenozide residues in cotton seed were determined by method 34-95-133 (described by Szuter, 1998a, and

finalized as method 34-96-88) which had an LOQ of 0.025 mg/kg. Residues of methoxyfenozide in cotton seed oil and gin trash were determined by the same method, which had an LOQ of 0.01 mg/kg for refined oil and 0.05 mg/kg for cotton gin trash. Recoveries of methoxyfenozide from stored samples were calculated by dividing the concentrations found in the stored samples by the amount fortified prior to storage and were corrected for procedural recoveries, which were in the ranges 89.3-110% for cottonseed, 84.5-110% for refined oil samples and 80-114% for cotton gin trash. Table 39 summarizes the results.

Table 39. Frozen storage stability data for methoxyfenozide in cotton seed and cotton seed processed samples (Szuter, 1998a and 1998b).

Matrix	Storage period, months	Fortification mg/kg	Apparent remainder [1/] %	Corrected remainder [2/] %	Reference
Cottonseed	0	1.0	100	100	34-98-107
	1		100	94	
	3		100	99	
	6		110	100	
	9		110	100	
	12		78	85	
	23.5		76	86	
Refined oil	0	1.0	99	100	34-98-40
	1		99	89	
	3		110	96	
	6		96	110	
	9		86	100	
	12		99	97	
Gin Trash	0	1.0	100	100	34-98-40
	1		89	83	
	3		90	80	
	6		93	88	
	9		74	88	
	12		80	70	

[1/] Values represent average of two determinations.
[2/] Residues corrected for procedural recovery.

Maize and maize products

Storage stability studies of methoxyfenozide in frozen maize grain, maize meal and refined maize oil were conducted (Filchner, 2000b and 2000c, reports No. 34-00-10 and 34-00-32; Haneur, 2000, report No. 34-00-25). Control samples of corn grain, corn meal and refined oil were fortified with methoxyfenozide at a concentration of 1.0 mg/kg and the fortified samples were stored frozen at approximately -20°C, under the same conditions as the samples from the residue trials. Residues of methoxyfenozide were determined by method TR 34-98-186 (Deakyne, 1998, report No. 34-98-186), using HPLC with UV detection. The LOQ for the method was 0.02 mg/kg for all corn matrices. Recoveries of methoxyfenozide from stored samples were calculated by dividing the concentrations found in the stored samples by the amount fortified prior to storage and were corrected for procedural recoveries, which were in the ranges 83.6-104% for corn grain, 90-105% for corn meal and 95-107% for refined oil. Table 40 summarizes the results.

Table 40. Frozen storage stability data for methoxyfenozide in maize grain and maize processed samples (Filchner, 2000b and 2000c; Haneur, 2000).

Matrix	Storage period, days	Fortification mg/kg	Apparent remainder [1/], %	Corrected remainder [2/], %	Reference
Maize (corn) grain	0	1.0	95	100	34-00-10
	105		90	92	
	161		91	93	
	222		90	110	
	278		95	110	
	341		99	93	
	397		97	94	
Maize meal	0	1.0	97	100	34-00-25
	34		83	90	
	62		84	93	
	92		93	92	

Matrix	Storage period, days	Fortification mg/kg	Apparent remainder [1/], %	Corrected remainder [2/], %	Reference
Maize meal	127	1.0	85	86	34-00-25
Refined maize oil	0	1.0	100	100	34-00-32
	31		100	96	
	60		95	100	
	92		100	99	
	122		97	94	
	184		95	97	

[1/] Values represent average of two analyses.
[2/] Residues corrected for procedural recovery.

Wheat forage

The effect of storage at -20°C on the stability of residues of methoxyfenozide in wheat forage samples, stored for four years, was assessed by re-assay of the A-ring labelled ^{14}C-methoxyfenozide treated sample from the confined accumulation study on rotational crops (Gu, 2000, report No. 34-00-30). The extraction and chromatographic methods originally used in the confined rotational crop study were followed and the results compared to those initially obtained in the rotational crop study (Table 41).

Table 41. Metabolite distribution in extractable fractions of A-ring 30 DAT wheat forage (Gu, 2000).

Residue	Re-assay results [1/]		Original study results [2/]	
	% TRR	mg/kg	% TRR	mg/kg
Methoxyfenozide	0.69	0.005	0.70	0.005
RH-117236	1.4	0.010	1.1	0.008
RH-151055	28	0.20	29	0.21
RH-152072	58	0.41	46	0.33

[1/] Re-assay was conducted after approximately 4 years of frozen storage at approximately -20°C.
[2/] Kim-Kang, H. 1998, report No. 34-98-99.

Bovine products

The stability of methoxyfenozide in frozen samples of milk, muscle, liver, and kidney, and of the metabolite RH-1518 in samples of liver and kidney, from lactating dairy cows was investigated (Bender, 1998b, report No. 34-98-178). Control samples of cattle milk, muscle, liver and kidney were homogenized in dry ice and fortified with methoxyfenozide at 1.0 mg/kg, using a methanol solution (milk was defrosted prior to fortification). Control samples of liver and kidney were similarly fortified with RH-1518 at 0.92 mg/kg. Samples were stored in a freezer at -20°C in the dark over a period at least equal to that during which the feeding study samples had been stored prior to analysis. Samples were analyzed at approximately 3 week intervals.

Milk samples and muscle samples were analyzed for residues of methoxyfenozide by HPLC-UV, whereas liver and kidney samples were analyzed with HPLC-MS. Procedural recoveries of methoxyfenozide were in the ranges 71-86% from milk, 78-84% from muscle, 101-105% from liver and 87-111% from kidney. The methods were finalized as method 34-98-106 (Chen, J.). Procedural recoveries of the metabolite, RH-1518, were in the ranges 85-97% from liver and 77-101% from kidney. The results are presented in Table 42.

A residue storage stability study was also carried out on eggs (Bender, 2000c, Report No. 34-00-33). Eggs (without shells) were fortified with either methoxyfenozide or RH-1518 at 0.1 mg/kg and stored at -20°C for 93 days. Egg samples were analyzed by method 34-99-11. Procedural recoveries were in the ranges 84-99% for methoxyfenozide and 81-112% for RH-1518. The results are included in Tables 42 and 43.

Table 42. Storage stability of methoxyfenozide in bovine products and hen eggs (Bender, 1998b and 2000c).

Matrix	Storage period, days	Fortification, mg/kg	Apparent remainder [1/] %	Corrected remainder [2/] %	Reference
Milk	0	1.0	78	100	34-98-178
	36		77	100	
	57		72	97	
	77		73	99	
	85		81	97	
	106		76	92	
Muscle	0	1.0	81	100	34-98-178
	21		85	110	
	42		85	110	
	63		84	100	
	83		82	100	
	104		80	96	
	124		82	99	
	144		82	110	
	165		80	100	
Liver	0	1.0	100	100	34-98-178
	19		110	100	
	42		94	93	
	89		90	84	
	111		100	93	
	131		96	93	
	132		99	93	
	153		88	85	
	174		88	85	
	218		100	98	
	240		100	97	
	261		94	90	
Kidney	0	1.0	100	100	34-98-178
	26		100	110	
	54		98	100	
	96		99	100	
	139		92	96	
	181		110	97	
	222		110	100	
	265		110	99	
Eggs	0	0.1	89	100	34-00-33
	34		95	110	
	62		94	110	
	93		100	100	

[1/] Values represent average of two analyses.
[2/] Residues corrected for procedural recovery.

Table 43. Storage stability of the metabolite RH-1518 in poultry products (Bender, 1998b and 2000c).

Matrix	Storage period, days	Fortification, mg/kg	Apparent recovery [1/] %	Corrected recovery [2/] %	Reference
Liver	0	0.92	89	100	34-98-178
	17		61	69	
	45		57	64	
	68		60	68	
	89		70	75	
	111		67	72	
	132		64	68	
	154		59	69	
	176		54	63	
	197		57	66	
	230		55	62	
	252		60	68	
	273		59	67	
Kidney	0	0.92	91	100	34-98-178
	26		100	130	
	54		86	110	
	96		80	98	
	139		79	97	
	181		87	86	
	222		84	84	
	265		78	78	
Eggs	0	1.0	99	100	34-00-33
	34		100	110	
	62		100	100	
	93		91	100	

[1/] Values represent average of two determinations.
[2/] Residues corrected for procedural recovery.

USE PATTERN

Methoxyfenozide belongs to the diacylhydrazine class of insecticides and has a novel mode of action that mimics the action of the moulting hormone of Lepidopterous (moths, butterflies) larvae. Upon ingestion, larval stages of the order Lepidoptera undergo an incomplete and developmentally premature moult, which is ultimately lethal. This process interrupts, and rapidly halts, feeding. Feeding typically ceases within hours of ingestion, although complete mortality of the larvae may take several days. Affected larvae often become lethargic and develop discoloured areas or bands between segments. Methoxyfenozide has virtually no effect on any order of insects or arthropods, except the Lepidoptera. This selectivity allows beneficial insects (including bees) and other arthropods to function unimpeded.

The formulated product is mixed with water and applied as foliar spray or broadcast spray treatments, using aerial or ground equipment equipped for conventional insecticide spraying on crops. Methoxyfenozide must be ingested by insect larvae to be fully effective. Consequently, the timing of application is largely dependent on the feeding behaviour of the target pest. For cryptic (internal) feeding larvae, application must be made prior to the time that surface feeding occurs i.e., just prior to initiation of egg hatch. For foliar or surface feeding larvae, application may be made during active feeding. Re-application may be required to protect new flushes of foliage, rapidly expanding fruit or for extended infestations.

Methoxyfenozide is registered for use in a variety of crops in several countries. The uses according to the Good Agricultural Practice (GAP), for which field trial data were submitted, are listed in Tables 44 to 47. Many uses outside the USA, especially in Europe, are pending registration and have not been included in the tables. Uses with approved GAP but for which no data were submitted have not been included in the table.

Table 44. GAP data for methoxyfenozide uses on fruit and tree nut crops.

Crop	Country	Formulation type/conc.	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. or max./season	
Pome fruit							
Apples, pears	UK	SC 240 g/l	Foliar	0.096-0.144	0.0096	3	14
Pome fruit [1/]	USA	SC 240 g/l WP 800 g/kg	Foliar	0.10-0.34	0.01-0.072	1.12 kg ai/ha/ season	14
Stone fruit							
Cherries	USA	SC 240 g/l WP 800 g/kg	Foliar	0.134-0.28 (0.12-0.25 lb/acre)	0.014-0.06	1.0 kg ai/ha/ season	7
Berries and other small fruit							
Grapes	USA	SC 240 g/l WP 800 g/kg	Foliar	0.07-0.28 (0.06-0.25 lb/acre)	0.02-0.075	0.84 kg ai/ha/ season	30
Tree nuts							
Almond	USA	SC 240 g/l WP 800 g/kg	Foliar	0.13-0.426 (0.12-0.38 lb/acre)	0.01-0.09	1.12 kg ai/ha/ season	14
Hazelnut	USA	SC 240 g/l WP 800 g/kg	Foliar	0.13-0.28 (0.12-0.25 lb/acre)	0.01-0.06	1.12 kg ai/ha/ season	14
Walnut	USA	SC 240 g/l WP 800 g/kg	Foliar	0.13-0.426 (0.12-0.38 lb/acre)	0.01-0.09	1.12 kg ai/ha/ season	14
Walnut	Chile	SC 240 g/l	Foliar	0.054-0.096	0.004-0.005	2	7

[1/] Pome fruit = apples, crab apples, loquat, mayhaw, pears incl. oriental pears, quinces.

Table 45. GAP data for methoxyfenozide uses on vegetable crops.

Crop	Country	Formulation type/conc.	Application				PHI days
			Method	Rate kg ai/ha	Spray conc. kg ai/hl	No. or max/season	
Brassica vegetables							
Brassica (cole) crops [1/]	USA	SC 240 g/l WP 800 g/kg	Foliar	0.07-0.28 (0.06-0.25 lb/acre)	0.04-0.30	1.12 kg ai/ha/ season	1
Fruiting vegetables other than cucurbits							
Fruiting vegetables [2/]	USA	SC 240 g/l WP 800 g/kg	Foliar	0.07-0.28 (0.06-0.25 lb/acre)	0.04-0.30	1.12 kg ai/ha/ season	1
Tomato	Australia	SC 240 g/l	Foliar	0.3-0.4	0.03-0.04	3	0
Eggplant	Malaysia	SC 240 g/l	Foliar	0.1	0.023	As required	14
Sweet corn (corn on cob)	USA	SC 240 g/l	Foliar	0.07-0.13 (0.06-0.12 lb/acre)	0.04-0.14	1.12 kg ai/ha/ season	3
Sweet corn (corn on cob)	USA	WP 800 g/kg	Foliar	0.07-0.28		1.12 kg ai/ha/season	3
Leafy vegetables							
Leafy vegetables [3/]	USA	SC 240 g/l WP 800 g/kg	Foliar	0.07-0.28	0.04-0.30	1.12 kg ai/ha/ season	1
Chinese cabbage [4/]	USA	SC 240 g/l	Foliar	0.067-0.28 (0.06-0.25 lb/acre)	0.04-0.15	1.12 kg ai/ha/ season	1
Collards [4/]	USA	SC 240 g/l	Foliar	0.067-0.28 (0.06-0.25 lb/acre)	0.04-0.15	1.12 kg ai/ha/ season	1
Kale [4/]	USA	SC 240 g/l	Foliar	0.067-0.28 (0.06-0.25 lb/acre)	0.04-0.15	1.12 kg ai/ha/ season	1
Mustard greens [4/]	USA	SC 240 g/l	Foliar	0.067-0.28 (0.06-0.25 lb/acre)	0.04-0.15	1.12 kg ai/ha/ season	1
Legume vegetables							
Beans (longbeans)	Malaysia	SC 240 g/l	Foliar	0.1	0.023	As required	14
Soybeans	Argentina	SC 240 g/l	Foliar	0.019-0.028	0.010-0.014	1	7
Soybeans	Brazil	SC 240 g/l	Foliar	0.014-0.022	0.004-0.011	1	7
Stalk and stem vegetables							
Celery	Israel	SC 240 g/l	Foliar	0.12-0.18	0.024-0.18	Not specified	7
Celery [5/]	USA	SC 240 g/l	Foliar	0.07-0.28	0.04-0.30	1.12 kg ai/ha	1
Rhubarb [5/]	USA	SC 240 g/l	Foliar	0.07-0.28	0.04-0.30	1.12 kg ai/ha	1

[1/] Brassica (cole) crops, including but not limited to: broccoli, broccoli raab, Brussels sprouts, cabbages, cauliflowers, cavalo □ broccoli, Chinese broccoli, Chinese cabbages (bok choy, napa), Chinese mustard cabbages (gai choy), collards, kale, kohlrabi, mizuna, mustard greens, mustard spinach, rape greens.

[2/] Fruiting vegetables: egg plants, ground cherries, pepino, peppers (including bell, chilli, pimento), tomatillo, tomatoes.
[3/] Leafy vegetables (except *Brassica*), including but not limited to: amaranth, arugula, cardoon, celery, celtuce, chervil, Chinese celery, corn salad, dandelion, dock, edible-leaved chrysanthemum, endive (escarole), Florence fennel, garden cress, garden purslane, garland chrysanthemum, lettuce (head, leaf), New Zealand spinach, orach, parsley, radicchio, rhubarb, spinach, Swiss chard, upland cress, vine spinach, winter purslane.
[4/] Included with Brassica (cole) crop group in the USA label.
[5/] Included with leafy vegetable group in the USA label.

Table 46. GAP data for methoxyfenozide uses on cereal crops and primary feed commodities.

Crop	Country	Formulation type/conc.	Application				PHI days
			Method	Rate kg ai/ha	Spray conc. kg ai/hl	No. or max/season	
Maize (field corn)	USA	SC 240 g/l WP 800 g/kg	Foliar	0.07-0.13 (0.06-0.12 lb/acre)	0.07– 0.28	1.12 kg ai/ha/ season	21 (grain, forage, fodder)
Maize	Brazil	SC 240 g/l	Foliar	0.036-0.043	0.01-0.02	1	7
Maize	Mexico	SC 240 g/l (OS)[???]	Foliar	0.03-0.04	0.01-0.013	1	30
Maize	Mexico	SC 240 g/l (HP)[???]	Foliar	0.03-0.04	0.01-0.013	1	30
Sweet corn, forage and fodder	USA	SC 240 g/l	Foliar	0.07-0.13 (0.06-0.12 lb/acre)	0.04-0.14	1.12 kg ai/ha/ season	3 for forage; 21 for fodder
Rice	Japan	SC 200 g/l	Broadcast	0.2		3	14

Table 47. GAP data for methoxyfenozide uses on oilseed crops.

Crop	Country	Formulation type/conc.	Application				PHI days
			Method	Rate, kg ai/ha	Spray conc., kg ai/hl	No. or max/season	
Cotton	Australia	SC 240 g/l	Foliar	0.4-0.6		3	28
Cotton	Mexico	SC 240 g/l (OS)	Foliar	0.03-0.12	0.015-0.08	2	14
Cotton	Mexico	SC 240 g/l (HP)	Foliar	0.03-0.12	0.015-0.08	2	14
Cotton	USA	SC 240 g/l	Foliar	0.07- 0.448 (0.06-0.4 lb/acre)	0.14-0.9	1.12 kg ai/ha/ season	14
Cotton	USA	WP 800 g/kg	Foliar	0.06-0.448 (0.05-0.4 lb/acre)	0.12-0.24	1.12 kg ai/ha/ season	14

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised trials were reported for numerous commodities and the trials are summarized in Tables 48-97. Commodities are listed in the order of the Codex Classification System. Each residue value listed may be the average of multiple analytical measurements and/or replicate (typically two) samples.

The residue values for trials conducted according to a maximum national/regional GAP (maximum application rate, minimum PHI) are underlined and used in the calculation of maximum residue levels and supervised field trial median residue levels (STMRs).

The commodities and trials locations reported to the Meeting are as follows:

Commodity	Table number	Country of trials
Citrus fruits - oranges	48	Italy, Spain, Portugal
Citrus fruits - oranges	49	Italy, Spain, Portugal, Greece
Citrus fruits - mandarin oranges	50	Italy, Spain, Portugal
Citrus fruits - mandarin oranges	51	Italy, Spain, Portugal, Greece
Pome fruits - apple	52	USA
Pome fruits - apple	53	USA
Pome fruits - apple	54	USA
Pome fruits - pear	55	USA
Pome fruits - pear	56	USA
Pome fruits - pear	57	USA
Pome fruits - apple and pear	58	France, Italy, Spain
Pome fruits - apple and pear	59	UK, Germany, Belgium, France
Pome fruits - apple and pear	60	UK, Germany, Belgium, France

Commodity	Table number	Country of trials
Pome fruits - apple and pear	61	Spain, France, Italy
Stone fruits - peach	62	USA
Stone fruits - cherry	63	USA
Stone fruits - plum	64	USA
Stone fruits - peach and nectarine	65	Spain, France, Italy
Grapes	66	USA
Grapes	67	Greece, Italy, France, Portugal, Spain
Grapes	68	Portugal, France, Spain, Italy, Germany
Broccoli	69	USA
Cabbage (head)	70	USA
Pepper	71	USA
Pepper	72	Portugal, Spain, Italy, France, Netherlands
Pepper	73	Italy, Spain
Pepper	74	Belgium, Germany
Tomato	75	USA
Tomato	76	Australia
Tomato	77	Germany, Belgium, Netherlands, Spain, Portugal, Italy, France
Egg plant	78	Malaysia
Sweet corn	79	USA
Lettuce (leaf)	80	USA
Lettuce (head)	81	USA
Spinach	82	USA
Mustard green	83	USA
Soya bean	84	Brazil
Long bean	85	Malaysia
Celery	86	USA
Rice	87	Japan
Maize (field corn)	88	USA
Maize (field corn)	89	Mexico
Maize (field corn)	90	Brazil
Cotton seed	91	USA
Cotton seed	92	Mexico
Cotton seed	93	Australia
Pecans	94	USA
Almonds	95	USA
Maize (field corn) fodder, forage	96	USA
Sweet corn fodder, forage	97	USA

Citrus fruits

Nine supervised field trials on oranges were conducted in 1997 and 1998 in Italy, Spain, Portugal and Greece (Seym and Deissler, 1998a and 1999a, reports No. 34-99-02 and 34-99-140; Walz-Tylla, 1999a, report No. 34-99-133). In five of these trials, a suspension concentrate (SC) formulation containing 240 g/l methoxyfenozide was applied twice to orange trees, at 0.192 kg ai/ha per application. In four of the trials, the first application was applied at 0.288 or 0.240 kg ai/ha and the second at 0.192 kg ai/ha. All applications were made at intervals of 10 to 14 days, with the last application being made 14 days before harvest. The product was applied with about 5 l/ha mineral oil. The spray volume was 2000 l/ha. Registration of the GAP is pending, according to the manufacturer, as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	Interval, days	
SC 240 g/l	Foliar	0.19	0.0096	2	10	14

Table 48. Residues data summary from supervised trials on oranges in Italy, Spain, and Portugal (Seym and Deissler, 1998a).

Report, trial, location	Crop variety	Application			Dates/No. of treatments	Growth stage at last treatment	Commodity	Residue [3/, 4/] (mg/kg)	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-02	Orange	0.19	2000	0.0096	11.22.97	BBHC 83	Whole fruit	0.31	0
Trial: 703745	Navelina	0.19	2000	0.0096	12.02.97	(begin		0.21	7
IT 17-						ripening)		0.20	14
Montalbano,								0.21	21
Italy							pulp	<0.05	14
							peel	0.57	14
							Total fruit (calc.) [2/]	0.21	14
34-99-02	Orange	0.24	2500	0.0096	11.10.97	BBHC 86-87	Whole fruit	0.32	0
Trial: 703753	Consumo	0.19	2000	0.0096	11.20.97	(advanced		0.27	7
SPA 6:						ripening and		0.34	14
Tarragona,						fruit		0.24	21
Spain						coloration)	pulp	<0.05	14
							peel	0.95	14
							Total fruit (Calc) [2/]	0.29	14
34-99-02	Orange	0.29	3000	0.0096	11.10.97	BBHC 85	Whole fruit	0.36	0
Trial: 705195	Consumo	0.19	2000	0.0096	11.20.97	(advanced		0.22	7
SPA 6:						ripening and		0.22	14
Tarragona,						fruit		0.26	21
Spain						coloration)	pulp	<0.05	14
							peel	0.94	14
							Total fruit (Calc) [2/]	0.26	14
34-99-02	Orange	0.19	2000	0.0096	10.28.97	BBHC 83	Whole fruit	0.11	0
Trial: 705209	Dalman	0.19	2000	0.0096	11.08.97	(begin		0.095	7
POR 1:						ripening)		0.057	14
Santarem,								0.057	21
Portugal							pulp	<0.05	14
							peel	0.37	14
							Total fruit (calc) [2/]	0.13	14
34-99-02	Orange	0.19	2000	0.0096	10.21.97	BBHC 81	Whole fruit	0.20	0
Trial: 705217	Navelina	0.19	2000	0.0096	11.08.97	(begin		0.24	7
IT 20:						ripening)		0.18	14
Siraycusa,								0.18	21
Italy							pulp	<0.05	14
							peel	0.60	14
							Total fruit (calc) [2/]	0.17	14

[1] Calculated from the kg ai/ha and spray volume.
[2] Calculated from residues in the pulp and peel.
[3] Registration of the GAP has not been finalized by national authorities.
[4] Analysis method 00470, LC-MS/MS, average recoveries: fruit/pulp, 102%; peel, 89%; LOQ = 0.05 mg/kg.

Table 49. Residues data summary from supervised trials on oranges in Italy, Spain, Greece, and Portugal (Walz-Tylla, 1999a).

Report, trial, location	Crop, variety	Application			Dates or No. of treatments	Growth stage at last treatment	Commodity	Residue [2/, 3/] (mg/kg)	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-133	Orange	0.19	2000	0.0096	11.09.98	BBCH 85	Whole fruit	0.18	0
Trial: 810533	Navelina	0.19	2000	0.0096	11.19.98	(advanced		0.13	15
SPA 6:						ripening)			
Tarragona,							pulp	<0.05	15
Spain							peel	0.52	15

Report, trial, location	Crop, variety	Application			Dates or No. of treatments	Growth stage at last treatment	Commodity	Residue [2/, 3/] (mg/kg)	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-133 Trial: 810541 IT 24: Carlentini. Italy	Orange Navelina	0.24 0.19	2500 2000	0.0096 0.0096	10.24.98 11.03.98	BBHC 82-83 (ripening)	Whole fruit pulp peel	0.25 0.19 <0.05 0.44	0 14 14 14
34-99-133 Trial: 814857 POR 2: Santarem, Portugal	Orange Dalman	0.29 0.19	3000 2000	0.0096 0.0096	11.06.98 11.16.98	BBHC 85-86 (advanced ripening and fruit coloration)	Whole fruit pulp peel	0.17 0.16 <0.05 0.31	0 14 14 14
34-99-133 Trial: 814865 GR 13: Enias, Greece	Orange Navelate	0.19 0.19	2000 2000	0.0096 0.0096	11.06.98 11.16.98	BBHC 83 (begin ripening)	Whole fruit pulp peel	0.30 0.14 <0.05 1.40	0 14 14 14

[1/] Calculated from kg ai/ha and spray volume.
[2/] Registration of the GAP has not been finalized by national authorities.
[3/] Analysis method 00551, LC-MS/MS, average recoveries: fruit/pulp, 98%; peel, 89%; LOQ = 0.05 mg/kg.

Table 50. Residues data summary from supervised trials on mandarins in Italy, Spain, and Portugal (Seym and Deissler, 1999a).

Report, trial, location	Crop, variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2/, 3/] (mg/kg)	PHI days
		kg ai/ha	water L/ha	kg ai/hl [1/]					
34-99-140 Trial: 703761 It 20: Sicily, Italy	Mandarin Montreal	0.182 0.182	1900 1900	0.0096 0.0096	10.17.97 10.27.97	BBCH 80 (beginning of ripening)	Whole fruit pulp peel	0.52 0.32 0.27 0.30 <0.05 0.99	0 7 14 21 14 14
34-99-140 Trial: 703788 SPA 2: Valencia, Spain	Mandarin Marisol	0.192 0.192	2000 2000	0.0096 0.0096	09.12.97 09.22.97	BBHC 82 (ripening)	Whole fruit Pulp peel	0.36 0.20 0.13 0.16 <0.05 1.0	0 7 14 21 14 14
34-99-140 Trial: 705284 SPA 6: Tarragona, Spain	Mandarin Clemenules	0.288 0.288	3000 3000	0.0096 0.0096	10.13.97 10.23.97	BBHC 81-82 (ripening)	Whole fruit pulp peel	0.37 0.38 0.39 0.36 <0.05 1.3	0 7 14 21 14 14
34-99-140 Trial: 705292 POR 2: Santarem, Portugal	Mandarin Not Provided	0.192 0.192	2000 2000	0.0096 0.0096	09.25.97 10.07.97	BBCH 82 (ripening)	Whole fruit pulp peel	0.23 0.17 0.085 0.11 <0.05 0.44	0 7 14 21 14 14
34-99-140 Trial: 705306 SPA 6: Tarragona, Spain	Mandarin Clemenvilla	0.192 0.192	2500 2000	0.0096 0.0096	11.07.97 11.17.97	BBHC 87 (advanced ripening)	Whole fruit pulp peel	0.35 0.42 0.45 0.33 <0.05 1.2	0 7 14 22 14 14

[1/] Calculated from kg ai/ha and spray volume.
[2/] Registration of the GAP has not been finalized by national authorities.
[3/] Analysis method 00470, LC-MS/MS, average recoveries: fruit/pulp, 98%; peel, 89%; LOQ = 0.05 mg/kg.

Table 51. Residues data summary from supervised trials on mandarins in Italy, Spain, Greece, and Portugal (Walz-Tylla, 1999b).

Report, trial, location	Crop, variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2/, 3/] (mg/kg)	PHI days
		kg ai/ha	water L/ha	kg ai/hl [1/]					
34-99-137	Mandarin	0.19	2000	0.0096	10.14.98	BBCH 79	Whole fruit	0.36	0
Trial: 810568	Monreal	0.19	2000	0.0096	10.24.98	(fruit		0.24	14
IT 14: Augusta,						development)	pulp	<0.05	14
Italy							peel	0.79	14
34-99-137	Mandarin	0.19	2000	0.0096	11.06.98	BBHC 81	Whole fruit	0.46	0
Trial: 814903	Satsuma	0.19	2000	0.0096	11.16.98	(begin		0.27	14
GR 13: Enias,						colouring)	pulp	<0.05	14
Greece							peel	0.84	14
34-99-137	Mandarin	0.19	2000	0.0096	10.09.98	BBHC 84	Whole fruit	0.28	0
Trial: 814911	Tangera	0.19	2000	0.0096	10.19.98	(advanced		0.21	14
POR 2:						ripening)	pulp	<0.05	14
Santarem,							peel	0.52	14
Portugal									
34-99-137	Mandarin	0.19	2000	0.0096	10.19.98	BBCH 82	Whole fruit	0.45	0
Trial: 814938	Orogrande	0.19	2000	0.0096	10.29.98	(begin		0.35	14
SPA 6:						colouring)	pulp	<0.05	14
Tarragona,							peel	0.52	14
Spain									

[1/] Calculated from kg ai/ha and spray volume.
[2/] Registration of the GAP has not been finalized by national authorities.
[3/] Analysis method 00551, LC-MS/MS, average recoveries: fruit/pulp, 100%; peel, 93%; LOQ = 0.05 mg/kg.

Pome fruits

Supervised trials on apples and pears were conducted in the USA and Europe, from 1996 to 1998 (Tables 52 to 61).

Twelve supervised trials on apples were conducted in the USA in 1996 and 1997. In addition, two trials were conducted in 1999, to develop residue bridging data between the 80W (800 g/kg WP) and 2F (240 g/l SC) formulations. Five supervised trials were conducted in 1996, and seven in 1997, in States representative of the major apple growing regions of the USA (Bender and Bergin, 1998, report No. 34-98-20; Bergin, 1998b, report No. 34-98-66). The trials consisted of six applications to apple trees of the 80W formulation, a wettable powder formulation containing 800 g/kg methoxyfenozide, at a nominal rate of 0.3 lb/acre (0.34 kg ai/ha) per application. The spray volume was 50 to 100 gal/acre (468 to 935 l/ha), giving a spray concentration equivalent to 0.036 to 0.072 kg ai/hl. The first four applications were made at 14 to 21-day intervals, in early- to mid-crop production season. The remainder of the applications were made at 14-day intervals. The total treatment was approximately 1.8 lb ai/acre (2.0 kg ai/ha). All applications were made as foliar sprays using air-blast equipment, except for one trial in 1997, where a single-nozzle mist blower was used. The GAP information for registered use of methoxyfenozide in the USA for pome fruit, including apples, is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. or max/season	Interval, days	
WP 800g/kg	Foliar	0.11-0.34 (0.1-0.3 lb/acre)	0.012-0.072	1.12 kg ai/ha/season	14-21	14
SC 240 g/l	Foliar	0.10-0.28 (0.09-0.25 lb/acre)	0.012-0.06	1.05 kg ai/ha/season	14-21	14

Sample harvest to analysis time was approximately 4.5 to 10 months. Samples were stored frozen (Tables 52 and 53).

Two bridging trials on apples were conducted in 1998 in the USA, using either the 2F or 80W formulation in each trial (Yoshida, 1999a, report No. 34-99-30). Treated plots received a total of 6 applications of either formulation, at the nominal rate of 0.3 lb ai/acre (0.336 kg ai/ha) per application. The total amount of each formulation applied was approximately 1.8 lb ai/acre (2.0 kg ai/ha). The first five applications were made at intervals of 14 to 21 days. All applications were made with ground-driven air-blast equipment. Whole apple fruits were collected 14 days after the last application (Table 54).

A total of six supervised trials on pears were conducted in States representative of the major pear growing areas in the USA. Four trials were conducted in California and Washington in 1996 (Carpenter, 1998a, report No. 34-98-77) (Table 55) and two in New York and Washington, in 1997 (Carpenter, 1998b, report No. 34-98-94) (Table 56). The trials consisted of six applications to pear trees of the 80W formulation, a wettable powder formulation containing 800 g/kg methoxyfenozide, at a nominal rate of 0.3 lb/acre (0.336 kg ai/ha) per application, applied at 14 to 21-day intervals. All applications were made as foliar sprays, using air-blast equipment, calibrated to deliver a spray volume of approximately 100-200 gallons of water per acre (935 to 1870 l/ha), or a spray concentration equivalent to 0.015 to 0.036 kg ai/hl. One trial in each year was designed as a decline study. The GAP information for the registered use of methoxyfenozide in the USA is as for pome fruit (see apple above). Sample harvest to analysis time was approximately 11–12.5 months for the 1996 trials and 4-8 months for the 1997 trials. Samples were stored frozen.

Two bridging trials were conducted on pears in 1998 in the USA, using either the 2F or 80W formulation in each trial (Yoshida, 1999b, report No. 34-99-31). Treated plots received a total of 6 applications of either formulation, at the nominal rate of 0.3 lb ai/acre (0.34 kg ai/ha). The total amount of each formulation applied was approximately 1.8 lb ai/acre (2.0 kg ai/ha). All applications were made with ground-driven air-blast equipment. Whole pear fruits were collected 14 days after the last application (Table 57).

A total of eighteen supervised trials were conducted on apples and pears in southern and northern regions of Europe, from 1997 to 1998 (Seym and Deissler, 1998b and 1999b, reports No. 34-98-191 and 34-99-178; Walz-Tylla, 1999c and 1999d, reports No. 34-99-128 and 34-99-130) (Tables 58-61). In all trials, methoxyfenozide (240 g/l SC) was applied three times to apple and pear trees, at intervals of 13 to 18 days, at a spray concentration of 0.04 % (0.0096 kg ai/hl) and water volume of 1000-1,500 l/ha (0.096–0.144 kg ai/ha). The GAP in the UK for use of methoxyfenozide on apples and pears is summarized below:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No.	Interval, days	
SC 240 g/l	Overall spray	0.096 (foliar canopy ≤ 2m) 0.14 (foliar canopy ≥ 3 m)	0.0096	1 - 3	14	14

Registration of the GAP is pending in many EC countries. Samples from all other trials were stored after sampling at –18ºC or below. All samples remained in frozen storage for about 7 months until analysis. The analytical method was validated at 0.05 mg/kg.

Table 52. Residues data summary from supervised trials on apples in the USA (Bender and Bergin, 1998).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue, mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water, l/ha					
34-98-20 Trial: 21696-030 Alton, NY WP 800 g/kg	Apple, Joni Mac	0.34 0.34 0.34 0.34 0.34 0.34	0.036 0.036 0.036 0.036 0.036 0.036	930 940 940 940 940 930	06.13.96 07.02.96 07.17.96 07.31.96 08.14.96 08.28.96	At maturity	Whole fruit	0.30	14
34-98-20 Trial: 21696-031 Hereford, PA WP 800 g/kg	Apple, Red Delicious	0.34 0.33 0.34 0.34 0.34 0.34	0.035 0.035 0.035 0.035 0.035 0.035	960 950 960 960 960 960	06.26.96 07.11.96 07.30.96 08.13.96 08.27.96 09.10.96	At maturity	Whole fruit	0.36	14
34-98-20 Trial 21696-032 Danbury, North Carolina WP 800 g/kg	Apple, Red Delicious	0.34 0.34 0.34 0.34 0.34 0.33	0.072 0.072 0.072 0.072 0.072 0.070	470 470 470 470 470 470	06.05.96 06.25.96 07.09.96 07.30.96 08.15.96 08.29.96	At maturity	Whole fruit	0.20	14

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue, mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water, l/ha					
34-98-20 Trial: 21696-033 Conklin, MI WP 800 g/kg	Apple, McIntosh	0.33 0.33 0.34 0.34 0.34 0.34	0.046 0.045 0.051 0.050 0.049 0.050	720 730 670 680 690 680	06.12.96 06.26.96 07.10.96 07.24.96 08.07.96 08.21.96	At maturity	Whole fruit	0.62	14
34-98-20 Trial: 21696-034 Harrah, WA WP 800 g/kg	Apple, Red Delicious	0.33 0.33 0.33 0.33 0.33 0.33	0.036 0.036 0.036 0.036 0.035 0.035	920 920 920 920 930 930	06.18.96 07.03.96 07.17.96 07.31.96 08.14.96 08.28.96	At maturity	Whole fruit	0.916, 0.888 1.0 0.940, 0.568 0.578, 0.775	7 14 21 28
34-98-20 [3/] Trial: 21696-035 Wapato, WA WP 800 g/kg	Apple, Red Delicious								

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis (TR 34-95-55) by HPLC in acetonitrile/water, UV detection, average recovery 91.7 ± 9.5%, LOQ 0.01 mg/kg.
[3/] Data lost.

Table 53. Residues data summary from supervised trials on apples in the USA (Bergin, 1998b).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [3/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-98-66 Trial 21697-027 Noblesville, IN WP 800 g/kg	Apple, Gold Rush	0.34 0.34 0.34 0.34 0.34 0.34	0.065 0.065 0.065 0.065 0.065 0.065	520 510 520 520 510 510	07.17.97 08.01.97 08.18.97 09.01.97 09.04.97 09.19.97	At maturity 2.5-3 in. fruit	Whole fruit	2.9 [2/] 2.3 [2/]	13
34-98-66 Trial: 21697-028 Eckert, CO WP 800 g/kg	Apple, Red Delicious	0.33 0.33 0.34 0.33 0.34 0.34	0.059 0.059 0.059 0.059 0.059 0.060	560 550 560 560 560 570	07.11.97 07.25.97 08.13.97 08.27.97 09.08.97 09.20.97	At maturity 3-4 in. fruit	Whole fruit	0.40	14
34-98-66 Trial 21697-029 Porterville, CA WP 800 g/kg	Apple, Granny Smith	0.34 0.34 0.34 0.34 0.33 0.34	0.011 0.011 0.011 0.011 0.011 0.011	3100 3070 3100 3100 3200 3400	07.10.97 07.24.97 08.07.97 08.21.97 09.04.97 09.18.97	At maturity 6 in. fruit	Whole fruit	0.23	14
34-98-66 Trial: 21697-030 Buena, WA WP 800 g/kg	Apple, Rome Apple	0.350 0.340 0.340 0.340 0.340 0.350	0.037 0.036 0.036 0.036 0.036 0.036	940 940 940 940 940 960	07.02.97 07.17.97 07.13.97 08.14.97 08.28.97 09.10.97	At maturity	Whole fruit	0.56	15
34-98-66 Trial: 21697-031 Granger, WA WP 800 g/kg	Apple, Rome Apple	0.340 0.340 0.340 0.340 0.340 0.330	0.036 0.036 0.036 0.036 0.036 0.035	940 940 930 930 930 930	07.02.97 07.17.97 07.31.97 08.14.97 08.18.97 09.10.97	At maturity	Whole fruit	1.0	15

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [3/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-98-66 Trial: 21697-032 Hereford, PA WP 800 g/kg	Apple, Red Delicious	0.330 0.330 0.330 0.340 0.340 0340	0.044 0.044 0.044 0.046 0.046 0.046	740 740 740 740 740 740	07.17.97 07.31.97 08.14.97 08.28.97 09.09.97 09.23.97	At maturity 2.25-3 in. fruit	Whole fruit	0.62 0.62 0.26 0.29	7 14 22 27
34-98-66 Trial: 21697-035 Payette, ID WP 800 g/kg	Apple, Red Delicious	0.330 0.330 0.340 0.340 0.340 0.340	0.036 0.036 0.036 0.036 0.036 0.036	920 920 940 940 940 940		At maturity 3 in. fruit	Whole fruit	0.25	14

[1/] Calculated from kg ai/ha and spray volume.
[2/] Application not according to re-treatment interval and local practice for method of application. A single-nozzle air-blast sprayer was used and there was a 3-day interval between the 4th and 5th applications.
[3/] Analysis (TR 34-95-55) by HPLC with UV detection, average recovery 92 ± 13%, LOQ 0.01 mg/kg.

Table 54. Residues data summary from bridging trials on apples in the USA (Yoshida, 1999a).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-30 Trial: 1559836 Hood River, OR WP 800 g/kg	Apple, Jonagold	0.34 0.34 0.34 0.35 0.32 0.34	0.034 0.039 0.039 0.037 0.025 0.048	990 870 880 950 810 800	07.15.98 07.29.98 08.12.98 08.26.98 09.09.28 09.23.98	At maturity	Whole fruit	0.43	14
34-99-30 Trial: 1559835 Alton, NY WP 800 g/kg	Apple, Golden Delicious	0.340 0.340 0.340 0.340 0.340 0.340	0.036 0.036 0.036 0.036 0.036 0.036	950 940 940 940 940 940	07.16.98 07.30.98 08.13.98 08.31.98 09.14.98 09.28.98	At maturity	Whole fruit	0.37	14
34-99-30 Trial: 1559836 Hood River, OR SC 240 g/l	Apple, Jonagold	0.340 0.340 0.340 0.340 0.340 0.340	0.035 0.038 0.039 0.036 0.041 0.041	970 890 870 940 820 820	07.15.98 07.29.98 08.12.98 08.26.98 09.09.28 09.23.98	At maturity	Whole fruit	0.52	14
34-99-30 Trial: 1559835 Alton, NY SC 240 g/l	Apple, Golden Delicious	0.340 0.340 0.340 0.340 0.340 0.340	0.036 0.036 0.036 0.036 0.036 0.036	940 940 940 940 940 940	07.16.98 07.30.98 08.13.98 08.31.98 09.14.98 09.18.98	At maturity	Whole fruit	0.60	14

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis (TR 34-95-55) by HPLC with UV detection, average recovery 91.9 ± 6.2%, LOQ 0.01 mg/kg.

Table 55. Residues data summary from supervised trials on pears in the USA (Carpenter, 1998a)

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-98-77 Trial: 21696-060 Porterville, CA WP 800 g/kg	Pear, Bosc	0.34 0.34 0.34 0.33 0.34 0.34	0.24 0.24 0.24 0.24 0.24 0.24	1420 1400 1400 1400 1400 1400	05.17.96 05.31.96 06.14.96 06.28.96 07.12.96 07.26.96	Immature fruit	Whole fruit	0.92	14

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-98-77	Pear	0.34	0.24	1400	05.23.96	Immature	Whole fruit	0.39	14
Trial: 21696-061	20th	0.34	0.24	1400	06.06.96	fruit			
Orosi, CA	Century	0.34	0.24	1400	06.20.96				
WP 800 g/kg		0.34	0.24	1400	07.04.96				
		0.34	0.24	1400	07.18.96				
		0.34	0.24	1400	08.01.96				
34-98-77	Pear-	0.34	0.38	900	05.23.96	Green fruit	Whole fruit	0.36	14
Trial: 21696-062	Bartlett	0.34	0.38	900	06.07.96				
Zillah, WA		0.34	0.38	900	06.22.96				
WP 800 g/kg		0.33	0.38	900	07.06.96				
		0.34	0.38	900	07.20.96				
		0.34	0.38	900	08.03.96				
34-98-77	Pear-	0.34	0.38	900	05.23.96	At maturity	Whole fruit	0.36	7
Trial: 21696-063	Bartlett	0.34	0.38	900	06.07.96			0.39	
Buena, WA		0.34	0.38	900	06.22.96			0.31	14
WP 800 g/kg		0.33	0.38	900	07.06.96			0.17	21
		0.34	0.38	900	07.20.96			0.25	
		0.34	0.38	900	08.03.96			0.32	28
								0.34	

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis by HPLC with UV detection (TR 34-98-87 variant), average recovery 86.0 ± 12.1%, LOQ 0.01 mg/kg.

Table 56. Residues data summary from supervised trials on pears in the USA (Carpenter, 1998b).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-98-94	Pear,	0.34	0.036	940	06.27.97	Immature	Whole fruit	0.17	7
Trial: 21697-033	Bartlett	0.34	0.036	940	07.04.97	fruit			
Block 3		0.34	0.036	940	07.11.97	4.3-5.9 cm		0.27	14
Alton, NY		0.34	0.036	940	07.18.97				
WP 800 g/kg		0.34	0.036	940	08.08.97				
		0.34	0.036	940	08.15.97				
34-98-94	Pear,	0.34	0.036	940	06.27.97			0.31	21
Trial: 21697-033	Bartlett	0.34	0.036	940	07.04.97				
Block 2		0.34	0.036	940	07.11.97			0.28	28
Alton, NY		0.34	0.036	950	07.18.97				
WP 800 g/kg		0.34	0.036	940	07.25.97				
		0.34	0.036	940	08.08.97				
34-98-94	Pear,	0.33	0.017	1900	07.09.97	Fruit about	Whole fruit	0.35	14
Trial: 21697-034	D'Anjou	0.33	0.017	1900	07.23.97	95% mature			
Soap Lake, WA		0.33	0.017	1900	08.06.97				
WP 800 g/kg		0.33	0.017	1900	08.20.97				
		0.33	0.017	1900	09.04.97				
		0.33	0.017	1900	09.18.97				

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis (TR 34-98-87 variant) by HPLC with UV detection, average recovery 96.7 ± 20.8%, LOQ 0.01 mg/kg.

Table 57. Residues data summary from bridging trials on pears in the USA (Yoshida, 1999b).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-31	Pear,	0.34	0.036	940	06.24.98	Immature	Whole fruit	0.74	14
Trial: 1559837	Bartlett	0.34	0.036	940	07.03.98	fruit			
Alton, NY		0.34	0.036	940	07.10.98	6.4-7.2 cm			
WP 800 g/kg		0.34	0.036	940	07.17.98	diameter			
		0.34	0.036	940	07.30.98				
		0.34	0.036	940	08.14.98				

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-31	Pear,	0.34	0.016	2100	07.15.98	Immature	Whole fruit	0.50	14
Trial: 1559838	Bosc	0.34	0.013	2600	07.29.98	fruit			
Hood River, OR		0.34	0.016	2100	08.12.98	7.1cm			
WP 800 g/kg		0.34	0.015	2300	08.26.98	diameter			
		0.34	0.014	2400	09.09.98				
		0.34	0.015	2300	09.23.98				
34-99-31	Pear,	0.34	0.36	940	06.24.98	Immature	Whole fruit	0.68	14
Trial: 1559837	Bartlett	0.34	0.36	940	07.03.98	fruit			
Alton, NY		0.34	0.36	940	07.10.98	6.4-7.2 cm			
SC 240 g/l		0.34	0.36	940	07.17.98	diameter			
		0.34	0.36	940	07.30.98				
		0.34	0.36	940	08.14.98				
34-99-31	Pear,	0.34	0.016	2100	07.15.98	Immature	Whole fruit	0.74	14
Trial: 1559838	Bosc	0.32	0.012	2600	07.29.98	fruit			
Hood River, OR		0.34	0.016	2100	08.12.98	7.1cm			
SC 240 g/l		0.35	0.015	2400	08.26.98	diameter			
		0.34	0.014	2400	09.09.98				
		0.34	0.015	2300	09.23.98				

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis (TR 34-98-87) by HPLC with UV detection, average recovery 85.6 ± 13.6%, LOQ 0.01 mg/kg.

Table 58. Residues data summary from supervised trials on apples and pears in France, Italy and Spain (Seym and Deissler, 1998b).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/hg	kg ai/hl					
34-98-191 [1/]	Apple,	0.11	1100	0.0096	07.09.97	BBCH 75	Whole fruit	0.20	0
Trial: 703702	Golden	0.11	1100	0.0096	07.23.97	(fruit half		0.13	7
FRA 2 – Avignon,	Delicious	0.12	1250	0.0096	08.06.97	final size)		0.13	14
France								0.11	21
SC 240 g/l								0.11	28
34-98-191 [1/]	Apple,	0.14	1500	0.0096	08.07.97	BBCH 83	Whole fruit	0.12	0
Trial: 703710	Florina	0.14	1500	0.0096	08.21.97	(start of		0.09	7
IT 14 - Ravenna		0.14	1500	0.0096	09.04.97	ripening)		<0.05	14
Italy								<0.05	21
SC 240 g/l								<0.05	28
34-98-191 [1/]	Apple,	0.12	1250	0.0096	07.15.97	BBCH 83	Whole fruit	0.28	0
Trial: 703737	Golden	0.12	1250	0.0096	07.28.97	(start of		0.25	7
SPA 1- Girona,	Delicious	0.12	1250	0.0096	08.11.97	ripening)		0.23	14
Spain								0.15	21
SC 240 g/l								0.16	28
34-98-191 [1/]	Apple,	0.096	1000	0.0096	08.12.97	BBCH 87	Whole fruit	0.16	0
Trial: 705187	Granny	0.096	1000	0.0096	08.26.97	(fruit ripe		0.10	7
FRA-2: Avignon,	Smith	0.096	1000	0.0096	09.09.97	for		0.10	14
France						picking)		0.10	21
SC 240 g/l								<0.05	28
34-98-191 [2/]	Pear,	0.14	1425	0.0096	06.30.97	BBCH 79	Whole fruit	0.15	0
Trial: 703729	William's	0.14	1425	0.0096	07.14.97	(fruit 90%		0.12	7
IT 14- Ravenna		0.14	1425	0.0096	07.28.97	final size)		0.07	14
Italy								<0.05	21
SC 240 g/l								<0.05	28

[1/] Analysis method 00470, electrospray LC-MS/MS, average recovery 85%, LOQ 0.05 mg/kg.
[2/] Analysis method 00470, electrospray LC-MS/MS, average recovery 86%, LOQ 0.05 mg/kg.

Table 59. Residues data summary from supervised trials on apples and pears in the UK, Germany, Belgium and, France (Seym and Deissler, 1999b).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-178 [1/] Trial: 703672 ENG2- North east of London, UK SC, 240 g/l	Apple, Fiesta	0.086 0.086 0.086	900 900 900	0.0096 0.0096 0.0096	09.02.97 09.16.97 09.30.97	BBCH 83 (start of ripening)	Whole fruit	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	0 7 14 21 28
34-99-178 [1/] Trial: 703680 DVG5- North east of Cologne, Germany SC, 240 g/l	Apple, James Grieve	0.14 0.14 0.14	1500 1500 1500	0.0096 0.0096 0.0096	07.09.97 07.23.97 08.06.97	BBCH 81 (start of ripening)	Whole fruit	0.22 0.17 <u>0.13</u> 0.12 0.06	0 7 14 21 28
34-99-178 [1/] Trial: 705144 BNL1- Geebets, Belgium SC, 240 g/l	Apple, Jonagold	0.14 0.14 0.14	1500 1500 1500	0.0096 0.0096 0.0096	08.08.97 08.23.97 09.05.97	BBCH 83–85 (start to advanced ripening)	Whole fruit	0.15 0.08 <u>0.11</u> 0.09 0.07	0 7 14 21 28
34-99-178 [1/] Trial: 705179 FRA 3- St Denis en val, France SC, 240 g/l	Apple, Granny Smith	0.12 0.12 0.12	1200 1200 1200	0.0096 0.0096 0.0096	08.14.97 08.28.97 09.12.97	BBCH 80 (start of ripening)	Whole fruit	0.16 0.11 0.096 <u>0.11</u> 0.064	0 7 14 21 28
34-99-178 [2/] Trial: 703699 DVG5 – Tönisvorst, Germany SC, 240 g/l	Pear, Vereins Dechant	0.14 0.14 0.14	1400 1400 1400	0.0096 0.0096 0.0096	08.25.97 09.08.97 09.22.97	BBCH 81	Whole fruit	0.20 0.17 <u>0.14</u> 0.05 <0.05	0 7 14 21 28

[1/] Analysis method 00470, electrospray LC-MS/MS, average recovery 85%, LOQ 0.05 mg/kg.
[2/] Analysis method 00470, electrospray LC-MS/MS, average recovery 86%, LOQ 0.05 mg/kg.

Table 60. Residues data summary from supervised trials on apples and pears in Germany, France, the UK and Belgium (Walz-Tylla, 1999c).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	water l/ha	kg ai/hl					
34-99-128 [1/] Trial: 810509 BNL1- Geetbets, Belgium SC, 240 g/l	Apple Jonagold	0.144 0.144 0.144	1500 1500 1500	0.0096 0.0096 0.0096	08.17.98 08.31.98 09.18.98	BBCH 85-86 (advanced ripening)	Whole fruit	0.08 <u>0.06</u>	0 15
34-99-128 [1/] Trial: 810525 FRA3- Uchizy, France SC, 240 g/l	Apple Starkrimson	0.120 0.120 0.120	1250 1250 1250	0.0096 0.0096 0.0096	07.23.98 08.06.98 08.20.98	BBCH 84 (start of ripening)	Whole fruit	0.16 <u>0.15</u>	0 14
34-99-128 [1/] Trial: 814946 ENG2 – Thurston, England SC, 240 g/l	Apple Golden Delicious	0.096 0.096 0.096	1000 1000 1000	0.0096 0.0096 0.0096	09.02.98 09.16.98 09.29.98	BBCH 85 (advanced ripening)	Whole fruit	0.06 <u><0.05</u>	0 14
34-99-128 [2/] Trial: 814954 DVG10– Höfchen, Germany SC, 240 g/l	Pear Conference	0.096 0.096 0.096	1000 1000 1000	0.0096 0.0096 0.0096	07.20.98 08.05.98 08.22.98	BBCH 85 (advanced ripening)	Whole fruit	0.14 <u>0.08</u>	0 14

[1/] Analysis method 00551, electrospray LC-MS/MS, average recovery 98%, LOQ 0.05 mg/kg.
[2/] Analysis method 00470, electrospray LC-MS/MS, average recovery 89%, LOQ 0.05 mg/kg.

Table 61. Residues data summary from supervised trials on apples and pears in Spain, France and Italy (Walz-Tylla, 1999d).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-130 [1/] Trial: 810517 SPA6- Pere Pescador, Spain SC, 240 g/l	Apple Suprema	0.14 0.14 0.14	1500 1500 1500	0.0096 0.0096 0.0096	08.17.98 08.31.98 09.18.98	BBCH 85-86 (Advanced ripening)	Whole fruit	0.08 <u>0.06</u>	0 15
34-99-130 [1/] Trial: 814962 IT 22- Montemarzino, Italy SC, 240 g/g	Apple Golden Delicious	0.12 0.12 0.120	1250 1250 1250	0.0096 0.0096 0.0096	07.23.98 08.06.98 08.20.98	BBCH 84 (Start of ripening)	Whole fruit	0.16 <u>0.15</u>	0 14
34-99-130 [2/] Trial: 814970 FRA2 – Avignon, France SC, 240 g/l	Pear Williams	0.14 0.14 0.14	1500 1500 1500	0.0096 0.0096 0.0096	06.18.98 07.02.98 07.17.98	BBCH 76 (Fruit 60% full size)	Whole fruit	0.13 <u>0.15</u>	0 13
34-99-130 [2/] Trial: 814989 IT 14- Ravenna, Italy SC, 240 g/l	Pear Williams	0.14 0.14 0.14	1500 1500 1500	0.0096 0.0096 0.0096	06.30.98 07.14.98 07.28.98	BBCH 81 (Start of ripening)	Whole fruit	0.13 <u>0.09</u>	0 15

[1/] Analysis method 00551, electrospray LC-MS/MS, average recovery 98%, LOQ 0.05 mg/kg.
[2/] Analysis method 00551, electrospray LC-MS/MS, average recovery 89%, LOQ 0.05 mg/kg.

Stone fruits

Supervised trials on stone fruits were conducted in the USA and Europe during 1998 and 1999 (Tables 62-65).

A total of 21 supervised trials were reported on stone fruits in locations representative of the major growing areas of the United States (Guo *et. al.,* 2000, report No. 34-00-75). Six trials were on cherries, nine on peaches and six on plums. For each commodity, at least one trial was a decline study and another was a bridging study, intended to compare results using the SC formulation with those obtained using the WP formulation. Supervised trials on plums and on peaches received 6 applications of the WP formulation, containing 800 g/kg methoxyfenozide, at a nominal rate of 0.3 lb ai/acre (0.336 kg ai/ha) per application. For the trials on cherries, three treatments were applied at a rate of 0.3 lb ai/acre (0.34 kg ai/ha) per application. The GAP information for the registered uses of methoxyfenozide in the U.S. for stone fruits is as follows:

Crop	Formulation type/conc.	Method	Application				PHI days
			Rate kg ai/ha	Spray conc. kg ai/hl	max/season kg ai/ha/season	Interval days	
Peaches, plums, nectarines, prunes	SC 240 g/l WP 800 g/kg	Foliar	0.13–0.28 (0.12–0.25 lb/acre)	0.014–0.06	1.1 kg ai/ha/season	10-18	7
Cherries	SC 240 g/l	Foliar	0.13–0.28 (0.12–0.25 lb/acre)	0.014–0.06	0.95 kg ai/ha/season	10-18	7

Samples from all trials were harvested 7 days after the final application. All stone fruit samples were frozen immediately after collection and maintained at about –25ºC (-14ºF) until analysis, in about 335 days.

Five supervised trials, three on peaches and two on nectarines, were conducted in France, Italy and Spain in 1998 (Walz-Tylla, 1999e, Report No. 34-99-131). Four additional trials, three on peaches and one on nectarines, were conducted in Spain and Italy in 1999 (Hoffman, 1999, Report No. 34-00-02). A suspension concentrate (SC) containing 240 g/l methoxyfenozide was applied twice to peach and nectarine trees at a spray concentration of 0.05% (0.012 kg ai/hl). The rate per application was

0.13-0.18 kg ai/ha. Treatments were conducted at 14-day intervals, with the last application made 7 days prior to harvest. There is no finalized GAP.

Table 62. Residues data summary from supervised trials on peaches in the USA (Guo *et. al.*, 2000).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-00-75	Peach	0.34	0.045	750	06.10.99	Fruit 5.6–7.3	Whole fruit		
Trial: 1559907	Baby gold	0.34	0.045	760	06.25.99	cm diameter		1.4	7
East Williamson,		0.34	0.045	750	07.13.99				
NY		0.34	0.045	750	07.24.99				
		0.34	0.045	760	08.10.99				
WP, 800 g/kg		0.34	0.045	750	08.25.99				
34-00-75	Peach	0.34	0.035	960	05.06.99	Fruit 2–2.5 in	Whole fruit	0.32	7
Trial: 1559908	Contender	0.34	0.035	960	05.17.99	(5.1–6.4 cm)			
Monetta, SC		0.34	0.035	960	05.27.99	diameter			
		0.34	0.036	940	06.07.99				
WP, 800 g/kg		0.34	0.036	940	06.19.99				
		0.34	0.036	940	07.05.99				
34-00-75	Peach	0.34	0.013	2600	05.20.99	Ripe fruit	Whole fruit	0.64	7
Trial: 1559909	O'Henry	0.34	0.013	2600	06.01.99	2.5 in (6.4 cm)			
Lakeview, CA		0.35	0.013	2600	06.15.99	diameter			
		0.34	0.013	2600	06.29.99				
WP, 800 g/kg		0.34	0.013	2700	07.09.99				
		0.34	0.013	2600	07.21.99				
34-00-75	Peach	0.34	0.053	640	05.15.99	Fruit 2.5-3 in	Whole fruit	1.0	0
Trial: 1559910	Redskin	0.34	0.052	650	05.22.99	(6.4-7.6 cm)		0.91	0
Winterville, GA		0.34	0.044	770	06.01.99	diameter		0.88	7
		0.34	0.11	310	06.11.99			0.42	14
WP, 800 g/kg		0.34	0.048	710	06.21.99			0.53	14
		0.34	0.040	840	07.01.99			0.23	21
								0.33	21
34-00-75	Peach	0.34	0.036	940	06.15.99	Fruit 2.5–3 in	Whole fruit	0.78	7
Trial: 1559911	Reliance	0.34	0.036	940	06.25.99	(6.4-7.6 cm)			
Egg Harbor, WI		0.35	0.037	950	07.06.99	diameter,			
		0.34	0.036	940	07.16.99	80% red			
WP, 800 g/kg		0.34	0.036	940	07.27.99				
		0.34	0.036	940	08.06.99				
34-00-75	Peach	0.34	0.039	870	05.06.99	Fruit maturing	Whole fruit	4.2 [3/]	7
Trial: 1559912	La Flesiana	0.34	0.039	880	05.13.99			3.8 [3/]	7
Burr, TX		0.34	0.039	870	05.21.99				
		0.34	0.039	870	05.31.99				
WP, 800 g/kg		0.34	0.039	870	06.07.99				
		0.35	0.039	890	06.14.99				
34-00-75	Peach	0.34	0.024	1400	05.11.99	Maturing,	Whole fruit	0.98	
Trial: 1559913	O'Henry	0.34	0.024	1400	05.21.99	beginning to			
Madera, CA		0.34	0.024	1400	06.01.99	colour			
		0.34	0.024	1400	06.11.99				
WP, 800 g/kg		0.34	0.024	1400	06.22.99				
		0.34	0.024	1400	07.02.99				
34-00-75	Peach	0.34	0.015	2300	05.25.99	Mostly mature	Whole fruit	0.88	7
Trial: 1559914	Carson	0.34	0.014	2400	06.04.99	fruit			
Woodville, CA		0.34	0.015	2300	06.17.99				
		0.34	0.014	2400	07.02.99				
WP, 800 g/kg		0.36	0.014	2500	07.19.00				
		0.34	0.015	2300	08.02.99				
34-00-75	Peach	0.34	0.013	2600	06.04.99	Fruit 3-4 in		0.54	7
Trial: 1559915	Red Sun	0.34	0.013	2600	06.16.99	(7.6-10.2 cm)			
Porterville, CA		0.34	0.013	2600	07.01.99	in diameter,			
		0.34	0.013	2600	07.13.99	colouring and			
WP, 800 g/kg		0.34	0.013	2600	07.26.99	softening			
		0.34	0.013	2600	08.05.99				

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-00-75 [4/]	Peach	0.33	0.013	2600	06.04.99	Fruit 3-4 in (7.6-10.2 cm) in diameter, colouring and softening	Whole fruit	0.50	
Trial: 1559915	Red Sun	0.34	0.013	2600	06.16.99				
Porterville, CA		0.33	0.013	2600	07.01.99				
		0.33	0.013	2600	07.13.99				
SC, 240 g/l		0.34	0.013	2600	07.26.99				
		0.32	0.013	2400	08.05.99				

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method (TR-34-99-26) HPLC with UV detection, average recovery 98.1%, LOQ 0.02 mg/kg.
[3/] Not included due to deviations from GAP (application timing).
[4/] Bridging study.

Table 63. Residues data summary from supervised trials on cherries in the USA (Guo *et. al.*, 2000).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/h [1/]	Water l/ha					
34-00-75	Cherry, sour, Montmorency	0.34	0.0	940	06.15.99	Fruit red and nearly ripe	Whole fruit	0.52	7
Trial: 1559901		0.34	0.036	940	06.25.99				
Sturgeon Bay, WI		0.34	0.036	940	07.09.99				
WP, 800 g/kg									
34-00-75	Cherry, sour, Montmorency	0.34	0.056	610	05.20.99	Fruit very early maturity, 100% red	Whole fruit	1.2	0
Trial: 1559902		0.34	0.056	610	06.11.99			0.90	0
Conklin, MI		0.34	0.056	610	06.25.99			0.43	7
WP, 800 g/kg								0.28	14
								0.29	14
								0.38	21
								0.31	21
34-00-75	Cherry, sweet, Bing	0.34	0.036	950	04.26.99	85% colour change to yellow and 25% pink		0.26	7
Trial: 1559903		0.34	0.036	940	05.06.99				
Hughson, CA		0.34	0.036	940	05.18.99				
WP, 800 g/kg									
34-00-75	Cherry, sweet, Bing	0.35	0.036	960	05.14.99	Fruit 100% pink to red		0.56	7
Trial: 1559904		0.34	0.036	940	05.24.99				
Hughson, CA		0.34	0.036	940	06.03.99				
WP, 800 g/kg									
34-00-75	Cherry, sweet, Bing	0.34	0.036	930	05.24.99	Medium to dark red fruit	Whole fruit	0.19	7
Trial: 1559905		0.34	0.037	920	06.14.99				
Fruitland, ID		0.33	0.036	920	06.24.99				
WP, 800 g/kg									
34-00-75	Cherry, sweet, Bing	0.34	0.018	1900	05.05.99	Fruit 80% ripe; colour progressing	Whole fruit	0.34	7
Trial: 1559906		0.34	0.018	1900	05.24.99				
Ephrata, WA		0.33	0.017	1900	06.16.99				
WP, 800 g/kg									
34-00-75 [3/]	Cherry, sweet, Bing	0.33	0.018	1800	05.05.99	Fruit 80% ripe; colour progressing	Whole fruit	0.28	7
Trial: 1559906		0.35	0.108	1900	05.24.99				
Ephrata, WA		0.33	0.017	1900	06.16.99				
SC, 240 g/l									

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR-34-99-26, HPLC with UV detection, average recovery 98%, LOQ 0.02 mg/kg.
[3/] Bridging study.

Table 64. Residues data summary from supervised trials on plums and prunes in the USA (Guo *et. al.*, 2000).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-00-75	Plum	0.34	0.037	930	06.16.99	Fruit 90% ripe	Whole fruit	0.34	6
Trial: 1559916	Alderman	0.34	0.036	930	06.30.99				
Buffalo, MN		0.34	0.036	940	07.14.99				
		0.34	0.036	930	07.29.99				
WP, 800 g/kg		0.34	0.036	940	08.11.99				
		0.34	0.036	940	08.17.99				
34-00-75	Plum	0.34	0.015	2300	06.10.99	Fruit near maturity	Whole fruit	0.16	7
Trial: 1559918	Angeleno	0.34	0.015	2200	06.23.99				
Terra Bella, CA		0.34	0.015	2200	07.07.99				
		0.34	0.015	2200	07.20.99				
WP, 800 g/kg		0.34	0.015	2300	07.30.99				
		0.34	0.015	2200	08.10.99				
34-00-75	Plum	0.34	0.024	1400	05.10.99	Fruit colouring	Whole fruit	0.13	7
Trial: 1559919	Fortune	0.34	0.024	1400	05.20.99				
Madera, CA		0.34	0.024	1400	05.31.99				
		0.34	0.024	1400	06.10.99				
WP, 800 g/kg		0.34	0.024	1400	06.21.99				
		0.35	0.024	1400	07.01.99				
34-00-75	Plum	0.34	0.015	2300	06.11.99	Late season;	Whole fruit	0.15	0
Trial: 1559920	Angeleno	0.34	0.015	2300	06.23.99	Fruit 2.5 inch		0.17	0
Poplar, CA		0.34	0.015	2300	07.09.99	(6.4 cm) in		0.19	7
WP, 800 g/kg		0.34	0.015	2300	07.19.99	diameter		0.19	14
		0.34	0.015	2300	07.29.99			0.22	14
		0.34	0.014	2400	08.13.99			0.25	21
								0.22	21
34-00-75	Plum	0.34	0.020	1700	06.08.99	Fruit ripe for picking	Whole fruit	0.14	8
Trial: 1559921	Italian	0.34	0.020	1700	06.21.99				
Banks, OR		0.34	0.020	1700	07.05.99				
WP, 800 g/kg		0.34	0.020	1700	07.16.99				
		0.34	0.017	2000	08.19.99				
		0.34	0.017	2000	09.02.99				
34-00-75	Plum	0.34	0.024	1400	06.04.99	Ripe to ripening fruit	Whole fruit	0.20	7
Trial: 1559922	French	0.34	0.024	1400	06.14.99				
Madera, CA	Prune	0.34	0.024	1400	06.24.99				
		0.34	0.024	1400	07.09.99				
WP, 800 g/kg		0.34	0.024	1400	07.22.99				
		0.34	0.024	1400	08.12.99				
34-00-75 [3/]	Plum	0.33	0.035	930	06.16.99	Fruit 90% ripe	Whole fruit	0.30	6
Trial: 1559916	Alderman	0.34	0.035	930	06.30.99				
Buffalo, MN		0.34	0.036	940	07.14.99				
		0.34	0.035	930	07.29.99				
SC, 240 g/l		0.34	0.036	950	08.11.99				
		0.34	0.036	950	08.17.99				

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR-34-99-26, HPLC with UV detection, average recovery 101.9%, LOQ 0.02 mg/kg.
[3/] Bridging study.

Table 65. Residues data summary from supervised trials on peaches and nectarines in Spain, France and Italy (Walz-Tylla, 1999e; Hoffman, 1999).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-131 [1/]	Peach	0.15	1200	0.012	06.02.98	BBCH 75	Whole fruit	0.09	0
Trial: 810851	Mery Gen	0.15	1200	0.012	06.16.98	Half final size		0.07	7
FRA2 – Eyragues	Free							0.07, 0.13	14
France								0.09	21
SC, 240 g/l									

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-131 [1/] Trial: 812943 IT 16 – Cisterna, Italy SC, 240 g/l	Peach Fairtime	0.18 0.18	1500 1500	0.012 0.012	08.04.98 08.18.98	BBCH 78 80% of final size	Whole fruit	0.12 0.10 <0.05 <0.05	0 7 14 21
34-99-131 [1/] Trial: 814997 SPA1 –La Fortesa, Spain SC, 240 g/l	Peach Merril July Lady	0.138 0.138	1200 1200	0.012 0.012	06.17.98 07.01.98	BBCH 86 Advanced colouring	Whole fruit	0.20 0.11 0.11 0.07	0 8 14 21
34-99-131 [1/] Trial: 815004 FRA2 –Eyragues, France SC, 240 g/l	Nectarine Belinda	0.18 0.18	1500 1500	0.012 0.012	07.23.98 08.06.98	BBCH 89 Ripe for consumption	Whole fruit	0.24 0.21	0 7
34-99-131 Trial: 815012 SPA1-Vilamacolum, Spain SC, 240 g/l	Nectarine Snow-green	0.18 0.18	1500 1500	0.012 0.012	06.09.98 06.23.98	BBCH 86 Advanced colouring	Whole fruit	0.11 0.09 0.13 0.07	0 7 13 20
34-00-02 [2/] Trial: 1999 0303/0 SPA1 – La Forteza, Spain SC, 240 g/l	Peach AM 40	0.12 0.12	1000 1000	0.012 0.012	06.01.99 06.15.99	BBCH 87	Whole fruit	0.20 0.13 0.08 0.09	0 8 14 21
34-00-02 [2/] Trial: 1999 0304/9 IT 14 – Ravenna, Italy SC, 240 g/l	Peach Red Haven	0.165 0.165	1400 1400	0.012 0.012	06.30.99 07.14.99	BBCH 85	Whole fruit	0.09 0.06	0 7
34-00-02 [2/] Trial: 1999 0305/7 SPA1 –La Fortesa, Spain SC, 240 g/l	Peach Royal Glory	0.15 0.15	1200 1200	0.012 0.012	06.15.99 06.29.99	BBCH 89	Whole fruit	0.17 0.10	0 7
34-00-02 [2/] Trial: 1999 0306/5 IT14 – Ravenna, Italy SC, 240 g/l	Nectarine Sweet Lady	0.132 0.132	1100 1100	0.012 0.012	08.03.99 08.17.99	BBCH 87	Whole fruit	0.25 0.15	0 7

[1/] Analysis method 00551, electrospray LC-MS/MS, average recovery 97%, LOQ 0.05 mg/kg.
[2/] Analysis method 00551, electrospray LC-MS/MS, average recovery 96%, LOQ 0.05 mg/kg.

Small fruits and berries

Supervised trials on grapes were conducted in the USA and Europe during 1997 to 1998 (Tables 68-70).

Thirteen supervised trials were conducted in major grape producing areas of the USA in 1998-1999 (Yoshida, 1999c, report No. 34-99-77). Nine of the trials were conducted in California and one each in New York, Pennsylvania, Oregon and Washington. All trials consisted of two plots, one control and one treated. Treated plots received 3 applications of a WP formulation containing 800 g/kg methoxyfenozide at a rate of 0.25 lb ai/acre (0.28 kg ai/ha) per application, using ground-driven air-blast equipment. The second application was made about 35 days after the first treatment and the third application was made 10-14 days after the second. Bridging trials were also conducted with an SC formulation containing 240 g/l methoxyfenozide. For these trials, 3 applications of the SC formulation were made at a rate of 0.25 lb ai/acre (0.28 kg ai/ha). The GAP information for registered uses of methoxyfenozide on grapes is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. or max/season	Interval, days	
WP 800 g/kg SC 240 g/l	Foliar	0.07 – 0.28 (0.06 - 0.25 lb/acre)	0.02 - 0.075	0.84 kg ai/ha/season	10-18	30

Samples were maintained in freezers for up to 164 days before analysis (Table 66).

Five supervised trials were conducted on table grapes in 1997, in Greece, Italy, France and Portugal (Seym and Deissler, 1999m, report No. 34-99-117). Four additional trials were conducted in 1998 in Italy, France and Spain (Walz-Tylla, 1999f, report No. 34-99-126). In all nine trials, four applications of an SC formulation containing 240 g/l methoxyfenozide were applied at a rate of 0.096 kg ai/ha per application. The spray volume was 1000 l/ha in Italy, Spain and Portugal and 100 l/ha in France. Treatments were made at intervals of 13 to 16 days, with the last application 7 days prior to harvest (Table 67). There is no registered GAP.

Two supervised trials were conducted on grape vines in 1996 in Portugal and France (Heinemann and Seym, 1998a, Report No. 34-98-190). Three separate trials were also conducted in different regions in Germany at about the same period (Seym and Deissler, 1998d, report No. 34-99-135). In 1997, three additional trials were conducted in Spain, Italy and southern France and six in Germany and northern France (Seym and Deissler, 1999c and 1999d, reports No. 34-99-120 and 34-99-124). All fourteen trials applied treatments of an SC formulation containing 240 g/l methoxyfenozide at 13- to 21-day intervals, at the rate of 0.096 kg ai/ha, with the last treatment 14 days before harvest. The spray volume was 1000-1600 l/ha, except in France, where the spray volume used was 100 l/ha (Table 68). There is no registered GAP.

Table 66. Residues data summary from supervised trials on grapes in the USA (Yoshida, 1999c).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-77 Trial: 1559825 Ducor, CA WP, 800 g/kg	Grapes Crimson seedless (red)	0.28 0.29 0.30	0.056 0.057 0.057	500 510 520	07.27.98 08.31.98 09.10.98	Mature, crop height 6 ft,	Bunches of grapes	0.41 0.37 0.29 0.26 0.20 0.16 0.14	0 0 14 14 30 40 40
34-99-77 Trial: 1559826 Paso Robles, CA WP, 800 g/kg	Grapes Cabernet (red)	0.29 0.34 0.29	0.035 0.035 0.031	830 960 930	07.09.98 08.13.98 08.24.98	Beginning of ripening, crop height 6 ft	Bunches of grapes	0.78 1.1 0.26 0.41 <u>0.45</u> 0.23 0.19	0 0 14 14 30 40 40
34-99-77 Trial: 1559820 Orefield, PA WP, 800 g/kg	Grapes Niagara (white)	0.29 0.30 0.29	0.043 0.043 0.043	680 680 660	06.16.98 07.20.98 07.30.98	3/4 to 7/8 in (1.9 – 2.2 cm) berry; crop height 7-8 ft	Bunches of grapes	<u>0.21</u>	29
34-99-77 Trial: 1559821 Dundee, NY WP, 800 g/kg	Grapes DeChaunac (red)	0.28 0.28 0.28	0.060 0.060 0.060	470 470 470	07.10.98 08.14.98 08.24.98	Post bloom early maturity	Bunches of grapes	<u>0.84</u>	30
34-99-77 Trial: 1559832 Hood River, OR WP, 800 g/kg	Grapes Chardonnay (White)	0.28 0.28 0.28	0.013 0.014 0.013	2200 2000 2100	07.07.98 08.30.98 09.09.98	At maturity. crop 5-8 ft	Bunches of grapes	<u><0.02</u>	30
34-99-77 Trial: 1559829 Biola, CA WP, 800 g/kg	Thompson seedless (white)	0.28 0.28 0.28	0.040 0.040 0.040	710 700 700	06.17.98 07.22.98 07.31.98	Small berries, crop 5-6 ft	Bunches of grapes	<u>0.26</u>	31

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-77 Trial: 1559830 Terra Bella, CA WP, 800 g/kg	Grapes Thompson seedless (white)	0.28 0.28 0.28	0.042 0.042 0.040	670 670 690	06.26.98 07.30.98 08.09.98	Post-verasion Crop 4 ft	Bunches of grapes	0.21	30
34-99-77 Trial: 1559823 Woodland, CA WP, 800 g/kg	Grapes Cabernet (red)	0.28 0.28 0.28	0.059 0.034 0.034	480 800 800	07.06.98 08.13.98 09.03.98	Full colour change, crop 6-10 ft	Bunches of grapes	0.46	29
34-99-77 Trial: 1559824 Temecula, CA WP, 800 g/kg	Grapes Chardonnay (white)	0.290 0.270 0.280	0.040 0.040 0.040	720 690 720	07.03.98 08.07.98 08.18.98	Post-verasion Crop 5-6 ft	Bunches of grapes	0.34	30
34-99-77 Trial: 1559827 Fresno, CA WP, 800 g/kg	Grapes Thompson seedless (white)	0.280 0.280 0.280	0.040 0.040 0.040	700 700 700	06.17.98 07.22.98 07.31.98	Small berries, crop 5-6 ft	Bunches of grapes	0.33	31
34-99-77 Trial: 1559831 George, WA WP, 800 g/kg	White Reisling (white)	0.280 0.280 0.280	0.030 0.030 0.030	940 930 940	07.15.98 08.21.98 08.31.98	14-16 mm berry size	Bunches of grapes	0.39	30
34-99-77 Trial 1559822 Upper Lake, CA WP, 800 g/kg	Grapes Sauvignon Blanc (white)	0.28 0.28 0.29	0.060 0.062 0.060	460 450 480	07.14.98 08.19.98 08.29.98	13-14º brix, crop 8 ft	Bunches of grapes	0.52	30
34-99-77 Trial: 1559828 San Obispo, CA WP, 800 g/kg	Grapes Chardonnay (white)	0.280 0.280 0.280	0.030 0.030 0.030	940 950 950	08.07.98 09.16.98 09.28.98	Beginning of ripening, crop 7 ft	Bunches of grapes	0.32	30
34-99-77 Trial: 1559827 [3/] Fresno, CA SC, 240 g/l	Grapes Thompson seedless (white)	0.280 0.280 0.280	0.040 0.040 0.040	700 700 700	06.17.98 07.22.98 07.31.98	Small berries, crop 5-6 ft	Bunches of grapes	0.26	31
34-99-77 Trial: 1559828 San Obispo, CA SC, 240 g/l[3]	Grapes Chardonnay (white)	0.280 0.280 0.280	0.029 0.030 0.030	700 900 950	08.07.98 09.16.98 09.28.98	Beginning of ripening, crop 7 ft	Bunches of grapes	0.52	30

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR-34-98-186, HPLC with UV detection, average recovery 97.8%, LOQ 0.02 mg/kg.
[3/] Bridging study.

Table 67. Residues data summary from supervised trials on table grapes in Greece, Italy, France, Portugal and Spain (Seym and Deissler, 1999m; Walz-Tylla, 1999f).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-117 Trial: 703591 [2/] GRI 1: Athens, Greece SC, 240 g/l	Table grapes Soultanina (white)	0.096 0.096 0.096 0.096	1000 1000 1000 1000	0.0096 0.0096 0.0096 0.0096	07.15.97 07.29.97 08.14.97 08.28.97	BBCH 87 (softening of berries)	Bunches of grapes	0.40 0.32 0.27 0.26 0.25	0 3 7 7 14
34-99-117 Trial: 703605 [2/] IT 16: Latina, Italy SC, 240 g/l	Table grapes Matilde (white)	0.096 0.096 0.096 0.096	1000 1000 1000 1000	0.0096 0.0096 0.0096 0.0096	06.30.97 07.14.97 07.28.97 08.11.97	BBCH 85 (softening of berries)	Bunches of grapes	0.37 0.28 0.30 0.32	0 3 7 14
34-99-117 Trial: 703613 [2/] FRA 2: Avignon, France SC, 240 g/l	Table grapes Chasselas (white)	0.096 0.096 0.096 0.096	100 100 100 100	0.096 0.096 0.096 0.096	06.23.97 07.07.97 07.21.97 08.04.97	BBCH 81 (beginning of ripening)	Bunches of grapes	0.40 0.74 0.20 0.29	0 3 8 14

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-117	Table grapes	0.096	1000	0.0096	06.30.97	BBCH 85	Bunches of	0.54	0
Trial: 705357 [2/]	Blush	0.096	1000	0.0096	07.14.97	(softening of	grapes	0.57	3
IT 17: Bari,	(red)	0.096	1000	0.0096	07.28.97	berries)		0.49	7
Italy		0.096	1000	0.0096	08.11.97			0.56	7
SC, 240 g/l								0.48	14
34-99-117	Table grapes	0.096	1000	0.0096	06.26.97	BBCH 81	Bunches of	0.47	0
Trial: 705365 [2/]	Alfonso	0.096	1000	0.0096	07.09.97	(beginning of	grapes	0.36	3
POR 2: Lisbon,	Lavalé	0.096	1000	0.0096	07.22.97	ripening)		0.32	7
Portugal	(red)	0.096	1000	0.0096	08.04.97			0.32	14
SC, 240 g/l									
34-99-126	Table grapes	0.096	1000	0.0096	07.07.98	BBCH 85	Bunches of	0.12	0
Trial: 810487 [3/]	Blush	0.096	1000	0.0096	07.21.98	(softening of	grapes	0.31	7
IT 17: Bari,	(red)	0.096	1000	0.0096	08.04.98	berries)	Berries	0.30	7
Italy		0.096	1000	0.0096	08.18.98				
SC, 240 g/l									
34-99-126	Table grapes	0.096	100	0.096	07.01.98	BBCH 85	Bunches of	0.14	0
Trial: 810495 [3/]	Chasselas	0.096	100	0.096	07.15.98	(softening of	grapes	0.13	7
FRA 2: Avignon,	(white)	0.096	100	0.096	07.29.98	berries)	Berries	0.10	7
France		0.096	100	0.096	08.12.98				
SC, 240 g/l									
34-99-126	Table grapes	0.096	1000	0.0096	06.19.98	BBCH 88	Bunches of	0.14	0
Trial: 815020 [3/]	Cardinal	0.096	1000	0.0096	07.03.98	(ripe for	grapes	0.08	7
SPA 2: Valencia,	(red)	0.096	1000	0.0096	07.17.98	harvest)			
Spain		0.096	1000	0.0096	07.31.98				
SC, 240 g/l									
34-99-126	Table grapes	0.096	1000	0.0096	07.02.98	BBCH 81	Bunches of	0.25	0
Trial: 815039 [3/]	Italia	0.096	1000	0.0096	07.16.98	(beginning of	grapes	0.14	7
IT 16: Latina,	(white)	0.096	1000	0.0096	07.30.98	ripening)	Berries	0.09	7
Italy		0.096	1000	0.0096	08.13.98				
SC, 240 g/l									

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method 00470, electrospray LC-MS/MS, average recovery 99%, LOQ 0.05 mg/kg.
[3/] Analysis method 00470, electrospray LC-MS/MS, average recovery 94%, LOQ 0.05 mg/kg.

Table 68. Residues data summary from supervised trials on grapes (wine) in Portugal, France, Spain, Italy and Germany (Heinemann & Seym, 1998a; Seym & Deissler, 1998d, 1999c, 1999d).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-98-190	Grapes	0.096	1000	0.0096	08.08.96	BBCH 89	Bunches of	0.56	0
Trial: 605883 [2/]	Periquita	0.096	1000	0.0096	08.22.96	(ripe for	grapes	0.14	7
POR 2: Lisbon,	(red)	0.096	1000	0.0096	09.05.96	harvest)		0.14	14
Portugal								0.12	21
SC, 240 g/l									
34-98-190	Grapes	0.096	100	0.096	08.12.96	BBCH 87	Bunches of	0.29	0
Trial: 605891 [2/]	Clairette	0.096	100	0.096	08.26.96	(softening of	grapes	0.22	7
FRA 2: Sorgues,	(white)	0.096	100	0.096	09.09.96	berries)		0.17	14
France								0.18	21
SC, 240 g/l									
34-99-120	Grapes	0.096	100	0.096	07.31.97	BBCH 85	Bunches of	0.070	0
Trial: 703532 [3/]	Syrah	0.096	100	0.096	08.14.97	(softening of	grapes	0.063	7
FRA 2: Orange,	(red)	0.096	100	0.096	08.28.97	berries)		0.12	14
France								0.13	21
SC, 240 g/l							Berries	0.075	14
34-99-120	Grapes	0.096	1000	0.0096	07.31.97	BBCH 87	Bunches of	0.12	0
Trial: 703583 [3/]	Cabernet	0.096	1000	0.0096	08.13.97	(softening of	grapes	0.079	14
SPA 1,	Sauvignon	0.096	1000	0.0096	08.26.97	berries)	Berries	0.080	14
Barcelona, Spain	(red)								
SC, 240 g/l									

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-120 Trial: 705446 [3/] IT 22: Alessandria, Italy SC, 240 g/l	Grapes Cortese (white)	0.096 0.096 0.096	1000 1000 1000	0.0096 0.0096 0.0096	07.24.97 08.07.97 08.21.97	BBCH 84 (berries bright in colour)	Bunches of grapes Berries	0.68 0.57 0.43 0.32 <u>0.42</u>	0 7 14 21 14
34-99-124 Trial: 703524 [3/] FRA 3: Brinay, France SC, 240 g/l	Grapes Sauvignon (white)	0.096 0.096 0.096	100 100 100	0.096 0.096 0.096	07.21.97 08.11.97 08.25.97	BBCH 83 (berries bright in colour)	Bunches of grapes Berries	0.16 0.16 0.14 0.12 0.10 <u>0.096</u>	0 7 14 21 28 14
34-99-124 Trial: 703540 [3/] DSW1: Albig, Germany SC, 240 g/l	Grapes Portugieser (red)	0.096 0.096 0.096	1600 1600 1600	0.006 0.006 0.006	08.05.97 08.19.97 09.03.97	BBCH 81 (berries begin to ripen)	Bunches of grapes Berries	0.30 0.20 0.22 0.002, 0.001 <u>0.22</u>	0 7 14 21 14
34-99-124 Trial: 703559 [3/] DSW1: Freinsheim, Germany SC, 240 g/l	Grapes Reisling (white)	0.096 0.096 0.096	1600 1600 1600	0.006 0.006 0.006	08.19.97 09.03.97 09.17.97	BBCH 81-85 (From start of ripening to softening of berries)	Bunches of grapes Berries	0.28 0.30 0.26 <u>0.21</u>	0 14 21 14
34-99-124 Trial: 703567 [3/] FRA 3: Brinay, France SC, 240 g/l	Grapes Pinot Meunier (red)	0.096 0.096 0.096	100 100 100	0.096 0.096 0.096	07.21.97 08.11.97 08.25.97	BBCH 85	Bunches of grapes Berries	0.45 0.25 0.26 <u>0.26</u>	0 14 21 14
34-99-124 Trial: 705411 [3/] DSW1: Albig, Germany SC, 240 g/l	Grapes Müller-Thurgau (white)	0.096 0.096 0.096	1600 1600 1600	0.006 0.006 0.006	08.05.97 08.19.97 09.03.97	BBCH 81	Bunches of grapes Berries	0.23 0.20 0.16 <u>0.16</u>	0 14 21 14
34-99-124 Trial: 705438 [3/] FRA 3: Clery St. André, France SC, 240 g/l	Grapes Gamay (red)	0.096 0.096 0.096	100 100 100	0.096 0.096 0.096	07.21.97 08.11.97 08.25.97	BBCH 85	Bunches of grapes Berries	0.61 0.52 0.50 0.58 <u>0.58</u>	0 14 21 14
34-99-135 Trial: 605867 [2/] DSW1: Albig, Germany SC, 240 g/l	Grapes Portugieser (red)	0.096 0.096 0.096	1600 1600 1600	0.006 0.006 0.006	08.07.96 08.20.96 09.04.96	BBCH 81-85	Bunches of grapes	0.19 0.16 0.12 <u>0.17</u> 0.12	0 7 14 21 28
34-99-135 Trial: 605875 [2/] DSW1: Albig, Germany SC, 240 g/l	Grape Müller-Thurgau (white)	0.096 0.096 0.096	1600 1600 1600	0.006 0.006 0.006	08.07.96 08.20.96 09.04.96	BBCH 85	Bunches of grapes	0.17 0.16 <u>0.15</u> 0.14 0.15	0 7 14 21 28
34-99-135 Trial: 607525 [2/] DSW3, Kirrweiler, Germany SC, 240 g/l	Grapes Müller-Thurgau (white)	0.096 0.096 0.096	1600 1600 1600	0.006 0.006 0.006	08.02.96 08.16.96 08.30.96	BBCH 81	Bunches of grapes	0.34 0.30 <u>0.28</u> 0.18 0.25	0 7 14 21 28

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method 00470, electrospray LC-MS/MS, average recovery 107%, LOQ 0.05 mg/kg.
[3/] Analysis method 00470, electrospray LC-MS/MS, average recovery 99%, LOQ 0.05 mg/kg.

Brassica vegetables

Supervised trials on broccoli and cabbage were reported from the USA in 1998. Details of these trials are summarized in Tables 69-70.

Eight field trials on broccoli were conducted in major vegetable growing areas in the United States in 1998-1999 (Carpenter, 1999d, report No. 34-99-76). All treated plots received four applications (except for one trial in Texas which received 5 applications) of the WP formulation containing 800 g/kg methoxyfenozide, at a rate of 0.25 lb ai/acre or 0.28 kg ai/ha per application, with the last application made one day before harvest. The applications were made at intervals of 7 to 14 days. The total nominal treatment was 1 lb ai/acre/season or 1.12 kg ai/ha/season. The bridging trials were also treated with an SC (240 g/l) formulation of methoxyfenozide, at the rate of 0.25 lb ai/acre or 0.28 kg ai/ha. The equipment used for foliar application was either a tractor-mounted boom sprayer or a powered backpack sprayer. A minimum of 14.5 gallons of water/acre (136 l/ha) was used for each application. The GAP for registered use of methoxyfenozide on brassica vegetables, including broccoli, in the USA is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications or max/season	Interval, days	
WP 800 g/kg SC 240 g/l	Foliar	0.07 – 0.28 (0.06 – 0.25 lb ai/acre)	0.04 – 0.30	1.12 kg ai/ha/season	10-14	1

Each sample consisted of a minimum of 12 heads (approximately 2.5 lbs). All samples were frozen upon collection and maintained frozen until analysis, up to 2-5 months (Table 69).

Table 69. Residues data summary from supervised trials on broccoli in the USA (Carpenter, 1999d).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue, mg/kg [2/]	PHI, days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-76 Trial:61198058 Ulvade, TX WP, 800 g/kg	Broccoli, Legacy	0.27 0.28 0.27 0.28 0.27	0.19 0.15 0.19 0.20 0.19	140 190 140 140 140	10.12.98 10.22.98 11.05.98 11.16.98 11.30.98	At maturity	Broccoli heads	0.52	1
34-99-76 Trial: 61198059 Oznard, CA WP, 800 g/kg	Broccoli, Emperor	0.29 0.28 0.28 0.28	0.053 0.053 0.053 0.053	550 530 540 540	10.20.98 10.27.98 11.04.98 11.11.98	At maturity	Broccoli heads	0.76	1
34-99-76 Trial: 61198060 Corvallis, OR WP, 800 g/kg	Broccoli, Arcadia	0.29 0.30 0.29 0.28	0.11 0.11 0.11 0.11	270 280 280 270	08.13.98 08.27.98 09.10.98 09.24.98	At Maturity	Broccoli Heads	0.70	1
34-94-76 [3/] Trial: 61198061 Porterville, CA WP, 800 g/kg	Broccoli, Liberty	0.30 0.30 0.30 0.30	0.12 0.12 0.12 0.12	240 240 240 240	12.01.98 12.08.98 12.15.98 12.22.98	At maturity	Broccoli heads	1.4	1
34-99-76 [4/] Trial: 61198062 Somerton, AZ WP, 800 k/kg	Broccoli, Everest	0.30 0.30 0.29 0.29	0.13 0.13 0.13 0.13	230 230 230 230	11.04.98 11.18.98 11.26.98 12.03.98	At maturity	Broccoli heads	1.6	1
34-99-76 [3/] Trial: 611980632 San Ardo, CA WP, 800 g/kg	Broccoli, Green Belt	0.30 0.30 0.30 0.30	0.11 0.11 0.11 0.11	280 280 280 280	10.01.98 10.08.98 10.22.98 10.29.98	At maturity	Broccoli heads	0.74 0.89 0.58 0.34	1 3 7 10
34-99-76 [4/] Trial: 61198061 Porterville, CA SC, 240 g/l	Broccoli, Liberty	0.27 0.26 0.26 0.27	0.11 0.11 0.11 0.11	240 240 230 230	12.01.98 12.08.98 12.15.98 12.22.98	At maturity	Broccoli heads	0.98	1

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue, mg/kg [2/]	PHI, days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-76 [4/]	Broccoli,	0.27	0.11	240	11.04.98	At	Broccoli	1.6	1
Trial: 61198062	Everest	0.26	0.11	230	11.18.98	maturity	heads		
Somerton, AZ		0.26	0.11	240	11.26.98				
SC, 240 g/l		0.25	0.11	220	12.03.98				

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR 34-98-186, HPLC with UV detection, average recovery 86%, LOQ 0.02 mg/kg.
[3/] Decline study.
[4/] Bridging study.

Nine supervised trials on cabbage were conducted in 1998-1999 in major vegetable-growing areas of the USA (Carpenter, 1999d, report No. 34-99-76). Conditions were as for broccoli, above. A minimum of 15.2 gallons of water/acre (142 l/ha) was used for each application. The proposed GAP for use of methoxyfenozide on brassica vegetables, including cabbage, is given above under broccoli. Each sample consisted of a minimum of 12 cabbage heads (approximately 2.5 lbs). All samples were frozen upon collection and maintained frozen until analysis, up to 2-5 months (Table 70).

Table 70. Residues data summary from supervised trials on cabbage in the USA (Carpenter, 1999d).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-76	Cabbage	0.30	0.13	230	08.27.98	Maturity	Cabbage head	0.93	1
Trial: 61198064	Rio Verde	0.29	0.13	220	09.08.98		with wrapper		
Hamburg, PA		0.29	0.13	220	09.16.98		leaves		
WP, 800 g/kg		0.29	0.08	350	09.29.98		Cabbage head	<0.05	1
							no wrapper	<0.05	
							leaves		
34-99-76	Cabbage	0.28	0.12	230	11.11.98		Cabbage head	2.2	1
Trial: 61198065	Stonehead	0.29	0.11	270	11.18.98		with wrapper		
Oviedo, FL		0.30	0.11	280	11.25.98		leaves		
WP, 800 g/kg		0.30	0.11	280	12.02.98		Cabbage head	0.26	1
							no wrapper	0.36	
							leaves		
34-99-76	Cabbage	0.28	0.12	230	12.03.98		Cabbage head	3.3	1
Trial: 61198066	Pennant	0.30	0.11	270	12.17.98		with wrapper		
Uvalde, TX		0.30	0.11	280	12.29.98		leaves		
WP, 800 g/kg		0.30	0.11	280	01.11.99		Cabbage head	0.075	1
							no wrapper	0.024	
							leaves		
34-99-76	Cabbage	0.30	0.11	280	10.29.98	Maturity	Cabbage head	0.57	1
Trial: 61198067	Supreme	0.30	0.11	280	11.06.98		with wrapper		
King City, CA	Vantage	0.30	0.11	280	11.13.98		leaves		
WP, 800 g/kg		0.30	0.11	280	11.21.98		Cabbage head	0.20	1
							no wrapper	0.23	
							leaves		
34-99-76 [4/]	Cabbage	0.28	0.13	220	06.08.98	Maturity	Cabbage head	0.88	1
Trial: 61198068	Market	0.29	0.13	230	06.18.98		with wrapper		
East Brunswick,	Prize	0.29	0.13	230	07.02.98		leaves		
NJ		0.29	0.13	230	07.16.98				
WP, 800 g/kg									
34-99-76 [4/]	Cabbage	0.30	0.16	190	07.15.98	Maturity	Cabbage head	0.67	1
Trial: 61198069	Quisto	0.30	0.17	180	07.22.98		with wrapper		
Arkansaw, WI		0.29	0.15	190	08.05.98		leaves		
WP, 800 g/kg		0.30	0.16	190	08.18.98				
34-99-76 [3/]	Cabbage	0.31	0.11	290	07.14.98	Maturity	Cabbage head	4.7	0
Trial: 61198070	Blue Gem	0.30	0.11	280	07.21.98		with wrapper	3.4	1
North Rose,		0.30	0.11	280	07.28.98		leaves	2.5	3
NY		0.29	0.11	280	08.04.98			6.2	7
WP, 800 g/kg								1.5	10

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-76 [4/] Trial: 61198068 East Brunswick, NJ SC, 240 g/l	Cabbage Market Prize	0.26 0.21 0.26 0.26	0.10 0.091 0.10 0.10	230 230 230 230	06.08.98 06.18.98 07.02.98 07.16.98	Maturity	Cabbage head with wrapper leaves	0.75	1
34-99-76 [4/] Trial: 61198069 Arkansaw, WI SC, 240 g/l	Cabbage Quisto	0.28 0.28 0.28 0.27	0.15 0.15 0.15 0.15	190 190 190 190	07.15.98 07.22.98 08.05.98 08.18.98	Maturity	Cabbage head with wrapper leaves	0.56	1

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR 34-98-186, HPLC with UV detection, average recovery 87%, LOQ 0.02 mg/kg.
[3/] Decline study.
[4/] Bridging study.

Fruiting vegetables

Supervised trials on fruiting vegetables (tomatoes, peppers, and egg plants) were conducted in the USA, Australia, Malaysia and Europe from 1997–1999. Trials on peppers were conducted in the USA and Europe (Tables 73 to 76) while those on tomatoes were conducted in Australia, Europe and the USA (Tables 71 to 73). Trials on egg plants were conducted in Malaysia (Table 80).

A total of thirteen supervised trials were conducted on bell and non-bell peppers in the USA (Bergin, 1999b, report No. 34-99-48). All treated plots received four applications of the 80W formulation of methoxyfenozide (800 g ai/l), at the rate of 0.25 lb ai/acre (0.28 kg ai/ha) per application, with the last application one day before harvest. The applications were made at 7- to 14-day intervals. The total nominal treatment rate was 1.0 lb ai/acre/season (1.12 kg ai/ha/season). All applications were made using tractor-, ATV-, or backpack-mounted boom sprayer equipment, delivering spray volumes ranging from 13.7 to 54.2 gallons/acre (128 to 507 l/ha). The registered GAP for the use of methoxyfenozide on fruiting vegetables, including peppers is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. or max/season	Interval, days	
SC 240 g/l WP 800 g/kg	Foliar	0.07 – 0.28 (0.06 – 0.25 lb ai/acre)	0.04 -0.30	1.12 kg ai/ha (1 lb ai/acre)	7 – 14	1

Duplicate samples, weighing 1.5-12 lbs of bell peppers and 4.3-7.5 lbs of non-bell peppers, were harvested from each plot. Samples were frozen upon collection and maintained frozen for up to 4-7.5 months, before analysis (Table 71).

Fourteen residue trials on peppers were conducted in glasshouses in Italy, Spain, Portugal, France, Germany, Belgium and the Netherlands from 1997 to 1999. These trials are described in four separate reports (Seym, and Deissler, 1999e, report No. 34-99-180; Walz-Tylla, 1999g, 1999h and 1999i, reports No. 34-99-182, 34-99-183 and 34-99-184). In each of the trials, an SC formulation containing 240 g/l of methoxyfenozide was applied three times to pepper plants at a rate of up to 0.19 kg ai/ha per application, in a maximum water volume of 2000 l/ha. The interval between applications was 7 days, with the last application made one day prior to harvest. There is no registered GAP. Samples were frozen within 24 hours and stored at -18°C, or below, for about 9 months until analysis.

Fourteen supervised trials on tomatoes were conducted in 1998 in major tomato growing areas of the USA (Bergin, 1999c, report No. 34-99-47). Each of the treated plots received four applications of the 80W (WP) formulation containing 800 g/kg methoxyfenozide, at the rate of 0.25 lb ai/acre (0.28 kg ai/ha) per application. In the bridging trials, one of the treated plots received the SC formulation containing 240 g/l methoxyfenozide, at the same rate as the 80W formulation. For all treatments, the interval between applications was 7 to 14 days. All applications were made using a tractor-, ATV-, or backpack-mounted boom sprayer, with spray volumes 20-55 gallons/acre (180-510 l/ha). The GAP for registered uses of methoxyfenozide on fruiting vegetables, including tomatoes, is given above under peppers. Each sample consisted of a minimum of 4.7 lbs (2.1 kg) of

tomatoes. All samples were frozen upon collection and maintained frozen until analysis until analysis, up to 2.5 to 7 months.

Thirteen supervised trials on tomatoes were conducted in various tomato growing areas in Australia from 1996 to 1999 (Hamblin, 1997, report No. PJH 208/97; Seidel, 1999a and 1999b, reports No. JES 610/99 and JES 611/99; English, 1999a, 1999b and 1999c, reports No. JME 250/97, JME 291/99 and JME 292/99; Mai, 1999, report No. 99/3162). In each of the trials, six treatments of an SC formulation containing 240 g/l methoxyfenozide were applied at the rate of 0.3 to 0.4 kg ai/ha per application, at intervals of 7 to 17 days between applications. Treatments were applied either by low-volume boom sprayers or high-volume sprays and, in some cases, with a miscible oil mixed with the product (e.g. D-C-Trate, D-C-Tron). The registered use on tomatoes in Australia is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No.	Interval, days	
SC 240 g/l	Foliar	0.30 or 0.40 (calculated)	0.03 or 0.04 [2/]	3	7	None [1/]

[1/] Label states that no PHI is necessary if applied as directed.
[2/] Higher rate for bush tomatoes.

Thirteen glasshouse trials on tomatoes were conducted from 1997 to 1999 in Germany, the Netherlands, Spain, Portugal, Italy, France and Belgium (Walz-Tylla, 1999j and 1999k, reports No. 34-98-138 and 34-00-03; Seym, and Deissler, 1999f, report No. 34-99-122). In each of the trials, tomato plants were treated with an SC formulation containing 240 g/l methoxyfenozide, at a rate corresponding to a spray concentration of 0.04% methoxyfenozide (0.0096 kg ai/hl). Treatments were conducted at intervals of 7 days, with the last application made 1 day prior to harvest. There is no registered GAP. Samples of about 2 to 6 kg of tomatoes were taken at each interval. Samples were immediately frozen and stored at -18°C until dispatch to the laboratory where they remained frozen until analysis. Sampling to analysis intervals were 7 to 11 months.

Two supervised trials on egg plants were conducted in Selangor, Malaysia in 1998 (Boh, 1998a, report No. Boh-1). Each plot received 4 applications of the 2F (SC) formulation of methoxyfenozide (240 g ai/l) at a rate of either 0.15 kg ai/ha or 0.3 kg ai/ha per application. Treatments were made at 7–day intervals. The GAP for registered uses of methoxyfenozide on egg plants in Malaysia is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No.	Interval, days	
SC 240 g/l	Foliar	0.1	0.023	As required	7	14

Table 71. Residues data summary from supervised trials on peppers in the USA (Bergin, 1999b).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-48 Trial: 61298014 Lucama, NC WP, 800 g/kg	Bell pepper California Wonder	0.28 0.28 0.28 0.29	0.16 0.16 0.15 0.16	180 180 190 180	06.11.98 06.25.98 07.09.98 07.23.98	Fruiting	Whole pepper	0.041	1
34-99-48 Trial: 61298015 Carlyle, IL WP, 800 g/kg	Bell pepper Better Bell	0.28 0.28 0.28 0.28	0.12 0.12 0.10 0.10	240 240 270 280	07.07.98 07.17.98 07.30.98 08.06.98	Fruiting	Whole pepper	0.049	1
34-99-48 Trial: 61298016 East Bernard, TX WP, 800 g/kg	Bell pepper Capistrono	0.28 0.29 0.29 0.28	0.20 0.21 0.22 0.15	140 140 130 130	07.08.98 07.17.98 08.01.98 08.14.98	Maturing fruit	Whole pepper	0.12	1
34-99-48 Trial: 61298017 Groom, TX WP, 800 g/kg	Non-bell pepper Anaheim	0.28 0.29 0.27 0.28	0.18 0.21 0.18 0.21	190 140 150 130	08.18.98 08.28.98 09.08.98 09.16.98	1.6 - 2.4 cm peppers	Whole pepper	0.48	1

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-48 Trial: 61298018 Porterville, CA WP, 800 g/kg	Non-bell pepper Anaheim	0.28 0.28 0.29 0.28	0.10 0.10 0.15 0.10	280 280 190 280	07.08.98 07.22.98 08.07.98 08.18.98	Green fruit	Whole pepper	0.94	1
34-99-48 [3/] Trial: 61298019 Hobe Sound, FL WP, 800 g/kg	Bell pepper Boynton Bell	0.27 0.29 0.28 0.27	0.061 0.057 0.067 0.059	440 510 420 460	10.21.98 10.28.98 11.06.98 11.13.98	Fruiting	Whole pepper	0.39 0.34 0.35 0.31 0.36	0 1 3 7 10
34-99-48 [3/] Trial: 61298020 San Ardo, CA WP, 800 g/kg	Bell Pepper California Wonder 300	0.29 0.28 0.28 0.28	0.10 0.10 0.10 0.10	280 280 280 280	08.31.98 09.08.98 09.15.98 09.25.98	Fruit maturing	Whole pepper	0.048 0.044 0.044 0.044 0.040 0.050	0 1 3 7 10
34-99-48 [4/] Trial: 61298021 Boynton Beach, FL WP, 800 g/kg	Bell pepper Boynton Bell	0.29 0.28 0.28 0.28	0.063 0.058 0.061 0.062	460 480 460 450	10.11.98 10.23.98 11.03.98 11.17.98	Fruiting	Whole pepper	0.14	1
34-99-48 [4/] Trial: 61298022 Arroyo Grande, CA WP, 800 g/kg	Bell pepper Jupiter	0.27 0.27 0.27 0.28	0.12 0.12 0.12 0.20	230 240 240 240	09.03.98 09.10.98 09.17.98 09.24.98	Mature	Whole pepper	0.16	1
34-99-48 [4/] Trial: 61298023 Claude, TX WP, 800 g/kg	Non-bell pepper Jalapeno	0.28 0.29 0.28 0.28	0.020 0.22 0.15 0.19	140 130 190 150	07.29.98 08.10.98 08.18.98 08.31.98	1.6 - 2.4 cm peppers	Whole pepper	0.26	1
34-99-48 [4/] Trial: 61298021 Boynton Beach, FL SC, 240 g/l	Bell pepper Boynton Bell	0.29 0.29 0.28 0.29	0.063 0.060 0.061 0.064	460 480 460 450	10.11.98 10.23.98 11.03.98 11.17.98	Fruiting	Whole pepper	0.16	1
34-99-48 [4/] Trial: 61298022 Arroyo Grande, CA SC, 240 g/l	Bell pepper Jupiter	0.28 0.28 0.28 0.28	0.12 0.12 0.12 0.12	230 240 240 240	09.03.98 09.10.98 09.17.98 09.24.98	Mature	Whole pepper	0.20	1
33-99-48 [4/] Trial: 61298023 Claude, TX SC, 240 g/l	Non-bell pepper Jalapeno	0.29 0.28 0.29 0.29	0.21 0.22 0.15 0.19	140 130 190 150	07.29.98 08.10.98 08.18.98 08.31.98	1.6 - 2.4 cm peppers	Whole pepper	0.40	1

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR 34-98-186, HPLC with UV detection, average recovery 96%, LOQ 0.01 mg/kg.
[3/] Decline study.
[4/] Bridging study.

Table 72. Residues data summary from supervised glasshouse trials on peppers in Portugal, Spain, Italy, France and the Netherlands (Seym and Deissler, 1999e).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last application	Commodity	Residue mg/kg [1/]	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-180 Trial: 703656 Alenquer, Portugal, POR 2 SC, 240 g/l	Pepper, Clovis	0.058 0.067 0.077	600 700 798	0.0097 0.0096 0.0096	05.20.97 05.26.97 06.02.97	BBCH 67-81	Whole fruit	0.21 0.076 0.086 0.10	0 1 3 5

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last application	Commodity	Residue mg/kg [1/]	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-180 Trial: 703664 Almeria, Spain SPA 5 SC, 240 g/l	Pepper, Roldan	0.096 0.106 0.106	1000 1100 1100	0.0096 0.0096 0.0096	04.21.97 04.28.97 05.05.97	BBCH 81	Whole fruit	0.23 0.11 0.17 0.16	0 1 3 4
34-99-180 Trial: 705314 Ragusa, Italy IT 20 SC, 240 g/l	Pepper, Trevi	0.086 0.086 0.086	900 900 900	0.0096 0.0096 0.0096	11.14.97 11.21.97 11.28.97	BBCH 72	Whole fruit	0.32 0.26 0.22 0.19	0 1 3 5
34-99-180 Trial: 705322 Avignon, France FRA 2 SC, 240 g/l	Pepper, Elisa	0.067 0.077 0.086	700 800 900	0.0096 0.0096 0.0096	06.10.97 06.17.97 06.24.97	BBCH 69	Whole fruit	0.040 0.055 0.046 0.042	0 1 3 5
34-99-180 Trial: 705330 Barcelona, Spain, SPA 6 SC, 240 g/l	Pepper, Irina	0.096 0.096 0.106	1000 1000 1100	0.0096 0.0096 0.0096	07.15.97 07.22.97 07.29.97	BBCH 76	Whole fruit	0.24 0.27 0.19 0.14	0 1 3 6
34-99-180 Trial: 705349 Rotterdam, Netherlands NIE 1 SC, 240 g/l	Pepper, Cardio	0.134 0.144 0.154	1400 1500 1600	0.0096 0.0096 0.0096	04.28.97 05.05.97 05.12.97	BBCH 79	Whole fruit	0.15 0.12 0.13 0.12	0 1 3 5

[1/] Analysis method 00470, electrospray LC-MS/MS, average recovery 95%, LOQ 0.05 mg/kg.

Table 73. Residues data summary from supervised glasshouse trials on peppers in Italy and Spain (Walz-Tylla, 1999g).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [1/]	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-182 Trial: 0154/2 Ragusa, Italy IT 24 SC, 240 g/l	Pepper, Soldi	0.077 0.077 0.077	800 800 800	0.0096 0.0096 0.0096	03.16.99 03.23.99 03.30.99	Several stages of fruit development in same plant	Whole fruit	0.08 0.07 0.13	0 1 3
34-99-182 Trial: 0163/1 Ragusa, Italy IT 24 SC, 240 g/l	Pepper, Soldi	0.11 0.12 0.12	1100 1200 1200	0.0096 0.0096 0.0096	03.17.99 03.24.99 03.31.99	Several stages of fruit development in same plant	Whole fruit	0.15 0.18 0.16	0 1 3
34-99-182 Trial: 0165/8 Almaria, Spain SPA 5 SC, 240 g/l	Pepper, Antonto	0.11 0.11 0.11	1100 1100 1100	0.0096 0.0096 0.0096	02.09.99 02.09.99 02.16.99	Several stages of fruit development in same plant	Whole fruit	0.29 0.27 0.20	0 1 3
34-99-182 Trial: 0166/6 Barcelona, Spain, SPA – 6 SC, 240 g/l	Pepper, Italiano	0.11 0.11 0.12	1100 1100 1100	0.0096 0.0096 0.0096	07.05.99 07.13.99 07.20.99	Several stages of fruit development in same plant	Whole fruit	0.22 0.23 0.14	0 1 3

[1/] Analysis method 00551, HPLC-MS/MS, average recovery 88%, LOQ 0.05 mg/kg.

Table 74. Residues data summary from supervised glasshouse trials on peppers in Belgium and Germany (Walz-Tylla, 1999h and 1999i).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at final treatment	Commodity	Residue mg/kg [1/]	PHI days
		kg ai/ha	Water l/ha	kg ai/hl					
34-99-183 Trial: 0153/4 Mechelen, Belgium, BNL 1 SC, 240 g/l	Pepper, Meteor	0.15 0.16 0.17	1600 1700 1800	0.0096 0.0096 0.0096	04.20.99 04.27.99 05.04.99	Several stages of fruit development in same plant	Whole fruit	0.28 0.27 0.23 0.19	0 1 3 6
34-99-184 Trial: 810592 Rheinland, Germany DVG 9 SC, 240 g/l	Pepper, Rumba	0.058 0.058 0.058	600 600 600	0.0096 0.0096 0.0096	08.04.98 08.11.98 08.18.98	Several stages of fruit development in same plant	Whole fruit	0.08 0.05 0.07	0 1 3
34-99-184 Trial: 815047 Mechelen, Belgium, BNL 1 SC, 240 g/l	Pepper, Meteor	0.14 0.15 0.15	1500 1550 1550	0.0096 0.0096 0.0096	03.31.98 04.07.98 04.14.98	Several stages of fruit development in same plant	Whole fruit	0.22 0.21 0.19	0 1 3
34-99-184 Trial: 815055 Mechelen, Belgium, BNL 1 SC, 240 g/l	Pepper, Meteor	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	06.02.98 06.09.98 06.16.98	Several stages of fruit development in same plant	Whole fruit	0.21 0.19 0.19	0 1 3

[1/] Analysis method 00551, HPLC-MS/MS, average recovery 88%, LOQ 0.05 mg/kg.

Table 75. Residues data summary from supervised trials on tomatoes in the USA (Bergin, 1999c).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-47 Trial: 61298001 Germansville, PA WP, 800 g/kg	Tomato, Better Boy	0.28 0.28 0.28 0.28	0.078 0.078 0.078 0.078	360 360 360 360	07.29.98 08.05.98 08.12.98 08.19.98	Early maturity	Whole fruit	0.16	1
34-99-47 Trial: 61298002 Lucama, NC WP, 800 g/kg	Tomato, Beef Stake	0.29 0.28 0.28 0.28	0.15 0.15 0.16 0.16	190 190 180 180	07.02.98 07.16.98 07.13.98 08.13.98	Fruiting	Whole fruit	0.052	1
34-99-47 Trial: 61298003 Boynton Beach, FL WP, 800 g/kg	Tomato, Solar Set	0.28 0.28 0.28 0.28	0.078 0.070 0.078 0.078	360 400 360 380	10.11.98 10.23.98 11.03.98 11.17.98	Fruiting	Whole fruit	0.088	1
34-99-47 Trial: 61298004 Carlyle, IL WP, 800 g/kg	Tomato, Better Boy	0.28 0.28 0.28 0.28	0.12 0.12 0.10 0.12	240 240 270 240	07.07.98 07.17.98 07.30.98 08.13.98	Fruiting	Whole fruit	0.13	1
34-99-47 Trial: 61298005 Visalia, CA WP, 800 g/kg	Tomato, 3155 Processing	0.28 0.28 0.28 0.28	0.096 0.10 0.10 0.10	290 280 280 280	07.21.98 08.04.98 08.18.98 09.01.98	Mature	Whole fruit	0.20	1
34-99-47 Trial: 61298006 Hickman, CA WP, 800 g/kg	Tomato, UC 82B	0.28 0.28 0.29 0.28	0.12 0.12 0.12 0.12	240 230 240 230	09.08.98 09.15.98 09.23.98 09.29.98	Red fruit	Whole fruit	No data	
34-99-47 [4/] Trial: 61298007 King City, CA WP, 800 g/kg	Tomato, Mountain Fresh	0.28 0.28 0.28 0.28	0.10 0.10 0.10 0.10	280 280 280 280	07.28.98 08.11.98 08.25.98 09.08.98	Maturity	Whole fruit	0.12	1

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	kg ai/hl [1/]	Water l/ha					
34-99-47 [4/]	Tomato,	0.28	0.12	240	08.03.98	Maturity	Whole fruit	0.28	1
Trial: 61298008	Halley	0.28	0.12	230	08.10.98				
Woodland, CA	3155	0.28	0.12	230	08.17.98				
WP, 800 g/kg		0.29	0.13	230	08.25.98				
34-99-47 [3/]	Tomato,	0.27	0.053	510	10.22.98	Maturity	Whole fruit	0.075	0
Trial: 61298009	Solar Set	0.28	0.055	510	10.29.98			0.082	1
Hobe Sound,		0.28	0.056	500	11.06.98			0.10	1
CA		0.28	0.054	520	11.13.98			0.19	3
WP, 800 g/kg								0.088	7
								0.068	10
34-99-47 [3/]	Tomato,	0.27	0.096	280	05.28.98	Fruit ripening	Whole fruit	0.12	0
Trial: 61298010	Roma	0.27	0.096	280	06.11.98			0.14	1
Sommerton, AZ		0.28	0.10	280	06.25.98			0.12	3
WP, 800 g/kg		0.28	0.10	280	07.08.98			0.11	7
								0.050	10
34-99-47 [3/]	Tomato,	0.29	0.11	270	07.20.98	Maturity	Whole fruit	0.60	0
Trial: 61298011	Red cherry	0.29	0.10	280	08.03.98			1.8	1
Dinuba, CA		0.28	0.10	270	08.17.98			0.31	3
WP, 800 g/kg		0.28	0.10	270	09.01.98			0.58	7
								0.32	10
34-99-47 [3/]	Tomato,	0.28	0.10	280	06.29.98	Maturity	Whole fruit	0.45	0
Trial: 61298012	Red cherry	0.28	0.10	280	07.13.98			0.94	1
Porterville, CA		0.28	0.10	280	07.27.98			0.72	3
WP, 800 g/kg		0.28	0.10	280	08.10.98			1.4	7
								0.80	10
34-99-47 [4/]	Tomato,	0.28	0.12	230	07.28.98	Maturity	Whole fruit	0.12	1
Trial: 61298007	Mountain	0.28	0.12	230	08.11.98				
King City, CA	Fresh	0.28	0.12	230	08.25.98				
SC, 240 g/l		0.28	0.12	230	09.08.98				
34-99-47 [4/]	Tomato,	0.29	0.10	280	08.03.98	Maturity	Whole fruit	0.33	1
Trial: 61298008	Halley	0.27	0.096	280	08.10.98				
Woodland, CA	3155	0.27	0.096	280	08.17.98				
SC, 240 g/l		0.28	0.10	280	08.25.98				

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR 34-98-186 (preliminary version of TR 34-99-74), HPLC with UV detection, average recovery 95.4%, LOQ 0.02 mg/kg.
[3/] Decline study.
[4/] Bridging study.

Table 76. Residues data summary from supervised trials on tomatoes in Australia (Hamblin, 1997; Seidel, 1999a & 1999b; English, 1999a, 1999b & 1999c).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Com-modity	Residue mg/kg		PHI days
		kg ai/ha	Water l/ha	kg ai/hl						
PJH 208/97 [4/]	Tomato,	0.3	200	0.15	01.14.97	End of	Whole		+ D-C	
Gooloogong,	XPH				01.31.97	flowering,	fruit		Trate [3/]	
NSW, Australia	flowering,				02.10.97	fruit set		0.47	0.54	0
SC, 240 g/l					02.21.97			0.30	0.52	1
					02.28.98			0.17	0.73	3
					03.17.97			0.60	0.99	5
PJH 208/97 [4/]	Tomato,	0.4	200	0.20	01.14.97	End of	Whole	0.81	0.93	0
Gooloogong,	XPH				01.31.97	flowering,	fruit	0.97	1.0	1
NSW, Australia	12047				02.10.97	fruit set		0.16	0.39	3
SC, 240 g/l					02.21.97			0.85	0.91	5
					02.28.98					
					03.17.97					
JES 610/99 [5/]	Tomato,	0.4 [1/]	170	0.22	12.01.98	20% fruit ripe	Whole		+D-C	
Jerilderie, NSW	Heinz				12.07.98		fruit		Tron [3/]	
Australia	9504				12.15.98			0.48	0.56	0
SC, 240 g/l					12.21.98			0.50	0.25	1
					12.28.98			0.32	0.29	3
					01.03.99			0.23	0.26	5

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Com-modity	Residue mg/kg		PHI days
		kg ai/ha	Water l/ha	kg ai/hl						
JES 610/99 [5/] Jerilderie, NSW Australia SC, 240 g/l	Tomato, Heinz 9504	0.4	1000	0.04 [2/]	12.01.98 12.07.98 12.15.98 12.21.98 12.28.98	20% fruit ripe	Whole fruit	0.73 0.55 0.30 0.39	0.57 0.31 0.32 0.28	0 1 3 5
JES 611/99 [6/] Rochester, Victoria Australia SC, 240 g/l	Tomato, Heinz 1935	0.4 [1/]	170	0.23	01.27.99 02.03.99 02.10.99 02.17.99 02.23.99 03.01.99	30% fruit ripe	Whole fruit	 1.4 1.6 1.0 0.8	+D-C Tron [3/] 1.4 1.5 0.91 1.2	 0 1 3 5
JME 250/97 [7/] Bowen, Queensland Australia SC, 240 g/l	Tomato, Eagle	0.3	220 to 250	0.12 to 0.14	09.05.96 09.12.96 09.18.96 09.25.96 10.02.96 10.09.96	Some mature fruit	Whole fruit	 0.11 0.18 0.18 0.09 0.15	+D-C Tron [3/] 0.11 0.16 0.13 0.14 0.25	 0 1 3 5 7
JME 250/97 [7/] Bowen, Queensland Australia SC, 240 g/l	Tomato, Eagle	0.4	220 to 250	0.16 to 0.18	09.05.96 09.12.96 09.18.96 09.25.96 10.02.96 10.09.96	Some mature fruit	Whole fruit	0.15 0.17 0.18 0.09 0.11	0.13 0.15 0.11 0.21 0.15	0 1 3 5 7
JME 291/99 [8/] Bowen, Queensland Australia SC, 240 g/l	Tomato, Tempest	0.3	220	0.14	10.14.98 10.21.98 10.29.98 11.05.98 11.10.98 11.17.98	Fruit starting to colour	Whole fruit	0.12 0.29 0.24 0.25		0 1 3 6
JME 250/97 [7/] Bowen, Queensland Australia SC, 240 g/l	Tomato, Tempest	0.4	220	0.18	10.14.98 10.21.98 10.29.98 11.05.98 11.10.98 11.17.98	Fruit starting to colour	Whole fruit	0.12 0.22 0.26 0.13		0 1 3 6
JME 291/99 [8/] Bowen, Queensland Australia SC, 240 g/l	Tomato, Tempest		run-off	0.03	6/11.17.98	Fruit starting to colour	Whole fruit	0.21 0.28 0.25 0.40		0 1 3 6
JME 250/97 [9/] Bowen, Queensland Australia SC, 240 g/l	Tomato, Tempest		Run-off	0.04	6/11.17.98	Fruit starting to colour	Whole fruit	0.26 0.27 0.23 0.57		0 1 3 6
JME 292/99 [10/] Bowen, Queensland Australia SC, 240 g/l	Tomato Guardian	0.4	220	0.18	05.03.99 05.10.99 05.17.99 05.23.99 05.31.99 06.04.99	Ready for harvest	Whole fruit		0.14 0.05 0.08 0.11	0 1 3 7
JME 250/97 [10/] Bowen, Queensland Australia SC, 240 g/l	Tomato Guardian		Run-off	0.04	05.03.99 05.10.99 05.17.99 05.23.99 05.31.99 06.04.99	Ready for harvest	Whole fruit	0.13 0.07 <0.05 0.12		0 1 3 7

[1/] Applied by low-volume method.
[2/] Applied by high-volume spray.
[3/] Brand of miscible oil used with the formulation.
[4/] Method 34-95-55, HPLC with UV detection, average recovery 77%, LOQ 0.05 mg/kg (Shields, 1997b, report No. 97/1291).
[5/] Method 34-95-55, HPLC with UV detection, average recovery 83%, LOQ 0.05 mg/kg (Shields, 1999a, report No. 99/5210).
[6/] Method 34-95-55, HPLC with UV detection, average recovery 116%, LOQ 0.05 mg/kg (Shields, 1999b, report No. 99/1395).
[7/] Method 34-95-55, HPLC with UV detection, average recovery 106%, LOQ 0.05 mg/kg (Shields, 1997a, report No. 96/4959).
[8/] Method 34-95-55, HPLC with UV detection, average recovery 104%, LOQ 0.05 mg/kg (Shields, 1999a, report No. 99/5210).
[9/] Method 34-95-55, HPLC with UV detection, average recovery 104%, LOQ 0.05 mg/kg.
[10/] Method 34-95-55, HPLC with UV detection, average recovery 87%, LOQ 0.05 mg/kg.

Table 77. Residues data summary from supervised trials on tomatoes in glasshouses in Germany, Belgium, the Netherlands, Spain, Portugal, Italy and France (Walz-Tylla, 1999j and 1999k; Seym and Deissler, 1999f).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha [1/]	Water l/ha	kg ai/hl					
34-98-138 [2/] Trial: 810576 Rheinland, Germany SC, 240 g/l	Tomato, Ferrari	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	08.11.98 08.18.98 08.25.98	Several stages of fruit development	Whole fruit	0.18 0.12 0.06	0 1 3
34-98-138 [2/] Trial: 810584 Mechelen, Belgium SC, 240 g/l	Tomato, Tradino	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	06.02.98 06.09.98 06.16.98	Several stages of fruit development	Whole fruit	0.14 0.14 0.10	0 1 3
34-98-138 [2/] Trial: 815063 Mechelen, Belgium SC, 240 g/l	Tomato, Blitz	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	04.28.98 05.05.98 05.12.98	Several stages of fruit development	Whole fruit	0.14 0.12 0.09	0 1 3
34-99-122 [3/] Trial: 703621 Rheinland, Germany, DVG 8 SC, 240 g/l	Tomato, Suso	0.19 0.19 0.192	2000 2000 2000	0.0096 0.0096 0.0096	08.11.97 08.18.97 08.25.97	Several stages of fruit development	Whole fruit	0.29 0.12 0.14 0.10	0 1 3 7
34-99-122 [3/] Trial: 703648 Barcelona, Spain, SPA 6 SC, 240 g/l	Tomato, Alboran	0.16 0.16 0.16	1700 1700 1700	0.0096 0.0096 0.0096	07.01.97 07.08.97 07.15.97	Several stages of fruit development	Whole fruit	0.17 0.18 0.20 0.15	0 1 3 6
34-99-122 [3/] Trial: 705152 Santarem, Portugal POR 1 SC, 240 g/l	Tomato, Indalo	0.14 0.14 0.14	1500 1500 1500	0.0096 0.0096 0.0096	06.13.97 06.20.97 06.27.97	Several stages of fruit development	Whole fruit	0.17 0.12 0.10 0.09	0 1 3 5
34-99-122 [3/] Trial: 705373 Almeira, Spain SPA 5 SC, 240 g/l	Tomato, Brilliance	0.17 0.18 0.19	1750 1900 2000	0.0096 0.0096 0.0096	04.21.97 04.28.97 05.05.97	Several stages of fruit development	Whole fruit	0.21 0.17 0.20 0.14	0 1 3 4
34-99-122 [3/] Trial: 705381 Ragusa, Italy IT 20 SC, 240 g/l	Tomato, Rita	0.17 0.17 0.17	1800 1800 1800	0.0096 0.0096 0.0096	11.14.97 11.21.97 11.28.97	Several stages of fruit development	Whole fruit	0.47 0.38 0.46 0.37	0 1 3 5

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha [1]	Water l/ha	kg ai/hl					
34-99-122 [3] Trial: 705403 Avignon, France, FRA 2 SC, 240 g/l	Tomato, Cecile	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	06.16.97 06.23.97 06.30.97	Several stages of fruit development	Whole fruit	0.09 0.13 0.11 0.12	0 1 3 5
34-00-03 [4] Trial: 0143/7 Antwerpen Netherlands NIE 7 SC, 240 g/l	Tomato, Jamaica	0.17 0.18 0.19	1800 1900 2000	0.0096 0.0096 0.0096	03.24.99 03.31.99 04.07.99	Several stages of fruit development	Whole fruit	0.10 0.08 0.06	0 1 3
34-00-03 [4] Trial: 0148/8 Rudesheim Germany DSW 1 SC, 240 g/l	Tomato, Vanes	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	05.25.99 06.01.99 06.08.99	Several stages of fruit development	Whole fruit	0.12 0.12 0.11 0.08	0 1 3 5
34-00-03 [4] Trial: 0161/5 Mechelen Belgium, BEL 3 SC, 240 g/l	Tomato, Star- fighter	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	04.01.99 04.08.99 04.15.99	Several stages of fruit development	Whole fruit	0.27 0.21 0.29 0.20 0.17	0 1 1 3 5
34-00-03 [4] Trial: 0508/4 Rudesheim Germany DSW 1 SC, 240 g/l	Tomato, Eurore	0.19 0.19 0.19	2000 2000 2000	0.0096 0.0096 0.0096	05.25.99 06.01.99 06.04.99	Several stages of fruit development	Whole fruit	0.18 0.11 0.10 0.13	0 1 3 5

[1] Calculated from kg ai/hl and spray volume.
[2] Analysis method 00551, HPLC-MS/MS, average recovery 97%, LOQ 0.05 mg/kg.
[3] Analysis method 00551, HPLC-MS/MS, average recovery 101%, LOQ 0.05 mg/kg.
[4] Analysis method 00551, HPLC-MS/MS, average recovery 94%, LOQ 0.05 mg/kg.

Table 78. Residues data summary from supervised trials on egg plants in Malaysia (Boh, 1998a).

Report, trial, location, formulation	Crop variety	Application			Date or number of applications	Growth stage at last treatment	Commod-ity	Residue, mg/kg [2]	PHI days
		kg ai/ha [1]	Water l/ha	kg ai/hl					
Boh-1 Selangor, Malaysia SC, 240 g/l	Egg plant Pintung Long	0.15	500	0.03	07.13.98 07.20.98 07.27.98 08.03.98	At maturity	Whole fruit	0.50, 0.52, 0.43, 0.48 0.28, 0.40, 0.32, 0.33 0.29, 0.39, 0.31, 0.33 0.19, 0.14, 0.13, 0.15 0.11, 0.12, 0.17, 0.13 (0.13 avg)	0 3 7 10 14
Boh-1 Selangor, Malaysia SC, 240 g/l	Egg plant Pintung Long	0.30	500	0.06	07.13.98 07.20.98 07.27.98 08.03.98	At maturity	Whole fruit	0.82, 0.48, 0.72, 0.67 0.41, 0.61, 0.63, 0.55 0.29, 0.20, 0.36, 0.28 0.18, 0.21, 0.24, 0.21 0.10, 0.10, 0.10, 0.10 0.14, 0.12, 0.18, 0.15	0 3 7 10 14 21

[1] Calculated from kg ai/hl and spray volume.
[2] Analysis method 34-95-55, HPLC with UV detection, average recovery 104%, LOQ 0.05 mg/kg.

Twelve supervised trials were conducted during 1998 in the US to evaluate the residues of methoxyfenozide in sweet corn, specifically: kernels plus cob with husk removed; forage; and stover (fodder) (Filchner and Carpenter, 2000, report No. 34-00-15, Tables 79 and 97). Each treated plot received four applications at 0.25 lb ai/acre (0.28 kg ai/ha) per application of either the 80W (WP) or 2F (SC) formulation of methoxyfenozide, with a total of 1.12 kg ai/ha for the season, at intervals of 7-10 days between applications. All applications were made with either a tractor-mounted boom sprayer, an ATV-mounted sprayer, or a CO_2-powered backpack sprayer. The GAP in the US for registered use of methoxyfenozide on sweet corn (corn on the cob) is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray volume (l/ha)	No. or max/season	Interval, days	
SC 240 g/l WP 800 g/kg	Foliar	0.07 – 0.13 (SC) (0.06 – 0.12 lb ai/acre) 0.07 – 0.27 [1/] (WP) (0.06 – 0.24 lb ai/acre)	94 min	1.1 kg ai/ha/season	5 - 10	3

[1/] Up to silking stage only, 0.13 kg ai/ha thereafter.

Samples of ears (kernel and cob with husk removed, K+CWHR) of the raw agricultural commodity (RAC) were harvested 3 days after the final application. All harvest samples were frozen immediately and maintained frozen until analysis. Sampling to analysis intervals for all samples ranged from 9.5 to 16 months.

Table 79. Residues data summary from supervised trials on sweet corn in the USA (Filchner and Carpenter, 2000).

Report, trial, location, formulation	Crop variety	Application		Date or number of applications	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	Water l/ha					
34-00-15 [3/] Trial: 98-0178/ 61198022 Hamburg, PA WP, 800 g/kg	Sweet corn, Fortune	0.28 0.28 0.26 0.28	580 490 490 490	07.17.98 07.27.98 08.03.98 08.11.98	Tassel	Ears [1/]	<0.02	3
34-00-15 [3/] Trial: 98-0204/ 61198023 North Rose, NY WP, 800 g/kg	Sweet corn, Tuxedo	0.29 0.27 0.28 0.28	240 220 230 230	07.21.98 07.28.98 08.04.98 08.11.98	Tassel	Ears [1/]	<0.02	3
34-00-15 [3/] Trial: 98-0214/ 61198024 Montezuma, GA WP, 800 g/kg	Sweet corn, Silver Queen	0.28 0.28 0.28 0.28	190 190 190 190	07.20.98 08.03.98 08.17.98 08.24.98	Milk	Ears [1/]	<0.02	3
34-00-15 [3/] Trial: 98-0149/ 61198025 O'Brein, FL WP, 800 g/kg	Sweet corn, Abbott & Cobb 8100	0.28 0.28 0.28 0.28	190 190 190 190	06.03.98 06.20.98 06.17.98 06.29.98	Dry silk	Ears [1/]	<0.02	3
34-00-15 [3/] Trial: 98-0218/ 61198026 Theilman, MN WP, 800 g/kg	Sweet corn, Seneca Appaloosa SH2	0.28 0.27 0.28 0.28	190 190 190 190	08.18.98 08.15.98 08.23.98 08.30.98	Milk	Ears [1/]	<0.02	3
34-00-15 [3/] Trial: 98-0328/ 61198027 Madera, CA WP, 800 g/kg	Sweet corn, Sweetie 82	0.28 0.28 0.29 0.29	200 200 210 210	09.14.98 09.21.98 09.28.98 10.05.98	Mature	Ears [1/]	<0.02	3
34-00-15 [3/] Trial: 98-0193/ 61198028 Ephrata, WA WP, 800 g/kg	Sweet corn, Jubilee	0.28 0.28 0.28 0.28	140 140 140 140	07.23.98 07.30.98 08.07.98 08.14.98	Milk	Ears [1/]	<0.02	3
34-00-15 [3/] Trial: 98-0334/ 61198029 Corvallis, OR WP, 800 g/kg	Sweet corn, SS Jubilee	0.28 0.28 0.28 0.28	190 190 180 190	08.19.98 09.02.98 09.16.98 09.26.98	Mature	Ears [1/]	<0.02	3
34-00-15 [4/] Trial: 98-0224/ 61198030 Fitchburg, WI WP, 800 g/kg	Sweet corn, Empire	0.28 0.28 0.28 0.28	330 330 340 320	07.29.98 08.10.98 08.18.98 08.25.98	Milk	Ears [1/]	<0.02	3

Report, trial, location, formulation	Crop variety	Application		Date or number of applications	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	Water l/ha					
34-00-15 [4/] Trial: 98-0157/ 61198031 Nobelsville, IN WP, 800 g/kg	Sweet corn, Silver Queen	0.28 0.28 0.28 0.28	190 190 190 190	07.16.98 07.23.98 07.31.98 08.11.98	Milk	Ears [1/]	<0.02	3
34-00-15 [4/] Trial: 98-0224/ 61198030 Fitchburg, WI SC, 240 g/l	Sweet corn, Empire	0.26 0.26 0.26 0.26	330 330 340 320	07.29.98 08.10.98 08.18.98 08.25.98	Milk	Ears [1/]	<0.02	3
34-00-15 [4/] Trial: 98-0157/ 61198031 Nobelsville, IN SC, 240 g/l	Sweet corn	0.27 0.26 0.26 0.26	190 190 190 190	07.16.98 07.23.98 07.31.98 08.11.98	Milk	Ears [1/]	<0.02	3
34-00-15 [5/] Trial: 98-0145/ 61198032 Cunningham, KS WP, 800 g/kg	Sweet corn Super sweet	0.28 0.28 0.28 0.28	120 120 110 110	07.01.98 07.15.98 07.18.98 07.22.98	Late milk	Ears [1/]	<0.02 <0.02 <0.02 <0.02	0 2 2 7
34-00-15 [5/] Trial: 98-0164/ 61198033 New Holland, OH WP, 800 g/kg	Sweet corn Golden Nuggets	0.28 0.28 0.28 0.28	120 120 110 110	07.16.98 07.23.98 07.31.98 08.07.98	Early milk	Ears [1/]	<0.02 <0.02 <0.02 <0.02	0 3 7 10

[1/] Ears = kernels + cob with husk removed.
[2/] Analysis method 34-98-186 (preliminary version of 34-99-74), HPLC with UV detection, average recovery 79%, LOQ 0.02 mg/kg.
[3/] Raw agricultural commodity (RAC) trial.
[4/] Bridging trial.
[5/] Decline trial.

Leafy vegetables

Supervised trials on leaf lettuce, head lettuce, spinach and mustard greens were conducted in the USA in 1998 and 1999 (Tables 80 - 83). The GAP registered in the USA for the leafy vegetable group is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate kg ai/ha	Spray conc., kg ai/hl	No. of applications or max/season	Interval, days	
SC 240 g/l WP 800 g/kg	Foliar	0.07 – 0.28	0.04 – 0.30 (calculated)	1.12 kg ai/ha	10 - 14	1

Sixteen supervised trials were conducted on lettuce plants (eight on leaf lettuce and eight on head lettuce) in 1998-1999 (Carpenter, 1999e, report No. 34-99-75). Plots were treated with either the 80W (WP) or 2F (SC) formulation of methoxyfenozide. Each treated plot received four applications of 0.25 lb ai/acre (0.28 kg ai/ha) per application, at intervals of 7 days between applications. The total treatment was nominally 1 lb ai/acre (1.12 kg ai/ha). The equipment used for the foliar application was either a tractor-mounted boom sprayer or CO_2-powered backpack sprayer with a spray boom. The minimum spray volume was 14.6 gallons water/acre (138 l/ha) for leaf lettuce and 14.9 gallons/acre (139 l/ha) for head lettuce.

On each sampling occasion, duplicate samples (3-9 lb) of whole leaf lettuce plants were collected from each untreated control and treated plot. Each sample consisted of 12 whole plants. In the head lettuce trials, samples with wrapper leaves and two without wrapper leaves were collected from both the treated and control plots. Each sample weighed 9.25-16.5 lbs and consisted to twelve whole lettuce heads. Samples were frozen immediately and transported to the laboratory, where they were maintained frozen at a temperature range of about -15.9 to +20.1ºF (-27 to -7ºC), for up to approximately 7 months for leaf lettuce and 4 months for head lettuce.

Eight supervised trials were conducted on spinach (Carpenter, 1999e, report No. 34-99-75). Plots were treated with either the 80W (WP) or 2F (SC) formulation of methoxyfenozide. Each treated plot received four applications of 0.25 lb ai/acre (0.28 kg ai/ha) per application, at intervals of 7 to 10 days between applications. The total nominal application was 1 lb ai/acre/season or 1.12 kg ai/ha/season. The equipment used for the foliar application was either a tractor-mounted boom sprayer or CO_2-powered backpack sprayer with a spray boom. The minimum spray volume was 14.1 gallons water/acre (132 l/ha) in all trials The GAP information for registered uses of methoxyfenozide on spinach is as indicated above. Each sample weighed a minimum of 2.5 lbs and represented a minimum of 12 plants. All samples were frozen upon collection and maintained frozen for 2 to 8 months, prior to analysis.

Seven supervised trials were carried out on mustard greens in major vegetable growing areas of the USA from 1998-1999 (Carpenter, 1999d, report No. 34-99-76). Plots were treated with either the 80W or 2F formulation of methoxyfenozide. Each treated plot received four applications of 0.25 lb ai/acre (0.28 kg ai/ha) per application, at intervals of 7-10 days. The total treatment rate was nominally 1 lb ai/acre or 1.12 kg ai/ha. The equipment used for the foliar application was either a tractor-mounted boom sprayer or a CO_2-powered backpack sprayer. A minimum of 10.1 gallons of water/acre (94 l/ha) was used for each application. Mustard greens belong to the EPA crop group of brassica (cole) crops. The registered use of methoxyfenozide on mustard greens follows the same GAP as indicated for other leafy vegetables above. Each sample consisted of a minimum of 12 individual plants (approximately 2.5 lbs). All samples were frozen upon collection and maintained frozen up to approximately 7 months until analysis.

Table 80. Residues data summary from supervised trials on leaf lettuce in the USA (Carpenter, 1999e).

Report, trial, location, formulation	Crop variety	Application		Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [1/]	PHI days
		kg ai/ha	Water l/ha					
34-99-75	Leaf lettuce,	0.29	230	06.15.98	At maturity	Whole	3.4	1
Trial: 61198034	Black-	0.28	220	06.22.98		commodity		
North Rose, NY	seeded	0.30	240	06.29.98				
WP, 800 g/kg	Simpson	0.30	240	07.06.98				
34-99-75	Leaf lettuce,	0.29	240	11.09.98	At maturity	Whole	13	1
Trial: 61198035	504 Green	0.29	340	11.16.98		commodity		
Hobe Sound, FL		0.29	330	11.23.98				
WP, 800 g/kg		0.30	330	11.30.98				
34-99-75	Leaf lettuce,	0.29	190	10.08.98	At maturity	Whole	12	1
Trial: 61198036	Red Giant	0.29	190	10.15.98		commodity		
King City, Ca		0.29	190	10.22.98				
WP, 800 g/kg		0.30	190	10.29.98				
34-99-75 [2/]	Leaf lettuce,	0.30	190	11.10.98	At maturity	Whole	17	1
Trial: 61198037	Hacienda	0.29	190	11.17.98		commodity		
Porterville, CA		0.30	200	11.25.98				
WP, 800 g/kg		0.30	190	12.02.98				
34-99-75 [2/]	Leaf lettuce,	0.30	240	11.04.98	At maturity	Whole	10	1
Trial: 61198038	Desert	0.30	240	11.14.98		commodity		
Somerton, AZ	Green	0.31	240	11.21.98				
WP, 800 g/kg		0.30	240	11.30.98				
34-99-75 [3/]	Leaf lettuce,	0.30	140	12.08.98	At maturity	Whole	18	0
Trial: 61198039	Green	0.29	140	12.18.98		commodity	16	3
Maricopa, AZ	Vision	0.32	150	01.01.99			11	7
WP, 800 g/kg		0.30	140	01.14.99			6.3	10
34-99-75 [2/]	Leaf lettuce,	0.27	190	11.10.98	At maturity	Whole	18	1
Trial: 61198037	Hacienda	0.27	190	11.17.98		commodity		
Porterville, CA		0.26	190	11.25.98				
SC, 240 g/l		0.26	190	12.02.98				

Report, trial, location, formulation	Crop variety	Application		Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [1/]	PHI days
		kg ai/ha	Water l/ha					
34-99-75 [2/]	Leaf lettuce,	0.27	240	11.04.98	At maturity	Whole	8.2	1
Trial: 61198038	Desert	0.26	230	11.14.98		commodity		
Somerton, AZ	Green	0.28	250	11.21.98				
SC, 240 g/l		0.27	240	11.30.98				

[1] Analysis method 34-98-186 (preliminary version of 34-99-74), HPLC with UV detection, average recovery 85.78%, LOQ 0.02 mg/kg.
[2] Bridging trial.
[3] Decline trial.

Table 81. Residues data summary from supervised trials on head lettuce in the USA (Carpenter, 1999e).

Report, trial, location, formulation	Crop variety	Application		Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha					
34-99-75 [1/]	Head lettuce,	0.29	540	10.10.98	At maturity	Whole lettuce heads,	7.9	1
Trial: 61198040	Target	0.29	530	10.20.98		with wrapper leaves		
Oxnard, CA		0.29	530	10.27.98		Whole lettuce heads,	0.13	1
WP, 800 g/kg		0.29	530	11.03.98		no wrapper leaves	0.091	1
34-99-75 [1/]	Head lettuce,	0.28	240	11.09.98		Whole lettuce heads,	4.8	1
Trial: 61198041	Southbay	0.29	340	11.16.98		with wrapper leaves		
Hobe Sound, FL	M90MT	0.29	340	11.23.98		Whole lettuce heads,	0.039	1
WP, 800 g/kg		0.29	320	11.30.98		no wrapper leaves	0.051	1
34-99-75 [1/]	Head lettuce,	0.29	190	10.08.98		Whole lettuce heads,	6.2	1
Trial: 61198042	Jupiter	0.29	190	10.15.98		with wrapper leaves		
Greenfield, CA		0.29	190	10.22.98		Whole lettuce heads,	0.059	1
WP, 800 g/kg		0.30	190	10.29.98		no wrapper leaves	0.14	1
34-99-75 [1/ 3/]	Head lettuce,	0.29	190	11.16.98	At maturity	Whole lettuce heads,	6.5	1
Trial: 61198043	Sharp	0307	190	11.23.98		with wrapper leaves		
Porterville, CA	Shooter	0.30	190	12.07.98				
WP, 800 g/kg		0.29	190	12.14.98				
34-99-75 [1/ 3/]	Head lettuce,	0.30	230	11.04.98	At maturity	Whole lettuce heads,	1.6	1
Trial: 6119804	HMX7197,	0.22	230	11.18.98		with wrapper leaves		
Somerton, AZ	Vanguard	0.30	240	12.01.98				
WP, 800 g/kg	type	0.30	240	12.11.98				
34-99-75 [1/ 4/]	Head lettuce,	0.29	140	01.05.99	At maturity	Whole lettuce heads,	6.7	0
Trial: 61198039	Cibola	0.30	140	01.18.99		with wrapper leaves	6.0	1
Maricopa, AZ		0.30	140	01.31.99			5.1	3
WP, 800 g/kg		0.29	140	02.09.99			5.5	7
							3.2	10
34-99-75 [2/ 3/]	Head lettuce,	0.26	190	11.16.98	At maturity	Whole lettuce heads,	9.6	1
Trial: 61198043	Sharp	0.26	190	11.23.98		with wrapper leaves		
Porterville, CA	Shooter	0.27	200	12.07.98				
SC, 240 g/l		0.26	190	12.14.98				
34-99-75 [2/ 3/]	Head lettuce,	0.26	230	11.04.98	At maturity	Whole lettuce heads,	5.4	1
Trial: 6119804	HMX7197,	0.27	240	11.18.98		with wrapper leaves		
Somerton, AZ	Vanguard	0.27	240	12.01.98				
SC, 240 g/l	type	0.28	240	12.11.98				

[1/] Analysis method 34-98-186 (preliminary version of 34-99-74), HPLC with UV detection, average recovery 86%, LOQ 0.02 mg/kg.
[2/] Analysis method 34-98-186 (preliminary version of 34-99-74), HPLC with UV detection, average recovery 86.34%, LOQ 0.02 mg/kg.
[3/] Bridging trial.
[4/] Decline trial.

Table 82. Residues data summary from supervised trials on spinach in the USA (Carpenter, 1999e).

Report, trial, location, formulation	Crop variety	application		Date or Number of Treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha					
34-99-75 [1/] Trial: 61198052 Germansville, PA WP, 800 g/kg	Spinach, Tyee	0.29 0.30 0.30 0.30	380 400 380 380	10.06.98 10.15.98 10.26.98 11.05.98	At maturity	Whole spinach plants	23	1
34-99-75 [1/] Trial: 61198053 Winterville, GA WP, 800 g/kg	spinach, Bloomsdale	0.30 0.30 0.30 0.29	230 240 240 250	10.14.98 10.22.98 11.04.98 11.18.98	At maturity	Whole spinach plants	12	1
34-99-75 [1/] Trial: 61198054 Oxnard, CA WP, 800 g/kg	spinach, Spring Field	0.30 0.29 0.30 0.29	210 540 540 530	09.16.98 09.15.98 10.02.98 10.09.98	At maturity	Whole spinach plants	14	1
34-99-75 [1/ 3/] Trial: 61198055 East Bernard, TX WP, 800 g/kg	Spinach, Bloomsdale	0.30 0.30 0.30 0.29 1.2	140 130 140 140 540	11.30.98 12.07.98 12.17.98 12.28.98	At maturity	Whole spinach plants	16	1
34-99-75 [1/ 3/] Trial: 61198056 Center, CO WP, 800 g/kg	Spinach, Unipak 151	0.30 0.30 0.29 0.29	190 190 180 180	06.27.98 07.06.98 07.13.98 07.21.98	At maturity	Whole spinach plants	9.8	1
34-99-75 [1/ 4/] Trial: 61198057 Porterville, CA WP, 800 g/kg	Spinach, Hybrid 424	0.29 0.30 0.30 0.29	230 230 230 230	12.15.98 12.22.98 12.29.98 01.05.99	At maturity	Whole spinach plants	38 43 41 39 31	0 1 3 7 10
34-99-75 [2/ 3/] Trial: 61198055 East Bernard, TX SC, 240 g/l	Spinach, Bloomsdale	0.28 0.27 0.27 0.26	140 140 140 130	11.30.98 12.07.98 12.17.98 12.28.98	At maturity	Whole spinach plants	18	1
34-99-75 [2/ 3/] Trial: 61198056 Center, CO SC, 240 g/l	Spinach, Unipak 151	0.27 0.25 0.25 0.26 1.0	200 190 190 190 760	06.27.98 07.06.98 07.13.98 07.21.98	At maturity	Whole spinach plants	10	1

[1/] Analysis method 34-98-186 (preliminary version of 34-99-74), HPLC with UV detection, average recovery 90%, LOQ 0.02 mg/kg.
[2/] Analysis method 34-98-186 (preliminary version of 34-99-74), HPLC with UV detection, average recovery 86%, LOQ 0.02 mg/kg.
[3/] Bridging trial.
[4/] Decline trial.

Table 83. Residues data summary from supervised trials on mustard greens in the USA (Carpenter, 1999d).

Report, trial, location, formulation	Crop variety	Application		Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [1/]	PHI days
		kg ai/ha	Water l/ha					
34-99-76 [2/] Trial: 98-0267 61198071 Metcalf, MS WP, 800 g/kg	Mustard greens, Broad Leaf	0.28 0.28 0.28 0.29	94 94 95 95	11.16.98 11.23.98 11.30.98 12.07.98	14 to 18 inches high; 6 to 8 leaves	Leaves	10	1
34-99-76 [2/] Trial 98-0143 61199072 Arkansaw, WI WP, 800 g/kg	Mustard greens, India Mustard; Florida Broadleaf	0.28 0.28 0.29 0.28	180 190 190 190	06.30.98 07.07.98 07.15.98 07.22.98	12 to 14 inches high; vegetative stage	Leaves	12	1

Report, trial, location, formulation	Crop variety	Application kg ai/ha	Application Water l/ha	Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [1/]	PHI days
34-99-76 [4/]	Mustard	0.29	230	11.18.98	14 to 18 inches	Leaves	16	0
Trial 98-0270	greens,	0.30	230	11.25.98	high; mature crop		17	1
61198075	Florida	0.30	230	12.02.99			14	3
Porterville, CA	Broadleaf	0.23	240	12.09.99			10	7
WP, 800 g/kg							7.5	10
34-99-76 [3/]	Mustard	0.28	230	10.14.98	4 to 16 inches high,	Leaves	16	1
Trial 98-0268	greens,	0.28	240	10.22.98	5 to 14 true leaves			
61198073	Florida	0.28	250	11.04.98				
Winterville, GA	Broadleaf	0.28	250	11.11.98				
WP, 800 g/kg								
34-99-76 [3/]	Mustard	0.28	130	11.30.98	10 to 13 inches	Leaves	18	1
Trial 98-0269	greens,	0.29	140	12.07.99	high; 8 to 10 leaves			
61198074	Florida	0.28	130	12.17.98				
East Bernard, TX	Broadleaf	0.29	140	12.28.98				
WP, 800 g/kg								
34-99-76 [3/]	Mustard	0.27	230	10.14.98	4 to 16 inches high;	Leaves	17	1
Trial 98-0268	greens,	0.26	240	10.22.98	5 to 14 true leaves			
61198073	Florida	0.26	240	11.04.98				
Winterville, GA	Broadleaf	0.27	250	11.11.98				
SC, 240 g/l								
34-99-76 [3/]	Mustard	0.26	130	11.30.98	10 to 13 inches	Leaves	14	1
Trial 98-0269	greens,	0.26	140	12.07.99	high; 8 to 10 leaves			
61198074	Florida	0.27	140	12.17.98				
East Bernard, TX	Broadleaf	0.27	140	12.28.98				
SC, 240 g/l								

[1/] Analysis method 34-98-186 (preliminary version of 34-99-74),, HPLC with UV detection, average recovery 87%, LOQ 0.02 mg/kg.
[2/] Raw agricultural commodity (RAC) trial.
[3/] Bridging trial.
[4/] Decline trial.

Legume vegetables

Two supervised trials on soya beans were conducted in Brazil in 1997 (Steckelberg, 1998a, Report No. AgReg10377). Treated plots received two applications of an SC formulation, containing 240 g/l methoxyfenozide, at the rate of either 0.022 kg ai/ha or 0.043 kg ai/ha per application, at intervals of 10 days. The GAP in Brazil for registered uses of methoxyfenozide on soybeans is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	Interval, days	
SC 240 g/l	Foliar	0.014 – 0.022	0.004 – 0.011	As required	10	7

About 1 kg samples were collected, frozen and maintained frozen for about 5 months until analysis.

Table 84. Residues data summary from supervised trials on soya beans in Brazil (Steckelberg, 1998a).

Report, trial, location, formulation	Crop variety	Application kg ai/ha	Application Water l/ha	Application kg ai/hl [1/]	Date or number of treatments	Growth stage at last treatment	Comm-odity	Residue g/kg [2/]	PHI days
AgReg10377	Soya	0.022	150	0.015	(2) 05.02.97; 05.12.97	Maturation	Seed	0.04	0
Trial:	bean,				(2) 04.25.97; 05.05.97	of seeds		0.03	7
RI 3379709	IAC8				(2) 04.18.97; 04.28.97			0.02	14
Sao Paolo,					(2) 04.11.97; 04.21.97			<0.02	21
Brazil					(2) 04.04.97; 04.14.97			<0.02	28
SC, 240 g/l									
AgReg10377	Soya	0.043	150	0.029	(2) 04.25.97; 05.05.97	Maturation	Seed	0.03	7
Trial:	bean,				(2) 04.18.97; 04.28.97	of seeds		0.04	14
RI 3379709	IAC8								
Sao Paolo,									
Brazil									
SC, 240 g/l									

[1/] Calculated from kg ai/ha and spray volume.

[2/] Analysis method 34-95-55, HPLC with UV detection, average recovery 82%, LOQ 0.02 mg/kg.

Two supervised trials were conducted on long beans in Malaysia (Boh, 1998b, Report No. Boh-2). A suspension concentrate formulation containing 240 g/l methoxyfenozide was applied four times, either at 0.15 kg ai/ha or 0.3 kg ai/ha (twice the recommended rate) per application. The interval between applications was 7 days. The GAP for registered uses of methoxyfenozide on long beans in Malaysia is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	Interval days	
SC 240 g/l	Foliar	0.1	0.023	As required	7	14

Table 85. Residues data summary from supervised trials on long beans in Malaysia (Boh, 1998b).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commo-dity	Residue, mg/kg [2/]	PHI, days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
Boh-2 MARDI, Serdang, Malaysia	Long beans Kajang Panjang Renek	0.15	500	0.03	09.01.98 09.08.98 09.15.98 09.22.98	Mature pods	Pods	1.30, 1.26, 2.16 0.35, 0.29, 0.05, 0.05, 0.26, 0.07 <0.05, <0.05, <0.05 <0.05, <0.05, <0.05 <0.05, <0.05, <0.05	0 3 7 10 14 21
Boh-2 MARDI, Serdang, Malaysia	Long beans Kajang Panjang Renek	0.3	500	0.06	09.01.98 09.08.98 09.15.98 09.22.98	Mature pods	Pods	3.13, 3.48, 1.26 0.57, 0.69, 0.65 0.18, 0.20, 0.18 0.17, 0.14, 0.22 0.17, 0.20, 0.05 <0.05, <0.05, <0.05	0 3 7 10 14 21

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method 34-95-55, HPLC with UV detection, average recovery 88%, LOQ 0.05 mg/kg.

Stalk and stem vegetables

Eight supervised trials were conducted on celery (Carpenter, 1999e, Report No. 34-99-75). All treated plots received four applications of the 80W formulation of methoxyfenozide, containing 800 g/kg ai, at the rate of 0.25 lb ai/acre (0.28 kg ai/ha) per application, at intervals of 7 to 10 days. Each of the bridging trials also received four applications of the SC formulation, containing 240 g/l methoxyfenozide, at the same nominal rate as the 80W formulation. The total nominal application was 1 lb ai/acre/season or 1.12 kg ai/ha /season. The equipment used to deliver the foliar application was either a tractor-mounted boom sprayer or CO_2-powered backpack sprayer with a spray boom. The minimum spray volume was 17.6 gallons water/acre (165 l/ha) in all trials. The GAP for registered uses of methoxyfenozide on celery is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. or max/season	Interval days	
SC 240 g/l WP 800 g/kg	Foliar	0.07 – 0.28	0.04 -0.30	1.1 kg ai/ha	10 - 14	1

Each sample weighed a minimum of 2.5 lbs and was collected from a minimum of 12 plants. All samples were frozen upon collection and maintained frozen for 1.5-7 months, prior to analysis.

Table 86. Residues data summary from supervised trials on celery in the USA (Carpenter, 1999e).

Report, trial, location, formulation	Crop variety	Application		Date or number of treatments	Growth stage at last treatment	Commodity	Residue [1/] mg/kg	PHI days
		kg ai/ha	Water l/ha					
34-99-75 Trial: 61198046 Hobe Sound, FL WP, 800 g/kg	Celery 683	0.29 0.29 0.29 0.30	330 340 460 470	11.23.98 12.02.98 12.09.98 12.16.98	At maturity	Whole celery plant	19 [2/] 20 [2/]	1

Report, trial, location, formulation	Crop variety	Application kg ai/ha	Application Water l/ha	Date or number of treatments	Growth stage at last treatment	Commodity	Residue [1/] mg/kg	PHI days
34-99-75 Trial: 61198047 Kent City, MI WP, 800 g/kg	Celery Florida 683K	0.29 0.29 0.29 0.29	180 180 180 180	06.29.98 07.07.98 07.15.98 07.23.98	At maturity	Whole celery plant	5.5	1
34-99-75 Trial: 61198048 Watsonville, CA WP, 800 g/kg	Celery Conquistador	0.28 0.29 0.29 0.30	360 370 360 380	10.28.98 11.10.98 11.24.98 12.08.98	At maturity	Whole celery plant	7.8	1
34-99-75 [3/] Trial: 61198049 King City, CA WP, 800 g/kg	Celery Conquistador	0.23 0.30 0.30 0.30	280 280 280 280	10.23.98 10.29.98 11.06.98 11.17.98	At maturity	Whole celery plant	3.4	1
34-99-75 [3/] Trial: 61198050 Somerton, AZ WP, 800 g/kg	Celery Conquistador	0.36 0.29 0.30 0.29	170 160 170 170	11.20.98 11.30.98 12.11.98 12.21.98	At maturity	Whole celery plant	0.48	1
34-99-75 [4/] Trial: 61198051 Oxnard, CA WP, 800 g/kg	Celery Santa Ana	0.30 0.29 0.29 0.29	550 530 530 530	10.12.98 10.16.98 10.27.98 11.09.98	At maturity	Whole celery plant	3.0 1.9 0.55 0.75	0 3 7 10
34-99-75 [3/] Trial: 61198049 King City, CA SC, 240 g/l	Celery Conquistador	0.26 0.26 0.26 0.26	280 280 280 280	10.23.98 10.29.98 11.06.98 11.17.98	At maturity	Whole celery plant	7.2	1
34-99-75 [4/] Trial: 61198050 Somerton, AZ SC, 240 g/l	Celery Conquistador	0.32 0.27 0.27 0.26	170 170 170 170	11.20.98 11.30.98 12.11.98 12.21.98	At maturity	Whole celery plant	0.72	1

[1/] Analysis method 34-98-186 (preliminary version of 34-99-74), HPLC with UV detection, average recovery 99%, LOQ 0.02 mg/kg.
[2/] Trial excluded because of black heart disease. Crop not marketable by day 7 after treatment.
[3/] Bridging trial.
[4/] Decline trial.

Cereal grains

Two supervised trials on rice were conducted in Japan in 1997 (Yajima, 1998, Report No. 8597AgReg). Rice plants were treated with three applications of a dust formulation (DP), containing 5 g/kg methoxyfenozide, at the rate of 0.2 kg ai/ha per application. Treatments were made every 7 days. The GAP in Japan for use of methoxyfenozide on rice is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	Interval, days	
DP 5 g/kg	Broadcast	0.2	Not applicable	3	7	14

Table 87. Residues data summary from supervised trials on rice in Japan (Yajima, 1998).

Report, trial, location, formulation	Crop variety	Application kg ai/ha	Application Water l/ha	Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [1/]	PHI days
8597/AgReg Fukui Prefecture, Japan Dust, 5g/kg	Rice, Hanaechize	0.2		3/ 08.12.97 3/ 08.05.97 3/ 07.30.97	Maturity	Hulled grain	≤0.02 <0.02 <0.02	14 21 28
8597/AgReg Hyogo Prefecture Japan Dust, 5 g/kg	Rice, Kinuhikari	0.2		3/ 09.13.97 3/ 09.07.97 3/ 09.30.97	Maturity	Hulled grain	≤0.02 <0.02 <0.02	14 21 28

[1/] Analysis method, HPLC with UV detection, recovery 88-93%, LOQ 0.02 mg/kg.

Supervised trials on field maize (corn) were conducted in the USA, Mexico and Brazil, from 1997 to 1998 (Tables 90-92).

Twenty-five supervised field trials of methoxyfenozide were conducted in 1998 in major field corn growing areas in the USA (Carpenter, 2000, Report No. 34-00-14). Plots were treated with either 80W (wettable powder containing 800 g/kg methoxyfenozide) or 2F (suspension concentrate containing 240 g/l methoxyfenozide) formulations. Each treated plot received four applications of 0.25 lb ai/acre (0.28 kg ai/ha) per application, with a total nominal application rate of 1.0 lb ai/acre (1.12 kg ai/ha). Applications were made at 7- to 14-day intervals. Either a tractor-mounted boom sprayer or a CO_2-powered backpack sprayer with spray boom was used to apply the product. The USA GAP for the use of methoxyfenozide on maize (field corn) is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray volume, l/ha	No. or max/season	Interval, days	
SC 240 g/l WP 800 g/kg	Foliar	0.07 – 0.13 (0.06 – 0.12 lb ai/acre)	47 minimum	1.12 kg ai/ha/season	Not specified	21 (grain, forage, fodder)

Treated and control maize grain, forage and fodder samples were harvested from all trials on Day 21 ± 2 days. For grain samples, ears were removed from 12 plants and the grain removed from the ear. The remaining stalk was used for the fodder sample. All samples were frozen immediately after collection and maintained frozen while awaiting analysis. The intervals between sampling and analysis were 3-17 months (Tables 88 and 96).

Two supervised trials on maize were conducted from 1997 to 1998 in Mexico (Hawkins, 1999, Report No. 34-99-27). Each trial received two applications of the 2F formulation, containing 240 g/l methoxyfenozide, at the rate of either 0.04 kg ai/ha or 0.08 kg ai/ha per application. The GAP in Mexico for registered uses of methoxyfenozide on maize is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	Interval, days	
SC 240 g/l	Foliar	0.03 – 0.04	0.01 – 0.013	1-4	Not specified	30

Grain samples were harvested at normal maturity, 77 or 156 days after the last application. All harvest samples were immediately frozen until shipment to the laboratory. The samples of grain were milled. After homogenization, the samples were kept frozen until analysis. The intervals between sampling and analysis were 8-10 months.

Two supervised trials on maize were conducted in Brazil in 1997 (Steckelberg, 1998b and 1998c, Reports No. AgReg10375 and AgReg10376). Each of the trials consisted of one application of an SC formulation, containing 240 g/l methoxyfenozide, at the rate of either 0.043 kg ai/ha or 0.086 kg ai/ha. Samples of grain were collected on days 0, 7, 14, 103 and 148 after treatment at the recommended dose of 0.043 kg ai/ha. Plots receiving twice the recommended dose (0.086 kg ai/ha) of methoxyfenozide were sampled on days 7 and 14 after the last application. For trial RI 3379710, samples were taken 7 days after the last application. The GAP in Brazil for use of methoxyfenozide on maize is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	Interval, days	
SC 240 g/l	Foliar	0.036 – 0.043	0.01 – 0.02	1	Not specified	7

Table 88. Residues data summary from supervised trials on maize (corn) grain in the USA (Carpenter, 2000).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-00-14 [3/] Trial: 98-0234 61198001 Hamburg, PA WP, 800 g/kg	Field maize, Dekalb DK 493	0.28 0.28 0.28 0.28	270 270 270 270	0.10 0.10 0.10 0.10	09.10.98 09.18.98 09.25.98 10.02.98	Mature	Grain Grain	<0.02 0.033	21 21

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1]					
34-00-14 [3] Trial: 98-0212/ 61198002 Montezuma, GA WP, 800 g/kg	Field maize, Pioneer 3167	0.28 0.28 0.30 0.28	190 190 190 190	0.15 0.15 0.16 0.15	08.17.98 08.24.98 09.02.98 09.09.98	Late dough	Grain	<0.02	21
34-00-14 [3] Trial: 98-0199/ 61198003 Webster City, IA WP, 800 g/kg	Field maize, Cropland Genetics N4640BT	0.28 0.28 0.28 0.28	190 190 190 190	0.15 0.15 0.15 0.15	08.25.98 09.01.98 09.08.98 09.17.98	R6 = Maturity	Grain	<0.02	21
34-00-14 [3] Trial: 98-0223/ 61198004 Theilman, MN WP, 800 g/kg	Field maize, Pioneer 3751	0.27 0.27 0.27 0.27	190 190 190 190	0.15 0.15 0.15 0.15	08.23.98 08.30.98 09.08.98 09.17.98	Dent	Grain	<0.02	21
34-00-14 [3] Trial: 98-0233/ 61198005 Campbell, MN WP, 800 g/kg	Field maize, DB5086	0.28 0.28 0.28 0.28	94 93 94 94	0.30 0.30 0.30 0.30	08.19.98 08.26.98 09.11.98 09.16.98	Early dent	Grain	<0.02	21
34-00-14 [3] Trial: 98-0170/ 61198006 Osceola, NE WP, 800 g/kg	Field maize, N3030 Bt	0.28 0.28 0.28 0.28	190 190 190 190	0.15 0.15 0.15 0.15	08.06.98 08.13.98 08.25.98 09.01.98	Late dough	Grain	<0.02	21
34-00-14 [3] Trial: 98-0180/ 61198007 Sheridan, IN WP, 800 g/kg	Field maize, Pioneer 34G81	0.25 0.28 0.28 0.28	170 190 190 190	0.15 0.15 0.15 0.15	08.06.98 08.20.98 09.03.98 09.10.98	Dough to dent	Grain	<0.02	21
34-00-14 [3] Trial: 98-0333/ 61198008 Northwood, ND WP, 800 g/kg	Field maize, Pioneer 3979	0.28 0.28 0.28 0.28	190 190 190 190	0.15 0.15 0.15 0.15	08.21.98 08.28.98 09.04.98 09.14.98	Dent	Grain	<0.02	21
34-00-14 [3] Trial: 98-0187/ 61198009 Lime Springs, IA WP, 800 g/kg	Field maize, Pioneer 3730	0.28 0.27 0.28 0.29	180 180 180 180	0.16 0.15 0.16 0.16	08.05.98 08.12.98 08.19.98 09.02.98	Black layer	Grain	<0.02	21
34-00-14 [3] Trial: 98-0327/ 61198010 Fitchburg, WI WP, 800 g/kg	Field maize, AP9121	0.29 0.29 0.52 0.49	330 330 340 320	0.087 0.087 0.15 0.15	08.25.98 09.01.98 09.15.98 09.28.98	Dent	Grain	<0.02	21
34-00-14 [3] Trial: 98-0182/ 61198011 New Holland, OH WP, 800 g/kg	Field maize, SC 1068	0.27 0.28 0.26 0.26	120 140 130 130	0.22 0.20 0.20 0.20	08.13.98 08.27.98 09.10.98 09.24.98	Dent	Grain	<0.02	21
34-00-14 [3] Trial: 98-0195/ 61198012 Williamston, MI WP, 800 g/kg	Field maize, DK 471	0.28 0.28 0.28 0.28	190 190 180 190	0.15 0.15 0.15 0.15	08.19.98 08.28.98 09.04.98 09.14.98	Dent	Grain	<0.02	21
34-00-14 [3] Trial: 98-0185 61198013 Dow, IL WP, 800 g/kg	Field maize, 8541 IT	0.28 0.28 0.28 0.28	190 190 190 190	0.15 0.15 0.15 0.15	08.05.98 08.17.98 08.27.98 09.08.98	Dough to dent	Grain	<0.02	20

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1]					
34-00-14 [3] Trial: 98-0200 61198014 Eakly, OK WP, 800 g/kg	Field maize, N7639 BT	0.27 0.24 0.26 0.30	120 130 120 130	0.22 0.21 0.21 0.23	07.24.98 08.04.98 08.17.98 08.28.98	Dough to dent	Grain	<0.02	20
34-00-14 [4] Trial: 98-0186 61198015 Dow, IL WP, 800 g/kg	Field maize, 8541 IT	0.28 0.28 0.28 0.28	190 190 190 190	0.15 0.15 0.15 0.15	08.05.98 08.17.98 08.27.98 09.08.98	Late dent	Grain	<0.02	20
34-00-14 [4] Trial: 98-0171 61198016 York, NE WP, 800 g/kg	Field maize, N3030 Bt	0.28 0.28 0.28 0.28	190 190 190 190	0.15 0.15 0.15 0.15	08.05.98 08.14.98 08.25.98 09.03.98	Late dough	Grain	<0.02	20
34-00-14 [4] Trial: 98-0186 61198015 Dow, IL SC, 240 g/l	Field maize, 8541 IT	0.26 0.26 0.26 0.26	190 190 190 190	0.14 0.14 0.14 0.14	08.05.98 08.17.98 08.27.98 09.08.98	Late dent	Grain	<0.02	20
34-00-14 [4] Trial: 98-0171 61198016 York, NE SC, 240 g/l	Field maize, N3030 Bt	0.26 0.27 0.27 0.27	190 190 190 190	0.14 0.14 0.14 0.14	08.05.98 08.14.98 08.25.98 09.03.98	Late dough	Grain	<0.02	20
34-00-14 [4] Trial: 98-0163 61198017 Cunningham, KS WP, 800 g/kg	Field maize, 2564 Golden Harvest	0.32 0.25 0.26 0.27	130 120 120 120	0.24 0.21 0.22 0.23	07.22.98 08.05.98 08.12.98 08.25.98	Dent	Grain	<0.02	20
34-00-14 [4] Trial: 98-0179 61198018 Noblesville, IN WP, 800 g/kg	Field maize, DK 545 BtY	0.28 0.28 0.28 0.28	190 190 190 190	0.15 0.15 0.15 0.15	08.06.98 08.20.98 09.02.98 09.10.98	Dent	Grain	<0.02	21
34-00-14 [4] Trial: 98-0163 61198017 Cunningham, KS SC, 240 g/l	Field maize, 2564 Golden Harvest	0.30 0.24 0.24 0.25	130 120 120 120	0.23 0.20 0.21 0.22	07.22.98 08.05.98 08.12.98 08.25.98	Dent	Grain	<0.02	20
34-00-14 [4] Trial: 98-0179 61198018 Noblesville, IN SC, 240 g/l	Field maize, DK 545 BtY	0.26 0.26 0.26 0.26	190 190 190 190	0.14 0.14 0.14 0.14	08.06.98 08.20.98 09.02.98 09.10.98	Dent	Grain	<0.02	21
34-00-14 [5] Trial: 98-0219 61198019 Britton, SD WP, 800 g/kg	Field maize, DB 5086	0.28 0.28 0.28 0.28	94 93 93 93	0.30 0.30 0.30 0.30	08.27.98 09.14.98 09.21.98 09.28.98	Mature	Grain Grain Grain Grain	<0.02 <0.02 <0.02 <0.02	0 7 14 21
34-00-14 [5] Trial: 98-0230 61198020 Columbia, MO WP, 800 g/kg	Field maize, 33915 IR	0.31 0.32 0.32 0.36	240 240 230 260	0.13 0.13 0.14 0.14	08.26.98 09.02.98 09.15.98 09.29.98	Dent	Grain Grain Grain Grain	<0.02 <0.02 <0.02 <0.02	0 7 14 21
34-00-14 Trial: 98-0229 61198021 Columbia, MO WP, 800 g/kg [6]	Field maize, 33915 IR	0.31 0.32 0.32 0.36	240 240 230 260	0.13 0.13 0.14 0.14	08.26.98 09.02.98 09.15.98 09.29.98	Dent	Grain	<0.02	21

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method 34-98-186 (preliminary version of 34-00-38), HPLC with UV detection, average recovery from maize grain 84%, LOQ for maize grain 0.02 mg/kg.
[3/] Raw agricultural commodity (RAC) trial.
[4/] Bridging trial.
[5/] Decline trial.
[6/] Processing trial.

Table 89. Residues data summary from supervised trials on maize (corn) in Mexico (Hawkins, 1999).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2/] mg/kg	PHI [3/] days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-27	Field maize,	0.04	120	0.032	12.11.97		Grain	<0.01	156
Trial: 98-0123	Asgrow 7475				12.17.97	whorl		<0.01	156
61298001						1 m high,			
Sinaloa, Mexico		0.08	120	0.064	12.11.97		Grain	<0.01	156
SC, 240 g/l					12.17.97	whorl, 1 m high		<0.01	156
34-00-14	Field maize,	0.04	120	0.032	12.22.97		Grain	<0.01	77
Trial: 98-0124	Dekalb 880				12.30.97	whorl, 1 m high		<0.01	
61398002									
Tamaulipas, Mexico		0.08	120	0.064	12.22.97		Grain	<0.01	77
SC, 240 g/l					12.30.97	whorl, 1 m high		<0.01	

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method 34-98-186 (preliminary version of 34-00-38), HPLC with UV detection, average recovery from maize grain 93.6%, LOQ 0.01 mg/kg.
[3/] Intended for early treatment use, 77-156 days before harvest of grain; no other information provided.

Table 90. Residues data summary from supervised trials on maize (field corn) in Brazil (Steckelberg, 1998b and 1998c).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment [2/]	Commodity	Residue [3/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
AgReg10375	Maize,	0.043	200	0.022	04.25.97	Plants 0.7m	Grain	3.43	0
Trial: RI 3379712	Piranão					high		0.40	7
São Paulo, Brazil								0.14	14
SC, 240 g/l								0.06	103
								<0.01	148
		0.086	200	0.043	04.25.97	Plants 0.7m high	Grain	0.52	7
								0.60	14
AgReg10376	Maize,	0.043	200	0.022	04.15.97	Vegetative growth of	Grain	0.11	7
Trial: RI 3379710	Dina 170								
São Paulo, Brazil		0.086	200	0.043		leaves and stalk	Grain	0.37	7
SC, 240 g/l									

[1] Calculated from kg ai/ha and spray volume.
[2] May be immature grain commodity at 0-14 day PHI.
[3] Analysis method 34-95-55, HPLC with UV detection, average recovery from corn grain 83%, LOQ 0.1 mg/kg.

Oil seeds

Supervised trials on cotton were reported from the United States, Mexico and Australia. Summaries of the trials data are presented in Tables 93 to 95.

Twelve supervised trials were conducted during 1995 to1997, at sites within the major cotton growing areas in the USA (Bender, 1996a, Report No. 34-96-89; Bergin, 1998c, Report No. 34-98-86; Yoshida, 1999d, Report No. 34-99-32). In each of the trials, five applications of a wettable powder formulation containing 800 g/kg methoxyfenozide were made at a rate of 0.4 lb ai/acre (0.45 kg ai/ha) per application, to make a total of 2 lb ai/acre (2.24 kg ai/ha). Intervals between applications were 14-21 days, with the last application occurring 14 days prior to normal harvest. Treatments in 4 of the trials were conducted using two different spray volumes: a standard spray volume (10-30 gallons/acre

or 94-280 l/ha) and an ultra-low volume (1 gallon/acre or about 9.4 l/ha). The GAP information for registered uses of methoxyfenozide in the USA on cotton is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray volume, l/ha	No. or max/season	Interval, days	
WP 800 g/kg	Foliar	0.07-0.45 (0.06-0.4 lb ai/acre)	19 aerial, 47 ground, minimum	1.1 kg ai/ha/season	10-14	14

Bolls were harvested mechanically, using picker- or stripper-equipment. After harvest, samples were shipped to the processing facility and placed in frozen storage upon arrival (1 day). At some sites, samples were frozen in the field and maintained as such until processing. The cotton samples from the raw agricultural commodity (RAC) trials were processed to provide the cotton RAC samples, consisting of undelinted cottonseed (also referred to as ginned seed) and cotton gin trash, representative of that produced commercially. Only ginned seed was obtained from the decline trials. Samples were maintained frozen, at or below -10°C, with sampling to analysis intervals ranging from 6 to 17 months.

Two supervised trials on cotton were conducted in Mexico during 1997 and 1998 (Hawkins, 1998, Report 34-98-143). The trials included determination of residues of methoxyfenozide in cottonseed and its crude oil processed fraction. The trials consisted of three applications of an SC formulation (2F), containing 240 g/l methoxyfenozide, at the rate of either 0.12 kg ai/ha or 0.24 kg ai/ha per application, at 7-day intervals between applications. GAP information on the registered uses of methoxyfenozide in Mexico on cotton is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray conc., kg ai/hl	No. of applications	Interval, days	
SC 240 g/l	Foliar	0.1-0.12	0.07-0.08	2	Not specified	14

Two cotton samples from each control plot, and four from each treated plot, were collected by hand, 14 days after the last application. These were ginned and the undelinted cottonseed immediately frozen until analysis. One of the control samples and two samples from each treatment rate were analyzed for residues. The remaining samples were processed to produce the crude oil. All harvested samples were immediately frozen and shipped frozen, for ginning into cottonseed samples. The samples used to produce crude oil were kept frozen until processing. Cottonseed and cottonseed oil samples were maintained frozen until analysis. The sampling to analysis intervals ranged from 219 to 272 days. The samples were analyzed by Method TR-34-97-001 (Desai, 1997) which determined methoxyfenozide in cottonseed and its processed fractions (including crude oil) with an LOQ of 0.01 mg/kg and LOD of 0.003 mg/kg. The average fortification recovery for cottonseed (n=4) was 85.% ± 5.7%. The average fortification recovery for cottonseed oil (n=4) was 97.%±15 % (Table 136).

Six supervised trials were conducted in cotton growing areas of Australia, from 1996 to 1998 (De Monte, 1998a and 1998b, Reports No. ADM 055/98 and ADM 011/98; Bullen, 1998, Report No. EMH 364/98; Pickering, 1999, Report No. ADM 085/99; Hawes, 1998, Report No. AJH 007/98; and Litzow, 1999, Report No. AJH 032/99). In each trial, cotton plants were treated with three applications of an SC formulation, containing 240 g/l methoxyfenozide, at the rate of 0.4 to 0.8 kg ai/ha per application, with the last treatment applied 21 to 36 days before harvest. Treatments were made at intervals of 7 to 14 days. The GAP in Australia for the registered use of methoxyfenozide on cotton is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray volume, l/ha	No. of applications	Interval, days	
SC 240 g/l	Foliar	0.4 – 0.6	100 (ground) 25 (aerial)	3	10	28

Samples of cottonseed and forage were collected 21, 28 and 36 days after the final application. In four of the trials, raw cotton was removed from all open bolls and separated into lint and seed components by hand ginning, discarding the lint. In the other two trials, raw cotton was ginned and the undelinted samples collected. Forage samples consisted of terminal 15 cm sections of

the main and lateral branches. In two of the trials, cotton gin trash was generated by randomly sampling leaves, bracts, stems, petioles and other plant material. The cotton gin trash samples were dried before freezing. About 0.5-1.0 kg of each sample was taken and frozen until analysis.

Residues of methoxyfenozide were determined following method TR 34-96-22 (Bender, 1996a, Report No. 34-96-89) , in which HPLC with UV detection was used for quantification. The method had an LOQ of 0.05 mg/kg for cottonseed and 0.1 mg/kg for forage. The methods are described in the relevant analytical reports (Shields, 1997c, 1997d, 1997e, 1997f and 1998, Reports No. 97/1506, 96/1536 & 2248, 96/2700, 97/2025, 98/2132 & 98/2409). Average recoveries were 101-105% from cottonseed at 0.05-5.0 mg/kg; 95-98% from forage at 0.1-102 mg/kg; and 99-100% from cotton gin trash at 100-200 mg/kg.

Table 91. Residues data summary from supervised trials on cotton in the USA (Bender, 1996a; Bergin, 1998c; Hawkins, 1998; Yoshida).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-96-89 Trial: 95-0166 Waller, TX WP, 800 g/kg	Cotton DPL-50	0.45	118	0.38	06.19.95 07.03.95 07.22.95 08.08.95 08.22.95	At maturity	Cottonseed	0.080 [2/]	14
34-96-89 Trial: 95-0180 Proctor, AR WP, 800 g/kg	Cotton Stoneville 453	0.45	94	0.48	08.02.95 08.16.95 08.30.95 09.13.95 09.27.95	At maturity	Cottonseed	0.51 [2/]	14
		0.45	9.4	4.8	08.02.95 08.16.95 08.30.95 09.13.95 09.27.95	At maturity	Cottonseed	0.26	14
34-96-89 Trial: 95-0181 Meigs, GA WP, 800 g/kg	Cotton DPL 5415	0.45	94	0.48	07.21.95 08.09.95 08.23.95 09.12.95 10.02.95	At maturity	Cottonseed	0.10 [2/]	14
		0.45	9.4	4.8	07.21.95 08.09.95 08.23.95 09.12.95 10.02.95	At maturity	Cottonseed	0.013	14
34-96-89 Trial: 95-0195 Levelland, TX WP, 800 g/kg	Cotton Paymaster HS-26	0.45	140	0.32	07.19.95 08.07.95 08.23.95 09.06.95 08.25.95	At maturity	Cottonseed Gin trash	0.22 [2/] 7.1	15 15
		0.45	9.4	4.8	07.19.95 08.07.95 08.23.95 09.06.95 08.25.95	At maturity	Cottonseed	0.26	15
34-96-89 Trial: 95-0216 Reedley, CA WP, 800 g/kg	Cotton GC-510	0.45	280	0.16	07.13.95 08.03.95 08.21.95 09.18.95 10.02.95	At maturity	Cottonseed Gin trash	0.50 [2/] 17	14
		0.45	9.4	4.8	08.19.95 08.03.95 08.21.95 09.18.95 10.02.95	At maturity	Cottonseed	0.14	14

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-96-89	Cotton	0.45	280	0.16	07.13.95	At maturity	Cottonseed	0.44 [2/]	7
Trial: 95-0217	GC-717				08.03.95				
Reedley, CA					08.21.95				
WP, 800 g/kg					09.18.95				
					10.02.95				
		0.45	280	0.16	07.05.95	At maturity	Cottonseed	0.52	14
					07.24.95				
					08.14.95				
					08.28.95				
					09.25.95				
		0.45	280	0.16	07.05.95	At maturity	Cottonseed	0.44	21
					07.19.95				
					08.03.95				
					08.24.95				
					09.18.95				
		0.45	280	0.16	07.13.95	At maturity	Cottonseed	0.35	28
					08.03.95				
					08.21.95				
					09.18.95				
					10.09.95				
34-98-86	Cotton	0.45	140	0.32	07.12.96	60% mature	Cottonseed	0.37 [3/]	14
Trial: 96-0250/	Suregrow 125	0.46	140	0.33	07.26.96	bolls open			
21696-054		0.44	140	0.31	08.12.96		Gin trash	15	14
Oil Trough, AR		0.44	140	0.31	08.28.96				
WP, 800 g/kg		0.45	140	0.32	09.18.96				
34-98-86	Cotton	0.43	140	0.31	06.12.96	Maturity	Cottonseed	1.0 [3/]	7
Trial: 96-0251/	Suregrow 125	0.44	140	0.31	06.26.96			0.94	14
21696-055		0.43	140	0.31	07.17.96			1.1	21
Uvalde, TX		0.44	140	0.31	07.31.96			0.65	28
WP, 800 g/kg		0.46	140	0.33	08.14.96				
34-98-86	Cotton	0.45	180	0.25	08.09.96	50% mature	Cottonseed	1.3 [3/]	14
Trial: 96-0312/	Delta Pine	0.45	180	0.25	08.23.96	bolls open			
21696-057	2156	0.45	160	0.28	09.06.96		Gin trash	9.9	14
Claude, TX		0.45	160	0.28	09.20.96				
WP, 800 g/kg		0.45	160	0.28	10.10.06				
34-98-86	Cotton	0.45	16	2.9	08.05.96	30%-50%	Cottonseed	1.2 [3/]	16
Trial: 96-0313/	Paymaster	0.45	15.	3.0	08.20.96	bolls open			
21696-059A	HS-200	0.44	16	2.8	09.04.96		Gin trash	9.4	16
Dill City, OK		0.44	14.	3.0	09.24.96				
WP, 800 g/kg		0.45	15.	3.0	10.08.96				
34-98-86	Cotton	0.45	17	2.7	08.15.96	At maturity	Cottonseed	1.0 [3/]	7
Trial: 96-0341/	Paymaster	0.45	17	2.6	09.04.96			0.42	14
21696-059B	HS-200	0.45	15	2.9	09.19.96			0.52	21
Alfalfa, OK		0.45	15	2.9	10.04.96			0.34	27
WP, 800 g/kg		0.44	15	2.9	10.22.96				
34-98-86	Cotton	0.45	13	3.4	09.08.97	60% bolls	Cottonseed	0.98 [3/]	14
Trial: 98-0029/	Delta Pine	0.46	13	3.5	09.29.97	Open			
21697-041	2156	0.46	15	3.0	10.13.97				
Goodnight, TX		0.45	16	2.9	10.27.97		Gin trash	3.8	14
WP, 800 g/kg		0.46	16	2.9	11.14.97				
34-99-32	Cotton	0.44	18	2.4	09.11.98	80% open	Cottonseed	0.51 [3/]	14
Trial: 98-0317/	Quickee RR	0.45	19	2.4	09.15.98	bolls			
1559839		0.45	13	3.5	10.09.98				
Groom, TX		0.45	13	3.5	10.13.98		Gin trash	12	14
WP, 800 g/kg		0.45	13	3.4	11.05.98				
34-99-32	Cotton	0.45	256	1.7	09.24.98	60-70% boll	Cottonseed	1.5 [3/, 5/]	14
Trial: 98-0318/	Maxxa	0.44	25	1.7	09.26.98	split		1.2	14
1559840 [2/]		0.44	25	1.7	10.17.98			0.75	14
Porterville, CA		0.44	26	1.7	100.20.98		Gin trash	40	14
WP, 800 g/kg		0.44	26	1.7	11.10.98				

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-99-32	Cotton	0.46	18	2.5	09.11.98	80% open	Cottonseed	0.41 [3/]	14
Trial: 98-0317/	Quickee RR	0.46	19	2.4	09.15.98	bolls			
1559839		0.46	13	3.6	10.09.98				
Groom, TX		0.46	13	3.6	10.13.98		Gin trash	18	14
SC, 240 g/l		0.46	13	3.5	11.05.98				
34-99-32	Cotton	0.45	26	1.7	09.24.98	60-70% boll	Cottonseed	1.5 [4/, 5/]	14
Trial: 98-0318/	Maxxa	0.45	25	1.7	09.26.98	split		0.93	14
1559840 [2/]		0.44	25	1.7	10.17.98			0.85	14
Porterville, CA		0.45	26	1.7	100.20.98		Gin trash	53	14
SC, 240 g/l		0.45	26	1.7	11.10.98				

[1/] Calculated from kg ai/ha and spray volume.

[2/] Analysis method TR-34-95-133 (Wu, 1995), HPLC in acetonitrile/water with UV detection, average recovery 89.2% (cotton seed), 88% (gin trash), LOQ 0.025 mg/kg (cotton seed), 0.05 mg/kg (gin trash).

[3/] Analysis method TR 34-97-001 (Desai, 1997), HPLC in acetonitrile/water with UV detection, average recovery 90.3% (cotton seed), 78.4% (gin trash), LOQ 0.01 mg/kg (cotton seed),0.05 mg/kg (gin trash).

[4/] Analysis method TR-34-96-88, HPLC in acetonitrile/water with UV detection, average recovery 98.4% (cotton seed), 85.7% (gin trash), LOQ 0.01 mg/kg (cotton seed), 0.05 mg/kg (gin trash).

[5/] All results from this trial were considered invalid. The final application of methoxyfenozide was made to defoliated plants.

Table 92. Residues data summary from trials on cotton in Mexico (Hawkins, 1998).

Report, trial, location, formulation	Crop Variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue mg/kg [2/]	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-98-143	Cotton,	0.120	250	0.048	10.21.97	At maturity	Cottonseed	0.020	14
Trial: 98-0067	PM 1220	0.120	250	0.048	10.28.97				
2359702		0.120	250	0.048	11.04.97				
Tamaulipas		0.240	250	0.096	10.21.97	At maturity	Cottonseed	0.034	14
Mexico		0.240	250	0.096	10.28.97			0.020	
SC, 240 g/l		0.240	250	0.096	11.04.97				
34-98-143	Cotton,	0.120	250	0.048	10.23.97	At maturity	Cottonseed	0.11	14
Trial: 98-0068/	Delta Pine 50	0.120	250	0.048	10.29.97				
2359701		0.120	250	0.048	11.05.97				
Chiapas		0.240	250	0.096	10.23.97	At maturity	Cottonseed	0.086	14
Mexico		0.240	250	0.096	10.29.97			0.29	
SC, 240 g/l		0.240	250	0.096	11.05.97				

[1/] Calculated from kg ai/ha and spray volume.

[2/] Analysis method TR-34-97-001 (Desai, 1997), HPLC in acetonitrile/water with UV detection, average recovery 90.3% (cotton seed), 78.4% (gin trash), LOQ 0.01 mg/kg (cotton seed), 0.05 mg/kg (gin trash).

Table 93. Residues data summary from trials on cotton in Australia (De Monte, 1998a and 1998b; Bullen, 1998; Pickering, 1999; Hawes, 1998; Litzow, 1999).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [4/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
ADM 011/98	Cotton,	0.3	110	0.27	02.14.96	Late boll fill	Cotton seed [2/]	<0.05	21
Boggabilla	CS 50				02.26.96			<0.05	28
New South Wales					03.05.96			<0.05	36
SC, 240 g/l		0.4	110	0.36	02.14.96	Late boll fill	Cotton seed [2/]	<0.05	21
					02.26.96			<0.05	28
					03.05.96			<0.05	36
		0.6	110	0.54	02.14.96	Late boll fill	Cotton seed [2/]	<0.05	21
					02.26.96			<0.05	28
					03.05.96			<0.05	36

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [4/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
EMH/ 364/98	Cotton,	0.3	120	0.24	03.07.96	40-50% bolls	Cotton seed [2/]	<0.05	23
Darling Downs,	Siokra 1-4				03.18.96	open		<0.05	29
Queensland					03.28.96			<0.05	36
SC, 240 g/l		0.4	120	0.32	03.07.96	40-50% bolls	Cotton seed [2/]	<0.05	23
					03.18.96	open		<0.05	29
					03.28.96			<0.05	36
		0.6	120	0.48	03.07.96	40-50% bolls	Cotton seed [2/]	0.17	23
					03.18.96	open		<u><0.05</u>	29
					03.28.96			<0.05	36
AJH 007/98	Cotton,	0.4	63	0.64	03.04.97	5% cracked bolls	Cotton seed [2/]	<0.05	21
Narrabri, NSW	Sicala V-2i				03.14.97			<0.05	28
SC, 240 g/l					03.24.97			<0.05	35
		0.6	63	0.95	03.04.97	5% cracked bolls	Cotton seed [2/]	0.07	21
					03.14.97			<u><0.05</u>	28
					03.24.97			<0.05	35
		0.8	63	1.3	03.04.97	5% cracked bolls	Cotton seed [2/]	0.08	21
					03.14.97			<0.05	28
					03.24.97			<0.05	35
ADM 055/98	Cotton,	0.4	110	0.36	02.24.97	Late boll fill	Cotton seed [2/]	<0.05	21
Brookstead,	CS 189+				03.04.97			<0.05	28
Queensland					03.13.97			<0.05	35
SC, 240 g/l		0.6	110	0.54	02.24.97	Late boll fill	Cotton seed [2/]	0.07	21
					03.04.97			<u><0.05</u>	28
					03.13.97			<0.05	35
		0.8	110	0.73	02.24.97	Late boll fill	Cotton seed [2/]	0.15	21
					03.04.97			0.06	28
					03.13.97			<0.05	35
AJH 032/99	Cotton,	0.4	88	0.46	02.25.98	80% bolls open	Cotton seed [2/]	0.84	21
Boggabri, NSW	CS 8S	+ oil			03.06.98			2.75	27
SC, 240 g/					03.17.98			0.56	35
							Cotton	165	21
							trash [3/]	69	27
								247	35
		0.6	88	0.68	02.25.98	80% bolls open	Cotton seed [3/]	1.4	21
		+ oil			03.06.98			<u>1.6</u>	27
					03.17.98			1.3	35
							Cotton	148	21
							trash [3/]	83	27
								95	35
		0.6	88	0.68	02.25.98	80% bolls open	Cotton seed [3/]	0.81	21
					03.06.98			<u>4.8</u>	27
					03.17.98			1.1	35
							Cotton	156	21
							trash [3/]	109	27
								58	35
ADM 085/99	Cotton,	0.4	100	0.39	02.13.98	50% bolls open	Cotton seed [3/]	1.43	21
Norwin,	CS 189+	+ oil			02.23.98			0.27	27
Queensland					03.05.98			1.18	35
SC, 240 g/l							Cotton	141	21
							trash [3/]	102	27
								89	28
		0.6	100	0.58	02.13.98	50% bolls open	Cotton seed [3/]	0.19	21
		+ oil			02.23.98			1.8	27
					03.05.98			<u>4.9</u>	35
							Cotton	190	21
							trash [3/]	213	27
								190	35
		0.6	100	0.58	02.13.98	50% bolls open	Cotton seed [3/]	0.09	21
					02.23.98			1.4	27
					03.05.98			<u>3.2</u>	35
							Cotton	194	21
							trash [3/]	188	27
								136	35

[1/] Calculated from kg ai/ha and spray volume.
[2/] Delinted cotton seed samples.
[3/] Cotton seed expressed on a fresh weight basis; gin trash, adjusted to dry weight, assuming 83.3% moisture.
[4] Analysis method 34-96-22 (Desai, 1996), HPLC with UV detection, average recovery 105% (cotton seed), 95% (forage), LOQ 0.05 mg/kg (cotton seed), 0.1 mg/kg (forage).

Tree nuts

Ten supervised trials on tree nuts (5 on almonds and 5 on pecans) were conducted in major almond and pecan growing areas in the USA during the 1999 growing season (Ross, 2001, Report No. 34-01-24). Trees were treated with five foliar applications of either 2F (8 trials) or 80W (2 trials) formulations of methoxyfenozide, at a nominal rate of 0.4 lb ai/acre (0.448 kg ai/ha) per application, for a total treatment of 2 lb ai/acre (2.24 kg ai/ha). The first application was made early in the season. Four additional applications were made 14-28 day intervals. The GAP for registered uses of methoxyfenozide on tree nuts in the USA is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray volume, l/ha	No. or max/season	Interval, days	
SC 240 g/l WP 800 g/kg	Foliar	0.13-0.43 (0.12-0.38 lb ai/acre)	Ground: 470 l/ha for trees in 4th leaf; 940 l/ha for trees in $\geq$5th leaf. Aerial: 190 l/ha.	1.12 kg ai/ha/season	8-14	14

Nuts were harvested by hand, 14 days after the last application. In two decline trials, nuts were harvested at 7, 14, 21, and 28 days after the last application. Each sample was obtained from a minimum of four individual trees. Samples were gathered from the ground after being knocked from the trees. Almond samples were separated into hulls and nuts-with-shell, while pecans were collected as whole nuts. Samples were immediately frozen and remained frozen during shipment, storage and processing (with the exception of almond hulls), until analysis. Almond hull samples were processed by spreading the hulls to dry, then grinding the dried hulls and re-freezing the powdered samples. Nut samples were processed by shelling the frozen nuts and grinding the nutmeat in a food processor with dry ice. The interval between sampling and analysis was approximately 535 days. The trials data are summarized in Tables 94-95.

Table 94. Residues data summary from supervised trials on pecans in the USA (Ross, 2001).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Comm-odity	Residue [4/] mg/kg	PHI days
		kg ai/hl [1/]	kg ai/ha	Water l/ha					
34-01-24 [5/] Trial: 00-0004/ 1559923 Chula, GA SC, 240 g/l	Pecan, Sumnersly	0.056	0.45 0.45 0.45 0.45 0.45	801 786 756 771 845	08.10.99 08.24.99 09.07.99 09.21.99 10.10.99	Very early shuck split	Kernels	<0.02	15
34-01-24 Trial: 00-0003/ 1559924 Nashville, GA SC, 240 g/l [7/]	Pecan Stuart	0.058	0.45 0.45 0.45 0.45 0.45	776 776 769 761 781	08.11.99 [2/,3/] 08.25.99 [2/,3/] 09.08.99 [2/,3/] 09.22.98 [3/] 10.06.99 [2/,3/] 10.20.99 [2/]	Early shuck split	Kernels	0.030 0.028 0.024 0.018 0..017 0.017 0.016	7 7 14 21 21 28 28
34-01-24 Trial: 00-0001/ 1559925 Cary, MS SC, 240 g/l [6/]	Pecans Kiowa	0.051	0.46 0.45 0.45 0.45 0.46	884 884 883 679 896	07.09.99 07.27.99 08.16.99 09.06.99 09.27.99	Nuts 2.25 to 2.5 in. long	Kernels	<0.02	15
34-01-24 Trial: 00-0006/ 1559926 Uvalde, TX SC, 240 g/l [5/]	Pecans Stuart	0.066	0.46 0.45 0.34 0.45 0.44	687 702 683	08.03.99 08.17.99 09.03.99 09.20.99 10.11.99	Shuck split on 30% of nuts	Kernels	0.034	14

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Comm-odity	Residue [4] mg/kg	PHI days
		kg ai/hl [1]	kg ai/ha	Water l/ha					
34-01-24	Pecans	0.051	0.45	878	07.09.99	Nuts 2.25	Kernels	<0.02	15
Trial: 00-0001/	Kiowa		0.46	885	07.27.99	to 2.5 in			
1559925			0.44	672	08.16.99	long			
Cary, MS			0.45	898	09.06.99				
WP, 800 g/kg [6]			0.44	814	09.27.99				
34-01-24	Pecans	0.048	0.48	987	08.25.99	Beginning	Kernels	0.027	14
Trial: 00-0005/	Pawnee		0.46	961	09.13.99	split			
1559927			0.46	947	09.29.99				
Lubbock, TX			0.45	940	10.18.99				
SC, 240 g/l [5]			0.44	923	11.03.99				

[1] Calculated from kg ai/ha and spray volume.
[2] Treatments for 7- and 14-day PHI samples.
[3] Treatments for 21- and 28-day PHI samples.
[4] Analysis method TR 34-00-61, HPLC with UV detection, average recovery 92%, LOQ 0.02 mg/kg (kernels), 0.05 mg/kg (hulls).
[5] Raw agricultural commodity (RAC) trial.
[6] Bridging trial.
[7] Decline trial.

Table 95. Residues data summary from supervised trials on almonds in the USA (Ross, 2001).

Report, trial, location, formulation	Crop variety	Application		Date or number of treatments	Growth stage at last treatment	Comm-odity	Residue [3] mg/kg	PHI days
		kg ai/ha	Water l/ha					
34-01-24 [4]	Almonds	0.45	2219	07.14.99	Immature fruit,	Kernels	0.021	14
Trial: 99-0131/	Fritz	0.44	2283	07.28.99	2-3 in. diameter			
1559928		0.44	2356	08.11.99				
Bakersfield, CA		0.45	2421	08.25.99		Hulls	26	14
SC, 240 g/l		0.45	2397	09.08.99				
34-01-24 [4]	Almonds	0.44	2353	07.05.99	Fruit, 3 in.	Kernels	0.021	14
Trial: 99-0130/	Mission	0.45	2473	07.19.99	diameter			
1559929		0.45	2437	08.02.99				
Porterville, CA		0.44	2509	08.16.99		Hulls	6.4	
SC, 240 g/l		0.45	2465	08.30.99				
34-01-24 [5]	Almonds	0.45	2283	06.25.99	Immature fruit,	Kernels	<0.02	14
Trial: 99-0124/	Carmel	0.45	2418	07.09.99	2-3 in. diameter			
1559930		0.44	2449	07.23.99				
Poplar, CA		0.45	2399	08.06.99		Hulls	10	14
SC, 240 g/l		0.44	2422	08.20.99				
34-01-24 [4]	Almonds	0.45	1396	06.14.99	Nuts, 2-3 in.	Kernels	0.074	14
Trial: 99-0126/	Non Pareil	0.45	1404	06.28.99	length			
1559932		0.46	1433	07.12.99				
Madera, CA		0.45	1412	07.26.99		hulls	16	
SC, 240 g/l		0.45	1386	08.09.99				
34-01-24 [5]	Almonds	0.45	2262	06.25.99	Immature fruit,	Kernels	0.02	14
Trial: 99-0124/	Carmel	0.46	2435	07.09.99	2-3 in. diameter			
1559930		0.45	1532	07.23.99				
Poplar, CA		0.45	2390	08.06.99		Hulls	10	14
WP, 800 g/kg		0.46	2449	08.20.99				

Report, trial, location, formulation	Crop variety	Application		Date or number of treatments	Growth stage at last treatment	Comm-odity	Residue [3] mg/kg	PHI days
		kg ai/ha	Water l/ha					
34-01-24 [6]	Almonds	0.45	1395	06.14.99 [1/2]	Nuts, 1.5–2 in. length	Kernels	0.054	7
Trial: 99-0127/	Non Pareil	0.45	1399	06.28.99 [1/2]			0.047	7
1559933		0.45	1397	07.12.99 [1/2]			0.036	14
Fresno, CA		0.44	1395	07.26.99 [2]			0.030	21
WP, 800 g/kg		0.45	1403	08.09.99 [1/2]			0.030	21
				08.23.99 [1]			0.031	28
							0.033	28
						Hulls	34.7	7
							32.6	7
							35	14
							30.2	21
							18.5	21
							28.8	28
							27.1	28

[1] Treatments for 7- and 14-day PHI samples.
[2] Treatments for 21- and 28-day PHI samples.
[3] Analysis method TR 34-00-61, HPLC with UV detection, average recovery 92%, LOQ 0.02 mg/kg (kernels), 0.05 mg/kg (hulls).
[4] Raw agricultural commodity (RAC) trial.
[5] Bridging trial.
[6] Decline trial.

Straw, fodder and forage of cereal grains and grasses

Twenty-five supervised field trials for methoxyfenozide use on maize were conducted in 1998, in major field corn growing areas in the USA (Carpenter, 2000, Report No. 34-00-14). Plots were treated with either 80W (wettable powder containing 800 g/kg methoxyfenozide) or 2F (suspension concentrate containing 240 g/l methoxyfenozide) formulations. Each treated plot received four applications of 0.25 lb ai/acre (0.28 kg ai/ha) per application, giving a total nominal application rate of 1.0 lb ai/acre (1.12 kg ai/ha). Applications were made at 7- to 14-day intervals. Either a tractor-mounted boom sprayer or a CO_2-powered backpack sprayer with spray boom was used to apply the product. The GAP for use of methoxyfenozide on maize (field corn) in the USA is as follows:

Formulation type/conc.	Method	Application				PHI, days
		Rate, kg ai/ha	Spray volume, l/ha	No. or max/season	Interval, days	
SC 240 g/l WP 800 g/kg	Foliar	0.07 – 0.13 (0.06 – 0.12 lb ai/acre)	47 minimum	1.12 kg ai/ha/season	Not specified	21 (grain, forage, fodder)

Treated and control maize grain, forage and fodder samples were harvested from all trials on day 21 ± 2 days. In addition, for decline trials, samples for all matrices were also taken on days 0, 7, 14 and 28. For forage, two samples were collected from each untreated control plot and two samples were independently collected from each treated plot at each sampling times. Samples were harvested manually, from a minimum of 12 individual plants in different areas of the plot. Forage samples were taken from three groups of four plants, each of which was cut into thirds (top, middle and bottom). Composite samples consisted of the top third from the first four plants, the middle third of the second four plants and the bottom third of the remaining four plants. Most forage samples included ears. Duplicate samples were collected from each plot. For grain samples, ears were removed from the 12 plants and the grain was shelled from the ear. The remaining stalk was used for the fodder sample. Composite samples of fodder were prepared in the manner described for forage. Additional corn plants were chopped from the remaining rows of treated and control plots from the trial intended for processing, after samples of the raw agricultural commodity had been collected. The chopped corn was packed into plastic cans, sealed and allowed to ferment for three weeks. At the end of this time, the containers were opened and silage samples were taken. Sampling of silage was done 42 days after the last application. All samples were frozen immediately after collection and maintained frozen until analysis. Sampling to analysis intervals for all maize samples was 3-17 months (Table 96, corresponding data on grain are given in Table 88).

In sweet corn trials in the USA in 1998 (see Table 79), samples of forage and fodder were also taken (Filchner and Carpenter, 2000, Report No. 34-00-15) and formed the source of the data given in Table 97. The USA GAP for use of methoxyfenozide on sweet corn for forage or fodder is as follows:

Formulation type/conc.	Method	Application				PHI days
		Rate, kg ai/ha	Spray volume (l/ha)	No. or max/season	Interval, days	
SC 240 g/l WP 800 g/kg	Foliar	0.07-0.13 (SC) (0.06-0.12 lb ai/acre) 0.07-0.27 [1/] (WP) (0.06-0.24 lb ai/acre)	94 min	1.1 kg ai/ha/season	5 - 10	3 forage 21 fodder

[1/] Up to silking stage only, 0.13 kg ai/ha thereafter.

Table 96. Residues data summary from supervised trials on maize forage and fodder in the USA (Carpenter, 2000).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Com-modity	Residue [2/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-00-14 [3/]	Maize,	0.28	280	0.10	08.20.98	Soft dough	Forage	5.9	21
Trial: 98-0234/	Dekalb	0.28	280	0.10	08.31.98		Forage	6.3	21
61198001	DK 493	0.28	270	0.10	09.10.98				
Hamburg, PA		0.28	270	0.10	09.18.98				
WP, 800 g/kg		0.28	270	0.10	09.10.98	Mature	Fodder	19	21
		0.28	270	0.10	09.18.98		Fodder	17	21
		0.28	270	0.10	09.25.98				
		0.28	270	0.10	10.02.98				
34-00-14 [3/]	Maize,	0.28	180	0.15	07.20.98	Milk	Forage	0.49	21
Trial: 98-0212/	Pioneer 3167	0.28	180	0.15	08.03.98			0.87	21
61198002		0.28	180	0.15	08.17.98				
Montezuma, GA		0.28	180	0.15	08.24.98				
WP, 800 g/kg		0.28	180	0.15	08.17.98	Late dough	Fodder	3.6	21
		0.28	180	0.15	08.24.98			6.1	21
		0.30	180	0.16	09.02.98				
		0.28	180	0.15	09.09.98				
34-00-14 [3/]	Maize,	0.28	180	0.15	07.22.98	R5 = dent	Forage	3.0	21
Trial: 98-0199/	Cropland	0.28	180	0.15	07.31.98		Forage	1.8	21
61198003	Genetics	0.28	180	0.15	08.11.98				
Webster City, IA	N4640BT	0.28	180	0.15	08.25.98				
WP, 800 g/kg		0.28	180	0.15	08.25.98	R6 =	Fodder	12	21
		0.28	180	0.15	09.01.98	maturity			
		0.28	180	0.15	09.08.98				
		0.28	180	0.15	09.17.98				
34-00-14 [3/]	Maize,	0.27	180	0.15	07.31.98	Milk	Forage	4.1	19
Trial: 98-0223/	Pioneer 3751	0.27	180	0.15	08.08.98		Forage	4.9	19
61198004		0.27	180	0.15	08.15.98				
Theilman, MN		0.27	180	0.15	08.23.98				
WP, 800 g/kg		0.27	180	0.15	08.23.98	Dent	Fodder	11	21
		0.27	180	0.15	08.30.98		Fodder	11	21
		0.27	180	0.15	09.08.98		Fodder	14	21
		0.27	180	0.15	09.17.98		Fodder	15	21
34-00-14 [3/]	Maize,	0.28	94	0.30	08.19.98	Early dent	Forage	12	21
Trial: 98-0233/	DB5086	0.28	93	0.30	08.26.98		Forage	6.9	21
61198005		0.28	94	0.30	09.02.98				
Campbell, MN		0.28	94	0.30	09.11.98				
WP, 800 g/kg		0.28	94	0.30	08.19.98	Early dent	Fodder	8.4	23
		0.28	93	0.30	08.26.98		Fodder	7.6	23
		0.28	94	0.30	09.11.98				
		0.28	94	0.30	09.16.98				

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Com-modity	Residue [2/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-00-14 [3/]	Maize,	0.28	190	0.15	07.08.98	Milk	Forage	3.8	21
Trial: 98-0170/	N3030 Bt	0.28	190	0.15	07.19.98			3.8	21
61198006		0.28	190	0.15	07.27.98				
Osceola, NE		0.28	190	0.15	08.06.98				
WP, 800 g/kg		0.28	190	0.15	08.06.98	Late dough	Fodder	29	21
		0.28	190	0.15	08.13.98		Fodder	35	21
		0.28	190	0.15	08.25.98				
		0.28	190	0.15	09.01.98				
34-00-14 [3/]	Maize,	0.28	170	0.16	07.23.98	Milk	Forage	0.11	21
Trial: 98-0180/	Pioneer	0.28	190	0.15	08.06.98		Forage	0.10	21
61198007	34G81	0.28	190	0.15	08.13.98				
Sheridan, IN		0.28	190	0.15	09.20.98				
WP, 800 g/kg		0.25	170	0.15	08.06.98	Dough to	Fodder	31	21
		0.28	190	0.15	08.20.98	dent	Fodder	36	21
		0.28	190	0.15	09.03.98				
		0.28	190	0.15	09.10.98				
34-00-14 [3/]	Maize,	0.28	190	0.15	08.04.98	Hard dough	Forage	5.8	21
Trial: 98-0333/	Pioneer 3979	0.28	190	0.15	08.14.98			2.8	21
61198008		0.28	190	0.15	08.21.98				
Northwood, ND		0.28	190	0.15	08.28.98				
WP, 800 g/kg		0.28	190	0.15	08.21.98	Dent	Fodder	6.9	21
		0.28	190	0.15	08.28.98		Fodder	7.5	21
		0.28	190	0.15	09.04.98				
		0.28	190	0.15	09.14.98				
34-00-14 [3/]	Maize,	0.28	180	0.16	07.22.98	Dough	Forage	1.4	21
Trial: 98-0187/	Pioneer 3730	0.28	180	0.16	07.29.98		Forage	2.7	21
61198009		0.28	180	0.16	08.05.98				
Lime Springs, IA		0.27	180	0.15	08.12.98				
WP, 800 g/kg		0.28	180	0.16	08.05.98	Black layer	Fodder	8.7	21
		0.27	180	0.15	08.12.98	(after dent)	Fodder	12.6	21
		0.28	180	0.16	08.19.98				
		0.29	180	0.16	09.02.98				
34-00-14 [3/]	Maize,	0.28	330	0.086	07.29.98	Dough	Forage	1.1	21
Trial: 98-0327/	AP9121	0.28	330	0.084	08.10.98		Forage	1.4	21
61198010		0.28	340	0.083	08.18.09				
Fitchburg, WI		0.28	320	0.087	08.25.98				
WP, 800 g/kg		0.29	330	0.087	08.25.98	Dent	Fodder	4.9	21
		0.29	330	0.087	09.01.98		Fodder	5.0	21
		0.52	340	0.15	09.15.98				
		0.49	320	0.15	09.28.98				
34-00-14 [3/]	Maize,	0.28	120	0.23	07.16.98	Blister	Forage	11.	21
Trial: 98-0182/	SC 1068	0.28	140	0.20	07.23.98		Forage	3.6	21
61198011		0.28	130	0.22	08.96.98				
New Holland, OH		0.28	130	0.22	08.13.98				
WP, 800 g/kg		0.27	120	0.22	08.13.98	Dent	Fodder	32	21
		0.28	140	0.20	08.27.98		Fodder	26	21
		0.26	130	0.20	09.10.98				
		0.26	130	0.20	09.24.98				
34-00-14 [3/]	Maize,	0.28	190	0.15	07.13.98	Blister	Forage	0.54	21
Trial: 98-0195/	DK 471	0.28	190	0.15	07.27.98			0.76	21
61198012		0.28	180	0.15	08.03.98				
Williamston, MI		0.28	180	0.15	08.12.98				
WP, 800 g/kg		0.28	190	0.15	08.19.98	Dent	Fodder	25	21
		0.28	190	0.15	08.28.98		Fodder	34	21
		0.28	180	0.15	09.04.98				
		0.28	180	0.15	09.14.98				

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Com-modity	Residue [2/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-00-14 [3/]	Maize,	0.28	190	0.15	07.07.98	Tassel	Forage	4.4	21
Trial: 98-0185	8541 IT	0.28	190	0.15	07.14.98		Forage	4.1	21
61198013		0.28	190	0.15	07.28.98				
Dow, IL		0.28	190	0.15	08.05.98				
WP, 800 g/kg		0.28	190	0.15	08.05.98	Dough to	Fodder	21	20
		0.28	190	0.15	08.17.98	dent		21	20
		0.28	190	0.15	08.27.98				
		0.28	190	0.15	09.08.98				
34-00-14 [3/]	Maize,	0.28	120	0.23	07.08.98	Milk	Forage	2.6	21
Trial: 98-0200	N7639 BT	0.28	120	0.24	07.16.98			1.1	21
61198014		0.28	120	0.22	07.24.98				
Eakly, OK		0.28	130	0.21	08.04.98				
WP, 800 g/kg		0.27	120	0.22	07.24.98	Dough to	Fodder	7.1	20
		0.24	120	0.21	08.04.98	dent	Fodder	8.2	20
		0.26	120	0.21	08.17.98				
		0.30	130	0.23	08.28.98				
34-00-14 [4/]	Maize,	0.28	190	0.15	07.07.98	Tassel	Forage	5.0	21
Trial: 98-0186	8541 IT	0.28	190	0.15	07.14.98		Forage	6.6	21
61198015		0.28	190	0.15	07.28.98				
Dow, IL		0.28	190	0.15	08.05.98				
WP, 800 g/kg		0.28	190	0.15	08.05.98	Late dent	Fodder	12	20
		0.28	190	0.15	08.17.98		Fodder	11	20
		0.28	190	0.15	08.27.98				
		0.28	190	0.15	09.08.98				
34-00-14 [4/]	Maize,	0.28	190	0.15	07.04.98	16-18 leaf	Forage	2.0	21
Trial: 98-0171	N3030 Bt	0.28	190	0.15	07.17.98			2.9	21
61198016		0.28	190	0.15	07.24.98				
York, NE		0.28	190	0.15	08.05.98				
WP, 800 g/kg		0.28	190	0.15	08.05.98	Late dough	Fodder	59	20
		0.28	190	0.15	08.14.98		Fodder	60	20
		0.28	190	0.15	08.25.98				
		0.28	190	0.15	09.03.98				
34-00-14 [4/]	Maize,	0.26	190	0.14	07.07.98	Tassel	Forage	5.8	21
Trial: 98-0186	8541 IT	0.26	190	0.14	07.14.98		Forage	4.8	21
61198015		0.26	190	0.14	07.28.98				
Dow, IL		0.26	190	0.14	08.05.98				
SC, 240 g/l		0.26	190	0.14	08.05.98	Late dent	Fodder	14.	20
		0.26	190	0.14	08.17.98		Fodder	17.	20
		0.26	190	0.14	08.27.98				
		0.26	190	0.14	09.08.98				
34-00-14 [4/]	Maize,	0.26	190	0.14	07.04.98	16-18 leaf	Forage	2.9	21
Trial: 98-0171	N3030 Bt	0.26	190	0.14	07.17.98		Forage	3.0	21
61198016		0.26	190	0.14	07.24.98				
York, NE		0.26	190	0.14	08.05.98				
SC, 240 g/l		0.26	190	0.14	08.05.98	Late dough	Fodder	46	20
		0.27	190	0.14	08.14.98		Fodder	53	20
		0.27	190	0.14	08.25.98				
		0.27	190	0.14	09.03.98				
34-00-14 [4/]	Maize,	0.28	130	0.22	06.17.98	Blister	Forage	0.16	19
Trial: 98-0163	2564 Golden	0.28	120	0.24	06.24.98		Forage	0.14	19
61198017	Harvest	0.28	120	0.24	07.01.98				
Cunningham, KS		0.28	120	0.24	07.15.98				
WP, 800 g/kg		0.32	130	0.24	07.22.98	Dent	Fodder	18	20
		0.25	120	0.21	08.05.98		Fodder	17	20
		0.26	120	0.22	08.12.98				
		0.27	120	0.23	08.25.98				

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Com-modity	Residue [2] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1]					
34-00-14 [4]	Maize,	0.28	190	0.15	07.23.98	Milk to	Forage	1.5	21
Trial: 98-0179	DK 545 BtY	0.28	190	0.15	08.06.98	dough	Forage	1.8	21
61198018		0.28	190	0.15	08.13.98				
Noblesville, IN		0.28	190	0.15	08.20.98				
WP, 800 g/kg		0.28	190	0.15	08.06.98	Dent	Fodder	58	21
		0.28	190	0.15	08.20.98		Fodder	86	21
		0.28	190	0.15	09.02.98				
		0.28	190	0.15	09.10.98				
34-00-14 [4]	Maize,	0.26	130	0.20	06.17.98	Blister	Forage	0.13	21
Trial: 98-0163	2564 Golden	0.27	120	0.23	06.24.98		Forage	0.11	21
61198017	Harvest	0.27	120	0.23	07.01.98				
Cunningham, KS		0.26	120	0.23	07.15.98				
SC, 240 g./l		0.30	130	0.23	07.22.98	Dent	Fodder	25	20
		0.24	120	0.20	08.05.98			19	20
		0.24	120	0.21	08.12.98				
		0.25	120	0.22	08.25.98				
34-00-14 [4]	Maize,	0.26	190	0.14	07.23.98	Milk to	Forage	1.7	21
Trial: 98-0179	DK 545 BtY	0.26	190	0.14	08.06.98	dough	Forage	1.1	21
61198018		0.26	190	0.14	08.13.98				
Noblesville, IN		0.26	190	0.14	08.20.98				
SC, 240 g./l		0.26	190	0.14	08.06.98	Dent	Fodder	86	21
		0.26	190	0.14	08.20.98		Fodder	120	21
		0.26	190	0.14	09.02.98		Fodder	110	21
		0.26	190	0.14	09.10.98				
34-00-14 [5]	Maize,	0.28	94	0.30	08.27.98	Dent	Forage	23	0
Trial: 98-0219	DB 5086	0.28	93	0.30	09.04.98		Forage	20	7
61198019		0.28	93	0.30	09.14.98		Forage	2.5	14
Britton, SD		0.28	93	0.30	09.21.98		Forage	3.0	21
WP, 800 g/kg							Forage	3.0	21
							Forage	2.4	28
		0.28	94	0.30	08.27.98	Mature	Fodder	30	0
		0.28	93	0.30	09.14.98		Fodder	10	7
		0.28	93	0.30	09.21.98		Fodder	8.4	14
		0.28	93	0.30	09.28.98		Fodder	19	21
							Fodder	7.2	21
							Fodder	13	28
34-00-14 [5]	Maize,	0.28	240	0.12	07.24.98	Milk	Forage	12	0
Trial: 98-0230	33915 IR	0.28	240	0.12	08.03.98		Forage	8.2	6
61198020		0.28	230	0.12	08.12.98		Forage	5.4	14
Columbia, MO		0.28	260	0.12	08.20.98		Forage	2.8	21
WP, 800 g/kg							Forage	2.8	21
							Forage	2.4	28
		0.31	240	0.13	08.26.98	Dent	Fodder	8.8	0
		0.32	240	0.13	09.02.98		Fodder	3.9	7
		0.32	230	0.14	09.15.98		Fodder	5.3	14
		0.36	260	0.14	09.29.98		Fodder	3.7	21
							Fodder	4.0	21
							Fodder	6.1	28
34-00-14 [6]	Maize,	0.28	240	0.12	07.24.98	Milk	Forage	6.0	21
Trial: 98-0229	33915 IR	0.28	240	0.12	08.03.98		Forage	8.6	21
61198021		0.28	230	0.12	08.12.98		Silage	1.4	42
Columbia, MO		0.28	260	0.11	08.20.98		Silage	3.0	42
WP, 800 g/kg		0.31	240	0.13	08.26.98	Dent	Fodder	1.3	21
		0.32	240	0.13	09.02.98		Fodder	3.4	21
		0.32	230	0.14	09.15.98				
		0.36	260	0.14	09.29.98				

[1] Calculated from kg ai/ha and spray volume.
[2] Analysis method TR 34-98-186 (preliminary method for 34-99-74), HPLC with UV detection, average recovery 96% from forage, 91% from fodder, LOQ 0.04 mg/kg (forage, silage and fodder).
[3] Raw agricultural commodity (RAC) trial.
[4] Bridging trial.
[5] Decline trial.
[6] Processing trial.

Table 97. Residues data summary from supervised trials on sweet corn forage and fodder in the USA (Filchner and Carpenter, 2000).

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-00-15 [3/]	Sweet corn,	0.28	580	0.049	07.17.98	Tassle	Forage	6.1	3
Trial: 98-0178/	Fortune	0.28	490	0.058	07.27.98				
61198022		0.26	490	0.054	08.03.98				
Hamburg, PA		0.28	490	0.058	08.11.98				
WP, 800 g/kg							Fodder	1.0	21
34-00-15 [3/]	Sweet corn,	0.29	240	0.12	07.21.98		Forage	15	3
Trial: 98-0204/	Tuxedo	0.27	220	0.12	07.28.98				
61198023		0.28	230	0.12	08.04.98				
North Rose, NY		0.28	230	0.12	08.11.98				
WP, 800 g/kg							Fodder	9.4	21
34-00-15 [3/]	Sweet corn,	0.28	180	0.15	07.20.98	Milk	Forage	3.4	3
Trial: 98-0214/	Silver Queen	0.28	190	0.15	08.03.98				
61198024		0.28	190	0.15	08.17.98				
Montezuma, GA		0.28	190	0.15	08.24.98				
WP, 800 g/kg							Fodder	5.9	21
34-00-15 [3/]	Sweet corn,	0.28	190	0.15	06.03.98	Dry silk	Forage	0.20	3
Trial: 98-0149/	Abbott &	0.28	190	0.15	06.20.98				
61198025	Cobb 8100	0.28	190	0.15	06.17.98				
O'Brein, FL		0.28	190	0.15	06.29.98				
WP, 800 g/kg									
34-00-15 [3/]	Sweet corn,	0.28	190	0.15	08.18.98	Milk	Forage	22.	3
Trial: 98-0218/	Seneca	0.27	190	0.15	08.15.98				
61198026	Appaloosa	0.28	190	0.15	08.23.98				
Theilman, MN	SH2	0.28	190	0.15	08.30.98				
WP, 800 g/kg							Fodder	20.	21
34-00-15 [3/]	Sweet corn,	0.28	200	0.14	09.14.98	Mature	Forage	7.2	3
Trial: 98-0328/	Sweetie 82	0.28	200	0.14	09.21.98				
61198027		0.29	210	0.14	09.28.98				
Madera, CA		0.29	210	0.14	10.05.98				
WP, 800 g/kg							Fodder	8.2	21
34-00-15 [3/]	Sweet corn,	0.28	140	0.198	07.23.98	Milk	Forage	4.6	3
Trial: 98-0193/	Jubilee	0.28	140	0.198	07.30.98				
61198028		0.28	140	0.200	08.07.98				
Ephrata, WA		0.28	140	0.202	08.14.98				
WP, 800 g/kg							Fodder	8.2	21
34-00-15 [3/]	Sweet corn,	0.28	190	0.150	08.19.98	Mature	Forage	4.4	3
Trial: 98-0334/	SS Jubilee	0.28	190	0.150	09.02.98				
61198029		0.28	180	0.152	09.16.98				
Corvallis, OR		0.28	190	0.147	09.26.98				
WP, 800 g/kg							Fodder	2.0	21
34-00-15 [4/]	Sweet corn,	0.28	330	0.085	07.29.98	Milk	Forage	6.2	3
Trial: 98-0224/	Empire	0.28	330	0.084	08.10.98				
61198030		0.28	340	0.083	08.18.98				
Fitchburg, WI		0.28	320	0.087	08.25.98				
WP, 800 g/kg							Fodder	8.4	21
34-00-15 [4/]	Sweet corn,	0.28	190	0.15	07.16.98	Milk	Forage	1.5	3
Trial: 98-0157/	Silver Queen	0.28	190	0.15	07.23.98				
61198031		0.28	190	0.15	07.31.98				
Nobelsville, IN		0.28	190	0.15	08.11.98				
WP, 800 g/kg							Fodder	46	21
34-00-15 [4/]	Sweet corn,	0.265	330	0.081	07.29.98	Milk	Forage	6.2	3
Trial: 98-0224/	Empire	0.264	330	0.079	08.10.98			9.0	
61198030		0.265	340	0.079	08.18.98				
Fitchburg, WI		0.265	320	0.083	08.25.98				
SC, 240 g/l							Fodder	3.0	21
								3.9	

Report, trial, location, formulation	Crop variety	Application			Date or number of treatments	Growth stage at last treatment	Commodity	Residue [2/] mg/kg	PHI days
		kg ai/ha	Water l/ha	kg ai/hl [1/]					
34-00-15 [4/]	Sweet corn,	0.266	190	0.14	07.16.98	Milk	Forage	1.0	3
Trial: 98-0157/		0.264	190	0.14	07.23.98			1.5	
61198031		0.264	190	0.14	07.31.98				
Nobelsville, IN		0.264	190	0.14	08.11.98				
SC, 240 g/l							Fodder	20	21
								21	
34-00-15 [5/]	Sweet corn,	0.28	118	0.24	07.01.98	Late milk	Forage	1.1	0
Trial: 98-0145/	Super sweet	0.28	115	0.24	07.15.98			0.65	2
61198032		0.28	114	0.24	07.18.98			0.097	2
Cunningham, KS		0.28	114	0.24	07.22.98			0.27	7
WP, 800 g/kg								1.4	9
							Fodder	3.4	21
								4.1	21
								3.6	29
								4.9	34
34-00-15 [5/]	Sweet corn,	0.28	118	0.24	07.16.98	Early milk	Forage	0.61	0
Trial: 98-0164/	Golden	0.28	115	0.24	07.23.98			0.52	3
61198033	Nuggets	0.28	114	0.24	07.31.98			0.14	3
New Holland, OH		0.28	114	0.24	08.07.98			0.14	7
WP, 800 g/kg								0.15	10
							Fodder	16	21
								19	21
								13	28
								20	35

[1/] Calculated from kg ai/ha and spray volume.
[2/] Analysis method TR 34-98-186 (preliminary method for 34-99-74), HPLC with UV detection, average recovery 97% from forage, 95% from fodder, LOQ 0.04 mg/kg (forage and fodder).
[3/] Raw agricultural commodity (RAC) trial.
[4/] Bridging trial.
[5/] Decline trial.

FATE OF RESIDUES IN STORAGE AND PROCESSING

Processing

Processing studies were conducted on oranges, apples, grapes, tomatoes, corn, fresh prunes, and cottonseed.

Oranges

Processing studies on oranges were conducted in Europe and are summarized in Table 98. Residues of methoxyfenozide in/on orange fruit, peel, pulp, and marmalade were determined, following spray application in the field, in Spain (Walz-Tylla, 1999l, Report No. 34-99-134). Residues arising in the field part of the study have been detailed in Table 49 (Report 34-99-133, trial 810533 in Tarragona, Spain). An SC containing 240 g/l methoxyfenozide was applied twice to orange trees with a spray concentration of 0.0096 kg ai/hl. The product was applied, with the addition of oil, at intervals of 10 days, with the last treatment made 15 days prior to harvest. Orange fruit were sampled from various points in the treated trees on day 15 after the last application. Oranges were taken and processed into marmalade, simulating commercial practice. Oranges were washed in standing water and peeled with a knife. The peel was cut into small strips. The fruit pulp was minced with a mixer and subsequently passed through a strainer, to separate pulp waste and fruit purée. Sugar, gelling agent and peel strips were added to the fruit purée and the mixture was heated to 98-100ºC for about 3 minutes. After cooking, the marmalade was cooled and stored deep frozen for 9 days at -18°C or below. The marmalade was then shredded with dry ice and portions were transferred into polystyrene boxes and stored at -18°C until analysis (Table 98).

In a second study, residues of methoxyfenozide in/on orange fruit, peel, pulp, juice, and marmalade were determined following two spray applications in trial fields in Italy and Spain (Seym, and Deissler, 1998c, Report No. 34-99-01). Residues arising in the field part of the study have been detailed in Table 48 (Report 34-99-02, specifically trials 703745 in Montalbano, Italy and 705195 in

Tarragona, Spain). An SC containing 240 g/l methoxyfenozide was applied twice to orange trees with a spray concentration of 0.0096 kg ai/ha. The product was applied, with the addition of oil, at intervals of 10 days, with the last treatment made 14 days prior to harvest. Orange fruit were sampled (7-34 kg) from various points in the treated trees on day 14 after the last application. At least 4 kg of the sampled fruit was separated into pulp, peel, and whole fruit. The remaining 25-26 kg was processed into juice and marmalade. The preparation of marmalade was done according to household practice, as described in the preceding paragraph. The processing procedures for juice simulated commercial practice but were on a laboratory scale. The oranges (20.7-22.7 kg) were washed in standing water and the peel removed with a knife. The peeled oranges were pressed into pulp waste and raw juice. After pressing, the raw orange juice was pasteurized at 89ºC. After pasteurization, the orange juice was transferred into bottles, which were stored at -18°C or below until analysis, in about 7 months (Table 98).

Table 98. Processing factors for orange products (Walz-Tylla, 1999l; Seym, and Deissler, 1998c).

Report, trial, commodity, fraction	Residues, mg/kg	Processing factor
34-99-134, Trial 810533 (Spain)		
Orange fruit (RAC)	0.13	
Pulp [1/]	<0.05	0.4
Peel	0.52	4
Marmalade	0.10	0.8
34-99-01, Trial 703745 (Italy)		
Orange fruit (RAC)	0.20	
Pulp[1]	<0.05	0.3
Peel	0.57	2.9
Marmalade	0.10	0.5
Juice	<0.05	0.3
34-99-01, Trial 705195) (Spain)		
Orange fruit (RAC)	0.22	
Pulp [1/]	<0.05	0.2
Peel	0.94	4.3
Marmalade	0.24	1.1
Juice	<0.05	0.2
Average processing factors for oranges		
Pulp [1/]		0.3
Peel		3.7
Marmalade		0.8
Juice		0.3

[1/] Not the animal feed item "citrus pulp".

Apples

Processing studies on apples were conducted in Germany, Belgium, France and the USA during 1997 to 1998. Studies on pears were also conducted in the USA in 1998.

Methoxyfenozide residues were determined in apples and processed apple fractions (fruit, washed fruit, sauce, juice, pomace) from samples taken from trees that had received three applications of a 240 g/l suspension concentrate formulation, at the rate of 0.14 kg ai/ha (0.0096 kg ai/hl) per application, a total application of 0.43 kg ai/ha. Three trials were conducted, one each in Germany, Belgium (Seym, and Deissler, 1999g, Report No. 34-99-179) and France (Walz-Tylla, 1999m, Report No. 34-99-129). Residues arising in the field part of the study have been detailed in Tables 59 and 60 (Report 34-99-178, trial 703680 in Germany and Trial 705144 in Belgium; Report 34-99-128, Trial 810525 in France). In each of the trials, the treatments were applied at intervals of 13-15 days, with the last application 14 days prior to harvest. Apples (36–48 kg) were taken from the treated plots and either washed, according to household practice, or processed into apple sauce, juice, wet and dry pomace, simulating commercial practice but on a laboratory scale.

According with household practice, apples were washed in standing water, then cut into small pieces with a knife and weighed. The apple pieces were subsequently shredded with dry ice, transferred to polystyrene boxed and stored deep frozen at -18°, or below, until analysis.

In a simulated commercial practice for production of sauce, after washing and cutting the apples, water was added at 250 ml/2 kg apples and mixture heated to 98 - 100ºC for about 15 minutes. The product was passed through a strainer to separate apple sauce from pomace. Sugar was added to the apple sauce, which was then placed in jars. The apple sauce was pasteurized and transferred to polystyrene boxes and stored frozen until analysis.

In a simulation of juice commercial production, after washing, cutting and shredding the apples, the mash was pressed into raw juice and wet pomace. The juice was heated to about 80-85ºC, cooled to 40- 50ºC and enzymes added (Novo Pectinex 3XL and Novo Amylase AG). The juice was then decanted and pasteurized at 88ºC for 0.58-0.86 min. After pasteurization, the juice was transferred to bottles and frozen until analysis. Part of the wet pomace was dried at about 70ºC, to a water content of 8–8.7%. Wet and dry pomace samples were shredded with dry ice and stored frozen until analysis. All processed samples remained frozen for 5-6 months until analysis.

One of the five trials conducted in 1996 in the USA, to determine the residues of methoxyfenozide on apple fruit (RAC), following six applications of the 80W formulation of methoxyfenozide at the rate of 0.3 lb ai/acre (0.336 kg ai/ha) per application, included a determination of residues in processed fractions (Bender and Bergin, 1998, Report No. 34-98-20, Table 52). Field treatments were made at 14-day intervals, with the last application made 14 days before harvest. Samples from the trial in New York state were collected for RAC analysis, along with bulk fruit samples, which were then processed into apple juice and wet pomace. Apples were processed following procedures that simulated commercial practices but at laboratory scale. Apples (105 lbs, 48 kg) were ground in hammer mill and the resulting mash was placed in cheese cloths in a hydraulic press at 2200-3000 psi. The process yielded 73.5 lbs (33 kg) of juice and 24.6 lbs (11 kg) of wet pomace. The juice was not pasteurized.

Table 99. Processing factors for apple products (Seym, and Deissler, 1999g; Walz-Tylla, 1999m; Bender and Bergin, 1998).

Report, trial/processed fraction	Residues [1/], mg/kg	Processing factor
34-99-179, Trial 703680 (Germany)		
Fruit	0.13	
Washed fruit	0.10	0.8
Apple sauce	0.056	0.4
Apple juice	<0.05 (0.028)	0.4
Apple pomace, wet	0.26	2
Apple pomace, dried	0.97	7
34-99-179, Trial 705114 (Belgium)		
Fruit	0.11	
Washed fruit	0.073	0.7
Apple sauce	<0.05 (0.041)	0.4
Apple juice	<0.05 (0.017)	0.4
Apple pomace, wet	0.25	2
Apple pomace, dried	0.89	8
34-99-129, Trial 810525 (France)		
Fruit	0.13	
Washed fruit	0.09	0.7
Apple sauce	0.05	0.4
Apple juice	<0.05 (0.02)	0.4
Apple pomace, wet	0.30	2
Apple pomace, dried	0.91	7
34-98-20, Trial 21696030 (USA)		
Apple fruit	0.27	
Apple pomace, wet	1.6	6
Apple juice	0.058	0.2

Report, trial/processed fraction	Residues [1/], mg/kg	Processing factor
Average processing factors for apples		
Washed fruit		0.7
Sauce		0.4
Juice		0.3
Pomace, wet		3
Pomace, dried		7

[1/] Average of duplicate analyses of single sample.

Stone fruits

Processing studies were conducted in Italy and the USA on peaches and plums, respectively, processing peaches into preserves and plums into prunes (dried plums).

A study was conducted in Italy in 1998 to determine residues of methoxyfenozide in peach fruit after washing or processing into preserves (Walz-Tylla, 1999n, Report No. 34-99-132). The corresponding trial of residues at harvest was previously detailed under Report No. 34-99-131, Trial 812943 (Table 65). A methoxyfenozide SC formulation containing 240 g/l active ingredient was applied twice to peach trees in a spray concentration of 0.012 kg ai/hl. The treatments were made at intervals of 14 days, with the last application 14 days before harvest. Peach fruit samples for processing were collected only from treated trees. Three samples were taken, each weighing about 10–11 kg.

Peaches were washed in standing water and processed following domestic practices. The stones were then removed and the fruits shredded with dry ice in a cutter. The homogenized sample was frozen and maintained frozen until analysis.

Washed fruits were peeled and the stones removed with a knife. The peeled and stoned peaches were placed in preserving cans and immersed in a solution of sugar. Then the preserves were pasteurized at 90-97°C. After pasteurization, the peach preserves were minced and transferred to polystyrene boxes for storage at -18°C until analysis.

In one of the field trials on plums in the USA (Trial 1559922) samples were processed into dried prunes (Guo *et al.*, 2000, Report No. 34-00-75). Plum trees received six applications of methoxyfenozide at the rate of 0.3 lb ai/acre (0.336 kg ai/ha) per application. Samples (approximately 90 lbs) were harvested 7 days after the last application. The bulk sample was shipped, without freezing, to the processing plant. Once processed, samples were frozen, shipped to the laboratory while frozen and stored frozen until analysis. Processing was as follows:

Dry cleaning. Leaves, twigs and stems were removed from the plums and the separated fruit and extraneous materials were weighed.

Cold water dip. The plums were dipped in cold water, kettle was filled with approximately 15 gallons of water and the fruit poured in. The mixture was stirred with a paddle, allowing light extraneous material to float to the surface where it was removed. Surface dirt on the plums was also removed. The water in the kettle was maintained at 72°C.

Drying. After a cold water dip which lasted about 48 hours, the plums were placed into a forced-air drier and dried at 185-196ºF until the moisture content was less than 25%. The moisture content at the end of the process was 17.99%.

Sweat-box storage. The samples were placed in plastic bags, sealed and then stored at about 70ºF for 21 days to allow moisture equilibration.

Rehydration. The prunes were rehydrated to a typical retail moisture content of 28-32%, by immersing them in 177ºF water until they absorbed sufficient water to attain the desired moisture level. To do this, the weight of the pit (stone) was measured and the percentages attributable to pit and flesh were calculated. The moisture content of the equilibrated dried fruit flesh was measured and the amount of water required for rehydration of 3 lbs of dried prunes was calculated. The dried prunes were dipped in water until they weighed the target rehydrated weight, then transferred into plastic bags, sealed and placed in storage at about 70ºF for at least 24 hours, to allow the moisture to equilibrate. The prunes were removed from storage, their moisture content measured and then placed in the freezer until analysis. The finished moisture content was 34 %.

Results for processing of peaches (Italy) and plums (USA) are summarized in Table 100.

Table 100. Processing factors for peach and plum products (Guo *et al.*, 2000; Walz-Tylla, 1999n).

Report, trial, processed fraction	Residues, mg/kg	Processing factor
34-99-132, Trial 812943		
Whole peaches	<0.05	-
Washed peaches	<0.05	-
Peach preserves	<0.05	-
34-00-75, Trial 1559922		
Whole plums	0.19 [1/]	
Prunes	0.26	1.4

[1/] Average from four samples (0.23, 0.099, 0.23 and 0.26 mg/kg).

Grapes

Processing studies on grapes were conducted in the USA and some countries in Europe during 1996 to 1998. The processing factors are summarized in Table 101.

Residues of methoxyfenozide were determined on fresh grapes and processed grape commodities, following three applications at 0.25 lbs ai/acre (0.28 kg ai/ha) per application in the USA (Yoshida, 1999c, Report No. 34-99-77). Samples of 250-300 lbs of grapes were collected 30 days after the last application and sent to the processing plant, without freezing. After processing into juice, raisins, red wine and white wine, the processed samples were immediately frozen and sent to the laboratory where they remained frozen until analysis. Red wine samples were obtained from grapes from Trial 1559823; white wine samples from Trial 1559824; juice from Trial 1559829; and raisins from Trial 1559830 (Table 66). Procedures were as follows.

Juice and pomace. Grapes were weighed , then crushed using a portable stemmer/crusher. As the grapes were crushed, skins, some seeds and juice were collected in a metal trough, fitted with a screen on top. Free-run fresh juice was collected directly into plastic bottles. The crushed grapes were then transferred to a bladder press and pressed for 3 minutes, to express the remaining juice. To clarify the combined juice, sulfur dioxide (as potassium metabisulfite) was added, as required, to prevent fermentation and oxidation. The juice, in a demijohn, was placed in cold storage (2-4ºC) for 2 days, to allow settling of solids. The top 2 gallons of clarified juice was siphoned into a clean plastic container, then passed through a small diatomaceous earth filter into clean plastic bottles. After pressing, samples of wet pomace were placed in plastic bags and the remaining wet pomace was converted into either fermented dry or oven-dry pomace.

Fermented dry pomace. The wet pomace was sprinkled with yeast, covered and left to ferment for approximately 4 days, after which the fermented dry pomace was collected.

Oven-dry pomace. Wet pomace was transferred to wooden trays with plastic mesh linings. The loaded trays were placed in a dehydrating tunnel. Warm air (65-77ºF) was passed over the wet pomace for approximately 16 to 24 hours, until it contained less than 10% moisture. The dried pomace was scraped from the trays and placed in plastic bags.

Red wine. Fresh red grapes were weighed then crushed using the stemmer-crusher. The free-run juice, grape skins and seeds were collected by hand and placed in the bladder press. After pressing, the skins and seeds were removed by hand into plastic buckets. The juice was collected and placed in the buckets with the skins and seeds, to produce a composite known as must. Sulfur dioxide was added (as potassium metabisulfite) to the must at the rate of 30 mg/l. The must was inoculated with active dry wine yeast and mixed twice a day to ensure that the skins and juice were in good contact with each other. The mixture was fermented at 15ºC until complete, then placed in a bladder press and the pomace pressed for 5 minutes. The liquid was collected in 5 gallon glass demijohns and allowed to settle. Once settled, in approximately 4 days, the wine was clarified, racked and the sulfur dioxide content adjusted to 60 mg/l. The wine was cold stabilized at -1-4°C, filtered and bottled. At bottling, the sulfur dioxide content was adjusted to 0.8 mg/l.

White wine. White grapes were weighed, then crushed using the stemmer-crusher. The free-run juice, grape skins and seeds were collected by hand and placed in the bladder press and, after pressing, the skins and seeds were removed by hand into plastic buckets. The juice was collected and placed in the buckets with the skins and seeds. Sulfur dioxide was added (as potassium metabisulfite) to the must at the rate of 30 mg/l. The liquid was allowed clarify for approximately 14-20 hours at

40-50ºF. The liquid was then racked (separated) and allowed to warm to approximately 60ºF. The liquid was inoculated with active dry wine yeast and allowed to ferment at 60ºF. The resulting wine was transferred to demijohns and allowed to settle. Once settled, in approximately 4 days, the wine was clarified, racked and the sulfur dioxide content adjusted to 60 mg/l. The wine was stabilized at 30-40ºF, filtered, bottled and, at bottling, the sulfur dioxide content was adjusted to 0.8 mg/l.

Raisins. Fresh grapes were laid out on wet-strength paper raisin trays, on terraces in the field, approximately 23 pounds of fruit per tray. After approximately one week, the drying grapes were checked, turning trays and clusters on the trays. After another 10 days, the paper trays were rolled up, with the dry grapes in them. The trays and raisins were left in a rolled state and collected after about 14-21 days of drying and then placed in wooden sweat-boxes. The sweat-boxes were transferred to cold storage (less than 15ºC) for about one month, to allow the moisture to equilibrate. The unprocessed dried fruit then entered the processing line, passing across a screen shaker into a cap stemmer, to separate the raisins from the clusters and remove the cap stems. The stems and cap stems, forming part of the raisin waste, were separated from the fruit on a slotted shaker. The raisins then passed under a vacuum unit, adjusted to remove remaining stems and low quality raisins, which were also included in the raisin waste fraction. The raisins were then passed through a sizing and grading shaker, to remove the smaller raisins. The larger raisins then passed through two more vacuum units to remove sub-standard raisins. Separated A- and B-grade raisins were passed into a water tank for washing and then rehydrated to a moisture content acceptable to consumers. Following rehydration, the raisins passed through a recliner and a final vacuum cleaning unit before visual inspection.

Six trials were conducted in Europe (one in France, one in Italy and four in Germany), to determine residues in must and wine after treatment of grape vines with three applications of a suspension concentrate formulation containing 249 g/l methoxyfenozide, at a rate of 0.096 kg ai/ha (Seym and Deissler, 1999h, 1999i and 1999j, Reports No. 34-99-136, 34-99-121 and 34-99-125). Treatments were applied at intervals of 14 days, with the last application made 14 or 21 days before harvest. To obtain sufficient material for processing, 54 to 112 kg samples of bunches of grapes were harvested randomly from various sections of the treated plots. Grapes were frozen and transported frozen. Two samples were kept frozen until analysis, while the remainder were processed into must and wine. Processing procedures, similar to those in the USA trials, followed commercial practice and were as follows.

Red must/wine. Grapes (about 60 kg) were crushed and de-stemmed in an electric grape crusher. The resulting mash was heated for 3 minutes, with stirring, at 80ºC. The mash was then pressed at 0.2–2 bar and the resultant must treated with potassium metabisulfite ("hyposulfite") and bentonite, the latter to prevent a protein haze. Sugar was added to achieve an Oechsle value of 95º. Pure-culture yeast was added to commence fermentation. The first racking occurred after 30 days, with the addition of more potassium metabisulfite. A second racking occurred 122 later, followed by bottling. In some instances (French and Italian trials), after alcoholic fermentation was achieved, a malolactic fermentation process was accelerated by the addition of lactic acid bacteria. This process was carried out at ambient temperature in the absence of air.

White must/wine. White grape must was prepared as above and a small amount was frozen for analysis. The remaining must was transferred to a plastic container and treated with 60 mg/l sulfur dioxide, 10 g/hl potassium caseinate, 30 g/hl bentonite and 25 g/hl yeast. After 2 days, another 25 g/hl yeast was added and the must was fermented for 12 days. The first racking was performed and 30 mg/l sulfur dioxide was added to the young wine. After storage and clarification for 55 days at –5 to -6ºC, the second racking was performed and additional sulfur dioxide added. The wine was then filtered and bottled.

Samples of fresh grapes, must and wine were analyzed for methoxyfenozide residues using method 00470, in which residues were quantified by HPLC-MS/MS. The LOQ for the method was 0.05 mg/kg for all matrices. Fortification recoveries were 98-103% from grapes, 102% from must and 102% from wine.

Two trials were conducted in Portugal and France to determine residues of methoxyfenozide in grape juice and wine after processing grapes harvested from vines treated three times with a suspension concentrate formulation containing 249 g/l active ingredient, at the rate of 0.096 kg ai/ha. The treatments were applied at 14-day intervals, with the last application made 14 days prior to harvest (Heinemann and Seym, 1998b, Report No. 34-98-189). For processing, 53-63 kg samples of bunches of grapes were harvested from treated plots and immediately frozen. The samples were kept frozen for up to 168 days until shipped to the processing facility. After processing, the processed components were kept frozen until analysis. Processing into juice and wine followed commercial procedures which, in general, were similar to those described above but the juice was produced as follows.

Grape juice. The treated frozen bunches of grapes were separated into berries, stalks and stems. The berries were washed in standing water and crushed into a mash, using a punctured-disk mill. The mash was pressed into raw juice and wet pomace. The raw juice was heated to 95ºC for 30 seconds, cooled to 50-55ºC and then de-pectinized by addition of a pectolytic enzyme. The juice was then decanted, filtered and pasteurized at about 85ºC. Samples were stored frozen until analysis.

Residues of methoxyfenozide in grapes and the processed fractions were determined according to method 00470, using electrospray HPLC–MS/MS. The fortification recoveries at 0.05 to 0.50 mg/kg levels were 97-110% from bunches of grapes, 95-111% from juice and 97-102% from wine. The LOQ was 0.05 mg/kg for all matrices.

Processing trials were conducted in Greece and Italy to determine the residues of methoxyfenozide in juice and raisins. The grapes had been treated four times at a rate of 0.096 kg ai/ha at intervals of 14-16 days, with the final application 7 days before harvest (Seym and Deissler, 1999k, Report No. 34-99-119). Samples of bunches of grapes (32-40 kg) for processing were taken from treated plots, immediately frozen and maintained frozen until processing. Processing procedures followed commercial practice. The procedure for preparing juice was similar to that described above, whilst that for raisins was as follows.

Raisins. Bunches of grapes were separated into stems, berries and stalks. The berries were dried for about 12-17 hours at 65ºC, to a water content of 11-12%. The raisins were then washed in standing water and the water content measured to be 14-17%. Samples of the raisins were transferred to polystyrene boxes and frozen until analysis.

Residues of methoxyfenozide were determined according to method 00470 by electrospray HPLC-MS/MS. Fortification recoveries at 0.05 and 0.5 mg/kg levels were 93-102% from bunches of grapes and berries; 99-101% from juice; and 95-101% from raisins. The LOQ was 0.05 mg/kg for all matrices.

An additional processing trial was conducted in Italy, to determine residues of methoxyfenozide in raisins, produced from grapes treated four times with a 240 g ai/l suspension concentrate formulation, at a rate of 0.096 kg ai/ha per application, applied at intervals of 14 days, with the last application 7 days before harvest (Walz-Tylla, 1999o, Report No. 34-99-127). Samples of bunches of grapes (about 20 kg) were taken from treated plots and immediately frozen. The deep frozen materials were shipped to the laboratory for processing and analysis. Samples were kept frozen up to nine months, until analysis. The processing procedure followed commercial practice and was similar to those described for Greek and Italian raisins.

Residues of methoxyfenozide were determined according to method 00470, using electrospray HPLC-MS/MS. Recoveries at fortification levels of 0.05-0.5 mg/kg were 84-99% from bunches of grapes and berries and 75-95% from raisins. The LOQ was 0.05 mg/kg.

Table 101. Processing factors for grape products (Yoshida, 1999c; Seym and Deissler, 1999h, 1999i, 1999j, 1999k; Heinemann and Seym, 1998b; Walz-Tylla, 1999o).

Report, trial and processed fraction	Residues, mg/kg	Processing factor
34-99-77, Trial 1559829 (USA)		
Bunches of grapes	0.26	
Clarified juice	0.072	0.3
Unclarified juice	0.046	0.2
34-99-77, Trial 1559830 (USA)		
Bunches of grapes	0.21	
Raisins	0.28	1.3
34-99-77, Trial 1559823 (USA)		
Bunches of grapes	0.46	
Red wine	0.17	0.4
34-99-77, Trial 1559824 (USA)		
Bunches of grapes	0.33	
White wine	0.10	0.3
34-99-121, Trial 703532 (France)		
Bunches of grapes	0.12	
Must	0.20	1.7
Wine	0.052	0.5
34-99-121, Trial 705446 (Italy)		
Bunches of grapes	0.43	
Must	0.55	1.3
Wine	0.56	1.3
34-99-125, Trial 73540 (Germany)		
Bunches of grapes	0.22	
Must	0.06	0.3
Wine	0.05	0.2
34-99-136, Trial 605867 (Germany)		
Bunches of grapes	0.17	
Must	<0.05	0.3
Wine	<0.05	0.3
34-99-136, Trial 605875 (Germany)		
Bunches of grapes	0.14	
Must	<0.05	0.4
Wine	<0.05	0.4
34-99-136, Trial 607525 (Germany)		
Bunches of grapes	0.18	
Must	<0.05	0.3
Wine	0.053	0.3
34-98-189, Trial 605883 (Portugal)		
Bunches of grapes	0.14	
Juice	<0.050	0.4
Wine	0.056	0.4
34-98-189, Trial 605891(France)		
Bunches of grapes	0.17	
Juice	<0.050	0.3
Wine	<0.050	0.3
34-99-119, Trial 703591 (Greece)		
Bunches of grapes	0.27	
Raisin	0.64	2.4
Juice	<0.050	0.2
34-99-119, Trial 705357 (Italy)		
Bunches of grapes	0.52	
Raisin	1.6	3.0
Juice	<0.05	0.1
34-99-127, Trial 810487 (Italy)		
Bunches of grapes	0.31	
Raisins	0.66	2.1
Raisins	0.64	2.1

Report, trial and processed fraction	Residues, mg/kg	Processing factor
Average processing factors		
Juice		0.3
Raisins		2.6
Must		0.7
Wine		0.4

Tomatoes

Several processing studies on tomatoes were conducted in European countries and in the USA, from 1997 to 1999.

A processing trial was conducted on tomatoes in 1998 in the USA (Bergin, 1999c, Report No. 34-99-47). Tomatoes were harvested from plants treated with four foliar applications (ground equipment) of the 80W formulation of methoxyfenozide, at the rate of 0.25 lb ai/acre (0.28 kg ai/ha) per application, at intervals of 14 days, and were washed and processed into juice, puree and paste. The bulk samples were harvested one day after the last application and shipped (at ambient temperature) to the processing facility on the same day. The processed fractions were frozen, and shipped and stored frozen, until analysis. The total sampling to analysis interval was 163 days.

The following fractions were generated and sampled: unwashed tomatoes, washed tomatoes, juice, puree and paste. The sample of unwashed tomatoes was assembled from randomly chosen fruit from each box, as the contents were transferred into the first washing flume. In the wash step, tomatoes were sprayed with re-circulating wash water, then a rinse water. Samples of washed tomatoes were collected by random selection of fruit after the second spray wash. Samples of processed tomato were placed in a freezer within one hour of sampling. Processing of the tomato samples followed commercial practices, as follows.

Juice. Washed tomatoes were crushed in a grinder, then heated to 204ºF and passed through a screen, to remove peel and seeds and produce the juice.

Purée. Juice was converted to purée using a vacuum evaporator. The juice was re-circulated through the evaporator, measuring total solids content until within the specified range (8-16%). The purée was collected in a drum and weighed.

Paste. Purée was transferred to a vacuum kettle evaporator and evaporated to form paste, with a total solids content of 24%.

Two separate processing trials were conducted in Belgium and in Germany, to determine residues of methoxyfenozide in tomato products produced from tomatoes that had been treated with three applications of a suspension concentrate formulation containing 240 g ai/l, at 0.0096 kg ai/hl per application, at intervals of 7 days. The last application was made one day before harvest (Walz-Tylla, 1999p and 1999q, Reports No. 34-00-04 and 34-99-139). Tomato fruit (a total of about 48 kg) from the treated plot were collected randomly from various points within the plants. After sampling, fruit were frozen within 24 hours and shipped to the processing facility, where processed fractions were prepared and kept frozen for about 4 months until analysis. Washing and peeling was done according to household practice, while preparation of juice, preserves (canned tomato) and paste simulated commercial practices. Procedures for the preparation of tomato juice and paste were similar to those described for the trial in the USA.

Washed tomatoes. Tomatoes were washed in standing water. After washing, they were cut into small pieces with a knife and weighed. The small tomato pieces were stored frozen for 6 days, after which the frozen samples were shredded with dry ice, transferred to polystyrene boxes and frozen until analysis. In the trial in Belgium, the washing water was also weighed, transferred into bottles and frozen.

Peeled tomatoes. Tomatoes were immersed and washed in lukewarm water. After a few minutes, the skins were removed The peeled tomatoes were cut into small pieces and stored frozen for 6 days, after which they were shredded with dry ice, transferred to polystyrene boxes and frozen until analysis. Again, in the Belgian trial, the washing water was weighed and stored frozen.

Tomato preserves. The tomatoes were immersed and washed in lukewarm water. After a few minutes, the peel was removed and the peeled tomatoes placed into preserving cans, with tomato juice

added. The tomato preserves were then pasteurized at 102ºC, minced in a mixer and transferred into polystyrene boxes and stored frozen until analysis.

Processing trials to determine the residues of methoxyfenozide in tomato and tomato processed fractions (fruit, washed fruit, peeled fruit, juice, paste, and preserves) were also conducted in Germany and Italy, using fruit from plants treated with a suspension concentrate formulation at the rate of 0.192 kg ai/ha (0.0096 kg ai/hl) per application (Seym and Deissler, 1999l, Report No. 34-99-123). Applications were made at intervals of 7 days, with the last performed one day before harvest. The field trials were conducted as described in Report 34-99-122 (Trials 703621 in Rheinland, Germany and 705381 in Ragusa, Italy). Samples of mature tomatoes (about 47-49 kg) were collected at random from treated plots for processing. Washing and peeling were done using household practice, while production of juice, paste and preserves simulated commercial practice, on a laboratory scale. Processing procedures were the same as those described in the previous sections. After processing, samples of each fraction were shredded in dry ice and stored frozen for about 11 months until analysis.

Table 102. Processing factors for tomato products (Bergin, 1999c; Walz-Tylla, 1999p, 1999q; Seym and Deissler, 1999l).

Report, trial and processed fraction	Residues, mg/kg	Processing factor
34-99-47, Trial 61298013 (USA)		
Tomato	0.22	
Washed tomato	0.03	0.1
Tomato juice	0.038	0.2
Tomato puree	0.058	0.3
Tomato paste	0.16	0.8
34-00-04, Trial 0161/5 (Belgium)		
Whole tomato	0.25	
Washed tomato	0.14	0.6
Peeled tomato	<0.05	0.2
Preserves	0.05	0.2
Paste	0.56	2.2
Juice	0.08	0.3
Washing water	0.08	0.3
Wet pomace	0.91	3.6
Peeling water w/ peels	0.13	0.5
34-99-139, Trial 810576 (Germany)		
Whole tomato	0.12	
Washed tomato	0.05	0.4
Peeled tomato	<0.05	0.4
Preserves	<0.05	0.4
Paste	0.36	3.0
Juice	0.05	0.4
34-99-123, Trial 703621 (Germany)		
Whole fruit	0.12	
Fruit, washed	<0.05	0.4
Fruit, peeled	<0.05	0.4
Juice	<0.05	0.4
Preserves	<0.05	0.4
Paste	0.20	1.7
34-99-123, Trial 705381 (Italy)		
Whole fruit	0.38	
Fruit, washed	0.22	0.6
Fruit, peeled	0.07	0.2
Juice	0.15	0.4
Preserves	0.098	0.3
Paste	1.3	3.4

Report, trial and processed fraction	Residues, mg/kg	Processing factor
Average processing factors for tomato		
Washed tomato		0.4
Peeled tomato		0.3
Tomato juice		0.3
Tomato puree		0.3
Tomato paste		2.0
Preserves		0.3
Washing water		0.3
Wet pomace		3.6
Peeling water with peel		0.5

Maize (field corn)

In a maize residue trial conducted in the USA, additional samples of maize grain were taken and processed into different fractions (Carpenter, 2000, Report No. 34-00-14). The trial was conducted in Columbia, MO (trial 61198021), where plants received four applications of an 80W formulation of methoxyfenozide, at the rate of about 0.3 kg ai/ha (approximately 0.26 lb ai/acre) per application. Grain samples were harvested 21 days after the last application and immediately processed according to commercial practices, as described below.

Wet Milling. After determining the moisture content of the raw agricultural commodity, the sample was dried in an oven at 110-150ºF, until the moisture content was 10-13%. After drying, the sample was placed in a dust-generation room to remove light impurities, which were separated using a Kice aspirator. After aspiration, the sample was screened and foreign particles removed. The cleaned corn was steeped in water (120-130ºF) containing 0.1-0.2% sulfur dioxide for 22-48 hours. At the end of this period, the corn was passed through a mill to remove the majority of the hull and germ. The germ and hull were then dried and separated using aspiration. The cornstock (without germ and hull) was ground in a mill and passed through a 43 µm screen. Screened-out material was discarded. Starch and gluten passing through the screen were separated into component parts by batch centrifugation. The germ was conditioned to 12% water content, heated to 88-104ºC, flaked and pressed in an expeller, to liberate part of the crude oil. The resultant fractions were crude oil and presscake, containing residual crude oil. The presscake was placed in batch extractors and immersed in hexane at 49-60ºC. After 30 minutes, the hexane was drained and fresh hexane added, to repeat the cycle two more times. The final two washings were for 15-30 minutes each. After the final draining, warm air was forced through the extracted presscake, to remove residual hexane. The miscella (hexane solution of crude oil) was then passed through a recovery unit, to separate the crude oil from hexane. Crude oil was heated to 73-90ºC to remove residual hexane. Crude oil recovered by pressing and solvent extraction was combined and refined according to AOCS method Ca9a52. After refining, the refined oil and soapstock were separated. The refined oil was bleached and deodorized. The wet milling process resulted in samples of hull, germ, gluten, starch, presscake from the expeller, crude oil from the expeller, presscake after solvent extraction, crude oil after solvent extraction, refined oil and soapstock. In addition, aspirated grain fractions were collected.

Dry Milling. The initial steps were the same as those for *wet milling.* After drying and removal of light impurities, the maize grain sample was conditioned to 20-22% water content and allowed to temper for 2-2.5 hours. After tempering, the maize grain was milled and the resultant cornstock dried at 54-71ºC for 30 minutes. The cornstock was allowed to cool and passed through a shaker screen. Material screened out was processed into grits, germ and hull (bran). Material passing through the screen was separated into medium and small grits, coarse meal, meal and flour. The material above the screen was passed through a Kice aspirator, to separate hull material with attached germ from the large grits and germ. The hull material with attached germ was passed through a mill and aspirated, to separate the hull from the germ. The large grits and germ from the first aspiration were separated on a gravity separator. The germ fractions were combined and dried to 7-10% moisture. The material passing through the shaker screen was separated using a Great Western sample shifter. The germ was heated and flaked, prior to extraction with hexane. Extraction produced a miscella from which crude oil was separated and further refined. The *dry milling* process resulted in hull, large grits, medium grits, small grits, coarse meal, meal, flour, flaked germ after solvent extraction, crude oil from solvent extraction, refined oil and soapstock. The processed

fractions were frozen and maintained frozen until analysis. The sampling to analysis intervals ranged from 92 days for refined oil to 105 days for grits and flour. Aspirated grain samples and the processed fractions from wet and dry milling were analyzed using method TR-34-98-186 (34-99-74). The LOQ for the method was 0.02 mg/kg for all matrices. Table 103 summarizes the results.

Table 103. Processing factors for maize (corn) products (Carpenter, 2000).

Crop, report	Processed fraction	Residue, mg/kg	Processing factor
Maize	Corn grain	<0.02 (0.0067)	
(field corn)	Meal	<0.02 (0.0074)	
34-00-14	Grits	<0.02 (0.00)	
	Flour	<0.02 (0.0085)	
	Refined oil (dry milled)	<0.02 (0.014)	
	Aspirated grain	0.59	≥30 [1/]
	Starch	<0.02	
	Refined oil (wet milled)	0.036	≥1.8 [1/]

[1/] The calculations include results with substantial uncertainty, i.e. <LOQ for the RAC. The LOQ value was used in the calculations.

Cotton seed

A cotton seed processing study was conducted in the USA, using samples harvested from plants treated with five applications of an 80W formulation of methoxyfenozide, at the rate of 0.4 lbs ai/acre (0.448 kg ai/ha) per application, at intervals of 14 to 21 days (Bender, 1996b, Report No. 34-96-83). Cotton samples (about 45 kg) were harvested 14 days after the final application, using picker harvesting equipment, and shipped to a processing facility, where the simulated commercial practice was as follows.

Processing method. The cotton was stick-extracted to remove burrs, sticks and other extraneous plant parts (gin trash) and to produce lint cotton, which was then ginned to remove most of the lint. The ginned seed, with approximately 11-15% lint remaining, was delinted to remove remaining lint. After delinting, about 3% of the lint remained with the seed. A huller then mechanically cracked and screened the seed, to separate most of the hull material from the kernel. In practice, if the moisture content of the kernels exceeds 12%, they are dried in an oven. The kernel material was heated to 77-86ºC for 15 to 30 minutes. After heating, the kernel material was flaked and fed into an extruder, where steam was injected into the product. The resultant material (collets) was extracted with hexane to produce a miscella. Some miscella was removed and passed through an evaporator. Crude oil was recovered and refined. The process produced samples of ginned cotton seed, cotton lint, gin trash, delinted cotton seed, linters, linter motes, hulls, kernels, solvent extracted meal, crude oil, refined oil and soapstock.

Processed fractions were frozen and maintained frozen, for up to 240 days, until analysis.

Cotton samples were collected from two cotton field trials in Mexico, 14 days after the last treatment of the plants with three applications of a 2F formulation of methoxyfenozide, at a rate of either 0.12 kg ai/ha or 0.24 kg ai/ha per application, at intervals of 7 to 10 days (Hawkins, 1998, Report No. 34-98-143). From each of the field trials, one control and two treated cottonseed samples were shipped to the laboratory, where they were processed to produce crude oil. Undelinted cotton seed samples were first passed through a delinter, to remove most of the remaining lint. A Bauer mill was used to crack the seed mechanically, then hulls and kernels were separated by screening. The kernel material was dried, then flaked in a flaking roll. The oil was extracted with hexane, then the solvent was removed under vacuum to yield the crude oil. After processing, the crude oil sample was frozen until analysis. Sampling to final analysis times were approximately 7-9 months.

Table 104 Processing factors for cottonseed products (Bender, 1996b; Hawkins, 1998).

Report, trial, commodity fraction	Residue, mg/kg	Processing factor
34-96-83, Trial 269501 (USA)		
Undelinted cotton seed	0.11	
Hulls	0.015	0.14
Crude oil	<0.05 [1/]	≤0.45
Refined oil	<0.05 [1/]	≤0.45
Meal	<0.015 [2/]	≤0.45
34-98-143, Trial 2359702 (Mexico) – 1X		
Cotton seed	0.020	
Crude oil	<0.01	0.5
34-98-143, Trial 2359702 (Mexico) – 2X		
Cotton seed	0.027	
Crude oil	<0.01	0.37
34-98-143, Trial 2359701 (Mexico) – 1X		
Cotton seed	0.12	
Crude oil	0.015	0.13
34-98-143, Trial 2359701 (Mexico) – 2X		
Cotton seed	0.19	
Crude oil	0.026	0.14
Average cotton seed processing factors		
Hulls		0.14
Crude oil		0.32
Refined oil		≤0.45
Meal		≤0.45

[1/] Average values for refined and crude oil were 0.025 and 0.021 mg/kg, respectively. As these were lower than the LOQ, the LOQ value of 0.05 mg/kg was used in the calculations.

[2/] Meal residues were below the estimated LOD (limit of detection) of 0.015 mg/kg. The LOQ (0.05 mg/kg) was used.

Residues in the edible portion of food commodities

Citrus

In field trials conducted in Europe, data on residues in orange and mandarin pulp and peel were obtained, in addition to those for the whole fruit. Table 105 shows the data for pulp and peel. In all trials, the residue level in the pulp was below the LOQ of 0.05 mg/kg, while residues in the peel were 0.31-1.4 mg/kg (average 0.734 mg/kg).

Table 105. Summary of data on the distribution of methoxyfenozide residues in/on oranges and mandarins, from treatments made according to GAP (Seym and Deissler, 1998a, 1999a; Walz-Tylla, 1999a, 1999b).

Commodity, report, country, year	Application				PHI, days	Residues, mg/kg	
	Formulation	kg ai/ha	kg ai/hl	No.		Pulp	Peel
ORANGES: proposed GAP in EU	SC 240 g/l	0.192	0.0096	2	14		
34-99-02, Italy, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.571
34-99-02, Spain, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.946
34-99-02, Spain, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.941
34-99-02, Portugal, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.373
34-99-02, Italy, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.599
34-99-133, Spain, 1998	SC 240 g/l	0.192	0.0096	2	15	<0.05	0.52
34-99-133, Italy, 1998	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.44
34-99-133, Portugal, 1998	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.31
34-99-133, Greece, 1998	SC 240 g/l	0.192	0.0096	2	14	<0.05	1.4
MANDARINS: proposed GAP in EU	SC 240 g/l	0.192	0.0096	2	14		
34-99-140, Italy, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.993
34-99-140, Spain, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	1.02
34-99-140, Portugal, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.44
34-99-140, Spain, 1997	SC 240 g/l	0.192	0.0096	2	14	<0.05	1.25
34-99-137, Italy, 1998	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.79
34-99-137, Greece, 1998	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.84
34-99-137, Portugal, 1998	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.52
34-99-137, Spain, 1998	SC 240 g/l	0.192	0.0096	2	14	<0.05	0.52
Average residues						<0.05	0.734

Pome fruits

The bridging trials on apples and pears, conducted in the USA in 1998, included studies on peeling and washing in which one set of samples per treatment was washed while the other set was peeled. The third set of sample remained unmodified and are referred to as "whole fruit" (Yoshida, 1999a and 1999b, Reports No. 34-99-30 and 34-99-31). Residues were concentrated mainly in the peel and washing removed variable amounts of that residue (Table 106).

Table 106. Residues of methoxyfenozide on apples and pears (pome fruit) after washing or peeling (Yoshida, 1999a, 1999b).

Report, trial, commodity	Residue, mg/kg	Processing factor
34-99-30, Trial 1559836 (OR, USA)		
Whole apples (mean)	0.43	
Washed apples	0.18	0.4
Peeled apples	0.059	0.1
34-99-30, Trial 1559836 (OR, USA)		
Whole apples (mean)	0.52	
Washed apples	0.30	0.6
Peeled apples	0.086	0.2
34-99-30, Trial 1559835 (NY, USA)		
Whole apples (mean)	0.37	
Washed apples	0.40	1
Peeled apples	0.12	0.3
34-99-30, Trial 1559835 (NY, USA)		
Whole apple (mean)	0.60	
Washed apples	0.69	1
Peeled apples	0.14	0.2
34-99-31, Trial 1559837 (NY, USA)		
Whole pears (mean)	0.74	
Washed pears	0.73	1
Peeled pears	0.071	0.1
34-99-31, Trial 1559837 (NY,USA)		
Whole pears (mean)	0.68	
Washed pears	0.67	1
Peeled pears	0.055	0.1
34-99-31, Trial 1559838 (OR, USA)		
Whole pears (mean)	0.50	
Washed pears	0.21	0.4
Peeled pears	0.054	0.1
34-99-31Trial 1559838 (OR, USA)		
Whole pears (mean)	0.74	
Washed pears	0.53	0.7
Peeled pears	0.088	0.1
Averages	Washing	0.76
	Peeling	0.15

Head cabbages

In trials in the USA on head cabbage, samples with and without wrapper leaves were analyzed for residues of methoxyfenozide. The results showed that most of the residues remained in the outer leaves of the cabbage head. See Table 107.

Table 107. Methoxyfenozide residues on/in cabbage samples, with and without wrapper leaves (Carpenter, 1999d).

Location, year, report	Cabbage, head, with wrapper leaves	Cabbage, head, without wrapper leaves	Reduction factor
PA, USA, 1998, 34-99-76	1.0	<0.05	0.050
	0.86	<0.05	0.058
FL, USA, 1998, 34-99-76	2.2	0.25	0.11
	2.1	0.36	0.17
TX, USA, 1998, 34-99-76	2.7	0.075	0.028
	3.9	0.024	0.006
CA, USA, 1998, 34-99-76	0.64	0.20	0.31
	0.50	0.23	0.46
Average	1.7	0.15	0.09

Head lettuce

In some trials in the USA on head lettuce, samples with and without wrapper leaves were analyzed for residues of methoxyfenozide. The results showed that most of the residues remained in the outer leaves. Table 108 summarizes the results.

Table 108. Methoxyfenozide residues on/in head lettuce samples, with and without wrapper leaves (Carpenter, 1999e).

Location, year, report	Lettuce, head, with wrapper leaves	Lettuce, head, without wrapper leaves	Reduction factor
CA, USA, 1998, 34-99-75	6.2	0.13	0.021
	9.6	0.091	0.0095
FL, USA, 1998, 34-99-75	5.4	0.039	0.0072
	4.3	0.051	0.012
CA, USA, 1998, 34-99-75	7.0	0.059	0.0084
	5.5	0.14	0.025
Average	6.3	0.086	0.014

RESIDUES IN ANIMAL PRODUCTS

Farm animal feeding studies

Cows

A cow feeding study was conducted in 1997, to determine the extent to which methoxyfenozide residues transfer from animal feed commodities to the edible tissues and milk (Bender, 1998c, Report No. 34-98-95). Ten lactating dairy cows (Holstein) were divided into three groups and fed at different dose levels, with 3 cows each in the low- (T-1) and mid-level- (T-II) dose groups and 4 cows in the high- (T-III) dose group. One cow in T-III was maintained for a further 7 days after cessation of dosing. Three control animals were also included in the study. The amount of methoxyfenozide administered daily to each cow in the was as follows: T-I (15 ppm in feed) 415.4 mg; T-II (45 ppm in feed) 1246 mg; and T-III (150ppm) 4154 mg. The cows were dosed orally, via gelatin capsules, once daily after the morning milking, for 28 days.

The actual dose levels administered, on a feed concentration basis, were higher than the target levels. Actual dose levels, i.e., concentration of methoxyfenozide in the feed on a dry matter basis, were recalculated in 2001 from reported feed consumption of each cow (Bender, 2001, Report No. 34-01-61). In the T-I group, nominally dosed at 15 ppm, the actual concentrations of methoxyfenozide in the feed were 14.7-17.4 ppm; average 16.5 ppm. In the T-II group (45 ppm nominal dose), the actual concentrations were 47.1-64.1 ppm; averaging 53.6 ppm in the feed of animals from which milk samples were collected and 50 ppm for those from which tissue samples were collected. Actual dose levels for the T-III group (150 ppm nominal dose) were 157-196 ppm; averaging 183 ppm in animals from which milk samples were collected and 178 ppm in those from which tissues were collected.

Milk was collected twice daily and samples from each milking were pooled to form a composite daily sample. Samples were taken on days 1, 2, 4, 7, 10, 14, 17, 21, 24, 28 and 35 and

were frozen and maintained frozen until analysis. In addition, milk samples collected on day 28 from one control cow and the 4 high-dose cows (T-III), as well as milk samples collected on day 35 from one cow in the T-III group, were separated into skimmed milk and cream fractions. Skimmed milk and cream samples were also frozen after fractionation. Sampling to analysis intervals were: milk including skimmed milk, 60-113 days; cream, 175-182 days; fat, 22-81 days;, muscle, 107-161 days; liver and kidney, 174-231 days.

Food consumption, milk production and animal health was generally unaffected by the methoxyfenozide dose, with the exception of one cow in T-III group which developed toxic mastitis. Due to severe weight loss, this animal was removed from the study and replaced by another. Milk from days 21, 24 and 28, and tissues, from the sick animal were not analyzed. One other animal received trauma to the nose during dosing and a further animal developed mastitis, which did not require medical treatment although it led to reduced milk production.

Milk and skimmed milk samples were analyzed for residues of methoxyfenozide using method TR 34-96-183 (Bender, 1998c, Report No. 34-98-95, Appendix IVA) while cream samples were analyzed using method TR 34-96-116 (Bender, 1998c, Report No. 34-98-95, Appendix IVB). Analysis for methoxyfenozide residues in fat samples was by HPLC with UV detection (TR 34-96-116). Liver and kidney samples were extracted with methanol and partitioned into hexane. In liver and kidney, in addition to methoxyfenozide, the metabolite, RH-1518 (glucuronide conjugate of the A-ring phenol), was also determined. For the metabolite, RH-1518, the extracts were concentrated, purified on C-18 and carbon SPE columns. To determine methoxyfenozide residues, the extracts were partitioned using dichloromethane/water, followed by basic alumina and carbon SPE clean-up. Both compounds were determined by HPLC with MS detection (TR 34-98-67 (Bender, 1998c, Report No. 34-98-95, Appendix IVD). The LOQ for methoxyfenozide residues was 0.01 mg/kg for all matrices, with an LOD of 0.003 mg/kg, while the LOQ for the metabolite, RH-1518, was 0.02 with an LOD of 0.006 mg/kg in liver and kidney samples.

Residues of methoxyfenozide in whole milk are summarized in Table 109, while those in skimmed milk and cream are presented in Table 112. The residues in milk samples apparently reached a plateau by day 10. Residues in cream and skimmed milk at day 28 were 0.066-0.213 mg/kg and 0.0043–0.0072 mg/kg, respectively (Table 110). Seven days after cessation of dosing (day 35), residues in cream and skimmed milk (from one cow in T-III) were 0.0076 and <0.003 mg/kg, respectively. A low-level residue was detected in cream derived from the one untreated control cow (0.0047 mg/kg). Based on the average residues in whole milk, the concentration factor into cream was 4.3.

Residue data for edible tissues are shown in Table 111. Residues in all tissues were directly proportional to the dose rates of methoxyfenozide. Fat and liver contained the highest residue levels but did not exceed 0.3 mg/kg at the highest dose rate (T-III, 178 mg/kg dose group). Muscle samples showed no detectable residues in the T-I and T-II dose groups, while, in the T-III group, the residue was less than the LOQ (<0.01 mg/kg). Liver and kidney samples were analyzed for residues of methoxyfenozide and RH-1518. Liver samples contained detectable residues in all dose groups with average total residues of 0.02-0.3 mg/kg. Kidney samples showed residues in the T-II and T-III dose groups, with average total residues of 0.01-0.06 mg/kg in these groups.

Table 109. Summary of methoxyfenozide residues in whole milk (Bender, 1998c).

	Feeding level, ppm, nominal (actual)	Group	Day 1	Day 2	Day 4	Day 7	Day 10	Day 14	Day 17	Day 21	Day 24	Day 28 [1/]	Day 35
Mean [2/]	15 (16.5)	I	<0.003	<0.003	<0.003	<0.003	0.0038	0.0033	<0.003	0.0037	<0.003	0.0041	NA
Values			<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	
			<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	<0.003	0.005	<0.003	<0.003	
			<0.003	<0.003	<0.003	<0.003	0.0055	0.0039	<0.003	<0.003	<0.003	0.0063	

	Feeding level, ppm, nominal (actual)	Group	Day 1	Day 2	Day 4	Day 7	Day 10	Day 14	Day 17	Day 21	Day 24	Day 28 [1/]	Day 35
Mean [2/]	45 (53.6)	II	<0.003	0.0039	0.0036	0.0053	0.0033	0.0033	0.0034	0.0036	0.0039	0.0047	NA
Values			<0.003	0.0048	0.0046	0.0076	<0.003	0.0039	0.0043	0.0035	0.0038	0.0056	
			<0.003	0.0040	<0.003	0.0052	0.0038	<0.003	<0.003	0.0042	<0.003	<0.003	
			<0.003	<0.003	0.0032	<0.003	<0.003	<0.003	<0.003	<0.003	0.0050	0.0054	
Mean [2/]	150 (183)	III	0.0042	0.024	0.029	0.050	0.030	0.027	0.028	0.030	0.027	0.028	<0.003
Min.			<0.003	0.0100	0.0078	0.0162	0.0137	0.0138	0.0155	0.0132	0.0154	0.0146	
Max.			0.0105	0.0370	0.0484	0.0996	0.0464	0.0432	0.0408	0.0572	0.0428	0.0452	
Values			<0.003	0.0100	0.0078	0.0162	0.0137	0.0138	0.0155	0.0132	0.0154	0.0146	<0.003
			<0.003	0.0124	0.0144	0.0230	0.0187	0.0177	0.0223	0.0154	0.0176	0.0237	
			0.0032	0.0355	0.0460	0.0616	0.0395	0.0337	0.0338	0.0334	0.0337	0.0271	
			0.0105	0.0370	0.0484	0.0996	0.0464	0.0432	0.0408	0.0572	0.0428	0.0452	

LOQ = 0.01 mg/kg; LOD (limit of detection) = 0.003 mg/kg. Report assigned '0.000' to residues <LOD.
NA = not applicable; no samples taken.
[1/] End of dosing.
[2/] Mean value was calculated using LOD for values <0.003 mg/kg.

Table 110. Summary of methoxyfenozide residues in cream and skimmed milk from cows fed at the 150 ppm dose level (Bender, 1998c).

	Feeding level, ppm nominal (actual)	Group/cow	Study day	Residues, mg/kg		Concentration factor [1/]	
				Cream	Skimmed milk	Cream	Skimmed milk
Mean	150 (183)	III/13	28	0.12	0.0054	4.3	0.19
Value				0.0715	0.0046		
				0.2130	0.0072		
				0.1290	0.0054		
				0.0655	0.0043		
Mean		III/13	35 [2/]	0.0076	<0.003 (ND) [3/]	NA [4/]	NA [4/]

LOQ = 0.01 mg/kg; LOD = 0.003 mg/kg
[1/] Concentration factor from whole milk, based on average residue of 0.028 mg/kg in Group III whole milk on day 28.
[2/] Single sample, 7 days after dosing ceased.
[3/] ND = non-detectable, <LOD of 0.003 mg/kg.
[4/] NA = not applicable; no residues in whole milk on day 35.

Table 111. Residues of methoxyfenozide and its metabolite RH-1518 in cattle tissues (Bender, 1998c).

	Dose nom. (act.)	Group	Fat	Muscle	Liver			Kidney		
			methoxy-fenozide, mg/kg	methoxy-fenozide, mg/kg	methoxy-fenozide, mg/kg	RH-1518 [5/] mg/kg	total [1/, 4/] mg/kg	methoxy-fenozide, mg/kg	RH-1518 [5/] mg/kg	total [1/, 4/] mg/kg
Mean [2/,3/]	15 (16.5)	I	0.0082	<0.003 (ND)	0.0075	0.010	0.018	<0.003 (ND)	<0.004 (ND)	<0.007 (ND)
Values			<0.003	<0.003	0.0062	0.0140	0.0202	<0.003	<0.004	<0.007
			0.0106	<0.003	0.0069	0.0032	0.0101	<0.003	<0.004	<0.007
			0.0109	<0.003	0.0094	0.0123	0.0217	<0.003	<0.004	<0.007
Mean [2/,3/]	45 (54)	II	0.041	<0.003 (ND)	0.028	0.026	0.055	0.0032	0.0075	0.011
Values			0.0183	<0.003	0.0241	0.0218	0.0459	<0.003	0.0084	0.0114
			0.0231	<0.003	0.0301	0.0347	0.0648	<0.003	0.0084	0.0114
			0.0820	<0.003	0.0305	0.0230	0.0535	0.0038	0.0056	0.0094
Mean [2/,3/]	150 (183)	III	0.28	0.0073	0.13	0.10	0.23	0.026	0.029	0.055
Values			0.1560	<0.003	0.0928	0.1176	0.2104	0.0210	0.0190	0.0400
			0.2600	0.0103	0.1450	0.1120	0.2570	0.0251	0.0465	0.0716
			0.4400	0.0085	0.1510	0.0784	0.2294	0.0336	0.0218	0.0554

LOQ for methoxyfenozide = 0.01 mg/kg; LOD = 0.003 mg/kg.
LOQ for RH-1518 = 0.02 mg/kg (0.013 mg/kg as methoxyfenozide equivalent); LOD = 0.006 mg/kg (0.004 mg/kg as methoxyfenozide equivalent).
[1/] Total residues = methoxyfenozide + RH-1518, using LOD in the calculation for values <LOD.
[2/] ND = non-detectable, <LOD, indicated as "0" in study.
[3/] Means for individual tissues were calculated using LOD for the ND.
[4/] Mean value for total residue (methoxyfenozide + RH-1518) was calculated using LOD of 0.006 mg/kg for ND.
[5/] As methoxyfenozide equivalents, i.e. RH-1518 x 368/530.

Poultry

A hen feeding study was conducted in the USA in 1999, to determine the extent to which residues of methoxyfenozide transfer from animal feed commodities to edible tissues and eggs of poultry (Bender, 2000c, Report no. 34-00-33). Forty-two hens (White Leghorn) were used in the study and were divided into four groups, by dosing level. The T-I group consisted of 10 chickens dosed at 2 ppm in the feed; the T-II group of 10 chickens was dosed at 6 ppm in the feed; and the T-III group, consisting of 12 chickens, was dosed at 20 ppm in the feed. A control group of another 10 chickens were dosed with placebo for 18 consecutive days. The control, T-I and T-II groups were further divided into 3 sub-groups consisting of 3, 3 and 4 chickens. The T-III group was divided into 3 sub-groups of 3, 3 and 6 chickens. At the end of the dosing period, all chickens in the control, T-I and T-II, together with three chickens in each of the T-III treatment sub-groups, were sacrificed. The remaining three chickens in the T-III group were depurated for 7 days, prior to sacrifice.

Actual dose levels administered, based on feed consumption during the study, were higher than the target dose levels. Actual dose levels in the T-I group (2 ppm, nominal dose) were 2.3-2.5 ppm; average 2.37 ppm. In the T-II group, the actual feed concentration (6 ppm nominal dose) was 7.4-7.7 ppm; average 7.58 ppm. The actual dose level in the T-III group (20 ppm nominal dose) was 23.2-23.7 ppm; average 23.49 ppm.

Egg samples were collected twice daily, prior to initiation of dosing and on days 1, 3, 7, 10, 14, 17, 21, 24, 28 and 35. The egg samples were composited by sub-group and treatment level, in order to obtain sufficient sample size, and frozen before analysis. Within 24 hours of administration of the final dose, 39 hens were sacrificed. The remaining 3 hens from the T III group were given a 7-day recovery period, during which they consumed only untreated food, and were then sacrificed. Samples of liver, fat and muscle were taken from each chicken at sacrifice and composited by sub-group. The following samples were collected from each bird, if available: liver, entire organ; fat, approximately 30 g of subcutaneous and abdominal fat combined; muscle, approximately 50 g of breast and 50 g of thigh muscle. The breast and thigh muscle tissues were combined to give a single muscle sample for each sub-group. All samples were frozen immediately and maintained frozen until analysis. Sampling to analysis intervals were: liver, 12-70 days; fat and muscle, 27-43 days; and eggs, 61-80 days.

Samples were analyzed using Method 34-99-11. Samples of all matrices were analyzed for residues of methoxyfenozide; liver and egg samples were also analyzed for the major metabolite RH-1518. Fat samples (combined subcutaneous and abdominal fat) were extracted with hexane and the residues partitioned into acidic methanol. Muscle samples (combined thigh and breast muscle) were extracted with acidic methanol, the residues partitioned into dichloromethane and cleaned up using basic alumina column chromatography and carbon solid-phase extraction. Liver and egg matrices were extracted with methanol, initially cleaned-up by partition with hexane and the methanolic extract was divided into two portions. One portion was purified by partition into dichloromethane, followed by basic alumina column chromatography and carbon solid-phase extraction, and analyzed to determine methoxyfenozide. The other portion was purified by C-18 solid phase extraction and analyzed to determine RH-1518. Analysis was carried out by HPLC with MS detection. Confirmatory analysis was carried out by MS/MS. The method LOQ for both methoxyfenozide and RH-1518 in all matrices was 0.01 mg/kg. The LOD was 0.003 mg/kg. Recoveries of methoxyfenozide from eggs were 62-93% (mean 82%), from fat were 85-98% (mean 92%), from muscle were 83-96% (mean 88%) and from liver were 89-95% (mean 92%). Recoveries of RH-1518 from eggs were 55-110% (mean 88%) and from liver were 73-120% (mean 98%).

Although residues of methoxyfenozide in animal commodities have been shown to be stable under conditions of frozen storage, residues of RH-1518 in beef liver may decline up to 30% when stored frozen for 7 months. All treated liver samples were analyzed within 21 days of collection. Fat and muscle samples were analyzed within 43 days.

The residue data for eggs are summarized in Table 112. No detectable residues of either the parent or the metabolite, RH-1518, were found in eggs at the T-1 dose level. The highest residue (parent + RH-1518) detected in the T-II dose level was ≤0.005 mg/kg, which was less than the LOQ of 0.01 mg/kg. At the T-III dose level, the highest residue (parent + RH-1518) was 0.008 mg/kg, or approximately the LOQ (0.01 mg/kg) At the same dose level, the highest residue of methoxyfenozide was 0.004 mg/kg, near the limit of detection (0.003 mg/kg).

Table 112. Residues in eggs from chickens dosed for 28 days with methoxyfenozide (Bender, 2000c).

Nominal (actual) feeding level, ppm	Group	Days of dosing		Methoxyfenozide, mg/kg	RH-1518 [1/], mg/kg	Total residue, mg/kg
2	T-I	1	Mean [2/]	<0.003 [3/]	<0.002	<0.005
(2.4)			Values	<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
		3	Mean	<0.003	<0.002	<0.005
			Values	<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
		7	Mean	<0.003	<0.002	<0.005
			Values	<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
6	T-II	1	Mean	0.003	<0.002	<0.005
(7.6)			Values	<0.003	<0.002	<0.005
				0.0032	<0.002	<0.0032
				<0.003	<0.002	<0.005
		3	Mean	<0.003	<0.002	<0.005
			Values	<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
		7	Mean	<0.003	<0.002	<0.005
			Values	<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
20	T-III	1	Mean	<0.003	<0.002	<0.005
(23.5)			Values	<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
		3	Mean	<0.003	<0.002	<0.005
			Values	<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
				<0.003	<0.002	<0.005
		7	Mean	<0.003	0.0028	0.0058
			Values	<0.003	0.0031	0.0061
				<0.003	0.0026	0.0056
				<0.003	0.0026	0.0056
		10	Mean	0.0038	0.0040	0.0078
			Values	0.0054	0.0033	0.0087
				<0.003	0.0038	0.0068
				<0.003	0.0049	0.0079
		14	Mean	<0.003	0.0036	0.0066
			Values	<0.003	0.0037	0.0067
				<0.003	0.0035	0.0065
				<0.003	0.0037	0.0067

Nominal (actual) feeding level, ppm	Group	Days of dosing		Methoxyfenozide, mg/kg	RH-1518 [1/], mg/kg	Total residue, mg/kg
20 (23.5)	T III	17	Mean	0.0030	0.0037	0.0068
			Values	<0.003	0.0046	0.0076
				0.0030	0.0021	0.0051
				<0.003	0.0048	0.0078
		21	Mean	<0.003	0.0053	0.0068
			Value	<0.003	0.0037	0.0067
				<0.003	0.0033	0.0063
				<0.003	0.0042	0.0072
		24	Mean	<0.003	0.0027 (<0.003)	0.0057 (<0.006)
			Values	<0.003	<0.003	<0.006
				<0.003	0.0027	0.0057
				<0.003	0.0024	0.0054
		28	Mean	<0.003	0.0040	0.0070
			Values	<0.003	0.0050	0.0080
				<0.003	0.0041	0.0071
				<0.003	0.0029	0.0059

LOQ for methoxyfenozide = 0.01 mg/kg; LOD (limit of detection) = 0.003 mg/kg.
LOQ for RH-1518 = 0.01 mg/kg; LOD = 0.003 mg/kg (0.002 mg/kg as methoxyfenozide equivalents).
[1/] As methoxyfenozide equivalents, i.e. RH-1518 x 368/530.
[2/] Means for individual tissues were calculated using LOD for the ND.
[3/] ND = non-detectable, <LOD, indicated as "0" in study.

In the feeding study, residues of methoxyfenozide were non-detectable in the fat and muscle tissues of all feeding groups. In liver, the combined residue of methoxyfenozide and metabolite RH-1518 was 0.03 mg/kg, at the highest feeding level, although parent methoxyfenozide was <0.003 mg/kg. Residues of RH-1518 (as methoxyfenozide equivalents) in T-I group livers averaged: <0.01 mg/kg (0.0023–0.0049 mg/kg); 0.016 mg/kg (<0.006-0.0418 mg/kg) in T-II group; and 0.0306 mg/kg (0.0211-0.0418 mg/kg) in the T-III group. The total average residues of parent + RH-1518 the liver were <0.01 mg/kg at the T-I feeding level, 0.015 mg/kg at the T-II feeding level, and 0.024 mg/kg at the T-III feeding level. Seven days after cessation of treatment (T-III group), no residues of RH-1518 were detected in liver. The results for poultry tissues are summarized in Table 113.

Table 113. Residues in tissues of chickens dosed with methoxyfenozide for 28 days (Bender, 2000c).

Nominal (actual) feeding level, ppm	Group	Dosing day		Fat	Muscle	Liver		
				Methoxy-fenozide mg/kg	Methoxy-fenozide mg/kg	Methoxy-fenozide mg/kg	RH-1518 [4/] mg/kg	Total residue [1/, 3/] mg/kg
2 (2.4)	T-I	28	Mean [2/]	<0.003 (ND)	<0.003 (ND)	<0.003 (ND)	<0.004	<0.00
			Min	<0.003	<0.003	<0.003	<0.004	<0.007
			Max	<0.003	<0.003	<0.003	<0.004	<0.007
6 (7.6)	T-II	28	Mean [2]	<0.003 (ND)	<0.003 (ND)	<0.003 (ND)	0.0121	0.0151
			Min	<0.003	<0.003	<0.003	<0.004 (ND)	<0.007 (ND)
			Max	<0.003	<0.003	<0.003	0.029	0.0321
20 (23.5)	T-III	28	Mean [2]	<0.003 (ND)	<0.003 (ND)	<0.003 (ND)	0.0213	0.0243
			Min	<0.003	<0.003	<0.003	0.0147	0.0177
			Max	<0.003	<0.003	<0.003	0.0300	0.0330
	T-III	7-day withdrawal		<0.003 (ND)	<0.003 (ND)	<0.003 (ND)	ND	ND

LOQ for methoxyfenozide = 0.01 mg/kg; LOD = 0.003 mg/kg.
LOQ for RH-1518 = 0.02 mg/kg (0.014 mg/kg as methoxyfenozide equivalents); LOD = 0.006 mg/kg (0.004 mg/kg as methoxyfenozide equivalents).
ND = non-detectable; <LOD, indicated as "0" in study.
[1/] Total residues = methoxyfenozide + RH-1518, using ½ LOD in calculation.
[2/] Means for individual tissues were calculated using LOD for ND values.
[3/] Mean value for total residue (methoxyfenozide + RH-1518) was calculated using LOD of 0.006 mg/kg for ND, except when all values for the group were ND.
[4/] As methoxyfenozide equivalents, i.e. RH-1518 x 368/530.

RESIDUES IN FOOD IN COMMERCE AND AT CONSUMPTION

The manufacturer reported that there were no monitoring data available on residues of methoxyfenozide in food in international trade.

NATIONAL RESIDUE LIMITS

Methoxyfenozide is a relatively new pesticide but some MRLs have already been established by national regulatory authorities. Table 114 provides information on existing national MRLs, based primarily on information provided by the manufacturer and supplemented by information from the governments of Australia and the Netherlands.

Table 114. National MRLs.

Country	Commodity	MRL (mg/kg)
Argentina	Soya beans	0.01 grain
	Cotton (seed, oil)	0.01
	Tomatoes (greenhouse)	0.2
	Apples/Pears	0.5
	Peaches	0.5
Australia	Cotton seed	3
	Tomatoes	3
	Edible offal	0.01
	Meat	0.01
	Milk	0.01
Brazil	Soya beans	0.05
	Cotton	0.5
	Corn	0.5
	Tomatoes	0.1
	Apple, Pear	0.5
	Citrus	0.05
	Coffee	0.01
	Potatoes	0.05
Canada	Pome fruit	1.5
Hungary	Grapes (wine)	1
	Apples and pears	0.3
Ireland	Apples and pears	0.3
Israel	Tomatoes and peppers	0.5
Japan	Rice	0.1
	Tea	5.0
	Chinese cabbages	1.0
	Apples	2.0
Mexico	Corn	0.01
	Cotton	0.05
UK	Apples/pears	0.3
	Grapes	1.0
USA (definition: parent only) [1/]	Almond hulls	25
	Apple pomace, wet	7
	Artichokes, globe	3
	Brassica, head and stem group	7
	Brassica, leafy sub-group	30
	Cattle fat	0.5
	Cattle meat	0.02
	Corn, field, forage	15
	Corn, field, grain	0.05
	Corn, field, refined oil	0.20
	Corn, field, fodder	125
	Corn, sweet. forage	30
	Corn, sweet K+KWHR	0.05
	Corn, sweet, fodder	60
	Cotton seed	2
	Cotton gin trash	35
	Fruit, pome group	1.5
	Fruit, stone group, except fresh plums	3
	Goat, fat	0.5

Country	Commodity	MRL (mg/kg)
USA (definition: parent only) [1/]	Goat, meat	0.02
	Grain, aspirated fractions`	2
	Grapes	1
	Grapes, raisins	1.5
	Hog, fat	0.1
	Hog, meat	0.02
	Horse, meat	0.02
	Horse, fat	0.5
	Leaf petioles sub-group	25
	Leafy greens sub-group	30
	Longans	2
	Lychees	2
	Milk	0.1
	Nut, tree group	0.1
	Pistachios	0.1
	Plums, fresh	0.3
	Poultry, fat	0.02
	Poultry, meat	0.02
	Rambutans	2
	Sheep, fat	0.5
	Sheep, meat	0.02
	Spanish limes	2
	Vegetable, fruiting group	2
USA (definition: parent + glucuronide metabolite) [1/]	Cattle, liver	0.4
	Cattle, meat by-product, except liver	0.1
	Eggs	0.02
	Goat, liver	0.4
	Goat, meat by-product, except liver	0.1
	Hog, liver	0.1
	Hog, meat by-product, except liver	0.02
	Horse, liver	0.4
	Horse, meat by-product, except liver	0.1
	Poultry, liver	0.1
	Poultry, meat by-product, except liver	0.02
	Sheep, liver	0.4
	Sheep, meat by-product, except liver	0.1

[1/] 40 CFR 180.544.

APPRAISAL

Methoxyfenozide, *N-tert*-butyl-*N'*-(3-methoxy-*o*-toluoyl)-3,5-xylohydrazide or 3-methoxy-2-methylbenzoic acid 2-(3,5-dimethylbenzoyl)-2-(1,1-dimethylethyl)hydrazide, is a substituted dibenzohydrazide and an insecticide that functions by accelerating the moulting process. It was considered for the first time by the present Meeting.

Animal metabolism

The Meeting received information of the metabolism of methoxyfenozide in rats, goats, and hens.

Goats. The metabolism, distribution, and elimination of [^{14}C]methoxyfenozide, labelled in the methoxyphenyl (A) ring, the dimethylphenyl (B) ring, or the *tert*-butyl group, were studied in lactating dairy goats. The methoxyfenozide was administered orally in gelatin capsules to lactating goats once a day at dietary equivalents of 45, 32, or 61 ppm, for 7 consecutive days. Over the treatment period, 81-88% of the administered dose was eliminated in the faeces (74-84%) and urine (5-7%). The major accumulation was in liver, where up to 0.14% of the total dose was found. The total radioactive residue (TRR) was <0.010-0.037 mg/kg in milk, 0.26-1.2 mg/kg in liver, 0.045-0.20 mg/kg in kidney, <0.010-0.017 mg/kg in leg muscle, <0.010-0.023 mg/kg in loin muscle, and 0.018-0.053 mg/kg in fat, expressed as methoxyfenozide. In general, residues were highest in the *tert*-butyl-labelled samples and lowest in dimethylphenyl-labelled samples.

About 85% of the TRR was extracted from milk and 28-72% of the TRR was identified. Some 98% was extracted from liver, 87-94% from kidney, and 68-98% from muscle, while the

corresponding proportions of TRR identified were 22-68% from muscle, 68-84% from fat, 44-76% from kidney, and 40-56% from liver. An additional 50% was characterized, in kidney and liver extracts from the *tert*-butyl labelled residue, as lactose and triglycerides (*see* below).

The milk from the last day of dosing contained methoxyfenozide as the major component (14%-35% of the TRR), the only significant metabolite(s) being a B-ring alcohol-carboxylic acid and/or an A-ring phenol-B-ring alcohol (<10% of the TRR).

Methoxyfenozide was the major component of the radioactive residue in fat (68-81% of the TRR) and muscle (20%) and constituted 2-3% of the TRR in liver and kidney. The major component of the radioactive residue in liver (23-30% of the TRR, 0.075-0.27 mg/kg equivalents) and kidney (25-42% of the TRR, 0.015-0.049 mg/kg) was the glucuronide conjugate of the A-ring phenol, formed by the demethylation of the methoxy group of the parent compound. It was present at low levels in the other samples (0.54%-8.1% of the TRR, <0.001-0.004 mg/kg).

The other metabolites identified in milk and most tissues, from various labels at low levels, were the A-ring phenol or demethylated parent, 0.72-7.4% of the TRR, <0.001-0.069 mg/kg; the B-ring carboxylic acid, 0.25-4.2% of the TRR, <0.001-0.013 mg/kg; the glucuronide conjugate of the A-ring phenol with an additional OH group *ortho* or *para* to the glucuronide moiety, 0.32-18.2% of the TRR, <0.001-0.024 mg/kg; and the A-ring phenol glucuronide, B-ring monoalcohol, 0.16-14.1% of the TRR, <0.001-0.12 mg/kg.

The metabolite profiles were broadly similar from the three labels, with radioactivity from the *tert*-butyl group prominent in the fat-soluble fraction. The hexane extracts of *tert*-butyl-labelled liver and kidney were subjected to hydrolysis and additional analytical procedures demonstrated the incorporation of radioactivity into triglycerides. From the results of HPLC, TLC, LC-MS, and LC-MS/MS analyses, triglyceride structures were proposed for several major components of the residue. Triglycerides accounted for 18% of the TRR (0.21 mg/kg as methoxyfenozide) and 22% of the TRR (0.043 mg/kg) in *tert*-butyl-labelled liver and kidney, respectively. The incorporation of radioactivity into lactose in *tert*-butyl-labelled milk (23-31% of the TRR, 0.007-0.011 mg/kg) was also demonstrated.

Hens. After oral doses of [methoxyphenyl-^{14}C]methoxyfenozide (A-ring), [dimethylphenyl-^{14}C]methoxyfenozide (B-ring), or [*tert*-butyl-^{14}C]methoxyfenozide to laying hens for 7 consecutive days at the equivalent of 58-68 ppm in the diet, the TRRs were 0.005-0.10 mg/kg in eggs, 0.28-1.57 mg/kg in liver, 0.009-0.027 mg/kg in dark muscle, 0.007-0.014 mg/kg in light muscle, 0.042-0.072 mg/kg in fat, and 0.042-0.052 mg/kg in skin with fat. Total recovery of the administered dose was 84-93%. In general, ^{14}C residues were highest in *tert*-butyl-labelled samples and lowest in dimethylphenyl-labelled samples, as in the ruminant study.

Approximately 81-98% of the TRR was characterized or identified in eggs and tissues from all labels, except *tert*-butyl-labelled light muscle (TRR = 0.014 mg/kg, 48% characterized or identified). Methoxyfenozide was identified in eggs and tissues from treatments with all labels, except the dimethylphenyl label in liver. It was the major residue component in methoxyphenyl- and dimethylphenyl-labelled dark muscle, fat, and skin with fat, and in *tert*-butyl-labelled fat and skin with fat (11-55% of the TRR, 0.001-0.032 mg/kg), and was a minor residue in eggs, methoxyphenyl- and *tert*-butyl-labelled liver, and *tert*-butyl-labelled dark and light muscle (0.26-8.1% of the TRR, <0.001-0.006 mg/kg). The glucuronide conjugate of the A-ring phenol was identified in eggs and tissues from treatments with all labels, except methoxyphenyl- and dimethylphenyl-labelled dark muscle. It was a major metabolite in eggs, methoxyphenyl- and dimethylphenyl-labelled liver, and *tert*-butyl-labelled light muscle and skin with fat (10-30% of the TRR, 0.001-0.054 mg/kg), and was present at low levels in the other samples (1.8-9.7% of the TRR, 0.001-0.007 mg/kg). The A-ring phenol or demethylated parent was identified in eggs and tissues with all labels at levels of 1.5-11% of the TRR (<0.001-0.044 mg/kg). Two other metabolites were identified at significant levels: the glucuronide conjugate of the A-ring phenol with an additional -OH group *ortho* or *para* to the glucuronide moiety (1.1-9.5% of the TRR, <0.001-0.009 mg/kg), and the A-ring phenol glucuronide-B-ring monoalcohol (2.2-28% of the TRR, <0.001-0.047 mg/kg). The B-ring carboxylic acid was also identified at <6% of the TRR in eggs and various tissues.

As in goats, the metabolite profiles were broadly similar from the three labels and the incorporation of radioactivity into triglycerides in *tert*-butyl-labelled liver (≤56% of the TRR, ≤0.89 mg/kg) and kidney (≤32% of the TRR, ≤0.18 mg/kg) was demonstrated.

The Meeting concluded that the metabolism of methoxyfenozide is similar in poultry and ruminants. The major residue component in muscle, fat, and milk was the parent, which was present at low levels (<5% of the TRR) in eggs, liver, and kidney. The main metabolite in eggs, liver, and kidney was the glucuronide conjugate of the A-ring phenol. The residues were concentrated in the fat relative to the muscle by factors of about 2-5. This is consistent with the log P_{ow} for methoxyfenozide of 3.7, which suggests slightly greater solubility in fat than muscle.

The metabolites found in rats were qualitatively the same as those in goats and hens.

Plant metabolism

Studies on cotton, apples, grapes and rice were reported.

Cotton. The total radioactive residues were 0.072, 0.054, and 0.057 mg/kg in hulled kernels and 0.089, 0.107, and 0.162 mg/kg in hulls and lint 21 days after two applications each of A-ring-, *tert*-butyl- and B-ring-labelled [^{14}C]methoxyfenozide at 1 kg ai/ha. The TRRs in whole cotton seed calculated from the total weight of kernels, hulls and lint were 0.081 mg/kg (methoxyphenyl label), 0.080 mg/kg (*tert*-butyl label), and 0.11 mg/kg (dimethylphenyl label). In whole cotton plants, the TRR decreased from 72 mg/kg (methoxyphenyl label), 60 mg/kg (*tert*-butyl label) and 86 mg/kg (dimethylphenyl label) in immature plants harvested after 7 days to 17 mg/kg (methoxyphenyl label), 13 mg/kg (*tert*-butyl label), and 17 mg/kg (dimethylphenyl label) in mature plants harvested after 21 days.

Solvent extractions released ≥75% of the TRR, and about 65-86% of the TRR was characterized or identified in whole cotton seed. Methoxyfenozide was the only residue component identified, accounting for 46% of the TRR (0.038 mg/kg) in methoxyphenyl-labelled, 67% (0.054 mg/kg) in *tert*-butyl labelled, and 57% (0.063 mg/kg) in dimethylphenyl-labelled whole cotton seed.

Apples. The total radioactive residues were 0.23 and 0.28 mg/kg in or on apples collected 14 and 36 days (normal harvest) after two applications of [methoxyphenyl-^{14}C]methoxyfenozide at 1 kg ai/ha.

Over 93% of the TRR was characterized or identified in apples. Methoxyfenozide was the major component identified, accounting for 91% of the TRR (0.26-0.27 mg/kg) in apples collected after 14 and 36 days. Two metabolites, identified in 14- and 36-day apples, were the B-ring monoalcohol at 1.4% of the TRR (0.004 mg/kg) and the B-ring dialcohol at 0.08% of the TRR and 0.11% of the TRR (both 0.003 mg/kg). The half-life of methoxyfenozide on apple foliage and fruit was estimated at 23 ± 8 days and 12 ± 9 days, respectively.

Grapes. The total radioactive residues were 0.75 mg/kg in grapes collected 27 days (normal harvest) after two applications of [*tert*-butyl-^{14}C]methoxyfenozide at 1 kg ai/ha. The TRR was 110 mg/kg in grape foliage at harvest.

Approximately 93% of the TRR was characterized or identified. Methoxyfenozide was the major residue component identified, accounting for 81% of the TRR (0.60 mg/kg). Two metabolites identified were the B-ring monoalcohol at <2.3% of the TRR (<0.017 mg/kg) and the glucose conjugate of the A-ring phenol at 3.6% of the TRR (0.027 mg/kg).

The TRRs in grapes and grape foliage sampled between the first and second applications and after the second application to harvest and beyond (for foliage) were monitored. The half-lives of methoxyfenozide were determined to be 13-21 days on grapes and 11-26 days on foliage.

Rice. Radiolabelled methoxyfenozide (A-ring, B-ring, and *tert*-butyl) was applied to rice 70 and 107 days after planting. The B-ring- and *tert*-butyl-labelled compounds were applied at 0.6 kg ai/ha in both applications. The A-ring material was applied first at 0.62 kg ai/ha, then at 0.31 kg ai/ha. Samples of grain (panicles) and foliage were collected 62 days after the second treatment. The panicles were separated into chaff and brown rice. The radioactive residue in plants ranged from 6.6 to 10 mg/kg, in grain 0.52 to 0.71 mg/kg, and in straw 21-44 mg/kg.

Solvent extraction released 88-91% of the TRR from rice straw. The major component was methoxyfenozide, 65-69% of the TRR. Identified metabolites were the B-ring-monoalcohol (0.9-1.4% of the TRR), the A-ring phenol or demethylated methoxyfenozide (2.7-2.9% of the TRR), the A-ring phenol B-ring monoalcohol (2.1-2.3%), the B-ring carboxylic acid (1.2-1.6%) and the glucose conjugate of the A-ring phenol (1.5-2.4%). A total of 75-78% of the TRR was identified.

The main component of the residue in the grain was again methoxyfenozide, 52-59% of the TRR. Identified metabolites were the B-ring monoalcohol (1.1-4.1% of the TRR), the A-ring phenol (3.2-7.5%), the B-ring carboxylic acid (1.6-2.9%), the B-ring dialcohol (0.4-0.7%), and the glucose conjugate of the A-ring alcohol (1.8-2.3%).

Summary. The Meeting concluded that the metabolism studies on apples, rice, grapes and cotton adequately elucidated the nature of residues arising from foliar application of methoxyfenozide to various types of crop. The major component of the residue is methoxyfenozide, typically 50-90%. The metabolism of methoxyfenozide in plants is slow but occurs via the same pathways as in animals. The primary routes of metabolism involve demethylation of the A-ring methoxy group, to produce a phenol which is then conjugated with sugars, and the oxidation of the methyl groups on the B-ring, to produce alcohols, acids or both. B-ring alcohols and acids also form glucose conjugates.

Environmental fate in soil

The aerobic degradation of radiolabelled methoxyfenozide was studied in four soils at an application rate of 0.75 kg ai/ha over a one-year period. The results demonstrated that methoxyfenozide is very persistent in soil, with 59-75% of the applied dose remaining after one year. Calculated first-order half-lives were 340-1100 days, depending on the soil. The major degradation pathway of methoxyfenozide in soil leads to incorporation into soil natural products, mainly humic and fulvic acids and to a lesser extent humins. Degradation also proceeds by oxidation of a methyl substituent of the B-ring to the acid (up to 3.2% of the applied dose), followed by mineralization to carbon dioxide (5.5% of the applied dose). No other degradation product exceeding 2% of the applied radioactivity was identified. Total recoveries during the test period were 90-123%.

Confined rotational crop studies were conducted with methoxyfenozide labelled in the A-ring, B-ring, and *tert*-butyl group. Three applications of each, formulated as an emulsifiable concentrate, were made to bare soil at a total application rate of 2.2 kg ai/ha, equivalent to the maximum registered rate in the USA. Mustard, white radish and wheat were planted in the treated soil at three different plant-back intervals, 30, 90 and 365 days (nominal) after the last application. Crops were harvested at an intermediate stage and at maturity.

Total radioactive residue levels were <0.05-0.3 mg/kg in all crops, except wheat forage and straw at 1-3 mg/kg. Residue levels were similar in immature and mature crops harvested from the same plant-back intervals, except in wheat. In general, the total residues found in mature and immature crop samples decreased significantly with increasing plant-back time. The highest TRRs, averaging about 3 mg/kg, were found in wheat straw samples at 30 days plant-back. Wheat forage residues averaged only about one-third those found in wheat straw. Wheat grain residues, at <0.05 mg/kg, were the lowest in any of the crops investigated. In general, most of the residue in all crops was readily extractable, but wheat straw and grain contained a large amount of bound radiolabel.

In mustard leaves at 30 days plant-back, the parent methoxyfenozide was present up to 0.027 mg/kg (about 21% of the TRR) and individual metabolites were all below 0.01 mg/kg.

In radish leaves at 30 days plant-back, individual residue components (up to 21 components were characterized) were all <0.05 mg/kg. The parent compound was found at up to 0.013 mg/kg (about 18% of the TRR). The *N*-glycosyl conjugate of the B-ring monoalcohol was detected at up to 0.035 mg/kg (about 13%) while the glucose conjugate of the A-ring phenol was detected at up to 0.028 mg/kg, (12~13% of the TRR). Radish roots contained the highest levels of unmetabolized parent of any of the crops investigated. Methoxyfenozide was the main residue component in mature roots of radishes planted 30 days after treatment, 0.022-0.033 mg/kg (up to 41% of the TRR).

Residues in wheat forage ranged from 0.72 to 1.5 mg/kg at a 30-day plant-back. The parent compound was a very minor component, less than 1% of the TRR (maximum 0.009 mg/kg). The two major components of the extracted residue were the malonylglycosyl conjugate of the A-ring phenol (up to 0.70 mg/kg, 48% of the TRR) and the glucose conjugate of the A-ring phenol (up to 0.36 mg/kg, 24% of the TRR).

Wheat straw contained the highest residues of any crop, 2-4 mg/kg at 30 days plant-back. Only about 45-50% of the straw residue was extractable. The main residue in the extract was the A-ring phenol (demethylated methoxyfenozide), which was present at up to 1.4 mg/kg and up to about 37% of the TRR.

Wheat grain contained the lowest residue levels, about 0.05 mg/kg. Only 11-23% of the grain residue was extractable. The main component of the extractable residue was the A-ring phenol, at a concentration of 0.006 mg/kg (about 15% of the TRR).

Potentially quantifiable residues of methoxyfenozide transformation products were found in wheat forage and straw at a 365-day plant-back interval. These included the A-ring phenol and its glucose and malonylglycosyl conjugates.

Residues of methoxyfenozide were determined in rotational crops at two trial locations (Texas and California) in the USA. Leaf lettuce, used as the cover crop, was planted in plots at each location for subsequent planting of rotational crops. Five applications of methoxyfenozide 80W were made at 7-10 days intervals to the lettuce crop at 0.45 kg ai/ha per application with a season total of 2.2 kg ai/ha. The leaf lettuce cover crop was harvested and removed from the plot 1-3 days after the last application. Rotational crops representative of leafy crops (mustard greens), fruiting vegetables (tomatoes), cucurbits (cucumbers), root vegetables (turnips), cereal grains (wheat), legumes (soya beans), and bulb crops (onions) were planted 6-7 days after the last application.

The methoxyfenozide contents of the rotational crops harvested at maturity were mustard greens 0.12 mg/kg, turnip tops 0.004 mg/kg, turnip roots 0.021 mg/kg, onions 0.055 mg/kg, wheat hay 0.031 mg/kg, wheat straw 0.057 mg/kg, soya beans 0.02 mg/kg, soya forage 1.2 mg/kg, soya hay 1.1 mg/kg, tomatoes <0.02 mg/kg, wheat grain <0.02 mg/kg. The wheat and soya samples were also analyzed for the A-ring phenol and its glucose conjugate. Neither was detected in wheat grain or soya beans (<0.05 mg/kg) but both were found in the animal feed commodities, at levels as high as 4.1 mg/kg of the conjugate and 2.2 mg/kg of the phenol.

The Meeting concluded that methoxyfenozide persists in the soil. The major pathways of transformation are incorporation into soil natural products and slow oxidation of a methyl substituent of the B-ring to form the acid. The Meeting also concluded that methoxyfenozide and/or its degradation products may accumulate in rotational crops, particularly in forages and fodders.

Environmental fate in water

The hydrolytic stability of methoxyfenozide was evaluated in sterile buffer solutions at pH 5, 7, and 9. The concentration of combined unlabelled and *tert*-butyl-labelled material was 1 µg/ml. Samples were incubated in the dark at 25°C for 30 days. Methoxyfenozide was stable for the 30-day test period. The calculated half-life values were 600 days at pH 5, 1600 days at pH 7 and 700 days at pH 9.

Methods of analysis

Numerous methods were presented for the determination of methoxyfenozide in plant commodities, both for data collection in field trials and processing studies and for monitoring and enforcement of national MRLs. Enforcement methods included independent laboratory validations. Methods that determine both methoxyfenozide and the glucuronide conjugate of the A-ring phenol were reported for meat, milk, fat, poultry, and eggs.

In all methods, an extraction suitable for the sample was followed by a standard clean-up, such as solid-phase extraction and Florisil chromatography, and analysis by HPLC. Detection was by UV (240 nm), MS (*m/z* 367.3) or MS/MS (*m/z* 369 → 149). HPLC-MS and HPLC-MS/MS were

used for metabolites. The methods typically had limits of quantification of 0.01-0.05 mg/kg for methoxyfenozide in plant commodities, 0.01 mg/kg for methoxyfenozide in milk, eggs, and meat, and 0.01-0.02 mg/kg for the glucuronide conjugate of the A-ring phenol.

Enforcement methods based on the above procedures were developed with HPLC and UV detection as the primary analytical method for many plant commodities (apples, pears, grapes, peppers, tomatoes, leafy vegetables, brassica vegetables, pecans, almonds, almond hulls, sweet corn, corn forage and fodder, cotton seed, cotton gin trash) and some animal commodities (milk, fat, bovine muscle, chicken liver, eggs) and HPLC-MS and/or HPLC-MS/MS as the confirmatory method and the primary method for the glucuronide conjugate of the A-ring phenol in animal commodities and for methoxyfenozide in bovine liver and kidney and chicken muscle. The methods underwent successful independent laboratory validations. Methods were also radio-validated using samples from the goat metabolism and confined rotational crop studies.

Neither the parent nor metabolites are determined by standard multi-residue methods.

The Meeting concluded that adequate methods exist for the determination of methoxyfenozide in a variety of plant commodities, with limits of quantification of 0.01 or 0.02 mg/kg in most instances, and for the determination of methoxyfenozide in poultry and bovine commodities at LOQs of 0.01 mg/kg. It was noted that the method for methoxyfenozide in bovine liver and kidney and chicken muscle for methoxyfenozide was HPLC-MS and/or HPLC-MS/MS and may not be practicable for some laboratories.

Stability of residues in stored analytical samples

The stability of methoxyfenozide in numerous plant commodities and of methoxyfenozide and the glucuronide conjugate of the A-ring phenol in bovine and poultry commodities, stored frozen at about -20°C, was reported. Methoxyfenozide was stable for at least the indicated periods in the following commodities: apples 365 days, apple juice 283 days, wet apple pomace 302 days, tomatoes 372 days, head lettuce 365 days, cotton seed 9 months, refined cotton seed oil 12 months, cotton gin trash 6 months, maize grain 397 days, maize meal 127 days, maize oil 184 days, milk 106 days, bovine muscle 165 days, bovine liver 261 days, bovine kidney 265 days, eggs 93 days.

The Meeting concluded that methoxyfenozide is stable for 6-12 months in various plant commodities and for about 100 days in animal commodities stored frozen.

Definition of the residue

Studies of metabolism in a variety of crops and environmental fate demonstrated that methoxyfenozide did not undergo extensive transformation. The major component of residues in soil and plants is the parent compound, methoxyfenozide. The main residue component in goat milk and tissues (except liver and kidney) is again methoxyfenozide, as it is in poultry tissues except liver, kidney, and eggs. In goat and poultry liver and kidney, as well as in eggs, the A-ring phenol glucuronide is the main residue component and the parent is about 10% of the metabolite and below the typical level of quantification. The glucuronide conjugate constituted a significant proportion of the total radioactive residues in eggs (30%), goat kidney (42%), goat liver (29%), hen liver (20%), and hen kidney (36%). However, the feeding studies described below revealed approximately equal concentrations of methoxyfenozide and the metabolite in poultry eggs and bovine kidney and liver.

The distribution of methoxyfenozide between fat and muscle in the ruminant and poultry metabolism studies indicate that it is fat-soluble. The methoxyfenozide concentration in fat was approximately 10 times that in muscle in goats and about 3-8 times that in muscle in hens. The log of the partition coefficient, 3.7, also indicates solubility in fat.

The Meeting concluded that the residue should be defined as methoxyfenozide for compliance with MRLs and for dietary intake estimation in both plant and animal commodities.

The compound is fat-soluble in its distribution between meat muscle and fat, but not in its distribution in milk.

Results of supervised trials on crops

Oranges and mandarins. Supervised field trials were conducted on oranges and mandarins in various parts of Europe but there is no finalized GAP, so the trials could not be evaluated.

Apples. Supervised field trials were conducted in the USA and in Europe. GAP in the USA requires WP 800 g/kg or SC 240 g/l, 0.34 or 0.28 kg ai/ha, 1.1 kg ai/ha per season, 14-day PHI. This corresponds to no more than 4 applications per season. All US trials were with 6 applications at 14-day intervals with a total application of 2.0 kg ai/ha. Two of the trials were conducted as residue decline studies. The half-life of the methoxyfenozide residue was about 20 days and, as it was found in the apple metabolism study that the half-life of the total radioactive residue was 12 ± 9 days, it can be estimated that the residue contribution from the first two applications would have decreased to ≤20% of the initial value by the time of the last application (56-70-day interval). The trials may therefore be considered as being according to maximum GAP. The residues of methoxyfenozide from trials according to maximum GAP in the USA, in ranked order median underlined, were 0.20, 0.23, 0.25, 0.30, 0.36, 0.37, 0.40, 0.43, 0.52, 0.56, 0.60, 0.62, 0.62, 1.0, 1.0 mg/kg. The HR is 1.0 mg/kg and the STMR is 0.43 mg/kg.

The proposed GAP in northern and southern Europe, Austria, Belgium, France, Germany, Greece, Hungary, Ireland, Italy, Luxembourg, The Netherlands, Portugal, Spain, and Switzerland have not been adopted by the respective national authorities. The recently finalized GAP for the UK specifies a 240 g/l SC formulation applied at 0.6 l product /ha (0.14 kg ai/ha in 1500 l water/ha) for large trees and 0.4 l product/ha (0.096 kg ai/ha in 1000 l water/ha) for smaller trees, 3 applications and a 14-day PHI. The spray concentration is 0.0096 kg ai/hl. The residues found on apples from trials complying with maximum UK GAP in France, Italy, Spain, Belgium, and Germany were <0.05 (3), 0.06, 0.06, 0.10, 0.11, 0.11, 0.13, 0.13, 0.15, 0.15 and 0.23 mg/kg.

The residues in the European trials appeared to be from a different population from those in the US trials, which were higher.

Pears. Supervised field trials were conducted in the USA and Europe. GAP in the USA is the same as for apples. In the US trials, as with apples, there were six applications, each at 0.34 mg/kg. Two residue decline studies on pears in Europe indicated a residue half-life of 14-20 days. Assuming a 20-day half-life, the residues from the first two applications would have decreased to ≤20% of the initial value by the time of the last application, so the trials may be considered as complying with maximum GAP. The residues of methoxyfenozide on pears from 10 trials at maximum GAP in the USA in ranked order (median underlined) were 0.27, 0.31, 0.35, 0.36, 0.39, 0.50, 0.68, 0.74, 0.74 and 0.92 mg/kg. The HR is 0.92 mg/kg and the STMR is 0.44 mg/kg.

Proposed GAP in northern and southern Europe, Austria, Belgium, France, Germany, Greece, Hungary, Ireland, Italy, Luxembourg, The Netherlands, Portugal, Spain, and Switzerland is SC 240 g/l, 0.0096 kg ai/hl, 3 applications, 14-day PHI. However, this GAP has not been finalized. Maximum GAP for the UK specifies a 240 g/l SC formulation applied at 0.6 l product /ha (0.14 kg ai/ha) in 15 l water/ha, 3 applications, and a 14-day PHI. For small trees (foliar canopy height 2 m or less), the application rate is reduced to 0.096 kg ai/ha in 10 hl water/ha. The spray concentration is 0.0096 kg ai/hl in both cases. The ranked order of residues in pear trials at the maximum UK GAP in Italy (2), France (1), and Germany (1) is 0.07, 0.08, 0.09, 0.14 and 0.15 mg/kg.

The European residues again appeared to be from a different population from those in the US trials, which were higher.

The residues from the 25 apple and pear trials in the USA were comparable, and could be combined to give 0.20, 0.23, 0.25, 0.27, 0.30, 0.31, 0.35, 0.36 (3), 0.37, 0.40, 0.43, 0.50, 0.52, 0.56, 0.60, 0.62, 0.62, 0.68, 0.74 (2), 0.92 and 1.0 (2) mg/kg. The Meeting estimated an STMR of 0.43 mg/kg and a maximum residue level of 2 mg/kg for pome fruit. The HR is 1.0 mg/kg.

Stone fruits. Trials were reported from the USA and various countries in Europe for the period 1998-1999. GAP for peaches, cherries, nectarines, and plums in the USA is SC 240 g/l or WP 800 g/kg, 0.28 kg ai/ha, 1.1 kg ai/ha per season, 7-day PHI. GAP for cherries specifies SC 240 g/l, 0.28 kg

ai/ha, 0.95 kg ai/ha per season, 7-day PHI. Compliance with the 1.1 kg ai/ha/season rate requires no more than 4 applications per season. All US trials were conducted with 6 applications at 14-day intervals. Several decline studies conducted on peaches, cherries, and plums in the USA and Europe showed residue half-lives of 7-21 days, and the half-life of methoxyfenozide in the grape metabolism study was 13-21 days. The residues from the first two applications would therefore have decreased to ≤20% of the initial value by the last application. As the fruits would also have been rather small at the time of the first two applications, the first two would have a minimal effect on the residue level compared with the final four applications and the trials may be considered to be in accord with maximum GAP.

The ranked order of residues on peaches from 9 trials at maximum GAP is 0.32, 0.50, 0.54, 0.64, 0.78, 0.88 (2), 0.98 and 1.4 mg/kg. The STMR is 0.78 mg/kg and the HR 1.4 mg/kg.

The ranked order of residues on cherries from 7 trials at maximum GAP is 0.19, 0.26, 0.28, 0.34, 0.43, 0.52 and 0.56 mg/kg, with an STMR of 0.34 mg/kg and an HR of 0.56 mg/kg.

The ranked order of residues on plums from 7 trials at maximum GAP is 0.13, 0.14, 0.16, 0.19, 0.29, 0.30 and 0.34 mg/kg, with an STMR of 0.19 mg/kg and an HR of 0.34 mg/kg.

The trials on peaches and nectarines in France, Italy, Spain, and Greece could not be evaluated because there is no finalized relevant GAP.

The Meeting noted the identical use patterns for peaches, cherries and plums and decided to combine the residues to give 0.13, 0.14, 0.16, 0.19 (2), 0.26, 0.28, 0.29, 0.30, 0.32, 0.34 (2), 0.43, 0.50, 0.52, 0.54, 0.56, 0.64, 0.78, 0.88 (2), 0.98, 1.4 mg/kg.

The Meeting estimated an STMR of 0.34 mg/kg and a maximum residue level of 2 mg/kg for stone fruit. The HR is 1.4 mg/kg.

Grapes. Supervised field trials on grapes were reported from Europe and the USA. Trials on table grapes in Greece, Italy, France, Spain, and Portugal, and on wine grapes in Portugal, France, Spain, Italy and Germany could not be evaluated as there is no finalized GAP. GAP in the USA is WP 800 g/kg or SC 240 g/l, 0.28 kg ai/ha, 0.84 kg ai/ha per season, 30-day PHI. The ranked order of residues in 15 trials at maximum GAP is <0.02, 0.20, 0.20, 0.21, 0.26, 0.26, 0.32, 0.33, 0.34, 0.39, 0.45, 0.46, 0.52, 0.52, 0.84 mg/kg.

Using only the US data with finalized GAP, the Meeting estimated an STMR of 0.33 mg/kg and a maximum residue level of 1 mg/kg. The HR is 0.84 mg/kg.

Broccoli. Eight supervised field trials were reported from the USA. GAP is WP 800 g/kg or SC 240 g/l, 0.28 kg ai/ha, 1.2 kg ai/ha per season, 1-day PHI. The ranked order of residues is 0.52, 0.70, 0.76, 0.89, 0.98, 1.4, 1.6, 1.6 mg/kg. The Meeting estimated an STMR of 0.94 mg/kg and a maximum residue level of 3 mg/kg. The HR is 1.6 mg/kg.

Cabbages. Nine supervised field trials on head cabbages were reported from the USA. The GAP is the same as for broccoli. The ranked order of residues is 0.56, 0.57, 0.67, 0.88, 0.93, 2.2, 3.3, 3.4, 6.2 mg/kg. The Meeting estimated an STMR of 0.93 mg/kg and a maximum residue level of 7 mg/kg. The HR is 6.2 mg/kg.

Tomatoes. Supervised field trials were reported from Australia, Germany, Belgium, The Netherlands, Spain, Portugal, Italy, France, and the USA. GAP in Australia is SC 240 g/l, 0.03 or 0.04 kg ai/hl, (0.3 or 0.4 kg ai/ha, calculated), 3 applications, 0-day PHI. Nine trials were conducted at maximum GAP (0.04 kg ai/hl and/or 0.4 kg ai/ha) and the ranked order of residues is 0.13, 0.14, 0.21, 0.26, 0.56, 0.57, 0.73, 1.0, 1.6 mg/kg.

Glasshouse trials in Germany, Belgium, The Netherlands, Spain, Portugal, Italy, and France could not be evaluated for lack of finalized GAP.

GAP in the USA is SC 420 g/l or WP 800 g/kg, 0.28 kg ai/ha, 1.2 kg ai/ha per season, 1-day PHI. Thirteen trials were conducted at maximum GAP, with residues in ranked order of 0.052, 0.088, 0.12, 0.12, 0.13, 0.14, 0.16, 0.19, 0.20, 0.28, 0.33, 0.94, 1.8 mg/kg.

As the residues from Australia and the USA represent similar use patterns and are from the same population the values were combined, giving 0.052, 0.088, 0.12, 0.12, 0.13, 0.13, 0.14, 0.14, 0.16, 0.19, 0.20, 0.21, 0.26, 0.28, 0.33, 0.56, 0.57, 0.73, 0.94, 1.0, 1.6, 1.8 mg/kg.

The Meeting estimated an STMR of 0.20 mg/kg and a maximum residue level of 2 mg/kg for tomatoes. The HR is 1.8 mg/kg.

Peppers. Supervised field trials were reported from the USA on peppers (bell and non-bell) and from Portugal, Spain, Italy, France, and The Netherlands on bell peppers. The 14 glasshouse trials in Europe could not be evaluated as there is no GAP. GAP in the USA is SC 240 g/l or WP 800 g/kg, 0.30 kg ai/ha, 1.1 kg ai/ha per season, 1-day PHI. The ranked order of residues on peppers from 13 trials at maximum GAP is 0.041, 0.049, 0.050, 0.12, 0.14, 0.16, 0.16, 0.20, *0.26*, 0.36, *0.40*, *0.48*, *0.94* mg/kg. The residues in non-bell peppers are in italics.

The Meeting estimated an STMR of 0.16 mg/kg and a maximum residue level of 2 mg/kg for peppers. The HR is 0.94 mg/kg.

Egg plants. Two trials were reported from Malaysia, one within 75% of maximum GAP with a residue value of 0.13 mg/kg.

The Meeting considered one trial insufficient to estimate a maximum residue level.

Sweet corn. Supervised field trials were reported from the USA, where GAP is 0.13 kg ai/ha in a minimum of 94 l/ha (ground and aerial) before tasselling and in a minimum of 190 l/ha after tasselling (ground equipment) for SC 240 g/l, and 0.28 kg ai/ha in 94 l/ha through silking and 0.13 kg ai/ha thereafter for WP 800 g/kg, 1.1 kg ai/ha per season, 3-day PHI. The 14 trials were conducted at 0.28 kg ai/ha but the residues (kernels + cob with husk removed) were all <0.02 mg/kg.

The Meeting estimated an STMR of 0 mg/kg and a maximum residue level of 0.02 mg/kg (*) for sweet corn (corn-on-the-cob). The HR was estimated as 0.02 mg/kg.

Lettuce (head and leaf). Supervised field trials were reported from the USA on leaf and head lettuce. GAP for leaf and head lettuce is 0.28 kg ai/ha, 1.1 kg ai/ha per season, 1-day PHI. Eight trials on leaf lettuce at maximum GAP gave the ranked order of residues of 3.4, 8.2, 10, 12, 13, 17, 18, 18 mg/kg.

The Meeting estimated an STMR of 12.5 mg/kg and a maximum residue level of 30 mg/kg for leaf lettuce. The HR is 18 mg/kg.

Eight trials on head lettuce at maximum GAP gave the ranked order of residues for head lettuce with wrapper leaves of 1.6, 4.8, 5.4, 6.0, 6.2, 6.5, 7.9, 9.6 mg/kg.

The Meeting estimated an STMR of 6.1 mg/kg and a maximum residue level of 15 mg/kg for head lettuce. The HR is 9.6 mg/kg.

Spinach. Supervised field trials were reported from the USA where GAP is the same as for lettuce. Eight trials at maximum GAP showed the ranked order of residues 9.8, 10, 12, 14, 16, 18, 23, 43 mg/kg.

The Meeting estimated an STMR of 15 mg/kg and a maximum residue level of 50 mg/kg for spinach. The HR is 43 mg/kg.

Mustard greens. Supervised field trials were reported from the USA. GAP is the same as for lettuce. Seven trials at maximum GAP gave the ranked order of residues 10, 12, 14, 16, 17, 17, 18 mg/kg.

The Meeting estimated an STMR of 16 mg/kg and a maximum residue level of 30 mg/kg for mustard greens. The HR is 18 mg/kg.

Soya beans. Two supervised trials on soya beans were reported from Brazil. The residue was 0.03 mg/kg in the one trial according to GAP.

The Meeting considered one trial inadequate to estimate a maximum residue level.

Long beans. Two supervised trials were reported from Malaysia, one within 75% of the maximum GAP with a residue of <0.05 mg/kg.

The Meeting considered one trial inadequate to estimate a maximum residue level.

Celery. Supervised trials for the foliar application of methoxyfenozide to celery were reported from the USA. GAP is SC 240 g/l or WP 800 g/kg, 0.28 kg ai/ha, 1.1 kg ai/ha per season, 1-day PHI. Seven trials were according to GAP. An eighth trial was rejected because of a disease which made the celery unmarketable. The ranked order of residues is 0.48, 0.72, 3.0, 3.4, 5.5, 7.2, 7.8 mg/kg.

The Meeting estimated an STMR of 3.4 mg/kg and a maximum residue level of 15 mg/kg. The HR is 7.8 mg/kg.

Rice. Residues in two supervised trials in Japan at maximum GAP were <0.02 (2) mg/kg.

The Meeting concluded that an STMR and/or maximum residue level could not be estimated from the limited data base.

Maize (field corn). Numerous supervised field trials were reported from the USA, where GAP is SC 240 g/l or WP 800 g/kg, 0.13 kg ai/ha in a 47 l/ha minimum spray volume (ground and aerial), 1.1 kg ai/ha per season, 21-day PHI. Methoxyfenozide residues in maize grain from 24 trials conducted at between 2 and 3 times maximum GAP (0.24-0.36 kg ai/ha) were all ≤0.02 mg/kg. One additional trial at a similar rate yielded 0.033 mg/kg on one of duplicate samples.

Two supervised trials were reported from Mexico, where GAP is SC 240 g/l, 0.04 kg ai/ha, 4 applications, 30-day PHI. The PHIs were 77 and 156 days.

Two supervised trials were reported from Brazil, where GAP is 0.043 kg ai/ha, 1 application, 7-day PHI. The residues were 0.11 and 0.40 mg/kg.

Although the trials in Brazil produced the highest residues, the Meeting considered two trials to be inadequate for the estimation of an STMR and/or maximum residue level. Moreover, the description of the plants at the time of the last application (0.7 m high) suggests harvest of an immature commodity.

The Meeting estimated an STMR and HR of 0.02 mg/kg and a maximum residue level of 0.02 mg/kg (*), based on the US trials.

Cotton seed. Supervised trials on cotton were reported from Australia, Mexico and the USA.

GAP in Australia is SC 240 g/l, 0.6 kg ai/ha, 3 applications at 10-day intervals, 28-day PHI. Eight trials at maximum GAP yielded residues in the seed of <0.05, <0.05, <0.05, <0.05, 1.6, 3.2, 4.8, 4.9 mg/kg. Those values below the LOQ are from delinted cotton seed with little or no boll opening at application. Those with finite values are for undelinted seed with substantial boll opening at the last application.

GAP in Mexico is SC 240 g/l, 0.12 kg ai/ha, 2 applications, 14-day PHI. Residues in two trials at maximum GAP were 0.02 and 0.11 mg/kg.

GAP in the USA is WP 800 g/kg, 0.45 kg ai/ha in a minimum of 19 l water/ha (aerial) or 47 l water/ha (ground), 1.1 kg ai/ha per season, 14-day PHI. The 1.1 kg ai/ha seasonal rate limits the number of applications at maximum rate to 2, but all US trials were conducted with 5 applications at 14-day intervals for a total seasonal rate of 2.2 kg ai/ha. As the trials in Australia indicate, however, the state of the boll (closed or open) is much more likely to affect the residue level than additional applications 28-70 days before harvest. Eighteen trials were at maximum GAP and the ranked order of residues in undelinted seed is 0.013, 0.08, 0.10, 0.14, 0.22, 0.26, 0.26, 0.37, 0.41, 0.50, 0.51, 0.51, 0.52, 0.52, 0.98, 1.1, 1.2, 1.3 mg/kg.

The trials in Australia, Mexico, and the USA were combined to give the ranked order <0.05 (4), 0.013, 0.020, 0.08, 0.10, 0.11, 0.14, 0.22, 0.26, 0.26, 0.37, 0.41, 0.50, 0.51, 0.51, 0.52, 0.52, 0.98, 1.1, 1.2, 1.3, 1.6, 3.2, 4.8, 4.9 mg/kg.

The Meeting estimated an STMR of 0.39 mg/kg and a maximum residue level of 7 mg/kg for cotton seed, with an HR of 4.9 mg/kg.

Tree nuts. Supervised trials on almonds and pecans were reported from the USA. GAP is SC 240 g/l or WP 800 g/kg, 0.43 kg ai/ha, 1.1 kg ai/ha per season, 14-day PHI. The ranked order of residues from trials at maximum GAP is <0.02 (3), 0.024, 0.027, 0.034 mg/kg in pecans and <0.02, 0.02, 0.021 (2), 0.036 0.074 mg/kg in almonds.

The combined ranked order of residues in pecans and almonds is <0.02 (4), 0.020, 0.021 (2), 0.024, 0.027, 0.034, 0.036, 0.074 mg/kg.

The Meeting estimated an STMR of 0.021 mg/kg and a maximum residue level of 0.1 mg/kg for tree nuts. The HR is 0.074 mg/kg.

Animal feed items

Almond hulls. In the US trials on almonds, described above, the ranked order of residues in hulls is 6.4, 10 (2), 16, 26, 35 mg/kg.

The Meeting estimated a maximum residue level of 50 mg/kg on a dry weight basis (90% dry matter), and an STMR of 13 mg/kg (not dry weight) for almond hulls.

Maize forage and fodder. Supervised field trials in the USA on maize are described above. The GAP application rate is 0.13 kg ai/ha with a 21-day PHI. As all the trials were at twice this rate (0.24-0.36 kg ai/ha) and showed finite residues at the 21-day PHI they were not evaluated. The evaluation of residues in sweet corn forage and fodder is described below.

Sweet corn forage and fodder. Supervised field trials were reported from the USA for the foliar application of methoxyfenozide to sweet corn according to the following GAP: SC 240 g/l or WP 800 g/kg, 0.13 kg ai/ha in a minimum of 94 l/ha (ground and aerial) before tasselling and in a minimum of 190 l/ha after tasselling for SC, 0.28 kg ai/ha through silking and 0.13 kg ai/ha thereafter for WP in 94 l water/ha (aerial and ground) before tasselling and in 190 l water/ha after tasselling, 1.1 kg ai/ha per season, 3-day PHI for forage and 21-day PHI for fodder. The 14 trials were conducted at 0.28 kg ai/ha/application. The ranked order of residues in forage for the 12 trials with the WP formulation at maximum GAP is 0.20, 0.52, 1.4, 1.5, 3.4, 4.4, 4.6, 6.1, 6.2, 7.2, 15, 22 mg/kg.

The Meeting estimated a maximum residue level of 50 mg/kg on a dry weight basis (48% dry matter), and an STMR of 4.5 mg/kg (not dry weight) for maize forage.

The ranked order of residues in fodder for the 11 trials with the WP formulation at maximum GAP is 1.0, 2.0, 4.9, 5.9, 8.2 (2), 8.4, 9.4, 20 (2), 46 mg/kg.

The Meeting estimated a maximum residue level of 60 mg/kg on a dry weight basis (83% dry matter) and an STMR of 8.2 mg/kg (not dry weight) for maize fodder.

Cotton gin by-products. Eight of the 18 cotton trials in the USA at maximum GAP, described above, included the determination of methoxyfenozide residues in cotton gin trash. The ranked order of residues in the gin trash is 3.8, 7.1, 9.4, 9.9, 12, 15, 17, 18 mg/kg.

The Meeting estimated an STMR of 11 mg/kg and an HR of 18 mg/kg for cotton gin by-products (trash containing 90% dry matter).

Fate of residues during processing

Processing studies on oranges, apples, grapes, tomatoes, maize (field corn), fresh prunes, and cotton seed were reported.

Two orange processing trials were conducted in Spain and one in Italy. Oranges were converted to marmalade and in two trials juice. The processing factors for juice were 0.3 and 0.2, average 0.25, and for marmalade 0.8, 0.5 and 1.1, average 0.8.

Single processing studies were conducted on apples in Germany, Belgium, France and the USA. The processing factors were as follows: apple sauce, 0.4, 0.4, 0.4, average 0.4; apple juice, 0.4, 0.4, 0.4, 0.2, average 0.3; apple pomace (wet), 2, 2, 2, 6, average 3; apple pomace (dry), 7, 8, 7, average 7.

From the STMR of 0.43 mg/kg for pome fruit and the average processing factors for apple juice and wet pomace, the Meeting estimated STMR-Ps of 0.13 mg/kg for apple juice and 1.3 mg/kg for apple pomace (wet). From the HR of 1 mg/kg for pome fruit and the average processing factor of 7 for dry apple pomace, the Meeting estimated a maximum residue level of 7 mg/kg for apple pomace (dry).

A processing study on the preparation of peach preserves was reported from Italy but neither the RAC nor the preserves contained a quantifiable residue.

A study on processing plums to dried prunes was reported from the USA. The processing factor was 1.3 which, when applied to the HR and STMR for stone fruits (1.4 and 0.34 mg/kg), provides a maximum residue level, an STMR-P and an HR-P of 3, 0.44 and 1.8 mg/kg respectively for prunes (dried plums).

Processing studies were conducted on grapes in France, Italy, Germany, Portugal, Greece, and the USA. The processing factors for grape juice were 0.3 (USA), 0.4 (Portugal), 0.3 (France), 0.2 (Greece), and 0.1 (Italy), average 0.3; for dried grapes (raisins) 1.3 (USA), 2.4 (Greece), 3.1 (Italy), and 2.1 (Italy), average 2.2; for wine 0.4 (USA), 0.3 (USA), 1.3 (Italy), 0.2 (Germany), 0.3 (Germany), 0.4 (Germany), 0.3 (Germany), 0.4 (Portugal), 0.4 (France) and 0.3 (France), average 0.4.

From the processing factors and the STMR for grapes (0.33 mg/kg), the Meeting estimated STMR-Ps for grape juice, raisins, and wine of 0.10 mg/kg, 0.73 mg/kg, and 0.13 mg/kg, respectively.

From the processing factor for raisins (2.2) and the STMR and HR for grapes (0.33 and 0.84 mg/kg), the Meeting estimated a maximum residue level of 2 mg/kg, an STMR-P of 0.73 mg/kg and an HR-P of 1.8 mg/kg for dried grapes (raisins).

Tomato processing studies were carried out in the USA, Belgium, Germany, and Italy. The processing factors for juice were 0.2 (USA), 0.3 (Belgium), 0.4 (Germany), 0.4 (Germany) and 0.4 (Italy), average 0.3; for tomato paste 0.7 (USA), 2.2 (Belgium), 1.7 (Germany), 3.0 (Germany) and 3.4 (Italy), average 2.2; and for wet tomato pomace 3.6 (Belgium). The processing factor for peeling was 0.3 (average of 0.2 (Belgium), 0.4 (Germany), 0.4 (Germany), and 0.2 (Italy)).

From the processing factors and the STMR for tomato (0.20 mg/kg), the Meeting estimated STMR-Ps for tomato juice (0.060 mg/kg), tomato paste (0.44 mg/kg) and peeled tomatoes (0.06 mg/kg).

Wet milling and dry milling studies on maize grain were reported from the USA but the residue on the RAC was not quantifiable (0.0067 or <0.02 mg/kg). The only processed commodities with quantifiable residues were aspirated grain fractions (0.59 mg/kg) and refined oil from wet milling (0.036 mg/kg). Processing factors could not be calculated.

Cotton seed processing studies were reported from Mexico and the USA. In the four trials in Mexico, only crude oil was analyzed. The processing factors were hulls 0.14, crude oil 0.47 (USA), 0.5 (Mexico), 0.37 (Mexico), 0.13 (Mexico) and 0.14 (Mexico), average 0.32; cotton seed meal 0.45.

From the STMR for cotton seed (0.39 mg/kg) and the processing factors, the Meeting estimated STMR-Ps for cotton seed oil (crude) of 0.12 mg/kg, for cotton seed hulls of 0.055 mg/kg and for cotton seed meal of 0.18 mg/kg.

Residues in the edible portion of food commodities

The residue levels in peel and pulp were determined in the citrus trials in Europe. The pulp residue was <0.05 mg/kg in all mandarin and orange trials. No GAP is available for citrus fruits in Europe.

Pome fruit (apple and pear) trials included studies on peeling and washing. Washing reduced methoxyfenozide residues by an average factor of 0.76 (range 0.4-1). Peeling had a more dramatic effect, with an average factor of 0.15 (range 0.1-0.3).

In the head cabbage trials in the USA, residues were determined both with and without the wrapper leaves. The wrapper leaves are commonly removed before sale at the retail level and/or by the consumer. The average reduction factor was 0.09 (range 0.006-0.46).

In the head lettuce field trials in the USA, residues were determined both with and without the wrapper leaves, which are normally removed. The average reduction factor was 0.014 (range 0.0072-0.021).

Residues in animal commodities

Dietary burden in animals

The Meeting estimated the dietary burden of methoxyfenozide residues in farm animals, on the basis of diets listed in Appendix IX of the FAO Manual. Calculation from MRLs, high residues (HR) and STMR-P values provides the levels in feed appropriate for estimating MRLs for animal commodities, while calculation from the STMR and STMR-P values for feed is used to estimate STMR values for animal commodities.

Table 115. Estimated maximum dietary burden of farm animals.

Commodity	Codex group	Residue (mg/kg)	Basis	% Dry matter	Residue dry wt (mg/kg)	Proportion of diet (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Almond hulls	AM	50 dry wt.	MRL		50						
Apple pomace, wet	AB	1.3	STMR-P	40	3.25	15	5	0	0.49	0.16	
Maize grain	GC	0.02	MRL	88	0.023			80			0.018
Maize forage	AF	50 dry wt.	MRL		50	40	50	0	20	25	
Maize fodder	AS	60 dry wt.	MRL		60						
Cotton seed	SO	7	MRL	88	8.0	25	25	0	2.0	2.0	
Cotton seed hulls	AM	0.064	STMR-P	90	0.074						
Cotton meal	-	0.21	STMR-P	89	0.24			20			0.05
Cotton gin by-products	AM	18	HR	90	20	20	20	0	4.0	4.0	
TOTAL						100	100	100	26	31	0.07

Table 116. Estimated median dietary burden of farm animals.

Commodity	Codex group	Residue (mg/kg)	Basis	% Dry matter	Residue dry wt (mg/kg)	Proportion of diet (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Almond hulls	AM	13	STMR	90	14			0			
Apple pomace, wet	AB	1.3	STMR-P	40	3.25	40	20	0	1.3	0.64	
Maize grain	GC	0.02	STMR	88	0.023			80			0.018
Maize forage	AF	4.5	STMR	40	11	40	50	0	4.4	5.5	
Maize fodder	AS	8.2	STMR	83	9.9			0			
Cotton seed	SO	0.46	STMR	88	0.52		10	0		0.052	
Cotton hulls	AM	0.064	STMR-P	90	0.071			0			
Cotton meal	-	0.21	STMR-P	89	0.24			20			0.05
Cotton gin by-products	AM	11	STMR	90	12	20	20	0	2.4	2.4	
TOTAL						100	100	100	8.1	8.6	0.07

The estimated maximum dietary burdens of methoxyfenozide for beef cattle, dairy cattle and poultry are 26 ppm, 31 ppm and 0.07 ppm, respectively, and the estimated median dietary burdens are 8.1 ppm, 8.6 ppm and 0.07 ppm, respectively.

Feeding studies

Three cows at each level were dosed orally with methoxyfenozide at the equivalent of 16, 54, or 180 ppm in the diet for 28 consecutive days. Milk was collected daily and analyzed from days 1, 2, 4, 7, 10, 14, 17, 21, 24 and 28. The cows were slaughtered within 24 h of the last dose and tissues were collected and analyzed for methoxyfenozide and the glucuronide conjugate of the A-ring phenol.

Residues in all milk samples from the 16 and 54 ppm feeding levels were below the LOQ (0.01 mg/kg) for methoxyfenozide. Methoxyfenozide was detected (>0.003 mg/kg) in some samples, the highest values being 0.0063 mg/kg in day 28 milk at the 16 ppm feeding level and 0.0076 mg/kg in day 7 milk at the 54 ppm level. Quantifiable residues were found in milk from the 180 ppm group. The residue reached a plateau of 0.03-0.05 mg/kg on days 7-10 and an average of 0.027-0.030 mg/kg for days 10-28. The highest residue was 0.10 mg/kg from a single cow on day 7.

Day 28 milk from the 180 ppm feeding level was separated into cream and skimmed milk. The residue in the cream was 0.12 mg/kg, in the skimmed milk 0.0054 mg/kg, and in the whole milk 0.028 mg/kg. The concentration factor for cream relative to whole milk was 4.3. This does not represent a significantly higher solubility in milk fat than in whole milk. No information was provided on the residues in cream from the other feeding levels.

Fat, muscle, liver, and kidney from each of the cows at each of the feeding levels were analyzed for methoxyfenozide. The glucuronide conjugate of the A-ring phenol was also determined in liver and kidney.

In fat, methoxyfenozide was quantifiable at all feeding levels: 0.011 mg/kg maximum(<0.01 mg/kg average) at 16 ppm; 0.082 mg/kg maximum (0.041 mg/kg average) at 54 mg/kg; 0.44 mg/kg maximum (0.28 mg/kg average) at 180 ppm.

Methoxyfenozide was not detected in muscle at the 16 and 54 ppm feeding levels (limit of detection, 0.003 mg/kg). At the 180 ppm feeding level, the maximum residue was 0.10 mg/kg and the average was estimated at 0.0073 mg/kg (LOQ 0.01 mg/kg).

Methoxyfenozide residues were not quantifiable in liver (<0.01 mg/kg) at the 16 ppm feeding level. The highest residue was 0.0094 mg/kg. At the 54 ppm feeding level, the highest methoxyfenozide residue was 0.030 mg/kg, average 0.028 mg/kg, while the highest and average residues of the glucuronide conjugate of the A-ring phenol were 0.035 mg/kg and 0.026 mg/kg, respectively. At the 180 ppm feeding level, the highest and average residues of methoxyfenozide were 0.15 mg/kg and 0.13 mg/kg, and of the glucuronide conjugate of the A-ring phenol 0.12 mg/kg and 0.10 mg/kg.

In kidneys, methoxyfenozide was not detected at the 16 ppm feeding level and it was below the limit of quantification at the 54 ppm feeding level. At the 180 ppm level, the highest and average methoxyfenozide residues were 0.034 mg/kg and 0.026 mg/kg and those of the glucuronide conjugate of the A-ring phenol 0.046 mg/kg and 0.029 mg/kg.

The Meeting noted that the methoxyfenozide and glucuronide conjugate residue levels were approximately equal in kidney and liver in the feeding studies, whereas in the metabolism study the metabolite concentration was about 10 times that of the parent.

In a poultry feeding study, 10 or 12 hens in each of three feeding groups were dosed orally at the equivalent of 2.4, 7.6, and 24 ppm methoxyfenozide for 28 consecutive days. Eggs were collected each day by group and the hens were killed within 24 h of the last dose. Tissues were analyzed for methoxyfenozide.

At the 2.4 ppm feeding level, residues in eggs were below the limit of detection on days 1, 3, and 7, as they were at the 7.6 ppm level except in one of three samples (0.0032 mg/kg) on day 1. The estimated limit of detection for methoxyfenozide was 0.003 mg/kg.

At the 24 ppm feeding level, residues of methoxyfenozide in eggs were below the limit of detection over the entire 28 days, except in one sample on day 10 (0.0054 mg/kg) and one sample on day 17 (0.0030 mg/kg). Residues of the glucuronide conjugate of the A-ring phenol became detectable on day 7 but never reached the limit of quantification (0.01 mg/kg).

Residues of methoxyfenozide never reached the limit of detection (0.003 mg/kg) in fat, muscle, or liver at any feeding level. At the 7.6 and 24 ppm feeding levels, the glucuronide conjugate of the A-ring phenol in liver averaged 0.013 and 0.021 mg/kg, respectively.

Maximum residue levels in animal commodities

The Meeting agreed that the residues in cows from the 54 ppm feeding level could be extrapolated to the 31 ppm maximum dietary burden for dairy cattle, to estimate maximum residue levels for ruminant commodities. The 16 ppm feeding level could not be used as the residues were undetectable or unquantifiable.

The Meeting further agreed that the residues from the 16 mg/kg feeding level, all below the LOQ, could be used without extrapolation as the residues at the 8.1 and 8.6 median feeding levels for beef and dairy cattle, respectively.

Table 117. Estimated maximum and median residue levels in animal commodities, derived from treated feeds.

Dietary burden (ppm) Feeding level [ppm]	Methoxyfenozide residue [1] (mg/kg)								
	Milk [2]	Muscle		Liver		Kidney		Fat	
	Mean	Highest	Mean	Highest	Mean	Highest	Mean	Highest	Mean
MRL dairy cattle (31)	0.0027	0.0017		0.017		0.0022		0.047	
[54] [3]	0.0047	<0.003		0.0305		0.0038		0.0820	
STMR dairy cattle (8.6)	0.0041		0.003		0.0075		0.003		0.0082
[16] [4]	0.0041		<0.003		0.0075		<0.003		0.0082

[1] Methoxyfenozide only.
[2] Day 28 milk.
[3] Extrapolation.
[4] Direct application of the 16 ppm level, owing to high uncertainty in the values (undetectable or below the LOQ).

The Meeting estimated a maximum residue level for milk of 0.01 mg/kg and an STMR of 0.0041 mg/kg. Although methoxyfenozide was distributed preferentially in the fat of muscle, the concentration factor from whole milk to cream at the 180 ppm feeding level was only 4.3 to 1. No measurements were made on cream at the feeding level of relevance, 54 ppm. The Meeting estimated maximum residue levels for mammalian meat (fat) of 0.05 mg/kg and for mammalian offal of 0.02 mg/kg, and STMRs for mammalian muscle at 0.003 mg/kg, for offal at 0.0075 mg/kg, and for mammalian fat (trimmable) at 0.0082 mg/kg. The HRs are 0.017 mg/kg for mammalian offal, 0.0017 mg/kg for mammalian meat muscle and 0.047 mg/kg for mammalian meat fat.

The Meeting concluded that residues of methoxyfenozide are unlikely in poultry commodities at the maximum and median dietary burden levels of 0.07 ppm. At the lowest feeding level, 2.4 ppm, methoxyfenozide and the glucuronide conjugate of the A-ring phenol were generally undetectable in eggs (<0.003 mg/kg). Methoxyfenozide was detected in one sample at the 2.4 ppm feeding level and in several samples at higher feeding levels, so the estimated maximum residue level is 0.01 mg/kg and the STMR 0 mg/kg in eggs. At all feeding levels, residues were undetectable (<0.003 mg/kg) in muscle and fat, so the estimated maximum residue level is 0.01 (*) mg/kg for poultry meat, and the STMR 0 mg/kg for poultry muscle and fat. In liver, residues were undetectable at all feeding levels (<0.003 mg/kg). The maximum residue level for poultry offal is therefore 0.01(*) mg/kg and the STMR 0 mg/kg. The HRs are 0.003 mg/kg for eggs and 0 mg/kg for poultry offal and meat.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed in Table 118 are suitable for establishing maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue in plant and animal commodities for compliance with MRLs and for estimation of dietary intake: *methoxyfenozide.*

The residue is regarded as fat soluble in its distribution between meat muscle and fat, but not in milk.

Table 118. Summary of recommendations.

CCN	Commodity	Recommended MRL (mg/kg)	STMR or STMR-P (mg/kg)	HR or HR-P (mg/kg)
AM660	Almond hulls	50 (dry wt.)	13	
AB226	Apple pomace, dry	7	1.3 (wet)	
VB400	Broccoli	3	0.94	1.6
VB41	Cabbages, head	7	0.93	6.2
VS624	Celery	15	3.4	7.8
	Cherries		0.34	0.56
SO691	Cotton seed	7	0.46	4.9
DF269	Dried grapes (raisins)	2	0.73	1.8
MO105	Edible offal (mammalian)	0.02	0.0075	0.017
PE112	Eggs	0.01	0	0.003
FB269	Grapes	1	0.33	0.84
VL482	Lettuce, head	15	6.1	9.6
VL483	Lettuce, leaf	30	12.5	18
GC645	Maize	0.02 (*)	0.02	0.02
AS645	Maize fodder	60 (dry wt.)	8.2 (fresh wt.)	
AF645	Maize forage	50 (dry wt.)	4.5 (fresh wt.)	
MM95	Meat (from mammals other than marine mammals)	0.05 (fat)	0.003 (muscle) 0.0082 (fat)	0.0017 (muscle) 0.046 (fat)
ML106	Milks	0.01	0.0041	
VL485	Mustard greens	30	16	18
VO51	Peppers	2	0.16	0.94
FP9	Pome fruits	2	0.43	1.0
PM110	Poultry meat	0.01 (*) note	0 (muscle) 0 (fat)	0 (muscle) 0 (fat)
PO111	Poultry, edible offal of	0.01 (*) note	0	0
DF14	Prunes (dried plums)	3	0.44	1.8
VL502	Spinach	50 [1/]	15	43
FS0012	Stone fruits	2	0.34	1.4
VO447	Sweet corn (corn-on-the-cob)	0.02 (*)	0	0.02
VL448	Tomatoes	2	0.20	1.8
TN85	Tree nuts	0.1	0.021	0.074
FP226	Apples		0.43	1.0
JF226	Apple juice		0.13	
	Cotton seed hulls		0.055	
	Cotton seed meal		0.18	
OC691	Cotton seed oil, crude		0.12	
	Cotton gin byproducts		11 (fresh wt.)	18 (fresh wt.)
JF269	Grape juice		0.10	
FS254	Nectarines		0.78	1.4
FS247	Peaches		0.78	1.4
FP230	Pears		0.44	0.92
FS014	Plums (including prunes)		0.19	0.34
JF448	Tomato juice		0.06	
	Tomato paste		0.44	
-	Tomato, peeled		0.06	
-	Wine		0.13	

Note: Animal commodity, no residues expected from consumption of feed commodities with methoxyfenozide residues, as evaluated by the JMPR.

[1/] The information provided to the JMPR precludes an estimate that the dietary intake would be below the acute RfD in the case of spinach (children).

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of methoxyfenozide, based on the STMRs estimated for 42 commodities for the five GEMS/Food regional diets were in the range of 0-9% of the maximum ADI (Table 119). The ADI is 0-0.1 mg/kg bw/day. The Meeting concluded that the long-term dietary intake of residues of methoxyfenozide is unlikely to present a public health concern.

Table 119. International estimated daily intakes (IEDIs) of methoxyfenozide in GEMS/Food regional diets (ADI = 0-0.1 mg/kg bw/day).

Codex code	Commodity	STMR or STMR-P, mg/kg	Diets: g/person/day. Intake = daily intake: µg/person									
			Mid-East		Far-East		African		Latin American		European	
			diet	intake	diet	intake	diet	intake	diet	intake	diet	intake
JF 0226	Apple juice	0.13	4.5	0.6	0	0.0	0	0.0	0.3	0.0	3.8	0.5
VB 0400	Broccoli	0.94	0.5	0.5	1.0	0.9	0.0	0.0	1.1	1.0	2.7	2.5
VB 0041	Cabbages, head	0.93	ND	-	ND	-	ND	-	ND	-	ND	-
VS 0624	Celery	3.4	0.5	1.7	0.0	0.0	0.0	0.0	0.3	1.0	2.0	6.8
SO 0691	Cotton seed	0.46	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
OC 0691	Cotton seed oil, crude	0.15	3.8	0.6	0.5	0.1	0.5	0.1	0.5	0.1	0.0	0.0
MO 0105	Edible offal (mammalian)	0.0075	4.2	0.0	1.4	0.0	2.8	0.0	6.1	0.0	12.4	0.1
PE 0112	Eggs	0	14.6	0.0	13.1	0.0	3.7	0.0	11.9	0.0	37.6	0
FB 0269	Grapes (fresh, wine, excluding dried grapes)	0.1	15.8	1.6	1.0	0.1	0.0	0.0	1.3	0.1	13.8	1.4
DF 0269	Grapes, dried (= currants, raisins and sultanas)	0.73	0.3	0.2	0.0	0.0	0.0	0.0	0.3	0.2	2.3	1.7
VL 0482	Lettuce, head	6.1	2.3	14.0	0.0	0.0	0.0	0.0	5.8	35.4	22.5	137.3
VL 0483	Lettuce, leaf	12	2.3	27.6	0.0	0.0	0.0	0.0	5.8	69.6	22.5	270.0
MM 0095	Meat from mammals other than marine mammals: 20% as fat	0.0082	7.4	0.1	6.6	0.1	4.8	0.0	9.4	0.1	31.1	0.3
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	0.003	29.6	0.1	26.2	0.1	19.0	0.1	37.6	0.1	124.4	0.4
ML 0106	Milks	0.0041	116.9	0.5	32.1	0.1	41.8	0.2	160.1	0.7	289.3	1.2
VL 0485	Mustard greens	16	0.1	1.6	0.1	1.6	0.1	1.6	0.1	1.6	0.1	1.6
VO 0051	Peppers	0.16	3.4	0.5	2.1	0.3	5.4	0.9	2.4	0.4	10.4	1.7
FP 0009	Pome fruits	1	10.8	10.8	7.5	7.5	0.3	0.3	6.5	6.5	51.3	51.3
PM 0110	Poultry meat: 10% as fat	0	3.1	0.0	1.3	0.0	0.6	0.0	2.5	0.0	5.3	0.0
PM 0110	Poultry meat: 90% as muscle	0	27.9	0.0	11.9	0.0	5.0	0.0	22.8	0.0	47.7	0.0
PO 0111	Poultry, edible offal of	0	0.1	0.0	0.1	0.0	0.1	0.0	0.4	0.0	0.4	0.0
DF 0014	Prunes	0.44	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.5	0.2
VL 0502	Spinach	15	0.5	7.5	0.0	0.0	0.0	0.0	0.3	4.5	2.0	30.0
FS 0012	Stone fruits	0.34	7.3	2.8	1.0	0.4	0.0	0.0	0.8	0.3	23.3	8.9
VO 0447	Sweet corn (corn-on-the-cob)	0	0.0	0.0	0.0	0.0	4.4	0.0	0.0	0.0	8.3	0.0
VO 0448	Tomatoes (fresh)	0.2	44.1	8.8	5.7	1.1	14.6	2.9	25.5	5.1	34.9	7.0
JF 0448	Tomato juice	0.06	0.3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	2.0	0.1
-d	Tomato paste	0.44	5.8	2.6	0.2	0.1	0.3	0.1	0.0	0.0	4.0	1.8
-d	Tomatoes peeled	0.06	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	4.0	0.2
TN 0085	Tree nuts	0.012	1.1	0.0	13.5	0.2	4.5	0.1	17.8	0.2	4.6	0.1
Total intake (µg/person)=				82.1		12.6		6.2		127.1		524.9
Bodyweight per person (kg bw)=				60		55		60		60		60
ADI (µg/person)=				6000		5500		6000		6000		6000
%ADI=				1.4		0.2		0.1		2.1		8.7
Rounded %ADI=				1		0		0		2		9

Short-term intake

The acute RfD for methoxyfenozide is 0.9 mg/kg body weight. The international estimate of short term intake (IESTI) for methoxyfenozide was calculated for food commodities for which maximum residue levels, STMRs and/or HR values were established at this the Meeting. The results for the general population are shown in Table 120 and those for children up to 6 years old are given in Table 121.

The IESTI for spinach is 100% of the acute RfD for the general population and 310% of the acute RfD for children. The information provided to the Meeting precluded an estimate that the short-term dietary intake of residues in spinach by children would be below the acute reference dose. The Meeting noted that a conservative acute RfD was established and that a refinement is possible.

For all the other commodities considered, the percentage of the acute RfD varied from 0 to 10% for the general population and from 0 to 30% for children.. The Meeting concluded that the short-term intake of residues of methoxyfenozide in these commodities, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

Table 120. International estimates of short-term intake (IESTI) of methoxyfenozide by the general population (acute RfD = 0.9 mg/kg bw/day or 900 µg/kg bw/day).

Codex code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight				Variability factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Country	Body wt (kg)	Large portion, corrected, g/person	Unit wt, g	Country	% edible portion	Unit wt, edible portion, g				
FP 0226	Apples	-	1	USA	65.0	1348	138	USA	92	127	3	2a	24.65	3
VB 0400	Broccoli	-	1.6	USA	65.0	376	608	USA	78	474	3	2b	27.79	3
VB 0041	Cabbages, head	-	6.2	SAF	55.7	362	908	USA	79	717	3	2b	120.90	10
VS 0624	Celery (stalk)	-	7.8	FRA	62.3	225	40	USA	100	40	3	2a	38.17	4
FS 0013	Cherries	-	0.56	FRA	62.3	375	5	FRA	89	4	1	1	3.37	0
PE 0840	Chicken eggs	-	0.003	FRA	62.3	219	-	-	-	-	-	1	0.01	0
SO 0691	Cotton seed	-	4.9	USA	65.0	3	-	-	-	-	-	3	-	-
MO 0096	Edible offal of cattle, goats, horses, pigs & sheep	-	0.017	FRA	62.3	277	-	-	-	-	-	1	0.08	0
FB 0269	Grapes (fresh, wine, dried)	-	0.84	AUS	67.0	1004	125	FRA	94	118	3	2a	15.54	2
DF 0269	Grapes, dried (= currants, raisins and sultanas)	-	1.8	FRA	62.3	135	-	-	-	-	-	-	-	-
VL 0482	Lettuce, head	-	9.6	USA	65.0	213	539	USA	95	512	3	2b	94.18	10
VL 0483	Lettuce, leaf	-	18	NLD	63.0	152	10	USA	100	10	1	1	43.38	5
GC 0645	Maize (fresh, flour, oil)	-	0.02	FRA	62.3	260	-	-	-	-	-	3	-	-
MM 0095	Meat from mammals other than marine mammals: 20% as fat	-	0.046	AUS	67.0	104	-	-	-	-	-	1	0.07	0
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	-	0.0017	AUS	67.0	417	-	-	-	-	-	1	0.01	0
ML 0106	Milks	0.0041	-	USA	65.0	2466	-	-	-	-	-	3	0.16	0
VL 0485	Mustard greens	-	18	USA	65.0	228	-	-	-	-	-	-	-	-
FS 0245	Nectarines	-	1.4	USA	65.0	590	136	USA	92	125	3	2a	18.10	2
FS 0247	Peaches	-	1.4	SAF	55.7	685	98	USA	87	85	3	2a	21.51	2
FP 0230	Pears	-	0.92	USA	65.0	693	166	USA	91	151	3	2a	14.08	2
VO 0444	Peppers, chili	-	0.94	USA	65.0	90	45	USA	96	43	3	2a	2.56	0
VO 0445	Peppers, sweet	-	0.94	FRA	62.3	207	119	USA	82	98	3	2a	6.07	1

Codex code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight				Variability factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Country	Body wt (kg)	Large portion, corrected, g/person	Unit wt, g	Country	% edible portion	Unit wt, edible portion, g				
FS 0014	Plums (fresh, prunes)	-	0.34	USA	65.0	413	66	USA	94	62	3	2a	2.81	0
PM 0110	Poultry meat: 10% as fat	-	0	AUS	67.0	43	-	-	-	-	-	1	0.00	0
PM 0110	Poultry meat: 90% as muscle	-	0	AUS	67.0	388	-	-	-	-	-	1	0.00	0
PO 0111	Poultry, edible offal of	-	0	USA	65.0	248	-	-	-	-	-	1	0.00	0
DF 0014	Prunes	-	1.8	USA	65.0	303	6	FRA	83	5	1	1	6.99	1
VL 0502	Spinach (bunch)	-	43	NLD	63.0	820	340	USA	72	245	3	2a	893.60	100
VO 0447	Sweet corn (corn-on-the-cob)	-	0.02	USA	65.0	367	200	JPN	100	200	3	2a	0.24	0
VO 0448	Tomatoes (fresh, juice, paste, peeled)	-	1.8	USA	65.0	391	123	USA	100	123	3	2a	17.63	2
TN 0085	Tree nuts	-	0.074	JPN	52.6	107	-	-	-	-	-	-	-	-
-d	Wine only	0.13	-	AUS	67.0	1131	-	-	-	-	-	3	2.19	0

Table 121. International estimates of short-term intake (IESTI) of methoxyfenozide by children up to 6 years (acute RfD = 0.9 mg/kg bw/day or 900 µg/kg bw/day).

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight				Variab-ility factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Coun-try	Body wt (kg)	Large portion, corrected, g/person	Unit wt, g	Coun-try	% edible portion	Unit wt, edible portion, g				
FP 0226	Apples	-	1	USA	15.0	679	138	USA	92	127	3	2a	62.18	7
VB 0400	Broccoli	-	1.6	USA	15.0	164	608	USA	78	474	3	2b	52.56	6
VB 0041	Cabbages, head	-	6.2	SAF	14.2	220	908	USA	79	717	3	2b	288.30	30
VS 0624	Celery (stalk)	-	7.8	FRA	17.8	111	40	USA	100	40	3	2a	83.81	9
FS 0013	Cherries	-	0.56	FRA	17.8	297	5	FRA	89	4	1	1	9.34	1
PE 0840	Chicken eggs	-	0.003	FRA	17.8	134		-	-	-	-	1	0.02	0
SO 0691	Cotton seed	-	4.9	USA	15.0	1		-	-	-	-	3	-	-
MO 0096	Edible offal of cattle, goats, horses, pigs & sheep	-	0.017	FRA	17.8	203		-	-	-	-	1	0.19	0
FB 0269	Grapes (fresh, wine, dried)	-	0.84	JPN	15.9	388	125	FRA	94	118	3	2a	32.90	4
DF 0269	Grapes, dried (= currants, raisins and sultanas)	-	1.8	USA	15.0	59		-	-	-	-	-	-	-
VL 0482	Lettuce, head	-	9.6	NLD	17.0	84	539	USA	95	512	3	2b	141.70	20
VL 0483	Lettuce, leaf	-	18	NLD	17.0	102	10	USA	100	10	1	1	108.00	10
GC 0645	Maize (fresh, flour, oil)	-	0.02	FRA	17.8	148		-	-	-	-	3	-	-
MM 0095	Meat from mammals other than marine mammals: 20% as fat	-	0.046	AUS	19.0	52		-	-	-	-	1	0.13	0
MM 0095	Meat from mammals other than marine mammals: 80% as muscle	-	0.0017	AUS	19.0	208		-	-	-	-	1	0.02	0
ML 0106	Milks	0.0041	-	USA	15.0	1286		-	-	-	-	3	0.35	0
VL 0485	Mustard greens	-	18	USA	15.0	53		-	-	-	-	-	-	-
FS 0245	Nectarines	-	1.4	AUS	19.0	302	136	USA	92	125	3	2a	40.70	5
FS 0247	Peaches	-	1.4	AUS	19.0	315	98	USA	87	85	3	2a	35.81	4
FP 0230	Pears	-	0.92	UNK	14.5	279	166	USA	91	151	3	2a	36.87	4
VO 0444	Peppers, chili	-	0.94	AUS	19.0	31	45	USA	96	43	3	2b	4.53	1
VO 0445	Peppers, sweet	-	0.94	AUS	19.0	60	119	USA	82	98	3	2b	8.91	1
FS 0014	Plums (fresh, prunes)	-	0.34	FRA	17.8	254	66	USA	94	62	3	2a	7.23	1
PM 0110	Poultry meat: 10% as fat	-	0	AUS	19.0	22		-	-	-	-	1	0.00	0
PM 0110	Poultry meat: 90% as muscle	-	0	AUS	19.0	201		-	-	-	-	1	0.00	0
PO 0111	Poultry, edible offal of	-	0	USA	15.0	37		-	-	-	-	1	0.00	0
DF 0014	Prunes	-	1.8	AUS	19.0	170	6	FRA	83	5	1	1	13.43	1

Codex Code	Commodity	STMR or STMR-P mg/kg	HR or HR-P mg/kg	Large portion diet			Unit weight				Variab-ility factor	Case	IESTI µg/kg bw/day	% acute RfD rounded
				Coun-try	Body wt (kg)	Large portion, corrected, g/person	Unit wt, g	Coun-try	% edible portion	Unit wt, edible portion, g				
VL 0502	Spinach (bunch)	-	43	SAF	14.2	420	340	USA	72	245	3	2a	2755.39	310
VO 0447	Sweet corn (corn-on-the-cob)	-	0.02	UNK	14.5	161	200	JPN	100	200	3	2b	0.67	0
VO 0448	Tomatoes (fresh, juice, paste, peeled)	-	1.8	USA	15.0	159	123	USA	100	123	3	2a	48.60	5
TN 0085	Tree nuts	-	0.074	AUS	19.0	28		-	-	-	-	-	-	-
-	Wine only	0.13	-	AUS	19.0	4		-	-	-	-	3	0.03	0

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