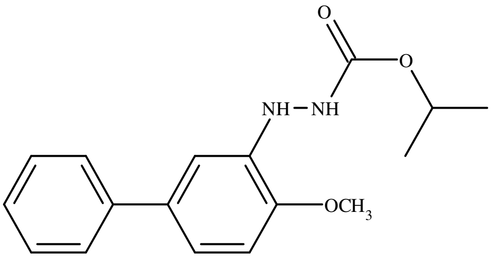
BIFENAZATE (219)

First draft was prepared by Denis Hamilton, Biosecurity, Queensland Department of Primary Industries and Fisheries, Australia

IDENTITY

ISO common name	Bifenazate
Synonyms:	D2341
IUPAC name	Isopropyl 2-(4-methoxybiphenyl-3-yl)hydrazinoformate
Chemical Abstracts name	1-methylethyl 2-(4-methoxy[1,1′-biphenyl]-3-yl) hydrazinecarboxylate
CAS Number	149877-41-8
CIPAC Number	736
Molecular formula	$C_{17}H_{20}N_2O_3$
Molecular mass	300.4
Structural formula	



PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient:

Property	Result	Ref
Colour (purity 99.7%)	White	Friedlander, 1998, GRL FR-11291
Odour (purity 99.7%)	Slight odour characteristic of aromatic compounds	Friedland, 1998, GRL FR-11293-01
Appearance (purity 99.7%)	Crystalline solid consisting of small needles of less than 1 mm in length	Friedlander, 1998, GRL FR-11292-01
Melting point (purity 98.1%)	121.5–123 °C	Dunn, 2003, GRL-12057
Relative density (purity 99.7%)	1.19 g/cm^3	Stevenson, 1998, GRL-FR-11296
Vapour pressure (purity 98.1%, light brown powder)	3.8×10^{-7} Pa at 25 °C[1]	Tremain, 2003, 666/079
Solubility in water (purity 98.1%, pale brown powder)	1.52 mg/L water at 20 °C 1.66 mg/L aqueous pH 5 buffer at 20 °C	Riggs, 2004, GRL-11907

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[1] Vapour pressure balance method with measurements over a temperature range of 100-113 °C and extrapolation to 25 °C.

Property	Result	Ref
Solubility in organic solvents at 20 °C (purity 99.7%)	ethyl acetate 102 g/L acetonitrile 96 g/L methanol 45 g/L toluene 24.7 g/L hexane 0.23 g/L n-octanol 8.9 g/L	Riggs, 1998, GRL-FR-11295
Octanol/water partition coefficient (purity 98.1%, pale brown powder)	Log P_{OW} = 3.5 at 38 °C	Riggs, 2003, GRL-12061
Hydrolysis rate (purity ≥ 98%)	Conditions: 25 °C, <1% acetonitrile in sterile buffers in the dark, approx 1 mg/L. pH DT_{50} 4 9.1 days 5 5.4 days 7 20 hours 9 1.6 hours	Shah, 1997, 6337-95-0006-EF-001
Dissociation constant in water	No evidence of dissociation in solutions where bifenazate is stable and could be tested, i.e. between pH 6.5 and 2.	Riggs, 2004, GRL-12062

Hydrolysis of bifenazate (Shah, 1997, 6337-95-0006-EF-001)

Bifenazate, at approximately 1 mg/L, was hydrolysed in sterile aqueous buffers in the dark. The buffers contained less than 1% acetonitrile as a co-solvent. The first step was an oxidation reaction producing bifenazate-diazene, which then hydrolysed to produce methoxy- and hydroxy- biphenyls. Bifenazate-diazene reached a maximum 21.3% (pH 4, day 14), 27.3% (pH 5, day 10), 58.5% (pH 7, 27 hours) and 23.5% (pH 9, 1.8 hours) of the starting material.

The data allowed estimates for hydrolysis DT_{50}s of bifenazate-diazene: 58, 50, 18 and 0.28 hours at pH 4, 5, 7 and 9 respectively.

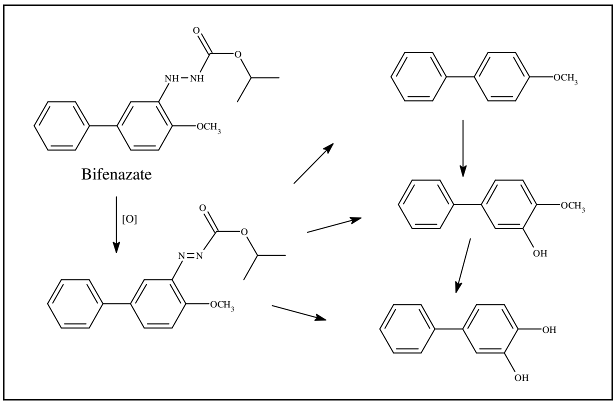
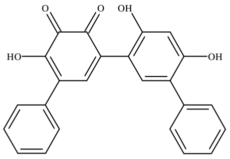


Figure 1. Proposed hydrolysis pathway for bifenazate (Shah, 1997, 6337-95-0006-EF-001).

Findak, DiFrancesco and Shah (1999, 6337-95-0006-EF-001-001) subsequently identified a product of hydrolysis at pH 5 and 7. The compound accounted for a maximum of approximately 14% and 24% of the ^{14}C during hydrolysis at pH 5 (day 30) and pH 7 (day 5).



Photolysis of bifenazate (Shah, 1997, 6337-95-0006-EF-001)

Bifenazate (^{14}C labelled), at approximately 1 mg/L, was photolysed in a sterile aqueous pH 5 acetate buffer in simulated sunlight, with 12 hours light and 12 hours dark for 6 days.

The photolysis DT_{50} for parent bifenazate was 0.72 days and for bifenazate + bifenazate-diazene it was 3.3 days. The dark solution DT_{50} for parent bifenazate was 1.8 days and for bifenazate

+ bifenazate-diazene the DT_{50} was 4.4 days. Three other products were identified in the photolysis solution: 3,4-dihydroxybiphenyl and 3-hydroxy-4-methoxybiphenyl accounted for 16% and 32% of the initial ^{14}C at the end of the 6 days, while 3-methoxybiphenyl reached a maximum of 13% of the ^{14}C after 54 hours of photolysis. Polar products and carbon dioxide were also produced.

Photolysis of bifenazate (Lewis, 2001, 217/27)

Bifenazate, at approximately 1 mg/L, was photolysed in a sterile aqueous pH 5 buffer in simulated sunlight filtered to remove wavelengths below 290 nm, with 12 hours light and 12 hours dark for 30 days. The intensity of the light was adjusted so as to approximate the same number of summer day's sunlight in UK or Florida. The DT_{50} for parent bifenazate was 0.9 days (10.8 hours). The initial half-life calculated from the 0–2 days data for bifenazate was 0.8 days (9 hours) and for combined bifenazate + bifenazate-diazene was 1.5 days (18 hours). The identified products of photolysis are shown in Figure 2. Polar products and carbon dioxide were also produced.

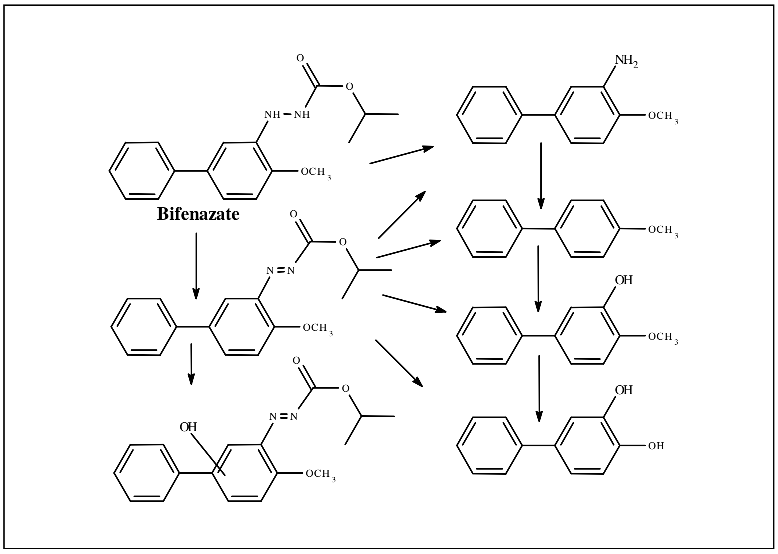


Figure 2. Proposed photolysis pathway for bifenazate (Lewis, 2001, 217/27)

Technical material:

Property	Result	Ref
Solubility in organic solvents at 20 °C	ethyl acetate 11.3 g/100 mL acetonitrile 11.1 g/100 mL methanol 5.07 g/100 mL toluene 2.62 g/100 mL hexane 0.0232 g/100 mL n-octanol 0.954 g/100 mL	Riggs, 1997, GRL-FR-10806

FORMULATIONS

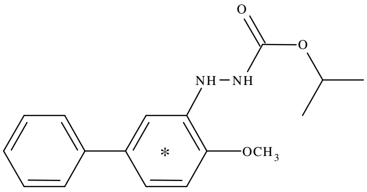
Bifenazate is available as commercial WP, SC and WG formulations.

Code	Description	Concentration	Examples
WP	wettable powder	500 g/kg	Acramite 50WP, Acramite 50WS (WP in water soluble bags)
SC	suspension concentrate	480, 240 or 200 g/L	Acramite 480 SC, Acramite 4L, Floramite 240 SC, Mitokohne 20 SC

Code	Description	Concentration	Examples
WG	water dispersible granules	750 g/kg	Acramite 75 WG

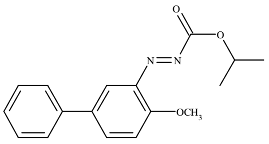
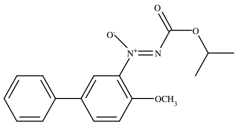
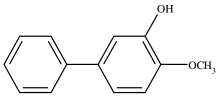
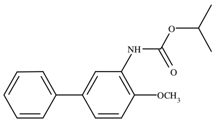
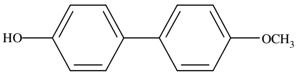
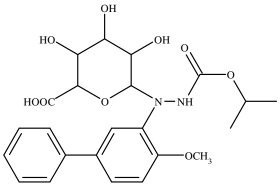
METABOLISM AND ENVIRONMENTAL FATE

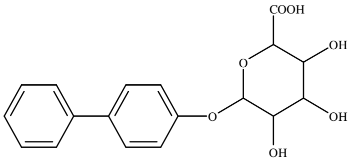
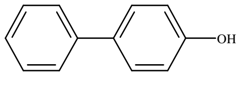
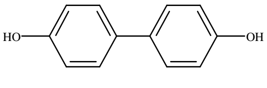
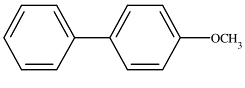
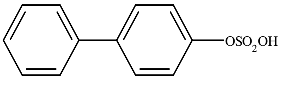
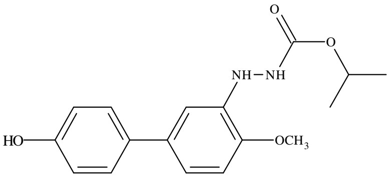
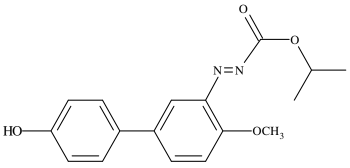
Animal and plant metabolism and environmental fate studies used bifenazate ^{14}C labelled in the substituted phenyl ring.



bifenazate ^{14}C labelled in the substituted phenyl ring

Structures, names and codes for metabolites are summarised below.

Bifenazate-diazene diazenecarboxylic acid, 2-(4-methoxy-[1,1'-biphenyl]-, 1-methylethyl ester CAS number: 149878-40-0 Code: D3598	
Bifenazate-diazene oxide diazenecarboxylic acid, 2-(4-methoxy-[1,1-biphenyl]-3-yl)-, 1-methylethyl ester 2-oxide Code: D4642	
3-hydroxy-4-methoxybiphenyl CAS number: 37055-80-4 Code: D9963	
Bifenazate-carbamate carbamic acid, (4-methoxy-[1,1'-biphenyl]-3-yl)-, 1-methylethyl ester Code: D6887	
4-hydroxy-4'-methoxybiphenyl CAS number: 16881-71-3 Code: D9477	
Bifenazate glucuronide	

4-hydroxybiphenyl glucuronide	
4-hydroxybiphenyl CAS number: 92-69-3 Code: A1530	
4,4'-dihydroxybiphenyl CAS number: 92-88-6 Code: D9569	
4-methoxybiphenyl CAS number: 613-37-6 Code: D1989	
4-hydroxybiphenyl sulphate	
4-hydroxy bifenazate	
4-hydroxy bifenazate-diazene	

Animal metabolism

The Meeting received animal metabolism studies with bifenazate in rats, lactating goats and laying hens.

Bifenazate is readily converted to bifenazate-diazene by mild oxidation. Primary metabolites are readily produced by removal of the side chain and by hydroxylation of the biphenyl rings. Glucuronide and sulphate conjugates are also produced.

Rats

McClanahan (1998, 95236) studied the distribution, metabolism and excretion of bifenazate following a single oral dose (10 or 1000 mg/kg bw) of [^{14}C]bifenazate in rats. Parent bifenazate and the following metabolites were identified in excreta: bifenazate glucuronide, bifenazate-diazene, 4-hydroxy bifenazate, 4-hydroxy bifenazate-diazene, 4-hydroxybiphenyl and its sulphate conjugate, 4,4'-dihydroxybiphenyl and its glucuronate and sulphate conjugates, 4-methoxybiphenyl and 4-hydroxy-4'-methoxybiphenyl and its conjugates.

Lactating goats

A lactating goat weighing 31 kg (day 1 and day 4) was dosed orally once daily for 4 consecutive days by gelatin capsule with 21 mg/animal/day of [^{14}C]bifenazate, equivalent to 10 ppm in the feed (McClanahan and Bayus, 1999, 96-0064) for a 2.10 kg/day feed consumption. Milk was collected twice daily; a day's sample began in the afternoon after dosing and ended with the morning milking preceding the next dose. Milk production averaged 1.78 kg/day during the dosing period. The animal was slaughtered approximately 8 hours after the final dose for tissue collection. Recovery of administered ^{14}C was 68%. Samples were stored in a freezer below -10 °C for the following intervals before the initial extraction for metabolite identification (milk 1 day; kidney 27 days; muscle, fat and liver 32 days). See Table 14 and Table 15 for freezer storage stability of tissues and milk of lactating goats.

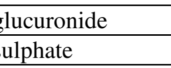
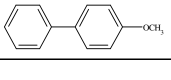
The majority of the administered ^{14}C was present in the excreta and stanchion wash (19.5% in urine, 46.5% in faeces). Milk accounted for 0.22% and tissues and blood 2.0% of the administered ^{14}C. The distribution of the radiolabel and identified metabolites in goat milk and tissues are summarised in Table 1. Residues of ^{14}C were higher in liver (1.77 mg/kg) than in other tissues.

In day 4 goat milk, 4-hydroxybiphenyl sulphate was the major identified residue at 0.019 mg/kg (41% of TRR). Bifenazate + bifenazate-diazene at 0.004 mg/kg comprised 9% of the TRR in milk. In muscle, the highest identified residue was 4-hydroxybiphenyl at 0.002 mg/kg (13% of TRR). Bifenazate + bifenazate-diazene comprised approximately 3–9% of the TRR in muscle. In fat, bifenazate was the major component, accounting for 53-58% of the TRR. Bifenazate + bifenazate-diazene comprised approximately 58–67% of the TRR in fat (0.070-0.072 mg/kg). The residue levels and patterns in omental and perirenal fat were quite similar.

In goat liver, only 10% of the TRR was extractable. The major identified metabolite was 4-hydroxybiphenyl glucuronide at 0.017 mg/kg (0.93% of TRR). Bifenazate + bifenazate-diazene comprised 0.98% of the TRR. The extracted liver was subjected to hydrolysis and extraction procedures to investigate the nature of the 87% non-extractable residues. The evidence suggested that some of the unextractable TRR was covalently bound to liver protein.

In goat kidney, 4-hydroxybiphenyl glucuronide and sulphate were the major identified components at 0.023 mg/kg, equivalent to approximately 14% of the TRR. Bifenazate + bifenazate-diazene comprised less than approx 2% of the TRR. Approximately 47% of the TRR was unextractable. The kidney was subjected to the same treatment as previously described for the liver, again suggesting that some of the unextractable ^{14}C was bound to protein.

Table 1. Distribution of ^{14}C residue and identified metabolites in tissues and milk of a lactating goat dosed orally for 4 days with 21 mg/animal/day of [^{14}C] bifenazate, equivalent to 10 ppm in the feed (McClanahan and Bayus, 1999, 96-0064).

Component	Concentration, mg/kg, expressed as parent							
	Loin muscle	Leg muscle	Omental fat	Peri-renal fat	Liver	Kidney	Milk, day 3	Milk, day 4
Total ^{14}C residue (TRR)	0.013	0.014	0.10	0.13	1.77	0.26	0.032	0.047
Extracted residue	0.008	0.008	0.090	0.096	0.175	0.13	0.031	0.044
Unextractable	0.005	0.005	0.021	0.013	1.54	0.12	0.002	0.002
Bifenazate	0.001	nd	0.061	0.066	0.011	0.003		0.0003
Bifenazate glucuronide					0.005			
Conjugates, includes 4-hydroxybiphenyl sulphate and glucuronide and bifenazate glucuronide	0.002	0.003	0.004	0.005				
4-hydroxybiphenyl 	0.002	0.002	0.006	0.007	0.012	0.009		0.0008
4-hydroxybiphenyl glucuronide					0.017	0.004		
4-hydroxybiphenyl sulphate					0.005	0.029	0.012	0.019
4-methoxybiphenyl 			0.003	0.004	0.006	0.005 1/		0.0017
Bifenazate-diazene			0.009	0.006	0.006			0.0039

nd: not detected. Detection limit approx 0.0001 mg/kg.

1/ In kidney, combined 4-methoxybiphenyl + bifenazate-diazene residue = 0.005 mg/kg.

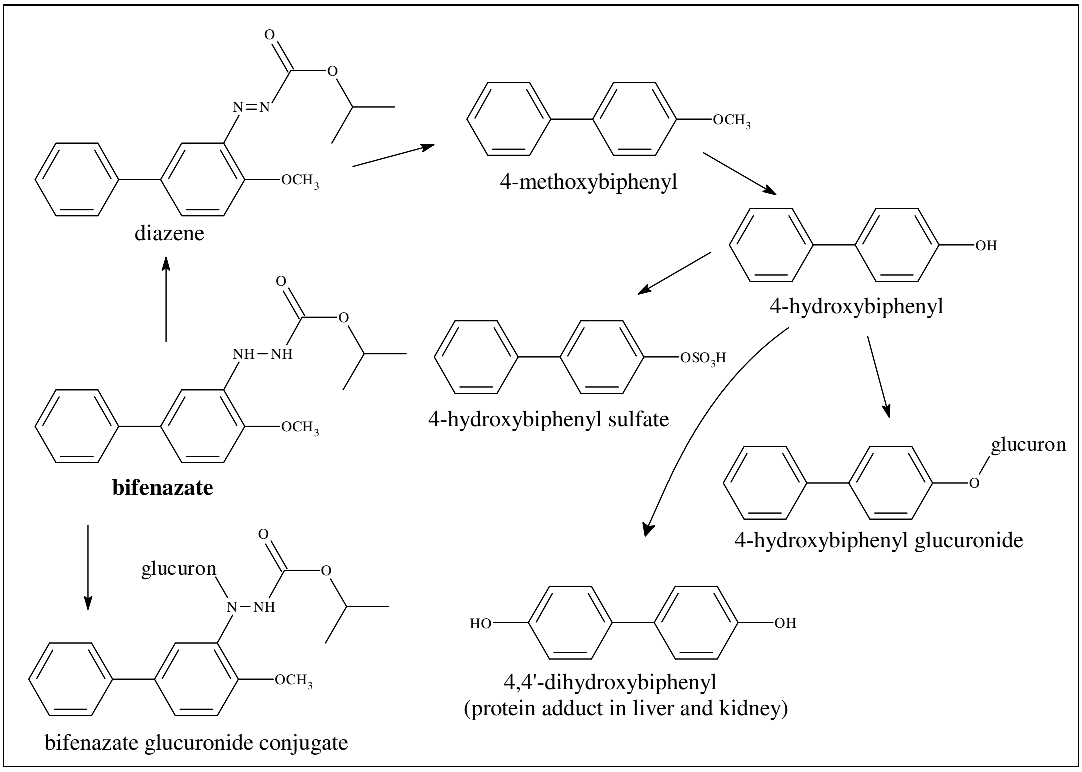


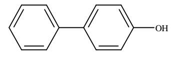
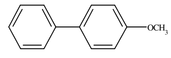
Figure 3. Proposed bifenazate metabolic pathway in lactating goats (McClanahan and Bayus, 1999, 96-0064).

Laying hens

A group of white leghorn laying hens (10 birds), approximately 25 weeks old, mean body weight 1.60 kg at study initiation and completion were dosed orally once daily via gelatin capsule for 4 consecutive days with 1.3 mg/bird/day of [^{14}C]bifenazate, equivalent to 10 ppm in the feed (McClanahan *et al.*, 1999, 96-0265) for a 127 g/day feed consumption. Eggs were collected twice daily. The birds were slaughtered approximately 8.5 hours after the final dose for tissue collection (breast and thigh muscle, skin and fat and liver). Recovery of administered ^{14}C was approximately 85%. Samples were stored in a freezer below -10 °C for 64 days before the initial extraction for metabolite identification. See Table 16 for freezer storage stability testing of residues in tissues and eggs of laying hens.

Most of the administered ^{14}C was present in excreta and cages (83.6%), with 1.38% in the tissues and 0.01% in the eggs. Residues were not detectable (< 0.005 mg/kg) in breast muscle and egg white. The highest ^{14}C residue was present in liver at 0.61 mg/kg, of which 0.19 mg/kg was extractable. The major identified residues in liver, skin + fat and egg yolk were hydroxybiphenyl, bifenazate-diazene and bifenazate, respectively. The distribution of residues is summarised in Table 2.

Table 2. Distribution of ^{14}C residue and identified metabolites in tissues and eggs of laying hens dosed orally for 4 days with 1.3 mg/bird/day of [^{14}C]bifenazate, equivalent to 10 ppm in the feed (McClanahan *et al.*, 1999, 96-0265).

Component		Concentration, mg/kg, expressed as parent					
		Skin + fat	Liver	Thigh muscle	Breast muscle	Egg white, day 4	Egg yolk, day 4
Total ^{14}C residue (TRR)		0.048	0.61	0.006	< 0.005	< 0.005	0.025
Extracted residue		0.025	0.19	0.002			0.012
Unextractable		0.015	0.37	0.003			0.007
Bifenazate		0.001	0.002	< 0.0005			0.005
Conjugates, mostly bifenazate glucuronide		0.001	0.017	< 0.0005			< 0.0005
4-hydroxybiphenyl		0.001	0.013	< 0.0005			0.001
4-methoxybiphenyl		0.005	0.001	< 0.0005			0.001
Bifenazate-diazene		0.008	0.002	< 0.0005			0.001

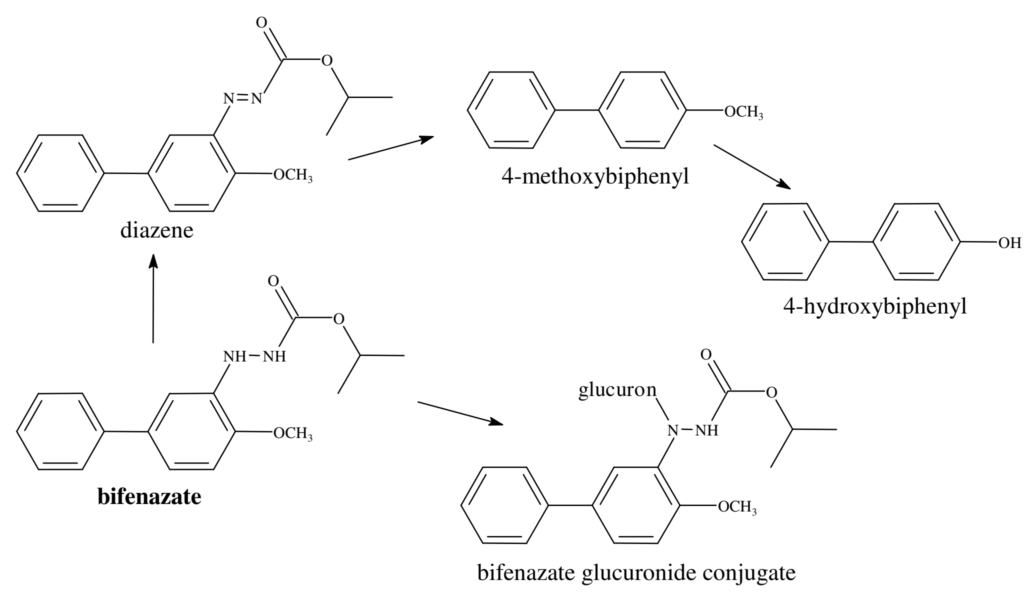


Figure 4. Proposed bifenazate metabolic pathway in laying hens (McClanahan *et al.*, 1999, 96-0265).

Plant metabolism

The Meeting received plant metabolism studies with bifenazate on oranges, apples, grapes, radish and cotton.

In plants, most of the resultant residue from the use of bifenazate was a surface residue. Parent bifenazate was the major component of the residue at shorter intervals and the major identified component at longer intervals after treatment. Bifenazate-diazene was usually also present, but at much lower levels than parent bifenazate. Very little of the residue translocated to the roots from treated radish foliage.

Oranges

In an orange metabolism study in California USA, Panthani and Hatzenbeler (1998, 6381-95-0028-EF-001) foliar sprayed Valencia oranges once with [^{14}C]bifenazate formulated as a WP (wettable powder) at 0.42 and 2.2 kg ai/ha and harvested mature fruit 43, 184, 274 and 442 days later for analysis. The mature fruits were maintained at refrigeration temperature (approx 5°C) for shipment to the laboratory and were processed immediately upon receipt from the field.

Surface residues in the oranges harvested 43 days after treatment, constituting 78% and 81% of the total residues for whole fruit, were removed with an acetonitrile rinse. Parent bifenazate was the major component of the surface residue (Table 3). Bifenazate-diazene was also identified in the surface rinse together with minor metabolites bifenazate-diazene oxide, 4-methoxybiphenyl and 3-hydroxy-4-methoxybiphenyl. The rinsed oranges were peeled and the peeled fruits were homogenized and centrifuged to produce pulp and juice. The TRR distribution was mostly into the peel (20% and 18% of the total residues for whole fruit) with very minor amounts into the pulp (0.9% and 0.9%) and juice (1.2% and 0.8%).

Total residue levels (TRR) in the oranges declined substantially for the fruit harvested at 184, 274 and 442 days after treatment (Table 3). The percentage of the residue on the fruit surface also declined (8% and 12% of the total residues for whole fruit at day 442). Bifenazate and bifenazate-diazene were identifiable components of the residue even at the longer intervals after treatment. At the longer intervals, polar materials constituted higher percentages of the residue. Most of the unextractable residue in peel was released by acid hydrolysis, but none of the individual components represented a residue exceeding 0.005 mg/kg.

In summary, for oranges most of the residues were surface residues, the majority of which was parent bifenazate. Very little of the residue appeared in orange pulp or juice.

Table 3. Residues in and on oranges treated with WP formulated [14C]bifenazate, at the equivalent of 0.42 and 2.2 kg ai/ha and harvested 43, 184, 274 and 442 days later (Panthani and Hatzenbeler, 1998, 6381-95-0028-EF-001).

Samples and metabolites	Concentration, mg/kg of ^{14}C expressed as parent	
	Treatment 0.42 kg ai/ha	Treatment 2.2 kg ai/ha
Whole fruit, 43 days, TRR	0.353	1.47
Unextractable TRR	0.024	0.082
Extractable TRR	0.330	1.38
bifenazate	0.266	1.16
bifenazate-diazene	0.026	0.088
bifenazate-diazene oxide	0.001	0.004
4-methoxybiphenyl	0.001	0.003
3-hydroxy-4-methoxybiphenyl	0.002	0.012
Surface wash, 43 days, TRR	0.275	1.18
bifenazate	0.259	1.12
bifenazate-diazene	0.016	0.060
bifenazate-diazene oxide	< 0.001	< 0.001
4-methoxybiphenyl	< 0.001	< 0.001
3-hydroxy-4-methoxybiphenyl	< 0.001	< 0.001
Peel, 43 days, TRR	0.070	0.259
Unextractable of peel, 43 days, TRR	0.022	0.075
Extractable of peel, 43 days, TRR	0.048	0.185
bifenazate	0.004	0.037
bifenazate-diazene	0.010	0.028
bifenazate-diazene oxide	0.001	0.004
4-methoxybiphenyl	0.001	0.003
3-hydroxy-4-methoxybiphenyl	0.002	0.012

Samples and metabolites	Concentration, mg/kg of ^{14}C expressed as parent	
	Treatment 0.42 kg ai/ha	Treatment 2.2 kg ai/ha
Pulp, 43 days, TRR	0.003	0.013
Unextractable of pulp, 43 days, TRR	0.001	0.004
Extractable of pulp, 43 days, TRR	0.003	0.009
bifenazate	0.001	0.001
bifenazate-diazene	< 0.001	< 0.001
bifenazate-diazene oxide	< 0.001	< 0.001
4-methoxybiphenyl	< 0.001	< 0.001
3-hydroxy-4-methoxybiphenyl	< 0.001	< 0.001
Juice, 43 days, TRR	0.005	0.012
bifenazate	0.003	0.001
bifenazate-diazene	< 0.001	< 0.001
bifenazate-diazene oxide	< 0.001	< 0.001
4-methoxybiphenyl	< 0.001	< 0.001
3-hydroxy-4-methoxybiphenyl	< 0.001	< 0.001
Whole fruit, 184 days, TRR	0.096	
bifenazate	0.035	
bifenazate-diazene	0.010	
Surface wash, 184 days, TRR	0.042	
Peel, 184 days, TRR	0.050	
Pulp, 184 days, TRR	0.002	
Juice, 184 days, TRR	0.003	
Whole fruit, 274 days, TRR	0.095	0.081
bifenazate	0.015	
bifenazate-diazene	0.006	
Surface wash, 274 days, TRR	0.021	0.020
Peel, 274 days, TRR	0.068	0.045
Pulp, 274 days, TRR	0.004	0.010
Juice, 274 days, TRR	0.003	0.005
Whole fruit, 442 days, TRR	0.013	0.032
bifenazate	0.001	
bifenazate-diazene	0.001	
Surface wash, 442 days, TRR	0.001	0.004
Peel, 442 days, TRR	0.009	0.020
Pulp, 442 days, TRR	0.002	0.005
Juice, 442 days, TRR	0.001	0.003

Apples

In an apple metabolism study in California USA, Panthani and Hatzenbeler (1998, 6850-96-0101-EF-001) foliar sprayed Granny Smith apples once with [^{14}C]bifenazate formulated as a WP (wettable powder) at 0.42 and 2.2 kg ai/ha and harvested the fruit at maturity 101 days later for analysis. The mature fruits were processed immediately after harvest.

Surface residues, constituting 55% and 66% of the total residues for whole apples, were removed with an acetonitrile rinse. Parent bifenazate was the major component of the surface residue (Table 4). Bifenazate-diazene was also identified in the surface rinse together with minor metabolites bifenazate-diazene oxide and 4-methoxybiphenyl. The rinsed apples were homogenized and centrifuged to produce pomace and juice. The TRR distribution was mostly into the pomace (35% and 26% of the total residues for whole apples) with lesser amounts into the juice (10% and 9%).

Very little of the residue was identified in pomace and juice components. The juice contained mainly polar fractions. None of the extractable components from the pomace or juice, apart from bifenazate and bifenazate-diazene, corresponded to available metabolite standards. Acid and base hydrolysis did not release any identifiable components.

In summary, for apples most of the residues were surface residues, the majority of which was parent bifenazate. Residues penetrating the fruit were mostly metabolized to polar compounds.

Table 4. Residues in and on apples treated with WP formulated [^{14}C]bifenazate, at the equivalent of 0.42 and 2.2 kg ai/ha and harvested 101 days later (Panthani and Hatzenbeler, 1998, 6850-96-0101-EF-001).

Samples and metabolites	Concentration, mg/kg of ^{14}C expressed as parent	
	Treatment 0.42 kg ai/ha	Treatment 2.2 kg ai/ha
Whole fruit TRR	0.088	0.37
Unextractable TRR	0.015	0.053
Extractable TRR	0.071	0.32
bifenazate	0.030	0.18
bifenazate-diazene	0.005	0.017
bifenazate-diazene oxide	0.001	0.003
4-methoxybiphenyl	< 0.001	0.001
bifenazate-carbamate 1/		0.001
Surface wash TRR	0.049	0.244
bifenazate	0.029	0.17
bifenazate-diazene	0.004	0.015
bifenazate-diazene oxide	0.001	0.003
4-methoxybiphenyl	< 0.001	0.001
bifenazate-carbamate		< 0.001
Pomace TRR	0.031	0.096
Unextractable of pomace TRR	0.015	0.053
Extractable of pomace TRR	0.014	0.038
bifenazate	0.001	0.001
bifenazate-diazene	0.001	0.001
bifenazate-diazene oxide	< 0.001	< 0.001
4-methoxybiphenyl	< 0.001	< 0.001
bifenazate-carbamate		< 0.001
Juice TRR	0.009	0.033
bifenazate	< 0.001	< 0.001
bifenazate-diazene	< 0.001	< 0.001
bifenazate-diazene oxide	< 0.001	< 0.001
4-methoxybiphenyl	< 0.001	< 0.001
bifenazate-carbamate		< 0.001

1/ Bifenazate-carbamate was a very minor component, detected in only the high-rate treatment extractable TRR from the whole fruit. The sponsor expressed the opinion that it may have been a small impurity in the labelled bifenazate.

Grapes

In a grape metabolism study in California USA, McManus and DeMatteo (2001, 2000-097) foliar sprayed Thompson seedless grapes with [^{14}C]bifenazate formulated as a WP (wettable powder) at 0.56 and 1.1 kg ai/ha and harvested the crop at maturity 30 days later for analysis. Foliage and fruit were also taken on day 0 after the single treatment.

Harvested samples were stored at temperatures below -10°C until processing within 30 days of harvest.

Grapes were thawed to room temperature and washed with methanol. The rinsed grapes were mixed with dry ice and homogenized to a fine powder. After the dry ice sublimed, the ground samples were centrifuged to produce juice and pomace. Pomace was rinsed with methanol, which was added to the juice. Almost the entire residue was in the surface rinsings (Table 5). HPLC analysis of the surface rinsings revealed only two peaks corresponding to bifenazate and bifenazate-diazene in the chromatogram and together accounting for 98% and 95% of TRR for the 0.56 and 1.1 kg ai/ha treatments respectively. Only trace amounts of ^{14}C were released from the pomace by acid, alkali and enzymic hydrolysis. HPLC analysis of grape juice revealed the presence of bifenazate and bifenazate-diazene and other trace metabolites.

The residues on grapes were mostly surface residues consisting essentially of bifenazate and bifenazate-diazene.

Table 5. Residues in and on grapes treated with WP formulated [^{14}C]bifenazate, at the equivalent of 0.56 and 1.1 kg ai/ha (McManus and DeMatteo, 2001, 2000-097).

Samples	Concentration, mg/kg of ^{14}C expressed as parent	
	Treatment 0.56 kg ai/ha	Treatment 1.1 kg ai/ha
Unwashed grapes TRR	3.5	12.0
Surface wash TRR	3.4	11.7
Aqueous juice TRR	0.075	0.11
Pomace TRR	0.10	0.24

Radish

In a plant metabolism study in USA, Charlton and Tecle (2002, 2001-147) sprayed the foliage of radish plants (variety French Breakfast) with [^{14}C]bifenazate formulated as a WS (water-dispersible powder) at 1.1 and 2.2 kg ai/ha and harvested the crop 7 days later for analysis. Harvested samples were stored in freezers or refrigerators and sample processing and analysis began within 32 days of harvest. Most of the ^{14}C remained on the foliage, with very little reaching the roots (Table 6). The nature of the residue in the roots was not investigated further because the levels were very low.

More than half of the residue associated with the foliage was removed with a surface wash of 50% acetonitrile in water. Parent bifenazate was the major component of the surface residue.

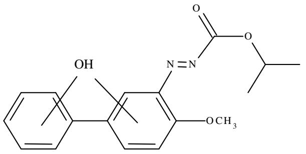
Washed and unwashed tops were extracted by homogenization with acetonitrile+methanol+water (1+1+1, v+v+v) to examine the nature of the residue by HPLC techniques. Bifenazate and bifenazate-diazene were the major identified components of the extractable residues. Another metabolite that appeared in small quantities in the extracts was identified as a ring-hydroxylated diazene. The position of the hydroxyl group on the biphenyl ring was not determined. Another minor metabolite was also observed, but its structure was not determined.

Table 6. Residues in and on radish plants treated with WS formulated [^{14}C]bifenazate, at the equivalent of 1.1 and 2.2 kg ai/ha (Charlton and Tecle, 2002, 2001-147).

Samples and metabolites	Concentration, mg/kg of ^{14}C expressed as parent	
	Treatment 1.1 kg ai/ha	Treatment 2.2 kg ai/ha
Unwashed tops TRR	13	21
Unextractable of unwashed tops TRR	0.74	1.7
Extract of unwashed tops TRR	14	20
bifenazate	1.7	14

Samples and metabolites	Concentration, mg/kg of ^{14}C expressed as parent	
	Treatment 1.1 kg ai/ha	Treatment 2.2 kg ai/ha
bifenazate-diazene	5.7	1.0
ring-hydroxylated bifenazate-diazene 1/	1.2	1.3
Surface wash TRR	7.9	17
bifenazate	7.3	15
bifenazate-diazene	0.2	1.3
ring-hydroxylated bifenazate-diazene	nd	nd
Washed tops TRR	5.7	10
Unextractable of washed tops TRR	0.39	1.4
Extract of washed tops TRR	4.8	7.7
bifenazate	0.9	4.0
bifenazate-diazene	1.7	0.8
ring-hydroxylated bifenazate-diazene	0.4	0.9
Roots TRR	0.0023	0.0043

nd: not detected.



ring-hydroxylated bifenazate-diazene

1/ $C_{17}H_{18}N_2O_4$

Cotton

In a cotton metabolism study in California USA, Panthani and Hatzenbeler (2000, 7137-97-0024-EF-001) foliar sprayed cotton plants (variety Maxxa), at late bloom to early boll set, once with [^{14}C]bifenazate formulated as a WP (wettable powder) at 0.56 and 2.2 kg ai/ha and harvested fuzzy seed, lint and gin trash 112 days later for analysis.

For each plant, cotton seed was removed by hand from the mature open bolls. Each plant was then stripped of all leaves, petioles, calyx and unopened immature bolls to represent the gin trash. The cotton seed was mechanically ginned to produce the lint and fuzzy seed. The gin trash was dried overnight under heat lamps and then portions were ground to a fine powder in a coffee grinder and the samples were placed in a freezer. Fuzzy seed and gin trash were shipped to the laboratory 3 and 10 days after harvest respectively. All samples were stored in a freezer below -5°C and were extracted and analysed within 30 days of harvest.

Bifenazate was extensively metabolised in cotton seed (Table 7). The majority of the hexane extractable residues were shown to be ^{14}C incorporated into triglycerides, while the aqueous acetonitrile extracts contained mainly polar compounds. Bifenazate, bifenazate-diazene, bifenazate-diazene oxide and 4-methoxybiphenyl were present at trace levels (each < 0.001 mg/kg). Acid and base catalysed hydrolyses did not release any recognized compound from possible conjugation.

A high proportion (77–82%) of the gin trash residue was extractable, with bifenazate approximately 50% of the extractable residue and bifenazate-diazene, bifenazate-diazene oxide and 4-methoxybiphenyl identified as minor residue components. Enzyme, acid and base catalysed hydrolysis of the unextractable material suggested that the ^{14}C was incorporated and covalently bound into natural compounds.

In summary, the residues in cotton seed from the use of bifenazate were extensively metabolized, with identified primary metabolites at very low levels. Parent bifenazate was the major identified component of the residue in cotton trash.

Table 7. Residues in cotton seed and gin trash from cotton plants treated with WP formulated [^{14}C]bifenazate, at the equivalent of 0.56 and 2.2 kg ai/ha and harvested 112 days later (Panthani and Hatzenbeler, 2000, 7137-97-0024-EF-001).

Samples and metabolites	Concentration, mg/kg of ^{14}C expressed as parent	
	Treatment 0.56 kg ai/ha	Treatment 2.2 kg ai/ha
Cotton seed, TRR	0.075	0.125
Unextractable of cotton seed, TRR	0.049	0.079
Extractable of cotton seed, TRR	0.026	0.046
bifenazate	< 0.001	< 0.001
bifenazate-diazene	< 0.001	< 0.001
bifenazate-diazene oxide/4-methoxybiphenyl	< 0.001	< 0.001
Gin trash, TRR	0.410	0.838
Unextractable of gin trash, TRR	0.150	0.288
Extractable of gin trash, TRR	0.317	0.685
bifenazate	0.154	0.338
bifenazate-diazene	0.018	0.051
bifenazate-diazene oxide/4-methoxybiphenyl	0.006	
4-methoxybiphenyl		0.009
bifenazate-diazene oxide		0.004

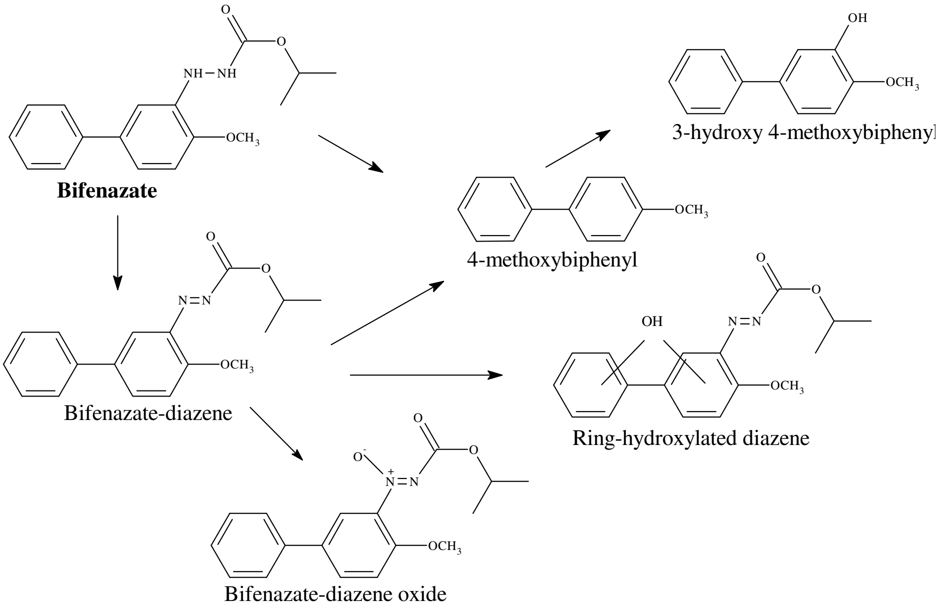


Figure 5. Proposed bifenazate metabolic pathway in crops.

Environmental fate in soil

The Meeting received information on crop rotational studies for bifenazate. Information on soil metabolism and field dissipation was not required because no bifenazate uses as seed treatments or on root crops, if there are such uses, were provided for evaluation.

Crop rotation studies

Information on the fate of radiolabelled bifenazate in a confined crop rotational study was made available to the meeting.

In a confined rotational crop study in USA (Findak, 2000, 6507-95-0124-EF-001) soil (Ohio loamy sand, 1.1% organic matter, 77% sand, 18% silt, 5.2% clay, pH 6.5) was treated directly with ^{14}C labelled bifenazate at a rate equivalent to 0.56 kg ai/ha and allowed to age under greenhouse conditions prior to sowing of the rotational crops. Crops of carrots, lettuce and wheat were sown into the treated soil in pots at intervals of 30, 125 and 360 days after treatment.

Immature lettuce plants were sampled at the 4-5 leaf stage. Immature carrot plants were sampled when carrots were approximately 6 mm in diameter. Wheat forage samples were taken approximately 5 weeks after sowing. The remainder of the crops were grown to maturity, subsequently harvested and analysed for ^{14}C (TRR) content (Table 8). A parallel treatment at the exaggerated rate of 5.6 kg ai/ha was used to assist in the identification of potential metabolites. Precautions were taken to prevent $^{14}CO_2$ from being released into the greenhouse from the treated soils and crops.

Samples were further examined by extraction (acetonitrile and acetonitrile/water). Where extractable residues exceeded 0.01 mg/kg they were examined by HPLC analysis but no parent compound or reference metabolite was observed. Low levels of unidentifiable components were observed as broad unresolved areas in the chromatograms.

The unextractable residual solids from the wheat straw and fractions from the wheat forage were subjected to acid, base and enzyme hydrolysis, but no parent bifenazate or recognizable metabolite was released.

Table 8. Confined rotational crop studies with ^{14}C labelled bifenazate. Soil was treated with ^{14}C bifenazate at a rate equivalent to 0.56 kg ai/ha (Findak, 2000, 6507-95-0124-EF-001).

Application country, year, ref.	Rotational crop (variety)	TSI 1/ days	THI 2/ days	Sample	TRR as bifenazate mg/kg	Extractable, % of TRR	Residues, mg/kg
Bare soil, USA, 1996, 6507-95-0124-EF-001							
	carrot (Nantes Coreless)	30	91	roots	0.033	36%	3/
		30	148	roots	0.007	na	na
	lettuce (Grand Rapids)	30	81	leaves	0.015	33%	3/
		30	163	leaves	0.014	29%	3/
	wheat (Clark)	30	65	forage	0.038	53%	3/
		30	192	straw	0.12	38%	3/
		30	192	chaff	0.031	13%	na
		30	192	grain	0.016	25%	na
	carrot (Nantes Coreless)	125	205	roots	0.010	40%	na
		125	252	roots	0.006	na	na
	lettuce (Grand Rapids)	125	169	leaves	0.013	23%	na
		125	203	leaves	0.005	na	na
	wheat (Clark)	125	169	forage	0.020	25%	3/
		125	309	straw	0.051	24%	3/
		125	309	chaff	0.025	16%	na
		125	309	grain	0.019	16%	na

Application country, year, ref.	Rotational crop (variety)	TSI 1/ days	THI 2/ days	Sample	TRR as bifenazate mg/kg	Extractable, % of TRR	Residues, mg/kg
	wheat (Clark)	360	391	forage	0.018	17%	na
		360	498	straw	0.033	29%	na
		360	498	chaff	0.015	53%	na
		360	498	grain	0.011	55%	na

na: not analysed.

1/ TSI: interval between treatment on soil and sowing of rotation crop, days.

2/ THI: interval between treatment on soil and harvest of rotation crop (or sampling of soil), days.

3/ HPLC analysis did not detect (LOQ 0.01 mg/kg) bifenazate or identifiable metabolites in any raw agricultural commodity.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for analytical methods for residues of bifenazate in raw agricultural commodities, processed commodities, feed commodities, animal tissues, milk and eggs.

Because bifenazate and bifenazate-diazene are readily interconverted by mild oxidation and reduction conditions, the measured residue includes both compounds. The analytical methods use a mild reduction with ascorbic acid to convert the bifenazate-diazene residue to bifenazate before the measurement step. Residues are typically extracted with acetonitrile and water acidified with acetic acid. After a partition clean-up and reduction with ascorbic acid, the residue is analysed by HPLC with coulometer detection.

Jablonski (1998, 6998-97-0237-CR-001) developed an HPLC method with coulometric detection for analysis of the combined residues of bifenazate and bifenazate-diazene in apples and citrus. The oxidative coulometric detection system is quite selective. Substituted hydrazines such as bifenazate are oxidised at 200 mV, but most sample matrix components are not.

Jablonski (1998, 6998-98-0051-CR-001) analysed apples and oranges from [^{14}C]bifenazate crop metabolism studies by the HPLC-coulometer method (6998-97-0237-CR-001) and by an HPLC radiometric method. The sample extraction procedure for the HPLC radiometric analysis was similar to the extraction procedure of method 6998-97-0237-CR-001. The HPLC-coulometer results were approximately 60% of those from the radiometric method (Table 9).

Table 9. Comparison of analyses for bifenazate and bifenazate-diazene on samples from ^{14}C crop metabolism studies by an HPLC-coulometer method (6998-97-0237-CR-001) and an HPLC radiometric method (Jablonski, 1998, 6998-98-0051-CR-001).

Sample	Residues of bifenazate + bifenazate-diazene (mg/kg) by method 6998-97-0237-CR-001	Residues of bifenazate + bifenazate-diazene (mg/kg) by HPLC radiometric method
Apple	0.107	0.186
Apple	0.117	0.170
Orange	0.178	0.342
Orange	0.196	0.327

Apples and citrus (Jablonski, 1998, 6998-97-0237-CR-001)

Analyte:	bifenazate and bifenazate-diazene	HPLC-coulometer	Method 6998-97-0237-CR-001
LOQ:	0.01 mg/kg		
Description	Residues are extracted twice from homogenized matrix with acetonitrile + acetic acid. An aliquot of filtered extract is partitioned with aqueous sodium sulphate and dichloromethane. The organic phase, which contains the residues, is dried and evaporated to near dryness. The residue is taken up in HPLC mobile phase (5% acetonitrile and 95% sodium acetate buffer 50 mM pH 4) containing ascorbic acid. The ascorbic acid reduces bifenazate-diazene to bifenazate. The oxidative coulometric detection system (150-200 mV) after the reversed phase HPLC measures the combined residue as bifenazate. Without the ascorbic acid, bifenazate may readily oxidise to bifenazate-diazene during the analysis. The coulometric detector provides a high degree of selectivity with little background interference observed. If the oil from some citrus samples causes interferences, a hexane wash should be introduced at an early stage of the cleanup.		

Fruit and fruit matrices: peaches, plums, grapes, grape juice, raisins, prunes (Wiedmann, 1999, RP-98018)

Analyte:	bifenazate and bifenazate-diazene	HPLC-coulometer	Method 7543-98-0072-CR-002
LOQ:	0.01 mg/kg		
Description	Residues are extracted twice from chopped matrix by blending with acetonitrile + acetic acid. Grape juice is mixed with the extraction solvent. An aliquot of filtered extract is partitioned with aqueous sodium sulphate and dichloromethane. The organic phase, which contains the residues, is dried and evaporated to near dryness. The residue is taken up in HPLC mobile phase (5% acetonitrile and 95% sodium acetate buffer 50 mM pH 4) containing ascorbic acid. The ascorbic acid reduces bifenazate-diazene to bifenazate. The oxidative coulometric detection system (200 mV) after the reversed phase HPLC measures the combined residue as bifenazate.		

Peaches, raisins and almonds (Wood, 2003, RP-02009)

Analyte:	bifenazate and bifenazate-diazene	LC-MS-MS	Method NCL ME 245
LOQ:	0.01 mg/kg		
Description	Homogenized matrix is extracted with 0.25% acetic acid in 70:30 acetonitrile:water. A portion of the extract is mixed with sodium chloride solution and the residues are partitioned into dichloromethane. The dichloromethane extract is evaporated to a small volume, then mixed with reducing solution (0.25% ascorbic acid in water:acetonitrile) and incubated in a water bath at 50 °C for 1 hour. A portion of the extract is then filtered through a syringe-tip filter and analysed by LC-MS-MS. The [M+H]$^+$ ion is used as the precursor ion for bifenazate. Transitions 301.1/198.1 (for quantification) and 301.1/170.1 are observed. For almonds, an extra step with a hexane wash is introduced after the extraction to remove the almond oil. The ascorbic acid reduction converts bifenazate-diazene residues to bifenazate so the method measures bifenazate and bifenazate-diazene as bifenazate. The method is suitable as a confirmatory method for bifenazate residues.		

Bovine liver, kidney, milk and fat (Wood, 2003, 2003-016)

Analyte:	bifenazate and bifenazate-diazene	LC-MS-MS	Method NCL ME 259
LOQ:	0.01 mg/kg		
Description	Homogenized bovine liver, kidney or milk is extracted with 0.25% acetic acid in 70:30 acetonitrile:water. A portion of the extract is diluted with water and subjected to a solid-phase-extraction column cleanup and a mild reduction with ascorbic acid ready for analysis by LC-MS-MS. The [M+H]$^+$ ion is used as the precursor ion for bifenazate. Transitions 301.1/198.1 (for quantification) and 301.1/170.1 are observed. Homogenized bovine fat is extracted with acetonitrile and excess lipid is removed by centrifugation, cooling and filtration. The extract is subjected to the ascorbic acid reduction and the remainder of the procedure follows that of the other tissues. The method is suitable as a confirmatory method for bifenazate residues.		

Bovine tissues and milk (Jablonski, 1999, 7473)

Analyte: bifenazate and bifenazate-diazene HPLC-coulometer Method 7473-98-0115-CR-001
LOQ: 0.01 mg/kg
Description Extraction, cleanup and ascorbic acid reduction procedures are similar to those described in Method NCL ME 259. The measurement step relies on HPLC with oxidative coulometric detection.

Recovery data from the internal and independent laboratory validation (ILV) testing are summarised in Table 10.

Table 10. Analytical recoveries for spiked bifenazate and diazene in various substrates. Diazene means bifenazate-diazene.

Commodity	Spiked analyte	Spike conc, mg/kg	n	Mean recov%	Range recov%	Method	Ref
almond	bifenazate	0.01-1.0	9	91	83-100	NCL ME 245	RP-02009
almond	diazene	0.01-1.0	9	99	92-112	NCL ME 245	RP-02009
almond hull	bifenazate	0.01-10	9	83	72-97%	6998-97-0237-CR-001	GRL-11866
almond hull	diazene	0.01-10	9	92	80-118%	6998-97-0237-CR-001	GRL-11866
almond kernel	bifenazate	0.01-0.5	9	92	87-95%	6998-97-0237-CR-001	GRL-11866
almond kernel	diazene	0.01-0.5	9	83	76-91%	6998-97-0237-CR-001	GRL-11866
apple	bifenazate	0.01	5	81	75-88	6998-97-0237-CR-001	6998-98-0051-CR-001
apple	bifenazate	0.01-0.10	14	91	71-114%	6998-97-0237-CR-001	6998-97-0237-CR-001
apple	bifenazate	0.01-0.10	6	82	75-89% [2]	6998-97-0237-CR-001	99214 ILV
apple	bifenazate	0.1	5	100	96-103	6998-97-0237-CR-001	6998-98-0051-CR-001
apple	diazene	0.01	5	79	74-82	6998-97-0237-CR-001	6998-98-0051-CR-001
apple	diazene	0.01-0.10	14	81	73-102%	6998-97-0237-CR-001	6998-97-0237-CR-001
apple	diazene	0.01-0.10	6	87	82-91%	6998-97-0237-CR-001	99214 ILV
apple	diazene	0.1	5	91	83-96	6998-97-0237-CR-001	6998-98-0051-CR-001
apricot	bifenazate	0.01-0.1	9	84	75-92	6998-97-0237-CR-001	GRL-11929
apricot	diazene	0.01-0.1	9	85	77-93	6998-97-0237-CR-001	GRL-11929
bovine fat	bifenazate	0.01	5	77	69-84	7473-98-0115-CR-001	7473
bovine fat	bifenazate	0.1	5	94	86-103	7473-98-0115-CR-001	7473
bovine fat	diazene	0.01	5	74	69-85	7473-98-0115-CR-001	7473
bovine fat	diazene	0.2	5	93	90-99	7473-98-0115-CR-001	7473
bovine fat	bifenazate	0.01, 0.1	10	103	97-110	NCL ME 259	2003-016
bovine fat	diazene	0.01, 0.1	10	92	76-106	NCL ME 259	2003-016
bovine kidney	bifenazate	0.01	5	93	83-99	7473-98-0115-CR-001	7473
bovine kidney	bifenazate	0.1	5	105	101-107	7473-98-0115-CR-001	7473
bovine kidney	diazene	0.01	5	77	70-81	7473-98-0115-CR-001	7473
bovine kidney	diazene	0.1	5	83	78-87	7473-98-0115-CR-001	7473
bovine kidney	bifenazate	0.01, 0.1	10	90	79-102	NCL ME 259	2003-016
bovine kidney	diazene	0.01, 0.1	10	85	80-88	NCL ME 259	2003-016
bovine liver	bifenazate	0.01	5	79	78-80	7473-98-0115-CR-001	7473
bovine liver	bifenazate	0.1	5	96	91-100	7473-98-0115-CR-001	7473
bovine liver	diazene	0.01	5	69	56-75	7473-98-0115-CR-001	7473
bovine liver	diazene	0.1	5	83	78-86	7473-98-0115-CR-001	7473
bovine liver	bifenazate	0.01, 0.1	10	106	100-110	NCL ME 259	2003-016
bovine liver	diazene	0.01, 0.1	10	84	79-90	NCL ME 259	2003-016
bovine milk	bifenazate	0.01, 0.1	10	95	87-103	7473-98-0115-CR-001	7473
bovine milk	diazene	0.01	5	77	69-89	7473-98-0115-CR-001	7473
bovine milk	diazene	0.1	5	99	90-106	7473-98-0115-CR-001	7473
bovine milk	bifenazate	0.01, 0.1	10	96	93-99	NCL ME 259	2003-016
bovine milk	diazene	0.01, 0.1	10	77	72-99	NCL ME 259	2003-016
bovine muscle	bifenazate	0.01	5	80	73-83	7473-98-0115-CR-001	7473
bovine muscle	bifenazate	0.1	5	116	102-121	7473-98-0115-CR-001	7473
bovine muscle	diazene	0.01	5	75	67-83	7473-98-0115-CR-001	7473
bovine muscle	diazene	0.1	5	104	99-109	7473-98-0115-CR-001	7473
cantaloupe	bifenazate	0.01-0.5	9	98	82-118%	6998-97-0237-CR-001	GRL-11662
cantaloupe	diazene	0.01-0.5	9	83	70-97%	6998-97-0237-CR-001	GRL-11662

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[2] A recovery of 131% for a 0.01 mg/kg spike was reported as an outlier.

Commodity	Spiked analyte	Spike conc, mg/kg	n	Mean recov%	Range recov%	Method	Ref
cucumber	bifenazate	0.01	2	112	111, 113	6998-97-0237-CR-001	GRL-11670
cucumber	bifenazate	0.1, 1.0	6	76	71-79	6998-97-0237-CR-001	GRL-11670
cucumber	diazene	0.01	3	77	69-87	6998-97-0237-CR-001	GRL-11670
cucumber	diazene	0.1, 1.0	6	97	83-117	6998-97-0237-CR-001	GRL-11670
grape juice	bifenazate	0.01-1.0	16	95	82-105	7543-98-0072-CR-002	RP-98018
grape juice	diazene	0.01-1.0	16	89	79-93	7543-98-0072-CR-002	RP-98018
grapes	bifenazate	0.01-1.0	16	82	72-99	7543-98-0072-CR-002	RP-98018
grapes	diazene	0.01-1.0	16	74	66-84	7543-98-0072-CR-002	RP-98018
orange	bifenazate	0.01	5	85	76-94	6998-97-0237-CR-001	6998-98-0051-CR-001
orange	bifenazate	0.01-0.50	15	87	76-100%	6998-97-0237-CR-001	6998-97-0237-CR-001
orange	bifenazate	0.1	5	91	90-2	6998-97-0237-CR-001	6998-98-0051-CR-001
orange	diazene	0.01	5	96	93-105	6998-97-0237-CR-001	6998-98-0051-CR-001
orange	diazene	0.01-0.50	15	87	70-110%	6998-97-0237-CR-001	6998-97-0237-CR-001
orange	diazene	0.1	5	80	75-84	6998-97-0237-CR-001	6998-98-0051-CR-001
peach	bifenazate	0.01-0.1	9	95	87-102	6998-97-0237-CR-001	GRL-11930
peach	diazene	0.01-0.1	9	92	80-103	6998-97-0237-CR-001	GRL-11930
peach	bifenazate	0.01-1.0	16	79	64-85	7543-98-0072-CR-002	RP-98018
peach	diazene	0.01-1.0	16	76	70-81	7543-98-0072-CR-002	RP-98018
peach	bifenazate	0.01-1.0	9	101	94-106	NCL ME 245	RP-02009
peach	diazene	0.01-1.0	9	101	96-109	NCL ME 245	RP-02009
pecans	bifenazate	0.01-0.5	9	88	70-118	6998-97-0237-CR-001	GRL-11868
pecans	diazene	0.01-0.5	9	87	77-108	6998-97-0237-CR-001	GRL-11868
peppers	bifenazate	0.01-0.5	9	88	70-120%	6998-97-0237-CR-001	GRL-11668
peppers	diazene	0.01-0.5	9	79	70-91%	6998-97-0237-CR-001	GRL-11668
plum	bifenazate	0.01-0.1	9	91	84-100	6998-97-0237-CR-001	GRL-11928
plum	diazene	0.01-0.1	9	83	71-90	6998-97-0237-CR-001	GRL-11928
plum	bifenazate	0.01-1.0	16	83	72-101	7543-98-0072-CR-002	RP-98018
plum	diazene	0.01-1.0	16	81	73-89	7543-98-0072-CR-002	RP-98018
prunes	bifenazate	0.01-1.0	16	81	71-91	7543-98-0072-CR-002	RP-98018
prunes	diazene	0.01-1.0	24	71	60-83	7543-98-0072-CR-002	RP-98018
raisins	bifenazate	0.01-1.0	16	80	71-96	7543-98-0072-CR-002	RP-98018
raisins	diazene	0.01-1.0	16	74	63-86	7543-98-0072-CR-002	RP-98018
raisins	bifenazate	0.01-1.0	9	101	91-114	NCL ME 245	RP-02009
raisins	diazene	0.01-1.0	9	100	94-108	NCL ME 245	RP-02009
strawberry	bifenazate	0.01-1.0	9	91	80-99%	6998-97-0237-CR-001	GRL-11940
strawberry	diazene	0.01-1.0	9	87	84-90%	6998-97-0237-CR-001	GRL-11940
summer squash	bifenazate	0.01-1.0	9	87	72-115%	6998-97-0237-CR-001	GRL-11664
summer squash	diazene	0.01-1.0	9	96	78-122%	6998-97-0237-CR-001	GRL-11664
tomato	bifenazate	0.01-0.5	9	100	88-120	6998-97-0237-CR-001	GRL-11666
tomato	diazene	0.01-0.5	9	94	75-114	6998-97-0237-CR-001	GRL-11666
tomato paste	bifenazate	0.01-0.5	8	82	76-87	6998-97-0237-CR-001	GRL-11666
tomato paste	diazene	0.01-0.5	9	100	84-119	6998-97-0237-CR-001	GRL-11666
tomato puree	bifenazate	0.01-0.5	9	87	81-95	6998-97-0237-CR-001	GRL-11666
tomato puree	diazene	0.01-0.5	9	84	70-94	6998-97-0237-CR-001	GRL-11666

Hackert Anderson and Koch (1999, 45552) examined the applicability of multiresidue methods to the residue analysis of bifenazate and bifenazate-diazene. Because both compounds exhibit fluorescence, testing began with an HPLC-fluorescence method. Bifenazate was not sufficiently stable in methanol and bifenazate-diazene was poorly separated from two decomposition peaks, so the method was not successful. In a GLC multiresidue method, acceptable chromatography was achieved on a DB-1 type column with NPD although some conversion of bifenazate to bifenazate-diazene was observed during chromatography. In the method without Florisil cleanup, recoveries of 24–43% were achieved with spiking an apple sample at 2 mg/kg. Interferences were too much to observe recoveries at 0.1 mg/kg. When a Florisil column cleanup was introduced, recoveries of both compounds were poor (2–22%). Partial conversion of bifenazate to bifenazate-diazene occurred during the cleanup.

In summary, none of the tested multiresidue methods was suitable for the analysis of bifenazate and bifenazate-diazene.

Extraction efficiency of analytical methods

Tissue and milk samples from a goat dosed orally for 4 consecutive days, with [^{14}C]bifenazate at the equivalent of 20 ppm in the feed as in a goat metabolism study, were used for radiovalidation of an analytical enforcement method for bifenazate in animal commodities (Gupta and Cassidy, 2005, 2005-013).

For the enforcement method, samples of milk, fat or liver were extracted by the procedures described for Method 7473-98-0115-CR-001 or Method NCL ME 259. For analysis of 4-hydroxybiphenyl and its sulphate conjugate, a portion of the extract was hydrolysed with hydrochloric acid for 2 hours at 60°C to convert the sulphate conjugate to free 4-hydroxybiphenyl before HPLC analysis.

The results for the enforcement method and the radiolabel measurement showed good agreement for residue analysis of bifenazate, bifenazate-diazene and 4-hydroxybiphenyl in milk, fat and liver (Table 11).

Table 11. Comparison of radiolabel measurement and enforcement analytical method for residues of bifenazate and metabolites in milk and tissues of a goat dosed orally with [^{14}C]bifenazate (Gupta and Cassidy, 2005, 2005-013). All residues are expressed as parent bifenazate.

Sample	Analyte	Determined from radiolabel detection, mg/kg	Enforcement method, mg/kg
Day 3 milk	4-hydroxybiphenyl + sulphate conjugate	0.0015	< 0.0025
Day 4 milk	4-hydroxybiphenyl + sulphate conjugate	0.0022	0.004
Fat	bifenazate + bifenazate-diazene	0.043	0.045
Liver	4-hydroxybiphenyl + sulphate conjugate	0.0062	0.006
Liver	bifenazate + bifenazate-diazene	0.0082	< 0.01

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues of bifenazate residues in apples, apricots, cantaloupe, cherries, cotton seed, cotton seed hulls, cotton seed meal, cotton seed refined oil, egg yolk, fat, gin trash, grape juice, grapes, kidney, liver, milk, mint, muscle, oranges, peaches, peppers, plums, potatoes, poultry liver, poultry muscle, poultry skin + fat, prunes, tomato, tomato paste and tomato puree.

Bifenazate residues (measured as bifenazate + bifenazate-diazene) are not particularly stable in some substrates. Stability is improved where the commodity is stored unchopped and in processed commodities presumably where enzymes are denatured. Bifenazate residues are stable in fat and milk, but are particularly unstable in kidney.

Storage stability data are recorded in the tables unadjusted for concurrent procedural recoveries. If the concurrent procedural recoveries were outside of the 70–120% range the data from that sampling occasion were not taken into account.

Buckrell (2001, GRL-FR-11667) fortified aliquots (approximately 20 g) of homogenised tomato matrix in glass jars with bifenazate for freezer storage stability testing at a temperature below -18°C. After each storage interval, an aged aliquot and a freshly fortified aliquot acting as a procedural recovery were analysed for bifenazate + bifenazate-diazene (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate).

Black (2002, GRL-FR-11853) fortified aliquots (approximately 20 g) of homogenised cherry matrix in plastic bags with bifenazate for freezer storage stability testing. After each storage interval, aged aliquots and freshly fortified aliquots acting as procedural recoveries were analysed (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate).

Benstead (2001, GRL-FR-11669) fortified aliquots (approximately 20 g) of homogenised peppers matrix in glass jars with bifenazate for freezer storage stability testing. After each storage interval, an aged aliquot and a freshly fortified aliquot acting as a procedural recovery were analysed for bifenazate + bifenazate-diazene (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate).

Benstead (2001, GRL-FR-11663) fortified aliquots (approximately 20 g) of homogenised cantaloupe matrix in glass jars with bifenazate for freezer storage stability testing. After each storage interval, an aged aliquot and a freshly fortified aliquot acting as a procedural recovery were analysed for bifenazate + bifenazate-diazene (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate).

Black (2002, GRL-FR-11911) fortified aliquots (approximately 20 g) of homogenised mint tops matrix in plastic bags with bifenazate for freezer storage stability. After each storage interval, aged aliquots and freshly fortified aliquots acting as procedural recoveries were analysed (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate).

Black (2003, GRL-11936) fortified aliquots (approximately 20 g) of homogenised potato tuber matrix in plastic bags with bifenazate for freezer storage stability testing. After each storage interval, aged aliquots and freshly fortified aliquots acting as procedural recoveries were analysed (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate). Because early results were questionable (low recoveries and residues not detected in stored samples), reserve samples were also analysed and two additional samples were run as a second trial.

Black (2004, GRL-12140) fortified aliquots (approximately 20 g) of chopped potato tuber in plastic bags with bifenazate for freezer storage stability testing. After each storage interval, aged aliquots and freshly fortified aliquots acting as procedural recoveries were analysed (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate). Because early results were questionable (low recoveries and residues not detected in stored samples), reserve samples were also analysed and two additional samples were run as a second trial.

Black (2005, GRL-12171) fortified aliquots (approximately 20 g) of sectioned plum tissue in plastic bags with bifenazate for freezer storage stability testing. After each storage interval, aged aliquots and freshly fortified aliquots acting as procedural recoveries were analysed (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate).

Black (2005, GRL-12172) fortified aliquots (approximately 20 g) of tomato paste and tomato puree in plastic bags with bifenazate for freezer storage stability testing. After each storage interval, aged aliquots and freshly fortified aliquots acting as procedural recoveries were analysed (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate).

Wiedmann and Korpalski (1999, RP-98019) fortified aliquots (approximately 20 g) of homogenates of peaches, grapes, apples, oranges, grape juice and prunes in glass jars with bifenazate and bifenazate-diazene for freezer storage stability testing. They also fortified some sample types on the intact surface (whole grapes and sections of apples and peaches) to determine the stability when stored as whole fruit. After each storage interval, aged aliquots and freshly fortified aliquots acting as procedural recoveries were analysed (Table 12). Analytical results were reported as bifenazate + bifenazate-diazene (as bifenazate). Homogenized tissues appeared to cause faster degradation of the residues than whole fruit. Residues were reasonably stable in processed commodities such as prunes and grape juice.

Table 12. Freezer storage stability data for bifenazate and metabolites spiked into matrices of apples, apricots, cantaloupe, cherries, cotton seed, cotton seed hulls, cotton seed meal, cotton seed refined oil, gin trash, grape juice, grapes, mint, oranges, peaches, peppers, plums, potatoes, prunes, tomato, tomato paste and tomato puree.

Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg	Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg
Tomato, homogenized matrix fortified with bifenazate at 0.1 mg/kg (Buckrell, 2001, GRL-FR-11667) storage temp below below -18°C.			Cherries, homogenized matrix, fortified with bifenazate at 0.1 mg/kg (Black, 2002, GRL-FR-11853), storage temp below below -20°C.		
1 month	80%	0.067	1 month	105% 113%	0.080 0.077 0.094
3 months	101%	0.075	3 months	92% 90%	0.046 0.044 0.054
6 months	96%	0.080	6 months	93% 81%	0.038 0.046 0.038
					30% decline in 2.6 months.
Peppers, homogenized matrix fortified with bifenazate at 0.1 mg/kg (Benstead, 2001, GRL-FR-11669), storage temp below below -18°C.			Cantaloupe, homogenized matrix fortified with bifenazate at 0.1 mg/kg (Benstead, 2001, GRL-FR-11663), storage temp below below -18°C. (30% decline in		
1 month	84%	0.083	1 month	83%	0.074
3 months	95%	0.076	3 months	90%	0.063
6 months	96%	0.077	6 months	95%	0.047
					30% decline in 3.9 months
Mint tops, homogenized matrix fortified with bifenazate at 0.1 mg/kg (Black, 2002, GRL-FR-11911), storage temp between -17.7°C and -26.5°C.			Potatoes, chopped tuber fortified with bifenazate at 0.1 mg/kg (Black, 2003, GRL-11936), storage temp between -22.1°C and -27.1°C.		
28 days	90% 79%	0.095 0.068 0.074	0	38%	
102 days	81% 88%	0.11 0.095 0.098	1 month	112% 101%	< 0.005 (3)
			Trial 2	93% 100 %	< 0.005 (3)
			1.5 months	45% 97%	< 0.005 (3)
			3 months	82% 30%	< 0.01 < 0.005 (2)
Potatoes, chopped tuber fortified with bifenazate at 0.1 mg/kg (Black, 2004, GRL-12140), storage temp between -23.4°C and -27.0°C.			Plums, sectioned tissue fortified with bifenazate at 0.1 mg/kg (Black, 2005, GRL-12171), storage temp between -22.4°C and -26.8°C.		
0	79% 70% 80%		0	78% 81% 76%	
1 week	103% 104%	0.014 0.018 0.019	1 week	73% 72% 78%	0.078 0.074
2 weeks	107% 89%	0.011 0.012 0.013	2 weeks	76% 75% 61%	0.073 0.071
4 weeks	89% 87%	0.012 0.017 0.020	4 weeks	70% 71% 77%	0.070 0.074
Tomato paste, fortified with bifenazate at 1 mg/kg (Black, 2005, GRL-12172), storage temp between -18.7°C and -27.2°C.			Tomato puree, fortified with bifenazate at 1 mg/kg (Black, 2005, GRL-12172), storage temp between -18.7°C and -27.2°C.		
0	80% 80% 78%		0	83% 85% 84%	
1 week	83% 83%	0.85 0.81 0.80	1 week	100% 92%	0.89 1.00 0.90
2 weeks	87% 82%	0.80 0.76 0.81	2 weeks	95% 90%	0.99 0.97 0.83
4 weeks	84% 87%	0.75 0.73 0.81	4 weeks	87% 89%	0.82 0.80 0.85
Cottonseed matrix, fortified at 0.1 mg/kg bifenazate (Wiedmann, 2000, 10495-1)			Cottonseed matrix, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann, 2000, 10495-1)		
0	78% 100% 98%		0	93% 85% 90%	
21 days	99%	0.038 0.038	21 days	88%	0.038 0.046
56 days	99%	0.059 0.067	56 days	83%	0.040 0.043
Gin trash, fortified at 0.1 mg/kg bifenazate (Wiedmann, 2000, 10495-1)			Gin trash, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann, 2000, 10495-1)		
0	76% 76% 83%		0	73% 75% 76%	
44 days	79%	0.051 0.055	44 days	84%	0.049 0.041
Cotton seed hulls, fortified at 0.1 mg/kg bifenazate (Wiedmann, 2000, 10495-1)			Cotton seed hulls, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann, 2000, 10495-1)		
0	89% 87% 77%		0	84% 83% 83%	
52 days	98%	0.070 0.063	52 days	93%	0.067 0.064

Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg	Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg
Cotton seed meal, fortified at 0.1 mg/kg bifenazate (Wiedmann, 2000, 10495-1)			Cotton seed meal, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann, 2000, 10495-1)		
0	95% 94% 99%		0	88% 91% 88%	
43 days	96%	0.058 0.055	43 days	92%	0.071 0.080
Cotton seed refined oil, fortified at 1 mg/kg bifenazate (Wiedmann, 2000, 10495-1)			Cotton seed refined oil, fortified at 1 mg/kg bifenazate-diazene (Wiedmann, 2000, 10495-1)		
0	55% 66% 70%		0	73% 77% 79%	
28 days	63% 1/	0.74 0.77	28 days	85%	0.72 0.75
Homogenized apricots fortified at 0.1 mg/kg bifenazate (Wesley, 2002, GRL11934) stored at -20.3 °C to -28.9 °C					
0	76%				
1 month	92% 77%	0.066 0.076 0.070			
1.5 months	84% 76%	0.065 0.059 0.061			
2 months	78% 72%	0.052 0.065			
Homogenized apples, fortified at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Homogenized apples, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	94% 92% 97% 109%		0	95% 96% 95% 97%	
7 days	91% 95%	0.078 0.080	7 days	94% 91%	0.086 0.081
14 days	72% 69%	0.056 0.057	14 days	70% 78%	0.065 0.063
21 days	68% 68% 1/	0.046 0.041	21 days	63% 67% 1/	0.048 0.048
29 days	73% 77%	0.053 0.058	29 days	81% 81%	0.065 0.063
42 days	67% 67% 1/	0.052 0.052	42 days	68% 72%	0.050 0.048
70 days	91% 88%	0.057 0.055	70 days	86% 79%	0.051 0.056
107 days	87% 88%	0.042 0.042	107 days	77% 81%	0.041 0.044
182 days	89% 91%	0.038 0.038	182 days	79% 71%	0.037 0.041
		30% decline in 106 days			30% decline in 97 days
Apples, fortified on the skin surface at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Apples, fortified on the skin surface at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	88% 93% 88% 90%		0	77% 76% 73% 76%	
14 days	81% 92%	0.088 0.092	14 days	81% 83%	0.075 0.090
28 days	94% 88%	0.078 0.090	28 days	79% 79%	0.080 0.069
56 days	88% 84%	0.098 0.086	56 days	89% 90%	0.070 0.068
126 days	92% 87%	0.093 0.096	126 days	90% 83%	0.073 0.051
224 days	94% 92%	0.098 0.088	224 days	86% 80%	0.072 0.082
Homogenized grapes (stems removed), fortified at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Homogenized grapes (stems removed), fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	92% 90% 95% 99%		0	88% 89% 86% 86%	
7 days	90% 92%	0.066 0.062	7 days	89% 89%	0.069 0.073
14 days	81% 79%	0.051 0.049	14 days	73% 74%	0.050 0.049
21 days	76% 79%	0.045 0.035	21 days	74% 75%	0.039 0.039
29 days	81% 90%	0.046 0.031	29 days	82% 78%	0.045 0.044
42 days	79% 72%	0.044 0.031	42 days	77% 74%	0.031 0.037
70 days	87% 83%	0.023 0.019	70 days	85% 87%	0.020 0.024
		30% decline in 23 days			30% decline in 22 days
Grapes (stems removed), fortified on the surface at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Grapes (stems removed), fortified on the surface at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	71% 81% 100% 95%		0	71% 76% 86% 89%	
14 days	107% 98%	0.088 0.088	14 days	93% 92%	0.086 0.086
28 days	94% 89%	0.083 0.081	28 days	84% 90%	0.074 0.074
56 days	85% 87%	0.079 0.070	56 days	86% 90%	0.079 0.068
126 days	96% 94%	0.081 0.083	126 days	94% 90%	0.073 0.078
224 days	84% 97%	0.073 0.076	224 days	82% 84%	0.066 0.067

Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg	Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg
Homogenized peaches (seeds removed), fortified at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Homogenized peaches (seeds removed), fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	89% 91% 91% 88%		0	88% 92% 90% 91%	
7 days	81% 84%	0.068 0.067	7 days	81% 79%	0.070 0.070
14 days	81% 80%	0.058 0.030	14 days	96% 86%	0.061 0.067
21 days	74% 79%	0.048 0.053	21 days	76% 67%	0.049 0.054
28 days	71% 71%	0.049 0.052	28 days	68% 72%	0.057 0.059
42 days	91% 84%	0.062 0.056	42 days	92% 89%	0.063 0.074
70 days	84% 81%	0.047 0.055	70 days	86% 81%	0.055 0.057
105 days	83% 85%	0.041	105 days	75% 82%	0.036 0.033
182 days	87% 84%	0.033 0.033	182 days	77% 80%	0.035 0.037
		30% decline in 126 days			30% decline in 92 days
Peaches, fortified on the skin surface at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Peaches, fortified on the skin surface at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	81% 86% 97% 93%		0	67% 66% 82% 86%	
14 days	98% 92%	0.075 0.084	14 days	80% 89%	0.066 0.072
28 days	83% 78%	0.058 0.059	28 days	83% 74%	0.050 0.042
56 days	91% 95%	0.060 0.077	56 days	79% 90%	0.052 0.044
126 days	101% 88%	0.049 0.062	126 days	89% 82%	0.035 0.040
223 days	98% 94%	0.063 0.067	223 days	77% 79%	0.044 0.056
Homogenized whole oranges, fortified at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Homogenized whole oranges, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	87% 84% 81% 89%		0	86% 82% 82% 85%	
7 days	79% 83%	0.063 0.063	7 days	78% 79%	0.068 0.071
14 days	89% 88%	0.056 0.066	14 days	79% 74%	0.061 0.062
28 days	92% 87%	0.070 0.059	28 days	81% 83%	0.060 0.076
40 days	91% 92%	0.064 0.062	40 days	72% 69%	0.069 0.065
75 days	95% 94%	0.066 0.068	75 days	87% 80%	0.069 0.072
105 days	82% 87%	0.055 0.050	105 days	72% 71%	0.058 0.066
186 days	95% 96%	0.052 0.051	186 days	81% 83%	0.065 0.068
Homogenized grape juice, fortified at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Homogenized grape juice, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	95% 95% 99% 98%		0	89% 89% 92% 93%	
7 days	99% 99%	0.089 0.105	7 days	87% 84%	0.085 0.087
14 days	98% 95%	0.096 0.098	14 days	88% 88%	0.084 0.083
28 days	94% 94%	0.096 0.101	28 days	86% 89%	0.085 0.082
40 days	91% 90%	0.091 0.089	40 days	81% 80%	0.081 0.079
75 days	97% 102%	0.103 0.096	75 days	90% 91%	0.089 0.084
107 days	96% 94%	0.095 0.095	107 days	82% 77%	0.087 0.086
186 days	99% 97%	0.104	186 days	83% 84%	0.085 0.087
Homogenized prunes, fortified at 0.1 mg/kg bifenazate (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.			Homogenized prunes, fortified at 0.1 mg/kg bifenazate-diazene (Wiedmann and Korpalski, 1999, RP-98019) stored at freezer temperature -24 °C to -20 °C.		
0	73% 72% 76% 72%		0	72% 70% 66% 67%	
7 days	76% 80%	0.075 0.069	7 days	71% 73%	0.070 0.068
14 days	75% 73%	0.082 0.073	14 days	77% 81%	0.063 0.063
28 days	83% 80%	0.073 0.070	28 days	78% 70%	0.064 0.061
42 days	80% 85%	0.072 0.073	42 days	72% 79%	0.057 0.062
70 days	86% 87%	0.067 0.066	70 days	75% 71%	0.056 0.052
105 days	84% 84%	0.071 0.068	105 days	79% 71%	0.056 0.057
182 days	86% 94%	0.080 0.078	182 days	70% 66% 1/	0.055 0.053

Jablonski (1999, 7475) tested the freezer storage stability of bifenazate, bifenazate-diazene and metabolite 4-hydroxybiphenyl fortified in bovine milk and tissues at 0.20 mg/kg (Table 13). It should be noted that the tests for 2 days storage were done in a follow-up experiment after substantial losses had been found after 14 days storage.

The compounds were stable in milk for the interval tested (202 days). The compounds were also reasonably stable in fat. In muscle, bifenazate and bifenazate-diazene declined within a few days with less than 50% remaining after 14 days. Metabolite 4-hydroxybiphenyl was much more stable. In liver and kidney, bifenazate and bifenazate-diazene also declined rapidly with less than 50% of the spiked concentration remaining after 2 days. Metabolite 4-hydroxybiphenyl was reasonably stable in liver and kidney with 65−85% of the spiked concentration remaining after 9−11 weeks of storage.

The instability of bifenazate-diazene in bovine kidney matrix was noted during the analytical recovery testing. At time 0, the recoveries were treated in the same way as the storage test samples, i.e., 5−10 minutes were allowed after fortification for the fortification solvent to evaporate before the jars were capped and placed in the freezer. In day 2 and day 14 procedural recoveries, samples were extracted immediately after fortification.

Table 13. Freezer storage stability testing of fortified bifenazate, bifenazate-diazene and 4-hydroxybiphenyl in bovine tissues and milk (Jablonski, 1999, 7475). Samples were stored at freezer temperature -24 °C to -20 °C.

Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg	Procedural recov %	4-hydroxybiphenyl mg/kg
MILK, fortified at 0.20 mg/kg bifenazate			MILK, fortified at 0.20 mg/kg of bifenazate-diazene		MILK, fortified at 0.20 mg/kg of 4-hydroxybiphenyl	
0 days	96% (n=6)		97% (n=6)		108% (n=6)	
14 days	100%	0.188	97%	0.194	104%	0.201
42 days	95%	0.168	88%	0.163	101%	0.183
85 days	86%	0.160	85%	0.157	102%	0.183
202 days	96%	0.162	78%	0.156	95%	0.167
MUSCLE, fortified at 0.20 mg/kg bifenazate			MUSCLE, fortified at 0.20 mg/kg of bifenazate-diazene		MUSCLE, fortified at 0.20 mg/kg of 4-hydroxybiphenyl	
0 days	105% (n=6)		89% (n=6)		96% (n=6)	
2 days	83%	0.100	83%	0.12		
14 days	102%	0.097	92%	0.031	103%	0.180
28 days	98%	0.042	90%	0.000	93%	0.155
86 days					98%	0.136
		30% decline in 10 days		30% decline in 2 days	30% decline in approx 100 days	
LIVER, fortified at 0.20 mg/kg bifenazate			LIVER, fortified at 0.20 mg/kg of bifenazate-diazene		LIVER, fortified at 0.20 mg/kg of 4-hydroxybiphenyl	
0 days	98% (n=6)		82% (n=6)		95% (n=6)	
2 days	73%	0.050	76%	0.0146		
14 days	106%	0.179	92%	0.032	98%	0.181
76 days					98%	0.175
KIDNEY, fortified at 0.20 mg/kg bifenazate			KIDNEY, fortified at 0.20 mg/kg of bifenazate-diazene		KIDNEY, fortified at 0.20 mg/kg of 4-hydroxybiphenyl	
0 days	91% (n=6)		57% (n=6)		91% (n=6)	
2 days	72%	0.092	69%	0.041		
14 days	95%	0.121	79%	0.003	99%	0.155
63 days					101%	0.133

Storage interval	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg	Procedural recov %	Bifenazate + bifenazate-diazene, mg/kg	Procedural recov %	4-hydroxybiphenyl mg/kg
FAT, fortified at 0.20 mg/kg bifenazate			FAT, fortified at 0.20 mg/kg of bifenazate-diazene		FAT, fortified at 0.20 mg/kg of 4-hydroxybiphenyl	
0 days	88% (n=6)		88% (n=6)		106% (n=6)	
14 days	75%	0.123	75%	0.148	103%	0.168
36 days	93%	0.147	81%	0.158	109%	0.168
95 days	104%	0.141	82%	0.156	107%	0.151

Procedural recoveries are means of duplicate samples (except as stated otherwise) and concentrations in test samples are means of 4 replicates.

Labelled bifenazate was spiked into control samples of goat milk (0.04 and 0.25 mg/kg), muscle (0.25 mg/kg), fat (0.25 mg/kg), liver (2.0 mg/kg) and kidney (0.25 mg/kg) for freezer storage stability testing. Samples were analysed by HPLC-LSC methods, initially and after 9 months of storage below -10 °C (McClanahan and Bayus, 1999, 96-0064). Stability data are summarised in Table 14 and Table 15. Metabolites 4-methoxybiphenyl and bifenazate-diazene are recorded as a combined residue because of only partial separation on the HPLC systems used for analysis. In cases where some resolution was achieved, bifenazate-diazene was a substantial part of the combined residue and sometimes the major part of the residue.

Bifenazate was stable to the fortification and extraction procedure for milk, muscle, fat and liver (day 0 samples). For kidney, only 27% of the fortified bifenazate was recovered from the day 0 sample, with 40% and 5% appearing as 4-methoxybiphenyl + bifenazate-diazene and 4-hydroxybiphenyl respectively.

Bifenazate was stable in the fat samples for 9 months freezer storage. In milk after 9 months freezer storage, bifenazate had declined to 23% of its fortification level, with 60% appearing as 4-methoxybiphenyl + bifenazate-diazene. In muscle after 9 months freezer storage, bifenazate had declined to 8–10% of its fortification level, with 40–44% appearing as 4-methoxybiphenyl + bifenazate-diazene and 26–30% unextractable from the matrix.

In kidney after 9 months freezer storage, bifenazate had declined to 1.2% of its fortification level, with 46% appearing as 4-methoxybiphenyl + bifenazate-diazene and 25% unextractable from the matrix. In liver after 9 months freezer storage, bifenazate had declined to 9% of its fortification level, with 64% appearing as 4-methoxybiphenyl + bifenazate-diazene and 14% unextractable from the matrix.

Samples from the lactating goat metabolism study (McClanahan and Bayus, 1999, 96-0064) were analysed by HPLC before and after freezer storage of 15–187 days to test the stability of incurred residues (muscle 79 days; fat 15 days; liver 111 days; kidney 187 days; milk 81 days). The qualitative appearance of the initial and final chromatograms was reasonably similar for milk, fat, liver and kidney. Substantial changes were apparent for muscle.

Table 14. Freezer storage stability testing of fortified [^{14}C]bifenazate in tissues of lactating goats (McClanahan and Bayus, 1999, 96-0064). Samples were stored below -10 °C.

	Concentration, mg/kg, expressed as parent							
	Loin muscle		Leg muscle		Omental fat		Peri-renal fat	
Component	initial	9 months	initial	9 months	initial	9 months	initial	9 months
Total ^{14}C residue (TRR)	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Extracted residue	0.25	0.17	0.24	0.17	0.25	0.24	0.24	0.24
Unextractable		0.064		0.074		0.007		0.006
Bifenazate	0.20	0.019	0.20	0.026	0.23	0.20	0.23	0.21
4-hydroxybiphenyl	nd	0.009	nd	0.011	nd	nd	nd	nd
4-methoxybiphenyl + bifenazate-diazene	0.037	0.10	0.032	0.11	0.012	0.028	0.009	0.027

nd: not detected. Detection limit approximately 0.0001 mg/kg.

Table 15. Freezer storage stability testing of fortified [^{14}C] bifenazate in tissues and milk of lactating goats (McClanahan and Bayus, 1999, 96-0064). Samples were stored below -10 °C.

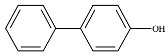
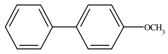
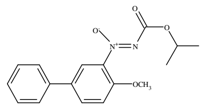
Component	Concentration, mg/kg, expressed as parent					
	Milk		Liver		Kidney	
	initial	9 months	initial	9 months	initial	9 months
Total ^{14}C residue (TRR)	0.04	0.25	2.0	2.0	0.25	0.25
Extracted residue	0.042	0.23	1.98	1.65	0.25	0.17
Unextractable		0.001		0.28		0.062
Bifenazate	0.034	0.057	1.7	0.18	0.067	0.003
4-hydroxybiphenyl	nd	0.003	nd	0.045	0.013	0.010
4-methoxybiphenyl + bifenazate-diazene	0.004	0.15	0.17	1.28	0.099	0.115

nd: not detected. Detection limit approximately 0.0001 mg/kg.

Bifenazate spiked into control samples of egg yolk, hen skin + fat and thigh muscle at approximately 0.10–0.13 mg/kg and liver at 0.60 mg/kg was tested for freezer storage stability. Samples were analysed by HPLC-LSC methods, initially and after 4.5−6 months of storage below -10 °C (McClanahan *et al*., 1999, 96-0265). Stability data are summarised in Table 16. Bifenazate residues were stable in egg yolk and liver. In thigh muscle, 45% of the bifenazate disappeared, with 14% and 11% appearing as 4-hydroxybiphenyl and bifenazate-diazene respectively. In skin + fat, 97% of the bifenazate disappeared with 4%, 4% and 59% appearing as 4-hydroxybiphenyl, 4-methoxybiphenyl and bifenazate-diazene respectively.

Samples from the laying hen metabolism study (McClanahan *et al*., 1999, 96-0265) were analysed by HPLC before and after freezer storage of 121−171 days to test the stability of incurred residues. The qualitative appearance of the initial and final chromatograms was reasonably similar for egg yolk, skin-with-fat and liver. Substantial changes were apparent for thigh muscle, but total residues in thigh muscle were very low (0.006 mg/kg).

Table 16. Freezer storage stability testing of fortified [^{14}C] bifenazate in tissues and eggs of laying hens (McClanahan *et al*., 1999, 96-0265). Samples were stored below -10 °C.

Component	Concentration, mg/kg, expressed as parent							
	Egg yolk		Skin + fat		Thigh muscle		Liver	
	initial	6 months storage	initial	4.5 months storage	initial	6 months storage	initial	4.5 months storage
Total ^{14}C residue	0.13	0.13	0.11	0.10	0.13	0.13	0.61	0.61
Extracted residue	0.13	0.13	0.10	0.077	0.13	0.11	0.60	0.59
Unextractable			nd	0.013		0.018		
Bifenazate	0.12	0.12	0.095	0.003	0.12	0.067	0.57	0.54
4-hydroxy biphenyl 	nd	nd	nd	0.004	nd	0.017	0.005	nd
4-methoxy biphenyl 	0.002	0.001	0.001	0.004	0.002	0.002	0.021	0.009
bifenazate-diazene 	0.008	0.006	0.004	0.056	0.006	0.013	0.012	0.009

nd: not detected. Detection limits for tissues and egg yolks were 0.005 and 0.003 mg/kg respectively.

USE PATTERN

Bifenazate is a selective acaricide which controls the motile stage of mites either by direct contact or through contact with foliar residues. Bifenazate blocks or closes the gamma-aminobutyric acid

(GABA) activated chloride channels of susceptible pests resulting in over-excitation of the peripheral nervous system.

Bifenazate products are mixed with water and applied as foliar sprays or broadcast treatments using aerial or ground equipment equipped for conventional spraying on crops. Bifenazate is not systemic in action; therefore complete coverage of both upper and lower leaf surfaces is necessary for effective control. Bifenazate is effective for the control of a variety of mite species, especially spider mites, red mites, and grass mites. The Meeting received information on bifenazate registered uses in Australia, Japan, Netherlands and USA. In The Netherlands, bifenazate is registered for use only in floriculture and on nursery trees and perennials.

Table 17. Registered field uses of bifenazate in Australia, Japan and USA. Labels for the following uses were available to the Meeting.

Crop	Country	Application						
		Form	Type	Rate kg ai/ha	Conc kg ai/hL	Min spray vol, L/ha	Max number	PHI days
Almonds	USA	500 WP	foliar	0.42-0.56		470	1	7
Apple	Australia	480 SC	foliar		0.031	1000	1	7
Apple	Japan	200 SC	foliar	0.27-1.4			1	7
Apricot	Australia	480 SC	foliar		0.031	1000	1	3
Cherry	Japan	200 SC	foliar	0.27-1.4			1	14
Cotton	USA	480 SC	foliar, aerial application	0.4-0.8		47	1	60
Cotton	USA	480 SC	foliar, ground application	0.4-0.8		190	1	60
Cucumber	Japan	200 SC	foliar	0.3-0.6			1	1
Cucurbit vegetables[3]	USA	500 WP	foliar	0.42-0.56		470	1	3
Egg plant	Japan	200 SC	foliar	0.3-0.6			1	1
Filbert	USA	500 WP	foliar	0.42-0.56		470	1	14
Fruiting vegetables[4]	USA	500 WP	foliar	0.42-0.56		470	1	3
Grapes	Japan	200 SC	foliar	0.27-1.4			1	21
Grapes	USA	500 WP	foliar	0.42-0.56		470	1	14
Hops	USA	500 WP	foliar	0.42-0.84		470	1	14
Lime	Japan	200 SC	foliar	0.27-1.4			1	7
Mandarin	Japan	200 SC	foliar	0.27-1.4			1	7
Melon	Japan	200 SC	foliar	0.3-0.6			1	1
Mint	USA	500 WP	foliar	0.42-0.84		470	1	7
Nectarine	Australia	480 SC	foliar		0.031	1000	1	3
Non-bearing crops	USA	500 WP	foliar	0.42-0.56		470	1	
Okra	USA	500 WP	foliar	0.42-0.56		470	1	3
Orange	Japan	200 SC	foliar	0.27-1.4			1	7
Other tree nuts[5]	USA	500 WP	foliar	0.42-0.56		470	1	7
Peach	Australia	480 SC	foliar		0.031	1000	1	3

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[3] Cucurbit vegetables include cucumbers, muskmelon, pumpkin, squash and watermelon.

[4] Fruiting vegetables include eggplants, peppers (bell and non-bell) and tomatoes. Use only on tomatoes greater than 1 inch in diameter when mature.

[5] Other tree nuts include Beech nut, Brazil nut, Butternut, Cashew, Chestnut, Hickory nut, Macadamia nut.

Crop	Country	Application						
		Form	Type	Rate kg ai/ha	Conc kg ai/hL	Min spray vol, L/ha	Max number	PHI days
Peach	Japan	200 SC	foliar	0.27-1.4			1	7
Pear	Australia	480 SC	foliar		0.031	1000	1	7
Pear	Japan	200 SC	foliar	0.27-1.4			1	1
Pecan	USA	500 WP	foliar	0.42-0.56		470	1	14
Pistachios	USA	500 WP	foliar	0.42-0.56		470	1	14
Plum	Australia	480 SC	foliar		0.031	1000	1	3
Plum	Japan	200 SC	foliar	0.27-1.4			1	2
Pome fruit[6]	USA	500 WP	foliar	0.42-0.56		470	1	7
Stone fruit[7]	USA	500 WP	foliar	0.42-0.56		470	1	3
Strawberries	Japan	200 SC	foliar	0.27-1.4			1	1
Strawberries	USA	500 WP	foliar	0.42-0.56		940	2	1
Tea	Japan	200 SC	foliar	0.4-0.8			1	14
Tomato	Japan	200 SC	foliar	0.3-0.6			1	1
Walnuts	USA	500 WP	foliar	0.42-0.56		470	1	14
Watermelon	Japan	200 SC	foliar	0.3-0.6			1	1

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for bifenazate uses on the following crops.

Citrus fruits	citrus	Table 19
Pome fruits	apples	Table 20
	pears	Table 21
Stone fruits	apricot	Table 22
	peach	Table 23
	plums	Table 24
	cherries	Table 25
Berry fruits	grapes	Table 26
	strawberries	Table 27
Tropical fruits	figs	Table 28
Cucurbits	cantaloupe	Table 29
	watermelon	Table 30
	cucumber	Table 31
	summer squash	Table 32
Fruiting vegetables	tomatoes	Table 33
	peppers	Table 34
	egg plant	Table 35
Tree nuts	almonds, pecans	Table 36
Oil seeds	cotton seed	Table 37
Herbs	mint	Table 38
	hops	Table 39
	tea	Table 40

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[6] Pome fruit include apple, crabapple, pear, quince.

[7] Stone fruit include nectarines, peach, plums/prunes.

Animal feeds	almond hulls, cotton gin trash	Table 41

Trials from Japan were available only in summary form.

Trials from USA and Canada were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are recorded unadjusted for recovery.

In most trials, duplicate field samples from an unreplicated plot were taken at each sampling time and were analysed separately. The mean of the two analytical results was taken as the best estimate of the residues in the plot and the means are recorded in the tables.

When residues were not detected they are shown as below the LOQ (e.g. < 0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are double underlined.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most trial designs used non-replicated plots. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Table 18. Summary of sprayers, plot size and field sample size in the US supervised trials.

Crop	Country	Year	Sprayer	Plot size	Sample size
Almond	USA	2001	tractor-mounted airblast	16-18 trees	1 kg
Apple	USA	1998	tractor-mounted airblast	180-560 m^2	24 fruit
Apricot	USA	2002	airblast sprayer	16-20 trees	2 kg
Cantaloupe	USA	2000	tractor-mounted boom, backpack	37-74 m^2	2 kg
Cherries	USA	2001	airblast, handgun sprayer	54-500 m^2	
Cotton seed	USA	1999, 2000	tractor-mounted boom, CO_2 powered, backpack, ATV	90-1200 m^2	0.5-15 kg
Cucumber	USA	2000	tractor-mounted boom, backpack boom	30-60 m^2	12 fruits
Grapes	USA	1998	tractor-mounted airblast	75-250 m^2	12 bunches
Hops	USA	1999	airblast	140-260 m^2	4 kg green
Mint tops	USA	2000, 2001	backpack boom, ATV-mounted boom, tractor-mounted boom	33-90 m^2	
Peach	USA	1998, 2002	airblast sprayer	12-17 trees	24 fruits
Pears	USA	1998	airblast sprayer	16-18 trees	2 kg
Pecan	USA	2001	tractor-mounted airblast	6 trees	1 kg
Peppers	USA	2000	tractor-mounted boom, backpack boom	14-60 m^2	
Plums	USA	1998	tractor-mounted airblast	15-16 trees	24-50 fruits
Strawberries	USA	1999	backpack sprayers	26-90 m^2	1 kg
Summer squash	USA	2000	tractor-mounted boom, backpack boom	20-90 m^2	
Tomato	USA	2000, 2001	tractor-mounted boom, backpack boom	8-110 m^2	12-15 fruit

Intervals of freezer storage between sampling and analysis were recorded for all trials and were compared with intervals in the freezer storage stability studies.

Table 19. Bifenazate residues in citrus fruit resulting from supervised trials in Japan.

CITRUS FRUITS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	1/	as bifenazate, mg/kg	
MANDARIN									
Japan, 1997 (Nankan 20 gou)	200 SC	1.2	0.02	6000	4	7 14 30 45	pu, pe pu, pe pu, pe pu, pe	0.02, 2.0 0.02, 0.90 0.02, 1.4 0.02, 1.2	Report No 1 Tokushima
Japan, 1997 (Nitinan 1 gou)	200 SC	2.0	0.02	10000	4	7 14 30 45	pu, pe pu, pe pu, pe pu, pe	< 0.02, 3.9 0.02, 3.7 < 0.02, 3.1 < 0.02, 2.4	Report No 2 Miyazaki
NATSUDAIDAI									
Japan, 1997 (Kawano-Natsudai)	200 SC	1.0	0.02	5000	1	7 14 30 45	pu, pe, wf pu, pe, wf pu, pe, wf pu, pe, wf	0.03, 0.72, 0.24 0.02, 0.64, 0.20 0.02, 0.37, 0.14 0.03, 0.44, 0.14	Report No 3, 4 Yamaguti
Japan, 1997 (Kawano-Natsudai)	200 SC	1.2	0.02	6000	1	7 14 30 45	pu, pe, wf pu, pe, wf pu, pe, wf pu, pe, wf	< 0.02, 0.61, 0.21 < 0.02, 0.38, 0.15 < 0.02, 0.30, 0.11 < 0.02, 0.07, 0.04	Report No 3, 4 Ehime
LIME									
Japan, 1997 (Sudati lime)	200 SC	1.2	0.02	6000	4	7 14 30 45	wf	0.27 0.08 0.10 0.10	Report No 5 Tokushima
Japan, 1997 (Kabosu lime)	200 SC	1.4	0.02	7000	4	7 14 21 28	wf	0.30 0.27 0.13 0.07	Report No 5 Ooita

1/ pu: pulp; pe: peel; wf: whole fruit.

Table 20. Bifenazate residues in apples resulting from supervised trials in USA and Japan.

APPLE	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/ 2/	
USA (NY) 1998, Idared	500 WP	2.8		470	1	7	whole fruit	1.3	RGC-98107 GRL-11419
USA (WA) 1998, Red Delicious	500 WP	2.8		460	1	7	whole fruit	2.0	DNJ-98107 GRL-11419
USA (NY) 1998, Monroe	500 WP	0.56	0.12	470	1	7 14 21	whole fruit	0.058 0.014 0.014	GRL-11346 RGC-98104
USA (PA) 1998, Red Delicious	500 WP	0.56	0.12	480	1	7 14 21	whole fruit	0.58 0.36 0.084	GRL-11346 RGC-98105
USA (PA) 1998, Law Rome	500 WP	0.56	0.12	480	1	7 14 21	whole fruit	0.20 0.13 0.074	GRL-11346 RGC-98112

APPLE	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/ 2/	
USA (GA) 1998, Golden Delicious	500 WP	0.55	0.12	470	1	7 14 21	whole fruit	0.16 0.052 0.086	GRL-11346 RCP-98105
USA (MI) 1998, Empire	500 WP	0.56	0.12	460	1	7 14 21	whole fruit	0.15 c 0.019 0.16 0.10	GRL-11346 JGC-98097
USA (MI) 1998, Red Max	500 WP	0.56	0.12	480	1	7 14 21	whole fruit	0.22 0.20 0.11	GRL-11346 JGC-98098
USA (CO) 1998, Golden Delicious	500 WP	0.56	0.12	460	1	7 14 21	whole fruit	0.23 c 0.068 0.20 0.016	GRL-11346 SWF-98101
USA (CA) 1998, Golden Delicious	500 WP	0.58	0.12	500	1	7 14 21	whole fruit	0.18 0.17 0.11	GRL-11346 CLS-98105
USA (WA) 1998, Red Delicious	500 WP	0.55	0.12	460	1	7 14 21	whole fruit	0.18 0.15 0.072	GRL-11346 DNJ-98102
USA (WA) 1998, Red Delicious	500 WP	0.56	0.12	470	1	7 14 21	whole fruit	0.37 0.15 0.17	GRL-11346 DNJ-98103
USA (OR) 1998, Jonagold	500 WP	0.54	0.12	470	1	7 14 21	whole fruit	0.17 c 0.01 0.13 0.078	GRL-11346 DNJ-98104
USA (OR) 1998, Gala	500 WP	0.55	0.12	470	1	7 14 21	whole fruit	0.049 0.017 0.024	GRL-11346 DNJ-98120
USA (NY) 1998, Empire	500 WP	0.56	0.12	470	1	3 7 14 20 30	whole fruit	0.11 0.19 0.13 0.13 0.15	GRL-11346 RGC-98103
USA (WA) 1998, Red Delicious	500 WP	0.56	0.12	470	1	3 7 14 21 28	whole fruit	0.48 0.38 0.36 0.25 0.22	GRL-11346 DNJ-98105
Japan, 1997 (Fuji)	200 SC	1.2	0.02	6000	4	7 14 21 28	whole fruit	0.28 0.43 0.11 0.13	Report No 6 Akita
Japan, 1997 (Fuji)	200 SC	1.2	0.02	6000	4	7 14 21 30	whole fruit	0.62 0.32 0.13 0.09	Report No 6 Nagano
Japan, 2003 (Tsugaru)	200 SC	1.2	0.02	6000	1	1 3 7	whole fruit	0.57 0.32 0.24	Report No 24 Iwate
Japan, 2003 (Tsugaru)	200 SC	1.0	0.02	5000	1	1 3 7	whole fruit	0.82 0.39 0.26	Report No 24 Fukushima

1/ mean of duplicate field samples (USA trials)

2/ c: sample from control plot.

Table 21. Bifenazate residues in pears resulting from supervised trials in USA and Japan.

PEAR	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/ 2/ 3/	
USA (NY) 1998, Bartlett	500 WP	0.55	0.12	470	1	7 14 21	whole fruit	0.10 0.036 0.025	GRL-11418 RGC-98108
USA (PA) 1998, Bartlett	500 WP	0.56	0.12	480	1	7 14 21	whole fruit	0.24 0.077 0.11	GRL-11418 RGC-98113
USA (CA) 1998, Bartlett	500 WP	0.55	0.13	440	1	7 14 21	whole fruit	0.14 0.034 0.025	GRL-11418 CLS-98106
USA (CA) 1998, Bartlett	500 WP	0.56	0.12	470	1	7 14 21	whole fruit	0.076 0.13 0.082	GRL-11418 CLS-98107
USA (WA) 1998, Bartlett	500 WP	0.54	0.12	450	1	7 14 21	whole fruit	0.16 0.12 c 0.014 0.12	GRL-11418 DNJ-98108
USA (WA) 1998, D'Anjou	500 WP	0.55	0.12	480	1	7 14 21	whole fruit	0.094 0.056 c 0.01 0.074	GRL-11418 DNJ-98109
USA (OR) 1998, Red Clapp	500 WP	0.55	0.12	460	1	7 14 21	whole fruit	0.097 c 0.01 0.095 0.043	GRL-11418 DNJ-98110
USA (WA) 1998, D'Anjou	500 WP	0.53	0.12	450	1	7 14 21	whole fruit	0.29 0.19 0.099	GRL-11418 DNJ-98121
Japan, 1998 (Housui)	200 SC	1.2	0.02	6000	4	7 14 21 28	whole fruit	0.45 0.36 0.11 0.12	Report No. 7 Nagano
Japan, 1998 (Kousui)	200 SC	1.2	0.02	6000	4	7 14 21 28	whole fruit	0.44 0.31 0.09 0.06	Report No. 7 Ooita
Japan, 2000 (Kousui)	200 SC	1.2	0.02	6000	1	1 3 7	whole fruit	0.42 0.26 0.32	Report No. 14 Nagano
Japan, 2000 (Kousui)	200 SC	1.2	0.02	6000	1	1 3 7	whole fruit	0.82 0.90 0.57	Report No. 14 Nagano
Japan, 2001 (Kousui)	200 SC	0.80	0.02	4000	1	1 3 7	whole fruit	0.54 0.34 0.28	Report No. 19 Fukushima
Japan, 2001 (Housui)	200 SC	0.40	0.02	2000	1	1 3 7	whole fruit	0.32 0.26 0.18	Report No. 19 Saitama
Japan, 2001 (Kousui)	200 SC	0.7	0.02	3500	1	1 3 7	whole fruit	0.56 0.50 0.15	Report No. 19 Ishikawa
Japan, 2001 (Kousui)	200 SC	1.0	0.02	5000	1	1 3 7	whole fruit	0.10 0.24 0.11	Report No. 19 Tokushima

1/ mean of duplicate field samples (USA trials)

2/ c: sample from control plot.

3/ Pear samples from the 8 US trials recorded in this table spent 15-16 months in frozen storage between harvest and analysis.

Table 22. Bifenazate residues in apricots resulting from supervised trials in USA.

APRICOT	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/	
USA (CA) 2002, Royal Blenheim	500 WP	0.56	0.12	460	1	1 3 7 14 21	whole fruit	0.49 0.36 0.44 0.349 0.21	CLS-02-102 RP-02007
USA (CA) 2002, Royal Rosa	500 WP	0.56	0.12	460	1	3	whole fruit	0.23	CLS-02-103 RP-02007
USA (CA) 2002, Royal Blenheim	500 WP	0.57	0.12	470	1	2	whole fruit	0.30	CLS-02-104 RP-02007
USA (WA) 2002, Rival	500 WP	0.57	0.12	480	1	3	whole fruit	0.73	DJN-02-101 RP-02007
USA (CA) 2002, Castlebright	500 WP	0.56	0.12	470	1	3	whole fruit	0.59	CEJ-02-101 RP-02007

1/ mean of duplicate field samples

Table 23. Bifenazate residues in peaches resulting from supervised trials in USA and Japan.

PEACH	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	2/	as bifenazate, mg/kg 1/	
USA (GA) 2002, Redskin	500 WP	0.56	0.12	470	1	3	whole fruit	0.44	KHG-02-101 RP-02007
USA (LA) 2002, Tex Royal	500 WP	0.56	0.12	480	1	3	whole fruit	0.45	AWD-02-901 RP-02007
USA (PA) 1998, Red Haven	500 WP	0.56	0.12	460	1	3 7 14	whole fruit	0.55 0.33 0.19	RGC-98-500 RP-02007 RP-98006
USA (SC) 1998, Harvesters	500 WP	0.55	0.12	480	1	3 7 14	whole fruit	0.23 0.14 0.12	RCP-98-102 RP-02007 RP-98006
USA (SC) 1998, Contender	500 WP	0.55	0.11	490	1	3 7 14	whole fruit	0.23 0.18 0.14	RCP-98-103 RP-02007 RP-98006
USA (GA) 1998, Redskin	500 WP	0.56	0.12	470	1	3 7 14	whole fruit	0.17 0.11 0.06	RCP-98-104 RP-02007 RP-98006
USA (MI) 1998, Red Haven	500 WP	0.55	0.12	450	1	3 7 14	whole fruit	0.22 0.19 0.05	JGC-98-099 RP-02007 RP-98006
USA (TX) 1998, June Gold	500 WP	0.56	0.12	480	1	3 7 14	whole fruit	1.2 1.0 0.73	AWD-98-202 RP-02007 RP-98006

PEACH	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	2/	as bifenazate, mg/kg 1/	
USA (CA) 1998, Carnival	500 WP	0.57	0.12	470	1	1	whole fruit	0.46	CEJ-98-108
						3		0.40	RP-02007
						7		0.26	RP-98006
						14		0.17	
						21		0.19	
USA (CA) 1998, O'Henry	500 WP	0.57	0.12	480	1	3	whole fruit	0.15	CEJ-98-109
						7		0.16	RP-02007
						14		0.10	RP-98006
USA (CA) 1998, Yodel	500 WP	0.57	0.13	440	1	3	whole fruit	0.13	CLS-98-110
						7		0.10	RP-02007
						14		0.03	RP-98006
USA (CA) 1998, Loadel	500 WP	0.56	0.12	470	1	4	whole fruit	0.26	CLS-98-111
						7		0.12	RP-02007
						14		0.09	RP-98006
Japan, 1998 (Akatsuki)	200 SC	0.80	0.02	4000	4	7	pulp	0.02	Report No. 8
						14	pulp	0.02	Fukushima
						21	pulp	< 0.02	
						28	pulp	< 0.02	
Japan, 1998 (Hakuhou)	200 SC	1.2	0.02	6000	4	7	pulp	< 0.02	Report No. 8
						14	pulp	< 0.02	Tokushima
						21	pulp	0.02	
						28	pulp	< 0.02	
Japan, 2003 (Akatsuki)	200 SC	0.8	0.02	4000	1	1	pu, pe	< 0.02, 8.8	Report No. 25 & 26
						3	pu, pe	< 0.02, 9.7	Fukushima
						7	pu, pe	< 0.02, 3.4	
Japan, 2003 (Akatsuki)	200 SC	1.4	0.02	7000	1	1	pu, pe	< 0.02, 6.9	Report No. 25 & 26
						3	pu, pe	< 0.02, 6.0	Nagano
						7	pu, pe	< 0.02, 3.8	

1/ mean of duplicate field samples for US trials.

2/ pu: pulp; pe: peel.

Table 24. Bifenazate residues in plums resulting from supervised trials in USA and Japan.

PLUMS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/	
USA (CA) 2002, Fortune	500 WP	0.56	0.12	470	1	3	whole fruit	0.034	CEJ-02-105 RP-02007
USA (MI) 1998, Stanley	500 WP	0.56	0.12	460	1	3	whole fruit	0.13	JGC-98-100
						7		0.08	RP-02007
						14		0.05	RP-98006
USA (CA) 1998, Fortune	500 WP	0.57	0.12	480	1	4	whole fruit	0.01	CEJ-98-110
						7		< 0.01	RP-02007
						14		< 0.01	RP-98006
USA (CA) 1998, Simka	500 WP	0.56	0.12	470	1	3	whole fruit	0.04	CEJ-98-111
						7		0.02	RP-02007
						14		0.01	RP-98006
USA (CA) 1998, French prune	500 WP	0.56	0.12	470	1	1	whole fruit	0.07	CLS-98-109
						3		0.06	RP-02007
						7		0.04	RP-98006
						14		0.04	
						21		0.02	

PLUMS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/	
USA (CA) 1998, Moyer	500 WP	0.56	0.13	450	1	3	whole fruit	0.04	CLS-98-108 RP-02007 RP-98006
						7		0.04	
						14		0.02	
USA (OR) 1998, Parsons	500 WP	0.56	0.12	470	1	1	whole fruit	0.04	DNJ-98-112 RP-02007 RP-98006
						3		0.03	
						7		0.02	
						14		0.02	
						21		0.01	
USA (WA) 1998, Friar	500 WP	0.57	0.12	470	1	3	whole fruit	0.04	DNJ-98-111 RP-02007 RP-98006
						7		0.03	
						14		0.01	
Japan, 2001 (Ooishiwase)	SC 200	0.80	0.02	4000	2	3	whole fruit	0.14	Report No. 22 Fukushima
						7		0.20	
						14		0.04	
Japan, 2001 (Ooishiwase)	SC 200	1.0	0.02	5000	2	3	whole fruit	0.05	Report No. 22 Nagano
						7		0.14	
						14		0.06	

1/ mean of duplicate field samples for US trials.

Table 25.Bifenazate residues in cherries resulting from supervised trials in USA and Japan.

CHERRIES	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/	
USA (CA) 2001, Kings	500 WP	0.57	0.03	1640	1	3	pitted fruits	0.29	07054.01-CA51
USA (CA) 2001, Brooks	500 WP	0.56	0.06	940	1	4	pitted fruits	0.23	07054.01-CA93
USA (CO) 2001, Montmorency	500 WP	0.55	0.04	1240	1	3	pitted fruits	1.6	07054.01-CO12
USA (ID) 2001, Lambert	500 WP	0.56	0.06	950	1	3	pitted fruits	0.11	07054.01-ID09
USA (ID) 2001, Montmorency	500 WP	0.56	0.06	930	1	3	pitted fruits	0.48	07054.01-ID10
USA (MI) 2001, Cavalier	500 WP	0.57	0.10	570	1	3	pitted fruits	0.20	07054.01-MI20
USA (MI) 2001, Emperor Francis	500 WP	0.58	0.10	580	1	3	pitted fruits	0.42	07054.01-MI21
USA (MI) 2001, Montmorency	500 WP	0.56	0.06	940	1	3	pitted fruits	0.89	07054.01-MI22
USA (MI) 2001, Montmorency	500 WP	0.55	0.06	930	1	3	pitted fruits	0.71	07054.01-MI23
USA (MI) 2001, Montmorency	500 WP	0.57	0.06	950	1	3	pitted fruits	1.2	07054.01-MI24

CHERRIES	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (MI) 2001, Montmorency	500 WP	0.56	0.05	930	1	3	pitted fruits	0.81	07054.01-MI25
USA (NJ) 2001, Montmorency	500 WP	0.57	0.06	1020	1	4	pitted fruits	0.18	07054.01-NJ17
USA (OR) 2001, Bing	500 WP	0.57	0.05	1170	1	4	pitted fruits	0.27	07054.01-OR13
USA (WA) 2001, Bing	500 WP	0.57	0.04	1420	1	3	pitted fruits	0.34	07054.01-WA22
Japan, 1998 (Koukanishiki)	200 SC	1.2	0.02	6000	4	14 21 28 42	whole fruits	0.17 0.21 0.03 0.02	Report No 9 Iwate
Japan, 1998 (Satonishiki)	200 SC	1.2	0.02	6000	4	14 21 28 42	whole fruits	0.45 0.30 0.06 0.20	Report No 9 Fukushima

1/ mean of duplicate field samples for US trials.

Table 26. Bifenazate residues in grapes resulting from supervised trials in USA and Japan.

GRAPES	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (NY) 1998, Seyval Blanc	500 WP	0.57	0.12	470	1	14 21	whole fruit	0.31 0.17	7545 RGC-98-110
USA (NY) 1998, Concord	500 WP	0.56	0.12	470	1	14 21	whole fruit	0.11 0.08	7545 RGC-98-111
USA (CA) 1998, Flame Seedless	500 WP	0.56	0.12	470	1	14 21	whole fruit	0.10 0.10	7545 CEJ-98-112
USA (CA) 1998, Thomson seedless	500 WP	0.57	0.12	470	1	14 21	whole fruit	0.07 0.06	7545 CEJ-98-113
USA (CA) 1998, Chenin Blanc	500 WP	0.58	0.13	450	1	14 21	whole fruit	0.05 0.02	7545 CEJ-98-114
USA (CA) 1998, Carigane	500 WP	0.58	0.12	470	1	14 21	whole fruit	0.33 0.17	7545 CLS-98-112
USA (CA) 1998, Palomino	500 WP	0.56	0.12	470	1	14 21	whole fruit	0.20 0.07	7545 CLS-98-113
USA (CA) 1998, Sauvignon Blanc	500 WP	0.58	0.12	480	1	14 21	whole fruit	0.21 0.19	7545 CLS-98-114

GRAPES	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (CA) 1998, Cabernet Sauvignon	500 WP	0.62	0.12	500	1	14 21	whole fruit	0.17 0.14	7545 CLS-98-115
USA (CA) 1998, Thomson seedless	500 WP	0.56	0.12	470	1	14 21	whole fruit	0.55 0.48	7545 CLS-98-116
USA (WA) 1998, Riesling	500 WP	0.55	0.12	460	1	14 21	whole fruit	0.17 0.15 c 0.06	7545 DNJ-98-118
USA (WA) 1998, White Riesling	500 WP	0.56	0.12	470	1	14 21	whole fruit	0.29 0.21	7545 DNJ-98-119
USA (CA) 1998, Thomson Seedless	500 WP	2.8		470	1	3 7 14 21 28	whole fruit whole fruit whole fruit whole fruit whole fruit	1.05 0.61 0.30 0.30 0.23	RP-98007 CEJ-98-115
USA (CA) 1998, Thomson Seedless	500 WP	2.9		480	1	3 7 14 21 28	whole fruit whole fruit whole fruit whole fruit whole fruit	1.1 0.72 0.19 0.08 0.12	RP-98007 CEJ-98-116
Japan, 1997 (Delaware) indoor	SC 200	0.80	0.02	4000	3	7 14 21 30 45	whole fruit	1.7 1.0 0.65 1.0 1.2	Report No. 17 Akita
Japan, 1997 (Kyohou) indoor	SC 200	0.80	0.02	4000	3	7 14 21 30 44	whole fruit	0.46 0.34 0.34 0.47 0.14	Report No. 17 Saitama
Japan, 1999 (Kyohou) indoor	SC 200	0.80	0.02	4000	3	14 21 28 42	whole fruit	0.16 0.13 0.15 0.19	Report No. 18 Nagano
Japan, 1999 (Delaware) indoor	SC 200	0.80	0.02	4000	3	14 21 28 42	whole fruit	2.2 1.1 0.88 0.68	Report No. 18 Ishikawa

1/ mean of duplicate field samples for US trials.

2/ c: sample from control plot.

Table 27. Bifenazate residues in strawberries resulting from supervised trials in USA and Japan.

STRAWBERRY	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (PA) 1999, Northeaster	500 WP	0.56	0.06	940	2	1 3	whole fruit	0.68 0.43	GRL-11517 RGC-99001
USA (GA) 1999, Chandler	500 WP	0.55	0.06	910	2	1 3	whole fruit	0.93 0.81	GRL-11517 RCP-99100
USA (FL) 1999, Camarosa	500 WP	0.61	0.04	1630	2	1 3	whole fruit	0.49 0.44	GRL-11517 PAK-99003
USA (IN) 1999, Tribute	500 WP	0.56	0.06	940	2	1 3	whole fruit	1.0 0.41	GRL-11517 JGC-99001
USA (CA) 1999, Selva	500 WP	0.54	0.06	900	2	1 3	whole fruit	0.63 0.62	GRL-11517 CLS-99101
USA (CA) 1999, PS-952	500 WP	0.55	0.06	920	2	1 3	whole fruit	0.23 0.29	GRL-11517 CLS-99102
USA (CA) 1999, Camarosa	500 WP	0.56	0.06	930	2	1 3	whole fruit	0.44 3.2 3/	GRL-11517 CEJ-99101
USA (OR) 1999, Totem	500 WP	0.56	0.06	940	2	1 3	whole fruit	0.53 0.24	GRL-11517 DNJ-99101
Japan, 1998 (Nyohou) indoor	200 SC	0.40	0.02	2000	1	1 3 7	whole fruit	0.89 0.53 0.36	Report No. 10 Gunma
Japan, 1998 (Nyohou) indoor	200 SC	0.50	0.02	2500	1	1 3 7	whole fruit	0.82 1.10 0.33	Report No. 10 Mie
Japan, 2002 (Nyohou) indoor	200 SC	0.50	0.02	2500	2	1 3 7	whole fruit	2.0 1.1 0.67	Report No. 23 Gifu
Japan, 2002 (Akihime) indoor	200 SC	0.50	0.02	2500	2	1 3 7	whole fruit	0.38 0.19 0.15	Report No. 23 Mie
Japan, 2002 (Nyohou) indoor	150 FT	100 g/ 400 m3 ~ 1.1 kg/ha			2	1 3 7	whole fruit	0.24 0.10 < 0.05	Report No. 23 Gifu
Japan, 2002 (Akihime) indoor	150 FT	100 g/ 400 m3 ~ 1.1 kg/ha			2	1 3 7	whole fruit	0.05 < 0.05 < 0.05	Report No. 23 Mie

1/ mean of duplicate field samples for US trials.

2/ c: sample from control plot.

3/ The two values at the 3-days PHI in report CEJ-99101 were 3.4 and 2.9 mg/kg. The authors of the report discounted the values as being due to analytical error, based on the reasons that the values were 7-8 times as high as in the other trials and also much higher than the 1-day sample from the same trial.

Table 28. Bifenazate residues in figs resulting from supervised trials in Japan.

FIGS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
Japan, 2003 (Masui-Dofin)	SC 200	0.60	0.02	3000	1	1 3 7	whole fruit	0.53 0.22 0.10	Report No. 27 Aichi, Anjo
Japan, 2003 (Masui-Dofin)	SC 200	0.60	0.02	3000	1	1 3 7	whole fruit	0.55 0.30 0.14	Report No. 27 Aichi, Nagakute

Table 29.Bifenazate residues in cantaloupe resulting from supervised trials in Canada, USA and Japan.

CANTALOUPE	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (TX) 2000, Hy-Mark	500 WP	0.57	0.18	310	1	1 3 7	fruit	0.31 0.10 0.05	07510.00-TX27
USA (GA) 2000, Vienna	500 WP	0.56	0.20	290	1	4	fruit	0.03	07510.00-GA15
USA (TX) 2000, Primo	500 WP	0.55	0.17	320	1	2	fruit	0.16 c 0.01	07510.00-TX28
USA (CA) 2000, Hymark	500 WP	0.54	0.18	300	1	3	fruit	0.04	07510.00-CA44
USA (CA) 2000, Sol Real	500 WP	0.54	0.18	300	1	4	fruit	0.04	07510.00-CA45
USA (WI) 2000, Super Star	500 WP	0.58	0.20	300	1	3	fruit	0.05	07510.00-WI04
USA (CA) 2000, Ambrosia	500 WP	0.57	0.28	200	1	3	fruit	0.04	07510.00-CA43
USA (NJ) 2000, Ambrosiaar	500 WP	0.57	0.14	400	1	3	fruit	0.08	07510.00-NJ35
Japan, 1999 (Natsukei2gou) indoor	200 SC	0.40	0.02	2000	2	1 3 7 14	pulp pulp pulp pulp	0.04 < 0.02 < 0.02 < 0.02	Report No. 16 Nagano
Japan, 1999 (Aruseinu) indoor	200 SC	0.40	0.02	2000	2	1 3 7 14	pulp pulp pulp pulp	< 0.02 < 0.02 < 0.02 < 0.02	Report No. 16 Ishikawa

1/ mean of duplicate field samples for trials in USA and Canada.

2/ c: sample from control plot.

Table 30. Bifenazate residues in watermelon resulting from supervised trials in Japan.

WATERMELON	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
Japan, 1998 (Benikodam) indoor	200 SC	0.40	0.02	2000	3	1	pulp	0.03	Report No. 11 Ishikawa.
						3	pulp	0.02	
						7	pulp	< 0.02	
						14	pulp	< 0.02	
						21	pulp	< 0.02	
Japan, 1998 (Madabowl-2gou) indoor	200 SC	0.40	0.02	2000	3	1	pulp	0.02	Report No. 11 Miyazaki..
						3	pulp	0.02	
						7	pulp	< 0.02	
						14	pulp	< 0.02	
						21	pulp	< 0.02	

Table 31. Bifenazate residues in cucumber resulting from supervised trials in Canada, USA and Japan.

CUCUMBER	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (MD) 2000, Regal (pickling cucumber)	500 WP	0.55	0.12	470	1	3	fruit	< 0.01	07511.00-MD09
USA (NJ) 2000, Dasher II	500 WP	0.56	0.14	400	1	3	fruit	0.22	07511.00-NJ20
USA (FL) 2000, Sawan Slicer Cucumber	500 WP	0.57	0.14	400	1	3	fruit	0.08	07511.00-FL44
USA (GA) 2000, Thunder, Hybrid Cucumber	500 WP	0.56	0.20	290	1	3	fruit	< 0.01	07511.00-GA16
USA (TX) 2000, Calypso pickling	500 WP	0.56	0.25	220	1	3	fruit	0.03	07511.00-TX29
Canada (ON), 2000, Dasher II	500 WP	0.55	0.28	200	1	2	fruit	0.07	07511.00-ON04
Canada (ON), 2000, Dasher II	500 WP	0.52	0.15	350	1	3	fruit	0.05	07511.00-ON03
USA (CA) 2000, Thunder cucumber	500 WP	0.54	0.18	300	1	3	fruit	0.04	07511.00-CA73
Japan, 2001 (Haruka) indoor	SC 200	0.50	0.02	2500	1	1	fruit	0.12	Report No. 20 Nagano
						3		0.05	
						7		< 0.01	

CUCUMBER	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
Japan, 2001 (Suiseisessei-2gou) indoor	SC 200	0.61	0.02	3040	1	1 3 7	fruit	0.14 0.08 < 0.01	Report No. 20 Miyazaki

1/ mean of duplicate field samples for US trials.

Table 32. Bifenazate residues in summer squash resulting from supervised trials in USA.

SUMMER SQUASH	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (NY) 2000, Yellow Crookneck	500 WP	0.54	0.13	410	1	4	fruit	0.04	07512.00-NY15
USA (NJ) 2000, Early Yellow Straightneck	500 WP	0.56	0.14	390	1	3	fruit	0.34	07512.00-NJ21
USA (FL) 2000, Gentry CS4 hybrid squash	500 WP	0.56	0.20	280	1	3	fruit	0.12	07512.00-FL45
USA (MD) 2000, Puma	500 WP	0.55	0.12	475	1	3	fruit	< 0.01	07512.00-MD02
USA (TX) 2000, Senator	500 WP	0.57	0.25	224	1	3	fruit	0.06	07512.00-TX30
USA (CA) 2000, Patty Groon Tint	500 WP	0.54	0.27	200	1	3	fruit	0.02	07512.00-CA46
USA (IN) 2000, Aristocrat	500 WP	0.56	0.24	230	1	3	fruit	0.01	07512.00-IN03

1/ mean of duplicate field samples

Table 33. Bifenazate residues in tomatoes resulting from supervised trials in Canada, USA and Japan.

TOMATOES	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (FL), 2000, FL47	500 WP	0.56	0.12	470	1	3	fruit	0.19	07266.00-FL42 GRL-FR-11673
USA (FL), 2000, Celebrity	500 WP	0.56	0.12	470	1	3	fruit	0.14	07266.00-FL43 GRL-FR-11673
USA (GA), 2000, Mountain Springs	500 WP	0.57	0.20	290	1	1 2 7 14	fruit	0.27 0.11 0.03 0.02	07266.00-GA14 GRL-FR-11673

TOMATOES	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (OH), 2000, Hypeel 696	500 WP	0.56	0.14	410	1	2	fruit	0.10	07266.00-OH12 GRL-FR-11673
USA (CA), 2000, Rio Grande	500 WP	0.54	0.18	300	1	3	fruit	0.07	07266.00-CA39 GRL-FR-11673
USA (CA), 2000, Yaqui	500 WP	0.54	0.18	300	1	3	fruit	0.03	07266.00-CA40 GRL-FR-11673
USA (CA), 2000, Celebrity	500 WP	0.58	0.16	370	1	3	fruit	0.03	07266.00-CA159 GRL-FR-11673
USA (CA), 2000, H9553	500 WP	0.58	0.16	360	1	3	fruit paste puree 2/	0.09 0.11 0.49	07266.00-CA41 GRL-FR-11673
USA (CA), 2000, Shady Lady	500 WP	0.58	0.15	380	1	3	fruit	0.04	07266.00-CA42 GRL-FR-11673
USA (CA), 2000, 3135 VF	500 WP	0.55	0.14	380	1	3	fruit	0.13	07266.00-CA107 GRL-FR-11673
USA (NJ), 2000, FT 4010	500 WP	0.57	0.14	400	1	3	fruit	0.04	07266.00-NJ19 GRL-FR-11673
USA (TX), 2001, Better Boy greenhouse	SC	0.56		960	1	0	fruit	0.21	08035-01-TX-03 GRL 11805
USA (NJ), 2001, Floralina greenhouse	SC	1.1		1140	1	0 0	fruit washed fruit	0.44 0.15	08035-01-NJ-04 GRL 11805
USA (TN), 2001, Celebrity greenhouse	SC	0.56		1400	1	0	fruit	0.16	08035-01-TN-02 GRL 11805
Canada (Ontario), 2000, 9478	500 WP	0.52	0.15	350	1	3	fruit	0.29	07266.00-ON02 GRL-FR-11673
Canada (Quebec), 2000, Aclaim	500 WP	0.20	0.025	800	1	3	fruit	0.02	07266.00-QC02 GRL-FR-11673
Japan, 2001 (House-Momotarou) indoor	SC 200	0.50	0.02	2500	1	1 7 14	fruit	0.32 0.11 0.10	Report No. 21 Ibaragi
Japan, 2001 (Rokusanmaru) indoor	SC 200	0.50	0.02	2500	1	1 7 14	fruit	0.11 0.07 0.03	Report No. 21 Nagano

1/ mean of duplicate field samples for US and Canadian trials.

2/ Processing factors: tomato paste 1.26 ; tomato puree 5.6.

Table 34. Bifenazate residues in bell peppers resulting from supervised trials in Canada and USA.

PEPPERS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (FL), 2000, Camelot – bell pepper	500 WP	0.56	0.20	280	1	3	fruit	1.1	07552.00-FL41 GRL-FR-11660
USA (GA), 2000, Ceystone – bell pepper	500 WP	0.56	0.12	480	1	2	fruit	0.52 c 0.02	07552.00-GA13 GRL-FR-11660
USA (OH), 2000, King Arthur – bell pepper	500 WP	0.57	0.14	420	1	2	fruit	0.15	07552.00-OH11 GRL-FR-11660
USA (MD), 2000, Boynton Bell – bell pepper	500 WP	0.56	0.12	480	1	3	fruit	0.32	07552.00-MD13 GRL-FR-11660
USA (CA), 2000, Ivan – bell pepper	500 WP	0.54	0.19	290	1	3	fruit	0.24	07552.00-CA37 GRL-FR-11660
USA (CA), 2000, Jupiter – bell pepper	500 WP	0.55	0.18	310	1	3	fruit	0.15	07552.00-CA38 GRL-FR-11660
Canada (Ontario), 2000, King Arthur – bell pepper	500 WP	0.61	0.20	310	1	3	fruit	0.23	07552.00-ON01 GRL-FR-11660
USA (TX), 2000, Capistrano – bell pepper	500 WP	0.55	0.12	450	1	3	fruit	0.13	07552.00-TX32 GRL-FR-11660
USA (TX), 2000, Tam Veracruz – Jalopeno non-bell pepper	500 WP	0.56	0.13	420	1	3	fruit	1.1 c 0.08	07552.00-TX52 GRL-FR-11660
USA (FL), 2000, Mesilla - non-bell pepper	500 WP	0.56	0.20	280	1	3	fruit	1.6 c 0.01	07552.00-FL40 GRL-FR-11660
USA (NM), 2000, Sandia - non-bell pepper	500 WP	0.54	0.17	320	1	3	fruit	0.54 c 0.03	07552.00-NM16 GRL-FR-11660

1/ mean of duplicate field samples.

2/ c: sample from control plot.

Table 35. Bifenazate residues in eggplant resulting from supervised trials in Japan.

EGGPLANT	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
Japan, 1997 (Kokuyou) indoor	200 SC	0.40	0.02	2000	1	1 3 7	whole fruit	0.41 0.33 0.09	Report No. 15 Ibaragi
Japan, 2000 (Itifuji) indoor	200 SC	0.40	0.02	2000	1	1 3 7	whole fruit	0.35 0.16 0.02	Report No. 15 Gunma

Table 36. Bifenazate residues in tree nuts resulting from supervised trials in USA.

TREE NUTS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
ALMONDS									
USA (CA), 2001, NonPareil	500 WP	0.84	0.18	470	1	3 7	kernel kernel	0.07 0.05	RP-01002 CEJ-01-101
USA (CA), 2001, NonPareil	500 WP	0.84	0.18	460	1	3 7	kernel kernel	0.04 0.03	RP-01002 CEJ-01-102
USA (CA), 2001, Mission	500 WP	0.85	0.18	470	1	3 7	kernel kernel	0.01 0.01	RP-01002 CEJ-01-103
USA (CA), 2001, Peerless	500 WP	0.85	0.18	470	1	3 7	kernel kernel	0.04 3/ 0.02 4//	RP-01002 CLS-01-101
USA (CA), 2001, Butte	500 WP	0.84	0.17	490	1	3 7	kernel kernel	0.11 c 0.025 0.10	RP-01002 CLS-01-102
PECANS									
USA (LA), 2001, Elliot, Summer and Kiowa	500 WP	0.85	0.18	480	1	14	kernel	< 0.01	RP-01001 AWD-01-901
USA (OK), 2001, Natives	500 WP	0.84	0.17	490	1	15	kernel	< 0.01	RP-01001 AWD-01-902
USA (NM), 2001, Western Schley	500 WP	0.89	0.18	500	1	14	kernel	0.014	RP-01001 SWG-01-001
USA (GA), 2001, Stewart	500 WP	0.85	0.19	460	1	14	kernel	0.013	RP-01001 KHG-01-001
USA (GA), 2001, Cape Fear	500 WP	0.84	0.18	470	1	14	kernel	< 0.01	RP-01001 KHG-01-002

1/ mean of 3 replicate field samples.

2/ c: sample from control plot.

3/ Almonds were dried for 9 days before hulling and shelling.

4/ Almonds were dried for 7 days before hulling and shelling.

Note: LOQ for almond hulls was 0.2 mg/kg.

Table 37. Bifenazate residues in cotton seed resulting from supervised trials on cotton in USA.

COTTON SEED	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/ 2/	
USA (SC) 2000, DP 458	500 WP	0.84	0.45	190	1	60	cotton seed	< 0.01	012351 RCP-00-001
USA (MI) 2000, DP 20B	500 WP	0.84	0.44	190	1	60	cotton seed	0.02	012351 RDH-00-101
USA (TX) 2000, Stoneville 489 2BRB	500 WP	0.83	0.46	180	1	60	cotton seed	< 0.01	012351 AWD-00-901
USA (NM) 2000, Acala 1517-95	500 WP	0.83	0.43	190	1	60	cotton seed	0.03	012351 SWF-00-204
USA (NM) 2000, Acala 1517-95	500 WP	0.84	0.44	190	1	60	cotton seed	0.06	012351 SWF-00-305
USA (TX) 2000, Paymaster HS2326	500 WP	0.84	0.46	180	1	65	cotton seed	0.04	012351 SWF-00-406
USA (CA) 2000, Acala Maxxa	500 WP	0.83	0.45	190	1	60	cotton seed	< 0.01	012351 CEJ-00-105
USA (CA) 2000, Acala Riata RR	500 WP	0.86	0.45	190	1	61	cotton seed	0.03	012351 CEJ-00-106
USA (SC) 1999, DP 458	500 WP	0.85	0.45	190	1	61	cotton seed	< 0.01	RP-99008 RCP-99-101
USA (MI) 1999, DPL 20B	500 WP	0.82	0.44	190	1	59	cotton seed	< 0.01	RP-99008 RDH-99-001
USA (LA) 1999, ST474	500 WP	0.84	0.44	190	1	60	cotton seed	< 0.01	RP-99008 AWD-99-903
USA (LA) 1999, DPL 458	500 WP	0.85	0.46	180	1	60	cotton seed	< 0.01	RP-99008 AWD-99-904
USA (OK) 1999, PM 145	500 WP	0.84	0.45	190	1	60	cotton seed	< 0.01	RP-99008 AWD-99-906
USA (TX) 1999, PM 2200RR	500 WP	0.85	0.45	190	1	59	cotton seed	< 0.01	RP-99008 SWF-99-801
USA (TX) 1999, Excess	500 WP	0.84	0.45	190	1	42	cotton seed	0.02	RP-99008 SWF-99-901
USA (TX) 1999, DP 2156	500 WP	0.84	0.45	190	1	59	cotton seed	0.01	RP-99008 SWF-99-10A
USA (NM) 1999, Acala 1517-95	500 WP	0.84	0.44	190	1	60	cotton seed	0.06	RP-99008 SWF-99-20A

COTTON SEED	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (NM) 1999, Acala 1517-95	500 WP	5.3			1	60	cotton seed meal refined oil	1.05 < 0.01 < 0.01	RP-99008 SWF-99-20A
USA (CA) 1999, CB 232	500 WP	0.82	0.45	180	1	61	cotton seed	< 0.01	RP-99008 CLS-99-109
USA (CA) 1999, Acala Maxxa	500 WP	0.85	0.45	190	1	60	cotton seed	0.04	RP-99008 CEJ-99-105
USA (CA) 1999, GC 500	500 WP	0.84	0.44	190	1	61	cotton seed	0.28	RP-99008 CEJ-99-106
USA (CA) 1999, GC 500	500 WP	5.3			1	60	cotton seed meal refined oil	2.64 < 0.01 < 0.01	RP-99008 CEJ-99-106

1/ mean of duplicate field samples.

2/ c: sample from control plot.

Table 38. Bifenazate residues in mint resulting from supervised trials in USA.

MINT	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/ 2/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (WA), 2000, Native spearmint	500 WP	0.82	0.17	480	1	8	mint tops	15.4	07386.00-WA49 GRL-FR-11712
USA (WI), 2000, Scotch spearmint, H. Darling strain	500 WP	0.84	0.27	310	1	8	mint tops	12.9	07386.00-WI21 GRL-FR-11712
USA (WI), 2000, Black Mitchem peppermint	500 WP	0.84	0.28	310	1	8	mint tops	18.1	07386.00-WI22 GRL-FR-11712
USA (WA), 2000, Scotch spearmint	500 WP	0.83	0.19	440	1	7	mint tops	6.6	07386.00-WA20 GRL-FR-11712
USA (WA), 2000, Native mint	500 WP	0.84	0.19	440	1	7	mint tops	6.4 c 0.01	07386.00-WA21 GRL-FR-11712
USA (WA), 2001, Spearmint (Native)	500 WP	1.8		290	1	7 7	mint tops mint oil	20 1.6 0.90 0.97 3/	RP-01015 DNJ-01-101

1/ mean of duplicate field samples.

2/ c: sample from control plot.

3/ 3 processing runs.

Table 39. Bifenazate residues in hops resulting from supervised trials in USA.

HOPS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	2/	1/	
USA (WA) 1999, Nugget	500 WP	0.85	0.18	470	1	13	dried hops	9.3	RP-99006 DNJ-99-103
USA (WA) 1999, Nugget	500 WP	0.85	0.18	470	1	14	dried hops	7.8	RP-99006 DNJ-99-104
USA (OR) 1999, Williamette	500 WP	0.85	0.18	470	1	14	dried hops	7.1	RP-99006 DNJ-99-105

1/ mean of 3 replicate field samples.

2/ Samples of green hops from the field were taken to commercial hop dryers for drying at 52 °C and, one day later, they were removed as approximately 1 kg of dried hops.

Table 40. Bifenazate residues in tea resulting from supervised trials in Japan.

TEA	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days	1/		
Japan, 1998 (Ooiwase)	200 SC	0.80	0.02	4000	3	7 14 21 7 14 21	leaves leaves leaves tea extract tea extract tea extract	6.5 0.82 < 0.1 1.6 0.22 < 0.1	Report No. 12 and 13 Shizuoka
Japan, 1998 (Yabukita)	200 SC	0.80	0.02	4000	3	7 14 20 7 14 20	leaves leaves leaves tea extract tea extract tea extract	24 0.53 0.10 5.4 0.14 < 0.1	Report No. 12 and 13 Fukuoka

1/ Tea extract: Tea leaves (6 g) were mixed with 360 ml of distilled water at 100 °C for 5 minutes. After filtration, the tea extract was analysed.

Table 41. Bifenazate residues in feed commodities resulting from supervised trials in USA.

FEED	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		1/ 2/	
ALMOND HULLS									
USA (CA), 2001, NonPareil	500 WP	0.84	0.18	470	1	3 7	hull hull	4.4 5.0	RP-01002 CEJ-01-101
USA (CA), 2001, NonPareil	500 WP	0.84	0.18	460	1	3 7	hull hull	7.3 6.9	RP-01002 CEJ-01-102

FEED	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/ 2/	
USA (CA), 2001, Mission	500 WP	0.85	0.18	470	1	3 7	hull hull	5.0 2.8	RP-01002 CEJ-01-103
USA (CA), 2001, Peerless	500 WP	0.85	0.18	470	1	3 7	hull hull	3.9 3/ 1.8 4/	RP-01002 CLS-01-101
USA (CA), 2001, Butte	500 WP	0.84	0.17	490	1	3 7	hull hull	6.0 5.1	RP-01002 CLS-01-102
COTTON GIN TRASH									
USA (NM) 2000, Acala 1517-95	500 WP	0.84	0.44	190	1	60	gin trash	3.8	012351 SWF-00-305
USA (TX) 2000, Paymaster HS2326	500 WP	0.84	0.46	180	1	65	gin trash	0.69	012351 SWF-00-406
USA (CA) 2000, Acala Riata RR	500 WP	0.86	0.45	190	1	61	gin trash	2.5 c 0.02	012351 CEJ-00-106
USA (SC) 1999, DP 458	500 WP	0.85	0.45	190	1	61	gin trash	0.88	RP-99008 RCP-99-101
USA (LA) 1999, DPL 458	500 WP	0.85	0.46	180	1	60	gin trash	1.3	RP-99008 AWD-99-904
USA (TX) 1999, PM 2200RR	500 WP	0.85	0.45	190	1	59	gin trash	0.07	RP-99008 SWF-99-801
USA (TX) 1999, Excess	500 WP	0.84	0.45	190	1	42	gin trash	0.46	RP-99008 SWF-99-901
USA (TX) 1999, DP 2156	500 WP	0.84	0.45	190	1	59	gin trash	0.39	RP-99008 SWF-99-10A
USA (NM) 1999, Acala 1517-95	500 WP	0.84	0.44	190	1	60	gin trash	4.0	RP-99008 SWF-99-20A
USA (NM) 1999, Acala 1517-95	500 WP	5.3			1	60	gin trash	30	RP-99008 SWF-99-20A
USA (CA) 1999, GC 500	500 WP	0.84	0.44	190	1	61	gin trash	18	RP-99008 CEJ-99-106
USA (CA) 1999, GC 500	500 WP	5.3			1	60	gin trash	110	RP-99008 CEJ-99-106

1/ mean of duplicate samples

2/ c: sample from control plot.

3/ Almonds were dried for 9 days before hulling and shelling.

4/ Almonds were dried for 7 days before hulling and shelling.

Note: LOQ for almond hulls was 0.2 mg/kg.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on the fate of bifenazate residues during the juicing of apples, the drying of prunes, the production of grape juice and raisins, the production of tomato paste and puree, the production of cotton seed oil and the processing of mint tops.

Gaydosh (2000, GRL-11419) processed apples, from two bifenazate field trials with exaggerated (5×) application rates, into juice and wet pomace using a small-scale process. The process was suitable for 20–50 kg of apples. Apples were ground in a hammer-mill and the wet mash was collected into clothsacks on a hydraulic press. The sack was pressed at 2200–3000 psi for a minimum of 5 minutes and juice was collected. The wet pomace cake within the bags was sampled as wet pomace. Residues were measured on whole fruit, juice and wet pomace (Table 42).

Table 42. Bifenazate residues in apple juice and wet pomace from processing trials in USA (Gaydosh, 2000, GRL-11419).

APPLE	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/ 2/	
USA (NY) 1998, Idared	500 WP	2.8		470	1	7 7	whole fruit whole fruit juice wet pomace	1.3 3/ 0.89 4/ 0.20 c 0.14 1.6	RGC-98107 GRL-11419
USA (WA) 1998, Red Delicious	500 WP	2.8		460	1	7 7	whole fruit whole fruit juice wet pomace	2.0 3/ 2.1 4/ 0.22 c 0.14 3.6	DNJ-98107 GRL-11419

1/ mean of duplicate samples

2/ c control juice from untreated apples. On a second analysis of these juice samples, no residue was detected (< 0.005 mg/kg).

3/ field samples taken at the same time as the fruit for processing

4/ sampled at the processing laboratory

Korpalski and Puhl (2000, RP-98006) described the processing to dried prunes of plums treated in the field with bifenazate (Table 43). At each of two sites plums (approximately 22 kg) were harvested for drying at maturity 3 days after a bifenazate treatment. In the process, plums were washed, placed in mesh bags and dried in a drying tunnel for 18 to 27 hours at 71–88 °C simulating a commercial process. Approximately 7 and 9 kg of prunes were produced from 22 kg of fresh plums at the two sites.

Table 43. Bifenazate residues in prunes resulting from processing trials in USA.

PLUMS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days		as bifenazate, mg/kg 1/	
USA (CA) 1998, French prune	500 WP	0.56	0.12	470	1	3 3	whole fruit whole fruit dried prune	0.06 2/ 0.02 3/ 0.01	CLS-98-109 RP-02007 RP-98006

PLUMS	Application					PHI	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
country, year (variety)	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.	days			
USA (OR) 1998, Parsons	500 WP	0.56	0.12	470	1	3	whole fruit dried prune	0.03 2/ < 0.01	DNJ-98-112 RP-02007 RP-98006

1/ mean of duplicate samples.

2/ field samples taken at the same time as the fruit for processing..

3/ sampled at the processing laboratory.

Korpalski (1999, RP-98007) described the processing of field-treated grapes into juice and raisins.

Approximately 22 and 45 kg of grapes were available for processing in the two trials. Fresh grapes were removed from storage and fed into a crusher/stemmer. The grape pulp was collected and the stems were discarded. The collected grape pulp was heated in a steam kettle to 52−57°C for 10 minutes and then to 60−66°C for another 10 minutes. The heated grape pulp was pressed to separate the juice and wet pomace, which was discarded. The juice was filtered and placed in the freezer for analysis.

Grapes for raisins were placed on trays or paper to dry. After 7 days the raisin samples were turned. After 14 days the stems were removed and the raisin samples were placed in bags and sent to the laboratory.

Korpalski and Puhl (2002, RP-01015) described the processing of mint tops to mint oil. Approximately 11 kg of fresh mint tops were subject to steam distillation in a cooker to produce 33−43 mL of oil in the 4 process runs (1 control and 3 treated samples). The oil samples were filtered, refrigerated and sent to the laboratory.

Dorschner (2002, 07266) descibed the processing of tomatoes to produce paste and puree. Tomatoes were first cleaned and then soaked for 3 minutes in a dilute sodium hydroxide solution, then thoroughly rinsed. Tomatoes were then chopped and rapidly heated to about 80°C and skin and seeds were separated from juice. Juice was evaporated to produce a puree. Further concentration and addition of salt produced a paste that was heated to approximately 85°C and canned. Residue data are summarised in Table 45.

Belcher (2000, RP-99008) described the processing of harvested seed cotton to cotton seed and refined oil. The seed cotton was first dried and the burrs, sticks and other trash removed. Ginning then removed most of the lint and the ginned seed was further delinted to produce cotton seed containing approximately 3% lint. A mill cracked the seed and removed most of the hull material. Kernel material was then heated to 80−90˚C for 15−30 minutes and then flaked and extruded with a flaking mill and steam treated. The material was then dried at 65−80˚C ready for solvent extraction with hot hexane. Hexane was evaporated from the extracted material to produce meal. After hexane was removed from the oil by vacuum evaporation, the oil was refined. Residue data for the meal and oil are summarised in Table 45.

Table 44. Bifenazate residues in grapes and processed commodities resulting from supervised trials in USA.

GRAPES country, year (variety)	Application					PHI days	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg 1/	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
USA (CA) 1998, Thomson Seedless	500 WP	2.8		470	1	14 14 14	whole fruit juice raisins	0.28 0.02 0.01 0.07 0.13	RP-98007 CEJ-98-115
USA (CA) 1998, Thomson Seedless	500 WP	2.9		480	1	15 15 15	whole fruit juice raisins	0.115 0.02 0.02 0.40 0.34	RP-98007 CEJ-98-116

1/ The residue on the whole fruit is the mean of duplicate field samples
The two residues for juice and raisins represent duplicate processing runs.

Table 45. Bifenazate residues in raw and processed commodities resulting from supervised trials on tomatoes, mint and cotton in USA.

CROP country, year (variety)	Application					PHI days	Commodity	Bifenazate + bifenazate-diazene as bifenazate, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water (L/ha)	no.				
TOMATOES USA (CA), 2000, H9553	500 WP	0.58	0.16	360	1	3	fruit puree paste	0.09 0.49 0.11	07266.00-CA41 GRL-FR-11673
MINT USA (WA), 2001, Spearmint (Native)	500 WP	1.8		290	1	7 7	mint tops mint oil	20 1.6 0.90 0.97 (3 processing runs)	RP-01015 DNJ-01-101
COTTON USA (NM) 1999, Acala 1517-95	500 WP	5.3			1	60	cotton seed hulls meal refined oil	1.05 0.11 < 0.01 < 0.01	RP-99008 SWF-99-20A
COTTON USA (CA) 1999, GC 500	500 WP	5.3			1	60	cotton seed hulls meal refined oil	2.64 0.92 < 0.01 < 0.01	RP-99008 CEJ-99-106

Table 46. Summary of processing factors for bifenazate residues. The factors are calculated from the data recorded in tables in this section.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors.	Mean or best estimate
Apple	wet pomace	1.8, 1.7	1.8
Apples	apple juice	0.23, 0.10	0.17
Cotton seed	cotton seed hulls	0.105, 0.35	0.23
Cotton seed	cotton seed meal	< 0.0095, < 0.0038	< 0.0038
Cotton seed	cotton seed refined oil	< 0.0095, < 0.0038	< 0.0038
Grapes	grape juice	0.054, 0.17	0.11
Grapes	raisins	0.36, 3.2	3.2
Mint tops	mint oil	0.080, 0.045, 0.049	0.057

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors.	Mean or best estimate
Plums	dried prunes	0.5, < 0.3	0.5
Tomato	tomato paste	1.26	1.3
Tomato	tomato puree	5.6	5.6

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The meeting received a lactating dairy cow feeding study, which provided information on likely residues resulting in animal tissues and milk from residues in the animal diet.

Groups of three lactating Holstein dairy cows (animals weighing 437−591 kg and 460−619 kg on days 1 and 28 respectively) were dosed once daily via gelatin capsule with bifenazate at 1 ppm (1×), 3 ppm (3×) and 10 ppm (10×) in the dry-weight diet, for 28 consecutive days (Wiedmann and Jablonski, 1999, 7474). Milk was collected twice daily for analysis and pooled from the morning and evening milkings. Butterfat and skim milk samples were taken on days 20 and 28. On day 29, within 24 hours of the final dose, the animals were slaughtered for tissue collection. Tissues collected for analysis were liver, kidney, perirenal fat, omental fat, round muscle and loin muscle. Animals consumed approximately 21−29 kg feed (approx 88% dry matter) each per day and produced approximately 16−24 kg milk per animal per day (means for each animal through the test period). Samples were analysed for bifenazate + bifenazate-diazene by HPLC-coulometer method 7473-98-0115CR-001 and for 4-hydroxybiphenyl and its sulphate conjugate by HPLC with fluorescence detection.

Residue samples were stored at freezer temperatures (below -10°C) awaiting extraction and analysis. Intervals of storage were: muscle, liver and kidney 1 day; fat 3−14 days; milk 5−112 days; butterfat 15−23 days and skim milk 95−103 days. Residues in muscle, liver and kidney decline very quickly, while the residues are much more stable in milk and fat matrices.

Residues of bifenazate + bifenazate-diazene did not exceed the LOQ (0.01 mg/kg) in loin muscle, round muscle, liver, milk or skim milk at the highest dosing level 10 ppm (Table 47). Residues were detected in the kidney of one animal at 0.01 mg/kg. Residues were present in omental and perirenal fat in the 3 ppm group (0.01−0.03 mg/kg) and the 10 ppm group (0.03−0.10 mg/kg), but not in the 1 ppm group. Residues were also present in butterfat from the 10 ppm group (0.01−0.03 mg/kg) but not from the 3 ppm group. Residues of 4-hydroxybiphenyl and its sulphate conjugate did not exceed the LOQ (0.01 mg/kg) in any sample of tissue, milk or butterfat.

Table 47. Residues in milk and tissues of lactating dairy cows (3 per group) dosed once daily via gelatin capsule with bifenazate at 1 ppm (1×), 3 ppm (3×) and 10 ppm (10×) in the dry-weight diet, for 28 consecutive days (Wiedmann and Jablonski, 1999, 7474).

Substrate	Residues, mg/kg 1/					
	Dosing, 1 ppm		Dosing, 3 ppm		Dosing, 10 ppm	
	bifenazate + bifenazate-diazene	4-hydroxy biphenyl + sulphate conj	bifenazate + bifenazate-diazene	4-hydroxy biphenyl + sulphate conj	bifenazate + bifenazate-diazene	4-hydroxy biphenyl + sulphate conj
Loin muscle					< 0.01 (3)	< 0.01 (3)
Round muscle					< 0.01 (3)	< 0.01 (3)
Liver					< 0.01 (3)	< 0.01 (3)
Kidney					0.01 < 0.01 (2)	< 0.01 (3)
Omental fat	< 0.01 (3)	< 0.01 (3)	0.01 0.02 0.02	< 0.01 (3)	0.03 0.06 0.07	< 0.01 (3)

Substrate	Residues, mg/kg 1/					
	Dosing, 1 ppm		Dosing, 3 ppm		Dosing, 10 ppm	
	bifenazate + bifenazate-diazene	4-hydroxy biphenyl + sulphate conj	bifenazate + bifenazate-diazene	4-hydroxy biphenyl + sulphate conj	bifenazate + bifenazate-diazene	4-hydroxy biphenyl + sulphate conj
Perirenal fat	< 0.01 (3)	< 0.01 (3)	0.02 0.03 0.02	< 0.01 (3)	0.10 0.07 0.07	< 0.01 (3)
Milk, days 1-28					< 0.01 (84)	< 0.01 (84)
Butterfat day 20			< 0.01 (3)	< 0.01 (3)	0.01 (3) 2/	< 0.01 (3)
Butterfat day 28			< 0.01 (3)	< 0.01 (3)	0.03 (2) 0.02 3/	< 0.01 (3)
Skim milk day 20					< 0.01 (3)	< 0.01 (3)
Skim milk day 28					< 0.01 (3)	< 0.01 (3)

1/ Concentration of bifenazate + bifenazate-diazene expressed as bifenazate. Concentration of 4-hydroxybiphenyl + sulphate conjugate expressed as 4-hydroxybiphenyl .

2/ Butterfat averaged 42.8% of milk sample due to separator failure. Therefore, residues are artificially low.

3/ Butterfat averaged 13% of milk sample.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was received on residues of bifenazate in food in commerce or at consumption.

NATIONAL MAXIMUM RESIDUE LIMITS

Information was provided on national residue definitions for bifenazate.

Australia (APVMA, 2006)

Sum of bifenazate and bifenazate diazene (diazenecarboxylic acid, 2-(4-methoxy-[1,1'-biphenyl-3-yl] 1-methylethyl ester), expressed as bifenazate .

Japan

Commodities of plant origin and fat: Sum of bifenazate and isopropyl 2-(4-methoxybiphenyl-3-yl)diazenylformate expressed as bifenazate.

Other commodities: Sum of bifenazate, isopropyl 2-(4-methoxybiphenyl-3-yl)diazenylformate and 4-hydroxybiphenyl expressed as bifenazate.

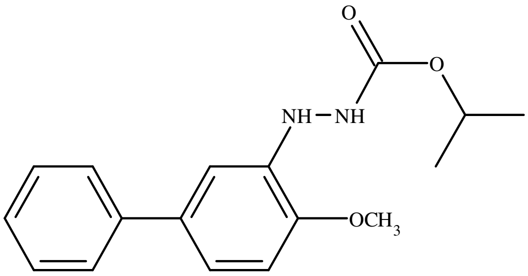
USA (USEPA, 2005)

Combined residues of bifenazate and diazinecarboxylic acid, 2-(4-methoxy-[1,1'-biphenyl]-3-yl), 1-methylethyl ester (expressed as bifenazate) for food crop commodities and animal fats.

Combined residues of bifenazate; diazinecarboxylic acid, 2-(4-methoxy-[1,1'-biphenyl]-3-yl), 1-methylethyl ester (expressed as bifenazate); 1,1'-biphenyl, 4-ol; and 1,1'-biphenyl, 4-oxysulfonic acid (expressed as 1,1'-biphenyl, 4-ol) for meat and meat byptoducts.

APPRAISAL

Bifenazate was considered for the first time by the present meeting. It is a selective acaricide which controls the motile stage of mites either by direct contact or through contact with foliar residues.



IUPAC: Isopropyl 2-(4-methoxybiphenyl-3-yl)hydrazinoformate

CAS: 1-methylethyl 2-(4-methoxy[1,1'-biphenyl]-3-yl)hydrazinecarboxylate

The Meeting received information on bifenazate metabolism and environmental fate, methods of residue analysis, freezer storage stability, national registered use patterns, supervised residue trials, fate of residues in processing and national MRLs. Australia and Japan submitted GAP information and labels to support MRLs for bifenazate.

Animal metabolism

The Meeting received animal metabolism studies with bifenazate in rats, lactating goats and laying hens. Bifenazate ^{14}C labelled in the substituted phenyl ring was used in all of the metabolism studies.

Bifenazate is readily converted to bifenazate-diazene (isopropyl 2-(4-methoxybiphenyl-3-yl)diazenoformate) by mild oxidation. Primary metabolites are readily produced by removal of the side chain and by hydroxylation of the biphenyl rings. Glucuronide and sulphate conjugates are also produced.

When rats were orally dosed with labelled bifenazate it was readily absorbed followed by extensive metabolism and excretion. Parent bifenazate and the following metabolites were identified in excreta: bifenazate glucuronide, bifenazate-diazene, 4-hydroxy bifenazate, 4-hydroxy bifenazate-diazene, 4-hydroxybiphenyl and its sulphate conjugate, 4,4'-dihydroxybiphenyl and its glucuronate and sulphate conjugates, 4-methoxybiphenyl and 4-hydroxy-4'-methoxybiphenyl and its conjugates. (See the toxicology report for more details of laboratory animal metabolism)

When a lactating goat was orally dosed with labelled bifenazate for 4 consecutive days at 21 mg/animal/day, equivalent to 10 ppm in the feed, most of the administered ^{14}C was excreted in the faeces (47%) and urine (19.5%). ^{14}C recovery was borderline at 68%. Residues in milk and tissues plus blood accounted for 0.22% and 2.0% of the dose respectively.

Metabolite 4-hydroxybiphenyl sulphate was the major identified component of the residue in milk (41% of TRR), while bifenazate and bifenazate-diazene comprised 9%. In muscle, residue levels were low with 4-hydroxybiphenyl the major identified component. In the fat, bifenazate was the major component at 53–58% of TRR. Residue levels and patterns in omental and perirenal fats were quite similar.

In goat liver, only 10% of the TRR was extractable. Bifenazate + bifenazate-diazene and 4-hydroxybiphenyl glucuronide were the main identified components, each comprising about 1% of TRR. In goat kidney, 4-hydroxybiphenyl glucuronide and sulphate accounted for approximately 14% of TRR. Bifenazate + bifenazate-diazene comprised less than 2% of TRR. In both liver and kidney, some of the unextractable TRR was apparently bound to protein.

The concentration of parent compound + bifenazate-diazene was substantially higher in the fat than in the other tissues suggesting that bifenazate (+ bifenazate-diazene) is a fat-soluble compound. No information was available on the residue distribution into the fat of goat milk.

When laying hens were orally dosed with labelled bifenazate for 4 consecutive days at 1.3 mg/bird/day, equivalent to 10 ppm in the feed, most of the administered 14C was excreted in the faeces (84%). ^{14}C recovery was approximately 85%. Residues in eggs and tissues accounted for 0.1% and 1.4% of the dose respectively. Residues were not detectable (< 0.005 mg/kg) in breast muscle and egg white.

The major identified residues in liver, skin + fat and egg yolk were 4-hydroxybiphenyl (0.013 mg/kg, 2% TRR), bifenazate-diazene (0.008 mg/kg, 17% TRR) and bifenazate (0.005 mg/kg, 20% TRR), respectively. The distribution of bifenazate and bifenazate-diazene in the tissues and egg yolk suggests fat solubility.

The metabolic pathways in goats and poultry were generally similar, but additional conjugates were identified in the goat.

Plant metabolism

The Meeting received plant metabolism studies with bifenazate on oranges, apples, grapes, radish and cotton.

In plants, most of the resultant residue from the use of bifenazate was a surface residue. Parent bifenazate was the major component of the residue at shorter intervals and the major identified component at longer intervals after treatment. Bifenazate-diazene was usually also present, but at much lower levels than parent bifenazate. Very little of the residue was translocated to the roots from treated radish foliage.

When Valencia orange trees were treated with a single application of WP formulated [^{14}C]bifenazate (0.42 and 2.2 kg ai/ha), approximately 80% of the residue was on the fruit surface 43 days after treatment, with bifenazate + bifenazate-diazene constituting 83% of TRR in and on the fruit. Bifenazate-diazene oxide, 4-methoxybiphenyl and 3-hydroxy-4-methoxybiphenyl were identified as minor components. After the rinsed oranges were separated into peel and the peeled fruits were homogenized to pulp and juice, the TRR distribution was mostly into the peel (approx 19% of TRR in whole fruit) with 0.9% in the pulp and 1% in the juice. Bifenazate was the only identified component in the juice at 0.003 and 0.001 mg/kg from the 0.42 and 2.2 kg ai/ha treatments respectively.

The TRR in oranges declined substantially at longer harvest intervals of 184, 274 and 442 days. Bifenazate and bifenazate-diazene were identified as components of the residue even at the longer intervals.

When Granny Smith apple trees were treated with a single application of WP formulated [^{14}C]bifenazate (0.42 and 2.2 kg ai/ha), approximately 60% of the residue was on the fruit surface 101 days after treatment, with bifenazate + bifenazate-diazene constituting 38% of TRR in and on the fruit. Bifenazate-diazene oxide and 4-methoxybiphenyl were identified as minor components. After the rinsed apples were homogenized and centrifuged to produce pomace and juice, the TRR distribution was mostly into the pomace (approx 30% of TRR in whole fruit) with approx 10% into the juice. Parent bifenazate and identified metabolites were not detected (< 0.001 mg/kg) in the juice.

Grape vines (variety Thompson Seedless) were treated with a single foliar application of WP formulated [^{14}C]bifenazate at 0.56 and 1.1 kg ai/ha and grapes were harvested 30 days later at maturity. Approximately 97% of the residue was surface residue. Bifenazate + bifenazate-diazene accounted for 98% and 95% of the TRR for the 0.56 and 1.1 kg ai/ha treatments respectively.

Radish plants (variety French Breakfast) were sprayed with a single foliar application of WS formulated [^{14}C]bifenazate at 1.1 and 2.2 kg ai/ha and harvested 7 days later for analysis. Most of the ^{14}C remained on the foliage (TRR 13 and 21 mg/kg) with little reaching the roots (0.0023 and 0.0043 mg/kg). The majority of the residue (60% and 80% TRR) remained on the surface. Bifenazate + bifenazate-diazene accounted for 57% and 71% of the TRR in and on the radish tops. A ring-hydroxylated bifenazate-diazene was identified as constituting approximately 1.3% of the TRR in radish tops.

After cotton plants (variety Maxxa), at late bloom to early boll set, were sprayed with a single foliar application of WP formulated [^{14}C]bifenazate (0.56 and 2.2 kg ai/ha), bifenazate and identifiable metabolites were present at very low levels (each < 0.001 mg/kg) in the cotton seed harvested 112 days after treatment. A high proportion (77–82%) of the cotton gin trash residue was extractable, with bifenazate approximately 50% of the extractable residue and bifenazate-diazene, bifenazate-diazene oxide and 4-methoxybiphenyl identified as minor residue components.

Environmental fate in soil

The Meeting received information on crop rotational studies for bifenazate. Information on soil metabolism and field dissipation was not required because no bifenazate uses as seed treatments or on root crops were provided for evaluation.

In a confined rotational crop study in USA a loamy sand soil was treated directly with ^{14}C labelled bifenazate at a rate equivalent to 0.56 kg ai/ha and allowed to age under greenhouse conditions prior to the sowing of the rotational crops. Crops of carrots, lettuce and wheat were sown into the treated soil in pots at intervals of 30, 125 and 360 days after treatment.

Immature lettuce was sampled at the 4-5 leaf stage. Immature carrot plants were sampled when carrots were approximately 6 mm in diameter. Wheat forage samples were taken approximately 5 weeks after sowing. The remainder of the crops were grown to maturity and subsequently harvested and analysed for ^{14}C (TRR) content. Samples were extracted and, where extractable residues exceeded 0.01 mg/kg, they were analysed by HPLC. No parent compound or reference metabolite was observed (LOQ 0.01 mg/kg). The unextractable residual solids from the wheat straw and fractions from the wheat forage were subjected to acid, base and enzyme hydrolysis, but no parent bifenazate or recognizable metabolite was released.

Methods of residue analysis

The Meeting received descriptions and validation data for analytical methods for residues of bifenazate in raw agricultural commodities, processed commodities, feed commodities, animal tissues, milk and eggs.

Because bifenazate and bifenazate-diazene are readily interconverted by mild oxidation and reduction conditions, the measured residue includes both compounds. The analytical methods use a mild reduction with ascorbic acid to convert the bifenazate-diazene residue to bifenazate before the measurement step. Residues are typically extracted with acetonitrile and water acidified with acetic acid. After a partition cleanup and reduction with ascorbic acid, the residue is analysed by HPLC with coulometric detection. The oxidative coulometric detection system is quite selective. Substituted hydrazines such as bifenazate are oxidised at 200 mV, but most sample matrix components are not.

LC-MS-MS has also been used in place of coulometric detection. The $[M+H]^+$ ion is used as the precursor ion for bifenazate. Transitions 301.1/198.1 (for quantification) and 301.1/170.1 are observed.

Numerous recovery data on a wide range of crop and animal commodity substrates and processed commodities were provided from validation testing of the methods, which showed that the methods were valid over the relevant concentration ranges. The validated LOQ was typically 0.01 mg/kg.

None of the tested multiresidue methods was suitable for the analysis of bifenazate and bifenazate-diazene.

Samples of apples and oranges from [^{14}C]bifenazate crop metabolism studies were extracted with acetonitrile + acetic acid and analysed by the HPLC-coulometer method and an HPLC-radiometric method. The HPLC-coulometer results were approximately 60% of those from the radiometric method.

Samples of fat and liver from a goat dosed orally for 4 consecutive days with [^{14}C]bifenazate at the equivalent of 20 ppm in the feed as in a goat metabolism study were analysed by the HPLC-coulometer method and by radiolabel measurement for bifenazate + bifenazate-diazene residues. Agreement was good for the fat tissue (0.043 and 0.045 mg/kg, radiolabel and enforcement respectively) while for the liver the level was too low for the enforcement method (0.0082 and < 0.01 mg/kg). Samples of milk and liver were hydrolysed with hydrochloric acid for 2 hours to convert the sulphate conjugate of 4-hydroxybiphenyl to the free metabolite for analysis. The analytical results for 4-hydroxybiphenyl, by a suggested enforcement method, were in good agreement with the radiolabel measurement for liver and in reasonable agreement for milk.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of residues of bifenazate and bifenazate-diazene in apples, apricots, cantaloupe, cherries, cotton seed, cotton seed hulls, cotton seed meal, cotton seed refined oil, egg yolk, fat, gin trash, grape juice, grapes, kidney, liver, milk, mint, muscle, oranges, peaches, peppers, plums, potatoes, poultry liver, poultry muscle, poultry skin + fat, prunes, tomato, tomato paste and tomato puree.

Bifenazate residues (measured as bifenazate + bifenazate-diazene) are not particularly stable in some substrates. Stability is improved where the commodity is stored unchopped and in processed commodities presumably where enzymes are denatured. Bifenazate residues are stable in fat and milk, but are particularly unstable in kidney. Bifenazate residues are unstable in potato tuber matrix to the extent that disappearance from spiked samples causes difficulty with analytical recovery testing.

In a number of substrates some losses appeared to occur at spiking or soon after, but these losses may not be relevant when assessing the stability of incurred residues.

Residues of bifenazate or bifenazate-diazene measured as the sum of bifenazate and bifenazate-diazene did not decline by more than 30% when spiked into the following substrates and stored in a freezer at temperatures below -18°C for the interval tested: homogenized tomato 6 months; homogenized peppers 6 months; homogenized mint tops 102 days; sliced plums 4 weeks; tomato paste 4 weeks; tomato puree 4 weeks; cottonseed refined oil 28 days; apples skin surface 224 days; grapes surface 224 days; peaches skin surface 223 days; homogenized oranges 186 days; grape juice 186 days; homogenized prunes 182 days; milk 202 days; fat 95 days.

Estimates were made of the time interval for a 30% decline of residues of bifenazate or bifenazate-diazene measured as the sum of bifenazate and bifenazate-diazene when spiked into the following substrates and stored in a freezer at temperatures below -18°C: homogenized cherries 2.6 months; homogenized cantaloupe 3.9 months; homogenized apples 106 days; homogenized grapes 22 days; homogenized peaches 92 days; muscle 10 days.

In some matrices, e.g. cotton seed, the stability data were variable and difficult to interpret precisely.

When bifenazate was spiked into control samples of egg yolk, hen skin + fat, thigh muscle and liver and stored for 6 months below -10 °C, residues were stable in egg yolk and liver. In thigh muscle, 45% of the bifenazate disappeared, with 14% and 11% appearing as 4-hydroxybiphenyl and bifenazate-diazene respectively. In skin + fat, 97% of the bifenazate disappeared with 4%, 4% and 59% appearing as 4-hydroxybiphenyl, 4-methoxybiphenyl and bifenazate-diazene respectively.

Samples from the laying hen metabolism study were analysed by HPLC before and after freezer storage of 121–171 days to test the stability of incurred residues. The qualitative appearance of the initial and final chromatograms were reasonably similar for egg yolk, skin-with-fat and liver. Substantial changes were apparent for thigh muscle, but total residues in thigh muscle were very low (0.006 mg/kg).

Definition of the residue

The composition of the residue in the metabolism studies, the available residue data in the supervised trials, the toxicological significance of metabolites, the capabilities of enforcement analytical methods and the national residue definitions already operating all influence the decision on residue definition.

Parent compound and metabolite bifenazate-diazene are readily interconverted, so both should be included in the residue definition.

In crop residue situations, parent compound comprised a substantial part of the residue for commodities that were directly sprayed, so bifenazate and bifenazate-diazene should constitute the residue definition for crops.

In goat fat, poultry fats and egg yolks, the sum of bifenazate and bifenazate-diazene was the major identifiable residue.

In goat muscle, liver, kidney and milk, 4-hydroxybiphenyl and its conjugates constituted the main identifiable residue. However, 4-hydroxybiphenyl may arise from sources other than bifenazate uses. It is a mammalian[8] and fungal[9] metabolite of biphenyl, a post-harvest fungicide used on citrus. It is also an industrial chemical used in the rubber industry[10]. Origins of 4-hydroxybiphenyl other than bifenazate mean that it would not be useful as part of an enforcement residue definition.

In the animal metabolism studies, the concentration of bifenazate + bifenazate-diazene was higher in the fat than in other tissues. In the dairy cow feeding study, the residue of bifenazate + bifenazate-diazene partitioned into the butter fat at the highest dosing level. The octanol-water partition coefficient of bifenazate (log P_{OW} = 3.5) also suggests that fat-solubility for the parent compound.

The Meeting recommended a residue definition for bifenazate for plants and animals.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake): sum of bifenazate and bifenazate-diazene (diazenecarboxylic acid, 2-(4-methoxy-[1,1'-biphenyl-3-yl] 1-methylethyl ester), expressed as bifenazate. The residue is fat soluble.

Results of supervised trials on crops

The Meeting received supervised trials data for bifenazate uses on citrus fruits (mandarin, natsudaidai, lime), pome fruits (apple, pear), stone fruits (apricot, peach, plum, cherry), berry fruits (grapes, strawberry), figs, cucurbit fruiting vegetables (cantaloupe, watermelon, cucumber, summer squash), fruiting vegetables (tomato, peppers, egg plant), tree nuts (almond, pecan), cotton and herbs (mint, hops, tea).

Trials from Japan were available only in summary form and could not be evaluated.

All other trials were from the USA. In most trials, duplicate field samples from an unreplicated plot were taken at each sampling time and were analysed separately. For the purposes of the evaluation, the mean of the two results was taken as the best estimate of the residue from the plot.

Labels (or translations of labels) were available from Australia, Japan describing the registered uses of bifenazate.

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[8] Wiebkin P, Fry JR, Jones CA, Lowing RK and Bridges JW. 1978. Biphenyl metabolism in isolated rat hepatocytes: effect of induction and nature of the conjugates. *Biochemical Pharmacology* 27:1899-1907.

[9] Schwartz RD, Williams AL and Hutchinson. 1980. Microbial production 4,4'-dihydroxybiphenyl: biphenyl hydroxylation by fungi. *Appl. Environ. Microbiol.* 39:702-708.

[10] Merck Index. 1996. 12th Edition. 7459 *p*-phenylphenol.

Pome fruits

Bifenazate is registered in USA for use on pome fruit trees at 0.42–0.56 kg ai/ha with a PHI of 7 days.

In 14 US trials on apples in 1998 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.049, 0.058, 0.16, 0.16, 0.17, 0.18, 0.18, 0.19, 0.20, 0.22, 0.23, 0.37, 0.38 and 0.58 mg/kg.

In eight trials on pears in USA in 1998 with conditions matching the registered use, residues of bifenazate + bifenazate-diazene were: 0.094, 0.097, 0.10, 0.13, 0.14, 0.16, 0.24 and 0.29 mg/kg. The Meeting noted that the pear samples had spent 15–16 months in frozen storage, which exceeded the proven frozen storage interval for apples (7–8 months) representing pome fruits. However, the residue levels appeared to be stable on the fruit surface for the interval tested and the residue trials were accepted as valid.

The Meeting decided to combine the apple and pear data to form a pome fruit crop group estimation (populations not significantly different – Mann-Whitney test). The combined pome fruit data (22 values), in rank order were: 0.049, 0.058, 0.094, 0.097, 0.10, 0.13, 0.14, 0.16, 0.16, 0.16, 0.17, 0.18, 0.18, 0.19, 0.2, 0.22, 0.23, 0.24, 0.29, 0.37, 0.38 and 0.58 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in pome fruits, of 0.7 and 0.175 mg/kg respectively.

Stone fruits

Bifenazate is registered in USA for use on stone fruit trees at 0.42–0.56 kg ai/ha with a PHI of 3 days.

In five US trials on apricots in 2002 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.23, 0.30, 0.44, 0.59 and 0.73 mg/kg.

In 12 US trials on peaches in 1998 and 2002 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.13, 0.16, 0.17, 0.22, 0.23, 0.23, 0.26, 0.40, 0.44, 0.45, 0.55 and 1.2 mg/kg.

In eight US trials on plums in 1998 and 2002 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.01, 0.03, 0.034, 0.04, 0.04, 0.04, 0.07 and 0.13 mg/kg.

In 14 US trials on cherries in 2001 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.29, 0.23, 1.6, 0.11, 0.48, 0.20, 0.42, 0.89, 0.71, 1.2, 0.81, 0.18, 0.27 and 0.34 mg/kg. The data were on pitted cherries, but the Meeting accepted the data as valid for MRL setting.

The residue data from peaches, apricots and cherries appeared to be from similar populations and were combined for a stone fruits group MRL. Residues on plums appeared to be much lower (significantly different from peach and cherry residues – Mann-Whitney test) than on the other stone fruits and were not included in the data set for STMR estimation.

Residue data on stone fruits in rank order (median underlined) were: 0.11, 0.13, 0.16, 0.17, 0.18, 0.20, 0.22, 0.23, 0.23, 0.23, 0.23, 0.26, 0.27, 0.29, 0.30, 0.34, 0.40, 0.42, 0.44, 0.44, 0.45, 0.48, 0.55, 0.59, 0.71, 0.73, 0.81, 0.89, 1.2, 1.2 and 1.6 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in stone fruits of 2 and 0.34 mg/kg respectively.

Grapes

Bifenazate is registered in USA for use on grape vines at 0.42–0.56 kg ai/ha with a PHI of 14 days.

In 12 US trials on grapes in 1998 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.05, 0.07, 0.10, 0.11, 0.17, 0.17, 0.20, 0.21, 0.29, 0.31, 0.33 and 0.55 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in grapes, of 0.7 and 0.185 mg/kg respectively.

Strawberries

Bifenazate is registered in USA for use on strawberries with two treatments at 0.42–0.56 kg ai/ha and a PHI of 1 day.

In seven US trials on strawberries in 1999 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.29, 0.49, 0.53, 0.63, 0.68, 0.93 and 1.0 mg/kg.

One strawberry trial had produced values of 3.4 and 2.9 mg/kg for its 3 day sample and 0.44 as the mean of the 1 day samples. The authors of the report discounted the high values as being due to analytical error, but found no specific cause. The trial was not included in this appraisal because of doubts about its validity.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in strawberries, of 2 and 0.63 mg/kg respectively.

Fruiting vegetables, cucurbits

Bifenazate is registered in USA for use on cucurbit vegetables at 0.42–0.56 kg ai/ha with a PHI of 3 days.

In eight US trials on cantaloupes in 2000 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.03, 0.04, 0.04, 0.04, 0.05, 0.08, 0.10 and 0.16 mg/kg.

In eight US trials on cucumbers in 2000 matching GAP, residues of bifenazate + bifenazate-diazene were: < 0.01, < 0.01, 0.03, 0.04, 0.05, 0.07, 0.08 and 0.22 mg/kg.

In seven US trials on summer squash in 2000 matching GAP, residues of bifenazate + bifenazate-diazene were: < 0.01, 0.01, 0.02, 0.04, 0.06, 0.12 and 0.34 mg/kg.

The Meeting decided to combine the data from cantaloupes, cucumbers and summer squash to support a cucurbit fruiting vegetables group MRL (populations not significantly different – Mann-Whitney test).

Residue data from 23 trials on cucurbit fruiting vegetables in rank order (median underlined) were: < 0.01, < 0.01, < 0.01, 0.01, 0.02, 0.03, 0.03, 0.04, 0.04, 0.04, 0.04, <u>0.04</u>, 0.05, 0.05, 0.06, 0.07, 0.08, 0.08, 0.10, 0.12, 0.16, 0.22 and 0.34 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in cucurbit fruiting vegetables of 0.5 and 0.04 mg/kg respectively.

Fruiting vegetables, other than cucurbits

Bifenazate is registered in USA for use on fruiting vegetables at 0.42–0.56 kg ai/ha with a PHI of 3 days.

In 12 US trials on tomatoes in 2000 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.03, 0.03, 0.04, 0.04, 0.07, 0.09, 0.10, 0.11, 0.13, 0.14, 0.19 and 0.29 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in tomatoes of 0.5 and 0.095 mg/kg respectively.

In eight US trials on bell peppers in 2000 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.13, 0.15, 0.15, 0.23, 0.24, 0.32, 0.52 and 1.1 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in sweet peppers of 2 and 0.235 mg/kg respectively.

In three US trials on non-bell peppers in 2000 matching GAP, residues of bifenazate + bifenazate-diazene were: 0.54, 1.1 and 1.6 mg/kg.

The Meeting noted that the residue data for non-bell peppers, a minor crop, were rather limited, but also noted that two values were equivalent to the high end of the bell pepper data with one slightly higher as expected.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in chili peppers of 3 and 1.1 mg/kg respectively.

The Meeting noted that the registered use of bifenazate referred to the fruiting vegetables group but was unable to recommend a group MRL because the residue levels on the three crops were too different.

Tree nuts

Bifenazate is registered in the USA for use on almonds and other tree nuts (including beech nut, Brazil nut, butternut, cashew, chestnut, hickory nut and Macadamia nut) at 0.42–0.56 kg ai/ha with a PHI of 7 days and on filberts, pecans, pistachios and walnuts with a PHI of 14 days.

In five trials on almonds in USA in 2001, the application rate was 0.84 kg ai/ha, 50% higher than the GAP rate, but acceptable for trials on tree nuts. In five US trials on almonds in 2001 harvested 7 days after treatment, residues of bifenazate + bifenazate-diazene in almond kernels were: 0.01, 0.02, <u>0.03</u>, 0.05 and 0.10 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in almonds of 0.2 and 0.03 mg/kg respectively.

In five US trials on pecans in 2001 where the application rate (0.85 kg ai/ha) was 50% higher than the GAP rate and a PHI of 14 days, residues of bifenazate + bifenazate-diazene in pecan kernels were: < 0.01 (3), 0.013 and 0.014 mg/kg. The Meeting noted that the application rate was higher than GAP, but the residues were close to the LOQ and could be used for evaluation.

The Meeting agreed to extrapolate the almond data to the tree nuts group and recommended a maximum residue level and an STMR value for bifenazate in tree nuts of 0.2 and 0.03 mg/kg respectively.

Cotton seed

Bifenazate is registered in USA for use on cotton at 0.4–0.8 kg ai/ha with a PHI of 60 days.

In 19 US trials on cotton in 1999 and 2000 matching GAP, residues of bifenazate + bifenazate-diazene in cotton seed were: < <u>0.01</u> (10), 0.01, 0.02, 0.03, 0.03, 0.04, 0.04, 0.06, 0.06 and 0.28 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in cotton seed of 0.3 and 0.01 mg/kg respectively.

Mint

Bifenazate is registered in USA for use on mint at 0.42–0.84 kg ai/ha with a PHI of 7 days.

In five US trials on mint in 2000 matching GAP, residues of bifenazate + bifenazate-diazene in mint tops were: 6.4, 6.6, <u>12.9</u>, 15.4 and 18.1 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in mint of 40 and 12.9 mg/kg respectively.

Hops

Bifenazate is registered in USA for use on hops at 0.42–0.84 kg ai/ha with a PHI of 14 days.

In three US trials on hops in 1999 matching GAP, residues of bifenazate + bifenazate-diazene in mint tops were: 7.1, 7.8 and 9.3 mg/kg.

The Meeting recognized that the database for hops was very limited. However, hops are a minor crop and the Meeting estimated a maximum residue level and an STMR value for bifenazate in hops of 20 and 7.8 mg/kg respectively.

Cotton gin trash

Bifenazate is registered in USA for use on cotton at 0.4–-0.8 kg ai/ha with a PHI of 60 days.

Residues were measured on cotton gin trash in 9 of the previously mentioned cotton trials where the application rates and PHIs matched label rates. Residues of bifenazate + bifenazate-diazene in cotton gin trash were: 0.07, 0.39, 0.69, 0.88, 1.3, 2.5, 3.8, 4.0 and 18 mg/kg. No maximum residue level was estimated for dry cotton fodder (cotton gin trash) because it is not traded internationally.

Almond hulls

In five trials on almonds in USA in 2001, the application rate was 0.84 kg ai/ha, 50% higher than the GAP rate, but acceptable for trials on tree nuts. In five US trials on almonds in 2001, harvested 7 days after treatment, residues of bifenazate + bifenazate-diazene in almond hulls were: 1.8, 2.8, 5.0, 5.1 and 6.9 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in almond hulls of 10 and 5.0 mg/kg respectively. The highest residue was 6.9 mg/kg.

Fate of residues during processing

The Meeting received information on the fate of bifenazate residues during the juicing of apples, the drying of prunes, the production of grape juice and raisins, the production of tomato paste and puree, the production of cotton seed oil and the processing of mint tops.

Apples from bifenazate field trials at exaggerated (5×) rates were ground in a hammer mill and the mash was collected in cloths sacks and pressed in a hydraulic press to produce the wet pomace and juice.

Plums were harvested 3 days after a bifenazate treatment, placed in mesh bags and dried in a drying tunnel for 18 to 27 hours at 71–88°C simulating a commercial process.

Fresh field-treated grapes were fed into a crusher/stemmer to produce a grape pulp that was heated in a steam kettle and then pressed to separate juice and wet pomace. Grapes for raisins were placed on trays or paper to dry with turning after 7 days. After 14 days the stems were removed to produce the raisins.

Approximately 11 kg of fresh mint tops were subject to steam distillation in a cooker to produce 33–43 mL of oil. The oil samples were then filtered and refrigerated.

Cotton seed was cracked and dried at 55–71°C to a kernel moisture level of 12%. After further heating, the kernel material was flaked, steam treated and extracted with hexane to produce meal and crude oil. Sodium hydroxide treatment of the crude oil produced the refined cotton seed oil.

Tomatoes were first cleaned and then soaked for 3 minutes in a dilute sodium hydroxide solution, then thoroughly rinsed. Tomatoes were then chopped and rapidly heated to about 80°C and skin and seeds were separated from juice. Juice was evaporated to produce a puree. Further concentration and addition of salt produced a paste that was heated to approximately 85°C and canned.

Calculated processing factors and the mean or best estimate are summarised in the following table.

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors (PF).	Median or best estimate PF
Apple	wet pomace	1.8, 1.7	1.8
Apples	apple juice	0.23, 0.10	0.17
Cotton seed	hulls	0.105, 0.35	0.23
Cotton seed	cotton seed meal	< 0.0095, < 0.0038	< 0.0038
Cotton seed	cotton seed refined oil	< 0.0095, < 0.0038	< 0.0038
Grapes	grape juice	0.054, 0.17	0.11
Grapes	raisins	0.36, 3.2	3.2
Plums	dried prunes	0.5, < 0.3	0.5
Tomato	tomato paste	1.26	1.3
Tomato	tomato puree	5.6	5.6

The processing factors for wet apple pomace (1.8) and apple juice (0.17) were applied to the estimated STMR for pome fruits (0.175 mg/kg) to produce STMR-P values for wet apple pomace (0.32 mg/kg) and apple juice (0.030 mg/kg).

The processing factor for dried prunes (0.5) was applied to the median residue for plums (0.04 mg/kg) to produce an STMR-P value for dried prunes (0.02 mg/kg).

The processing factors for raisins (3.2) and grape juice (0.11) were applied to the estimated STMR for grapes (0.185 mg/kg) to produce STMR-P values for raisins (0.59 mg/kg) and grape juice (0.020 mg/kg). The processing factor for raisins (3.2) was applied to the grape residue data (highest value 0.55 mg/kg) to produce an estimated highest value for dried grapes (1.76 mg/kg).

The Meeting estimated a maximum residue level for bifenazate in dried grapes (= currants, raisins, sultanas) of 2 mg/kg.

The processing factors for tomato puree (5.6) and tomato paste (1.3) were applied to the estimated STMR for tomatoes (0.095 mg/kg) to produce STMR-P values for tomato puree (0.53 mg/kg) and tomato paste (0.13 mg/kg).

The processing factors for cotton seed hulls (0.23), cotton seed meal (< 0.0038) and cotton seed refined oil (< 0.0038) were applied to the estimated STMR for cotton seed (0.01 mg/kg) to produce STMR-P values for cotton seed hulls (0.0023 mg/kg), cotton seed meal (0.00004 mg/kg) and cotton seed refined oil (0.00004 mg/kg).

Residues in animal commodities

Farm animal feeding

The meeting received a lactating dairy cow feeding study, which provided information on likely residues resulting in animal tissues and milk from residues in the animal diet.

Lactating Holstein cows were dosed with bifenazate at the equivalent of 1 (1×), 3 (3×) and 10 (10×) ppm in the dry-weight diet for 28 consecutive days. Milk was collected throughout and tissues were collected for residue analysis of bifenazate + bifenazate-diazene and metabolite 4-hydroxybiphenyl and its sulphate conjugate from animals slaughtered on day 29.

Residues of bifenazate + bifenazate-diazene did not exceed the LOQ (0.01 mg/kg) in loin muscle, round muscle, liver, milk or skim milk at the highest dosing level 10 ppm. Residues were detected in the kidney of one animal at 0.01 mg/kg.

Residues of 4-hydroxybiphenyl and its sulphate conjugate did not exceed the LOQ (0.01 mg/kg) in any sample of tissue, milk or butterfat.

Residues were present in omental and perirenal fat in the 3 ppm feeding group (0.01–0.03 mg/kg) and the 10 ppm feeding group (0.03–0.10 mg/kg), but not in the 1 ppm feeding group. Residues were also present in butterfat from the 10 ppm group (0.01–0.03 mg/kg) but not from the 3 ppm group.

The dairy cow feeding study confirms the fat-solubility of the residue, bifenazate + bifenazate-diazene and that fat is the target tissue.

Farm animal dietary burden

The Meeting estimated the dietary burden of bifenazate in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual. Calculation from highest residue and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed as dry weight.

Estimated maximum dietary burden of farm animals

Commodity	CC	Residue mg/kg	Basis	DM %	Residue dw mg/kg	*Diet content (%)*			Residue contribution (mg/kg)		
						Beef cattle	*Dairy cattle*	*Poultry*	Beef cattle	Dairy cattle	Poultry
Almond hulls	AM	6.9	highest residue	90	7.7						
Apple pomace, wet	AB	0.32	STMR-P	40	0.800	*40*	*20*		0.32	0.16	
Cotton fodder, dry	AM	18	highest residue	90	20.000	*20*	*20*		4.00	4.00	
Cotton seed	SO	0.28	highest residue	88	0.318	*25*	*25*		0.08	0.08	
Cotton seed hulls	AM	0.0023	STMR-P	90	0.003						
Cotton seed meal		0.00004	STMR-P	89	0.000			*20*			0.00
Total						*85*	*65*	*20*	4.40	4.24	0.00

Estimated mean dietary burden of farm animals

Commodity	CC	Residue mg/kg	Basis	DM %	Residue dw mg/kg	*Diet content (%)*			Residue contribution (mg/kg)		
						Beef cattle	*Dairy cattle*	*Poultry*	Beef cattle	Dairy cattle	Poultry
Almond hulls	AM	5.0	STMR	90	5.6	*10*	*10*		0.56	0.56	
Apple pomace, wet	AB	0.32	STMR-P	40	0.800	*40*	*20*		0.32	0.16	
Cotton fodder, dry	AM	1.3	median residue	90	1.444	*10*	*10*		0.14	0.14	
Cotton seed	SO	0.01	STMR	88	0.011	*25*	*25*		0.00	0.00	
Cotton seed hulls	AM	0.0023	STMR-P	90	0.003						
Cotton seed meal		0.00004	STMR-P	89	0.000			*20*			0.00
Total						*85*	*65*	*20*	1.02	0.86	0.00

Animal commodities, MRL estimation

For MRL estimation, the high residues in the tissues were calculated by interpolating the maximum dietary burden between the relevant feeding levels from the dairy cow feeding study and using the highest tissue concentrations from individual animals within those feeding groups. The high residues for butterfat were calculated similarly except that the mean butterfat concentrations from the relevant groups were used instead of the highest individual values.

Cattle

The STMR values for the tissues, milk and butterfat were calculated by interpolating the STMR dietary burdens between the relevant feeding levels from the dairy cow feeding study and using the mean tissue and milk concentrations from those feeding groups.

In the table, dietary burdens are shown in round brackets (), feeding levels and residue concentrations from the feeding study are shown in square brackets [] and estimated concentrations related to the dietary burdens are shown without brackets.

Dietary burden (ppm) Feeding level [ppm]	Milk	Butterfat	Muscle	Liver	Kidney	Fat
MRL						
	mean	mean	highest	highest	highest	highest
MRL beef cattle (4.4) [3, 10]			< 0.01 [< 0.01, < 0.01]	< 0.01 [< 0.01, < 0.01]	< 0.01 [< 0.01, 0.01]	0.044 [0.03, 0.10]
MRL dairy cattle (4.24) [3, 10]	< 0.01 [< 0.01, < 0.01]	0.013 [< 0.01, 0.03]				
STMR						
	mean	mean	mean	mean	mean	mean
STMR beef cattle (1.02) [0, 1]			< 0.01 [0, < 0.01]	< 0.01 [0, < 0.01]	< 0.01 [0, < 0.01]	< 0.01 [0, < 0.01]
STMR dairy cattle (0.86) [0, 1]	< 0.01 [0, < 0.01]	< 0.01 [0, < 0.01]				

The Meeting estimated dietary burdens for bifenazate in dairy cows to be 4.24 and 0.86 ppm (maximum and mean) and for beef cattle to be 4.4 and 1.02 ppm (maximum and mean), which are all less than feeding levels that produced residues below LOQ (< 0.01 mg/kg) in the milk, muscle and liver.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in milk of 0.01* and 0.01 mg/kg, respectively.

For kidney, there was one residue detection from three animals at the 10 ppm feeding level, so for a dietary burden of 4.4 ppm, the residue in kidney should not exceed 0.01 mg/kg. The kidney and liver residues were used to support an edible offal MRL recommendation.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in mammalian edible offal of 0.01* and 0.01 mg/kg, respectively.

By interpolation, the highest residue in fat was estimated as 0.044 mg/kg, while the STMR value was below the LOQ (0.01 mg/kg).

The Meeting estimated a maximum residue level for bifenazate in mammalian meat of 0.05 (fat) mg/kg. The associated STMR values for muscle and fat were 0.01 and 0.01 mg/kg.

By interpolation, the highest residue in butterfat was estimated as 0.013 mg/kg, while the STMR value was below the LOQ (0.01 mg/kg). The Meeting noted that, in this experiment, the yield of butterfat averaged 13% of the milk sample, suggesting that this "butterfat" may have contained only about 33% lipid (if the milk contained 4% lipid). This would mean that the highest residue would be approximately 0.039 mg/kg, expressed on the lipid content.

The Meeting estimated a maximum residue level and an STMR value for bifenazate in milk fats of 0.05 and 0.01 mg/kg, respectively.

Poultry

The dietary burden for poultry, currently based only on cotton seed meal is very low and is essentially zero. According to the poultry metabolism study, residues in poultry tissues and eggs were very low even for a 10 ppm dietary burden. Bifenazate residues, with current uses, are therefore not anticipated to occur in poultry tissues and eggs.

The Meeting estimated maximum residue levels of 0.01* (fat), 0.01* and 0.01* for bifenazate in poultry meat, poultry offal and eggs, respectively. The Meeting also estimated STMR values of 0 mg/kg for bifenazate residues in poultry meat (muscle 0 mg/kg; fat 0 mg/kg), poultry edible offal and eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue concentrations listed below are suitable for establishing MRLs and for assessing IEDIs.

Definition of the residue (for compliance with the MRL and for estimation of dietary intake): Sum of bifenazate and bifenazate-diazene (diazenecarboxylic acid, 2-(4-methoxy-[1,1'-biphenyl-3-yl] 1-methylethyl ester), expressed as bifenazate. The residue is fat soluble.

CCN	**Commodity**	**MRL, mg/kg**	**STMR or STMR-P, mg/kg**
AM 0660	Almond hulls	10	5.0
SO 0691	Cotton seed	0.3	0.01
DF 0269	Dried grapes (= Currants, Raisins, Sultanas)	2	0.59
MO 0105	Edible offal (Mammalian)	0.01*	0.01
PE 0112	Eggs	0.01*	0
VC 0045	Fruiting vegetables, Cucurbits	0.5	0.04
FB 0269	Grapes	0.7	0.185
DH 1100	Hops, dry	20	7.8
MM 0095	Meat (from mammals other than marine mammals)	0.05 (fat)	0.01 muscle 0.01 fat
FM 0813	Milk fats	0.05	0.01
ML 0106	Milks	0.01*	0.01
HH 0738	Mints	40	12.9
VO 0444	Peppers, Chili	3	1.1
VO 0445	Peppers, Sweet (including Pimento or pimiento)	2	0.235
FP 0009	Pome fruits	0.7	0.175
PM 0110	Poultry meat	0.01* (fat)	0 muscle 0 fat
PO 0111	Poultry, Edible offal of	0.01*	0
FS 0012	Stone fruits	2	0.34
FB 0275	Strawberry	2	0.63
VO 0448	Tomato	0.5	0.095
TN 0085	Tree nuts	0.2	0.03
JF 0226	Apple juice		0.030
	Apple pomace, wet		0.32
	Cotton seed hulls		0.0023
	Cotton seed meal		0.00004

CCN	Commodity	MRL, mg/kg	STMR or STMR-P, mg/kg
OR 0691	Cotton seed refined oil		0.00004
DF 0014	Plum, dried (prunes)		0.02
JF 0269	Grape juice		0.020
	Tomato paste		0.13
	Tomato puree		0.53

* At or about the limit of quantification.

Note: Bifenazate is a fat-soluble compound. Previously, the milk MRL would have been marked with an F to indicate a procedure for calculating "MRLs" for processed dairy products. Currently, bifenazate MRLs for milk and milk fat are available to support "MRLs" for processed dairy products.

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of bifenazate resulted in recommendations for MRLs and STMR values for raw and processed commodities. Where data on consumption were available for the listed food commodities, dietary intakes were calculated for the 13 GEMS/Food Consumption Cluster Diets. The results are shown in Annex 3 of the 2006 JMPR Report.

The IEDIs in the thirteen Cluster Diets, based on estimated STMRs were 1−20% of the ADI (0-0.01 mg/kg bw). The Meeting concluded that the long-term intake of residues of bifenazate from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The Meeting decided that it was unnecessary to establish an ARfD. The Meeting concluded that the short-term intake of bifenazate residues is unlikely to present a public health concern.

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