TEBUFENOZIDE (196)

EXPLANATION

Tebufenozide was first evaluated in 1996 for toxicology and residues and was subsequently reviewed to include a proposed MRL for kiwifruit in 1997 while data on grapes and pome fruit were re-evaluated in 1999. The manufacturer requested that tebufenozide be scheduled for evaluation by the 2001 JMPR to consider MRLs for other commodities to accommodate new registered uses in a number of countries.

The present Meeting received information requested by the 1996 JMPR on rotational crops, animal feeding studies, stability in stored samples and residues in raisins. Supervised trials on avocados, bush berries, Brassica vegetables, citrus, rape seed, cranberries, fruiting vegetables other than cucurbits, leafy vegetables, mint, stone fruit (excluding cherries), sugar cane, tree nuts and turnips were also reported as were various analytical methods. Information on current GAP was provided.

METABOLISM AND ENVIRONMENTAL FATE

Tebufenozide and its metabolites in this evaluation are generally designated by codes instead of chemical names (see below).

RH-5992	tebufenozide, *N-tert*-butyl-*N'*-(4-ethylbenzoyl)-3,5-dimethylbenzohydrazide
RH-9886	*N-tert*-butyl-*N'*-(4-ethylbenzoyl)-3-hydroxymethyl-5-methylbenzohydrazide
RH-1788	*N-tert*-butyl-*N'*-[4-(1-hydroxyethyl)benzoyl]-3,5-dimethylbenzohydrazide
RH-0282	*N-tert*-butyl-*N'*-[4-(1-hydroxyethyl)benzoyl]-3-hydroxymethyl-5-methylbenzohydrazide
RH-0126	*N-tert*-butyl-*N'*-[4-(1-hydroxyethyl)benzoyl]-3-carboxy-5-methylbenzohydrazide
RH-2703	*N-tert*-butyl-*N'*-(4-carboxymethylbenzoyl)-3,5-dimethylbenzohydrazide
RH-6595	*N-tert*-butyl-*N'*-(4-acetylbenzoyl)-3,5-dimethylbenzohydrazide
RH-9871	*N-tert*-butyl-*N'*-(4-acetylbenzoyl)-3-hydroxymethyl-5-methylbenzohydrazide
RH-2631	*N-tert*-butyl-*N'*-(4-acetylbenzoyl)-3-carboxy-5-methylbenzohydrazide
RH-0875	*N-tert*-butyl-*N'*-(4-ethylbenzoyl)-3,5-dicarboxybenzohydrazide
RH-9841 (RH-5992-olefin)	*N-tert*-butyl-*N'*-(4-vinylbenzoyl)-3,5-dimethylbenzohydrazide
RH-9526	Stearic acid conjugate of RH-9886

Note. The names of RH-0126, RH-2703, RH-2631 and RH-0875 are not strictly according to IUPAC usage, but have been used to show more clearly their relations to the other compounds

Animal metabolism

No additional information was provided.

Plant metabolism

No additional information was provided.

Environmental fate in soil

Residues in rotational crops. In a confined rotational crop study in 1991 in Madera, California, USA (Sharma and Bergin, 1996a) three separate plots were treated with four applications at 14-day intervals to bare ground, at the maximum US label rate of 0.28 kg ai/ha, of [^{14}C]tebufenozide labelled in the ethylphenyl ring (A), the dimethylphenyl ring (B), or the *tert*-butyl group (T). Wheat, collards and turnips were planted back in each plot 30, 90, 250 and 365 days (384 days for turnips) after the last applications. Samples of mature turnips, collards, grain and straw, and immature wheat forage were analysed by combustion to determine the total radioactive residue (TRR). Results with the three labels were comparable, except in wheat grain and straw at 30 days plant-back, in which some A values were higher and some T values lower than those for the B label. The average results are shown in Table 1.

Table 1. Total radioactive residues in rotational crops.

Crop	^{14}C, mg/kg as tebufenozide, mean of A, B and T labels			
	30 DAT	90 DAT	250 DAT	365/384 DAT
Wheat grain	0.4	0.06	0.07	0.07
Wheat straw	7.2	0.4	0.8	0.3
Wheat forage	2.6	0.3	0.1	0.1
Collards	0.1	0.03	0.08	0.006
Turnip top	0.5	0.06	0.08	0.03
Turnip root	0.08	0.007	0.008	0.007

DAT: days between last treatment and plant-back.

Extracts were analysed by a combination of HPLC and TLC. Wheat straw, which contained the highest residues, was analysed at each interval. Since the residues in wheat straw differed only in their magnitude and not the nature of the metabolites, only the 30- and 250-day samples of the remaining crop samples were analysed. Sample were stored for about 4 years before being analysed.

Wheat. Extraction of the residue with methanol and water containing acetic acid recovered 80% from wheat straw and forage and 52% from grain, but became less efficient at later samplings: about 60% from straw and forage and 15% from grain at 250 or 365 days plant-back. The major component in all samples was RH-1788, either as the free alcohol or conjugated with glucose or malonylglucose. In all the straw and in the 30-day plant-back grain samples, the amount of free RH-1788 was almost equal to the conjugated. Residues in forage were almost entirely conjugated, predominantly consisting of the malonylglucose conjugate of RH-1788. Low concentrations (<10%) of other metabolites, which could only be identified in wheat samples at 30 days plant-back, were the ketone RH-6595, the two alcohols RH-9886 and RH-0282 as well as their sugar conjugates, and RH-0126 and 9871. Less than 1% of the parent compound was present and only in 30 days plant-back straw and grain. The residues in 90, 250 and 365-day straw, forage and grain consisted mainly of RH-1788 and its sugar conjugates. The parent compound was not detected in any of these samples. In the 250-day grain samples the extracted residue amounted to only about 0.01 mg/kg. No quantifiable individual residues were detected in the 250-day grain. Residues as percentages of the TRR are shown in Table 2.

Table 2. Percentages of the TRR (mean of 3 labels) in wheat planted as a rotational crop.

Compound	Straw				Forage		Grain	
	DAT 30	DAT 90	DAT 250	DAT 365	DAT 30	DAT 250	DAT 30	DAT 250
Tebufenozide	1.2						1.2	
RH-1788	17	>29	>16	>9	<5	<1	11	
RH-1788-conj	>25	22	23	24	66	46	21	
RH-0282	8.1					<1	<4	
RH-0282-conj	2.0				7.4			
RH-6595	9.2				<5		1.4	
RH-9886	2.7	<6	<12	<6	<5	<1		
RH-9886-conj	<6							
RH-0126	1.4						<4	
RH-9871					<5		2.5	

[>] Metabolite also found in adjacent fraction(s) mainly containing other(s).
[<] Fraction contains other metabolites as well.

Additional extraction of the post-extraction solids from wheat forage and straw was by sequential incubation with the enzymes amylase and pronase, followed by extraction with EDTA, sodium chlorite, 20% KOH and 70% sulfuric acid to release the activity associated with large molecule natural products such as starch, proteins, pectin, lignins, hemicellulose and cellulose. Each step released 1-6% of the TRR from the straw and forage, indicating that the bound residue was either incorporated into natural complex molecules like starch and cellulose or tightly trapped in these natural biopolymers.

Since bound residue levels in grain were high, two samples of solvent-extracted grain were extracted by the sequential treatment with two different enzymes, followed by digestion with acid and base, each of which made 3-13% of the bound residue in the 30-day grain soluble. The extracted activity in each case contained too much substrate to allow any analysis. The remaining radioactivity was incorporated into natural grain constituents like starch and cellulose.

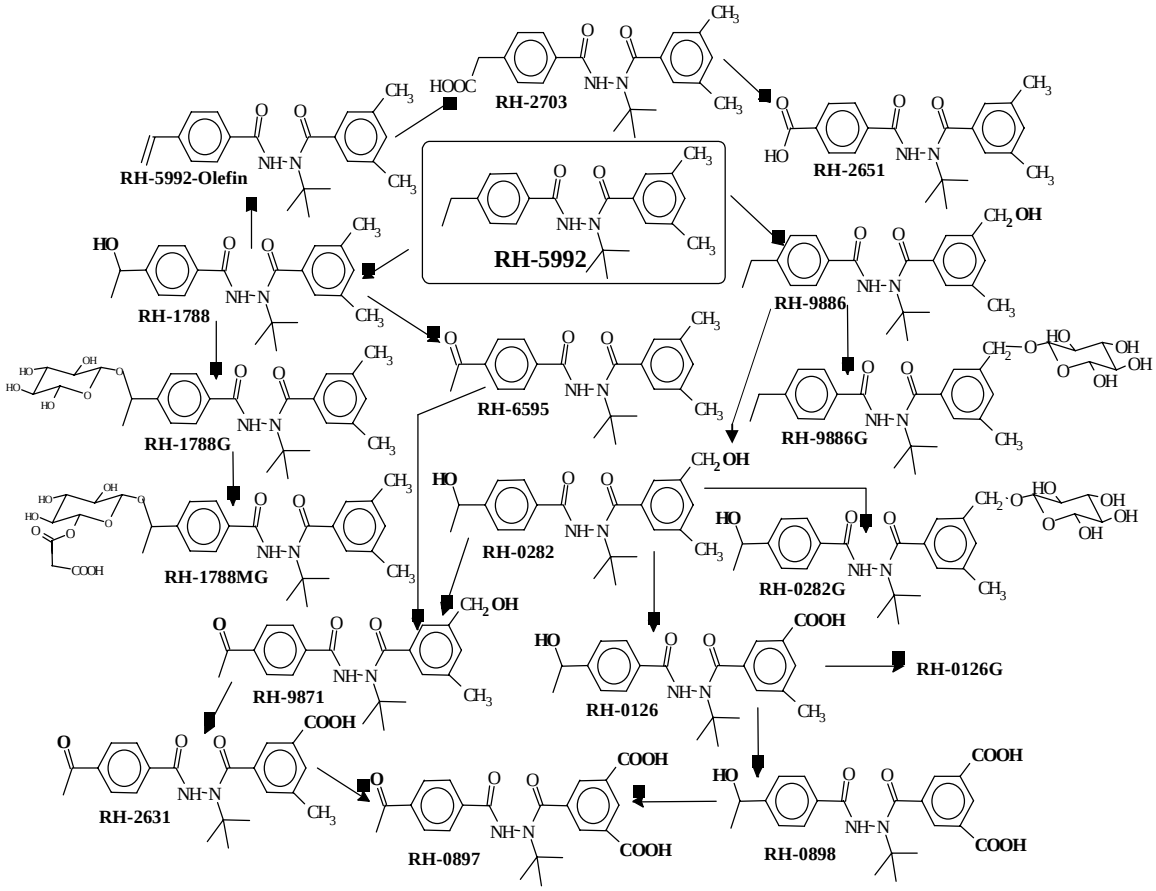
Collards. Residues in the collards planted back at 30 days were about 0.1 mg/kg as tebufenozide, of which 70% was extractable by solvents. The identified residues included the glucose conjugates of RH-1788 as well as small amounts of RH-9886 and 0282; altogether 42% of the TRR. The main individual component was the olefin RH-9841 (15%), in which the ethyl group on the A-ring had been converted into a vinyl substituent. Hydrolysis with cellulase enzyme liberated small quantities of several metabolites such as RH-1788, RH-6595, RH-9886, RH-2631 and RH-0282. The parent compound was undectable. Residues in 250-day collards were low and no single component was detected above 0.01 mg/kg.

Turnips. Residues were about 0.1 mg/kg in roots and 0.5 mg/kg in tops at 30 DAT plant-back, and had decreased to less than 0.01 mg/kg in the roots at the later intervals. In tops 89% and in roots 76% of the residue of 30-day plant-back samples was extractable by organic solvents, and was partitioned into CH_2Cl_2, EtOAc and MeOH before analysis. Low levels of a large number of metabolites were found, which resulted from the oxidation of tebufenozide. About 14% of the residues in both root and tops consisted of the sugar conjugates of RH-1788 that were also found in the wheat samples. The parent compound accounted for 6.7% of the TRR (0.03 mg/kg) in the tops, and was the main compound at 20% of the TRR (0.02 mg/kg) in the roots. RH-1788, RH-6595, RH-0282, RH-9886, RH-2703, RH-0875, RH-2631 and RH-9871 were also identified by a combination of HPLC and TLC and accounted for the remaining residue, each <5% of the TRR.

In summary, the main residues in the wheat samples were RH-1788 and its sugar conjugates. Only the turnip roots had a significant percentage of the residue as tebufenozide. The leafy crop

collards contained mainly sugar conjugates of RH-1788 and tebufenozide-olefin (RH-9841). On the basis of the results of the confined and field rotation studies, the significant residues were identified as tebufenozide, the alcohol metabolite RH-1788, and tebufenozide-olefin (RH-9841). Proposed metabolic pathways are shown in Figure 1.

Figure 1. Metabolic pathways of tebufenozide (RH-5992) in rotational crops.



Note: In the text, RH-5992-olefin is also referred to as RH-9841.

The metabolites found in rotational crops were similar to those identified in the crop metabolism studies reviewed by the 1996 JMPR, except that the parent compound was a minor component or undetectable, and large amounts of sugar conjugates were formed from the alcohol metabolites. Most of the metabolites found in rotational crops were also identified in rats, except the conjugates. In soil, the parent compound and RH-6595 were identified.

Two US field rotational crop studies each consisted of two trials, one in Tulare County, CA and the other in Willacy County, TX. One control and one treated plot were planted with leaf lettuce as a primary crop in each trial. Test plots were sprayed four times with a foliar-ground application of tebufenozide at 0.28 kg ai/ha ± 5%, 234 to 281 l/ha, per application at intervals of 9 to 12 days (Dong, 1998, 1999). The leaf lettuce was removed at maturity and the plots, divided into unequal subplots, were prepared for rotational planting according to normal agronomic practices. Rotational crops were then planted 30 and/or 120 ± 2 days after the last treatment (DAT) as shown in Table 3 below.

Table 3. Tebufenozide field rotation studies.

Both studies		Study A (34P-95-69)		Study B (34P-95-104)
Primary crop	Rotational crop group	30 DAT CA and TX	120 DAT CA and TX	30 DAT CA and TX
Leaf lettuce[1]	Leafy vegetables	Leaf lettuce	Leaf lettuce	Leaf lettuce
	Root crops	Radish	Radish	Radish
	Cereal Grains	Wheat	Wheat/sorghum	Sorghum
	Onion	Bulb onion	Green onion	Green onion
	Fruiting vegetables		Green pepper	Green pepper
	Cucurbits		Squash	Squash
	Legumes		Soya bean[2]	Soya bean

[1] 4 applications at 0.28 kg ai/ha.
[2] The soya beans at the TX site were replanted at 134 DAT.

One sample of each crop from the control plots and two from the treated plots were collected at normal harvest maturity, except forage, cereal grains and legume vegetables which were collected at appropriate growing stages.

The high-moisture samples, leaf lettuce, radish (tops and roots), squash, onions (green and bulb), and green peppers were processed and analysed for residues of tebufenozide and its olefin metabolite RH-9841 - the significant residues found in the confined study on rotational crops by Sharma and Bergin (1996a). Samples were analysed by the preliminary analytical method TR 34-97-91 (Deakyne, 1997) with LC-MS quantification. The LOQ was 0.01 mg/kg, the lower limit of detection (LOD) 0.003 mg/kg, and the sampling-to-analysis interval (SAI) 1-1.6 years (350 to 590 days).

No residues of tebufenozide or RH-9841 above the LOQ were observed in the 30-day plantings of any of these crops (Table 4).

Table 4. Residues in 30-day plant-back high-moisture rotational crops at sites in California and Texas, USA.

Group	Crop	Sample	No. of trials	Tebufenozide	RH-9841
Leafy vegetables	Leaf lettuce	Leaves	4	<LOQ	<LOD
Root crops	Radish	Top and root	4	<LOD	<LOD
Fruiting vegetables	Green bell pepper	Fruit	2	<LOD	<LOD
Cucurbit vegetables	Squash	Fruit	2	<LOD	<LOD
Onion	Green onion Bulb onion	Green onion Bulb onion	2 2	<LOQ <LOD	<LOD <LOD

For tebufenozide and RH-9841 LOQ is 0.01 mg/kg, and LOD 0.003 mg/kg.

Low-moisture crop samples (wheat, sorghum, and soya beans) were analysed for residues of tebufenozide and its alcohol metabolite RH-1788 using the preliminary analytical method TR 34-98-149 (Choo, 1998a) with LC-MS and/or LC-MS-MS quantification that measures residues of tebufenozide and RH-1788 in low-moisture crops with an LOQ of 0.02 mg/kg and an LOD of 0.006 mg/kg. Overall average recoveries from fortified crop samples (n=50) were 88.4% ± 8.1% for tebufenozide, and 79.2% ± 9.4% for RH-1788; the average recoveries at the LOQ level (0.02 mg/kg) at both sites in both studies (n=16) were 92.4% ± 9.7% for tebufenozide and 80.6% ± 11.7% for RH-1788. Samples were stored for 2-2.6 years before analysis (SAI of 718 to 954 days). The results are shown in Table 5.

No residues of tebufenozide above the 0.02 mg/kg LOQ were observed in any wheat, sorghum or soya bean components planted at 30 DAT, and none of RH-1788 above its 0.02 mg/kg LOQ in the grain or seed fractions, but residues of the latter were 0.28 mg/kg, 0.12 mg/kg, and 0.03 mg/kg in wheat straw and hay and soya bean forage respectively.

Table 5. Residues in 30-day plant-back low-moisture rotational crops.

Group	Crop	Sample	Site	No. of samples	Tebufenozide (mg/kg)	RH-1788 (mg/kg)	Total residue[1] (mg/kg)
Cereal Grain	Wheat	Grain	CA	2	<LOD	<LOD	<LOQ
			TX	2	<LOD	<LOD	<LOQ
		Forage	CA	2	<LOD	<LOD	<LOQ
			TX	2	<LOD	<LOD	<LOQ
		Hay	CA	2	<LOD	0.12	0.14
			TX	2	<LOD	0.034	0.053
		Straw	CA	2	<LOQ	0.28	0.30
			TX	2	<LOD	0.062	0.079
	Sorghum	Grain	CA	2	<LOD	<LOD	<LOQ
			TX	2	<LOD	<LOQ	<LOQ
		Forage	CA	2	<LOD	<LOD	<LOQ
			TX	2	<LOD	<LOD	<LOQ
		Hay	CA	2	<LOD	<LOD	<LOQ
			TX	2	<LOD	<LOD	<LOQ
		Straw	CA	2	<LOD	<LOD	<LOQ
			TX	2	<LOD	<LOD	<LOQ
Legume	Soya beans	Grain	TX	2	<LOD	<LOD	<LOQ
		Forage	CA	1	<LOD	0.033	0.052
			TX	2	<LOQ	<LOQ	<LOQ
		Hay	TX	2	<LOD	<LOD	<LOQ

[1] As parent equivalent, LOQ 0.02 mg/kg, LOD 0.006 mg/kg (tebufenozide and RH-1788)

Environmental fate in water/sediment systems

No additional information was provided

METHODS OF RESIDUE ANALYSIS

Analytical methods

Rotational crops. Tebufenozide and its metabolites RH-9841 and RH-1788 can be determined in rotational crops by the enforcement method TR 34-99-10 (Choo, 1999), based on the preliminary methods TR 34-97-91 (Deakyne, 1997) for high-moisture crops, and TR 34-98-149 (Choo, 1998a) for low-moisture crops.

For high-moisture crops such as root and leafy vegetables tebufenozide and RH-9841 are extracted by blending with acidic methanol. Sodium chloride solution is added and the extract is partitioned with hexane to remove oils. Residues are then partitioned into methylene chloride. The methylene chloride layer is evaporated to dryness and the residues are cleaned up on a basic alumina column. A carbon solid-phase extraction tube clean-up is also sometimes used as an optional additional step. For quantification an isocratic HPLC system with a C-18 column is used with negative ion MS detection of the 351 ion for tebufenozide and the sum of the 349 and 385 ions for RH-9841. The average recoveries were 95 ± 10% for tebufenozide and 88±12% for RH-9841, with a demonstrated LOQ of 0.01 mg/kg for both analytes.

For low-moisture rotation crops such as cereal grains tebufenozide and RH-1788 are extracted from the grain by refluxing, and the remaining non-grain fractions by blending or shaking, with acidic methanol. The sample extract is initially purified by the two liquid/liquid partitions described for high-moisture crops, then on a silica column. Soya beans and sorghum grain are cleaned up further by carbon SPE (solid-phase extraction) and non-grain fractions by phenyl SPE. Quantification of the residues is by gradient HPLC on a C-8 column. Negative ion MS detection is used to monitor the 351 ion for tebufenozide and the 367 ion for RH-1788. Average recoveries were 89 ± 10% for tebufenozide and 78 ± 11% for RH-1788, with a demonstrated LOQ of 0.02 mg/kg for both analytes. For confirmation of the residues, MS-MS can be used as an alternative detector, monitoring the 149 daughter ion of both analytes.

Citrus fruit. The method for the determination of tebufenozide in citrus fruits and their processed fractions, TR 34-00-09, is described by Choo (2000). It is derived from the preliminary methods TR 34-96-184 (Meng and Choo, 1996) for citrus and TR 34-97-119 (Choo, 1997) for processed fractions. Residues are extracted from whole fruit, juice and dry pulp by blending with methanol/0.1N HCl (90:10). A salt solution is added to the extract, which is then partitioned with hexane to remove wax, oil and hexane-soluble interferences. Citrus oil samples are partitioned directly with methanol/HCl/hexane. The methanol extract is then diluted with water and partitioned with dichloromethane. The dichloromethane layer is concentrated and further cleaned up successively on carbon and C-18 SPE columns. Analysis of the final extract is by HPLC with UV detection. The LOQ was 0.02 mg/kg, with average recoveries of 98 ± 7.3% for fruit, 98 ± 11% for juice, 90 ± 13% for dry pulp and 92 ± 9.1% for oil. A confirmatory method uses the same extraction and purification procedure with HPLC-MS for quantification of residues in whole fruit, juice and dry pulp and HPLC-MS-MS for oil.

Lettuce. The method TR 34-94-90 for the determination of tebufenozide residues in grapes by GLC (Mellet, 1993), which was evaluated by the 1996 JMPR , was validated for lettuce by Quintelas (2000). The average recovery was 96% ± 6% with an LOQ of 0.02 mg/kg.

Vegetables. The revised enforcement method for the determination of residues of tebufenozide in vegetable crops (TR 34-98-193) described by Chen *et al.* (1998) is similar to 34-93-119 reported by Deakyne (1993) and evaluated by the 1996 JMPR. It includes a new HPLC-MS confirmation of the residues detected and a revised calculation of fortification recoveries. These varied according to the sample but mean recoveries were above 80% from lettuce, cabbage, spinach, mustard greens, broccoli and celery, and overall 85 ± 9%. An LOQ of 0.01 mg/kg has been demonstrated for all samples except celery, which has an LOQ of 0.05 mg/kg. Residues determined by HPLC-UV were confirmed by HPLC-MS.

Sugar cane and its processed fractions. In method TR 34-97-115 (Filchner and Deakyne, 1997) residues of tebufenozide are extracted from stems and stalks, molasses, raw sugar and refined sugar by blending samples with acidic methanol/water (methanol/0.1 N aqueous HCl, 90:10). A salt solution is then added to the extract, which is partially purified by liquid-liquid partition, first with hexane (which is discarded), then with methylene chloride. The methylene chloride layer is concentrated and residues are further purified by carbon SPE followed by chromatography on basic alumina. HPLC on a C-18 column with UV detection is used for quantification. The LOQ was 0.01 mg/kg for all samples with average recoveries of 87 ± 8.4%. Confirmatory analysis is by HPLC-MS, with negative monitoring of the 351 ion.

Pecans. TR 34-96-198 (Cui, 1996) is an updated version of method TR 34-95-20 (Cui and Deakyne, 1994) evaluated by the 1996 JMPR, which describes several necessary precautions in the clean-up procedures. Before the Alumina-B open column clean-up, the extract must be completely dried before reconstitution. In the optional carbon solid-phase extraction the SPE tube must not be allowed to go

dry between the addition of eluents. Finally, the control correction factor was removed from the fortification recovery calculation. The LOQ of 0.01 mg/kg remained unchanged.

Oilseed and process fractions. Method TR 34-96-135 for cotton seed and processed fractions (Wu *et al.*, 1996) was used in supervised trials on rape and the processing studies with rape seed and mint. Residues of tebufenozide are extracted from whole cotton seed and its processed fractions by blending with methanol/0.1N HCl (90:10). A salt solution is added to the extract, which is then partitioned with hexane to remove wax, oil and hexane-soluble interferences. The methanol extract is diluted with water and partitioned with dichloromethane. The dichloromethane layer is concentrated and further cleaned up by chromatography on basic alumina. An additional silica column clean-up step is added for gin trash and a final clean-up by carbon SPE follows. Analysis of the final extract is by HPLC with UV detection. The LOQ was 0.01 mg/kg. Average recoveries were 98 ± 14% for whole cotton seed, 97 ± 11% for meal, 94 ± 11% for hulls, 94 ± 20% for refined oil and 88 ± 16% for gin trash. A confirmatory method uses the same extraction and purification procedure with HPLC-MS for quantification.

Animal commodities. The analytical method TR 34-96-109 (Burnett *et al.*, 1996) described below is based on the preliminary methods TR 34-95-98 for milk (Choo *et al.*, 1996b), TR 34-95-160 for muscle and kidney (Chen *et al.*, 1996), TR 34-95-159 for liver (Filchner *et al.*, 1995), and TR 34-95-161 for fat (Choo *et al.*, 1996a). Method TR 34-96-109 determines tebufenozide in all samples, and its metabolites RH-9886 in muscle and kidney, RH-0282 in milk, muscle and kidney, fatty acid conjugates of RH-9886 in milk and fat, and RH-2703 in liver. RH-9526, the stearic acid conjugate of RH-9886, was used to generate recovery data for the fatty acid conjugates through the method.

Milk samples are blended with methanol containing 10% water and filtered, and the filtrate divided into two equal portions. Residues of RH-9526 (and other fatty acid conjugates of RH-9886) are determined in the first portion after refluxing with hydrochloric acid for 2 hours to effect hydrolysis to the free alcohol. A hexane partition then removes fat contaminants. After adding aqueous sodium chloride, the residues of RH-9886 are partitioned into methylene chloride, which is evaporated to dryness and the residue cleaned up on a carbon SPE column. Residues of tebufenozide and RH-0282 are determined in the second portion of the milk filtrate, which is initially cleaned up by partitioning with hexane. The aqueous extract is then concentrated and the residues partitioned into methylene chloride. The methylene chloride layer is evaporated to dryness and the residue is cleaned up on a basic alumina column. The analytes in the two final extracts are determined by isocratic HPLC with a C-18 column and UV detection. Average recoveries were 84 ± 10% for RH-9526, 88 ± 12% for tebufenozide, and 90 ± 8.4% for RH-0282, with a demonstrated LOQ of 0.01 mg/kg for all three analytes.

Muscle and kidney samples are blended with methanol containing 10% 0.1 N hydrochloric acid and filtered. Sodium chloride solution is added and the extract partitioned with hexane to remove non-polar contaminants. After concentration and addition of additional sodium chloride, the residues are partitioned into methylene chloride. The methylene chloride layer is evaporated to dryness and the residue cleaned up on basic alumina and carbon SPE columns. The analytes in the final extract are determined by isocratic HPLC as before. Average recoveries were 87 ± 12% for tebufenozide, 93 ± 11 % for RH-9886 and 88 ± 11% for RH-0282, with a demonstrated LOQ of 0.02 mg/kg for all three analytes.

+ are homogenized with methanol/0.5 N hydrochloric acid (70:30) and centrifuged. After addition of aqueous sodium chloride solution, residues of tebufenozide and RH-2703 are partitioned into methylene chloride. RH-2703 is then partitioned into aqueous sodium bicarbonate solution. The methylene chloride fraction containing tebufenozide is concentrated to dryness and cleaned up on a basic alumina column. An optional carbon SPE step is also described for samples which require additional clean-up. The sodium bicarbonate fraction is acidified with 1 N hydrochloric acid and the free acid extracted into methylene chloride. This is evaporated to dryness and the residue cleaned up on a silica gel column. Analysis of the two final extracts is by HPLC as before. Average recoveries

were 93 ± 11% for tebufenozide, and 84 ± 11% for RH-2703, with a demonstrated LOQ of 0.02 mg/kg for both analytes.

Fat samples are blended with a mixture of methanol, water and concentrated hydrochloric acid (120:40:15). The mixture is then refluxed for two hours to hydrolyse the fatty acid conjugates of RH-9886 to the free alcohol. After cooling, the extract is filtered and partitioned with hexane to remove non-polar contaminants. The filtrate is treated with sodium chloride solution and residues of tebufenozide and RH-9886 are partitioned into methylene chloride. The methylene chloride fraction is washed with aqueous sodium bicarbonate solution, then concentrated to dryness. The residue is cleaned up on a carbon SPE column and the extract is chromatographed on basic alumina, separating tebufenozide from RH-9886. The eluates containing each analyte are concentrated to dryness and taken up in different mobile phase mixtures of acetonitrile and water. Quantification of the analytes in each final extract is by HPLC as before. Average recoveries were 89 ± 9.8% for tebufenozide and 78 ± 9.1% for RH-9526, with a demonstrated LOQ of 0.02 mg/kg for both analytes.

Confirmatory HPLC methods for all samples used modified mobile phases together with MS detection. In these methods, the negative ions monitored were 351, 367 and 383 for tebufenozide, RH-9886 and RH-0282 respectively. Detection of RH-2703 was by positive ion monitoring of the 383 ion.

Stability of pesticide residues in stored analytical samples

Wheat. Samples from the rotational crop study (Sharma and Bergin, 1996a) were stored for approximately 4 years, and the stability of residues during freezer storage was examined as part of the studycby comparing the TLC profile of a straw sample analysed after two years of storage with that of the same sample after 4 years' storage. The major components in straw were RH-6595, RH-1788 and its glucose conjugate. These represented respectively 14, 26 and 18% of the TRR after 2 years, and 11, 21 and 19% after 4 years.

The composition of the residue in extracts of forage stored for 4 years (which mainly contained the glucose and malonylglucose conjugates of RH-1788) was comparable to that of the same extracts stored for 4 years and 7 months.

Rice straw and grain. The storage stability of tebufenozide and its metabolites RH-1788, RH-6595 and RH-9886 in rice was examined by Sharma and Bergin (1996b). Samples of rice straw and grain from a rice metabolism study initiated in 1989 were analysed for the first time in 1991 i.e. after 2 years of storage (Randazzo, 1992). In 1996, these samples were re-analysed after another 5 years of frozen storage. Subsamples of each field sample were re-extracted and analysed by methods identical to those used in the metabolism study. It was found that tebufenozide was still the main compound, although its proportion of the TRR decreased slightly from 77.9 to 74.8% in rice straw and from 49.5 to 47.5% in rice grain. It was probably mainly converted to RH-9886, which approximately doubled from 1.0 to 1.9% of the TRR in straw and from 1.1 to 2.5% in grain. The proportion of the metabolites RH-1788 and RH-6595 decreased slightly too, in both straw and grain. A small amount of conjugated RH-1788 was identified in 1996 at the same level as in 1991, but then as one of the low level unknown components. The profile remained essentially the same in that by far the main component was tebufenozide, while the proportions of the metabolites were less than 5% each (except RH-1788 in rice grain, which was less than 10%). This stability study does not cover the first two years of storage.

Green onions (RH-9841). A study was conducted by Graves (2000b) to assess the frozen storage stability of RH-9841 in green onions to support the residue data for the rotational crop study (Doug, 1998). Green onion samples spiked with 1.0 mg/kg of RH-9841 on two dates 3 months apart and stored below -10ºC were analysed by the preliminary analytical method for rotational crops TR 34-97-91 (Deakyne, 1997). The sample to analysis intervals (SAIs) for all high-moisture crop samples in

the field rotational crop study (Dong, 1998) ranged from 350 to 590 days (less than 20 months). RH-9841 was stable during 24 months of frozen storage in green onion samples. There was a slight decrease (10%) in the corrected daily recovery over the 24 months of storage.

Citrus oil (Graves, 2000a). Commercial orange oil samples spiked with 1.0 mg/kg of tebufenozidewere stored at about -20ºC. Periodically one control, 2 fresh fortifications, and 3 aged fortifications were analysed by method TR 34-97-119 (Choo, 1997). There was no decrease in the recovery over the 15 months of storage.

Lettuce. 20 g samples of homogenized head lettuce were fortified with tebufenozide at a concentration of 1 mg/kg and stored in a freezer at -15 ± 10°C for 36 months (Choo, 1998b). Samples were analysed before storage and at various intervals by method TR 34-93-119 (Deakyne, 1993). Tebufenozide was found to be stable for the 36 months.

Animal commodities. Control samples of bovine milk, meat, liver and fat were fortified with tebufenozide and its relevant metabolites for each sample at a concentration of 1 mg/kg and stored in a freezer at -15 ± 10°C for 8 months (Choo, 1996). In addition to tebufenozide, milk was fortified with RH-0282 and RH-9526, meat with RH-9886 and RH-0282, liver with RH-2703, and fat with RH-9526. Samples were analysed before storage and then at various intervals by method TR 34-96-109 (Burnett, *et al.*, 1996). No analytes showed any signs of degradation. Residues were stable in the milk, liver, meat and fat samples for a minimum of 192, 203, 182 and 145 days respectively.

Blueberries, raspberries, cranberries, turnip roots and foliage, rape seed and processed fractions, mint and mint oil. Dorschner and Breuninger (1998a-f) conducted stability studies in conjunction with residue trials on these and the results are shown in Table 6. Recoveries are uncorrected for concurrent analytical recoveries, since these were not measured except in cranberries. For comparison, the (general) method recovery at approximately the same fortification level is shown as the 0-day SAI.

Table 6. Stability of residues in frozen storage (Dorschner and Breuninger, 1998).

Crop	Sample	Longest SAI in supervised trials	Fortification (mg/kg)	No.	SAI of sample (days)	Recovery (%)	Ref
Blueberry	fruit	186	1.07	1	0	97.2	1998d
			1.07	3	189	87.3	
Raspberry	fruit	305	0.99	4	0	101.6	1998c
			1.07	3	322	87.5	
Cranberry	fruit	127	2.2	2	0	85.3	1998a
			2.2	2	30	91	
			2.2	1	30 (fresh fort.)	91	
Turnip	roots	259	1.07	4	0	92.7	1998b
			1.07	3	279	89.5	
	tops	244	1.07	4	0	100.4	
			1.07	3	279	84.0	
Rape	seed	231	1.07	4	0	86.3	1998f
			1.07	3	236	77.9	
	meal	68	1.07	3	0	85.8	
			1.07	3	90	80.7	
	oil	68	1.07	3	0	90.7	
			1.02	3	83	83.1	
Mint	foliage	200	1.07	4	0	89.0	1998e
			1.07	3	279	70.2	
	oil	273	1.02	4	0	97.1	
			1.07	3	285	90.6	

USE PATTERN

The Meeting received updated information on the registered uses of tebufenozide. Table 7 shows only the approved GAP for the crops evaluated. Application intervals generally vary between 7 and 21 days.

Table 7. Registered uses of tebufenozide.

Crop	Country	Form	Application					PHI, days	Comments
			Method	Growth stage	Rate kg ai/ha	Spray conc kg ai/hl	Max no.		
Citrus fruits									
	Algeria	SC	high volume	ripening of fruits	0.19	0.019	4	21	
	Italy .	SC	high/low volume			0.017-0.019*	2	14	c2
	Morocco	SC	high volume	ripening of fruits	0.18	0.018	4	45	
	Portugal (.)	SC	high volume airblast	from young growing shoots		0.0144-0.018	2	7	c2
	Tunisia	SC	high volume	ripening of fruits	0.18	0.018	4	21	
	Spain .	SC	high volume			0.0144-0.018	2	14	c1
Stone fruits									
Stone fruits excl. cherries	New Zealand .	WP	high/low volume	from flowering	0.12		4	14	a, c4
Berries and small fruits									
Bush and cane berries (excl. cranberries)	USA	SC	Ground or aerial appl		0.07-0.28			14	a
Cranberries	USA	SC	Ground or aerial appl		0.28			30	a
Grapes	Algeria	SC	high volume	ripening of fruits	0.144	0.0144	3	21	
Grapes	Australia .	WP	high/low volume	from pre-flowering onwards		0.006*		21	a, c3
Grapes	France .	SC	airblast	ripening berries	0.144			21	
Grapes	Germany	SC	high volume	after blossom		0.012		28	c3
Grapes	Italy .	SC	high/low volume	from pre-flowering onwards		0.0144*		30	c3
Grapes	New Zealand .	WP	high/low volume	14-21 days and 1 day pre-bunch closure	0.12	0.006*	2	28	a
Grapes	Portugal (.)	SC	medium /high volume	beginning of ripening	0.144	0.0144*		14	
Grapes	Slovenia	SC	high volume	bunch closing	0.144	0.0144	2	21	
Grapes	Spain .	SC	high/low volume	ripening of fruits	0.144	0.012-0.0144*	4	21	
Grapes	Switzerland .	SC	high volume	after blossom	0.18	0.012	2	none	
Grapes	Tunesia	SC	high volume	ripening of fruits	0.144	0.0144	3		
Assorted tropical and sub-tropical fruits – inedible peel									

Crop	Country	Form	Application					PHI, days	Comments
			Method	Growth stage	Rate kg ai/ha	Spray conc kg ai/hl	Max no.		
Avocado	New Zealand .	WP	high/low volume	from pre-flowering	> 0.12	0.006*	4	21	a, c4
Brassica vegetables									
	Switzerland	SC	high volume			0.012	2	14	
	USA	SC	ground or aerial appl	from young crop/small plants	0.105-0.14			7	b
Cabbage	Slovenia	SC	high volume	directly after hatching	0.043-0.076	0.0096-0.017	1	14	
Fruiting vegetables other than cucurbits									
	USA	SC	ground or aerial appl.	from young crop/small plants	0.105-0.28			7	b, c2
Peppers, tomatoes and egg plant	Belgium .	SC	spraying		0.18	0.018-0.024	2	3	c1
Peppers and tomatoes	Spain .	SC	high volume			0.0144-0.018	3	3	
Tomatoes	Algeria	SC	high volume	ripening of fruits	0.144-0.19	0.0144-0.019	5	21	
Leafy vegetables including leafy brassica									
	USA	SC,	ground or aerial appl	from young crop/small plants	0.105-0.14			7	b
Lettuce, spinach	Switzerland	SC	high volume			0.012		14	
Root and tuber vegetables									
Turnips	USA	SC,	ground or aerial appl	from young crop/small plants	0.105-0.140			7	b
Stalk and stem vegetables									
Celery, celture, rhubarb, cardoon	USA	SC	ground or aerial appl	from young crop/small plants	0.105-0.140			7	b
Grasses for sugar or syup production									
Sugar cane	USA	SC	ground or aerial appl.		0.105-0.28			14	b, c2
Tree Nuts									
Tree nuts excl. pecans	USA	SC	ground or aerial appl		0.28-0.53			14	a, c2
Pecans	USA	SC	ground or aerial appl		0.14-0.28			14	a, c2
Walnuts	France .	SC	airblast	ripening of fruits		0.0144		21	
Walnuts	Spain	SC	high volume	ripening of fruits	0.29	0.0144		21	
Oilseed									
Rape seed (Canola)	USA	UL	ground or aerial appl	from young crop/small plants	0.14-0.28			14	b
Herbs									
Mint	USA	SC	ground or aerial appl.	from young crop/small plants	0.105-0.28			14	c2

. label available

(.) only a translation or summary of label in English available

* concentration for normal (high) volume application. For concentrate (low volume) spraying, adjust dilution rate accordingly (use same rate of product per hectare as in normal volume applications). In Australia do not use at rates greater than 5 times the dilute spraying rate

[1] maximum total application per season is 2.1 kg ai/ha

a) do not graze any treated area, do not feed treated crops to stock.

b) rotational crop restrictions: crops for which use of tebufenozide is registered no restrictions; all other crops 30 days re-cropping interval.

c) interval between applications: c1= 7 days, c2= 10 days, c3= 2 weeks, c4= 3 weeks

d) maximum total application per season 2.1 kg ai/ha

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received residue data from supervised field trials on citrus fruits, stone fruit, berry crops, cranberries, avocado, fruiting vegetables, turnip greens and roots, sugar cane, rape seed (canola) and mint, and supplementary data on residues in grapes, head lettuce, pecans, almonds and macademia nuts. Because of newly approved registered used previously reviewed trials on cabbage, broccoli, head and leaf lettuce, spinach, mustard greens, Chinese kale and celery were re-evaluated. Residues have not been corrected for analytical method recoveries except where indicated. Trials are listed in the following Tables, where residues resulting from trials according to GAP are underlined. All applications were with ground equipment.

Table 8	Oranges	Table 24	Head lettuce
Table 9	Lemons	Table 25	Head lettuce (JMPR 1996)
Table 10	Grapefruit	Table 26	Leaf lettuce (JMPR 1996)
Table 11	Mandarins	Table 27	Spinach (JMPR 1996)
Table 12	Peaches	Table 28	Mustard greens (JMPR 1996)
Table 13	Nectarines	Table 29	Chinese kale (JMPR 1996)
Table 14	Blueberries	Table 30	Turnip greens
Table 15	Raspberries	Table 31	Turnip roots
Table 16	Cranberries	Table 32	Celery (JMPR 1996)
Table 17	Grapes (JMPR 1996)	Table 33	Sugar cane
Table 18	Grapes	Table 34	Pecans (JMPR 1996)
Table 19	Avocado	Table 35	Pecans
Table 20	Cabbage (JMPR 1996)	Table 36	Almonds
Table 21	Broccoli (JMPR 1996)	Table 37	Macadamia nuts
Table 22	Tomatoes	Table 38	Rape
Table 23	Peppers	Table 39	Mint

Citrus fruits

Summer oil or other adjuvant with strong penetrating properties is mixed into the holding tank unless otherwise noted.

Oranges. The available data on whole oranges from Australia, Italy, Spain, and the USA are shown in Table 8. In field trials in Australia from 1994 to 1999 (Arlett, 2000) 2 to 3 applications of a WP formulation at the proposed GAP rate of 0.006 kg ai/hl and at exaggerated rates of 0.012 and 0.024 kg ai/hl were made to the foliage at approximately 14 day intervals (except in trial RTL 446/96, in which the intervals between applications were 45 and 21 days). Residues of tebufenozide were determined according to the method of Holzwarth and Schuld (1993a), modified so that the methylated derivative of tebufenozide was determined by GC-MS rather than GLC with an NPD with an LOQ of 0.02 mg/kg, except in trial DJR/171/00 for which the Agrifood Technology method TP/215/990201 (Bayer) was used (GLC/NPD; LOQ 0.05 mg/kg). The SAI ranged from 2 to 9 months.

In ten trials in Italy and Spain in 1996 and 1997 (Balluff, 1997a, 1999) two foliar applications of an SC formulation of tebufenozide at 0.018 kg ai/hl, which corresponds to approved GAP, were made , with an interval of 13-16 days. In each of the trials, oranges were collected on day 0 and 13 or 14 days after the second application. In addition in 3 of the 5 1996 trials, samples were also taken at days 3 and 7. Day 13/14 samples were peeled as part of the processing. In 1996 the peel, pulp and whole fruit samples were analysed by method TR 34-96-184 (HPLC-UV) (Meng and Choo, 1996) with an LOQ of 0.02 mg/kg for whole fruit and pulp, and 0.04 mg/kg for peel which was subjected to an additional alumina column clean-up. In the 1997 trials, whole fruit, peel and pulp samples were analysed by the GLC method AL 013/92-0 (Schuld and Holzwarth, 1994) with an LOQ of 0.02 mg/kg. The analytical laboratory made slight modifications to the method and used mass spectrometry for detection. Residues in whole fruit were calculated from the residues found in the peel and pulp samples and the weights of each. The SAI was up to 234 days.

In a further eleven field trials in geographically representative areas of the USA (Koals and Carpenter, 2000) 4 foliar applications of a WP formulation of tebufenozide were made at a rate of 0.34 kg ai/ha + 5%. The first was made in the early season, the second in mid-summer and the third and fourth 28 ± 2 and 14 ± 1 days before harvest. In all the trials, duplicate field samples of oranges were collected at maturity. All samples were analysed for tebufenozide by method TR 34-96-184. The LOQ was 0.02 mg/kg. The SAI ranged from 186 to 547 days.

Table 8. Residues of tebufenozide from supervised trials on oranges in Australia, Italy, Spain and the USA.

Country, year, location, variety	Application				PHI, days	Sample	Residue mg/kg	Ref, trial number
	Form	No	kg ai/ha	kg ai/hl				
Australia 1997, Ramco (SA) Washington Navel	WP	3	[3]	0.006	0 1 5 12	fruit fruit fruit fruit	0.42 0.38 0.42 0.42	Arlett, 2000 DJR 136/98[1]
Australia 1997, Leeton (NSW) Navel	WP	3	0.24	0.006	0 1 7 14	fruit fruit fruit fruit	0.35 0.41 0.37 0.42	Arlett, 2000 JES 542/98[1]
Australia 1994, Cobram (Vic) Valencia	WP	3	0.12	0.006	1 8 15 22	fruit fruit fruit fruit	0.20 0.16 0.20 0.20	Arlett, 2000 RTL 446/96 int: 45, 21 [2]
		3	0.24	0.012	1 8 15 22	fruit fruit fruit fruit	0.64 0.47 0.40 0.35	Arlett, 2000 RTL 446/96 int: 45, 21 [2]
		3	0.48	0.024	1 8 15 22	fruit fruit fruit fruit	0.68 0.63 0.62 0.77	Arlett, 2000 RTL 446/96 int: 45, 21[2]
		2	0.12	0.006	1 8 15 22	fruit fruit fruit fruit	0.17 0.17 0.12 0.07	Arlett, 2000 RTL 446/96 int.: 21 days [2]
		2	0.24	0.012	1 8 15 22	fruit fruit fruit fruit	0.18 0.46 0.32 0.36	Arlett, 2000 RTL 446/96 int.: 21 days[2]
Australia, 1999 Loxton North (SA) Valencia	WP	3	[3]	0.006	0 1 7 14	fruit fruit fruit fruit	0.12 0.14 0.15 0.09	Arlett, 2000 DJR 171/00[1]

Country, year, location, variety	Application				PHI, days	Sample	Residue mg/kg	Ref, trial number
	Form	No	kg ai/ha	kg ai/hl				
		3	[3]	0.012	0 1 7 14	fruit fruit fruit fruit	0.43 0.37 0.39 0.28	Arlett, 2000 DJR 171/00[1]
Italy 1996, Fondi Tarocco	SC	2	0.20, 0.21	0.018	0 14	fruit fruit* peel pulp	0.42 <u>0.25</u> 0.79 <u>0.053</u>	Balluff, 1997a 96I019R
Italy 1996, Catania Navelina	SC	2	0.52, 0.52	0.018	0 3 7 14	fruit fruit fruit fruit* peel pulp	0.48 0.24 0.43 <u>0.78</u> 2.69 <u>0.15</u>	Balluff, 1997a 96I020R
Italy 1996, Carlentini Navelina	SC	2	0.49, 0.48	0.018	0 3 7 14	fruit fruit fruit fruit* peel pulp	0.30 0.47 0.54 <u>0.60</u> 2.02 <u>0.11</u>	Balluff, 1997a 96I021R
Spain 1996, Palacios Navelina	SC	2	0.31, 0.30	0.018	0 3 7 14	fruit fruit fruit fruit* peel pulp	0.61 0.27 0.48 <u>0.43</u> 1.28 <u>0.13</u>	Balluff, 1997a 96S004R
Spain 1996, Lliria Navelina	SC	2	0.40, 0.39	0.018	0 14	fruit fruit* peel pulp	0.84 <u>0.39</u> 1.46 <u>0.021</u>	Balluff, 1997a 96S005R
Italy 1997, Catania Navelina	SC	2	0.54, 0.52	0.018	0 14	fruit fruit* peel pulp	0.29 <u>0.21</u> 0.75 <u>0.03</u>	Balluff, 1999 I97049R
Italy 1997, Lentini Navelina	SC	2	0.56, 0.55	0.018	0 14	fruit fruit* peel pulp	0.47 <u>0.56</u> 1.5 <u>0.13</u>	Balluff, 1999 I97050R
Spain 1997, Torrente Navelina	SC	2	0.62, 0.64	0.018	0 13	fruit fruit* peel pulp	0.56 <u>0.38</u> 1.2 <u>0.04</u>	Balluff, 1999 S97016R
Spain 1997, Anahuir Salustiana	SC	2	0.48, 0.47	0.018	0 14	fruit fruit* peel pulp	0.47 <u>0.48</u> 1.8 <u>0.05</u>	Balluff, 1999 S97017R
Spain 1997, Beniel Newhall	SC	2	0.47, 0.46	0.018	0 14	fruit fruit* peel pulp	0.37 <u>0.36</u> 1.1 <u>0.04</u>	Balluff, 1999 S97018R
USA 1995, LaBelle (FL), Hamlin	WP	4	0.34	0.037	7 14 21 28	fruit fruit fruit fruit	0.42, 0.43 mean 0.42 0.41[4], 0.42[5] 0.17, 0.28 mean 0.22 0.22, 0.28 mean 0.25	Koals 2000 95-0271 + 95-0274[2]
USA 1995, Alva (FL), Valencia	WP	4	0.34	0.037	14	fruit	0.52, 0.43 mean 0.47	Koals 2000, 95-0273[2]

Country, year, location, variety	Application				PHI, days	Sample	Residue mg/kg	Ref, trial number
	Form	No	kg ai/ha	kg ai/hl				
USA 1995, Raymondville (TX) Everhard Navel	WP	4	0.34	0.037	14	fruit	0.12, 0.14 mean 0.13	Koals 2000 95-0279[2]
USA 1996, Porterville (CA) Wahington Navel	WP	4	0.34	0.009	14	fruit	0.15, 0.12 mean 0.14	Koals 2000 96-0082[2]
USA 1996, Windermere (FL) Parson Brown	WP	4	0.34	0.037	14	fruit	0.16, 0.26 mean 0.21	Koals 2000 96-0241[2]
USA 1996, Raymondville (TX) Everhard Navel	WP	4	0.34	0.037	14	fruit	0.17, 0.23 mean 0.20	Koals 2000 96-0272[2]
USA 1996-1997, LaBelle (FL) Hamlin	WP	4	0.34	0.037	14 14 14	fruit peel pulp	0.47, 0.47 mean 0.47 1.04, 0.75 mean 0.88 0.069, 0.093 mean 0.081	Koals 2000 96-0304[2]
USA 1996, LaBelle (FL) Pineapple	WP	4	0.34	0.037	14 14 14	fruit peel pulp	0.29, 0.35 mean 0.32 0.68, 0.56 mean 0.62 0.098, 0.053 mean 0.075	Koals 2000 96-0306[2]
USA 1996, LaBelle (FL) Hamlin	WP	4	0.34	0.037	14	fruit	0.24, 0.46 mean 0.35	Koals 2000 96-0310[2]
USA 1996-1997, Porterville (CA) Navel	WP	4	0.34	0.009	14 14 14	fruit* peel pulp	0.25, 0.30 mean 0.28 0.56, 0.71 mean 0.64 0.082, 0.097 mean 0.089	Koals 2000 96-0336[2]
USA 1996-1997, Rich Grove (CA) Navel	WP	4	0.34	0.008	14 14 14	fruit* peel pulp	0.19, 0.24 mean 0.21 0.26, 0.38 mean 0.32 0.052, 0.11 mean 0.082	Koals 2000 96-0337[2]

* residue in whole fruit is calculated from peel and pulp samples and their respective weights
[1] 1 tree per plot, sampling is required from 4 individual trees. Sample size was about 1 kg instead of min 2 kg
[2] application in absence of a wetting agent or summer oil
[3] sprayed well in excess of run-off
[4] 0.41 is the mean of 2 field samples (0.36, 0.46)
[5] 0.42 is the mean of 2 field samples (0.45, 0.39)

Lemons. The lemon residue data from the USA and Australia are shown in Table 9.

In two field trials in Australia, one in 1997 and the other in 1999 (Arlett, 2000) 3 applications of a WP formulation of tebufenozide at the proposed GAP rate of 0.006 kg ai/hl, corresponding to 0.12 kg ai/ha per application, were made at approximately 14 day intervals to the leaves of the crop . Lemons were collected 0, 1, (3), 7 and 14 days after the last application. Analytical method AL013/92-0 (Holzwarth and Schuld, 1993a) with GC-MS instead of GLC and NPD was used in trial TAB 274/98 (LOQ=0.02 mg/kg), and the Agrifood Technology method TP/215/990201 (GC/NPD; LOQ=0.05 mg/kg) (Bayer) in trial DJR/173/00. Recoveries were low: 72% at 0.4 mg/kg in trial TAB 274/98 and 74% at 0.5 mg/kg in trial DJR/173/00. The SAI was 2 months.

In five field trials in the USA from 1995 to 1997 with a WP formulation of tebufenozide (Koals, 1999a) 4 foliar applications, each at 0.34 kg ai/ha ± 5%, were made by airblast sprayers. The interval between the third and fourth applications was 14 days, and between the first and second ranged from approximately 2 to 6 months. In 3 of the trials in California and the single trial in Florida, duplicate field samples of lemons were collected 13 or 14 days after the last application. In the fourth trial in California, samples were collected 7, 14, 21 and 28 days after the last application. Whole fruit, peel and pulp samples were analysed by method TR 34-96-184 (Meng and Choo, 1996) with an LOQ of 0.02 mg/kg. The SAI ranged from 220 to 520 days.

Table 9. Residues of tebufenozide from supervised trials on lemons in Australia and the USA. All WP formulations.

Country, year, location, variety	Application			PHI, days	Sample	Residue mg/kg	Ref, trial number
	No	kg ai/ha	kg ai/hl				
Australia 1997, Galston (NSW) Eureka Lemon	3	[1]	0.006	0 1 3 7 14	fruit fruit fruit fruit fruit	0.37 0.16 0.24 0.24 0.13	Arlett, 2000 TAB 274/98[2,3]
Australia 1999, Loxton North (SA) Lisbon	3	0.012	0.006	0 1 7 14	fruit fruit fruit fruit	0.48 0.52 0.38 0.41	Arlett, 2000 DJR 173/00[2,4]
USA 1995, Fallbrook (CA) Eureka	4	0.34	0.018-0.025	13	fruit	0.22, 0.21 mean 0.22	Koals, 1999a 95-0258[5]
USA 1995, Porterville (CA) Lisbon	4	0.34	0.008-0.009	14	fruit	0.23, 0.22 mean 0.22	Koals, 1999a 95-0278[5]
USA 1996, Clewiston (FL) Eureka	4	0.34	0.037	14	fruit	0.14, 0.12 mean 0.13	Koals, 1999a 96-0307[5]
USA 1996-1997, Porterville (CA) Lisbon	4	0.34	0.008-0.009	7 14 14 14 21 28	fruit fruit peel pulp fruit fruit	0.30 0.51, 0.32 mean 0.42 0.97, 0.50[6] mean 0.74 0.10, 0.11 mean 0.11 0.33 0.36	Koals, 1999a 96-0342[5]
USA 1996-1997, Porterville (CA) Lisbon	4	0.34	0.009	14 14 14	fruit peel pulp	0.24, 0.47 mean 0.35 0.49, 0.59 mean 0.54 0.070, 0.030 mean 0.050	Koals, 1999a 96-0344[5]

[1] sprayed well in excess of run-off
[2] recovery at the level of found residues was less than 80%. TAB 274/998: R=72% at 0.40 mg/kg (n=2), DJR 173/00: R=74% at 0.5 mg/kg (n=1)
[3] 2 trees per plot, sampling is required from 4 trees
[4] 1 tree per plot, sampling is required from 4 trees. Sample size was about 1 kg instead of min 2 kg
[5] application in absence of a wetting agent or summer oil
[6] average residue of 3 analytical samples (0.720, 0.330 and 0.459 mg/kg)

Grapefruit. The residue data from the USA are shown in Table 10. In six field trials in geographically representative areas of the USA in 1995, 1996 and 1997 (Koals, 1999b) 4 applications of a WP formulation of tebufenozide at 0.34 kg ai/ha ± 5% were made to the leaves of the crop at early season, mid-season and the third and fourth 28 ± 2 and 14 ± 1 days before harvest. In each of the trials, duplicate field samples of grapefruit were collected 13-14 days after the last application. All samples were analysed for tebufenozide by the method of Meng and Choo (1996) with an LOQ of 0.02 mg/kg. The SAI ranged from 277 to 673 days.

Table 10. Residues of tebufenozide from supervised trials on grapefruit in the USA. All 4 applications of a WP formulation.

Year, location, variety	Application		PHI, days	Sample	Residue mg/kg	Ref, trial number
	kg ai/ha	kg ai/hl				
1995, Porterville (CA) Mello Gold	0.34	0.009	14	fruit	0.15, 0.18 mean 0.17	Koals, 1999b 95-0249[1]
1995, Raymondville (TX), Rio Red	0.34	0.035- 0.037	7 14 21 28	fruit fruit fruit fruit	0.047, 0.098 mean 0.072 0.11, 0.095 mean 0.10 0.12, 0.12 mean 0.12 0.093, 0.12 mean 0.11	Koals, 1999b 95-0268[1]
1995, LaBelle (FL) White Marsh	0.34	0.037	14	fruit	0.30, 0.50 mean 0.40	Koals, 1999b 95-0272[1]
1996, Windermere (FL) Ruby Red	0.34	0.037	13	fruit	0.10, 0.075 mean 0.089	Koals, 1999b 96-0263[1]
1996, LaBelle (FL) White Marsh	0.34	0.037	14 14 14	fruit[2] peel pulp	0.27, 0.24 mean 0.25 0.68, 0.81 mean 0.74 0.15, 0.10 mean 0.12	Koals, 1999b 96-0308[1]
1996-1997, Porterville (CA) Mello Gold	0.34	0.009	14 14 14	fruit peel pulp	0.071, 0.065 mean 0.068 0.21, 0.22 mean 0.21 0.019, 0.026 mean 0.022	Koals, 1999b 96-0343[1]

[1] application in absence of a wetting agent or summer oil
[2] residue in whole fruit is calculated from peel/pulp samples and their respective weights

Mandarins. The results of trials in Australia, Spain and Italy are shown in Table 11.

In two trials in Australia in 1996 and 1997 (Arlett, 2000) one and three applications of a WP formulation of tebufenozide at the proposed GAP rate of 0.006 kg ai/hl and at 0.012 and 0.024 kg ai/hl were sprayed in excess to the point of run-off. Foliar applications were made to the crop at approximately 14-day intervals in trial JES 557/98 and at 23- and 19-day intervals in trial MJG 035/97. Single samples of mandarins were collected 1, 7, 14 and 21 days after the last application and residues were determined by the method of Holzwarth and Schuld (1993a) with GC-MS instead of GLC with an NPD. The LOQ was 0.02 mg/kg. The SAI was 5 to 6 months.

In trials in Spain in 1995 (Jousseaume, 1995) two foliar applications of an SC formulation of tebufenozide at the GAP rate of 0.018 kg ai/hl equivalent to 0.31 to 0.51 kg ai/ha were made to the crop at 14-15 day intervals. In most trials, single samples of treated mandarins were collected 0, 7, 14, 21 and 28 days after the last application, and 0, 7 and 14 days after the first. Samples were analysed for tebufenozide by HPLC method TR 34-96-184 (Meng and Choo, 1996). Peel samples were analysed by the same method with an additional clean-up before quantification. The LOQ was 0.02 mg/kg for pulp and fruit and 0.05 mg/kg for peel. The SAI ranged from 363 to 455 days.

In five trials in Italy and Spain in 1996 (Balluff, 1997b) 2 foliar applications of a SC formulation of tebufenozide at 0.017 kg ai/hl, corresponding to GAP, were made to the crop with an interval of 14 days. In each of the trials, treated samples of mandarins were collected 0 and 13 or 14 days after the last application. Day 13/14 samples were peeled as part of the processing. Peel, pulp and whole fruit were analysed by method TR 34-96-184 (Meng and Choo 1996). The LOQ was 0.02 mg/kg for all samples. The SAI ranged from 101 to 271 days.

Table 11. Residues of tebufenozide resulting from supervised trials on mandarins in Australia, Italy and Spain.

Country, year, location, variety	Application				PHI, days	Sample	Residue mg/kg	Ref, trial number
	Form	No	kg ai/ha	kg ai/hl				
Australia 1996, Bundaberg (Qld) Murcott	WP	1	0.18	0.006	1 7 14 21	fruit fruit fruit fruit	0.05 0.05 0.04 0.04	Arlett, 2000 MJG 035/97[1, 2, 3]
	WP	1	0.45	0.012	1 7 14 21	fruit fruit fruit fruit	0.09 0.10 0.09 0.09	Arlett, 2000 MJG 035/97[1, 2, 3]
	WP	1	0.91	0.024	1 7 14 21	fruit fruit fruit fruit	0.17 0.13 0.16 0.17	Arlett, 2000 MJG 035/97[1, 2, 3]
	WP	3	0.22, 0.28, 0.23	0.006	1 7 14 21	fruit fruit fruit fruit	0.08[4] 0.14 0.10 0.15	Arlett, 2000 MJG 035/97[1, 2, 3]
	WP	3	0.35, 0.34, 0.44	0.012	1 7 14 21	fruit fruit fruit fruit	0.14 0.35 0.33[4] 0.19	Arlett, 2000 MJG 035/97[1, 2, 3]
	WP	3	0.94, 0.91, 0.91	0.024	1 7 14 21	fruit fruit fruit fruit	0.34 0.46 0.34 0.17	Arlett, 2000 MJG 035/97[1, 2, 3]
Australia 1997, Leeton (NSW) Imperial	WP	3	0.24	0.006	0 1 7 14	fruit fruit fruit fruit	0.23 0.20 0.34[4] 0.25	Arlett, 2000 JES 557/98[2, 3]
Italy 1996, Belpasso Avana	SC	2	0.48, 0.49	0.017	0 14	fruit fruit[7] peel pulp	1.0[4] <u>0.59</u> 1.8 <u>0.082</u>	Balluff, 1997b 96I022R[5]
Italy 1996, Catania Avana	SC	2	0.49	0.017	0 14	fruit fruit[7] peel pulp	0.62[4] <u>0.30</u> 0.71 <u>0.14</u>[6]	Balluff, 1997b 96I023R[5]
Spain 1995, Sal Alcacer (Valentia) Clementine	SC	1	0.43	0.018	0 7 14	fruit fruit fruit	0.094 0.25 0.37	Jousseaume, 1995 96-0148
		2	0.43, 0.51	0.018	0 7 14 21 28	fruit fruit fruit fruit fruit	0.77 0.48 <u>0.95</u> 0.80 0.62	Jousseaume, 1995 96-0148
Spain 1995, Sal Turis (Valentia) Clementine	SC	1	0.35	0.018	0 7 14	fruit fruit fruit	0.41 0.50 0.45	Jousseaume, 1995 96-0149
		2	0.35, 0.40	0.018	0 7 14 21 28	fruit fruit fruit fruit fruit	0.83 0.79 0.48 <u>0.78</u> 0.42	Jousseaume, 1995 96-0149
Spain 1995, Sal Alcacer (Valentia) Clementine	SC	1	0.31	0.018	0 7 14	fruit fruit fruit	0.62 0.44 0.23	Jousseaume, 1995 96-0150

Country, year, location, variety	Application				PHI, days	Sample	Residue mg/kg	Ref, trial number
	Form	No	kg ai/ha	kg ai/hl				
		2	0.31, 0.35	0.018	0	fruit	0.94	Jousseaume, 1995 96-0150
					7	fruit	0.60	
					14	fruit	0.84	
					21	fruit	0.62	
					28	fruit	0.52	
Spain 1995, Sal Via (Valentia) Clementine	SC	1	0.41	0.018	0	fruit	0.41	Jousseaume, 1995 96-0189
					0	peel	0.97	
					0	pulp	0.12	
					7	fruit	0.37	
					7	peel	1.0	
					7	pulp	0.096	
					14	fruit	0.47	
					14	peel	0.76	
					14	pulp	0.071	
		2	0.41, 0.37	0.018	0	fruit	0.64	Jousseaume, 1995 96-0189
					0	peel	2.2	
					0	pulp	0.18	
					7	fruit	0.67	
					7	peel	2.0	
					7	pulp	0.18	
					14	fruit	0.52	
					14	peel	2.1	
					14	pulp	0.13	
					21	fruit	0.42	
					21	peel	1.4	
					21	pulp	0.18	
					28	fruit	0.60	
					28	peel	1.3	
					28	pulp	0.17	
Spain 1996, Alcala Clemenales	SC	2	0.33, 0.32	0.017	0	fruit	0.42[6]	Balluff, 1997b 96S006R[5]
					13	fruit[7]	0.30	
						peel	0.82	
						pulp	0.092	
Spain 1996, Alcala Oroval	SC	2	0.38, 0.37	0.017	0	fruit	0.74[4]	Balluff, 1997b 96S007R[5]
					13	fruit[7]	0.42	
						peel	1.3	
						pulp	0.076	
Spain 1996, Lliria Clemenales	SC	2	0.40, 0.41	0.017	0	fruit	1.2[4]	Balluff, 1997b 96S008R[5]
					14	fruit[7]	0.60	
						peel	1.9	
						pulp	0.069	

[1] application in absence of a wetting agent or summer oil
[2] recoveries of spiked samples at the level of found residues were <80%. MJG 035/97: 78, 70 and 69% at 0.040, 0.10 and 0.20 mg/kg respectively (n=1), JES 557/98: 67% at 0.20 mg/kg (n=2)
[3] 1 tree per plot, sampling is required from 4 trees. Sample size was about 0.5 - 1 kg, instead of min 2 kg
[4] average of 2 analytical samples
[5] recovery of peel samples spiked at 1.0 mg/kg was 77.4% (n=2)
[6] average of 3 analytical samples
[7] residue in whole fruit calculated from peel/pulp samples and their respective weights

Stone fruits

Peaches. In three field trials in 1996-1998 in New Zealand 3 or 6 applications of a WP formulation of tebufenozide at the GAP rate of 0.12 kg ai/ha and at an exaggerated rate of 0.24 kg ai/ha were made to the leaves of the crop at intervals of 17 to 35 days (Baynon, 1998a,b). From 3-4 days after the last application and every week thereafter single samples of peaches were collected and analysed by method TR 34-95-66 (HPLC-MS) with slight modifications (Deakyne *et al.*, 1995). In the 1996/97

trials the LOQ was 0.01 mg/kg, and in the 1997/1998 trial 0.03 mg/kg. The SAI was up to 4 months. The results in fruit without stone are shown in Table 12.

Table 12. Residues of tebufenozide in fruit without stone resulting from supervised trials on peaches in New Zealand.

Year, Location Variety	Application				PHI days	Residue, mg/kg	Reference/ Comments
	Form.	No.	kg ai/ha	kg ai/hl			
1996-1997, Hawkes Bay Golden Queen	WP	6	0.12	0.006	1 8 15 22	0.36 0.13 <u>0.10</u> 0.10	Baynon, 1998a FSLHBRE02[1]
		6	0.24	0.012	1 8 15 22	1.3 0.86 0.82 0.47	Baynon, 1998a FSLHBRE02[1]
1996/1997 Nelson Golden Queen	WP	6	0.12	0.006	1 8 15 22 29	0.20 0.15 <u>0.14</u> 0.10 0.06	Baynon, 1998a FSLNRE05[1]
		6	0.24	0.012	1 8 15 22 29	0.61 0.54 0.51 0.37 0.23	Baynon, 1998a FSLNRE05[1]
1997/1998 Hastings Elegant Lady	WP	3	0.12	0.006	3 10 17 25 31	0.28 0.13 0.08 <u>0.09</u> 0.03	Baynon, 1998b FSLH/08/98/R[1]
		3	0.24	0.012	10 17 25 31	0.44 0.31 0.28 0.14	Baynon, 1998b FSLH/08/98/R[1]

[1] No soil type or weather data available

<u>Nectarines</u>. The results of trials in New Zealand are shown in Table 13. In three field trials, 1996-1998, 3 or 4 applications of a WP formulation of tebufenozide at 0.12 kg ai/ha were sprayed on the leaves of the crop at 16-35-day intervals (Baynon, 1998a,b). Samples were collected and analysed as in the peach trials, with the same LOQs and an SAI of up to 4 months.

Table 13. Residues of tebufenozide in fruit without stone resulting from supervised trials on nectarines in New Zealand.

Year, location Variety	Application				PHI days	Residue, mg/kg	Reference/ Comments
	Form.	No.	kg ai/ha	kg ai/hl			
1996-1997, Hastings Tasty Gold	WP	4	0.12	0.006	0 7 14	0.19 0.13 <u>0.05</u>	Baynon, 1998a FSLHBR01[1]
		4	0.24	0.012	0 7 14	0.68 0.28 0.19	Baynon, 1998a FSLHBR01[1]

Year, location Variety	Application				PHI days	Residue, mg/kg	Reference/ Comments
	Form.	No.	kg ai/ha	kg ai/hl			
1997-1998, Hastings Fantasia	WP	3	0.12	0.006	3 10 17 25 31	0.34 0.33 <u>0.26</u> 0.09 0.07	Baynon, 1998b FSLH/07/98R[1, 2]
		3	0.24	0.012	3 10 17 25 31	0.58 0.27 0.22 0.17 0.15	Baynon, 1998b FSLH/07/98R[1, 2]
1997-1998, Nelson Red Gold	WP	4	0.12	0.006	0 7 14 21 28	0.32 0.15 <u>0.22</u> 0.14 0.21	Baynon, 1998b FSLH/06/98R[1]
		4	0.24	0.012	0 7 14 21 28	0.68 0.48 0.52 0.56 0.37	Baynon, 1998b FSLH/06/98R[1]

[1] No soil type or weather data available
[2] Residues in untreated samples were 0.12, 0.07, 0.07, 0.08, <0.03 mg/kg, at 3, 10, 17, 25, and 31 days, probably due to spray drift (decrease with time).

Berries and other small fruits

Blueberries. In eight field trials in geographically representative areas of the USA in 1996 (Dorschner and Breuninger, 1998d) (Table 14) 4 foliar applications of a WP formulation of tebufenozide were made to the crop at 0.29 kg ai/ha (maximum GAP) ± 5% at intervals of approximately 14 days (in the trial in Ohio each application was 15% less). Replicate samples were collected 12-15 days after the last application, and analysed by method TR 34-94-40 (Deakyne *et al.*, 1994). Slight modifications were made during analyses. The LOQ was 0.005 mg/kg. The SAI ranged from 121 to 186 days. The storage stability of tebufenozide was demonstrated in blueberries stored frozen for 189 days.

Table 14. Residues of tebufenozide resulting from supervised trials on blueberries in the USA in 1996 (Dorschner and Breuninger, 1998d).

Location variety	Application				PHI days	Residue, mg/kg	Report no.
	Form	No.	kg ai/ha	kg ai/hl			
Castle Hayne (NC) Blue Chip	WP	4	0.29	0.034	12	1.2, 1.1 mean <u>1.2</u>	96-NC12
Gainesville (FL) Choice	WP	4	0.29	0.021	13	1.3, 2.2 mean <u>1.7</u>	96-FL33
Wooster (OH) Early Blue	WP	4	0.25	0.056	15	0.35, 0.32 mean <u>0.34</u>	96-OH16
Aurora (OR) Blue Crop	WP	4	0.29	0.043	14	0.81, 1.4 mean <u>1.1</u>	96-OR22[1]
Chatsworth (NJ) Blue Crop	WP	4	0.29	0.077	14	0.75, 0.87 mean <u>0.81</u>	96-NJ19
Pennsylvania Furnace (PA) Blue Crop	WP	4	0.29	0.063	12	0.60, 0.53 mean <u>0.56</u>	96-PA01
Douglas (MI) Jersey	WP	4	0.29	0.062	14	0.32, 0.28 mean <u>0.30</u>	96-MI17

Location variety	Application				PHI days	Residue, mg/kg	Report no.
	Form	No.	kg ai/ha	kg ai/hl			
Douglas (MI) Jersey	WP	4	0.29	0.062	14	0.45, 0.55 mean 0.50	96-MI18

[1] no soil type data available

Raspberries. In five field trials in geographically representative areas of the USA in 1996 (Dorschner and Breuninger, 1998c) (Table 15) 4 applications of a WP formulation of tebufenozide at the maximum GAP rate of 0.29 kg ai/ha ± 5% were made to the leaves of the crop at approximately 14-day intervals. Sampling and analysis were as for blueberries. The LOQ was 0.01 mg/kg. The SAI ranged from 283 to 305 days, with demonstrated stability of tebufenozide in raspberries stored frozen for 322 days.

Table 15. Residues of tebufenozide resulting from supervised trials on raspberries in the USA, 1996 (Dorschner and Breuninger, 1998c).

Location Variety	Application				PHI days	Residue, mg/kg	Report no.
	Form.	No.	kg ai/ha	kg ai/hl			
Aurora (OR) Meeker	WP	4	0.30	0.032	14	0.56, 0.43 mean 0.50	96-OR21
Skagit County (WA) Meeker	WP	4	0.31	0.076	15	0.95, 0.78 mean 0.86	96-WA52
Burlington (WA) Meeker	WP	4	0.30	0.076	15	0.71, 0.94 mean 0.82	96-WA34
Greenwood (WI) Royalty	WP	4	0.30	0.25	13	0.32, 0.39 mean 0.36	96-WI15
Pennsylvania Furnace (PA) Titan	WP	4	0.29	0.063	12	0.55, 0.57 mean 0.56	96-PA02

Cranberries. In four field trials in geographically representative areas of the USA in 1996 (Dorschner and Breuninger, 1998a) (Table 16) 4 foliar applications of a WP formulation of tebufenozide at 0.29 kg ai/ha ± 5% were made at approximately 14-day intervals, except in the MA trial where each application was 31% lower. In a single trial in Canada 4 applications were made at the same rate but of an SP formulation. In all the trials 2 or 4 replicate samples of cranberries were collected 13-14 days and 25-29 days after the last application, and analysed for tebufenozide as before. The LOQ was 0.05 mg/kg. The SAI ranged from 109 to 127 days, with demonstrated storage stability in cranberries stored frozen for 30 days.

Table 16. Residues of tebufenozide resulting from supervised trials on cranberries in the USA and Canada, 1996 (Dorschner and Breuninger, 1998a).

Country, Location Crop variety	Application				PHI days	Residue, mg/kg	Report no.
	Form	No.	kg ai/ha	kg ai/hl			
US, Wisconsin Rapids (WI), Ben Lear	WP	4	0.29	0.14	14 27	0.19, <0.01 mean 0.10 0.040, 0.051 mean 0.046	96-WI01
US, Biron (WI) Ben Lear	WP	4	0.29	0.14	14 25	0.18, 0.10 mean 0.14 <0.01, <0.01 mean <0.01	96-WI02
US, Chatsworth (NJ) Early Black	WP	4	0.29	0.083	14 29	0.12, 0.072 mean 0.091 <0.01, 0.023 mean 0.016	96-NJ23
US, East Wareham (MA) Early Black	WP	4	0.20	0.040	13 27	0.074, 0.069 mean 0.072 0.034, 0.050 mean 0.042	96-MA01
Canada, Agassiz (BC) MacFarlin	SC	4	0.29	0.038	14 27	0.84, 0.61, 0.89, 0.42 mean 0.69 0.23, 0.21, 0.34, 0.32 mean 0.28	96-BC01/02

Grapes. French trials in 1990 and 1992 conducted according to GAP in Portugal were reported to the 1996 JMPR but could not be evaluated because Portugese GAP was pending. It is now confirmed. The data are shown in Table 17.

Table 17. Previously summarized data for residues of tebufenozide in grapes.

Country, year	Application				PHI days	Residue, mg/kg	Reference
	Form	No.	kg ai/ha	kg ai/hl			
France, 1990	SC	2	0.15	0.04	12	0.16	Gocha, 1995
France, 1992	SC	2	0.144	0.048	14	0.29	Gocha, 1995
France, 1992	SC	2	0.144	0.048	14	0.68 0.81	Gocha, 1995

In five field trials in Australia in 1995 and 1998 (Hamblin *et al.*, 2001) (Table 18) 2 or 3 applications of a WP formulation of tebufenozide at a rate of 0.006 kg/hl, 0.12 kg ai/ha were made to the leaves of the crop . The intervals were 14 or 15 days between the first and second applications and 14 to 63 days between the second and third in 1998, and 33 days between the two in 1995. In all the trials single samples of grapes were collected 21, 28 and 35 days after the last application and analysed for tebufenozide by method AL013/92-0 (Holzwarth and Schuld, 1993a) with GC-MS instead of NPD. The laboratory made slight modifications during analyses. The LOQ was 0.01 mg/kg. The SAI was up to 9 months.

Table 18. Residues of tebufenozide resulting from supervised trials on grapes in Australia (Hamblin *et al.*, 2001).

Year, location variety	Application				PHI days	Residue, mg/kg	Report no./ application interval, days
	Form	No.	kg ai/ha	kg ai/hl			
1995/1996 Irymple (Vic) M12 Sultana	WP	2	0.071	0.006	0 21 28 35	0.32 0.12 0.09, 0.13, 0.22, 0.18, 0.15 <0.01	SCM248/96 33
	WP	2	0.141	0.012	0 21 28 35	0.80 0.33 0.39 0.29	SCM248/96 33
1998/1999, Dixons Creek (Vic) Chardonnay	WP	2	[1]	0.06	83 90 97	0.24 0.11 0.09	RTL528/99 14
	WP	3	[1]	0.006	21 28 35	1.3 0.72 0.64	RTL528/99 14, 62
1998/1999, Herne Hill (WA) Table Grapes/Flame Seedless, Red Globe	WP	3	0.12	0.006	21 28 35	1.0 1.1 0.85	MWS427/99 14, 14
1998/1999, Young (NSW) Firmint, Harslevelo	WP	2	0.135	0.006	60 67 74	0.18 0.17 0.12	PJH285/99 14
	WP	3	0.135, 0.135 0.18	0.006	21 28 35	1.5 0.58 1.2	PJH285/99 14, 39
1998/1999, Coonawarra (SA) Shiraz	WP	2	0.094, 0.129	0.006	84 91 98	0.06 0.06 0.05	SCM296/99 15

Year, location variety	Application				PHI days	Residue, mg/kg	Report no./ application interval, days
	Form	No.	kg ai/ha	kg ai/hl			
	WP	3	0.094, 0.129 0.144	0.006	21 28 35	0.61 0.81 0.78	SCM296/99 15, 63

[1] 1st and 2nd application sprayed well in excess of run-off, 3rd application (where applicable) >0.18 kg ai/ha.

Avocados. In five field trials in Australia and New Zealand from 1998 to 2000 (Brookbanks *et al.*, 2001) 4 foliar applications of a WP formulation of tebufenozide at a rate of 0.006 kg ai/hl (New Zealand GAP), 0.012 kg ai/hl, and 0.060 kg ai/hl (a tenfold concentrate) were made at intervals of 20-22 days in New Zealand and 14-15 days in Australia. In most of the trials, replicate samples of avocados were collected 0, 7, 14 and 21 days after the last application. GAP in New Zealand specifies a PHI of 21 days and proposed GAP in Australia 14 days.

Samples in Australia were analysed by Agrifood Technology method TP/215/990201 (GLC with an NPD) (Bayer). In trial DCP015/99 corrected data are also shown because recoveries of samples were low (59 to 65% at 0.2 to 1.0 mg/kg). Samples from New Zealand were analysed by HPLC-MS method 34-94-66 (Deakyne *et al.*, 1995). The LOQ was 0.04-0.05 mg/kg. All samples were analysed without stone. Table 19 shows the results with and without adjustment for the weight of the stone. The SAI ranged from 4 to 12 months. In trial DCP 020/00 it rained after the last application and residues were considerably lower.

Table 19. Residues of tebufenozide from supervised trials on avocados in Australia and New Zealand (Brookbanks *et al.*, 2001).

Country, year, location variety	Application				PHI days	Residue in stoneless fruit, mg/kg	Calculated residue[1] mg/kg	Report no.
	Form	No.	kg ai/ha	kg ai/hl				
Australia, 1998, Toowoomba (Qld) Hass	WP	4	0.12	0.006	0 7 14 21	0.12 0.086 0.10 0.12	0.099 0.074 0.087 0.10	IMI 243/00 [2]
	WP	4	0.18	0.012	0 7 14 21	0.36 0.45 0.43 0.33	0.30 0.40 0.38 0.28	IMI 243/00 [2]
	WP	4	0.30	0.060[3]	0 7 14 21	0.16 0.18 0.13 0.091	0.13 0.15 0.11 0.075	IMI 243/00 [4]
Australia, 1999-2000, Walkamin (Qld) Sheppard	WP	4	0.050	0.006	0 7 14 21	0.044 0.042 0.044 <0.040	0.037 0.036 0.037 0.033	DCP 020/00) [4,5]
		4	0.10	0.012	0 7 14 21	0.13 0.083 0.10 0.080	0.11 0.069 0.082 0.066	DCP 020/00 [4,5]
Australia, 1999, Kairi (Qld) Hass	WP	4	0.042	0.006	0 7 14 21	0.20/0.31[6] 0.16/0.24[6] 0.18/0.28[6] 0.16/0.24[6]	0.18/0.27[6] 0.13/0.21[6] 0.16/0.25[6] 0.13/0.21[6]	DCP 015/99 [7,8]
		4	0.086	0.012	0 7 14 21	0.48/0.75[6] 0.37/0.58[6] 0.36/0.57[6] 0.33/0.52[6]	0.43/0.67[6] 0.33/0.51[6] 0.32/0.50[6] 0.28/0.45[6]	DCP 015/99 [7,8]

Country, year, location variety	Application				PHI days	Residue in stoneless fruit, mg/kg	Calculated residue[1] mg/kg	Report no.
	Form	No.	kg ai/ha	kg ai/hl				
		4	0.086	0.060[3]	0 7 14 21	0.51/0.80[6] 0.44/0.70[6] 0.48/0.76[6] 0.34/<u>0.53</u>[6]	0.46/0.72[6] 0.39 /0.61[6] 0.43/0.67[6] 0.30/<u>0.47</u>[6]	DCP 015/99 [7,8]
New Zealand, 1999-2000, Mangawhai Hass	WP	4	n.d.[9]	0.006	0 7 14 21 28	0.26 0.37 0.45 <u>0.21</u>[10] 0.32	0.22 0.31 0.38 <u>0.18</u>[10] 0.28	FSLA039 [11]
New Zealand, 1999-2000, Katikati Hass	WP	4	n.d. [9]	0.006	21	0.18, 0.20 mean <u>0.19</u>	0.16, 0.18 mean <u>0.17</u>	FSLA039 [11]

[1] in whole fruit with stone
[2] one tree per plot (sampling is required from 4 trees), duplicate plots, one analytical sample per treatment
[3] application by concentrate spraying (10x). Spray volume was 0.63 l/tree
[4] one tree per plot, unreplicated. Samples consisted of 5 fruit instead of min. 12. Duplicate analyses.
[5] it rained after the last application (35 mm)
[6] uncorrected/corrected for recovery (64%). Corrected results are given because recoveries from samples spiked at 0.2, 0.5 and 1.0 mg/kg were 65, 64 and 59% respectively
[7] one tree per plot, duplicate plots. The average value is taken because of small plot size
[8] recoveries from samples spiked at 0.2, 0.5 and 1.0 mg/kg were 65, 64 and 59% respectively
[9] n.d.: no data available, spray volume/tree 7.2 l (corresponds to 0.11 kg ai/ha if 250 trees/ha (average))
[10] mean of duplicate analyses
[11] samples stored for up to 6 months at –4°C, and then another 3 months at $\leq$ –10°C

<u>Brassica vegetables</u>

<u>Cabbage and broccoli</u>. Table 20 and Table 21 show residue data submitted to the 1996 JMPR on cabbages and broccoli from US trials which comply with currently approved US GAP. These data could not be evaluated in 1996 because US GAP was pending. The SAI ranged from 110 to 938 days.

Table 20. Previously submitted data for residues of tebufenozide in cabbage from trials in the USA.

Location, Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
OH, 1992	SC	7	0.14	0.048-0.054	0 8	0.004[1], 0.007 <0.01[1], <u>0.004</u>	Chen, 1994b
FL, 1992	SC	7	0.14	0.015	9	<0.01[1], <u>0.03</u>	Chen, 1994b
TX, 1993	SC	7	0.14	0.045-0.062	0 7 14	0.02[1], 0.05 <0.01[1], <u>0.30</u> <0.01[1], 0.06	Chen, 1994b
WI, 1993	SC	7	0.14	0.057-0.064	0 7	<0.01[1], 0.01 0.01[1], <u>0.04</u>	Chen, 1994b
VA, 1993	SC	7	0.14	0.037	7	<u>0.53</u>	Chen, 1994b
CA, 1991	SC	8	0.14	0.075	0 7	0.39 <u>0.17</u>	Chen, 1994b
NY, 1991	SC	8	0.14	0.026-0.028	0 7	1.5 <u>0.09</u>	Chen, 1994b
TX, 1991	SC	8	0.14	0.047	0 7	0.24[1], 1.1 0.01[1], <u>0.11</u>	Chen, 1994b
CA, 1993	SC	8	0.14	0.05	0 7 14	1.5 <u>1.0</u> 0.91	Chen, 1994b

Location, Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
GA, 1991	SC	9	0.14	0.075	0 7	0.07[1], 1.3 0.01[1], 0.38	Chen, 1994b
TX, 1994	SC	9	0.14	0.05-0.075	0 7	0.8 0.78	Dong, 1995b
TX, 1994	WP	9	0.14	0.05-0.075	0 7	1.2 1.3	Dong, 1995b
FL, 1994	SC	9	0.14	0.05	0 7	3.6 4.6	Dong, 1995b
FL, 1994	WP	9	0.14	0.05	0 7	5.2 4.3	Dong, 1995b

[1] Head without wrapper leaves

Table 21. Previously submitted data for residues of tebufenozide in broccoli from trials in the USA.

Location Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
VA, 1992	SC	7	0.14	0.038	7	0.33	Chen, 1994b
OR, 1991	SC	8	0.14	0.044-0.047	0 7	0.67 0.24	Chen, 1994b
TX, 1991	SC	8	0.14	0.047	0 7	0.45 0.11	Chen, 1994b
CA, 1991	SC	8	0.14	0.042	0 7	0.33 0.09	Chen, 1994b
TX, 1992	SC	8	0.14	0.047-0.050	6	0.01	Chen, 1994b
CA, 1993	SC	8	0.14	0.050	0 7 14	0.46 0.07 0.05	Chen, 1994b
OR, 1992	SC	9	0.14	0.030	7	0.12	Chen, 1994b
CA, 1994	SC	7	0.14	0.025	0 7	0.36 0.1	Dong, 1995b
CA, 1994	WP	7	0.14	0.025	0 7	0.32 0.11	Dong, 1995b
CA, 1994	SC	9	0.14	0.025	0 7	0.75 0.31	Dong, 1995b
CA, 1994	WP	9	0.14	0.025	0 7	0.94 0.34	Dong, 1995b

Fruiting vegetables

Tomatoes (including cherry tomatoes). The residue data from France, Greece, Italy, The Netherlands, Spain and the USA are shown in Table 22.

Field trials were conducted in France, Greece and Spain in 1995 (Bürstell *et al.*, 1996), and greenhouse trials in Italy, Greece, Spain and The Netherlands in 1996 (Sonder and Bürstell, 1997) and 1997 (Schreuder, 1998). 2-6 foliar applications of a SC formulation of tebufenozide at rates between 0.18 and 0.45 kg ai/ha were made at 7-12 day intervals. In The Netherlands, all trials were at the same location but were independent. Single samples of tomatoes were collected 0, 1, 3, 7, 10 and/or 14 days after the last application and analysed for tebufenozide by the GC-MS method AL 013/92-0 (Holzwarth and Schuld, 1993a-c). The LOQ was 0.02 mg/kg. The SAI ranged from 4 to 8 months.

In field trials in the USA in 1996 (Carpenter, 1997a) 4 foliar applications of a WP formulation of tebufenozide at 0.29 $\pm$ 5% kg ai/ha (maximum GAP rate) were made at 6-8 day intervals. Replicate field samples of tomatoes were collected in all trials at the 7-day PHI and in some trials also at 0, 3,

14 and 21 days. All tomato samples were analysed for tebufenozide by method TR 34-95-66 (Deakyne *et al.*, 1995). The LOQ was 0.02 mg/kg. The SAI ranged from 203 to 327 days.

Table 22. Residues of tebufenozide resulting from supervised trials on tomatoes.

Country, year, location variety	Site	Application				PHI days	Residue, mg/kg	Reference/ Report no.
		Form	No.	kg ai/ha	kg ai/hl			
Spain, 1995, Brenes/Sevilla (Andalucia) Red Hunter	F	SC	4	0.18	0.051	0 3 7 10 14	0.33 0.41 0.33 0.26 0.09	Bürstell, 1996 ESP 00 01
France, 1995, St.Sixte (Midi-Pyrénées) Cannery row	F	SC	6	0.18	0.030	0 3 7 10 14	0.24, 0.28 0.30, 0.34 0.17, 0.27 0.14, 0.12 0.16, 0.16	Bürstell, 1996 FRA 00 01/ FRA 00 02
Greece, 1995, Korifi (Macedonia) Rio Grande	F	SC	6	0.18	0.040	0 3 7 10 14	0.27 0.19 0.19 0.13 0.10	Bürstell, 1996 GRC 00 01
Spain, 1996, Ultera (Andalucia) Caruso	G	SC	4	0.38	0.018	0 3	0.42 0.34	Sonder, 1997 ESP 00 01[1]
Spain, 1996, Los Palacios (Andalucia) Genaro	G	SC	4	0.45	0.018	0 3	0.33 0.25	Sonder, 1997 ESP 00 02[1]
Greece, 1996, Esovalta (Macedonia) Arletta	G	SC	4	0.45	0.018	0 3	0.16 0.09	Sonder, 1997 GRC 00 01[1]
Italy, 1996, Zapponeta (Puglia) Maiorca	G	SC	4	0.27	0.018	0 3	0.28 0.19	Sonder, 1997 ITA 00 01[1]
Italy, 1996, Molfetta (Puglia) Granito	G	SC	4	0.27, 0.31	0.018	0 3	0.40 0.20	Sonder, 1997 ITA 00 02[1]
Netherlands, 1997, Haren (Gr) Aramato	G	SC	2	0.21	0.014 3	0 1 3 7	0.12 0.10 0.07 0.16	Schreuder, 1998 NLD 015 01[2]
Netherlands, 1997, Haren (Gr) Aramato	G	SC	2	0.21	0.014 3	0 1 3 7	0.12 0.09 0.11 0.11	Schreuder, 1998 NLD 015 02[2]
Netherlands, 1997, Haren (Gr) Aramato	G	SC	2	0.21	0.014 3	0 1 3 7	0.11 0.09 0.11 0.10	Schreuder, 1998 NLD 015 03[2]
Netherlands, 1997, Haren (Gr) Aramato	G	SC	2	0.21	0.014 3	0 1 3 7	0.09 0.09 0.08 0.10	Schreuder, 1998 NLD 015 04[2]
US, 1996, North Rose (NY) Floradade	F	WP	4	0.29	0.062	7	0.057, 0.060 mean 0.058	Carpenter, 1997a 96-0214 2079601
US, 1996, Lucama (NC) Campbell 1327	F	WP	4	0.29	0.10	7	0.28, 0.22 mean 0.25	Carpenter, 1997a 96-0229 2079602

Country, year, location variety	Site	Application				PHI days	Residue, mg/kg	Reference/ Report no.
		Form	No.	kg ai/ha	kg ai/hl			
US, 1996, Chipley (FL) Mountain Spring	F	WP	4	0.30	0.16	7	0.10, 0.15 mean 0.13	Carpenter, 1997a 96-0157 2079603
US, 1996, Sneads (FL) Mountain Spring	F	WP	4	0.30	0.16	0 3 7 14 21	0.12, 0.12 mean 0.12 0.088, 0.050 mean 0.069 0.060, 0.12 mean 0.089 0.058, 0.032 mean 0.045 0.063, 0.070 mean 0.068	Carpenter, 1997a 96-0158 2079604
US, 1996, Dow (IL) Mountain Fresh	F	WP	4	0.29	0.16	7	0.092, 0.098 mean 0.095	Carpenter, 1997a 96-0236 2079605
US, 1996, Maricopa (AZ) Romano	F	WP	4	0.29, 0.33	0.12	7	0.086, 0.084 mean 0.085	Carpenter, 1997a 96-0210
US, 1996, Arroyo Grande (CA) Shady Lady	F	WP	4	0.30	0.095, 0.19	7	0.018, 0.045 mean 0.031	Carpenter, 1997a 96-0211[3]
US, 1996, Porterville (CA) Celebrity	F	WP	4	0.29	0.10	7	0.11, 0.098 mean 0.11	Carpenter, 1997a 96-0280
US, 1996, Porterville (CA) 88-90	F	WP	4	0.29	0.10	0 3 7 14 21	0.44, 0.42 mean 0.43 0.47, 0.50 mean 0.49 0.41, 0.36 mean 0.38 0.52, 0.53 mean 0.53 0.21, 0.63 mean 0.42	Carpenter, 1997a 96-0227
US, 1996, Porterville (CA) 88-90	F	WP	4	0.29	0.094, 0.045	7	0.35, 0.28 mean 0.31	Carpenter, 1997a 96-0249[4]
US, 1996, Avila (CA) Cherry tomato Sweet Cherry	F	WP	4	0.29	0.045	7 14 21	0.14, 0.21 mean 0.17 0.15, 0.15 mean 0.15 0.12, 0.16 mean 0.14	Carpenter, 1997a 96-0173[3]
US, 1996, Nipomo (CA) Cherry tomato Sweet Cherry	F	WP	4	0.30	0.19	0 3 7 14 21	0.61, 0.76 mean 0.68 0.74, 0.64 mean 0.69 0.51, 0.52 mean 0.52 0.49, 0.54 mean 0.52 0.47, 0.33 mean 0.40	Carpenter, 1997a 96-0273[5]

F: field, G: greenhouse
[1] sand or sandy-clay soil, cold house
[2] rockwool soil
[3] fruit was picked only from the bottom of the vines because the upper, exposed vines had no ripe fruit
[4] 50% of tomato fruit sampled was green, 50% was red. Sufficient ripe samples were available.
[5] sample sizes were too small (0.5 to 0.8 kg instead of min 2 kg)

Peppers (bell and non-bell). Nine field trials were conducted in the USA in 1996 (Carpenter, 1997b) in which 4 foliar applications of a WP formulation of tebufenozide at the GAP rate of 0.29 + 5% kg ai/ha were made at 6-8 day intervals. Replicate field samples of peppers were collected in all trials at the 7-day PHI and in some trials also 0, 3, 14 and 21 days after the last application. All samples were analysed for tebufenozide by method TR 34-95-66 (Deakyne *et al.*, 1995). The LOQ was 0.02 mg/kg. Non-bell pepper samples received an additional Florisil column clean-up to remove interferences. The SAI ranged from 185 to 290 days. Results are shown in Table 23.

Table 23. Residues of tebufenozide resulting from supervised trials on peppers in the USA, 1996 (Carpenter, 1997b).

Location, variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form	No.	kg ai/ha	kg ai/hl			
Bell peppers							
Lucama (NC) Capistrano	WP	4	0.29	0.10	0 3 7 14 21	0.37, 0.21 mean 0.29 0.052, 0.058 mean 0.055 0.049, 0.054 mean 0.052 0.037, 0.018 mean 0.027 0.017, 0.016 mean 0.017	96-0223
Sneads (FL) Camelot	WP	4	0.29	0.16	7	0.11, 0.20 mean 0.16	96-0156
Columbia (IL) King Arshon	WP	4	0.29	0.16	7	0.056, 0.041 mean 0.048	96-0237[1]
Uvalde (TX) Grand Rio 66	WP	4	0.29	0.20	0 3 7 14 21	0.19, 0.082 mean 0.14 0.070, 0.072 mean 0.071 0.038, 0.090 mean 0.064 0.075, 0.030 mean 0.052 0.074, 0.044 mean 0.059	96-0168
Porterville (CA) California Wonder	WP	4	0.29	0.10	7	0.52, 0.76 mean 0.64	96-0245
San Arch (CA) California Wonder 300	WP	4	0.29	0.11	7	0.14, 0.21 mean 0.17	96-0244
Non-bell peppers							
Levelland (TX) Jalapeno	WP	4	0.29	0.16	7	0.046, 0.034 mean 0.040	96-0195
Lubbock (TX) Jalapeno	WP	4	0.29	0.16	7	0.030, 0.062 mean 0.046	96-0197
Porterville (CA) Mitla Chili	WP	4	0.29	0.11	7	0.11, 0.086 mean 0.097	96-0246

[1] recovery from a spiked sample at the level of found residues was 75%

Leafy vegetables

Lettuce, spinach and mustard greens. In eight trials on head lettuce in Europe 3 foliar applications of an SC formulation were made at 7-day intervals at 0.144 kg a.i./ha ± 5% (Heydkamp, 2000). Single samples of head lettuce were taken 3, 7, 14 and 21 days after the last application. The validated analytical method was GLC with specific thermoionic detection (Quintelas, 2000). The LOQ was 0.02 mg/kg. The SAI was up to 10 months. The results are shown in Table 24.

Table 24. Residues of tebufenozide resulting from supervised trials on head lettuce in Europe in 1999 (Heydkamp, 2000).

Country, location, variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form.	No.	kg ai/ha	kg ai/hl			
Italy, Callepio di Settala (MI), Batavia/Dublin	SC	3	0.144	0.0144	3 7 14 21	0.72 0.05 <0.02 <0.02	VP98-1-33I1
Italy, Triginto di Mediglia (MI), Iceberg/Camaro	SC	3	0.144	0.0144	3 7 14 19	1.9 1.8 0.06 0.05	VP98-1-33I2
France, Manziat Romana/Feuille de Chêne	SC	3	0.144	0.0144	3 7 14 21	1.0 0.17 0.06 <0.02	VP98-1-33F3

Country, location, variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form.	No.	kg ai/ha	kg ai/hl			
France, Lucenay Batavia	SC	3	0.144	0.0144	3 7 14 21	1.5 0.18 0.25 0.12	VP98-1-33F4
Spain, La Palma (Murcia) Iceberg/Lluma	SC	3	0.144	0.0144	3 7 14 21	2.1 0.59 0.34 0.15	VP98-1-33E5
Spain, Almusafes Batavia/Empire	SC	3	0.144	0.0144	3 7 14 21	1.8 0.92 0.59 0.37	VP98-1-33E6
Spain, Castellar (Val) Iceberg	SC	3	0.144	0.0144	3 7 14 21	4.2 2.4 0.67 0.21	VP98-1-33E7
Spain, Castellar (Val) Inverna	SC	3	0.144	0.0144	3 7 14 21	4.9 2.6 0.83 0.40	VP98-1-33E9

Tables 25 to 28 show previously submitted residue data (JMPR 1996) on head lettuce, leaf lettuce, spinach and mustard greens from US trials conducted according to currently approved US GAP. These results could not be evaluated in 1996 because US GAP was pending. The SAIs in head and leaf lettuce ranged from 264 to 1186 in trials by Chen, and from 178 to 344 days in trials by Dong. The SAI in spinach was about 750 days and in mustard greens ranged from 139 to 930 days.

Table 25. Previously submitted data for residues of tebufenozide in head lettuce in the USA.

Location Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
NJ, 1991	SC	7	0.14	0.043	0 7	0.03[1], 0.48 0.009[1], <u>0.092</u>	Chen, 1994a
CA, 1993	SC	7	0.14	0.042	0 7 14	0.41 <u>0.14</u> 0.009	Chen, 1994a
CA, 1991	SC	8	0.14	0.075	0 7	1.7[1], 5.1 0.053[1], <u>0.83</u>	Chen, 1994a
FL, 1991	SC	8	0.14	0.019-0.03	0 7 14	0.09[1], 1.0 0.018[1], <u>0.9</u> 0.006[1], 0.02	Chen, 1994a
TX, 1992	SC	9	0.14	0.044	0 7	1.5[1] <u>0.29</u>	Chen, 1994a
CA, 1994	SC	7	0.14	0.037	0 7	3.0 <u>2.3</u>	Dong, 1995a
CA, 1994	WP	7	0.14	0.037	0 7	3.8 <u>6.6</u>	Dong, 1995a
AZ, 1994	SC	7	0.14	0.05	0 7	3.5 <u>3.2</u>	Dong, 1995a
AZ, 1994	WP	7	0.14	0.050	0 7	4.4 <u>2.7</u>	Dong, 1995a

[1] Head without wrapper leaves

Table 26. Previously submitted data for residues of tebufenozide in leaf lettuce in the USA.

Location Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
NJ, 1991	SC	7	0.14	0.043	0 7	3.5 2.2	Chen, 1994a
CA, 1991	SC	8	0.14	0.042	0 6	5.7 1.7	Chen, 1994a
FL, 1991	SC	8	0.14	0.019	0 7	0.88 0.41	Chen, 1994a
TX, 1991	SC	9	0.14	0.044	0 7	2.7 0.69	Chen, 1994a
CA, 1994	SC	7	0.14	0.025	0 7	3.7 1.1	Dong, 1995a
CA, 1994	WP	7	0.14	0.025	0 7	3.5 2.5	Dong, 1995a
AZ, 1994	SC	7	0.14	0.050	0 7	3.3 3.2	Dong, 1995a
AZ, 1994	WP	7	0.14	0.05	0 7	3.6 2.6	Dong, 1995a

Table 27. Previously submitted data for residues of tebufenozide in spinach in the USA.

Location Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
VA, 1991	SC	6	0.14	0.05-0.058	0 7	10 7.1	Chen, 1994a
AZ, 1993	SC	7	0.14	0.074-0.076	0 7 14	4.4 0.99 0.13	Chen, 1994a
OK, 1993	SC	7	0.14	0.050-0.052	0 7	5.1 1.3	Chen, 1994a
CA, 1991	SC	8	0.14	0.075	0 7	5.5 8.1	Chen, 1994a
TX, 1992	SC	9	0.14	0.044	0 7	15 2.7	Chen, 1994a
CA, 1994	SC	7	0.14	0.025	0 7	7.0 3.9	Dong, 1995a
CA, 1994	WP	7	0.14	0.025	0 7	7.0 3.3	Dong, 1995a
TX, 1994	SC	7	0.14	0.05	0 7	8.3 3.8	Dong, 1995a
TX, 1994	WP	7	0.14	0.05	0 7	7.0 4.2	Dong, 1995a

Table 28. Previously submitted data for residues of tebufenozide in mustard greens in the USA.

Location Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
NJ, 1992	SC	7	0.14	0.043	7	5.6	Chen, 1994b
NJ, 1993	SC	7	0.14	0.043	0 7	4.1 1.6	Chen, 1994b
AZ) 1993/1994	SC	7	0.14	0.060	0 7 14	7.1 2.6 1.6	Chen, 1994b
CA, 1991	SC	8	0.14	0.075	0 7	8.2 3.9	Chen, 1994b
CA, 1991	SC	8	0.14	0.075	0 7	5.5 6.9	Chen, 1994b
CA, 1991	SC	8	0.14	0.075	0 7	5.6 4.4	Chen, 1994b

	Application						
CA, 1994	SC	7	0.14	0.025	0 7	4.3 0.65	Dong, 1995b
CA, 1994	WP	7	0.14	0.025	0 7	2.5 0.93	Dong, 1995b
TX, 1994	SC	8	0.14	0.050-0.075	0 7	5.1 1.9	Dong, 1995b
TX, 1994	WP	8	0.14	0.050-0.075	0 7	6.8 2.4	Dong, 1995b

The previously submitted residues from one trial on Chinese kale in Thailand are shown in Table 29. According to the manufacturers, this trial was in compliance with current GAP in Thailand (5 applications of 0.30 kg ai/ha, PHI 14 days), but no independent confirmation was reported to the JMPR.

Table 29. Previously submitted data for residues of tebufenozide in Chinese kale, Thailand, 1993.

Application				PHI	Residue,	Reference
Form.	No.	kg ai/ha	kg ai/hl	days	mg/kg	
SC	5	0.25	0.033-0.053	0 3 5 10 15	17.2 8.8 5.2 1.5 0.88	Ishii and Higuchi, 1993

Turnip greens and roots. In six new field trials on turnips in geographically representative areas of the USA in 1996 4 foliar applications of a WP formulation of tebufenozide were made at 0.295 ± 5% kg ai/ha at approximately 7-day intervals (Dorschner and Breuninger, 1998b). At three of the sites (GA, OH, TN), two separate plots were treated and root samples collected from one plot and foliage samples (turnip greens) from the other. At the TX site, plots were divided into two sub-plots, and tops and roots were harvested from different sub-plots. At the sites in CA and SC, both roots and tops were collected from the same plot but separated into two samples. In each of the trials, replicate samples of roots and tops were collected 6-8 days after the last application.

All root and foliage samples were analysed for tebufenozide by method TR 34-94-41 (Chen *et al.*, 1994c). The analytical laboratory made slight modifications during analyses. The LOQ was 0.01 mg/kg. The longest SAI was 244 days. Storage stability was demonstrated in turnip greens and roots stored frozen for 279 days. The results for turnip greens are given in Table 30 and for roots in Table 31.

Table 30. Residues of tebufenozide in turnip greens resulting from supervised trials in the USA, 1996 (Dorschner and Breuninger, 1998b).

Location, variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form.	No.	kg ai/ha	kg ai/hl			
Tifton (GA) Purple Top	WP	4	0.29	0.11	8	1.3, 1.3 mean 1.3	96-GA*04
Charleston (SC) Purple Top White Globe	WP	4	0.30	0.09	7	2.6, 1.5 mean 2.1	96-SC*01
Weslaco (TX) Purple Top White Globe	WP	4	0.31	0.11	6	8.3, 6.4 mean 7.4	96-TX*02
Celeryville (OH) Purple Top	WP	4	0.30	0.04	7	0.57, 0.31 mean 0.44	96-OH*03
Salinas (CA) Purple Top White Globe	WP	4	0.29	0.10, 0.08, 0.04, 0.04	8	0.34, 0.34 mean 0.34	96-CA*04

Location, variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form.	No.	kg ai/ha	kg ai/hl			
Crossville (TN) Purple Top White Globe	WP	4	0.29	0.12	7	2.1, 2.1 mean 2.1	96-TN02

Table 31. Residues of tebufenozide in turnip roots resulting from supervised trials in the USA, 1996 (Dorschner and Breuninger, 1998b).

Location variety	Application				PHI days	Residue, mg/kg	Report no.
	Form.	No.	kg ai/ha	kg ai/hl			
Tifton (GA) Purple Top	WP	4	0.29	0.11	8	0.18, 0.17 mean 0.18	96-GA*04
Charleston (SC) Purple Top White Globe	WP	4	0.30	0.09	7	0.09, 0.07 mean 0.08	96-SC*01
Weslaco (TX) Purple Top White Globe	WP	4	0.31	0.11	6	0.21, 0.23 mean 0.22	96-TX*02
Celeryville (OH) Purple Top	WP	4	0.30	0.040	7	0.02, 0.02 mean 0.02	96-OH*03
Salinas (CA) Purple Top White Globe	WP	4	0.29	0.10, 0.08, 0.04, 0.04	8	0.02, 0.03 mean 0.02	96-CA*04
Crossville (TN) Purple Top White Globe	WP	4	0.29	0.12	7	0.06, 0.12 mean 0.09	96-TN02

Celery. The previously submitted residue data (JMPR 1996) on celery from trials complying with currently approved US GAP are shown in Table 32. These data could not be evaluated in 1996 because US GAP was pending. In all except two trials only stalk samples were analysed. The SAI ranged from 71 to 525 days.

Table 32. Previously submitted data for residues of tebufenozide in celery in the USA.

Location Year	Application				PHI days	Residue, mg/kg	Reference
	Form.	No.	kg ai/ha	kg ai/hl			
MI, 1992	SC	7	0.14	0.045-0.047	0 6	1.4 0.47	Chen, 1994a
FL, 1992/1993	SC	7	0.14	0.015	9	0.1	Chen, 1994a
CA, 1993	SC	7	0.14	0.030	0 7	2.3[1], 0.29 1.3[1], 0.49	Chen, 1994a
CA, 1994	SC	7	0.14	0.025	0 7	0.38 0.64	Dong, 1995a
CA, 1994	WP	7	0.14	0.025	0 7	0.47 0.6	Dong, 1995a
MI, 1993	SC	8	0.14	0.050-0.055	0 6 13	1.2 1.2 0.64	Chen, 1994a
CA, 1993	SC	8	0.14	0.075	0 7	1.2[1], 0.15 0.41[1], 0.09	Chen, 1994a
MI, 1993	SC	8	0.14	0.100-0.111	0 6 13	4.5 3.2 1.5	Chen, 1994a
FL, 1995	SC	9	0.14	0.05	0 7	0.08 0.04	Dong, 1995a
FL, 1995	WP	9	0.14	0.05	0 7	0.08 0.05	Dong, 1995a

[1] stalk with foliage

Grasses

One field trial was conducted on sugar cane in the USA in 1994 with the SC formulation and seven in 1995 with the WP (Filchner, 1997a,b). In two further 1997 US field trials SC and WP formulations were compared (Bergin, 1998). Four foliar applications were made at the US GAP rate of 0.28 kg ai/ha at intervals of 14 to 21 days. Duplicate samples of sugar cane stems were collected 13-14 days after the last application. Residue decline was determined in 2 trials in 1995, and extra samples for processing were collected in the 1994 and 1997 trials.

Analyses were by method TR 34-94-41 (Chen *et al.*, 1994c) in 1994 and TR 34-95-66 (Deakyne *et al.*, 1995) in 1995. Both methods were adapted by the addition of a carbon solid phase clean-up to achieve an LOQ of 0.01 mg/kg. Samples from the 1997 trials were analysed by method TR 34-97-115 (Filchner and Deakyne, 1997), which has an LOQ of 0.01 mg/kg. The results are shown in Table 33. The SAI ranged from 115 to 404 days.

Table 33. Residues of tebufenozide in sugar cane stems resulting from supervised trials in the USA.

Year, location variety	Application				PHI days	Residue, mg/kg	Reference
	Form	No.	kg ai/ha	kg ai/hl			
1994, Washington (LA) 357	SC	4	0.28	0.20	14	0.31, 0.25 mean <u>0.28</u>	Filchner, 1997b 94-0168
1995, Church Point (LA) CP 70-321	WP	4	0.30	0.31	14	0.11, 0.14 mean <u>0.12</u>	Filchner, 1997a 95-0182
1995, Simmsport (LA) CP65-357	WP	4	0.29	0.31	13	0.10, 0.14 mean <u>0.12</u>	Filchner, 1997a 95-0192
1995, Cheneville (LA) CP65-357	WP	4	0.29	0.31	7 13 20 27	0.046, 0.066 mean 0.056 0.16, 0.16 mean <u>0.16</u> 0.10, 0.14 mean 0.12 0.068, 0.092 mean 0.080	Filchner, 1997a 95-0193
1995, Moorehaven (FL) CP-1133	WP	4	0.29	0.15	14	0.036, 0.034 mean <u>0.035</u>	Filchner, 1997a 95-0275
1995, Belleglade (FL) CP-1210	WP	4	0.29	0.15	14	0.032, 0.032 mean <u>0.032</u>	Filchner, 1997a 95-0276
1995, Belleglade (FL) CP-1210	WP	4	0.29	0.15	7 14 21 28	0.036, 0.023 mean 0.030 0.016, 0.010 mean <u>0.013</u> 0.000, 0.000 mean <0.01 0.006, 0.000 mean <0.01	Filchner, 1997a 95-0277
1995, Raymondville (TX) CP310	WP	4	0.29	0.31	14	0.055, 0.053 mean <u>0.054</u>	Filchner, 1997a 95-0280
1997, Port Allen (LA) CP-845	WP	4	0.29	0.24	13	0.63, 0.61 mean <u>0.62</u>	Bergin, 1998 97-0130
	SC	4	0.29	0.25	13	0.56, 0.52 mean <u>0.54</u>	Bergin, 1998 97-0130

<u>Tree nuts</u>

<u>Pecans</u>. Data on residues in pecans were submitted to the JMPR in 1996 but could not be evaluated since in 1996 the use of tebufenozide was not registered for any tree nuts except walnuts. US trials in 1993 on pecans that were within the range of the now-approved US GAP are shown in Table 34.

Table 34. Previously submitted US data on residues of tebufenozide in pecans (Cui and Desai, 1995b).

Location, Year	Application				PHI days	Residue, mg/kg
	Form.	No.	kg ai/ha	kg ai/hl		
TX, 1993	SC	6	0.28	0.035	0 14 28	<0.01 <0.01 <0.01
NM, 1993	SC	6	0.28	0.30	0 14 28	<0.01 <0.01 <0.01
AL, 1993	SC	6	0.28	0.019-0.031	0 14 28	<0.01 <0.01 <0.01
GA, 1993	SC	6	0.28	0.033	0 14 28	<0.01 <0.01 <0.01

In eight field trials on pecans in geographically representative areas of the USA in 1997 4 ground-applied foliar applications of either a WP or an SC formulation of tebufenozide were made at 0.54 kg/ha ± 5% (total application 2.1 kg ai/ha) at 14-83 day intervals (Bergin, 1999). Surfactant was tank-mixed with the WP formulation. Replicate samples of nuts were collected 14 days after the last application. The results are shown in Table 35.

All pecans were shelled and kernels were analysed for tebufenozide by the HPLC-UV method TR 34-95-20 (Cui and Desai, 1995a). The analytical laboratory made slight modifications during analyses. The LOQ was 0.01 mg/kg. The SAI ranged from 245 to 295 days.

Table 35. Residues of tebufenozide in pecan kernels resulting from supervised trials in the USA, 1997 (Bergin, 1999).

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Location, Variety	Application				PHI days	Residue, mg/kg	Trial no., appl. interval, days
	Form.	No.	kg ai/ha	kg ai/hl			
Cary (MS) Cape Fear	WP	4	0.54	0.10	14	<0.01, <0.01 mean <0.01	97-0114 17, 18, 42
Burr (TX) USDA 67-9-7	WP	4	0.54	0.10	14	<0.01, <0.01[1] mean <0.01	97-0115 14, 14, 53
Hawkinsville (GA) Stuart	WP	4	0.54	0.11	7 14 21 28	<0.01[1] <0.01, <0.01 mean <0.01 <0.01 <0.01	97-0110 $int^{7,14}$: 14, 48, 14 $int^{21, 28}$: 14, 34, 14
Silverton (TX), Western Sly	WP	4	0.54	0.10	14	<0.01, <0.01 mean <0.01	97-0129 14, 34, 14
Eastman (GA), Desirable	WP	4	0.54	0.09	14	<0.01, <0.01 mean <0.01	97-0113 21, 76, 20
	SC	4	0.54	0.09	14	<0.01, <0.01 mean <0.01	97-0113[2] 21, 62, 14
Hereford (TX), Wichita	WP	4	0.54	0.08	14	<0.01, <0.01 mean <0.01	97-0128 14, 19, 35
	SC	4	0.55	0.08	14	<0.01, <0.01 mean <0.01	97-0128 21, 14, 83

$int^{7,14}$: intervals for samples with PHIs of 7 and 14 days
$int^{21, 28}$: intervals for samples with PHIs of 21 and 28 days
[1] average of duplicate analyses
[2] surfactant was also inadvertently tank-mixed with the SC formulation

Almonds. In ten field trials in geographically representative areas of the USA in 1995/96 (Filchner, 1998) and 1998 (Yoshida, 1999) 4 foliar applications of either a WP or an SC formulation of tebufenozide at the maximum GAP rate of 0.54 kg/ha ± 5% (total application 2.1 kg ai/ha) were made with a first and second interval of 27-64 days and a third of 12-17 days in 1995/96, and 10-39-day intervals in 1998. Surfactant was tank-mixed. In all the trials, replicate samples of nuts were collected 14 days after the last application, and in all except one the hulls were removed in the field.

All kernel and hull samples were analysed by method TR 34-95-20 (Cui and Desai, 1994) with slight modifications. The LOQ was 0.01 mg/kg for the kernels. The SAI ranged from 174 to 330 days for the kernels and 164 to 549 days for hulls. The results are shown in Table 36.

Table 36. Residues of tebufenozide in almond kernels and hulls resulting from supervised trials in the USA.

Year, Location, Variety	Application				PHI days	Sample	Residue, mg/kg	Reference/appl. interval, days
	Form	No.	kg ai/ha	kg ai/hl				
1995, Arbuckle (CA) Non Pariel	WP	4	0.54	0.06	13	kernel hull	0.029, 0.040 mean 0.034 8.9, 7.9 mean 8.4	Filchner, 1998 95-0120 27, 35, 17
1995, Chico (CA) Non Pariel	WP	4	0.54	0.05	14	kernel hull	0.038, 0.052 mean 0.045 18, 16 mean 17	Filchner, 1998 95-0163 28, 48, 13
1995, Chico (CA) Non Pariel	WP	4	0.54	0.05	11	kernel hull	0.024, 0.034 mean 0.029 9.6, 9.5 mean 9.5	Filchner, 1998 95-0164 26, 45, 12
1995, Porterville (CA) Mission	WP	4	0.54	0.03	14	kernel	<0.01, <0.01 mean ≤0.01	Filchner, 1998 95-0165 33, 62, 14
1995 Sanger (CA) Butte	WP	4	0.54	0.03	14	kernel hull	0.023, <0.01 mean 0.017 12, 18 mean 15	Filchner, 1998 95-0174 29, 51, 12
1996, Madera (CA) Butte	WP	4	0.54[1]	0.06, 0.05, 2x 0.03	7 14 21 28	kernel hull kernel hull kernel hull kernel hull	0.050, 0.055 mean 0.052 22, 20 mean 21 0.036, 0.023 mean 0.029 10, 20 mean 15 0.038, 0.030 mean 0.034 12, 15 mean 14 0.030, 0.054 mean 0.042 21, 14 mean 17	Filchner, 1998 96-0184 28, 61, 15
1998, Terra Bella (CA) Non Pariel	WP	4	0.54	0.03	14	kernel hull	0.015, 0.032 mean 0.024 9.6, 12 mean 11	Yoshida, 1999 98-0319 [2] 14, 21, 14
	SC	4	0.54	0.03	14	kernel hull	<0.01, <0.01 mean ≤0.01 16, 16 mean 16	Yoshida, 1999 98-0319 [2] 14, 21, 14
1998, Brooks (CA) Carmel	WP	4	0.54	0.06	14	kernel hull	<0.01, 0.011 mean 0.010 23, 19 mean 21	Yoshida, 1999 98-0231 10, 39, 16
	SC	4	0.54	0.06	14	kernel hull	0.022, <0.01 mean 0.016 4.1, 16 mean 10	Yoshida, 1999 98-0231 10, 39, 16

[1] Second application was 17% low (0.49 kg ai/ha)
[2] Recovery from spiked kernel sample at 0.02 mg/kg was 61.1% (79.5% at 0.01 mg/kg)

Macadamia nuts. In six field trials in Australia from 1997 to 1999 5 foliar applications of a WP formulation of tebufenozide at a rate of 0.012 kg ai/hl, corresponding to 0.084 to 0.42 kg ai/ha, depending on the spray volumes used, were made to macadamia trees at intervals of 14 to 36 days

(14 to 111 days between the second and third). Single samples of nuts were collected 21, 28 and 35 days after the last application (Lewis and Vitelli, 2000).

All kernels samples were analysed for tebufenozide by method AL013/92-0 (Holzwarth and Schuld, 1993a) with GC-MS instead of GLC with NPD detection and slight modifications during analyses. The LOQ was 0.02 mg/kg. The SAI was up to 240 days (Table 37).

Table 37. Residues of tebufenozide in macadamia kernels resulting from supervised trials in Australia (Lewis and Vitelli, 2000).

Year, Location variety	Application				PHI days	Residue, mg/kg	Trial no/interval, days, between appl.
	Form.	No.	kg ai/ha	kg ai/hl			
1996/97, Kairi (Qld)	WP	5	hv[1]	0.006	21 28 35	<0.02 <0.02 <0.02	JME 231/97 [2-4] 15, 14, 14, 18
	WP	5	hv[1]	0.012	21 28 35	0.03 <0.02 0.03	JME 231/97 [2-4] 15, 14, 14, 18
1997/98, Wongabel (Qld) 344	WP	5	hv[1]	0.006	21 28 35	<0.02 <0.02 <0.02	JME 265/98 [4] 14, 111, 14, 14
	WP	5	hv[1]	0.009	21 28 35	<0.02 <0.02 <0.02	JME 265/98 [4] 14, 111, 14, 14
	WP	5	hv[1]	0.012	21 28 35	<0.02 <0.02 <0.02	JME 265/98 [4] 14, 111, 14, 14
1996/97, Bundaberg (Qld) A4	WP	5	hv[1]	0.006	21 28 32	<0.02 <0.02 <0.02	RAV 007/98 26, 31, 28, 36
	WP	5	hv[1]	0.012	21 28 32	0.02 <0.02 0.02	RAV 007/98 26, 31, 28, 36
1997/98, Bundaberg (Qld) 344	WP	5	0.21	0.006	21 28 35	0.03 <0.02 <0.02	RAV 051/98 28, 49, 19, 19
	WP	5	0.32	0.009	21 28 35	0.03 0.03 <0.02	RAV 051/98 28, 49, 19, 19
	WP	5	0.42	0.012	21 28 35	0.04 0.05 0.06	RAV 051/98 28, 49, 19, 19
1998/99, Bundaberg (Qld) 344	WP	5	0.13	0.006	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 28, 64, 20, 24
	WP	5	0.13	0.006	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 [2] 28, 64, 20, 24
	WP	5	0.13	0.030 (5x conc of 0.006)	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 28, 64, 20, 24
	WP	5	0.13	0.048 (8x conc of 0.006)	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 28, 64, 20, 24
	WP	5	0.26	0.012	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 28, 64, 20, 24
	WP	5	0.26	0.012	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 b 28, 64, 20, 24

Year, Location variety	Application				PHI days	Residue, mg/kg	Trial no/interval, days, between appl.
	Form.	No.	kg ai/ha	kg ai/hl			
	WP	5	0.26	0.060 (5x conc of 0.012)	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 28, 64, 20, 24
	WP	5	0.26	0.096 (8x conc of 0.012)	21 28 35	<0.02 <0.02 <0.02	RAV 073/99 28, 64, 20, 24
2000, Bundaberg (Qld) HAES 772	WP	5	0.17	0.012	0 14 21 28 35	0.03 <0.02 0.03 <0.02 0.03	RAV 113/00 14, 14, 13, 14
	WP	5	0.17	0.12 (as 10x conc of 0.012)	0 14 21 28 35	<0.02 <0.02 <0.02 <0.02 0.02	RAV 113/00 14, 14, 13, 14
	WP	5	0.25	0.18	0 14 21 28 35	0.08 0.05 0.06 0.05 0.03	RAV 113/00 14, 14, 13, 14

[1] high volume spray to the point of run-off, in trial RAV 007/98 spray volume was 2 l/tree
[2] a wetting adjuvant (BS 1000) was tank mixed
[3] tebufenozide was applied one day before 5th application, but heavy rain soon washed it off
[4] no weather data available

Rape (canola). In six field trials on rape in geographically representative areas of the USA and one in Canada in 1996 and 1997 four foliar applications of either the UL formulation (USA) or the SC formulation (Canada) of tebufenozide at the maximum US GAP rate of 0.28 kg/ha ± 5% were made at 12-15-day intervals. In all the trials, replicate samples of mature canola seed were collected 6-15 days after the last application. In addition, meal, soapstock and refined oil generated from canola were collected from two sites (Dorschner and Breuninger, 1998f).

All canola seed samples were analysed by the LC-MS method TR 34-96-135 (Wu *et al.*, 1996) with slight modifications. The LOQ was 0.01 mg/kg for the seed. The SAI ranged from 13 to 231 days. Storage stability was demonstrated for rape seed stored frozen for 236 days. The results are given in Table 38.

Table 38. Residues of tebufenozide in rape seed (canola) resulting from supervised trials in the USA and Canada (Dorschner and Breuninger, 1998f).

Country, year, location, variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form.	No.	kg ai/ha	kg ai/hl			
Canada, 1996, Minto (MB) 45A71	SC	4	0.29	0.14	6 (+8)[1]	0.29, 0.65 mean 0.47	96-MB04
US, 1996, Moxee (WA) Tobin	UL	4	0.28	0.074	9 9 9	0.95 1.6, 1.5 mean 1.6 1.1, 1.1 mean 1.1	96-WA*40/41/42[2, 3]
US, 1997, Tifton (GA) Bingo	UL	4	0.28	0.15	15	0.32, 0.30 mean 0.31	96-GA17 [4, 5]

Country, year, location, variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form.	No.	kg ai/ha	kg ai/hl			
US, 1996, Langdon (ND) Hyola 401	UL	4	0.27	0.15	14 (+7) [1]	0.49, 0.61, 0.47 mean 0.52	96-ND03
US, 1996, Carrington (ND) Hyola 401	UL	4	0.29	0.15	14	1.2, 1.1 mean 1.2	96-ND04

[1] After swathing, seed was left on the field to dry for several days. In ND, this mimics local commercial practice
[2] extremely hot and dry weather caused early crop maturation
[3] Silwet L-11 was added to each tank mix
[4] on the day of the last application it had rained 79 mm
[5] samples arrived thawed at the analytical laboratory 5 days after shipment

Mint. In five field trials in Wisconsin and Washington, USA, in 1996 (Table 39) 4 foliar applications of a WP formulation of tebufenozide at the maximum GAP rate of 0.295 kg ai/ha ± 5% were made at approximately 14-day intervals (Dorschner and Breuninger, 1998e). At the two Wisconsin sites an additional application was made because cool early season temperatures delayed maturity by approximately two weeks. In trial 96WI14, a frost killed the mint tops on the day of the first application. In all trials replicate samples of the foliage were collected 14 days after the last application. At two sites, additional foliage samples were collected for processing to oil. Foliage samples were analysed for tebufenozide by method TR 34-94-41 (HPLC-UV; Chen *et al.*, 1994c) with an additional clean up by Envi-Carbon solid-phase extraction and other slight modifications. The LOQ was 0.01 mg/kg. The SAI ranged from 155 to 200 days. Storage stability was demonstrated in mint foliage stored frozen for 279 days.

Table 39. Residues of tebufenozide in mint resulting from supervised trials in the USA, 1996 (Dorschner and Breuninger, 1998e).

Location variety	Application				PHI days	Residue, mg/kg	Trial no.
	Form.	No.	kg ai/ha	kg ai/hl			
Prosser (WA) Spearmint, Native	WP	4	0.29	0.10	14	8.2, 8.5 mean 8.4	96-WA*37
Mabton (WA) Spearmint, Native	WP	4	0.29	0.10, 0.08	14	8.0, 9.2 mean 8.6	96-WA*38
Mabton (WA) Pepermint	WP	4	0.29	0.08	14	8.2, 8.5 mean 8.3	96-WA*39
Marquette Co. (WI) Spearmint, Scotch	WP	5	0.30	0.15	14 (+1) [1]	6.0, 9.0 mean 7.5	96-WI13
Dalton (WI) Spearmint, Scotch	WP	5	0.30	0.15	14	2.9, 2.6 mean 2.8	96-WI14 [2]

[1] mint was collected after drying in field for 1 day
[2] frost killed the mint tops on day of first application

Animal feeding studies

Cows. Sixteen dairy cows, divided into groups of four, were dosed with tebufenozide (purity: 96.5%) with capsules at levels equivalent to 0, 6, 18 or 60 ppm ai in the diet for 28 consecutive days (Deakyne, 1996). Doses were based on the highest feed consumption over the previous 7 days: 25 kg (mean about 20 kg). One cow from each group was dosed for the 28 days before having a 3-day recovery period. Composited milk samples were taken from the morning and afternoon milkings. The sampling schedule was -1 day, 0 day and then at 3-day intervals to day 27 or day 30 (for the depurated

cows). No significant differences were noted in body weights between control and test animals during the quarantine and test periods. Autopsies showed no effects that appeared to be related to the test material.

Analytes for determination were selected from the significant residues found in the metabolism study in lactating dairy goats (JMPR 1996) and included tebufenozide, the free oxidation products RH-9886, RH-0282 and RH-2703, and fatty acid conjugates of RH-9886. In the metabolism study, multiple fatty acid conjugates of RH-9886 were identified. For residueanalysis the stearic acid conjugate (RH-9526) was synthesized for fortification purposes. Table 40 lists the analytes that were determined in each sample.

Table 40. Analytes determined in samples from cow feeding study.

Sample	Analyte				
	tebufenozide	RH-9886	RH-0282	RH-9526	RH-2703
Milk	X		X	X	
Meat	X	X	X		
Liver	X				X
Kidney	X	X	X		
Fat	X			X	

Samples were analysed by method TR 34-96-109 (Burnett *et al.*, 1996). The metabolite RH-9526 was, after hydrolysis, measured as RH-9886 and was used as a standard for all fatty acid conjugates of RH-9886. Milk samples were shaken vigorously by hand on the day of analysis; cream and skimmed milk were not analysed separately. For all analytes, the LOQ was 0.02 mg/kg in liver, kidney, muscle and fat, and 0.01 mg/kg in milk. SAIs were 183-250 days for milk, 151 days for liver, 240 days for meat and 274 days for fat. Table 41 shows the average residues of tebufenozide, RH-0282 and RH-9526 in milk in each dosed group and Table 42 the individual residues of tebufenozide and metabolites in the tissues.

Table 41. Average group residues (4 cows/group) by day in milk of cows fed tebufenozide.

Day	Tebufenozide, mg/kg	RH-0282, mg/kg	RH-9886[1], mg/kg
6 ppm feeding level			
-1	0[2]	0	0
0	0	0	0
3	0	0	0.004 (<LOQ)
6	0	0	0.005 (<LOQ)
9	0	0	0.003 (<LOQ)
13	0	0	0.002 (<LOQ)
16	0.002 (<LOQ)	0	0.003 (<LOQ)
20	0.002 (<LOQ)	0	0.004 (<LOQ)
23	0.003 (<LOQ)	0	0.004 (<LOQ)
27	0	0	0.002 (<LOQ)
30[3]	0	0	0
18 ppm feeding level			
-1	0	0	0
0	0.002 (<LOQ)	0.003 (<LOQ)	0
3	0.005 (<LOQ)	0	0.011
6	0.004 (<LOQ)	0	0.012
9	0.006 (<LOQ)	0.002 (<LOQ)	0.012
13	0.006 (<LOQ)	0.004 (<LOQ)	0.011
16	0.007 (<LOQ)	0	0.011
20	0.009 (<LOQ)	0	0.014
23	0.007 (<LOQ)	0	0.012
27	0.004 (<LOQ)	0	0.012
30[3]	0	0	0
60 ppm feeding level			

Day	Tebufenozide, mg/kg	RH-0282, mg/kg	RH-9886[1], mg/kg
-1	0	0	0.003 (<LOQ)
0	0.004 (<LOQ)	0	0.002 (<LOQ)
3	0.025	0.004 (<LOQ)	0.027
6	0.021	0.005 (<LOQ)	0.027
9	0.024	0.004 (<LOQ)	0.025
13	0.017	0.004 (<LOQ)	0.020
16	0.026	0.005 (<LOQ)	0.024
20	0.028	0.007 (<LOQ)	0.030
23	0.019	0.004 (<LOQ)	0.024
27	0.015	0.004 (<LOQ)	0.020
30[3]	0	0	0

[1] RH-9526 and all fatty acid conjugates of RH-9886 are reported as RH-9886
[2] A value of zero indicates that no peaks were detected and residues would be less than the LOD (<0.003 mg/l). All detected peaks were reported
[3] 3-day recovery

Table 42. Residues in tissue samples of 3 cows dosed with tebufenozide.

Sample	Dose (ppm)	Tebufenozide (mg/kg)	Metabolite A (mg/kg)	Metabolite B (mg/kg)
Fat			***RH-9526[1]***	
	6	0.029, 0.011 (2x)	0.005, 0.03, 0.02	
	18	0.109, <LOD, 0.063	0.007, 0.05, 0.02	
	60	0.063, 0.38, 0.23	0.15, 0.02, 0.012	
Muscle			***RH-9886***	***RH-0282***
	6	<LOD (3x)	<LOD (3x)	<LOD (3x)
	18	<LOD (2x), 0.022	<LOD (3x)	<LOD (3x)
	60	<LOD, 0.056, 0.028	0.003, 0.004, 0.005	0.008, 0.006 (2x)
Kidney			***RH-9886***	***RH-0282***
	6	<LOD (3x)	<LOD (3x)	<LOD (3x)
	18	<LOD (2x), 0.007	0.003 (2x), 0.004	0.005, 0.004, 0.009
	60	0.007, 0.006, 0.043	0.008, 0.007, 0.004	0.015, 0.014, 0.007
Liver			***RH-2703***	
	6	0.014, 0.008, 0.009	<LOD (3x)	
	18	0.041, 0.026, 0.014	<LOD (2x), 0.007	
	60	0.061, 0.101, 0.066	0.018, 0.066, 0.020	

LOQ for parent and metabolites 0.02 mg/kg, and LOD 0.006 mg/kg
[1] RH-9526 residues are reported as equivalent mg/kg of RH-9886, and represent all fatty acid conjugates of RH-9886.

Overall, residues of tebufenozide and the metabolites were at or below the LOQ in all samples except fat in the 2 lowest dose groups (6 and 18 ppm), and liver (mean 0.03 mg/kg) in the 18 ppm group. In fat both tebufenozide and RH-9526 were detected in all groups, the former at concentrations up to 0.03 mg/kg in the 6 ppm group and 0.1 mg/kg in the 18 ppm group. In the 60 ppm group tebufenozide levels were up to 0.03 mg/kg in milk, 0.04 mg/kg in kidney, 0.06 mg/kg in muscle, and 0.38 mg/kg in fat. No residues were detectable in milk, liver, muscle or kidney samples of any depurated cows, while in fat approximately 30% of the initial residue could still be detected.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information.

In processing

Processing studies were carried out on citrus fruits, peaches, grapes, tomatoes, sugar cane, rape seed and mint.

Citrus fruits. In two US studies on oranges and grapefruit (Dong, 2000) the crops were field-treated with tebufenozide at the proposed GAP rate (Koals, 1999b, Koals and Carpenter, 2000). Samples of orange and grapefruit (about 375 kg) were harvested and stored cold for 3 days. RAC samples were first rinsed with water, then washed in a brush washer with a fruit-cleaning detergent. The washed fruit was stored under ambient conditions until the following day, when it was extracted in an extractor and continuously pumped into a finisher which removed the excess ruptured juice sacs. The resulting fresh juice was sampled for analysis.

The oil/water/peel emulsion from the juice extractor was passed through another finisher which removed peel frits. The remaining water/oil emulsion was stored for a minimum of 5 hours to separate the water which was removed from the concentrated oil emulsion. After further storage at 5°C and centrifugation, additional water was removed and the oil fraction frozen, thawed, filtered, treated with anhydrous sodium sulfate and then refiltered to remove any remaining water. The remaining cold-pressed oil was sampled for analysis.

The solid peel residue from the juice extractor was transferred to a peel hopper and chopped before a liquid lime slurry was added. A press separated the wet pulp into press cake and liquor. The press cake was dried with air at 143°C, resulting in dried pulp containing about 10% moisture with a minimum of charring which was sampled for analysis.

Processed samples were analysed by method TR 34-97-119 (Choo, 1997), with minor modifications for the oil fraction. Residues in the whole fruit, juice and dried pulp were quantified by HPLC-UV and in citrus oil by HPLC-MS. The LOQ was 0.02 mg/kg for all samples. The SAI ranged from 357 to 406 days. The results are shown in Table 43.

Table 43. Residues of tebufenozide in orange and grapefruit products, Windermere, Florida, USA, 1966.

Crop, variety	TTR kg ai/ha	PHI days	Sample	Residues mg/kg	Processing factor	Reference
Orange, Parson Brown	1.38	14	unwashed fruit	0.21[1]		Koals 2000
			washed fruit	0.030[2]	0.14	96-0241
			dried pulp	0.17[2]	0.82	Dong 2000
			juice	<0.02[2]	<0.10	
			oil	4.8[2]	23.1	
Grapefruit, Ruby Red	1.38	13	unwashed fruit	0.089[1]		Koals, 1999b
			washed fruit	0.023	0.26	96-0263, 24:Dong 2000
			dried pulp	0.070	0.79	
			juice	<0.02	<0.23	
			oil	2.6	29.2	

TTR: total treatment rate
[1] Mean of duplicate field samples
[2] Mean of duplicate analyses

Peaches. Peaches from two field trials in New Zealand (Baynon, 1998a) treated at 1 and 2 times the GAP rate were canned at a commercial cannery. The stone was removed, and the peeled and sliced fruit added to cans, followed by a fruit juice/water/sugar syrup. The cans were sealed and heated at temperatures approaching 100°C. Fresh and canned fruit and canned syrup were analysed by method TR 34-95-96 (Deakyne *et al.*, 1995), with an LOQ of 0.01 mg/kg. The SAI was 3 months. The results are shown in Table 44.

Table 44. Residues of tebufenozide in canned peaches, New Zealand, 1996-97 (Baynon, 1998a).

Location variety	TTR kg ai/ha	PHI days	Residue (mg/kg)			Processing factor for canned fruit	Trial no.
			fruit	canned fruit	canned syrup		
Hawkes Bay Golden Queen	0.72	15	0.10	<0.01	<0.01	<0.10	FSLHBRE 02
		22	0.10	<0.01	<0.01	<0.10	
	1.44	15	0.82	<0.01	<0.01	<0.01	
		22	0.47	<0.01	<0.01	<0.02	
Nelson Golden Queen	0.72	22	0.10	<0.01	<0.01	<0.10	FSLNRE05
	1.44	22	0.37	<0.01	<0.01	<0.03	

Grapes. Six processing trials were conducted on grapes harvested in Australia (Hamblin *et al.*, 2001) (Table 45). In Trial RTL 412, residues were found in juice but not in whole grapes, so processing factors could not be determined. No processing factors were calculated for the 0.22 kg ai/ha treatment in trial SCM296/99, since the residues in whole grapes (0.05-0.06 mg/kg) were too low to make accurate estimates.

In the trial with sultana grapes, berries treated at either 0.14 or 0.28 kg ai/ha were processed into raisins by first drying on commercial grape drying racks, then spraying on the rack with an emulsion of vegetable oil and potash (pH 9.5-11) which is used in Australian dried fruit production to facilitate drying by removing the grapes' waxy bloom. When the grapes had dried to 18% moisture they were removed from the rack and finish-dried for a day in direct sunlight. Bunch stems were removed by hand, leaving only raisins and their stems.

Wine and pomace samples were prepared by hand-crushing 1.5-2 kg of grapes from 4 trials. The mixture was inoculated with re-hydrated active wine yeast and fermented on the skins for 7 days at 20-25° C with daily mixing. The must was pressed twice, each time at 0.14 MPa for 3 minutes with mixing of the pomace between pressings. The wine (about 1-1.5 l) was fermented to dryness at 20-25°C. Except in trial PJH151, sulfur dioxide was added after fermentation at 0.1 g/kg and the must allowed to settle at 4°C until clear.

Juice was prepared by pressing of 600 g grapes twice at 0.14 MPa for 3 minutes with mixing of the pomace between pressings, yielding approximately 350 ml of juice.

Wine, pomace, juice and raisins were analysed for tebufenozide by method AL013/92-0 (Holzwarth and Schuld, 1993a) with GC-MS instead of GLC with an NPD; the LOQ was 0.01 mg/kg. The SAI was up to 9 months.

Table 45. Residues of tebufenozide in grapes and processed products, Australia, 1995-99 ((Hamblin *et al.*, 2001).

Year, Location, Variety	TTR kg ai/ha	PHI, days	Sample	Residues mg/kg	Processing factor	Trial no.
1995/96 Irymple (Vic) M12 Sultana	0.14	28	whole fruit dried fruit	0.15[1] 0.10[2]	- 0.62	SCM248/96[3]
	0.28	28	whole fruit dried fruit	0.39 0.34	- 0.87	SCM248/96[3]
1998/99 Dixons Cr. (Vic) Chardonnay	[4]	83, 90, 97	whole fruit pomace wine	0.24, 0.11, 0.09 1.2, 1.6, 0.57 0.04, 0.04, 0.01	- 5.0, 15[5], 6.3 0.17, 0.36, 0.11	RTL528/99
	[6]	21, 28, 35	whole fruit pomace wine	1.3, 0.72, 0.64 5.2, 3.9, 5.6 0.29, 0.14, 0.21	- 4.1, 5.5, 8.7 0.23, 0.19, 0.33	RTL528/99
1998/99Young (NSW) Firmint, Harslevelo	0.27	60, 67, 74	whole fruit pomace wine	0.18, 0.17, 0.12 0.44, 0.63, 0.74 0.03, 0.07, 0.04	- 2.4, 3.7, 6.2 0.17, 0.41, 0.33	PJH 285/99
	0.45	21, 28, 35	whole fruit pomace wine	1.5, 0.58, 1.2 2.5, 2.6, 3.2 0.24, 0.20, 0.24	- 1.6, 4.5, 2.7 0.16, 0.34, 0.20	PJH 285/99
1998/99 Coonawarra SA Shiraz	0.22	84, 91, 98	whole fruit pomace wine	0.06, 0.06, 0.05 0.49, 0.73, 0.61 0.06, 0.04, 0.04	- - -	SCM296/99
	0.37	21, 28, 35	whole fruit pomace wine	0.61, 0.81, 0.78 2.3, 1.8, 3.0 0.26, 0.20, 0.15	- 3.8, 2.3, 3.8 0.43, 0.25, 0.19	SCM296/99
	6 g/hl	0, 14, 28, 35	whole fruit juice pomace wine	1.3, 0.68, 0.27, 0.35 0.12, 0.07, 0.05, 0.08 4.05, -, -, 0.97 0.30, 0.21, 0.09, 0.04	- 0.09, 0.10, 0.19, 0.23 3.0, -, -, 2.8 0.22, 0.31, 0.33, 0.11	PJH151[7]
	12 g/hl	0, 14, 28, 35	whole fruit juice pomace wine	2.6, 2.2, 0.83, 0.71 0.19, 0.24, 0.13, 0.09 9.8, -, -, 2.6 0.50, 0.46, 0.19, 0.22	- 0.07, 0.11, 0.16, 0.13 3.8, -, -, 3.6 0.19, 0.21, 0.23, 0.31	PJH151[7]
	6 g/hl	0, 14, 28, 35	whole fruit juice	<0.01 <0.01, 0.06, 0.04, 0.03	- -	RTL412[7]
	12 g/hl	0, 14, 28, 35	whole fruit juice	<0.01 0.13, 0.09, 0.04, 0.12	- -	RTL412[7]

[1] mean of 0.09, 0.13, 0.22, 0.18 and 0.15
[2] mean of 0.06, 0.03, 0.13, 0.10 and 0.16
[3] in the control dried fruit samples, residues varied from <0.01 to 0.04 mg/kg
[4] 2 applications of 0.006 kg ai/hl, sprayed well in excess of run-off
[5] outlier according to Dixons test, not used to estimate the mean processing factor for pomace
[6] 3 applications of 0.006 kg ai/hl, 1st and 2nd sprayed well in excess of run-off, 3rd >0.18 kg ai/ha
[7] analytical data were included in the analytical report of trial SCM 248/96. No field reports available. Application rates were 0.006 and 0.012 kg ai/hl, number of treatments and interval(s) unknown.

Tomatoes. Residues in processed fractions of tomatoes treated with tebufenozide at the GAP rate in the USA, and at approximately twice the GAP rate in Europe are shown in Table 46.

In the US trial (Carpenter, 1997a), about 150 kg tomato fruit was first washed with water and chlorinated water and then crushed. The mash was heated to 99°C, then screened to remove the seed and peel fraction (wet pomace) and produce juice. The juice was condensed under vacuum to form purée (9% natural tomato soluble solids, NTSS) and then canned and heated to 94°C for 5 minutes. A sub-sample of the purée was further condensed under vacuum to form paste (25% NTSS), which was also canned and heated to 94°C for 5 minutes. Samples were analysed by method TR 34-95-66

(Deakyne *et al.*, 1995). The LOQ in tomatoes was 0.02 mg/kg; the method was not validated for processed commodities. The SAI was 242 ± 2 days.

Processed commodities were prepared from tomatoes collected from four supervised trials in France, Greece and Spain (Bürstell *et al.*, 1996). 5 kg of tomatoes were washed in water at 25°C with gentle motion for 1 minute. The ratio of tomatoes to water was 1:2. The tomatoes were then divided into 2 portions, one for canning, the second for processing into juice, purée and paste. The canning process was one which retained the peel, since this was expected to leave the highest residues in the processed fruit. Washed, unpeeled tomatoes were drained, the calyx was removed, and the peel pricked several times with a fork. The tomatoes in jars filled with 1% salt water were then sterilized at 115-120°C for 20 minutes. The fruit and preserving liquid were later separated for analysis.

The second portion of washed tomatoes was chopped and blanched. A tenfold volume of water was added to the chopped fruit in a saucepan and the mixture heated to 70°C for 2-5 minutes. After blanching, the mashed pulp was separated from the pomace fraction (skin, pith and seeds) by sieving. After separation, part of the pulp was used to produce paste, and part for juice. To prepare paste, the sieved pulp was rotary-evaporated at a pressure of 300-100 mbar and a maximum water bath temperature of 65°C until the volume had been reduced to about one fifth. The paste was then bottled and sterilized by heating in an oven at 115-120°C for 20 minutes. Tomato juice was prepared by heating the sieved pulp in an oven at 113-117°C for 15 minutes.

All tomato samples were analysed by the GC-MS method AL 013/92-0 (Holzwarth and Schuld, 1993a-c). The LOQ was 0.02 mg/kg for fruit and fractions from the processing except juice, wash water and canning liquid where the LOQ was 0.01 mg/kg. The SAI ranged from 185 to 252 days.

Table 46. Residues of tebufenozide in tomatoes and processed products.

Country, year, location, variety	TTR kg ai/ha	PHI days	Sample	Residues mg/kg	Processing factor	Reference
US, 1996, Porterville (CA) 88-90	1.16	7	unwashed fruit washed fruit purée paste	0.35 0.090[1] 0.11[1] 0.29[1]	- 0.26 0.31 0.83	Carpenter 1997b 96-0249 [2,3]
France, 1995, St.Sixte (Midi-Pyrénées) Cannery row	1.08	3	unwashed fruit washed fruit wash water juice, unsterilized juice, sterilized wet pomage fruit, preserved canning liquid paste	0.24, 0.27 0.08, 0.12 <0.01, <0.01 0.05, 0.05 0.05, 0.06 0.24, 0.29 0.07, 0.08 0.01, 0.02 0.18, 0.20	- 0.33, 0.44 0.21, 0.22 0.29, 0.30 0.75, 0.74	Bürstell, 1996 FRA 00 01/ FRA 00 02

Country, year, location, variety	TTR kg ai/ha	PHI days	Sample	Residues mg/kg	Processing factor	Reference
Greece, 1995, Korifi (Macedonia) Rio Grande	1.08	3	unwashed fruit	0.18	-	Bürstell, 1996 GRC 00 01
			washed fruit	0.07	0.39	
			wash water	<0.01		
			juice, unsterilized	0.03		
			juice, sterilized	0.03	0.17	
			wet pomage	0.25		
			fruit, preserved	0.06	0.33	
			canning liquid	0.01		
			paste	0.13	0.72	
Spain, 1995, Brenes/Sevilla (Andalucia) Red Hunter	0.72	3	unwashed fruit	0.25	-	Bürstell, 1996 ESP 00 01
			washed fruit	0.09	0.36	
			wash water	<0.01		
			juice, unsterilized	0.03		
			juice, sterilized	0.03	0.12	
			wet pomage	0.31		
			fruit, preserved	0.05	0.20	
			canning liquid	<0.01		
			paste	0.15	0.60	

[1] mean of two laboratory replicates
[2] almost all of the fruit used for processing came from the field sample which had a residue of 0.35 mg/kg. In the replicate field sample, approximately 95% of the tomatoes were green and unsuitable for processing.
[3] no validation data for analysis of processed commodities available for the method used

Sugar cane. Four processing studies were conducted with sugar cane harvested from supervised trials according to GAP, two (trials 94-0168 and 95-0291) in the state of Hawaii (Filchner, 1997a,b) and two (both from trials 97-0130) in Louisiana (Bergin, 1998).

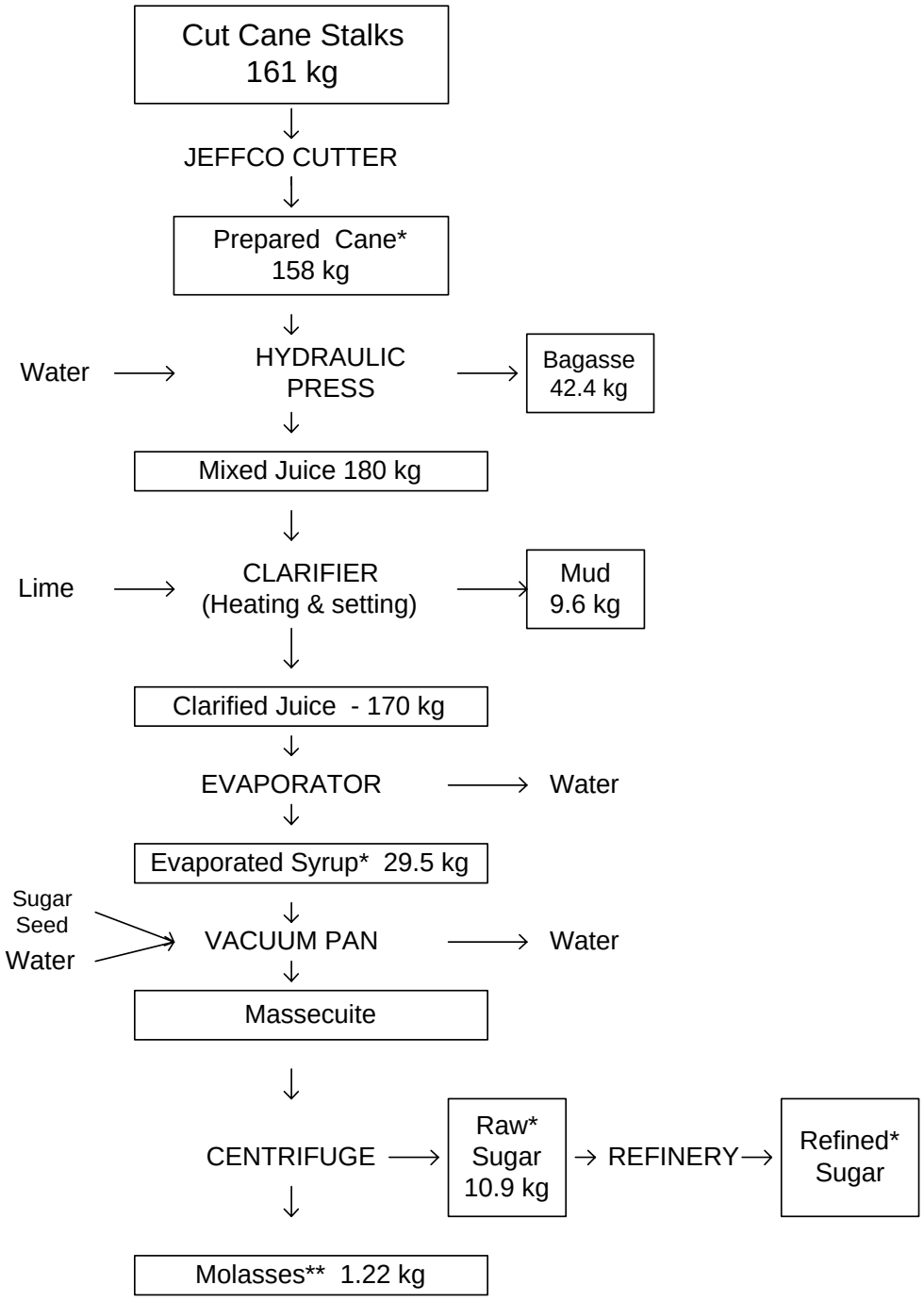
The processing procedures were the same in all the trials, although the samples analysed differed. The cane was processed into molasses and sugar by the procedure shown in Figure 2. The cane was chopped and, after water was added, pressed to produce juice and bagasse. The juice was mixed with lime, boiled for 3 minutes and then allowed to settle for 1 hour in the clarifier, yielding mud and clarified juice. In the evaporator the juice is concentrated to syrup, which is mixed with water and sugar seed in a vacuum pan and concentrated by boiling under vacuum to obtain A-strike massecuite. The massecuite is centrifuged to produce A-strike raw sugar and A-strike molasses (A-strike process). The A-strike molasses is blended with additional syrup, water and sugar seed and again concentrated in a vacuum pan to yield B-strike massecuite, which is centrifuged to produce B-strike raw sugar and B-strike molasses (B-strike process). The B-strike molasses and additional syrup, water and sugar seed are concentrated under vacuum to C-strike massecuite which is crystallized. The cured C-strike massecuite from the crystallizer is centrifuged to produce C-strike raw sugar and C-strike (final) molasses (C-strike process). Refined sugar was prepared from raw sugar dissolved in pure water. In trials 94-0168 and 95-0291 only the A-strike raw sugar was used for processing to refined sugar, whereas in trial 97-0130 A- and B-strike raw sugars were used. The raw sugar syrup was decolorized with activated charcoal, filtered and evaporated to crystallize the refined sugar, which was separated by centrifugation. Figure 2 shows the weights of the individual fractions isolated in the 1994 trial; the weight distribution was similar in the other 3 trials.

Method TR 34-95-66 (Deakyne *et al.*, 1995) was used in trial 95-0291, method TR 34-94-41 (Chen *et a.l*, 1994c) in trial 94-0168, and method TR 34-97-115 (Filchner and Deakyne, 1997) in trial 97-0130. The LOQ was 0.01 mg/kg in all methods. The SAI ranged from 115 to 169 days in trials 94-0168 and 97-0130 and was 397 days in trial 95-0291. The results are shown in Table 47.

Table 47. Residues of tebufenozide in sugar cane and processed products, USA.

Year, location variety	TTR kg ai/ha	PHI days	Sample	Residues, mg/kg	Processing factor	Reference
1994, Washington (LA), 357	1.12 (SC)	14	sugar cane	0.28[1]	-	Filchner, 1997b 94-0168
			raw sugar A	0.017[2]	0.06	
			raw sugar B	0.054	0.19	
			raw sugar C	0.060	0.21	
			refined sugar	<0.01[2]	<0.04	
			final molasses	0.32[2]	1.1	
			molasses A	0.49	1.75	
			molasses B	0.53	1.9	
			molasses blend	0.35	1.25	
			evaporated syrup	0.22	0.79	
1995, Waialua (HI), 73-6110	1.16 (WP)	14	sugar cane	-[3]	-	Filchner, 1997a 95-0291
			refined sugar	<0.01[2]	-	
			raw sugar	0.016[2]	-	
			final molasses	0.70[1]	-	
1997, Port Allen (LA), CP-845	1.16 (SC)	13	sugar cane	0.54[1]	-	Bergin, 1998 97-0130
			burned stems	0.27[1]	0.50	
			raw sugar A	0.26	0.48	
			raw sugar B	0.30	0.55	
			refined sugar	<0.01[2]	<0.019	
			final molasses	4.9[2]	9.0	
1997, Port Allen (LA), CP-845	1.16 (WP)	13	sugar cane	0.62[1]	-	Bergin, 1998 97-0130
			burned stems	0.29[1]	0.47	
			raw sugar A	0.24	0.39	
			raw sugar B	0.33	0.53	
			refined sugar	<0.01[2]	<0.016	
			final molasses	4.7[2]	7.6	

[1] mean of duplicate field samples
[2] mean of analytical duplicates
[3] no RAC sample was taken for analysis so processing factors cannot be calculated.



* Sampling points for analysis.
**Molasses returned to the vacuum pan to isolate one or more additional batches of raw sugar before the final molasses fraction is isolated; final molasses is subsampled for analysis.

Figure 2. Sugar cane processing scheme.

Rape seed (Canola). Treated and untreated canola seed was processed into refined oil (Dorschner and Breuninger, 1998f) by a small-scale process (Figure 3). Canola seeds were dried in an oven at 54-71°C to a moisture content of 7-10%. After aspiration and screening to separate foreign particles, whole cleaned seeds were flaked, heated to 82-99°C for 10-15 minutes, and then pressed in an expeller to liberate most of the crude oil. Residual crude oil remaining in the solid material

(presscake) was extracted by submerging three times in hexane at 43-52°C for 15-30 minutes and subsequent draining. After the final draining, warm air was forced through the extracted presscake to remove residual hexane. The final solvent-extracted presscake is the meal fraction. The drained crude oil/hexane mixture (miscella) was heated to 73-90°C to remove hexane.

Crude oil recovered from the expeller and from solvent extraction was combined and refined. It was first treated with phosphoric acid (40-45°C, 30 min, stirred at 70 rpm). After the addition of NaOH, the solution was mixed (40-45°C, 250 rpm; 20 min, and 60-65°C, 70 rpm, 10 min). The neutralized oil was allowed to settle for 1 hour at 60-65°C and then overnight in a refrigerator. The refined oil was decanted and filtered; the solid fraction is soapstock. All samples were analysed by method TR 34-96-135 (Wu *et al.*, 1996). The LOQ was 0.01 mg/kg for the seed and meal and 0.03 mg/kg for soapstock and oil. The longest SAI was 68 days for oil and meal fractions and 20 days for soapstock. Storage stability was demonstrated in oil stored frozen for 83 days, and meal for 90 days. The results are shown in Table 48.

Table 48. Residues of tebufenozide in processed rape (canola) seed products, USA, 1996 (Dorschner and Breuninger, 1998f).

Location, Variety	TTR kg ai/ha	PHI days	Sample	Residues, mg/kg	Processing factor	Trial no.
Moxee (WA) Tobin	1.12	9	seed meal soapstock refined oil	0.95 0.11 1.2 2.6	- 0.12 1.3 2.7	96-WA*40
Langdon (ND) Hyola 401	1.09	14 (+7) [1]	seed meal soapstock refined oil	0.52[2] 0.10 0.46 0.95	- 0.19 0.88 1.8	96-ND03

[1] After swathing, rape seed was left in the field to dry for 7 days. In ND, this mimics local commercial practice
[2] Mean of 3 replicate field samples (0.47, 0.61, 0.49)

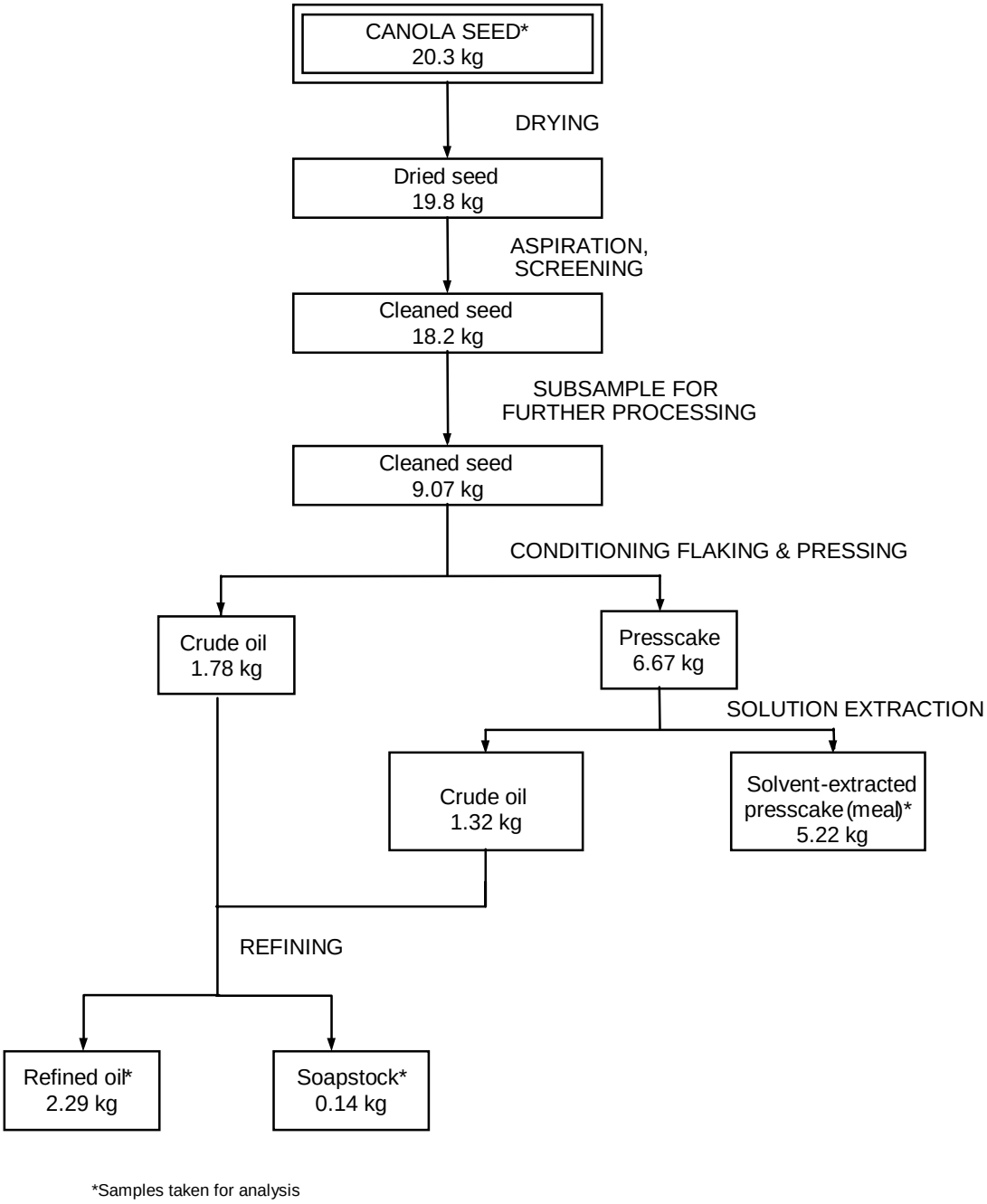


Figure 3. Rape seed processing.

Mint. Mint oil was producted from mint treated at the GAP rate in the USA in 1996 (Dorschner and Breuninger, 1998e) by steam distillation of the treated foliage according to a standard protocol. Approximately 3 kg of leaves in a cloth mesh bag were transferred to an electric boiler connected to a condenser and separator. The mint sample was tightly packed in the boiler to ensure that steam would pass through the sample. Steam was blown into the boiler and the distillate passed through the water-cooled condenser into a separatory funnel. After 1 hour the steam flow was disconnected and the water drained from the funnel. The oil was sampled and analysed by method TR 34-96-135 (Wu *et al.*, 1996), with an LOQ of 0.02 mg/kg. A Florisil clean-up to remove residual oil was added before basic alumina chromatography. The SAIs for mint oil were 225 and 273 days. Storage stability was demonstrated in mint oil stored frozen for 285 days.

Table 49. Residues of tebufenozide in mint and mint oil, USA, 1996 (Dorschner and Breuninger, 1998e).

Location, Variety	TTR kg ai/ha	PHI days	Sample	Residues, mg/kg	Processing factor	Trial no.
Prosser (WA) Spearmint, Native	1.16	14	leaves and stems oil	8.4[1] 0.33	- 0.04	96-WA*37
Marquette Co. (WI) Spearmint, Scotch	1.50	14 (+1) [2]	leaves and stems oil	7.5[1] 0.12	- 0.02	96-WI13

[1] mean of field duplicates
[2] the mint was collected after drying in the field for 1 day

Residues in the edible portion of food commodities

The available information is reported in the previous section.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported by the manufacturers.

Commodity	Country	MRL, mg/kg
Avocado	New Zealand	0.5
Bush berries, caneberries (excluding grapes and cranberries)	USA	3
Citrus	Brazil	0.5
Citrus	Greece	1
Citrus	Italy	1
Citrus	Morocco	0.5
Citrus	Portugal	1
Clementine, Mandarin	Spain	1
Citrus	Tunisia	0.5
Citrus	Uruguay	0.5
Cranberries	USA	1
Fruiting vegetables, other than cucurbits	USA	1
Eggplant	Belgium	0.2
Peppers	Belgium	0.2
Peppers	Mexico	1
Peppers	Spain	1
Tomato	Argentina	0.5
Tomato	Belgium	0.2
Tomato	Brazil	0.5
Tomato	Mexico	1
Tomato	Spain	1
Tomato	Uruguay	0.5
Grapes Dried grapes Grape pomace, dry	Australia	2 4 10
Head and stem Brassica Leafy Brassica vegetables	USA	5 10
Cabbage species	Switzerland	0.5
Kale	Brazil	2

Commodity	Country	MRL, mg/kg
Kale	Uruguay	2
Leafy green vegetables Leaf petioles (including celery)	USA	10 2
Lettuce	Switzerland	1
Spinach	Switzerland	1
Turnip, garden	USA	0.3 roots 9 greens
Mint	USA	10
Rape seed (Canola) Rape seed oil, edible	USA	2 4
Stone fruits excluding cherries	New Zealand	0.5
Sugar cane Sugar cane molasses	USA	1 3
Tree nuts including pistachio	USA	0.1 25 almond hulls
Walnut	France	0.5
Walnut	Mexico	0.1
Milk	USA	04**
Meat (from mammals other than marine mammas)	USA	08**
Edible offal (Mammalian)	USA	08**
Mammalian fats (except milk fats)	USA	0.1**

**includes residues of tebufenozide and its 3 metabolites

The Meeting was informed that the following MRLs for tebufenozide were pending.

Commodity name	Country	MRL, mg/kg
Avocado	Australia	1
Citrus	Australia	1
Citrus fruits	USA	0.8, oil 15
Lettuce	Spain	1.0
Macadamia nuts	Australia	0.05

APPRAISAL

The insecticide tebufenozide was first evaluated by the 1996 JMPR, which recommended MRLs for grapes, pome fruits, husked rice and walnuts. In 1997, an additional MRL for kiwifruit was recommended, and in 1999 data on pome fruits and grapes were re-evaluated. The present Meeting received information requested by the 1996 JMPR, including information about rotational crops, animal feeding studies, storage stability and data on residues on raisins. Furthermore, the results of new supervised trials and analytical methods for new and previously considered commodities were submitted, and information on currently registered GAP was provided.

The abbreviations used for metabolites are as follows:

RH-5992	tebufenozide, N-tert-butyl-N'-(4-ethylbenzoyl)-3,5-dimethylbenzohydrazide
RH-9886	N-tert-butyl-N'-(4-ethylbenzoyl)-3-hydroxymethyl-5-methylbenzohydrazide
RH-1788	N-tert-butyl-N'-[4-(1-hydroxyethyl)benzoyl]-3,5-dimethylbenzohydrazide
RH-0282	N-tert-butyl-N'-[4-(1-hydroxyethyl)benzoyl]-3-hydroxymethyl-5-methylbenzohydrazide

RH-0126	N-tert-butyl-N'-[4-(1-hydroxyethyl)benzoyl]-3-carboxy-5-methylbenzohydrazide
RH-2703	N-tert-butyl-N'-(4-carboxymethylbenzoyl)-3,5-dimethylbenzohydrazide
RH-6595	N-tert-butyl-N'-(4-acetylbenzoyl)-3,5-dimethylbenzohydrazide
RH-9871	N-tert-butyl-N'-(4-acetylbenzoyl)-3-hydroxymethyl-5-methylbenzohydrazide
RH-2631	N-tert-butyl-N'-(4-acetylbenzoyl)-3-carboxy-5-methylbenzohydrazide
RH-0875	N-tert-butyl-N'-(4-ethylbenzoyl)-3,5-dicarboxybenzohydrazide
RH-9841 (RH-5992-olefin)	N-tert-butyl-N'-(4-vinylbenzoyl)-3,5-dimethylbenzohydrazide
RH-9526	Stearic acid conjugate of RH-9886

Environmental fate

Soil

In 1996, the Meeting requested a detailed report of a completed study of uptake by rotational crops that the Meeting was informed was available. The current Meeting received the results of both confined and field studies of rotational crops.

In the study of confined rotational crops, tebufenozide labelled with ^{14}C in the ethylbenzoyl ring, the dimethylbenzoyl ring or the central carbon of the *tert*-butyl group was applied in four applications to bare ground, each at the maximum rate of use described on the label for crops in the USA that could be rotated. Rotational crops (wheat, collards and turnips) were planted back 30, 90, 250 and 365 days after last treatment of the initial crop.

High concentrations of residues were found in wheat at 30 days' plant-back: expressed as parent equivalents, 0.4 mg/kg TRR in grain, 7.2 mg/kg in straw and 2.6 mg/kg in forage. At 90 days plant-back, the TRR had fallen to 0.06 mg/kg in grain, 0.4 mg/kg in straw, and 0.3 mg/kg in forage. The concentrations at 250 and 365 days plant-back were comparable. The main component in all wheat samples was RH-1788, either as the free alcohol or conjugated to glucose or malonylglucose. The parent compound was present in only small amounts (1%) in samples of straw and grain at 30 days plant-back and was not detected in any of the wheat samples at longer plant-back intervals.

The concentration of residues in collards at 30 days plant-back was about 0.1 mg/kg. The residues included the glucose conjugates of RH-1788 and small amounts of glucose conjugates of RH-9886 and RH-0282; the conjugates constituted 42% of the TRR. The main individual component (15%) in collards was RH-5992-olefin (RH-9841). The parent compound was not found.

The concentrations of residues were about 0.1 mg/kg in turnip roots and 0.4 mg/kg in turnip tops at 30 days plant-back. The parent compound was the most prevalent component in turnip roots (20% of TRR, 0.02 mg/kg) and accounted for about 7% of the TRR (0.03 mg/kg) in turnip tops. Sugar conjugates of RH-1788 constituted 14% of the TRR in both turnip roots and tops.

Thus, the main residues in wheat samples were RH-1788 and its sugar conjugates. Only the turnip root crop had a significant percentage of tebufenozide. The leafy crop collard contained mainly sugar conjugates of RH-1788 and tebufenozide-olefin (RH-9841).

The metabolites found in rotational crops were similar to those identified previously in the studies of crop metabolism, except that the parent compound was either a minor component or undetectable and large amounts of sugar conjugates were formed from the metabolites. All the metabolites found in rotational crops, except the conjugated metabolites, have also been characterized in rats. In soil, the parent compound and RH-6595 were characterized.

In the field study of rotational crops, leaf lettuce was planted as the primary crop and tebufenozide was applied at maximum GAP rate in the USA. The lettuce was removed at maturity, and rotational crops were planted 30 and 120 days after the last application.

The high-moisture crops, including leaf lettuce, radish tops and roots, squash, green and bulb onion and green peppers, were analysed for residues of tebufenozide and its olefin metabolite RH-9841. These compounds were found at concentrations below the LOQ (0.01 mg/kg) in all crops tested.

The low-moisture crop samples (wheat, sorghum and soya beans), planted at 30 days plant-back, were analysed for residues of tebufenozide and its alcohol metabolite RH-1788. No residues of tebufenozide or RH-1788 were found in wheat or sorghum grain or soya bean seed, at concentrations above the LOQ (0.02 mg/kg). In the plant parts used for animal feed, residues of RH-1788 were found in wheat straw (0.28 mg/kg), wheat hay (0.12 mg/kg) and soya bean forage (0.03 mg/kg); no residues of RH-1788 were found in wheat forage, sorghum forage, fodder or stover or soya bean hay. No residues of the parent compound were found at concentrations above the LOQ (0.02 mg/kg) in any of the animal feed commodities from wheat, sorghum and soya bean.

Methods of analysis

Two analytical methods that had been evaluated by the 1996 JMPR were updated with respect to the methods used to measure residues of tebufenozide in six vegetable crops (lettuce, cabbage, spinach, mustard greens, broccoli and celery) and in pecans. The LOQs were unchanged. The GLC method for determining tebufenozide in grapes, evaluated by the 1996 JMPR, was validated for lettuce. The LOQ was 0.02 mg/kg.

An analytical method for determining tebufenozide in citrus fruit and its processed fractions includes the extraction and partitioning of whole citrus fruit, juice and dry pulp samples and direct partitioning of citrus oil samples. The samples are cleaned-up on a carbon and C-18 solid-phase extraction column and analysed by HPLC with UV detection. The LOQ was 0.02 mg/kg. A confirmatory method is based on the same extraction and purification procedure with HPLC–MS for quantification of residues in whole fruit, juice and dry pulp and HPLC–MS/MS for quantification of residues in oil.

A method for measuring residues of tebufenozide in sugar cane and sugar cane processed fractions and one for residues in cotton seed and cotton seed processed fractions were submitted. The method for cotton seed was used in trials on rape seed. After extraction, partitioning and further purification, HPLC–UV was used for quantification. The LOQ was 0.01 mg/kg for all matrices with both methods. The concentrations of residue obtained by HPLC–UV were confirmed by the HPLC–MS method.

A method was reported for the detection and quantification of residues of tebufenozide and its metabolites RH-9841 and RH-1788 in rotational crops. After extraction, partitioning and clean-up, residues were quantified by LC–MS. The LOQ was 0.01 mg/kg for tebufenozide and RH-9841 in high-moisture crops such as root and leafy vegetables, and 0.02 mg/kg for tebufenozide and RH-1788 in low-moisture crops such as grains.

In a method for enforcement of concentrations of residues of tebufenozide and its metabolites in animal commodities, tebufenozide was quantified in all matrices, RH-9886 in muscle and kidney, RH-0282 in milk, muscle and kidney, fatty acid conjugates of RH-9886 in milk and fat and RH-2703 in liver. Residues of fatty acid conjugates of RH-9886 were hydrolysed with hydrochloric acid, and the hydrolysed and normal extracts were partitioned, cleaned-up and then quantified by HPLC–UV. The LOQ was 0.01 mg/kg for all three analytes in milk and 0.02 mg/kg for all analytes in animal

tissues. The confirmatory HPLC methods for all matrices consisted of use of modified mobile phases with MS detection.

Stability of residues in stored analytical samples

As described by the 1996 JMPR, the stability of tebufenozide at –20°C has been demonstrated in apples (at least 33 months), apple juice (at least 6 months), grapes and wine (at least 12 months) and walnuts (at least 18 months). The 1996 JMPR requested representative data on the stability of residues on leafy vegetables for the full duration of the storage studies that the Meeting was informed were in progress, and on the stability of residues in analytical samples of rice stored for longer than the 20–21 days already reported. The present Meeting received reports on stability in storage for wheat, rice, green onion, citrus oil, lettuce and animal commodities.

The stability of RH-6595, RH-1788 and the RH-1788-glucose conjugate was tested in wheat straw derived from a study of rotational crops for only the last 2 years of a 4-year storage period. Although little or no degradation was observed, no information was available about a possible change in composition during the first 2 years of storage. Since degradation is usually not a linear process, extrapolation is not possible. The composition of the residue in extracts of wheat forage samples stored for 4 years, which contained mainly the glucose and malonylglucose conjugates of RH-1788, was comparable to that of the remainder of the extracts stored for 4 years and 7 months, but no information was available about stability during the first 4 years of storage.

The stability of tebufenozide and its metabolites RH-1788, RH-6595 and RH-9886 was examined in frozen stored samples of rice straw and grain from a study of metabolism. Samples were first analysed after 2 years and were re-analysed after another 5 years of frozen storage. The proportions of the metabolites remained essentially the same. Although this study did not cover the first 2 years of storage, it satisfied the request of the 1996 JMPR.

In support of the findings on the stability of tebufenozide residues in stored rotational crops, the metabolite RH-9841 was shown to be stable in green onion for at least 24 months at < –10 C. The interval between storage and analysis for high-moisture crop samples in the study of rotational crops was $\leq$ 20 months.

The stability of tebufenozide was demonstrated in orange oil frozen at –20ºC for at least 15 months, and in head lettuce stored at –15 ± 10 °C for up to 36 months.

In the supervised trials, the stability of tebufenozide was shown to be at least 189 days in blueberries, 322 days in raspberries, 30 days in cranberries, 279 days in turnip roots and foliage, 236 days in rape seed, 90 days in rape seed meal, 83 days in rape seed oil, 279 days in mint and 285 days in mint oil. The periods evaluated covered the interval between storage and analysis for the crops in the supervised trials.

The intervals between storage and analysis for leafy vegetables and tree nuts are covered by the data on the stability of head lettuce and walnuts, respectively. No studies of the stability of tebufenozide in storage were conducted with citrus fruit (interval between storage and analysis in supervised trials, $\leq$ 2 years), stone fruit (interval, $\leq$ 4 months), avocado (interval, $\leq$ 1 year), cabbage and broccoli (interval, $\leq$ 2.5 years), fruiting vegetables (interval, $\leq$ 11 months), celery (interval, $\leq$ 1.5 years) or sugar cane (interval, $\leq$ 14 months). The stability of tebufenozide in these commodities can be inferred from the stability of its residues in other crop matrices.

In animal commodities, the stability of tebufenozide and the metabolites of possible concern in each matrix during a certain duration at –15 ± 10 °C was tested. In milk (RH-0282 and RH-9526, 192 days), meat (RH-9886 and RH-0282, 203 days), liver (RH-2703, 182 days) and fat (RH-9526, 145 days), no decrease was found in the concentrations of tebufenozide and the metabolites measured.

Although these data do not cover the entire interval between storage and analysis of milk and fat samples in the feeding trial in cows (250 days for milk and 274 days for fat), as judged from the stability observed, there should be no concern that the measured concentrations of residues were influenced by the storage period.

Definition of the residue

In 1996, the Meeting agreed that the residue for compliance with MRLs and for estimating dietary intake should be defined as tebufenozide. The residue is fat-soluble.

The Meeting agreed that this residue definition would apply to both plant and animal commodities.

Results of supervised trials

Citrus fruit

Five trials in Spain and five trials in Italy on oranges were conducted according to Spanish and Italian GAP for citrus fruit (two applications at 0.018 or 0.019 kg ai/hl; PHI, 14 days). The concentrations of residues of tebufenozide in these trials were: 0.21, 0.25, 0.36, 0.38, 0.39, 0.43, 0.48, 0.56, 0.60 and 0.78 mg/kg in whole fruit and 0.021, 0.03, 0.04 (2), 0.05, 0.053, 0.11, 0.13 (2) and 0.15 mg/kg in pulp.

Five trials with oranges in Australia and nine in the USA, two trials on lemon in Australia and five in the USA, six trials with grapefruit in the USA and two trials with mandarin in Australia were conducted according to the respective pending national GAPs for citrus fruit. Trials based on pending GAP were not taken into consideration by the Meeting.

The concentrations of residues in mandarin in trials in Italy and Spain conducted according to approved GAP for citrus fruit, with a PHI of at least 14 days, were: 0.30 (2), 0.42, 0.59, 0.60 (2), 0.78, 0.84 and 0.95 mg/kg in whole fruit and 0.069, 0.076, 0.082, 0.092, 0.14 and 0.18 mg/kg in pulp.

As the concentrations of residues of tebufenozide were comparable in the whole commodity and in edible portions (pulp) of citrus fruits, the Meeting agreed to evaluate the combined data for oranges and mandarins to apply to citrus fruit. The concentrations of residues in citrus fruit were, in ranked order (median underlined): 0.21, 0.25, 0.30 (2), 0.36, 0.38, 0.39, 0.42, 0.43, 0.48, 0.56, 0.59, 0.60 (3), 0.78 (2), 0.84 and 0.95 mg/kg in whole fruit and 0.021, 0.03, 0.04 (2), 0.05, 0.053, 0.069, 0.076, 0.082, 0.092, 0.11, 0.13 (2), 0.14, 0.15 and 0.18 mg/kg in the edible portion (pulp).

The Meeting estimated a maximum residue level of 2 mg/kg for tebufenozide in citrus fruit, and an STMR value of 0.079 mg/kg and a highest residue of 0.18 mg/kg for tebufenozide in the edible part of citrus fruit (the pulp).

Stone fruit

The values for residues in peaches and nectarines were derived directly from measurements in fruit without stones, whereas the MRL for peaches and nectarines applies to residues measured in fruit without stones but calculated and expressed as the whole fruit. The weight of the stone is set at a default value of 10% of the total weight of the fruit (see table “Unit weights and edible %” prepared by GEMS/Food for the CCPR and JMPR; nectarines were assumed to resemble peaches). As correction for the weight of the stones resulted in only marginally different figures, the values for residues were used without correction.

Three trials on peach in New Zealand were conducted according to national GAP for stone fruit (four applications at 0.12 kg ai/ha; PHI, 14 days), yielding concentrations of residues of 0.09, 0.10 and 0.14 mg/kg; and three trials on nectarines conducted at GAP yielded concentrations of residues of 0.05, 0.22 and 0.26 mg/kg.

As the concentrations of residues in the trials on peach and nectarine were within the same range, the Meeting agreed to combine the data for mutual support. The concentrations of residues in peach and nectarine were, in ranked order: 0.05, 0.09, 0.10, 0.14, 0.22 and 0.26 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR value of 0.11 mg/kg and a highest residue of 0.23 mg/kg for tebufenozide in peaches and nectarines.

Berries

Eight field trials were conducted in the USA on blueberry according to national GAP (four applications of 0.29 kg ai/ha; PHI, 14 days), resulting in concentrations of residues of 0.30, 0.34, 0.50, 0.56, 0.81, 1.1, 1.2 and 1.7 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg, an STMR value of 0.685 mg/kg and a highest residue of 1.7 mg/kg for tebufenozide in blueberries.

Five trials on raspberries conducted in the USA according to GAP resulted in concentrations of residues of 0.36, 0.50, 0.56, 0.82 and 0.86 mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg, an STMR value of 0.56 mg/kg and a highest residue of 0.86 mg/kg for tebufenozide in raspberries.

Five trials on cranberry (one in Canada, four in the USA) were conducted according to GAP in the USA (four applications of 0.29 kg ai/ha; PHI, 30 days). The concentrations of residues were < 0.01, 0.016, 0.042, 0.046 and 0.28 mg/kg. The Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR value of 0.042 mg/kg and a highest residue of 0.28 mg/kg for tebufenozide in cranberries.

Residues of tebufenozide in grapes were evaluated by the 1996 and 1999 JMPR . The ranked order of concentrations of residues from 18 trials in France and Germany was 0.05, 0.06, 0.07, 0.08, 0.12, 0.18, 0.21, 0.22, 0.24, 0.26 (2), 0.27, 0.28 (3), 0.4, 0.42 and 0.5 mg/kg (see Annex 5, reference 87). Re-evaluation of data from four trials in France conducted according to current GAP in Portugal (three applications of 0.144 kg ai/ha; PHI, 14 days; GAP was pending in 1996) resulted in concentrations of residues of 0.16, 0.29, 0.68 and 0.81 mg/kg (Annex 5, reference 78). Trials on grapes conducted in Australia in 1995 and 1998 according to Australian GAP (0.006 kg ai/hl, ≤ 0.030 kg ai/hl for low volume spraying; PHI, 21 days) resulted in highest residues at least 21 days after the last treatment of 0.22, 0.39, 0.81, 1.1, 1.3 and 1.5 mg/kg. The four re-evaluated French trials and the Australian trials yielded higher concentrations and were considered to represent different data populations from the study in Germany and the previously considered French trials. Therefore the Meeting estimated the maximum residue level, the STMR value and the highest residue on the basis of the four re-evaluated French and six Australian trials.

The concentrations of residues in grapes in trials with Portuguese and Australian GAP were: 0.16, 0.22, 0.29, 0.39, 0.68, 0.81 (2), 1.1, 1.3 and 1.5 and mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg for tebufenozide in grapes to replace the previous recommendation of 1 mg/kg, an STMR value of 0.745 mg/kg and a highest residue of 1.5 mg/kg.

Avocado

Trials were conducted in Australia and New Zealand on avocado according the approved GAP of New Zealand (four applications of 0.006 kg ai/hl; PHI, 21 days). The results of one trial (Walkamin) were not used because there had been heavy rainfall after the last application and the values for residue in this trial were considerably lower than those in other trials. The concentrations of residues measured in stoneless fruit, but calculated for whole fruit, were: 0.08, 0.10, 0.17, 0.18, 0.28, 0.45 and 0.47 mg/kg. The concentrations in the edible portion of avocados (stoneless fruit) were: 0.09, 0.12, 0.19, 0.21, 0.33, 0.52 and 0.53 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg for tebufenozide in avocados and an STMR value of 0.21 mg/kg and a highest residue of 0.53 mg/kg for tebufenozide in the edible part of avocados (seeded avocados).

Cabbage

Trials on cabbage in the USA were summarized by the 1996 JMPR but, because there was no approved GAP at that time, no MRLs were proposed.

In 14 trials on cabbage conducted in the USA according to GAP for brassica (seven applications at 0.14 kg ai/ha; PHI, 7 days), the concentrations of residues were 0.004, 0.03, 0.04, 0.09, 0.11, 0.17, 0.30, 0.38, 0.53, 0.78, 1.0, 1.3, 4.3 and 4.6 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 0.34 mg/kg and a highest residue of 4.6 mg/kg for cabbage.

Broccoli

Trials on broccoli in the USA were summarized by the 1996 JMPR but, because there was no approved GAP at that time, no MRLs were proposed.

Eleven trials on broccoli conducted in compliance with GAP in the USA for brassica resulted in concentrations of residues of 0.01, 0.07, 0.09, 0.1, 0.11 (2), 0.12, 0.24, 0.31, 0.33 and 0.34 mg/kg.

The Meeting estimated a maximum residue level at 0.5 mg/kg, an STMR value of 0.11 mg/kg and a highest residue of 0.34 mg/kg for broccoli.

Tomato

Trials on tomato were performed in both greenhouses and the field. Five trials conducted in greenhouses in southern Europe in 1996 according to Spanish GAP (three applications of 0.018 kg ai/hl; PHI, 3 days) resulted in concentrations of residues of 0.09, 0.19, 0.20, 0.25 and 0.34 mg/kg. Four trials in greenhouses performed in The Netherlands according to Belgian GAP (two applications of 0.18 kg ai/ha; PHI, 3 days) resulted in concentrations of residues in tomatoes of 0.10, 0.11 (2) and 0.16 mg/kg. The concentrations in tomatoes from field trials in the USA that complied with GAP (four applications of 0.28 kg ai/ha; PHI, 7 days) were 0.031, 0.058, 0.085, 0.089, 0.095, 0.11, 0.13, 0.17, 0.25, 0.31, 0.52 and 0.53 mg/kg. As the results for tomatoes grown in the field and in greenhouses are comparable, the data can be combined. The concentrations of residues in trials conducted according to GAP, in ranked order, were: 0.031, 0.058, 0.085, 0.089, 0.09, 0.095, 0.10, 0.11 (3), 0.13, 0.16, 0.17, 0.19, 0.20, 0.25 (2), 0.31, 0.34, 0.52 and 0.53 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, an STMR value of 0.13 mg/kg and a highest residue of 0.53 mg/kg for tomato.

Peppers

Trials on peppers conducted in the USA according to GAP (four applications at 0.29 kg ai/ha; PHI, 7 days) gave concentrations of residues of 0.048, 0.052, 0.064, 0.16, 0.17 and 0.64 in bell peppers and 0.040, 0.046 and 0.097 in other peppers.

The Meeting agreed to combine the data for the two types of peppers, resulting in concentrations, in ranked order, of: 0.040, 0.046, 0.048, 0.052, 0.064, 0.097, 0.16, 0.17 and 0.64 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg, an STMR value of 0.064 mg/kg and a highest residue of 0.64 mg/kg for tebufenozide in peppers.

Leafy vegetables

Eight newly submitted trials on lettuce, head were conducted in Europe according to pending GAP and were therefore not considered by the Meeting. Newly submitted trials on turnip greens in the USA were not performed according to GAP (application rate too high) and were also not taken into consideration.

The results of trials on head lettuce, leaf lettuce, spinach and mustard greens were reviewed by the 1996 JMPR, but, as there was no approved GAP at that time no MRLs were proposed. As GAP for leafy vegetables is now registered in the USA, these trials can be evaluated.

Trials on leafy vegetables conducted in the USA in compliance with current GAP (seven applications at 0.14 kg ai/ha; PHI, 7 days) resulted in concentrations of residues of: 0.092, 0.14, 0.29, 0.83, 0.9, 2.3, 2.7, 3.2 and 6.6 mg/kg in lettuce, head; 0.41, 0.69, 1.1, 1.7, 2.2, 2.5, 2.6 and 3.2 mg/kg in lettuce, leaf; 0.13 (2), 2.7, 3.3, 3.8, 3.9, 4.2, 7.1 and 8.1 mg/kg in spinach and 0.65, 0.93, 1.6, 1.9, 2.4, 2.6, 3.9, 4.4, 5.6 and 6.9 mg/kg in mustard greens.

As the use patterns of tebufenozide in leafy vegetables are similar and the concentrations of residues are in the same range, the Meeting concluded that the data for leafy vegetable crops could be combined. This resulted in concentrations of residues of tebufenozide, in ranked order, of: 0.092, 0.13, 0.14, 0.29, 0.41, 0.65, 0.69, 0.83, 0.9, 0.93, 1.1, 1.3, 1.6, 1.7, 1.9, 2.2, 2.3, 2.4, 2.5, 2.6 (2), 2.7 (2), 3.2 (2), 3.3, 3.8, 3.9 (2), 4.2, 4.4, 5.6, 6.6, 6.9, 7.1 and 8.1 mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg, an STMR value of 2.45 mg/kg and a highest residue of 8.1 mg/kg for the crop group leafy vegetables.

Turnip roots

Trials on turnip roots in the USA were not conducted according to GAP (0.14 kg ai/ha; PHI, 7 days) and were therefore not considered The Meeting could not estimate a maximum residue level for tebufenozide residues in turnip roots.

Celery

The maximum residue level in celery applies to the whole commodity after removal of adhering soil and clearly decomposed or withered leaves. The data on residues in trials on celery submitted previously, which were conducted in compliance with currently approved GAP in the USA (seven applications at 0.14 kg ai/ha; PHI, 7 days), pertained mainly to celery stalks *without* foliage. In two samples of celery stalks with foliage, the concentrations of residues were 0.41 and 1.3 mg/kg. The concentrations of residues in the stalk were lower. Insufficient data were available to estimate a maximum residue level in celery.

Sugar cane

The concentrations of tebufenozide residues in sugar cane were derived from 10 trials in the USA that complied with GAP (four applications, 0.28 mg/kg; PHI, 14 days). The values were 0.013, 0.032, 0.035, 0.054, 0.12 (2), 0.16, 0.28, 0.54 and 0.62 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, an STMR value of 0.12 mg/kg and a highest residue of 0.62 mg/kg for tebufenozide in sugar cane stems.

Tree nuts

Four trials on pecan previously submitted to the JMPR, which were conducted within currently approved GAP for pecans (0.28 kg ai/ha; PHI, 14 days), resulted in undetectable residues (< 0.01 mg/kg). Further trials on pecans were conducted in the USA in 1997. Although the total amount of tebufenozide applied was equal to the maximum allowed (2.1 kg ai/ha per season), the application rate per treatment was twice as high and the number of applications twice as low as the critical GAP. Residues were undetectable (< 0.01 mg/kg). Since these results confirm those obtained in 1993, the Meeting decided to include them in the evaluation. The concentration of residues in the 12 trials on pecans was < 0.01 mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg as a practical limit of quantification for tebufenozide in pecans. In addition, the Meeting estimated an STMR value of 0.01 mg/kg and a highest residue of 0.01 mg/kg.

Ten trials were conducted on almonds in the USA in 1995–98 in accordance with GAP for tree nuts excluding pecans (0.53 kg ai/ha; PHI, 14 days). The concentrations of residues were < 0.01 (2), 0.010, 0.016, 0.017, 0.024, 0.029, 0.034, 0.042 and 0.045 mg/kg in almond nut kernel.

The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR value of 0.0205 mg/kg and a highest residue of 0.045 mg/kg for tebufenozide in almond nut kernel.

Information on residues in macadamia nuts was generated in Australia, where the GAP for macadamia nuts (five applications of 0.009 kg ai/hl or concentrate spraying; PHI, 28 days) is still pending. The data were therefore not considered by the Meeting.

Rape seed

One trial on rape seed was performed in Canada and six in the USA. The approved GAP in the USA is four applications of 0.28 kg ai/ha and a PHI of at least 14 days. In one trial in the USA conducted according to GAP, the concentration of tebufenozide residue was 1.2 mg/kg. Each of the other trials involved either a deviation from the PHI or a special circumstance such as thawing of samples during transport. The residue values that were probably underestimates were 0.31, 0.47 and 0.52 mg/kg; and the values that were probably overestimates were 0.95, 1.1 and 1.6 mg/kg. However, since the values are more or less within the same range, the Meeting agreed to use all of them. In ranked order, the concentrations of tebufenozide residues in rape seed were 0.31, 0.47, 0.52, 0.95, 1.1, 1.2 and 1.6 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg, an STMR value of 0.95 mg/kg and a highest residue of 1.6 mg/kg for rape seed.

Mint

Five trials on mint were conducted in the USA according to GAP (0.28 kg ai/ha; PHI, 14 days). The concentrations of residues were much lower in mint foliage from one site (2.9 and 2.6 mg/kg) than in

that from the other sites, perhaps due to a frost that killed the mint tops on the day of the first application at the former site. The values from that trial were therefore not used to estimate the maximum residue level. The concentrations of residues in mint foliage in the remaining four trials were 7.5, 8.3, 8.4 and 8.6 mg/kg.

The Meeting estimated a maximum residue level of 20 mg/kg, an STMR value of 8.35 mg/kg and a highest residue of 8.6 mg/kg for tebufenozide in mint.

Almond hulls

Ten residue trials were conducted on almonds in the USA in 1995–98 in accordance with approved GAP for tree nuts excluding pecans (0.53 kg ai/ha; PHI, 14 days). The concentrations of residues measured in hulls (used as feed for livestock) in nine of these trials were 8.4, 9.5, 10, 11, 15, 16, 17 (2) and 21 mg/kg.

The Meeting estimated an maximum residue level of 30 mg/kg, an STMR value of 15 mg/kg and a highest residue of 21 mg/kg for tebufenozide in almond hulls.

Fate of residues during processing

The 1996 JMPR requested information on tebufenozide residues in raisins, raisin culls and rice hulls. The present Meeting received a report on two supervised trials with grapes in which raisins were generated. Furthermore, processing studies on citrus fruit, peaches, tomatoes, sugar cane and rape seed were supplied.

Washing, the first step in processing citrus fruit, removed most of the residue in oranges (86%) and grapefruit (74%). The calculated processing factors for industrial processing of citrus fruits were (mean of two trials, one in orange and one in grapefruit) 26.1 for oil, 0.80 for dried pulp and <0.016 for juice.

On the basis of the STMR value for citrus whole fruit of 0.48 mg/kg, the Meeting estimated STMR-P values of 12.5 mg/kg for citrus oil, 0.38 mg/kg for dried pulp and 0.0077 mg/kg for citrus juice.

During canning of peaches, all the residues of tebufenozide originally present were depleted, and no measurable residues (> 0.01 mg/kg) were found in either the fruit or the syrup of canned peaches. On the basis of the processing factor for canned peaches of < 0.06 (mean of six trials) and the STMR value for peaches of 0.11 mg/kg, the Meeting estimated an STMR-P value of 0.0066 mg/kg for canned peaches.

Processing of grapes yields wine, wet pomace, juice and raisins. Two trials with dried grapes resulted in a processing factor of 0.74 for raisins. Eight trials on grapes that were processed into juice resulted in a processing factor of 0.13 for juice. The 1996 JMPR determined processing factors of 2.7 (mean of 1.6, 2.8 and 3.7) for wet pomace and 0.36 for mature wine (0.07–0.69; n = 14). Additional data from 26 studies of wine processing conducted in Australia showed that the residues in pomace were concentrated by factors of 1.6–8.7 with an average of 4.1 (n = 18); the concentrations of residues in wine resulted in processing factors of 0.11–0.43 with an average of 0.25 (n = 23). Combining these processing factors with those of the 1996 JMPR resulted in processing factors of 3.9 for wet pomace (n = 21) and 0.29 for wine (n = 37).

On the basis of the highest residue in grapes of 1.5 mg/kg, the Meeting estimated a maximum residue level of 2.0 mg/kg for tebufenozide in raisins and a highest residue of 1.11 mg/kg. On the basis of the STMR value for grapes of 0.745 mg/kg, the Meeting estimated an STMR-P value for

tebufenozide of 0.551 mg/kg in raisins, 0.097 mg/kg in grape juice, 2.9 mg/kg in wet pomace to replace the STMR-P value of 0.36 mg/kg, and 0.216 mg/kg in wine to replace the STMR-P value of 0.03 mg/kg.

Tomatoes were processed differently in the four trials conducted in Europe and the one trial in the USA, but, to the extent that the processes yielded the same products, the data were comparable. About two-thirds of the residue in tomatoes was removed by washing in all five trials. The calculated processing factors were 0.31 for purée (n = 1), 0.73 for paste (n = 5), 0.18 for sterilized juice (n = 4) and 0.28 for preserved fruit (n = 4).

On the basis of the STMR value for tomatoes of 0.13 mg/kg, the Meeting estimated STMR-P values of 0.04 mg/kg for purée, 0.095 mg/kg for paste, 0.023 mg/kg for tomato juice and 0.036 mg/kg for preserved tomatoes.

During isolation of refined sugar from sugar cane, all the residues of tebufenozide originally present were depleted; no residues were present at a concentration > 0.01 mg/kg in the resulting refined sugar in four separate studies. The mean processing factor for refined sugar in three trials was < 0.025. Residues of tebufenozide concentrate in molasses; the processing factor for molasses was 5.9 (n = 3).

On the basis of the STMR value for sugar cane stems of 0.12 mg/kg, the Meeting estimated an STMR-P value of 0.003 mg/kg for refined sugar and 0.71 mg/kg for molasses.

Rape seed was processed in two trials into meal, soapstock and refined oil, resulting in processing factors of 0.15 for meal, 1.1 for soapstock and 2.3 for refined oil. On the basis of the STMR value for rape seed of 0.95 mg/kg, the Meeting estimated an STMR-P value of 0.14 mg/kg for meal, 1.0 mg/kg for soapstock and 2.2 mg/kg for refined rape seed oil.

Two studies on the processing of mint oil from mint resulted in a mean processing factor of 0.03 for mint oil. On the basis of the STMR value for mint foliage of 8.35 mg/kg, the Meeting estimated an STMR-P value of 0.25 mg/kg for mint oil.

Residues in animal commodities

Dietary burden of farm animals

The Meeting estimated the dietary burden of tebufenozide residues for farm animals from the diets listed in Appendix IX of the *FAO Manual*. Calculation from the HR values provides the concentrations in feed suitable for estimating MRLs for animal commodities, while calculation from the STMR values for feed is suitable for estimating STMR values for animal commodities. In the case of processed commodities, the STMR-P value is used for both intake calculations.

Estimated maximum intake

Commodity	Group	Residue (mg/kg)	Basis	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Almond hulls	AM	30	MRL	90	33.3	10	10	–	3.3	3.3	–

Apple wet pomace	AB	0.4	STMR-P	40	1.0	40	20	–	0.4	0.2	–
Citrus dry pulp	AB	0.38	STMR-P	91	0.42	20	20	–	0.08	0.08	–
Rape seed meal	SO	0.14	STMR-P	88	0.16	10	15	15	0.02	0.02	0.02
Rice grain	GC	0.1	MRL	88	0.114	–	15	60	–	0.02	0.07
Rice straw[a]	AS	7.7	HR	90	8.6	10	10	–	0.86	0.86	–
Sugar cane molasses	DM	0.71	STMR-P	75	0.9	10	10	–	0.09	0.09	–
					Total	100	100	75	4.8	4.6	0.09

[a] 2.9, 3.9, 6.2 and 7.7 mg/kg (1996 JMPR); STMR = 5.05 mg/kg

Estimated mean intake

Commodity	Group	Residue (mg/kg)	Basis	Dry matter (%)	Residue, dry weight (mg/kg)	Choose diets (%)			Residue contribution (mg/kg)		
						Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Almond hulls	AM	15.5	STMR	90	17.2	10	10	–	1.7	1.7	
Apple wet pomace	AB	0.4	STMR-P value	40	1.0	40	20	–	0.4	0.2	
Citrus dry pulp	AB	0.38	STMR-P value	91	0.42	20	20	–	0.08	0.08	
Rape seed meal	SO	0.14	STMR-P value	88	0.16	10	15	15	0.02	0.02	0.02
Rice grain	GC	0.025	STMR	88	0.028	–	15	60	–	0.004	0.02
Rice straw[a]	AS	5.05	STMR	90	5.6	10	10	–	0.56	0.56	
Sugar cane molasses	DM	0.71	STMR-P value	75	0.95	10	10	–	0.10	0.10	
					Total	100	100	75	2.9	2.7	0.04

[a] 2.9, 3.9, 6.2, 7.7 mg/kg (1996 JMPR); STMR = 5.05 mg/kg

Feeding studies

The 1996 JMPR requested the results of a study in which cows were fed diets containing tebufenozide, which the Meeting was informed was in progress. The present Meeting received those results.

Four cows in each group were given a capsule containing tebufenozide at 0, 6, 18 or 60 ppm ai for 28 days. One cow from each group was observed for 3 days while on a normal diet after the end of the dosing period. Whole milk, fat, meat, kidney and liver samples were analysed. The analytes of interest included parent tebufenozide in all matrices, RH-9886 in muscle and kidney, RH-0282 in milk, muscle and kidney, fatty acid conjugates of RH-9886 in milk and fat and RH-2703 in liver.

In cows at the two lower concentrations, the values for residues were below the LOQ (0.01 mg/kg in milk and 0.02 mg/kg in other matrices) in milk, muscle and kidney, except for a residue at the LOQ in muscle of one cow at 18 ppm. The concentration of residue in milk reached a plateau within about 3 days. The concentration in cream was not reported. In milk, the highest average group concentration of residue was at the LOD of 0.003 mg/kg in cows at 6 ppm, 0.009 mg/kg at 18 ppm and 0.028 mg/kg at 60 ppm. The highest individual concentrations of residues at 6, 18 and 60 ppm were 0.029 mg/kg, 0.11 mg/kg and 0.38 mg/kg in fat, < 0.006 mg/kg, 0.02 mg/kg and 0.06 mg/kg in muscle, < 0.006 mg/kg, 0.009 mg/kg and 0.04 mg/kg in kidney and 0.014 mg/kg, 0.04 mg/kg and 0.10 mg/kg in liver.

No detectable residues of analytes were found in cows observed on a normal diet for 3 days after treatment, except in fat in which approximately 30% of the initial residue was still present.

The Meeting considered that a feeding study with poultry was not necessary, as the concentrations of residues in poultry feed do not exceed 0.1 mg/kg and residues are therefore not expected in poultry products.

Maximum residue levels

As the maximum dietary burden of beef and dairy cattle was 4.8 mg/kg, the concentrations of residues in tissues and milk can be taken directly from results of the feeding study with 6 ppm, without interpolation. The maximum concentrations expected in tissues at this level are: 0.029 mg/kg in fat, < 0.006 mg/kg in muscle and kidney, 0.014 mg/kg in liver and 0.003 mg/kg in milk.

The Meeting estimated maximum residue levels of 0.05 mg/kg for cattle meat (fat), 0.02* mg/kg for cattle kidney, 0.02* mg/kg for cattle liver and 0.01* mg/kg for milk.

The STMR dietary burden of beef and dairy cattle was 2.9 mg/kg, which is about one-half the lowest concentration used in the feeding studies. The Meeting estimated STMR and highest residue values of 0.006 mg/kg for cattle meat and kidney and 0.02 mg/kg for liver, and an STMR value of 0.003 mg/kg for cattle milk.

A study of metabolism in poultry treated orally, evaluated by the 1996 JMPR, showed that, when laying hens were treated at a concentration equivalent to 30 ppm in the feed for 7 days, the concentrations of parent compound were 0.005 mg/kg in eggs, 0.18 mg/kg in fat and undetectable in liver and muscle. The maximum dietary burden of poultry was calculated to be 0.09 mg/kg, which is 300 times lower than that used in the study of metabolism. Therefore, the Meeting agreed to recommend MRLs for poultry meat and eggs at the LOQ. The Meeting acknowledged that the analytical method for animal commodities had not been validated for eggs but accepted that the LOQ for cattle tissues could apply to poultry tissues. The Meeting estimated maximum residue levels for poultry meat (fat) and eggs of 0.02* mg/kg, an STMR value and a highest residue for poultry meat (fat) of 0.02 mg/kg and an STMR value and a highest residue for eggs of 0 mg/kg.

The Meeting was informed that a report on validation of the analytical method for residues of tebufenozide in chicken liver, muscle, fat and eggs was available and would be submitted to a future Meeting.

Further work or information

Desirable

1. Information on the level of residues in milk cream
2. A report on validation of the analytical method for animal commodities with respect to poultry meat and eggs that the Meeting was informed was available.

Dietary risk assessment

Long-term intake

STMR or STMR-P values for tebufenozide were estimated by the current Meeting for 43 plant commodities and animal products. STMR values for four additional plant commodities were estimated by the 1996, 1997 and 1999 Meetings. When data on consumption were available, these values were used in the estimates of dietary intake. The results are shown in Annex 3.

The International Estimated Daily Intakes for the five GEMS/Food regional diets, based on the estimated STMRs, were in the range of 1-20% of the ADI. The Meeting concluded that the chronic intake of residues of tebufenozide from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The international estimated short-term intake (IESTI) for tebufenozide was calculated for those plant commodities and animal products for which maximum residue levels and STMRs were estimated and for which consumption data were available. The results are shown in Annex 4. The IESTI represented 0-440% of the acute RfD for the general population and 0-1220% of the acute RfD for children. That representing 440% (general population) and 1220% (children) results from the consumption of leafy vegetables (spinach). The short-term intake of cabbage also exceeded the acute RfD in both groups, with 230% (general population) and 410% (children). For children, the estimated short-term intake of pomefruit (apple) and grapes exceeded the acute RfD with 210% and 190% respectively.

RECOMMENDATIONS

CCN	Commodity	MRL, mg/kg		STMR, STMR-P, mg/kg	HR, HR-P, mg/kg
		New	Previous		
TN 0660	Almonds	0.05	–	0.0205	0.045
AM 0660	Almond hulls	30		15	
FI 0326	Avocado	1	–	0.21	0.53
VB 0400	Broccoli	0.5	–	0.11	0.34
FB 0020	Blueberries	3	–	0.685	1.7
VB 0041	Cabbage, Head[1]	5	–	0.34	4.6
MO 1280	Cattle, kidney	0.02*		0.006	0.006
MO 1281	Cattle, liver	0.02*	–	0.02	0.02
MM 0812	Cattle meat (F)	0.05		0.006	0.006
ML 0812	Cattle milk	0.01*	–	0.003	
FC 0001	Citrus fruit Citrus oil Citrus dried pulp	2	–	0.079 12.5 0.38	0.18
JF 0001	Citrus juice			0.0077	
FB 0265	Cranberries	0.5	–	0.042	0.28
DF 0269	Dried grapes (currants, raisins and sultanas)	2	–	0.551	1.11
PE 0112	Eggs	0.02*		0	0
FB 0269	Grapes[1] Grape wet pomace Wine	2	1	0.745 2.9 0.216	1.5
JF 0269	Grape juice			0.097	
VL 0053	Leafy vegetables[1]	10	–	2.45	8.1
HH 0738	Mint Mint oil	20	–	8.35 0.25	8.6
FS 0245	Nectarines	0.5	–	0.11	0.23
FS 0247	Peaches Peaches, canned	0.5	–	0.11 0.0066	0.23

CCN	Commodity	MRL, mg/kg		STMR, STMR-P, mg/kg	HR, HR-P, mg/kg
		New	Previous		
TN 0672	Pecans	0.01*	–	0.01	0.01
VO 0051	Peppers	1		0.064	0.64
PM 0110	Poultry meat	0.02*	–	0.02	0.02
FB 0272	Raspberries	2		0.56	0.86
SO 0495	Rape seed Rape seed meal Rape seed soapstock	2	–	0.95 0.14 1.0	1.6
OC 0495	Rape seed oil			2.2	
GS 0659	Sugarcane stems Refined sugar Molasses	1	–	0.12 0.003 0.708	0.62
VO 0448	Tomato Tomato purée Tomato paste Tomatoes (preserved)	1	–	0.13 0.04 0.095 0.036	0.53
JF 0448	Tomato juice			0.023	

[1] The information provided to the JMPR precludes an estimate that the dietary intake would be below the acute RfD.Acute RfD: 0.05 mg/kg bw

Definition of the residue: for compliance with MRL and for estimation of dietary intake for plant and animal products*:* tebufenozide

The residue is fat-soluble

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