

ACETAMIPRID (246)

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EXPLANATION

Acetamiprid is a neonicotinoid insecticide with contact and stomach action against a range of *Hemiptera*, *Thysanoptera* and *Lepidoptera* plant pests, acting as an agonist of the nicotinic acetylcholine receptor in the insect central nervous system. It exhibits translaminar activity in plants and is authorised for use in North and South Americas, Europe and in a number of countries in Asia and the Pacific.

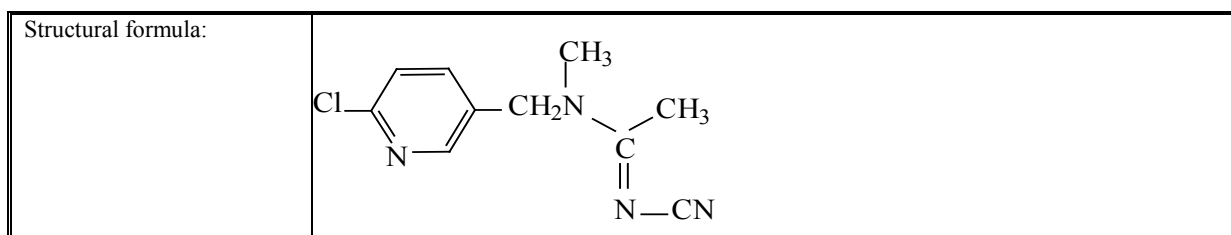
Residue and analytical aspects of acetamiprid were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, authorised uses, supervised field trials, the effects of processing, freezer storage stability, environmental fate in soil and rotational crop residues. Information on authorised uses was also provided by the Government of Japan.

In this evaluation, the values presented in the tables are as reported in the various studies, but in the accompanying text, they have generally been rounded to two significant digits. Abbreviations have also been used for the various acetamiprid metabolites mentioned in the study reports. These are:

IM-1-2	<i>N</i> ² -carbamoyl- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ¹ -methylacetamidine
IM-1-3	<i>N</i> -[(6-chloro-3-pyridyl)methyl]- <i>N</i> -methylacetamide
IM-1-4	<i>N</i> -methyl(6-chloro-3-pyridyl)methylamine
IM-2-1	<i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ² -cyanoacetamidine
IM-2-2	<i>N</i> ² -carbamoyl- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]-acetamidine
IM-2-3	<i>N</i> -[(6-chloro-3-pyridyl)methyl]acetamide
IM-2-4	(6-chloro-3-pyridyl)methylamine
IM-2-5	<i>N</i> ¹ -(6-chloropyridin-3-ylmethyl)-acetamidine
IM-0	(6-chloro-3-pyridyl)methanol
IM-0-Glc	(6-chloro-3-pyridyl)methyl-β-D-glucopyranoside
IC-0	6-chloronicotinic acid

IDENTITY

ISO common name:	Acetamiprid
Code number	NI-25
IUPAC name:	(<i>E</i>)- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ² -cyano- <i>N</i> ¹ -methylacetamidine
Chemical Abstracts name:	(<i>E</i>)- <i>N</i> -[(6-chloro-3-pyridinyl)methyl]- <i>N</i> ² -cyano- <i>N</i> -methylethanimidamide
CAS number	135410-20-7
CIPAC number	not allocated
FAO Specification	none
Molecular mass:	222.68 g/mol
Molecular formula	C ₁₀ H ₁₁ ClN ₄



Physical and chemical properties

A detailed chemical and physical characterisation of the active ingredient is given in Table 1.

Table 1 Physical and chemical data of acetamiprid (active ingredient)

Property	Results	Method (test material)	Reference
Melting point	98.9 °C	OECD 102 (99.7% purity)	RD-95148N Aceta_001
Relative density	1.330 g/cm ³ at 20 °C	EEC A3 CIPAC MT 3.2 (99.7% purity)	RD-9744 Aceta_002
Vapour pressure	< 1 × 10 ⁻⁶ Pa (1 × 10 ⁻⁸ mm Hg) at 25 °C	OECD 104 (> 99% purity)	RD-95115N Aceta_003
Physical state and colour	Pure active substance: white powder	(99.9% purity)	RD-9743 Aceta_004
Odour	Pure active substance: scentless	(99.9% purity)	RD-9743 Aceta_004
Solubility in water including effect of pH	Water solubility at 25 °C: in distilled water: 4.25 × 10 ³ mg/l pH 5: 3.48 × 10 ³ mg/l pH 7: 2.95 × 10 ³ mg/l pH 9: 3.96 × 10 ³ mg/l	OECD 105 (> 99% purity)	RD-95124N Aceta_005
Solubility in organic solvents	Solubility at 25 °C g/100g Acetone : > 20 Methanol : > 20 Ethanol : > 20 Methylene chloride : > 20 Chloroform : > 20 Acetonitrile : > 20 Tetrahydrofuran : > 20 Benzene : 2.44 Xylene : 4.01 n-Hexane : 6.54 ppm Carbon disulfide : 507 ppm	EPA 63-8 Shake Flask method (< 99% purity)	RD-95113N Aceta_006
Partition coefficient n-octanol / water	log Pow = 0.8	OECD 107 flask shaking method (> 99% purity)	RD-95112N Aceta_007
Hydrolysis rate	Acetamiprid is hydrolytically stable at pH 4, 5 and 7 at temperature tested (22 °C) and at pH 9 at 22 °C over a 35 day period. At pH 9, it is labelled at high temperatures, with half-life of 13 and 53 days at 45 °C and 35 °C, respectively.	OECD 111 BBA 55 US EPA 161-1(1982) ¹⁴ C-labelled substance (97.9% purity)	RD-95122N Aceta_008
Photochemical degradation	Testing conditions: 25 °C, 10 ppm solution Xenon lamp Distilled water: DT ₅₀ = 68.0 d River water: DT ₅₀ = 20.1 d	¹⁴ C-labelled substance (98.9% purity)	RD-95120 Aceta_009
Dissociation constant	pKa 0.68+/- 0.08.	OECD 112 spectrophotometry (> 99% purity)	RD-95114N Aceta_010

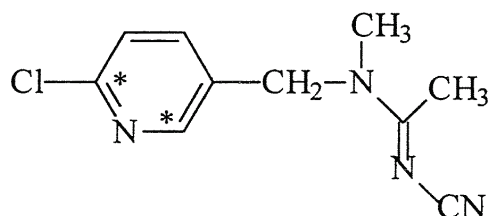
Property	Results	Method (test material)	Reference
Adsorption/ Desorption in soil	Five kinds of soils, i.e. sand, loamy sand, sandy loam, and two silt loam soils were tested. Adsorption: K_{oc} 71.09-138.39 mL/g First desorption: K_{oc} 98.19-345.14 mL/g Second desorption: K_{oc} 130.35-1178.09 mL/g	OECD 106 ^{14}C -labelled substance (> 99.9% purity)	RD-09564N Aceta_011

Formulations

Currently registered formulations containing acetamiprid include wettable powder formulations (700 g ai/kg), soluble granules (200 g ai/kg and 300 g ai/kg) and soluble powders (50 g ai/kg and 200 g ai/kg).

METABOLISM AND ENVIROMENTAL FATE

The Meeting received acetamiprid metabolism studies on animals (rats, lactating goats and laying hens), plants, soil and rotational crops. Acetamiprid radiolabelled in the 2 and 6 positions of the pyrimidine ring was used in these studies. The label positions are given below:

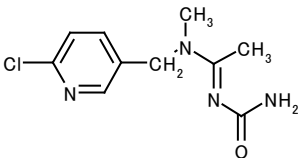
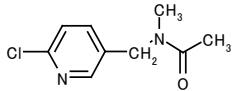
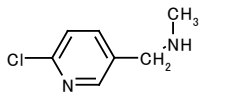
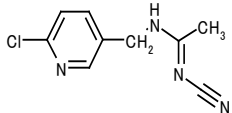
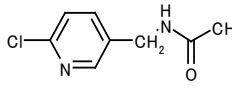
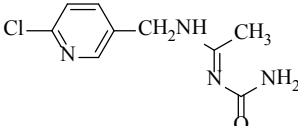
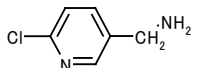
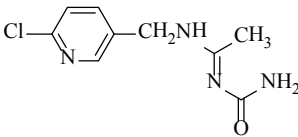
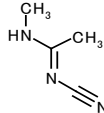
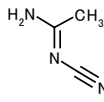
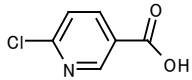
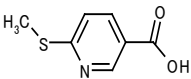


* denotes position of radiolabel, uniformly labelled in the 2 and 6 positions of the pyridine ring

Major metabolites identified in these studies and discussed in this evaluation are listed below.

Table 2 Major acetamiprid metabolites identified in plant and animal matrices

Code Number	Description/Denomination (IUPAC Name)	Study metabolite identified in	Structure
NI-25/ parent	Acetamiprid (<i>E</i>)- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ² -cyano- <i>N</i> ¹ -methylacetamidine	Apple, eggplant, cabbage, carrot, goat	
IM-0	(6-chloro-3-pyridyl)methanol	Apple, eggplant, cabbage, carrot, cotton	
IM-0-Glc	(6-chloro-3-pyridyl)methyl-β-D-glucopyranoside	apple, eggplant, cabbage, carrot, cotton	

Code Number	Description/Denomination (IUPAC Name)	Study metabolite identified in	Structure
IM-1-2	<i>N</i> ² -carbamoyl- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ¹ -methylacetamidine	carrot	
IM-1-3	<i>N</i> -[(6-chloro-3-pyridyl)methyl]- <i>N</i> -methylacetamide	Apple, cabbage, cotton, soils	
IM-1-4	<i>N</i> -methyl(6-chloro-3-pyridyl)methylamine	Apple, cabbage, carrot, cotton, soils	
IM-2-1	<i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ² -cyanoacetamidine	Apple, eggplant, cabbage, carrot, cotton, goat, hen	
IM-2-3	<i>N</i> -[(6-chloro-3-pyridyl)methyl]acetamide	Apple, cabbage, carrot, goat, hen	
IM-2-2 (IM-2-1- amide)	<i>N</i> ² -carbamoyl- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]-acetamidine	goat, hen	
IM-2-4	(6-chloro-3-pyridyl)methylamine	goat, hen	
IM-2-5	<i>N</i> ¹ -(6-chloropyridin-3-ylmethyl)-acetamidine	hen	
IS-1-1	<i>N</i> ² -cyano- <i>N</i> ¹ -methylacetamidine	cabbage	
IS-2-1	<i>N</i> ² -cyanoacetamidine	cabbage	
IC-0	6-chloronicotinic acid	Apple, cabbage, carrot, cotton, soils, goat, hen	
MeS-IC-0	6-methylthionicotinic acid		

Code Number	Description/Denomination (IUPAC Name)	Study metabolite identified in	Structure
AS-IC-0	6-hydroxycarbonylmethylthio-nicotinic acid		
IC-0-Gly	6-chloronicotinuric acid		
IM-1-5	<i>N</i> ^l -(6-Chloropyridin-3-ylmethyl)- <i>N</i> ^l -methylacetamidine	soils	

Animal metabolism

The Meeting received animal metabolism studies with ¹⁴C-acetamiprid in rats, goat and laying hens.

Rats

In rats, acetamiprid is rapidly and almost completely absorbed and is widely distributed into the tissues, being found at highest concentrations in GI tract, adrenal gland, liver and kidney, following oral administration to the rat. The major route of elimination was via the urine and bile (relevant but not a major route in excreta). The disappearance of radioactivity from the body of the rat was rapid and there was no indication of accumulation in any tissue. Less than 1% of the administered radioactivity was left in the tissues by day four following dosing. The major radioactive compounds in the excreta of rats were acetamiprid (approx. 5–7 %), the demethylated compound IM-2-1 (approx. 15–20 %), the nicotinic acid derivative IC-O (approx. 8–11%) and the IC-O glycine conjugate IC-O-Gly (approx. 10 %). In addition, MeS-IC-O, IM-1-4, IM-2-4, IM-O, IM-1-3 and IM-2-3 were detected, but they were less than 2% of dose. There were several unknown compounds in urine with a maximum abundance of 1 %.

The main metabolic pathway of acetamiprid in rats is the transformation to IM-2-1 by demethylation which is further metabolized to IC-O with the release of IS-1-1 and IS-2-1 after the cleavage from the side chains of IN-25 and IM-2-1.

Goats

The metabolism of acetamiprid in goats was investigated by Burri, 1997 [Ref: Aceta_012]. Two lactating goats (45 kg and 62 kg bodyweight respectively) were dosed twice daily for seven consecutive days at daily dose levels of 1.0 ppm diet (low dose) and 8.6 ppm diet (high dose) with [pyridine-2, 6-¹⁴C]-acetamiprid. The dose was administered in two doses per day via capsule (0.023 mg/kg bw/dose and 4.32 mg/kg bw/dose respectively) just after the morning and afternoon milking. The regular food consumption (2.0–2.4 kg/day) and milk production (0.8–1.0 L/day) and veterinary inspections indicated that the dose treatments did not affect the health of the animals. Milk, urine and faeces were collected throughout the dosing period and samples of meat, fat, liver and kidney were taken at sacrifice, 22 hours after the last administration.

The samples were analysed by LSC for total radioactivity. Milk samples were mixed with acetone to precipitate proteins, refrigerated overnight and centrifuged. The protein pellets were extracted once with acetone. The remaining aqueous layer was partitioned twice with methylene chloride, once with ethyl acetate and once with acidic ethyl acetate. Tissue samples were extracted with acetone, methanol, and the aqueous methanol. The extracts were mixed with acetonitrile followed by partition with hexane. Acetonitrile of aqueous phase was removed and partitioned with three times with methylene chloride, once with ethyl acetate and once with acidic ethyl acetate. All

layers were analysed for radioactivity by LSC. For the identification of the radioactivity the different layers were analysed by LC against reference compounds. The identification of metabolites found was confirmed by TLC and HPLC and in addition by LC/MS or GC/MS depending on the metabolites.

Most of the administered radiolabel was excreted via urine of the animal (about 89% of the low administered dose and 74% of the high administered dose). Faeces contained about 10% of the low dose and 21% of the high dose while in milk (0–174 hours) 0.17% and 0.62% of the applied radioactivity were found, respectively, with residues reaching a plateau after about 3 days. In tissues, radioactivity did not exceed 1.6% of the applied radioactivity (AR).

Table 3 Balance of radioactivity and distribution pattern in lactating goats after oral dosing with [pyridine-2, 6-¹⁴C]-acetamiprid twice daily for 7 days at the dietary equivalent of 1.0 ppm and 8.6 ppm

Animal	Goat 1		Goat 2	
Treatment	1.0 ppm dose		8.6 ppm dose	
	mg/kg equiv	% AR	mg/kg equiv	% AR
Urine		89.2		73.8
Faeces		10.1		21.3
Milk				
After am dose – day 1	0.008	0.013	0.097	0.023
After pm dose – day 1	0.004	0.014	0.104	0.046
After am dose – day 2	0.008	0.012	0.155	0.033
After pm dose – day 2	0.003	0.009	0.12	0.059
After am dose – day 3	0.008	0.013	0.179	0.038
After pm dose – day 3	0.002	0.007	0.129	0.057
After am dose – day 4	0.009	0.016	0.173	0.036
After pm dose – day 4	0.003	0.012	0.12	0.056
After am dose – day 5	0.008	0.012	0.16	0.036
After pm dose – day 5	0.003	0.011	0.12	0.059
After am dose – day 6	0.009	0.014	0.163	0.036
After pm dose – day 6	0.003	0.011	0.097	0.047
After am dose – day 7	0.008	0.015	0.142	0.03
After pm dose – day 7	0.003	0.013	0.108	0.054
21 h after last dose	0.001	0.000	0.065	0.012
Total in milk (0-174 hours)		0.17		0.62
Edible organs/tissues (at sacrifice, 21 h after last dose)				
Liver	0.01	0.06	0.493	0.42
Kidney	0.007	0.01	0.355	0.04
Muscle	< 0.001	0.02	0.064	1.16
Fat	< 0.001	< 0.01	0.012	0.06
Blood	0.003	0.08	0.076	0.27
Total in edible tissues/organs and blood		0.2		2.0
Content intestinal tract		0.4		0.6
Cage wash		1.7		2.4
Total excreted		101.2		98.1
Total recovered		101.8		100.7

The majority of radioactivity (89–98% TRR) was extracted with about 73–90% TRR being partitioned into the organic phases. Only a minor amount of the radioactivity remained in the aqueous phase (3–8%). About 94–96% of the milk radioactivity was found in the whey (defatted and deproteinised milk) with only about 3–5% being present in milk fat and precipitated milk proteins.

Table 4 Characterisation of radioactivity in liver, kidney, fat and muscle of lactating goats dosed orally (14×) with [pyridine-2, 6-¹⁴C]-acetamiprid for 7 days. Values are % TRR with mg/kg parent equivalents in brackets

Animal:	Goat 1			Goat 2			
Treatment:	Low dose (1.0 ppm diet)			High dose (8.6 ppm diet)			
Tissue/organ:	Liver	Kidney	Fat	Liver	Kidney	Fat	Muscle
Extraction:							
Extracted	89 (0.009)	97.6 (0.006)	99.7 (0.075)	94.8 (0.456)	96.9 (0.338)	87.1 (0.011)	91.8 (0.058)
Post extraction solids ^a	14.9 (0.001)	7.8 (0.001)	0.3 (< 0.001)	7.6 (0.037)	4.9 (0.017)	5.7 (0.001)	9.3 (0.006)
Total	103.9 (0.01)	105.4 (0.007)	100 (0.075)	102.4 (0.493)	101.8 (0.355)	92.8 (0.012)	101.1 (0.064)
Partitioning:							
Organic phase	73.2 (0.007)	88.5 (0.006)	–	90.6 (0.436)	90.2 (0.315)	–	86.6 (0.055)
Aqueous phase ^a	8.2 (0.001)	6.2 (< 0.001)	–	3.2 (0.015)	5.7 (0.02)	–	3.9 (0.002)
Total	81.4 (0.008)	94.7 (0.006)	–	93.8 (0.451)	95.9 (0.335)	–	90.5 (0.057)

^a not further analysed

Table 5 Characterisation of radioactivity in milk from lactating goats dosed orally with [pyridine-2, 6-¹⁴C]-acetamiprid twice daily and sampled 144–168 hours (7 days) after the first administration. Values are % TRR with mg/kg parent equivalents in brackets

Animal:	Goat 1		Goat 2	
Treatment:	Low dose (1.0 ppm diet)		High dose (8.6 ppm diet)	
Tissue/organ:	% Recovered	mg/kg eq	% Recovered	mg/kg eq
Milk fat	5.2	< 0.001	3.0	0.003
Whey				
acetone (1 st extraction)	89.8	0.004	90.5	0.106
acetone (2 nd extraction)	4.2	< 0.001	5.2	0.006
Whey total	94	0.005	95.7	0.112
Post extraction solids ^a	5.3	< 0.001	3.2	0.004
Total	104.5	0.005	101.9	0.119
Partitioning (whey):				
Organic phase	89.8	0.005	92	0.108
Aqueous phase ^a	4.2	< 0.001	3.7	0.004
Total	94	0.005	95.7	0.112

^a not further analysed

Analysis of the extracted radioactivity identified the IM-2-1 metabolite as the predominant residue, at 70-89% TRR in milk, liver and kidney. Acetamiprid (parent) was only found in milk, at about 9.6% TRR and 0.001 mg/kg (low dose) and 4% TRR and 0.005 mg/kg (high dose). In muscle, IM-2-2 was the predominant residue (about 50% TRR) and the IM-2-3 and IM-2-4 metabolites were also found at 6% and 13% TRR respectively.

Table 6 Identification of radioactivity in milk and tissues from lactating goats dosed orally for 7 days with [pyridine-2, 6-¹⁴C]-acetamiprid. Values are % TRR and mg/kg parent equivalents in brackets

	Milk		Liver		Kidney		Muscle	
Dose group	1.0 ppm	8.6 ppm	1.0 ppm	8.6 ppm	1.0 ppm	8.6 ppm	1.0 ppm	8.6 ppm
Compound	% TRR (mg/kg)	% TRR (mg/kg)	% TRR (mg/kg)	% TRR (mg/kg)	% TRR (mg/kg)	% TRR (mg/kg)	% TRR (mg/kg)	% TRR (mg/kg)
Parent	9.6 (0.001)	4.1 (0.005)	--	--	--	--	--	--
IM-2-1	78 (0.004)	76.8 (0.09)	70.2 (0.007)	89.2 (0.429)	85.2 (0.006)	86.0 (0.3)	--	9.6 (0.006)
IM-2-3	--	--	--	--	--	--	--	6.1 (0.004)
IM-2-4	--	--	--	--	--	--	--	13.2 (0.008)
IC-0	--	--	--	--	--	0.7 (0.003)	--	--
IM-2-2	--	--	--	--	--	--	--	49.8 (0.032)
Unidentified	2.2 < 0.001)	11.1 (0.013)	--	1.0 (0.005)	--	1.9 (0.006)	--	6.7 (0.004)
Not resolved		3.7 (0.004)		3.6 (0.017)		7.3 (0.026)		5.1 (0.003)
Total	89.8 (0.005)	95.7 (0.112)		93.8 (0.451)		95.9 (0.335)		90.5 (0.057)

Laying hens

The metabolism of acetamiprid was investigated in laying hens by Burri, 1997 [Ref: Aceta_013]. In this study, single daily doses of [pyridine-2, 6-¹⁴C]-acetamiprid were administered for 14 consecutive days to groups of laying hens (five animals each) at rates of 0.15 mg/hen/day and 1.5 mg/hen/day, equivalent to mean dietary concentrations of 1.1 ppm and 12.5 ppm respectively. The doses were administered each morning, after the morning egg collection and before feeding. Average food consumption was 127–144 g/hen/day and mean body weights ranged from 1.7–2 kg. Eggs and excreta were collected throughout the dosing period and samples of meat, liver and fat were taken at sacrifice.

The samples were analysed by LSC for total radioactivity. Eggs were washed and separated into yolks and whites. Pooled egg samples from each hen laid on day 6 and day 14 were mixed with acetone to precipitate proteins, refrigerated overnight and centrifuged. The protein pellets were extracted with acetone and the remaining aqueous layer was partitioned with acetone (1×), methanol (1×) and aqueous methanol (1×). Egg yolk was subjected to further partition between hexane and aqueous acetonitrile, with residues in the evaporated aqueous phase being then extracted with methylene chloride. Tissue samples were extracted with acetone, methanol, and the aqueous methanol and after mixing with acetonitrile were partitioned with hexane. After evaporation, the aqueous phase was partitioned with methylene chloride (3×), ethyl acetate (1×) and acidic ethyl acetate (1×). All layers were analysed for radioactivity by LSC. For the identification of the radioactivity the different layers were analysed by LC against reference compounds. The identification of metabolites found was confirmed by TLC and HPLC and in addition by LC/MS or GC/MS depending on the metabolites.

At sacrifice (24 hours after the last administration), total radiolabel recoveries were 95–99%, most of which was excreted or in the cage wash (93–97%). Small amounts of radioactivity were detected in edible organs/tissues (0.7–0.8%) and total radioactive residues in eggs were about 1.3% of the applied radiolabel, these reaching a plateau after about 8–11 days in the low and high dose groups respectively. In liver and skin, residues were about 0.1% of the applied radioactivity and were 0.3% in muscle.

Less than 10% of the TRR was not extracted in organic solvents and about 73-82% TRR was partitioned into the organic phases. Only a minor amount of the radioactivity remained in the aqueous phase (6-15%).

Table 7 Balance of radioactivity and distribution pattern in laying hens after oral daily doses of [pyridine-2, 6-¹⁴C]-acetamiprid

Animal	Group 1				Group 2			
Treatment	1.1 ppm dose				12.5 ppm dose			
	mg/kg equiv			% AR	mg/kg equiv			% AR
Eggs	yolk	white	total ^a		yolk	white	total ^a	
Day 1	0.000	0.002	0.0014		0.000	0.015	0.011	
Day 2	0.015	0.022	0.02		0.164	0.25	0.224	
Day 3	0.04	0.026	0.03		0.417	0.302	0.337	
Day 4	0.047	0.025	0.032		0.4	0.301	0.331	
Day 5	0.068	0.03	0.041		0.653	0.296	0.403	
Day 6	0.081	0.028	0.044		0.751	0.297	0.433	
Day 7	0.084	0.031	0.047		0.811	0.298	0.452	
Day 8	0.086	0.028	0.045		0.891	0.351	0.513	
Day 9	0.087	0.027	0.045		0.876	0.351	0.509	
Day 10	0.076	0.027	0.042		0.904	0.357	0.521	
Day 11	0.079	0.029	0.044		0.922	0.369	0.535	
Day 12	0.083	0.03	0.046		0.915	0.34	0.513	
Day 13	0.081	0.032	0.047		0.944	0.359	0.535	
Day 14	0.085	0.03	0.047		0.879	0.322	0.489	
Total in eggs				1.3	(0.944	0.369	0.542)	1.4
Edible organs/tissues ^b (at sacrifice, 24 h after last dose)								
Liver	0.034			0.1	0.568			0.1
Muscle	0.007			0.3	0.09			0.3
Fat	0.001			--	0.009			--
Skin	0.011			0.1	0.105			0.1
Blood	0.012			0.1	0.15			0.1
Plasma	0.008				0.115			
Developing eggs	0.084	0.027		0.1	0.978	0.315		0.2
Total in edible tissues/organs				0.7				0.8
Excreta				94.6				90.9
Cage wash				2.5				2.1
Total excreted				97.1				93.1
Total recovered				99				95.3

^a Assumed 30% yolk, 70% white

^b Assumed 40% muscle, 12% fat, 11% skin, 8% blood

Table 8 Characterisation of radioactivity from liver, muscle and skin of laying hens dosed orally with [pyridine-2, 6-¹⁴C]-acetamiprid for 14 days

Treatment:	Dose group 1 (1.1 ppm diet)						Dose group 2 (12.5 ppm diet)					
Tissue/organ:	Liver		Muscle		Skin		Liver		Muscle		Skin	
	% TRR mg/kg		% TRR mg/kg		% TRR mg/kg		% TRR mg/kg		% TRR mg/kg		% TRR mg/kg	
Extraction:												
Extracted	94.2	0.028	76.7	0.007	107.7	0.01	92.7	0.545	87.4	0.082	86.8	0.109
Post extraction solids	7.4	0.002	3.9	0.00	8.4	0.001	7.0	0.041	8.6	0.008	5.2	0.007
Total	101.6	0.03	80.6	0.007	116.1	0.011	99.7	0.586	96.0	0.09	92.0	0.116
Partitioning:												
Organic phase	72.7	0.022	78.7	0.005	–		75.7	0.445	81.8	0.073	79.2 ^a	0.091 ⁽¹⁾
											3.3 ^b	0.004 ⁽²⁾
Aqueous phase	15.2	0.004	6.7	0.001	–		12.7	0.075	6.5	0.006	5.7	0.007
Total	87.9	0.026	85.4	0.006	–		88.4	0.52	88.3	0.079	88.2	0.102

^a sum of methylene chloride/acetonitrile extracts and acetone-MeOH/ethyl acetate extracts^b methylene chloride/hexane extractsTable 9 Characterisation of radioactivity in eggs from hens dosed orally with [pyridine-2, 6-¹⁴C]-acetamiprid and sampled 7 days after the first administration. Values are % TRR with mg/kg parent equivalents in brackets

Animal:	Dose group 1 (1.1 ppm diet)				Dose group 2 (12.5 ppm diet)			
Tissue/organ:	Egg white		Egg yolk		Egg white		Egg yolk	
	Day 6	Day 14	Day 6	Day 14	Day 6	Day 14	Day 6	Day 14
	% TRR (mg/kg eq)	% TRR (mg/kg eq)	% TRR (mg/kg eq)	% TRR (mg/kg eq)	% TRR (mg/kg eq)	% TRR (mg/kg eq)	% TRR (mg/kg eq)	% TRR (mg/kg eq)
Extraction:								
Extracted	89.6 (0.025)	83.5 (0.028)	115 (0.051)	105.7 (0.064)	97.1 (0.295)	92.8 (0.31)	94.9 (0.719)	94.5 (0.849)
Post extraction solids	3.0 (0.001)	3.1 (0.001)	4.8 (0.002)	6.4 (0.004)	1.1 (0.003)	1.3 (0.004)	5.7 (0.043)	5.1 (0.046)
Total	92.6 (0.026)	86.6 (0.029)	119.8 (0.053)	112.1 (0.068)	98.2 (0.298)	94.1 (0.314)	100.6 (0.762)	99.6 (0.895)
Partitioning:								
Organic phase	–	–	70 (0.037)	67.5 (0.046)	–	–	68.7 (0.52)	65.7 (0.591)
Aqueous phase	–	–	26 (0.014)	26.8 (0.018)	–	–	26.2 (0.199)	28.8 (0.258)
Total	–	–	96 (0.051)	94.3 (0.064)	–	–	94.9 (0.719)	94.5 (0.849)

Analysis of the extracted radioactivity identified the IM-2-1 metabolite as the predominant residue, at 83-86% TRR in egg white, about 60% TRR in egg yolk, 65-69% TRR in liver and 53-62% TRR in muscle and skin. Other metabolites found at more than 10% TRR were IM-2-3 in muscle (17-21% TRR) and egg whites (9-11% TRR). IM-2-5 was the predominant residue in egg yolks (27% TRR) and IC-0 was found in skin at about 13% TRR. Acetamiprid (parent) was not found in any tissues or in eggs.

Table 10 Identification of radioactivity in eggs and tissues from hens dosed orally for 14 days with [pyridine-2, 6-¹⁴C]-acetamiprid

Identity	Egg white		Egg yolk		Egg total ^a		Liver		Muscle		Skin	
	mg/kg	% TRR	mg/kg	% TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	% TRR
High dose (12.5 ppm)												
Parent	-	-	-	-	-	-	-	-	-	-	-	-
IM-2-1	0.279	83.4	0.510	56.7	0.348	75.4	0.406	69.0	0.056	62.3	0.063	54.1
IM-2-3	0.031	9.4	0.032	3.6	0.031	7.7	0.021	3.6	0.015	17.3	0.012	10.5
IM-2-4	-	-	0.009	1.0	-	0.3	-	-	-	-	-	-
IC-0	-	-	-	-	-	-	-	-	-	-	0.015	12.8
IM-2-5	-	-	0.241	26.9	0.072	8.1	-	-	-	-	-	-
Unidentified	-	-	0.028	3.1	0.008	0.93	0.075	12.7	-	-	-	-
SUBTOTAL	0.310	92.8	0.820	91.3	0.463	92.4	0.502	85.3	0.071	79.6	0.090	77.4
Not resolved	0.004 ^b	1.3 ^b	0.029	3.2	0.012	1.9	0.018	3.1	0.008	8.7	0.012	10.8
TOTAL	0.314	94.1	0.849	94.5	0.475	94.2	0.52	88.4	0.079	88.3	0.102	88.2
Low dose (1.1 ppm)												
Parent	-	-	-	-	-	-	-	-	-	-		
IM-2-1	0.025	85.6	0.041	60.3	0.03	78.0	0.020	64.7	0.004	53.3		
IM-2-3	0.003	10.8	0.002	3.1	0.003	8.5	0.001	4.4	0.001	21.2		
IM-2-4	-	-	-	-	-	-	-	-	-	-		
IC-0	-	-	-	-	-	-	-	-	-	-		
IM-2-5	-	-	-	-	-	-	-	-	-	-		
Unidentified	-	-	0.02	30.4	0.006	9.1	-	-	-	-		
TOTAL	0.028	96.4	0.063	93.8	0.039	95.6	0.021	69.1	0.005	74.5		

^a Assumed 30% yolk, 70% white^b Not extracted

In summary, acetamiprid metabolism in animals is similar, with more than 95% of the residues being eliminated in excreta and less than 2% remaining in tissues or present in eggs or milk. Residues of the parent acetamiprid were not found (except at low levels in milk), and the predominant residue in most animal products was the IM-2-1 (53%-89% TRR) with IM-2-2 occurring in goat muscle at about 50% TRR). The IM-2-4 and IM-2-3 metabolites were also found in muscle at 13-21% TRR, with the IM-2-5 metabolite being found at about 27% TRR in egg yolks.

The proposed metabolic breakdown of acetamiprid in both goats and hens involves degradation to IC-0 or demethylation to IM-2-1 with the IM-1-2 metabolite converting to the amide (IM-2-2) or IM-2-3 and the subsequent formation of the IM-2-4 and IM-2-5 metabolites.

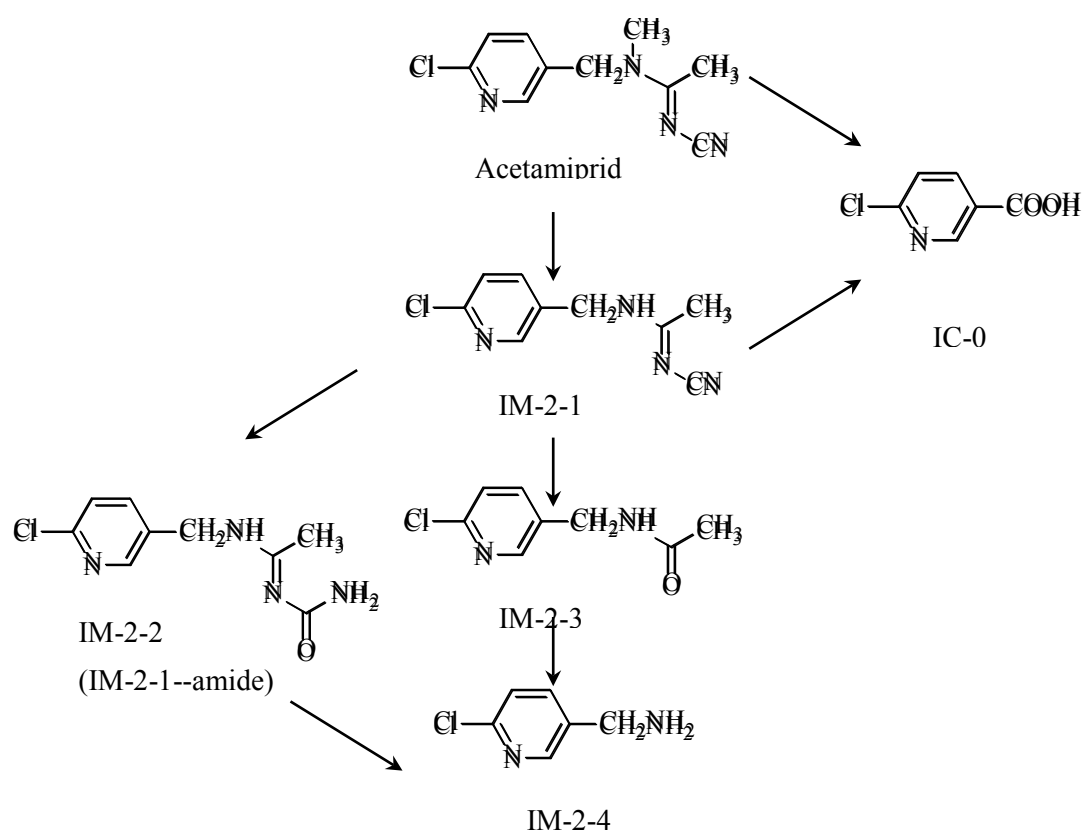


Figure 1 Proposed metabolic pathways of Acetamiprid in lactating goat

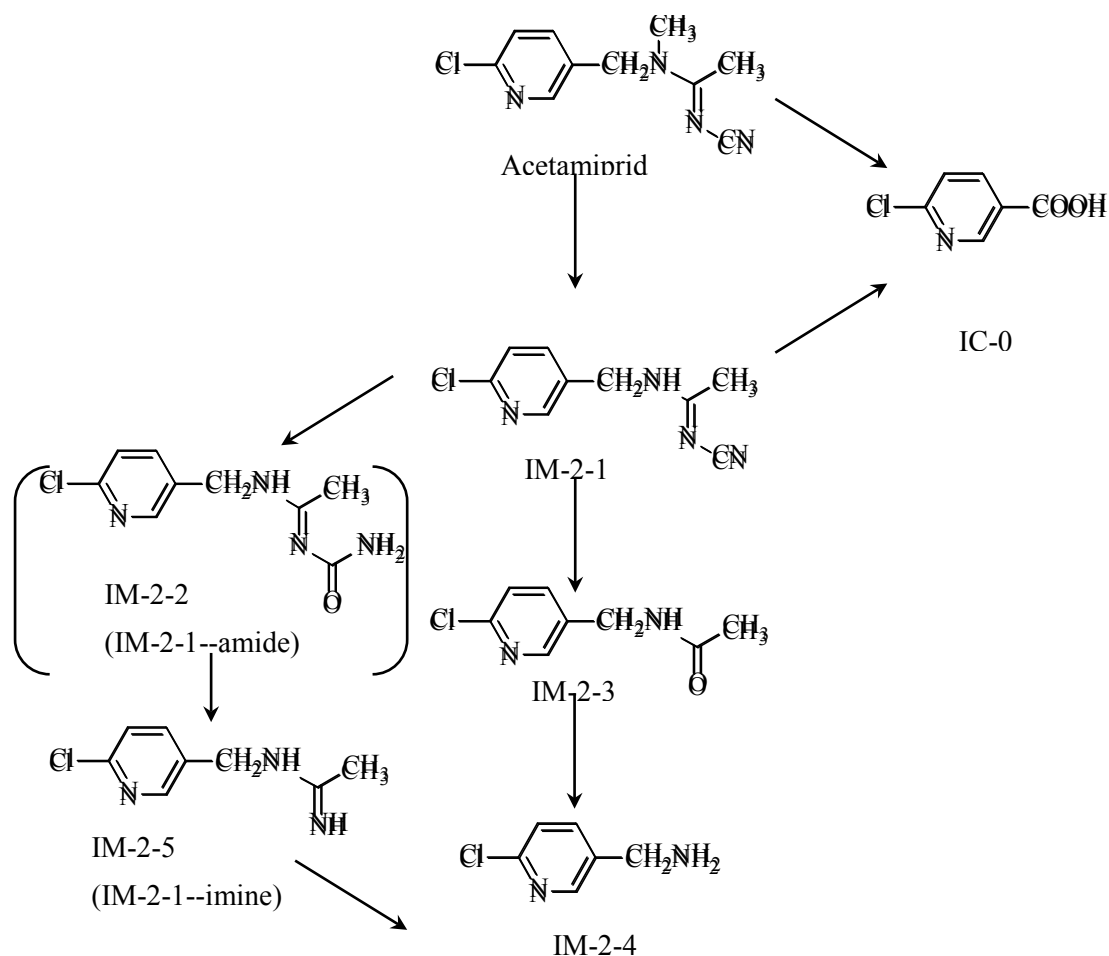


Figure 2 Proposed metabolic pathways of Acetamiprid in laying hen

Plant metabolism

The meeting received plant metabolism studies in apples, eggplant, cabbage, cotton and carrot following foliar applications of [pyridine-2, 6-¹⁴C]-acetamiprid and an additional study with cabbages treated with [CN-¹⁴C]-acetamiprid

Apples

In a study on apples conducted by Saito, 1997, [Ref: Aceta_014], 4-year old 'Tsugara' and 3-year old 'Fuji' apples trees, cultivated in a greenhouse, were sprayed with [pyridine-2, 6-¹⁴C]-acetamiprid at an rate equivalent to 10.5 g ai/hL (0.105 kg ai/ha) to seven individual fruits and 10.4 g ai/100 hL (0.208 kg ai/ha) to 44 leaves.

Samples of treated and untreated leaves were taken 0, 7, 14, 28, 62 and 90 days after the application and washed using methanol (50 mL/leaf). The washed leaves were homogenized with 75% aqueous methanol and filtered. The filtrate was partitioned with n-hexane and methylene chloride and radioactivity in all the fractions was determined by LSC. Characterisation and identification of the radioactivity was achieved by TLC, HPLC and LC-MS analysis.

The recovery of the radioactivity in leaves was greater than 98%, with surface residues decreasing from an initial level of 99.9% TRR down to about 43% TRR at the end of the 90-day study

period and the residues in the homogenate increasing accordingly. Translocated radioactivity in untreated leaves was less 0.04 mg/kg.

The majority of the radioactivity was extracted (93–99%) with methylene chloride and was mostly the unchanged parent compound, making up 90% or more of the TRR in the first 14 days after application, declining to 49% TRR after 90 days. The main metabolite found above 5% TRR was IM-2-1, present at about 10% TRR after 62 days and 15.6% TRR after 90 days. The only other metabolite present at more than 5% TRR was IM-0-Glc (max 8.3% TRR at day-90).

Table 11 Characterisation and distribution of radioactivity in apple leaves after a single foliar application of [pyridine-2, 6-¹⁴C]-acetamiprid

Days after application	0 days	7 days	14 days	28 days	62 days	90 days
	% TRR					
Surface wash	99.9	73.9	52.8	48.3	37.2	42.9
Homogenate (extracted)	na	25.4	45.5	47.9	58.1	50.6
Leaf total extracted	99.9	99.3	98.3	96.2	95.3	93.5
Post-extraction solids	0.1	0.7	1.7	3.8	4.7	6.6
	Concentration (mg/kg parent equivalents)					
TRR (mg/kg eq)	35.78	37.82	31.47	24.85	25.71	23.3
Surface wash	35.75	27.91	16.44	12.21	9.5	10.09
Homogenate (extracted)	na	9.64	14.5	11.7	15.0	11.75
Leaf total extracted	35.75	37.55	30.94	23.91	24.5	21.84
Post-extraction solids	0.04	0.26	0.54	0.94	1.21	1.46
Upper leaves (untreated)	na	0.01	0.00	0.02	0.02	0.04
Lower leaves (untreated)	na	0.00	0.00	0.01	0.01	0.03

na - not analysed

Table 12 Identification of radioactivity in apple leaves treated with [pyridine-2, 6-¹⁴C]-acetamiprid

Days after application	0 days	7 days	14 days	28 days	62 days	90 days
	% TRR					
Acetamiprid (parent)	97.4	94.4	89.7	80.2	61.1	49.0
IM-2-1	0.6	1.0	2.1	4.8	10.5	15.6
IM-1-3	nd	0.6	0.8	1.0	1.3	1.1
IM-2-3	nd	0.1	0.3	0.6	1.5	1.3
IM-1-4	nd	0.1	0.1	0.3	1.5	2.3
IC-0	nd	0.1	0.2	0.4	1.1	2.2
IM-0-Glc	nd	0.1	0.5	1.9	5.9	8.3
Not identified (total)	2.0	2.2	2.9	4.2	7.7	11.1
Post extraction solids	0.1	0.7	1.7	3.8	4.7	6.5
Total	100.0	99.3	98.3	97.2	95.4	97.4
	Concentration (mg/kg parent equivalents)					
Parent	34.85	35.71	28.03	19.98	15.69	11.5
IM-2-1	0.21	0.39	0.67	1.19	2.70	3.64
IM-1-3	nd	0.21	0.24	0.24	0.34	0.25
IM-2-3	nd	0.05	0.09	0.14	0.39	0.29
IM-1-4	nd	0.02	0.04	0.07	0.38	0.53

Days after application	0 days	7 days	14 days	28 days	62 days	90 days
	% TRR					
IC-0	nd	0.03	0.07	0.1	0.29	0.51
IM-0-Glc	nd	0.04	0.14	0.46	1.52	1.92
Not identified (total)	0.69	0.84	0.88	1.04	2.0	2.61
Post extraction solids	0.04	0.26	0.54	0.94	1.21	1.46
Total	35.78	37.56	30.70	24.18	24.52	22.70

Single fruit were collected after 0, 14, 28 and 62 days. After surface washing with methanol, the apples were peeled and sectioned into flesh and core. Analysis was conducted according to the procedure described for leaves and radioactivity was measured by LSC. Characterisation and identification of the radioactivity was achieved by TLC, HPLC and LC-MS analysis.

The recovery of the radioactivity in fruit was greater than 97% with surface residues decreasing to about 12% TRR after 14 days and to about 6% TRR in fruit sampled 28 and 62 days after application. Residues in flesh increased to 48% TRR after 14 days and to about 78% TRR at the end of the 62-day study period. Total residues in the core did not exceed 5.5% TRR and were not measurable in the stalk.

Acetamiprid (parent) was the predominant residue, making up more than 79% TRR. Minor metabolites (IM-2-1 and IM-0-Glc) were found at maximum 3.7%TRR and 1.8%TRR, respectively.

Table 13 Characterisation and distribution of radioactivity in apple fruits after a single application of [pyridine-2, 6-¹⁴C]-acetamiprid

Days after application	0 days	14 days	28 days	62 days
	% TRR			
Surface wash	99.9	12.2	6.1	5.6
Peel	0.1	34.3	29.9	15.5
Flesh	na	48.1	58.9	78.1
Core	na	5.5	2.4	2.3
Stalk	na	0.1	0.0	0.1
Total	100.0	100.1	97.1	101.5
	Concentration (mg/kg parent equivalents)			
Surface wash	0.48	0.17	0.03	0.02
Peel	0.00	0.5	0.16	0.04
Flesh	*1	0.70	0.31	0.24
Core	*1	0.07	0.01	0.01
Stalk	*1	0.00	0.00	0.00
Total	0.48	1.44	0.52	0.31

na - not analysed

Table 14 Identification of radioactivity in apple fruit treated with [pyridine-2, 6-¹⁴C]-acetamiprid

Days after application	0 days	14 days	28 days	62 days
	% TRR			
Acetamiprid (parent)	97.1	89.9	79.2	80.8
IM-2-1	0.6	2.3	2.1	3.7
IM-1-3	nd	nd	nd	1.2
IM-2-3	nd	0.1	0.1	nd
IM-1-4	nd	0.4	1.1	1.1
IC-0	nd	0.2	0.3	0.9

Days after application	0 days	14 days	28 days	62 days
	% TRR			
IM-0-Glc	nd	0.5	0.6	1.8
Not identified (total)	2.2	2.2	3.2	9.3
Post extraction solids (incl stalks)	0.1	4.3	10.5	2.4
Total	100.0	99.8	96.9	101.1
	Concentration (mg/kg parent equivalents)			
Acetamiprid (parent)	0.47	1.30	0.42	0.24
IM-2-1	0.00	0.03	0.01	0.01
IM-1-3	nd	nd	nd	0.00
IM-2-3	nd	0.00	0.00	nd
IM-1-4	nd	0.00	0.01	0.00
IC-0	nd	0.00	0.00	0.00
IM-0-Glc	nd	0.01	0.00	0.01
Not identified (total)	0.01	0.02	0.02	0.03
Post extraction solids (incl stalks)	0.00	0.06	0.06	0.01
Total	0.48	1.44	0.52	0.31

nd: not detected

Eggplant

The metabolism of acetamiprid in eggplants was investigated by Saito, 1997 [Ref: Aceta_015]. Leaves and fruit from two eggplants cultivated in a greenhouse were treated once with [pyridine-2, 6-¹⁴C]-acetamiprid at a concentration of 95 ppm.

Leaf and fruit samples were taken 7 and 14 days after the application and washed with methanol (50 mL/leaf). The washed leaves were homogenized, extracted twice with 75% aqueous methanol and the extracts were combined and adjusted to 50% aqueous methanol before partitioning with n-hexane and extracted twice with methylene chloride. A final clean-up was achieved using XAD-4 column and an elution with methanol. Radioactivity in all the liquid fractions was determined by LSC. Characterisation and identification of the radioactivity was achieved by TLC, HPLC and LC-MS analysis.

The recoveries of the radioactivity in leaves and fruits were more than 99% at 7 and 14 days after application. Most of the radioactivity was found in the surface washes (79–75% TRR in leaves and 84–70% TRR in fruit, with 20–30% TRR present in the homogenised extracts. Translocated radioactivity was negligible.

Table 15 Characterisation and distribution of radioactivity in eggplant leaves and fruits after a single foliar application of [pyridine-2, 6-¹⁴C]-acetamiprid

Days after application	7 days	14 days	7 days	14 days
	% TRR		mg/kg parent equivalents	
	Foliar application			
Surface wash	79.0	74.4	17.74	14.85
Homogenate (extracted)	20.2	25.1	4.53	5.02
Treated leaves (total)	99.2	99.5	22.27	19.87
Untreated leaves	--	--	0.01	0.01
Untreated fruit	--	--	0.00	0.00
	Fruit application			
Surface wash	84.2	69.9	0.34	0.82

Days after application	7 days	14 days	7 days	14 days
	% TRR		mg/kg parent equivalents	
Homogenate (extracted)	21.6	30.1	0.09	0.35
Treated fruits (total)	105.8	99.9	0.42	1.17
Untreated leaves	--	--	0.01	0.00
Untreated fruit	--	--	-	0.00

Acetamiprid was the major residue, making up about 85-89% TRR in leaves and 94-95% TRR in fruit. Of the three identified metabolites, IM-2-1 was present in fruit at 0.4% TRR and 1.8% TRR in leaves and the IM-0 metabolite and its glycoside were identified in leaves at 0.6% TRR and 4.6% TRR respectively.

Table 16 Identification of radioactivity in eggplant fruit and leaves treated with [pyridine-2, 6-¹⁴C]-acetamiprid

Compound	Foliar application				Fruit application			
	% TRR		mg/kg parent equivalents		% TRR		mg/kg parent equivalents	
Days after application	7 days	14 days	7 days	14 days	7 days	14 days	7 days	14 days
Acetamiprid	89.2	85.2	20.02	17.02	95.4	93.9	0.38	1.1
IM-2-1	1.0	1.8	0.22	0.35	0.4	nd	0.00	nd
IM-0	0.4	0.6	0.10	0.12	nd	nd	nd	nd
IM-0-Glc	2.4	4.6	0.54	0.92	nd	nd	nd	nd
Not identified (total)	5.7	6.4	1.27	1.28	2.1	0.17	0.01	0.02
Post extraction solids	0.8	0.9	0.18	0.19	1.5	2.6	0.01	0.03
Total	99.5	99.6	22.33	19.89	99.4	98.1	0.40	1.15

Cabbage

In a study conducted by Saito, 1997 [Ref: Aceta_016], cabbage plants were grown under greenhouse conditions and treated with [pyridine-2, 6-¹⁴C]-acetamiprid either as a foliar spray or as a pre-plant soil application. For the foliar application, cabbage plants at the 15-leaf stage were sprayed once with a 201 ppm solution of ¹⁴C-acetamiprid at a rate equivalent to 0.284 kg ai/ha. For the soil application, a 2.0 gram granule containing 42 mg ¹⁴C-acetamiprid was placed in the planting hole at the time the cabbage seedlings (6-7 leaves) were transplanted, approximating an application rate of 5.9 kg ai/ha.

Samples were collected at day 0, 7, 14, 21, 28 and 63 after the foliar application and at day 7, 14 and 28 after the soil application. Leaves (and heads at day-63) were surface washed with methanol, the washed leaves were extracted with 75% methanolic water, and the extracts were subjected to a liquid-liquid partition with n-hexane and methylene chloride. A final clean-up was achieved using XAD-4 column and elution with methanol. Radioactivity in all the liquid fractions was determined by LSC and characterisation of the radioactivity was achieved by TLC, HPLC and LC-MS analysis.

Total recovery rates were 95-100% for the foliar applications, with surface residues decreasing to 30–50% TRR in the 28 days after application and to about 12% at the end of the study period (day-63). Residues in the homogenate increased accordingly, from 15% TRR (day-0) to 83.5% TRR (day-63).

Table 17 Characterisation and distribution of radioactivity in cabbage after foliar application of [pyridine-2, 6-¹⁴C]-acetamiprid

Days after application	0 days	7 days	14 days	21 days	28 days	63 days
	% TRR					
Surface wash	85.1	36.5	49.0	37.2	30.8	12.1

Days after application	0 days	7 days	14 days	21 days	28 days	63 days
	% TRR					
Leaf homogenate (extracted)	14.4	58.8	43.1	47.4	61.3	80.4
Post extraction solids (leaf)	0.5	1.5	2.6	4.9	3.0	3.1
Total (leaf)	100.0	96.8	94.7	89.5	95.2	95.5
	Concentration (mg/kg parent equivalents)					
Surface wash	6.72	1.83	1.73	1.12	0.74	0.33
Leaf homogenate (extracted)	1.14	2.93	1.53	1.44	1.47	2.22
Post extraction solids (leaf)	0.04	0.08	0.09	0.15	0.07	0.08
Total (leaf)	7.9	4.84	3.36	2.71	2.28	2.63
Head	na	na	na	na	na	0.05
Root homogenate (extracted)	na	0.07	0.01	0.04	0.02	0.01
Post extraction solids (root)		0.02	0.02	0.02	0.04	0.01
Total (root)		0.09	0.03	0.06	0.06	0.02

na - Not analysed

For the soil application, the recovery of the radioactivity in cabbage leaves decreased from 97.2% down to 72.6% and the recovery in roots was 97–98% TRR. The radioactivity was readily translocated into leaves, reaching levels of about 2–3× the root concentrations during the 28-day study period.

Table 18 Characterisation and distribution of radioactive residues in cabbage after soil application of [pyridine-2, 6-¹⁴C]-acetamiprid

	% TRR			Concentration (mg/kg parent equivalents)		
Days after application	7 days	14 days	28 days	7 days	14 days	28 days
Leaf homogenate (extracted)	96.4	94.2	66.7	102.3	65.03	26.7
Post extraction solids (leaf)	0.8	3.2	5.8	0.08	3.2	5.8
Total (leaf)	97.2	97.5	72.6	103.2	67.07	28.43
Root homogenate (extracted)	85.3	81.7	71.7	38.23	30.42	6.95
Post extraction solids (root)	11.6	16	25.6	4.87	5.84	2.44
Total (root)	96.9	97.8	97.3	43.1	36.26	9.38

Acetamiprid was the major residue component in leaves following foliar application, present at about 67–91% TRR with residues of the IM-2-1 metabolite increasing over the study period to a maximum of 7.2% TRR (day-63). No parent residues were measured in cabbage heads (at day-63), with only the IC-0 metabolite being identified, at about 46% TRR (0.03 mg/kg).

Table 19 Identification of radioactivity in cabbage leaves treated with [pyridine-2, 6-¹⁴C]-acetamiprid

	Plants (tops)					Wrapper leaves	Head ^a
Days after application	0 days	7 days	14 days	21 days	28 days	63 days	63 days
	% TRR						
Parent	84.6	90.8	83.9	76.5	78.8	66.7	nd
IM-2-1	0.1	1.6	2.0	3.4	4.7	7.2	nd
IM-1-3	nd	0.1	0.1	0.2	0.2	nd	nd

	Plants (tops)					Wrapper leaves	Head ^a
Days after application	0 days	7 days	14 days	21 days	28 days	63 days	63 days
IM-2-3	nd	0.0	0.0	0.0	0.1	nd	nd
IM-0	nd	0.1	0.3	0.3	0.1	nd	nd
IC-0	nd	0.3	0.2	0.6	0.9	1.6	45.6
IM-0-Glc	nd	0.4	0.3	0.8	1.5	2.3	nd
Not identified	14.7	2.1	5.7	4.0	7.2	16.2	35.7
Post extraction solids	0.5	1.5	2.6	4.9	3.0	3.1	4.6
Total	100.0	97.1	95.1	90.7	96.6	97.1	85.9
Concentrations (mg/kg parent equivalents)							
Parent	6.69	4.54	2.97	2.32	1.89	1.84	nd
IM-2-1	0.01	0.08	0.07	0.10	0.11	0.20	nd
IM-1-3	nd	0.01	0.00	0.00	0.01	nd	nd
IM-2-3	nd	0.00	0.00	0.00	0.00	nd	nd
IM-0	nd	0.00	0.01	0.01	0.00	nd	nd
IC-0	nd	0.02	0.01	0.02	0.02	0.04	0.03
IM-0-Glc	nd	0.02	0.01	0.02	0.04	0.06	nd
Not identified	1.17	0.1	0.2	0.12	0.17	0.45	0.01
Post extraction solids	0.04	0.08	0.09	0.15	0.07	0.08	0.00
Total	7.90	4.86	3.37	2.75	2.32	2.68	0.06

^a without wrapper leaves

Acetamiprid was also the only major residue in leaves and roots from the soil application, present initially at about 90% TRR (leaves) and 78% TRR (roots), decreasing to 60% TRR (leaves) and 50% TRR (roots) after 28 days. The IM-1-4 metabolite was the only other identified metabolite present at more than 5% TRR, being found in roots after 28 days.

Table 20 Identification of radioactivity in cabbage leaves and roots after soil application of [pyridine-2, 6-¹⁴C]-acetamiprid

	Leaves			Roots		
Days after application	7 days	14 days	28 days	7 days	14 days	28 days
	% TRR					
Acetamiprid (parent)	90.2	86.5	60.5	77.6	67.7	50.3
IM-2-1	0.8	1.9	2.0	ND	N.D	ND
IM-1-4	0.2	0.9	1.3	0.5	2.6	7.6
IC-0	ND	0.8	0.7	ND	ND	ND
IM-0-Glc	ND	0.6	0.4	ND	ND	ND
Not identified	5.2	2.6	1.8	7.2	1.5	3.3
Post extraction solids	0.8	3.2	5.8	11.6	16.0	25.6
Total	97.2	96.5	72.4	96.9	87.8	87.0
Compound	Concentration (mg/kg parent equivalents)					
Acetamiprid (parent)	93.11	58.01	17.20	33.45	24.56	4.72
IM-2-1	0.84	1.25	0.56	ND	ND	ND
IM-1-4	0.18	0.60	0.37	0.21	0.94	0.71
IC-0	ND	0.53	0.19	ND	ND	ND
IM-0-Glc	ND	0.41	0.13	N.D	ND	ND

	Leaves			Roots		
Days after application	7 days	14 days	28 days	7 days	14 days	28 days
Not identified	5.33	1.7	0.51	3.1	0.53	0.3
Post extraction solids	0.90	2.04	1.73	4.87	5.84	2.44
Total	100.37	64.56	20.69	41.63	31.87	8.19

Cabbage [CN label]

The metabolism of acetamiprid in cabbage was further investigated by Kawai, 1997 [Ref: Aceta_017] in a study involving a foliar application of CN-¹⁴C-labelled acetamiprid at a concentration of 199 ppm (equivalent to 0.3 kg ai/1500 L/ha) to cabbage plants at about the 15-leaf stage.

Leaf samples were collected at day 0, 7, 14, 28 and 63 (maturity) and surface washed with methanol. The washed leaves were extracted with 75% methanolic water, and the extracts were partitioned with n-hexane. The methanol of the aqueous phase was removed and aqueous phase was centrifuged and filtered. The filtrate was treated with ODS solid phase extraction cartridge. Radioactivity in all the liquid fractions was determined by LSC. Characterisation and identification of the radioactivity was achieved by TLC, HPLC and LC-MS analysis.

The distribution of radioactivity in surface wash decreased from an initial level of about 86% TRR down to about 16% TRR after 63 days, with residues in the leaf homogenates increasing accordingly, to about 78% TRR at the end of the study period. Residues in roots and in cabbage heads did not exceed 0.01 mg/kg acetamiprid equivalents.

Table 21 Characterisation and distribution of radioactivity in cabbages after application of CN-¹⁴C-acetamiprid

Days after application	0 days	7 days	14 days	28 days	63 days
	% TRR				
Surface wash	85.78	49.24	35.83	26.1	15.77
Leaf homogenate	14.13	49.88	62.49	70.36	77.85
Post extraction solids (leaf)	0.1	0.88	1.67	3.54	6.38
Total (leaf)	100	100	100	100	100
	Concentration (mg/kg parent equivalents)				
Surface wash	4.34	2.38	1.39	1.3	0.49
Leaf homogenate	0.72	2.41	2.42	3.51	2.43
Post extraction solids (leaf)	0.00	0.04	0.06	0.18	0.2
Total (leaf)	5.06	4.98	3.87	4.04	3.20
Head	na	na	na	na	0.01
Root	na	0.02	0.04	0.03	0.01

na - not analysed

Acetamiprid was the major residue component in leaves, making up more than 98% TRR during the 7 days after application and decreasing to about 65% at day-68. Additional metabolites were IS-1-1, present initially at 2.4% TRR increasing to 15.6% of TRR and IS-1-1, found in later samples at up to about 10% TRR.

Table 22 Identification of radioactivity in cabbage leaves after a foliar application of CN-¹⁴C-acetamiprid

Days after application	0 days	7 days	14 days	28 days	63 days
	% TRR				
Parent	100.15	98.3	88.14	78.26	65.17
IM-2-1	nd	0.48	2.03	4.31	4.14
IS-1-1	nd	2.39	5.73	9.43	15.56
IS-2-1	nd	0.62	1.44	3.68	10.51
Not identified	1.05	0.45	1.03	0.56	0.94
Post extraction solids	0.10	0.88	1.67	3.54	6.38
Total	101.3	103.12	100.01	99.78	102.7
	Concentrations (mg/kg parent equivalents)				
Parent	5.07	4.75	3.41	3.90	2.03
IM-2-1	nd	0.02	0.08	0.21	0.13
IS-1-1	nd	0.12	0.22	0.47	0.48
IS-2-1	nd	0.03	0.06	0.18	0.33
Not identified	0.06	0.02	0.04	0.03	0.03
Post extraction solids	0.00	0.04	0.06	0.18	0.2
Total	5.13	4.98	3.87	4.97	3.2

Carrots

The metabolism of acetamiprid in carrots was investigated by Austin, 1997 [Ref: Aceta_018]. In this study, [pyridine-2, 6-¹⁴C]-acetamiprid was applied as a foliar spray to carrots, 2 months after sowing and again about 5 weeks later, at a rate equivalent to 100 g ai/ha.

Samples of carrots were taken just before the second application ('Interim samples') and at harvest, 2 weeks after the second application. After washing in water, the carrots were peeled and topped and radioactive residues extracted with acetone and with aqueous acetone (2×) with the peel samples also being treated with sodium chloride:methanol, aqueous ammonia and formic acid to improve extraction performance. Radioactivity in all fractions was measured by LSC and characterised by HPLC.

Total radioactivity in carrots at harvest was less than 0.1 mg/kg acetamiprid equivalents, mostly in the tops, with 78–90% (tops) and 66–88% (flesh) being extracted in acetone solvents. Extraction rates for the peel were lower, 52–65% TRR from the acetone extractions and a further 3% TRR following the more extensive extraction procedures.

Table 23 Total radioactive residues in carrots after foliar applications with [pyridine-2, 6-¹⁴C]-acetamiprid

Sampling time	Plant parts	TRR (mg/kg)	Extract		Unextracted	
			mg/kg	% TRR	mg/kg	% TRR
Interim (5 weeks after 1 st application)	Tops	0.0868	0.0674	77.56	0.0195	22.44
	Peel	0.0372	0.0198	52.33	0.0177	47.67
	Flesh	0.0172	0.0114	66.26	0.0058	33.74
Harvest (2 weeks after 2 nd application)	Tops	0.4463	0.4006	89.77	0.0456	10.23
	Peel	0.1347	0.0877	65.10	0.0450	33.37
	Flesh	0.0549	0.0481	87.6	0.0068	12.4
	Total carrot	0.0842	0.0626	79.36	0.0208	20.08

N.B. Total carrot is calculated on ratios of flesh to peel

In the interim harvest top samples the main components found were IM-1-4 (42.8% TRR) and IM-0-Glc (8.6% TRR). Twelve other components, five of which were identified by co-chromatography, were also found at levels < 5% TRR. In the peel, a number of identified and unknown metabolites were present, with one exception, less than 10% TRR while in the flesh, metabolites found at more than 10% TRR were IM-0 (about 14% TRR) and IC-0 (about 11% TRR). Acetamiprid was also measured in the tops and flesh at low levels (0.17% TRR and 0.62% TRR respectively).

Table 24 Identification of radioactivity in interim harvested (immature) carrots, 5 weeks after a single foliar application of [pyridine-2, 6-¹⁴C]-acetamiprid

	Interim Tops	Interim Peel	Interim Flesh	Interim Total
	% TRR (0.086 mg/kg)	% TRR (0.037 mg/kg)	% TRR (0.017 mg/kg)	% TRR (0.14 mg/kg)
Acetamiprid	0.17	-	0.62	0.2
IC-0	0.58	3.39	11.32	2.6
IM-1-4	42.8	4.45	7.22	28.3
IM-0-GLC	8.64	7.63	0.48	7.4
IM-0	2.4	6.23	13.77	4.8
IM-2-3	4.09	7.5	4.88	5.1
IM-1-2	-	1.01	-	0.3
IM-2-1	0.16	-	1.33	0.3
Unknown	3.21	1.2	-	2.3
Unknown	0.31	5.74	4.77	2.3
Unknown	-	13.76	-	3.6
Unknown	4.31	-	8.90	3.7
Unknown	2.55	-	-	1.6
Unknown	4.22	-	2.16	2.9
Unknown	3.88	-	2.13	2.6
Unknown	-	1.42	8.67	1.4
Unknown	0.24	-	-	0.1
Total characterised	77.55	52.33	66.26	69.5
Total identified	69.22	50.91	53.30	62.4
Post extraction solids	22.45	47.67	33.74	30.5

In the final harvest samples (taken 2 weeks after the second application), the main components found in the carrot tops were IM-0-Glc (33% TRR), parent acetamiprid (27% TRR) and IM-1-4 (15% TRR) with no other components exceeding 6% TRR. In the carrot peel the main components were acetamiprid (30% TRR) and IC-0 (17% TRR) with a similar pattern occurring in carrot flesh, with acetamiprid (34% TRR) and the IC-0 metabolite (31% TRR) being the major residues.

In the total carrot the major components found were acetamiprid (29% TRR) and IC-0 (26% TRR) with other minor metabolites each present at less than 6% TRR.

Table 25 Identification of radioactivity in mature carrots, 2 weeks after two foliar applications with [pyridine-2, 6-¹⁴C]-acetamiprid

	Carrot Tops	Carrot Peel	Carrot Flesh	Carrot Total
	% TRR (0.446 mg/kg)	% TRR (0.135 mg/kg)	% TRR (0.055 mg/kg)	% TRR (0.084 mg/kg)
acetamiprid	26.85	30.05	34.09	32.61
IC-0	1.45	16.61	31.10	25.80
IM-1-4	14.70	2.96	6.34	5.10

	Carrot Tops	Carrot Peel	Carrot Flesh	Carrot Total
	% TRR (0.446 mg/kg)	% TRR (0.135 mg/kg)	% TRR (0.055 mg/kg)	% TRR (0.084 mg/kg)
IM-0-Glc	32.93	6.25	4.81	5.34
IM-0	0.13	2.27	2.46	2.39
IM-2-3	4.97	0.23	-	-
IM-1-2	0.11	-	-	-
IM-1-3	0.10	0.21	-	-
IM-2-1	5.92	4.11	4.15	4.14
Unknown	0.00	2.40	3.08	2.83
Unknown	0.40	0.00	0.70	0.44
Unknown	0.50	-	-	-
Unknown	1.70	-	-	-
Total Characterised	89.77	65.10	86.73	78.64
Total Identified	87.16	62.70	82.95	75.37
Post extraction solids	10.23	34.90	12.40	20.64

Cotton

The metabolism of acetamiprid in cotton was investigated by Miller, 1999 [Ref: Aceta_019] following four foliar applications of [pyridine-2, 6-¹⁴C]-acetamiprid at rates equivalent to 0.127 kg ai/ha (1×) and 1.27 kg ai/ha (10×). Applications were applied at weekly intervals from about 12 weeks after planting and mature cotton samples were taken 28 days and 14 days after the last application.

Cotton seed (undelinted), lint, leaves and gin trash (stems, petioles, small leaves and closed bolls) and large mature leaves (probably present at the time of application) were analysed for total radioactive residues by combustion and LSC.

Total radioactive residues (TRR) were highest in the mature leaves and decreased between the 14-day pre-harvest interval (PHI) and 28-day PHI. Cotton seed TRRs were lower than the TRRs observed in gin trash.

Table 26 Total radioactive residues (TRR) in cotton after four foliar applications with [pyridine-2, 6-¹⁴C]-acetamiprid

Crop	PHI	Application rate	TRR (mg/kg)
Seed	14	1×	1.5
	28	1×	1.11
	28	10×	14.39
Gin trash	14	1×	2.81
	28	1×	1.56
	28	10×	18.97
Lint	14	1×	1.39
	28	1×	2.74
	28	10×	6.10
Leaves	14	1×	12.94
	28	1×	6.72
	28	10×	74.81

The raw agricultural commodities (RACs) gin trash and cotton seed at the 1× application rate were extracted with methanol:water (1:1), 0.1 N acetic acid in methanol, and 0.1 N ammonium hydroxide in methanol.

A high percentage of the radioactive residue in gin trash was extracted with methanol/water. Cotton seed required more acetic or basic conditions to extract the residues. Total extracted residues were higher for gin trash than for cotton seed.

The relative amounts of residues extracted with each solvent did not differ greatly between the 14-day and 28-day PHI for either gin trash or cotton seed. Extracts containing > 1% of the TRR were concentrated and analysed by HPLC.

Table 27 Characterisation and distribution of radioactivity in cotton seed and gin trash after four foliar applications of [pyridine-2, 6-¹⁴C]-acetamiprid

Matrix	Seed				Gin trash			
Days after application	14 days		28 days		14 days		28 days	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
MeOH/Water	22.3	0.33	22.2	0.24	78.3	2.2	91.0	1.42
0.1 N Acetic acid/MeOH	43.9	0.66	29.6	0.33	8.0	0.23	5.5	0.09
0.1 N NH ₄ OH/MeOH	10.3	0.16	10.0	0.11	1.0	0.03	0.8	0.01
Total Extracted	76.5	1.15	61.8	0.69	87.3	2.45	97.2	1.51
Post extraction solid	21.5	0.32	19.9	0.22	6.4	0.18	8.5	0.13
Total	97.8	1.47	81.6	0.91	93.7	2.63	105.6	1.65

Acetamiprid was the major residue identified in gin trash, making up 45–50% TRR. The metabolites IM-1-4, IM-0-Glc, IC-0, and IM-2-1 were also identified at significant levels in both the 14- and 28-day gin trash. Several unidentified peaks were also detected in each extract, all less than 0.03 ppm (\leq 1% of the TRR). The metabolite profiles were also very similar for the 14-day and 28-day PHI seed.

The major metabolite observed in cotton seed was IC-0 (45% TRR decreasing to 24% TRR after 28 days). The parent acetamiprid and the metabolites IM-1-3, IM-2-1 and IC-0-ME were also identified at significant levels and several unidentified peaks were also detected in each extract, all less than 0.04 ppm (< 2.5% of the TRR). The higher levels of acetamiprid observed in gin trash is most likely due to the direct exposure of gin trash to acetamiprid at application.

Table 28 Identification of radioactivity present at > 1% TRR in cotton seed and gin trash after four foliar applications with [pyridine-2, 6-¹⁴C]-acetamiprid

Sample	Cotton seed				Gin trash			
Days after application	14 days		28 days		14 days		28 days	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
acetamiprid	3.1	0.05	4.9	0.06	50.4	1.42	45.2	0.71
IC-0	45.7	0.69	24.2	0.27	3.9	0.11	5.2	0.08
IM-1-4	--	--	--	--	2.9	0.08	2.4	0.04
IM-0-Glc	0.4	0.01	--	--	5.0	0.14	5.0	0.08
IM-1-3	1.0	0.02	1.2	0.01	--	--	--	--
IM-2-1	8.2	0.12	6.0	0.07	9.4	0.26	8.4	0.13
IC-0-Me ^a	16.1	0.24	22.8	0.26	--	--	--	--
Total identified	74.5	1.13	59.1	0.67	71.6	2.01	66.2	1.04
Total characterised	18.3	0.28	20.5	0.24	7.8	0.22	10.3	0.17
Post extraction solids	1.1	0.02	0.8	0.01	1.7	0.05	0.8	0.01

^a Total IC-0-Me (bound and extracted) – possibly an artefact of methanolic extraction since not observed in cotton seed extracted with 0.1 N acetic acid in acetonitrile

In summary, the predominant residue in plant part exposed to foliar treatments is the parent compound, with low levels of the IM-2-1 metabolite being a common component in the plants

studied, but generally at levels of 10% TRR or less. Acetamiprid is also the predominant residue in cabbage and carrot roots following soil treatments. The other significant metabolite found in plant parts not directly treated was the IC-0 cleavage product, found in cabbage heads (0.03 mg/kg), carrot roots (0.04 mg/kg) and cotton seed (up to 0.69 mg/kg).

The proposed metabolic breakdown of acetamiprid in plants following foliar application involves demethylation to IM-2-1 and further degradation to IC-0-Glc, or conversion of the parent compound to IM-0, with subsequent conjugation with glucose to form the IM-0-Glc. Degradation can also involve formation of the IM-1-2 metabolite which rapidly degrades to IM-1-3 and either IM-2-3 or IM-1-4, both of which degrade to IC-0.

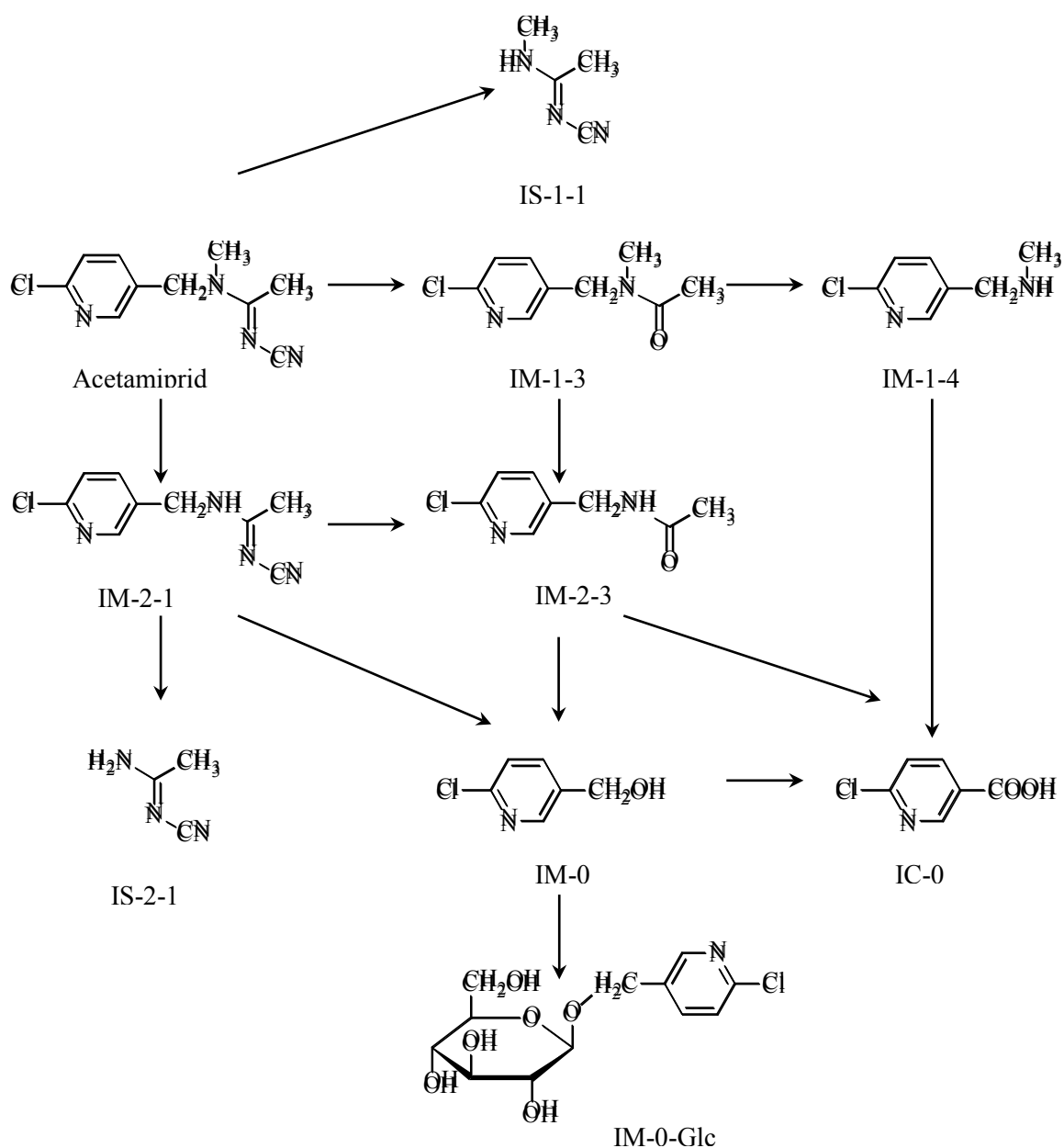


Figure 3 Proposed metabolic pathways of Acetamiprid in apple, cabbage or eggplant

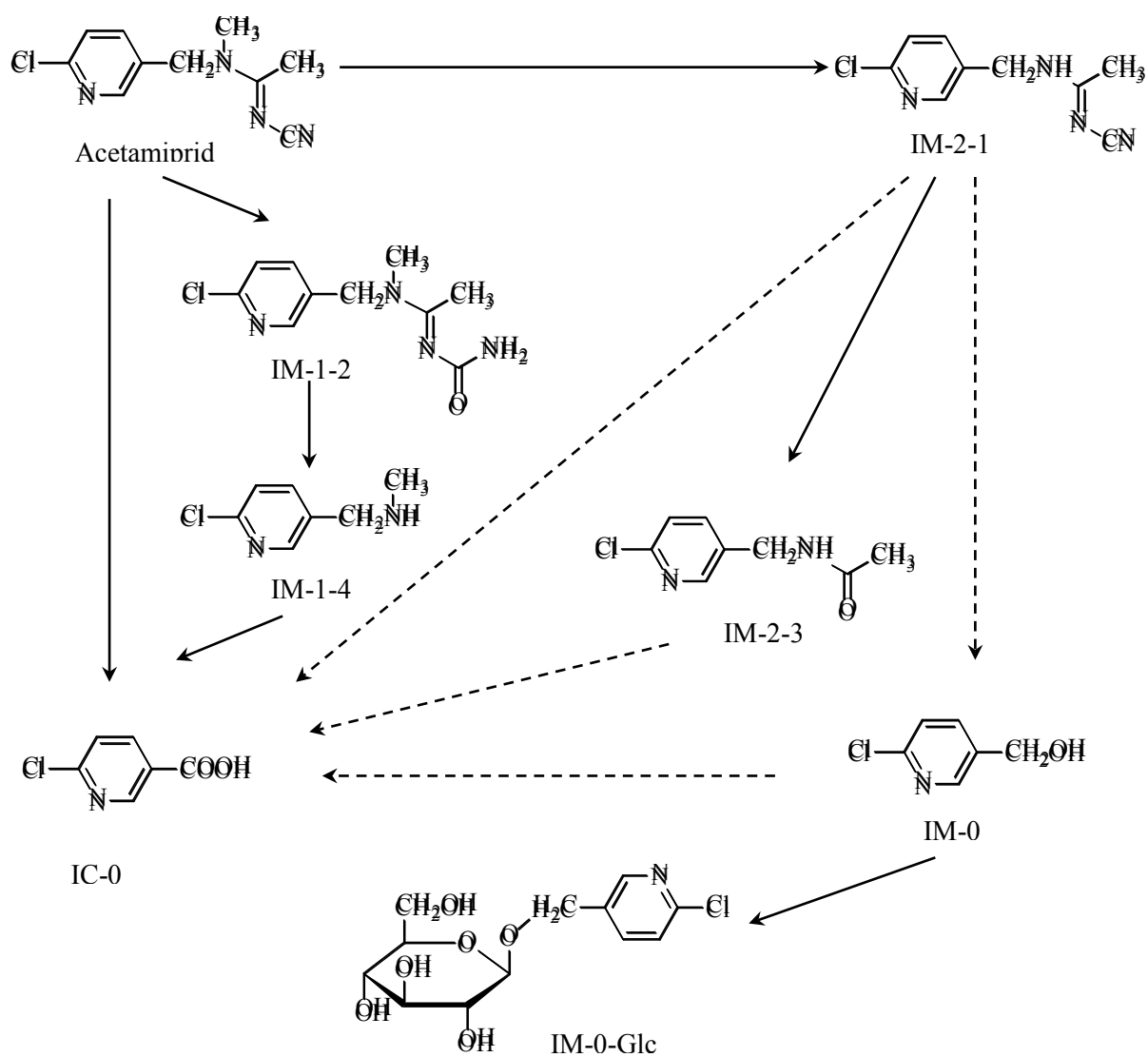


Figure 4 Proposed metabolic pathways of Acetamiprid in carrots

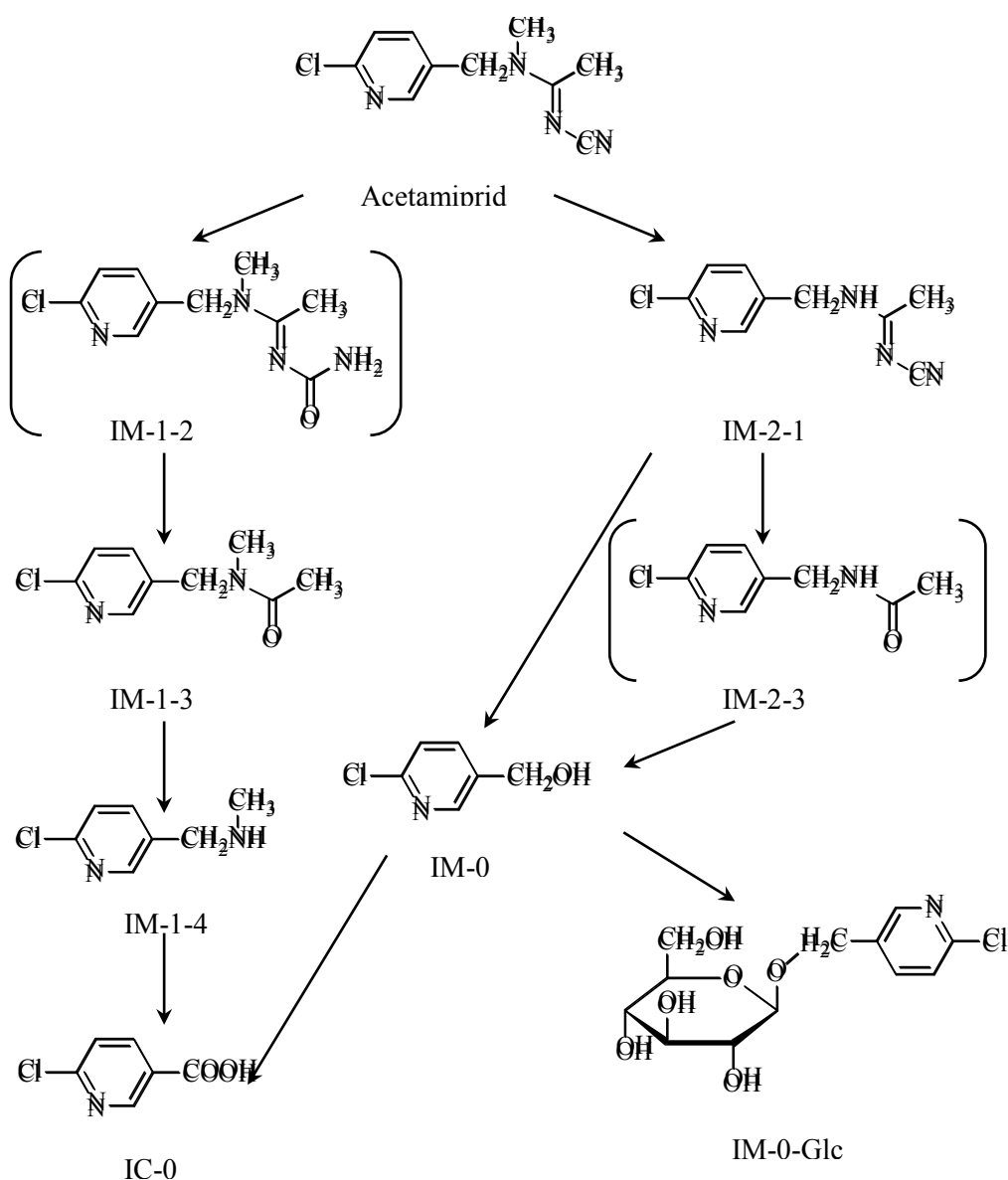


Figure 5 Proposed metabolic pathways of Acetamiprid in cotton

ENVIRONMENTAL FATE IN SOIL

Soil metabolism

Aerobic soil metabolism

An aerobic soil metabolism study was conducted by Feung, 1998 [Ref: Aceta_020] using [^{14}C -2, 6-pyridine]-acetamiprid. Study details and results are summarised below:

Test material: [^{14}C -2, 6-pyridine]-acetamiprid
 Duration: 365 days
 Moisture: 10.14%
 pH: 6.9

Dose rate: 0.672 kg ai/ha
 Temp: 25 °C
 Soil: loamy sand
 Percent Organic Matter: 1.2%

Half-life (parent):	8.2 days
¹⁴ C accountability:	91.0–101%
% acetamiprid remaining after	365 days: 0.79%
% mineralisation:	19.1% after 270 days, 11.7% after 365 days
% unextracted:	20.4% after 365 days

Metabolites	Max (% AR)	Day
IM-1-3	2.54%	60
IM-1-4	73.29%	120
IC-0	4.73%	60

The estimated aerobic soil metabolism half-life for acetamiprid at 25 °C was about 8.2 days with a significant amount of ¹⁴CO₂ (up to about 19% of the applied dose) being detected over the course of the study. The metabolite IM-1-4 was a major component of the radioactive residue, increasing to about 73% after 120 days and slowly decreasing thereafter. Two minor metabolites, IM-1-3 and IC-0 were also identified but at less than 5% of the applied dose during the study.

Rate of aerobic soil degradation

The rate of degradation of [¹⁴C-2, 6-pyridine]-acetamiprid under aerobic conditions was investigated by Burr, 1997 [Ref: Aceta_021] in three soils incubated at 20 °C and one soil at 10 °C in the dark. The soils selected were a sandy loam soil, a clay loam soil and a high pH calcareous silty clay loam. Flasks of the three soils were treated with the equivalent of 0.1 kg ai/ha and incubated in the dark for intervals up to 6 months. Volatile products were captured and soils were extracted with various solvents and residues were analysed by HPLC, TLC and LC-MS. Soil properties and study results are summarised below:

Table 29 Aerobic degradation of [pyridine-2, 6-¹⁴C]-acetamiprid in soils

Soil Reference	96/37		96/46	96/47
Soil type	Clay loam		Sandy loam	Silty clay loam
Soil pH	7.4		5.6	7.9-8.5
Organic carbon (%)	2.1		3.5	4.3
Incubation temperature	10 °C	20 °C		
Mean overall recovery (%) of applied radioactivity	97.2	96.9	97.6	97.6
DT ₅₀ (days)				
acetamiprid	7.92	5.45	2.56	0.84
IM-4			252	4.2
IC-0				4.8
DT ₉₀ (days)				
acetamiprid	49.5	34.6	15.0	3.64
IM-4				13.5
IC-0				15.6
% Mineralization at 182 days	3.5	10.4	23.9	59.5
% Unextracted at 182 days	24.3	38.2	26.3	17.8
Maximum % AR				
IM-1-3	1.9 (178 days)	3.0 (28 days)	3.1 (7 days)	1.6 (7 days)
IM-1-4	71.9 (30 days)	53.9 (14 days)	71.8 (14 days)	65.3 (3 days)
IC-0	3.3 (178 days)	10.5 (120 days)	3.6 (120 days)	10.4 (7 days)
IM-1-5	nd	nd	nd	20.0 (13 days)

nd: not detected

Estimated aerobic soil metabolism half-lives for acetamiprid at 20 °C ranged 0.84–7.92 days. A significant amounts of $^{14}\text{CO}_2$ (10.37–59.47%) of the applied dose was detected at 20 °C after 6 months.

In all three soils acetamiprid degraded rapidly. IM-1-4 was the major metabolite in all soils (maximum 54–72% AR). IC-0 was also observed as a significant metabolite (> 10% of applied radioactivity) in two soils (the clay loam at 20 °C and the silty clay loam at 20 °C). In addition, in the silty clay loam, a unique metabolite, found at up to 20% AR was identified as IM-1-5.

Rotational crops

The metabolism of acetamiprid in rotational crops was investigated by Mislanker, 199 [Ref: Aceta_022]. In this study, [pyridine-2, 6- ^{14}C]-acetamiprid was applied as a surface spray to bare sandy loam soil at a rate of 0.74 kg ai/ha and covered with additional soil. The soil used in this study had an organic matter content of 1.8%, a sand:silt:clay ratio of 70:19:11, a pH of 6.5 and a CEC of 9.6 meq/100g. A representative root crop (radish), a leafy vegetable (lettuce) and a small grain (sorghum or wheat) were planted in freshly cultivated soil (to a depth of 8 cm), 30, 60, 120 and 365 days after application. Samples of wheat and sorghum forage were collected at about half-maturity and all crops were also sampled at maturity (mustard and radish leaves, radish roots, sorghum and wheat grain and straw).

The samples from each rotation interval were processed and subsamples of each matrix were analysed for total radioactive residues (TRR) by combustion followed by liquid scintillation analysis. The organosoluble extracts were analysed by reverse phase HPLC. The LOQ for HPLC analyses was less than 0.001 ppm.

Total radioactive residues in samples from all plant-back intervals were less than 0.1 mg/kg except sorghum fodder from the 60-day plant-back interval (0.115 mg/kg). Acetamiprid was not found in any of the matrices. All the metabolites were less than 0.05 mg/kg in any matrix at any rotation. The metabolites which accounted for more than 0.01 mg/kg were identified by retention time comparison with authentic standards and confirmed by mass spectral analysis.

IM-1-4 was the major metabolite found in most of the matrices at 30, 60 and 120 DAT intervals, ranging from 0.001 mg/kg in 60 DAT radish root to a maximum of 0.04 mg/kg in sorghum forage. IM-0-Glc and IC-0 accounted for a maximum of 0.01 mg/kg (30 DAT sorghum forage) and 0.014 mg/kg (60 DAT sorghum fodder), respectively. In all other matrices these metabolites were less than 0.01 ppm.

Table 30 Acetamiprid and metabolite residues present in rotational crops after bare ground application of [pyridine-2, 6- ^{14}C]-acetamiprid

	Rotation (DAT) ^a	Harvest (DAP) ^b	TRR	Extracted		IM-1-4		IM-0-Glc		IC-0		Not identified		Total	
Sample				mg/k g	%TRR	mg/kg	%TR R	mg/kg	%TR R	mg/kg	%TR R	mg/kg	%TR R	mg/k g	%TR R
Mustard leaf	30	57	0.04 6	0.038	82.74	0.016	34.7	0.006	12.21	0.011	24.19	0.004	8.63	0.037	79.73
	60	63	0.05	0.04	79.14	0.021	42.25	0.004	8.81	0.007	14.46	0.004	7.87	0.037	73.38
	120	47	0.03 5	0.03	84.61	0.02	56.65	0.001	1.53	0.002	5.28	0.005	13.73	0.027	77.19
	365	39	0.01 7	0.006	35.86	0.001	2.94	0.002	9.9	0.000	2.87	0.002	7.98	0.004	23.69
Radish leaf	30	57	0.06 2	0.048	77.17	0.033	52.76	0.009	15.23	0.004	6.22	0.006	9.9	0.052	84.11
	60	68	0.01 9	0.009	50.93	0.004	21.43	0.002	9.61	0.001	7.44	0.001	4.26	0.008	42.74
	120	47	0.04 7	0.33	70.99	0.023	49.82	0.003	6.2			0.007	15.02	0.033	71.04

	Rotation (DAT) ^a	Harvest (DAP) ^b	TRR	Extracted		IM-1-4		IM-0-Glc		IC-0		Not identified		Total	
Sample				mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
	365	39	0.026	0.016	61.96	0.006	23.27	0.003	13.05	0.002	6.55	0.003	9.73	0.014	52.6
Radish root	30	57	0.044	0.027	61.71	0.008	17.3	0.002	4.52	0.003	6.65	0.010	23.42	0.023	51.9
	60	68	0.009	0.004	40.71	< 0.001	4.09	< 0.001	5.39	< 0.001	2.84	0.001	5.97	0.002	18.29
	120	47	0.012	0.008	63.95	0.001	10.85	< 0.001	1.11	< 0.001	2.75	0.002	17.16	0.003	31.87
	365	39	0.007	na	na	na	na	na	na	na	na	na	na	na	na
Forage	30 (S)	50	0.079	0.059	74.99	0.04	50.39	0.01	12.89			0.005 ^c	6.18 ^c	0.055	69.47
	60 (S)	69	0.045	0.029	64.48	0.018	39.78	0.005	10.89	< 0.001	0.74			0.023	51.41
	120 (W)	186	0.028	0.018	63.39	0.01	36.73					0.002	5.69	0.012	42.42
	365 (S)	49	0.015	0.01	67.21	0.006	41.93	< 0.001	2.68	0.001	3.68			0.007	48.47
Fodder	30 (S)	124	0.078	0.034	43.39	0.025	31.98	0.001	1.33	0.003	3.72			0.029	37.03
	60 (S)	109	0.115	0.061	52.96	0.029	25.14			0.014	12.58	0.008 0.001 ^c	7.31 0.87 ^c	0.053	45.9
	120 (W)	249	0.064	0.028	44.410	0.007	10.98	0.001	2.15	0.011	17.96			0.019	31.09
	365 (S)	130	0.018	0.009	49.030	0.005	28.09	0.001	5.5	0.001	4.55			0.007	38.04
Grain	30 (S)	124	0.01	0.002	21.590	na	na	na	na	na	na	na	na	na	na
	60 (S)	109	0.011	0.004	37.690	0.005	45.28							0.005	45.28
	120 (W)	249	0.002	na	na	na	na	na	na	na		na	na	na	na
	365 (S)	130	0.003	na	na	na	na	na	na	na		na	na	na	na

^a Planting days after application^b Harvest days after planting^c Metabolite IM-0

na – not analysed

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on analytical methods for the determination of residues of acetamiprid in crop commodities and for determining acetamiprid and its IM-2-1 metabolite in animal commodities.

These methods summarised below are suitable for the determination of residues of acetamiprid (and its IM-2-1 metabolite in animal commodities), as defined for both enforcement and/or risk assessment. The principle of most methods involves extraction steps using methanol or acetonitrile, liquid/liquid partition (commonly hexane) and further extraction into methylene chloride,

column chromatographic clean-up (silica gel, Florisil) and analysis by GC/ECD or HPLC or by LC-MS/MS.

Table 31 Summary of Analytical Methods for Acetamiprid

Plant material (vegetables, fruit - except citrus) Williams, 1999 [Ref: Aceta_024, Aceta_025]			
Analytes:	Acetamiprid	GC/ECD	Method EC-521 (RD-9420)
LOQ:	0.01 mg/kg		
Description	Acetamiprid is extracted with methanol. After filtration and dilution with brine, the mixture is partitioned with hexane. Acetamiprid remains in the aqueous layer and is extracted into methylene chloride. The methylene chloride extract is purified with fully-activated Florisil (elution with 50:50 acetone:hexane), redissolved in ethylacetate/hexane and further purified by column chromatography on Silica gel 60. The final residue is dissolved in acetone and measured by GC/ECD.		
Plant material (citrus fruit and oil) Williams, 1999 [Ref: Aceta_024, Aceta_026]			
Analytes:	Acetamiprid	HPLC	Method EC-521-M2
LOQ:	0.05 mg/kg		
Description	A modification of Method M1 above, developed for citrus matrices, with residues being extracted with acetonitrile instead of methanol and concentrated by evaporation before the hexane partitioning and methylene chloride extraction steps as used in Method M1 above. The same Florisil clean-up step is used, but further purification is by C-18 reverse phase column chromatography rather than silica gel. Acetamiprid residues in the final extract are measured by HPLC-UV (254 nm).		
Plant material Li, 2003 [Ref: Aceta_092]			
Analytes:	Acetamiprid	LC-MS/MS	Method KP-216
LOQ:	0.01 mg/kg		
Description	Acetamiprid is extracted twice with methanol:water (50:50) and centrifuged or filtered before SPE reversed phase cleanup (acetonitrile elution) and analysis by LC/MS/MS. MRM transitions for quantitation and confirmation are m/z 223 and m/z 126		
Animal commodities (Muscle, fat, liver, kidney, eggs, milk) Wais, 1997 [Ref: Aceta_029, Aceta_030]			
Analytes:	Acetamiprid, IM-2-1 metabolite	HPLC	Method AR 149-97
LOQ:	0.05 mg/kg (liver, kidney), 0.01 mg/kg (other matrices)		
Description	Samples are macerated with acetonitrile, the filtrate is concentrated by evaporation and mixed with brine before being partitioned with hexane (discarded) and methylene chloride. The dried methylene chloride extract is dissolved in hexane:acetone (80:20) and eluted through a Florisil column with hexane:acetone (1:1) and then through a C-18 SPE column with water:acetonitrile (85:15). Acetamiprid and the IM-2-1 metabolite residues in the final extract are measured by HPLC-UV (254 nm).		

Data collection methods

Method EC-521 (plant matrices)

The basic GC/ECD Method (EC-521), involving methanol extraction, hexane and methylene chloride partitioning with Florisil and silica gel clean-up steps, was developed for measuring acetamiprid residues in plant matrices and is summarized above.

A modification of this method (EC-521-M2), using acetonitrile extraction, the same partitioning steps, with Florisil and C-18 SPE clean-up steps and HPLC determination was developed for measuring acetamiprid residues in citrus matrices is also summarized above.

Recovery rates in a range of representative fruit and vegetable crops averaged about 93% and the calculated LOQs were 0.05 mg/kg for citrus matrices and 0.01 mg/kg for other crops.

Table 32 Validation recovery values for analytical method EC-521 for the determination of acetamiprid in crops using GC/ECD

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Cabbage	0.01 0.05	97.2 100.2	9.9 9.0	5 5	81.9 88.2	106 109	Aceta_024
Pears	0.01 0.05	82.3 84.4	12.5 8.9	5 5	68.1 71.6	95.7 90.3	
Raisins	0.01 0.05	95.2 99.2	4.7 9.8	5 5	91.4 84.4	101 110	
Cotton, seed	0.01 0.05	108.9 91.8	3.7 4.6	5 5	102 84.8	112 95.3	
Cotton, gin trash	0.01 0.05	88.4 88.1	16.1 9.0	5 5	73.4 76.9	112 98.9	
Broccoli	0.01 0.05	86.8 95.3	12.2 7.7	5 5	73.0 85.6	98.1 103	
Carrot, root	0.01 0.05	97.9 90.3	7.7 3.4	5 5	89.0 86.1	109 93.3	
Spinach	0.01 0.05	98 98.7	-	2 2	95 97.3	101 100	Aceta_025
Bell peppers	0.01 0.05	104 88.4	-	2 2	100 85.4	108 91.4	
Eggplant	0.01 0.05	101.7 103.5	-	2 2	92.4 103	111 104	
Cabbage	0.01 0.05	101 85.8	-	2 2	100 84	102 87.6	
Broccoli	0.01 0.05	95.9 87.7	-	2 2	95.7 82.9	96.1 92.4	
Celery	0.01 0.05	108 90.4	-	2 2	107 88.6	109 92.1	
Head lettuce	0.01 0.05	97.6 116	-	2 2	87.1 114	108 118	
Leaf lettuce	0.01 0.05	102.5 105	-	2 2	94.9 101	110 109	
Pear, whole	0.01 0.05	91.3 103	-	2 2	85.3 101	97.2 105	
Mustard greens	0.01 0.05	109 97.7	-	2 2	109 95.5	109 99.8	
Tomato, paste	0.01 0.05	89.8 94.6	-	2 2	84.1 93.1	95.4 96.0	
Tomato, puree	0.01 0.05	98.8 97.6	-	2 2	96.6 95.4	101 99.7	
Grape, whole	0.01 0.05	104 101	-	2 2	110 97.0	118 105	
Grape, raisins	0.01 0.05	88.9 88.6	-	2 2	87.3 87.9	90.5 89.3	
Grape, juice	0.01 0.05	91.7 80.6	-	2 2	91.5 79.1	91.9 82.0	
Grape, raisin waste	0.01 0.05	97.2 92.5	-	2 2	90.3 92.3	104 92.7	
Grape, wet pomace	0.01 0.05	108 84.9	-	2 2	108 82.5	108 87.2	

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Grape, dry pomace	0.01 0.05	82.0 84.6	-	2 2	80.9 84.4	83.0 84.8	
Apple, whole	0.01 0.05	82.1 95.3	-	2 2	76.8 94.6	87.3 96.0	
Apple, pomace	0.01 0.05	89.0 88.1	-	2 2	88.5 87.5	89.4 88.7	
Apple, juice	0.01 0.05	88.9 99.3	-	2 2	88.7 90.6	89.0 108	
Cotton, gin trash	0.01 0.05	78.9 83.7	-	2 2	78.7 83.6	79.1 83.7	
Cotton, hulls	0.01 0.05	83.8 83.2	-	2 2	81.1 82.4	86.5 83.9	
Cotton, meal	0.01 0.05	86.9 92.2	-	2 2	81.9 91.4	91.8 92.9	
Cotton, seeds	0.01 0.05	100.3 90.1	-	2 2	93.6 89.4	107 90.8	
Cotton, refined oil	0.01 0.05	103.4 89.6	-	2 2	98.7 88.6	108 90.6	
Carrots	0.01 0.05	98.0 89.6	-	2 2	95.0 87.9	101 91.3	
Pepper ^a	0.01 0.05	76 80	-	3 2	74 78	77 82	Aceta_027
Apple ^a	0.01 0.10	80 82	-	2 2	80 78	80 86	
Pear ^b (acidic extraction)	0.01 0.05	89 90		2 2	84 84	94 96	
Orange ^a	0.01 0.10	97 101	-	2 2	92 98	102 104	
Peach ^a	0.01 0.05	91 92	-	3 2	83 86	96 98	
Peach ^c (basic extraction)	0.01 0.05	71 72	-	2 2	71 72	71 72	
Tobacco ^d	0.01 0.05	71.5 73	-	2 2	70 72	73 74	
Cotton ^d	0.01 0.05	75 76	-	2 2	74 74	76 78	
Tomato	0.01 0.05 0.5	98 105 96		2 2 2	96 96 92	100 115 101	Aceta_052

^a A single Florisil clean-up step^b Extraction in acidic water (not methanol) and a single Florisil clean-up step^c Extraction in alkaline methanol and a single Florisil clean-up step^d Florisil and C-18 SPE clean-up

Table 33 Validation recovery values for analytical Method EC-521-M2 for the determination of acetamiprid in citrus commodities using GC/ECD

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Orange	0.05 1.0	98.6 94.9	-	1 1			Aceta_024

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Orange, oil	0.05 3.0	92.5 94.2	-	1 1			Aceta_026
Orange, dried pulp	0.05 1.5	71.3 77.6	-	1 1			
Orange, juice	0.05 1.5	88.2 96.3	-	1 1			
Lemon	0.05 0.5	91.1 91.4	-	2 2	90.9 91.0	91.2 91.8	
Grapefruit	0.05 0.5	106.3 93.1	-	2 2	105.0 89.1	107.5 97.1	
Orange	0.05 0.25 2.5	90 91.2 91.7		2 2 2	87.4 88.1 91.3	92.9 94.3 92	Aceta_048
Orange, juice	0.05 0.25 2.5	91.4 93.7 93.8		2 2 2	90.3 93.3 93.8	92.4 94 95.1	
Orange, dried pulp	0.05 0.25 2.5	75.1 78.8 81.2		2 2 2	66.5 78 79.4	83.7 79.5 83	
Orange, oil	0.05 0.25 2.5	96.8 94.4 96.9		2 2 2	95.1 94.4 94.9	96.8 94.7 96.9	

Method KP-216 (plant matrices)

A more recent LC-MS/MS method involving methanol extraction and SPE clean-up prior to analysis (as summarized above) was developed for plant matrices and was used in a number of US supervised field trials. The LOQ for this method is 0.01 mg/kg and validation data are summarized below.

Table 34 Validation recovery values for analytical method EC-521 for the determination of acetamiprid in crops using GC/ECD

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Tomato	0.01 0.05	89 90	-	2 2	82 78	96 102	Aceta_028
Cucumber	0.01 0.05	74 81	-	2 2	74 72	74 90	
Melon	0.01 0.05	102.5 88	-	2 2	102 86	103 90	
Plum, fruit	0.01 0.05	100 85	-	2 2	95 82	105 88	
Plum, dry fruit	0.01 0.05	89.5 93	-	2 2	87 90	92 96	
Almond, nutmeat	0.01 0.1 1.0	103 102 97	-	3 3 3	95 97 92	108 105 101	Aceta_050
Almond, hulls	0.01 0.1 1.0	112 98 80	-	3 3 3	104 96 77	117 100 83	
Cucumber	0.01 0.1 1.0	81 88 80	-	3 3 3	75 78 78	88 96 82	Aceta_051

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Green beans	0.01	88	-	3	81	96	Aceta_082
	0.1	87		3	82	94	
	1.0	88		3	84	91	
Blueberries	0.01	80	-	3	79	81	Aceta_083
	0.1	86		3	84	90	
	1.0	107		3	104	109	
Onion, bulb	0.01	99	-	3	97	100	Aceta_085
	0.1	103		3	93	115	
	1.0	100		3	75	116	
Strawberry	0.01	86	-	6	82	89	Aceta_086
	0.1	88		3	87	88	
	1.0	100		3	100	101	
Grape	0.01	96	-	3	95	98	Aceta_087
	0.1	85		3	83	88	
	1.0	94		3	92	96	

Method AR 149-97 (animal matrices)

The HPLC Method (AR 149-97), involving acetonitrile extraction, hexane and methylene chloride partitioning with Florisil and C-18 SPE clean-up steps, was developed for measuring residues of acetamiprid and its IM-2-1 metabolite in animal commodities and is summarized above. Calculated LOQs were 0.05 mg/kg for liver and kidney and 0.01 mg/kg for other animal matrices.

Table 35 Validation recovery values for analytical Method AR 149-97 for the determination of acetamiprid in animal commodities using HPLC-UV

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Milk, cattle	0.01	88.9	12.5	3	78.2	100.4	Aceta_029
	0.1	84.1	6.5	3	80.6	90.4	
Muscle, cattle	0.01	88.5	9.7	3	80.6	97.6	
	0.1	89.1	6.5	3	82.6	93.7	
Fat, cattle	0.01	93.5	4.1	3	89.8	97.5	
	0.1	98.0	0.8	3	97.2	98.7	
Liver, cattle	0.05	89.2	2.5	3	87.0	91.5	Aceta_030
	0.5	97.1	1.8	3	95.9	99.1	
Kidney, cattle	0.05	92.6	2.1	3	91.0	94.8	
	0.5	95.9	2.6	3	93.1	97.9	
Egg, hen	0.01	94.9	3.5	3	93.0	98.8	
	0.1	91.7	0.8	3	90.9	92.2	
Muscle, hen	0.01	97.5	5.3	3	92.7	103.0	
	0.1	93.6	0.7	3	92.8	94.0	
Fat, hen	0.01	96.9	-	2	96.1	97.6	
Liver, hen	0.05	93.3	-	2	93.1	93.5	

Table 36 Validation recovery values for analytical Method AR 149-97 for the determination of the IM-2-1 metabolite of acetamiprid in animal commodities using HPLC

Matrix/Analyte	Fortification levels (mg/kg)	Mean recovery (%)	Relative standard deviation (%)	No.	Range of recoveries (%)		Reference
					Low	High	
Milk, cattle	0.01 0.1	90.6 84.3	9.4 6.6	3 3	82.2 80.6	99.2 90.7	Aceta_029
Muscle, cattle	0.01 0.1	95.5 90.8	14.0 5.0	3 3	82.3 85.6	109.1 94.0	
Fat, cattle	0.01 0.1	94.5 98.0	3.0 0.8	3 3	91.3 97.1	96.6 98.6	
Liver, cattle	0.05 0.5	89.6 96.4	2.7 0.5	3 3	87.6 95.9	92.3 96.7	
Kidney, cattle	0.05 0.5	93.1 96.4	2.5 3.0	3 3	90.7 93.2	95.4 98.8	
Egg, hen	0.01 0.1	86.8 92.0	1.8 0.3	3 3	85.6 91.7	88.6 92.3	Aceta_030
Muscle, hen	0.01 0.1	95.1 93.9	6.4 0.7	3 3	89.6 93.1	101.7 94.4	
Fat, hen	0.01	100.4	-	2	99.6	101.2	
Liver, hen	0.05	93.1	-	2	92.5	93.6	

Enforcement methods

Multi-residue method PAM-1

A study by Rhoads, 1997 [Ref: Aceta_023], investigated the suitability of the US PAM-1 multiresidue protocols as residue enforcement method for acetamiprid in plant commodities. Oranges were used as the test matrix and recovery rates under Protocol D (without clean-up) were measured following fortification at 0.05 mg/kg and 0.25 mg/kg. Recoveries were less than 8% in samples spiked with 0.05 mg/kg and 37–41% in the 0.25 mg/kg spiked samples. Recovery rates following Protocol E (Florasil 303/304 C1 and 303/304 C2) methodology were less than 30%. It was concluded that the US PAM-1 multiresidue method was not suitable as an enforcement method for acetamiprid.

QuEChERS Multi-residue method

The multi-residue QuEChERS method was validated for the determination of acetamiprid in representative plant commodities with a high water content (apple), high oil content (sunflower seed), high starch/protein (maize grain) and high acid content (orange) in a study by Schwartz, 2008 [Ref: Aceta_093].

Homogenized samples were extracted with acetonitrile, mixed with MgSO₄, NaCl, and buffering citrate salts (pH 5–5.5) with the organic phase was separated by centrifugation and cleaned-up (freezing out, dispersive SPE, C18) before being acidified (formic acid) and analysed by LC-MS/MS for acetamiprid (223 m/z and 126 m/z) and the IM-2-1 metabolite (209 m/z) with confirmation using the 223 m/z > 90 m/z transition. For each fortification level (0.01 mg/kg, 0.1 mg/kg and 1.0 mg/kg and for both MS/MS transitions monitored, the average recoveries were in the range of 91 % and 103 %, and the relative standard deviations were ≤ 8 %.

The multi-residue QuEChERS method was also validated for the determination of acetamiprid and its IM-2-1 metabolite in animal commodities (muscle, liver, kidney, fat, eggs and milk) by Miya, 2010 [Ref: Aceta_094].

Homogenized samples were extracted with acetonitrile, mixed with MgSO_4 , NaCl, and buffering citrate salts (pH 5–5.5) with the organic phase was separated by centrifugation and cleaned-up (C18), diluted with methanol, filtered and analysed by LC-MS/MS for acetamiprid (223 m/z and 126 m/z) and the IM-2-1 metabolite (209 m/z) with confirmation using the 223 m/z > 90 m/z transition. For each fortification level (0.01 mg/kg, 0.1 mg/kg and 1.0 mg/kg and for both MS/MS transitions monitored, the average recoveries were in the range of 95 % and 108 %, and the relative standard deviations (RSD) were ≤ 8 %.

Stability of residues in stored analytical samples

Plant matrices

The Meeting received information on the stability of residues of acetamiprid in various substrates with a high water content (apple, cabbage, cucumber, grape, lettuce, tomato) a high starch content (potato), a high oil content (cotton seed) and a high acid content (orange) and their processed fractions, stored at ambient temperatures and at freezer temperatures.

In a study by Gieseke, 1999 [Ref: Aceta_031], residue stability of acetamiprid was investigated in a range of homogenised fruit, vegetables and processed commodity samples fortified with 0.25 mg/kg or 0.1 mg/kg acetamiprid and stored in the dark in sealed containers, either at room temperature for up to 6 days or at about -35 °C for up to 12 months (16 months for lettuce).

Duplicate ‘freezer-aged’ fortified samples were taken at the time of the initial fortification and at intervals during the storage periods and together with untreated samples freshly fortified on the day of analysis, were analysed for acetamiprid residues using Method EC-521 (or EC-521-M2 for the citrus matrices).

A further study was reported by Netzbund, 2003 [Ref: Aceta_032] on residue stability in homogenised potatoes spiked with 0.1 mg/kg acetamiprid and stored frozen for intervals up to 12 months. In this study, triplicate ‘freezer-aged’ fortified samples were taken at the time of the initial fortification and at intervals during the storage periods for analysis and together with duplicate untreated samples freshly fortified on the day of analysis, were analysed for acetamiprid residues.

For analysis, the samples were extracted in methanol, partitioned into methylene chloride, cleaned up through a Florisil column and residues were measured by LC/MS/MS (m/z ratios of 223.2, 126.2 and 56.2).

Acetamiprid residues were stable in stored samples of representative high water content, high oil content, high starch content and high acid content commodities and their processed fractions that were stored for up to 7 days at room temperature or up to 12 months at freezer temperatures (16 months for lettuce).

Table 37 Residue stability of acetamiprid in fortified plant matrices stored at room temperature. (Gieseke, 1999: Aceta_031)

Commodity	Storage interval (days)	Spike level (mg/kg)	Procedural recovery (%)	Recovery in stored samples (%)
Orange	0	0.25	89.3, 101.4	
	0	0.25	106.2, 92.2	96.2, 84.5
	2	0.25	103.3, 88.8	88.7, 96.4
	5	0.25	105.3, 96	86.7, 104.5
	7	0.25	101.4, 106.3	104.2, 93.9
Cotton seed	0	0.1	86.1, 84.1	
	0	0.1	82.8, 81.1	83.4, 79.5
	2	0.1	106, 82.3	76.2, 80.8
	5	0.1	75.4, 87	98.7, 83.8
	7	0.1	74.4, 76.5	72.4, 74.7

Commodity	Storage interval (days)	Spike level (mg/kg)	Procedural recovery (%)	Recovery in stored samples (%)
Cotton gin trash	0	0.25	89.9, 92.9	
	0	0.25	84.8, 82.4	84.6, 84.7
	3	0.25	91, 91.7	106.4, 87.3
	5	0.25	104.4, 105.6	83.8, 108.3
	7	0.25	103.8, 94.1	70.8, 86.8
Cotton seed oil	0	0.1	89.7, 83.6	
	0	0.1	101.6, 84.8	78.3, 82.8
	3	0.1	93, 102.4	85.9, 87.3
	5	0.1	89.7, 93.3	93.1, 76.9
	7	0.1	99.8, 87.3	84.8, 92.4
Tomato	0	0.1	77.1, 82.9	
	0	0.1	100.5, 94.3	90.9, 97.6
	2	0.1	111.3, 96.5	92, 89.6
	4	0.1	105.7, 101.5	99.1, 98.2
	7	0.1	91.7, 92.7	87.4, 95.2
Tomato paste	0	0.1	92.3, 87.4	
	0	0.1	92.9, 86.1	106.8, 101.8
	2	0.1	107.5, 95.8	97.5, 99.1
	4	0.1	103, 94.6	103, 100
	7	0.1	115.5, 116.9	120.2, 112.4
Grape	0	0.1	93.1, 87.5	
	0	0.1	75.8, 88.6	88.1, 101.2
	2	0.1	89, 88.8	95.9, 87.1
	5	0.1	87.8, 91.1	79.4, 92.8
	7	0.1	77.7, 91.5	84.2, 84.4
Grape raisins	0	0.1	99.1, 98.6	
	0	0.1	87.1, 98.6	93.2, 101.8
	2	0.1	92.6, 92.9	96.3, 91.2
	5	0.1	96.7, 89.4	91.6, 96.4
	7	0.1	95.9, 112.4	82.1, 85.2
	15		113, 97	105.7, 103.4
Grape juice	0	0.1	88.9, 98.3	
	0	0.1	88.4, 86.6	99.8, 83.1
	3	0.1	100, 104.5	90.7, 100.3
	5	0.1	97, 100.0	91, 91.7
	7	0.1	81.3, 84.3	89.3, 83.5

Table 38 Residue stability of acetamiprid in fortified plant matrices stored at freezer temperatures (Gieseke, 1999: Aceta_031)

Commodity	Storage interval (months)	Spike level (mg/kg)	Procedural recovery (%)	Recovery in stored samples (%)
Apple	0	0.1	93.4, 104.6	
	1	0.1	101, 96.4	87.8, 87.1
	3	0.1	84.3, 93.5	97.6, 99.8
	6	0.1	100.3, 97.5	89.6, 89.3
	12	0.1	102.5, 104.9	96.3, 93
Apple, juice	0	0.1	92, 104.6	
	1	0.1	80.4, 89	85.8, 101.1
	3	0.1	93, 109.8	107.5, 105.3
	6	0.1	99.2, 92.8	96.9, 93.8
	12	0.1	92.5, 90.3	88.8, 87.8
Apple, wet pomace	0	0.1	91.4, 74.1	
	1	0.1	68, 79.3	84.2, 82.5
	3	0.1	76.9, 88.1	79.8, 77.3
	6	0.1	81.3, 73.5	67.9, 81.2
	12	0.1	86, 82.1	81.7, 81.2

Commodity	Storage interval (months)	Spike level (mg/kg)	Procedural recovery (%)	Recovery in stored samples (%)
Cabbage	0	0.1	94.6, 93.6	
	1	0.1	98.9, 97.3	96.6, 99.4
	3	0.1	91.2, 93.2	94, 117.3
	6	0.1	95.9, 91.4	94.9, 96.5
	12	0.1	96.9, 95.5	95.9, 98.4
Cotton, gin trash	0	0.1	95.8, 106.9	
	1	0.1	100.8, 110.1	100.8, 107.2
	3	0.1	82.5, 80.1	78, 81.2
	6	0.1	94.1, 91.3	80.9, 90
	12	0.1	92.7, 88.2	89.3, 83.4
Cotton, seed hulls	0	0.1	72.8, 74.2	
	1	0.1	84.3, 88.1	78.8, 93.3
	3	0.1	79.3, 66.6	66, 61.7
	6	0.1	82.8, 76.2	81, 83.2
	12	0.1	77.6, 78	69.7, 76.1
Cotton, seed meal	0	0.1	87.8, 78.7	
	1	0.1	87.5, 101.1	86.6, 97.6
	3	0.1	82.8, 73.6	78.8, 80.6
	6	0.1	89.2, 83.9	82.9, 80.7
	12	0.1	74.8, 76.2	75.7, 76.5
Cotton, seed oil	0	0.1	78.4, 91	
	1	0.1	99.4, 98	92.1, 106.3
	3	0.1	86.5, 77.8	88.4, 86.1
	6	0.1	92.8, 102.2	114, 98.4
	12	0.1	83.4, 87.1	82.7, 79.9
Cotton, seed	0	0.1	94.1, 101.2	
	1	0.1	88.7, 89.2	78.4, 91.1
	3	0.1	89.3, 75.7	85.2, 78.5
	6	0.1	94.9, 97.3	93.9, 89
	12	0.1	83, 89.4	91.3, 93.8
Cucumber	0	0.1	108.5, 106.6	
	1	0.1	100, 84.1	94.4, 91.8
	3	0.1	88.3, 91.4	80.5, 90.5
	6	0.1	97.3, 113.2	100.4, 96
	12	0.1	98.1, 93.6	93.7, 92
Head lettuce	0	0.1	99.6, 96.8	
	1	0.1	88.7, 96.2	86.9, 97.2
	3	0.1	93.3, 93.8	94.8, 98.7
	6	0.1	97.7, 107.9	112.6, 111.3
	12	0.1	114.4, 99	105.4, 98.2
	16	0.1	82.9, 94.7	96.6, 91.4
Orange, dried pulp	0	0.5	83.4, 105.3	
	1	0.5	84.1, 78.8	84.5, 95.6
	3	0.5	85.9, 86.6	86.1, 87.8
	6	0.5	86.5, 84.4	84.7, 83.4
	12	0.5	84.9, 90.8	90.7, 85.7
Orange	0	0.5	91, 90.5	
	1	0.5	90.5, 91.8	88.1, 85.6
	3	0.5	96.2, 97	102.9, 87.4
	6	0.5	83.8, 95.7	79.4, 90.8
	12	0.5	93.4, 103	97.4, 98.9
Orange, juice	0	0.5	97.6, 98.1	
	1	0.5	95.1, 96.2	92.5, 92.4
	3	0.5	100.2, 98.7	100.2, 99.2
	6	0.5	96.7, 101.2	97.5, 95.1
	12	0.5	97.1, 98.6	95.8, 96.5

Commodity	Storage interval (months)	Spike level (mg/kg)	Procedural recovery (%)	Recovery in stored samples (%)
Orange, oil	0	0.5	92.8, 92.4	
	1	0.5	93.9, 94.6	93.9, 92.6
	3	0.5	103.7, 99.6	99.1, 99.6
	6	0.5	98.8, 98.5	96.6, 110.9
	12	0.5	97.3, 98.7	97.4, 97.4

Table 39 Residue stability of acetamiprid in fortified plant matrices stored at freezer temperatures (Netzband, 2003: Aceta_032)

Commodity	Storage interval (months)	Spike level (mg/kg)	Procedural recovery (%)	Recovery in stored samples (%)
Potatoes	0	0.1	61, 101	73, 93, 97
	1	0.1	88, 80	104, 66, 100
	2.4	0.1	97, 103	99, 103, 122
	8	0.1	79, 81	100, 91, 107

USE PATTERN

Information on acetamiprid GAP in Europe, Japan and USA was provided to the Meeting. Relevant uses listed on authorised labels or advised by national authorities are summarised in the following table.

Table 40 Registered or authorised uses of acetamiprid

Crop	Country	Application						PHI (days)	Remarks
		method	form	kg ai/ha	kg ai/hL	water L/ha	Max no		
Adzuki bean (dry)	Japan	foliar	SL, SP		0.0045-0.01		3	14	
Apple	Japan	foliar	SG, SP		0.005-0.01		3	1	
Apricot	Japan	foliar	SG, SP		0.005-0.01		3	7	
Blueberries	Japan	foliar	SG, SP		0.005		1	1	
Brassica leafy vegetables	Japan	foliar	SG, SP		0.005		1	7	
Brassica vegetables (Flowerhead)	Italy	foliar	SP	0.065-0.08	0.007-0.008		2	14	
Brassica vegetables (Head)	Italy	foliar	SP	0.065-0.08	0.007-0.008		2	7	except Brussel sprouts
Broccoli	Japan	foliar	SG, SP		0.01		3	14	
Brussel sprouts	Italy	foliar	SP	0.05-0.07	0.005-0.007		2	21	
Brussels sprouts	Japan	foliar	SG, SP		0.01		1	7	
Brussels sprouts, non-heading	Japan	foliar	SG, SP		0.01		2	7	
Bulb vegetables	USA	foliar	SG, WP	0.11-0.17			4	7	7 day intervals min
Bush & caneberries	USA	foliar	SG, WP	0.05-0.11		47 min (air) 187 min (ground)	5	1	7 day intervals min

Crop	Country	Application						PHI (days)	Remarks
		method	form	kg ai/ha	kg ai/hL	water L/ha	Max no		
Cabbage ("Choux")	France	foliar	SG	0.05			2	21	7 days apart
Cabbages, Head	Japan	foliar	SG, SP, SL		0.005-0.02		5	7	
Cauliflower	Japan	foliar	SG, SP		0.01		3	7	
Celery	Japan	foliar	SG, SP		0.005		2	7	
Cherry, Sweet	Japan	foliar	SP		0.005-0.01		1	3	
Chinese cabbage	Japan	foliar	SG, SP, SL		0.005-0.02		3	14	
Citrus	France	foliar	SG		0.005	1000	2	14	14 days apart
Citrus	Italy	foliar	SP	0.1	0.005-0.0075		2	14	
Citrus	Japan	foliar	SL, AL, SG, SP		0.005-0.01		3	14	
Citrus	Spain	foliar	SP	0.1	0.005	2000	1	14	Up to petal fall
Citrus	Spain	foliar	SP	0.1	0.005-0.01		2	14	
Citrus	USA	foliar	SG, WP	0.05-0.3		187 min (air) 935-14000 (ground)	5	7	7 day intervals min
Clover	USA	foliar	SG, WP	0.05-0.08		47 min (air) 187 min (ground)	1	56	
Cole crops	USA	foliar	SG, WP	0.04-0.08		47 min (air) 187 min (ground)	5	7	7 day intervals min
Common bean	Japan	foliar	SG, SP		0.005-0.01		3	1	
Cotton	Italy	foliar	SP	0.05	0.005		2	14	
Cotton	Spain	foliar	SP	0.04-0.06		500-800		14	
Cotton	USA	foliar	SG, WP	0.03-0.11		47 min (air) 140 min (ground)	4	28	7 day intervals
Cucumber	Japan	foliar	AL, SG, SL, SP		0.005-0.01		3	1	
Cucumber (indoor)	Spain	foliar	SG	0.05-0.08	0.01	1000-1700		3 (indoor)	
Cucurbits	France	foliar	SG	0.05		200-1000	2	3	cucumber, courgette, gherkin (7 days apart)
Cucurbits	USA	foliar	SG, WP	0.05-0.11		47 min (air) 187 min (ground)	5	0	5 day intervals min
Cucurbits (edible peel)	Italy	foliar	SP	0.1	0.01		2	7 (outdoor) 3 (indoor)	
Cucurbits (inedible peel)	Italy	foliar	SP	0.05	0.005		2	14	
Egg plant	Japan	foliar	AL, SG, SL, SP		0.004-0.01		3	1	
Fruiting vegetables	USA	foliar	SG, WP	0.03-0.08		47 min (air) 187 min (ground)	4	7	7 day intervals

Crop	Country	Application						PHI (days)	Remarks
		method	form	kg ai/ha	kg ai/hL	water L/ha	Max no		
Grapes	Japan	foliar	SG, SP		0.005-0.01		3	14	
Grapes & small vine fruit	USA	foliar	SG, WP	0.05-0.11		47 min (air) 187 min (ground)	2	3	14 day intervals min
Japanese apricot	Japan	foliar	AL, SG, SL, SP		0.003-0.01		3	1	
Leafy vegetables	France	foliar	SG	0.05		200-1000	2	7	lettuce, escarole, curly parsley (endive)
Leafy vegetables	USA	foliar	SG, WP	0.04-0.08		47 min (air) 187 min (ground)	5	7	7 day intervals min
Legume vegetables	Italy	foliar	SP	0.07-0.08	0.007-0.008		2	14	with and without pods
Legume vegetables	Japan	foliar	SG, SP		0.005		3	7	except green soya beans, common beans, peas (with pod)
Legume vegetables	USA	foliar	SG, WP	0.05-0.11		47 min (air) 187 min (ground)	3	7	7 day intervals min
Lettuce and similar	Italy	foliar	SP	0.05	0.005-0.007		2	7 (outdoor) 3 (indoor)	lettuce, escarole, curly parsley (endive), rucola, leafy brassicas
Lettuce and similar	Spain	foliar	SG	0.05		500-1000		7	
Lettuce, Head	Japan	foliar	SG, SP		0.005		3	7	
Lettuce, Leaf	Japan	foliar	SG, SP		0.005		1	7	
Low growing berries	USA	foliar	SG, WP	0.04-0.15		94 min (air) 187 min (ground)	2	1	7 day intervals min
Melon (outdoor)	Spain	foliar	SG	0.025-0.05		500-600		7 (outdoor)	
Melons (for pickling)	Japan	foliar	SP		0.005-0.01		3	1	
Melons, except Watermelon	Japan	foliar	SG, SP		0.0025		3	3	
Nectarine	Japan	foliar	SG, SP		0.005		3	3	
New Zealand Spinach	Japan	foliar	SG, SP		0.0025		3	14	
Oilseed rape	Italy	foliar	SP 5	0.05-0.06	0.01-0.013		2	40	
Okra	Japan	foliar	SG, SP		0.005		3	1	
Onion, Bulb	Japan	foliar	SG, SP		0.01		3	7	
Onion, Chinese	Japan	foliar	SG, SP		0.01		3	14	
Onion, Welsh	Japan	foliar	SG, SP		0.01		3	7	
Peach	Japan	foliar	SG, SP		0.005-0.01		3	1	
Pear	Japan	foliar	SG, SP		0.005-0.01		3	1	
Peas (with pod)	Japan	foliar	SG, SP		0.005		3	1	

Crop	Country	Application						PHI (days)	Remarks
		method	form	kg ai/ha	kg ai/hL	water L/ha	Max no		
Pepper (sweet)	Japan	foliar	SG, SP		0.005		2	1	
Peppers	Italy	foliar	SP	0.05-0.1	0.007-0.01		2	7 (outdoor) 3 (indoor)	
Peppers (Sweet, Chili)	Japan	foliar	SG, SP		0.0025		2	7	small
Plum, Japanese	Japan	foliar	SG, SP		0.005-0.01		3	7	
Pome fruit	France	foliar	SG		0.005	1000-1500	2	14	apple, pear, quince nashi (14 days apart)
Pome fruit	Italy	foliar	SP	0.05-0.1	0.004-0.008		2	14	
Pome fruit	Spain	foliar	SP		0.005	1000	2	14	1000-1500 l/ha for plums
Pome fruit	USA	foliar	SG, WP	0.05-0.17		94 min (air) 468 min (ground)	4	7	12 day intervals (min)
Potato	France	foliar	SG	0.03-0.05			2	7	20-30 days apart
Potato	Italy	foliar	SP	0.05-0.07	0.01-0.013		2	7	
Potato	Spain	foliar	SP	0.025-0.04		400		14	
Pulses ??	Italy	foliar	SP	0.07-0.08	0.007-0.008		2	14	
Pumpkin	Japan	foliar	SG, SP		0.01		2	1	
Solonacea	France	foliar	SG	0.05-0.1		200-1000	2	7 (outdoor) 3 (indoor)	Eggplant, peppers, tomato 10 days apart
Solonacea	Italy	foliar	SP	0.05-0.1	0.007-0.01		2	7 (outdoor) 5 (indoor)	except peppers
Solonacea	Spain	foliar	SG	0.025-0.08		500-1000 1000-1700		7 (outdoor) 3 (indoor)	Eggplant, peppers, tomato
Spinach	Japan	foliar	SG		0.0025		2	14	
Stem broccoli	Japan	foliar	SG, SP		0.005		2	1	
Stone fruit	France	foliar	SG		0.005	1000	2	14	apricot, cherry, peach, nectarine, plum (14 days apart)
Stone fruit	Italy	foliar	SP	0.05-0.09	0.004-0.006		2	14	
Stone fruit	Spain	foliar	SP		0.005	1000-1500		14	
Stone fruit	USA	foliar	SG, WP	0.05-0.17		94 min (air) 468 min (ground)	4	7	10 day intervals (min)
Strawberry	Japan	foliar	SG, SP		0.005-0.01		2		
Tomato	Japan	foliar	SG, SP		0.01		3	1	
Tomato (indoor)	USA	irrigation	SG 30	0.08	3.4 g ai/1000 plants		1	1	protected crops

Crop	Country	Application						PHI (days)	Remarks
		method	form	kg ai/ha	kg ai/hL	water L/ha	Max no		
Tomato (regular size)	Japan	foliar	AL, SL		0.004-0.005		3	1	
Tree nuts	USA	foliar	SG, WP	0.05-0.2		94 min (air) 468 min (ground)	4	14	14 day intervals (min)
Tuber, corm vegetables	USA	foliar	SG, WP	0.03-0.08		47 min (air) 187 min (ground)	4	7	7 day intervals min
Watermelon	Japan	foliar	SG, SP		0.005-0.01		3	3	

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised field trials involving foliar applications of acetamiprid to the following crops.

Crop	Commodity	Region	Table No
Citrus	lemon, mandarin, orange	Europe	41–43
Pome fruit	apple, pear	USA	44–45
Stone fruit	cherry, peach, plum	USA	46–48
Berries & small fruit	grape, strawberry	Canada,	USA 49–50
	blueberry, cane fruit	USA	50–52
Bulb vegetables	bulb onions, spring onions	USA	53–54
Brassica vegetables	broccoli, cabbage	USA	55–56
Fruiting vegetables, Cucurbits	cucumber, summer squash, melon	USA	57–59
Fruiting vegetables, other than Cucurbits	tomato	Canada,	USA 60
	peppers	USA	61–62
Leafy vegetables	lettuce	USA,	Europe 63–65
	spinach, mustard greens	USA	66–67
Legume vegetables	beans, peas	USA	68–69
Stalk & stem vegetables	celery	USA	70
Tree nuts	almonds, pecans	USA	71–72
Oilseeds	cotton seed	USA	73
Miscellaneous animal feed commodities	almond hulls, cotton gin trash	USA	74–75

Trials were generally well documented with laboratory and field reports. Laboratory reports included procedural recoveries from spiking at residues levels similar to those occurring in samples from supervised trials. Dates of analyses or duration of residue sample storage were also provided.

In trials where duplicate field samples from an unreplicated plot were taken at each sampling time and analysed separately, each figure is presented individually but the mean value has been used for intake risk assessment and residue estimation. Where duplicate analyses were conducted on the same field samples, only the mean residue values have been reported. Although all trials included control plots, no control data are recorded in the tables unless residues in control samples exceeded the LOQ. In such cases, the residues found are noted as “c = n mg/kg”. Residue data are recorded unadjusted for recovery and when residues were not detected they are shown as below the LOQ (e.g., < 0.01 mg/kg).

Residues and application rates have been rounded to two significant digits (or if close to the LOQ, the residue results have been rounded to one significant digit). Mean residues have been calculated from the unrounded residue values reported and then rounded. The results from trials used for the estimation of maximum residue levels have been underlined.

When multiple applications were made to a crop, the application rate, spray concentration and spray volume were not always identical from one application to the next. If the variation was small, only the final values for application rate, concentration and spray volume were recorded. For larger variations all values were recorded.

Intervals of freezer storage between sampling and analysis were recorded for all trials and were covered by the conditions of the freezer storage stability studies in representative matrices except where noted.

Citrus

Lemon

Results from supervised trials in Italy and Spain on lemons were provided to the Meeting. In these trials, 1-2 applications of acetamiprid (SP formulations) were applied to mature trees, 44-45 days apart as foliar sprays using backpack sprayers or hand guns, applying 2580-3500 litres water/ha. Plot sizes in these trials ranged from 30-180 square metres.

Unreplicated samples of at least 12 fruit were taken from each plot, frozen within 24 hours of sampling and stored frozen until whole fruit analysis for acetamiprid using GC/ECD or GC/NPD (Method EC-521), with an LOQ of 0.01 mg/kg. Recovery rates in untreated samples fortified with 0.01-1.16 mg/kg acetamiprid ranged from 77-128%. Sample storage intervals were less than 12 months except in one trial, where samples were stored for up to 15 months before analysis.

Table 41 Residues in lemons from supervised trials in Italy and Spain involving foliar applications of acetamiprid (SP formulations)

Location, Year (variety)	Form	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	matrix	PHI [days]	Residues [mg/kg]	
Italy Siracusa, Sicilia 1996 (Femminello Siracusano)	SP 20	2	0.35	0.012	Whole fruit	0 3 7 14 21	0.46 0.56 0.5 ^a 0.37 <u>0.45</u>	RD-01013, NTF/1/51/L, Aceta_33
Italy, Gregorio (CT), Az. Costanzo, 1996 (Femminello)	SP 20	2	0.35	0.01	Whole fruit	7 15	0.25 <u>0.15</u>	RD-00268, 96763BO1, Aceta_34
Spain, Orihuela, Alicante, 1995 (Verna)	SP 20	2	0.26	0.01	Whole fruit	0 7 14 21 28	0.18 0.08 <u>0.09</u> 0.04 0.03	RD-00261, 95726SE1, (15 month storage) Aceta_35

^a mean of duplicate analyses

Mandarin

Results from supervised trials in Italy and Spain on mandarins/tangerines were provided to the Meeting. In these trials, 2-3 applications of acetamiprid (SP formulations) were applied to mature trees, 38-55 days apart as foliar sprays using backpack sprayers or hand guns, applying 1600-3500 litres water/ha. Plot sizes in these trials ranged from 60-128 square metres.

Unreplicated samples of at least 12 fruit were taken from each plot, frozen within 24 hours of sampling and stored frozen for up to 5 months before whole fruit analysis for acetamiprid using GC/ECD or GC/NPD (Method EC-521), with an LOQ of 0.01 mg/kg. Recovery rates in untreated samples fortified with 0.008-1.11 mg/kg acetamiprid ranged from 70–115%.

Table 42 Residues in mandarins and tangerines from supervised trials in Italy and Spain involving foliar applications of acetamiprid (SP formulations)

MANDARIN Location, Year (variety)	Form	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
Italy Belpasso, Catania 1997 (Avana)	SP 20	2	0.35	0.014	Whole fruit	0 3 7 14 21	0.38 0.5 0.54 0.46 0.47	RD-01027 NTF/2/31/M Aceta_036
Italy Torremuzza Caruso 1996 (Tardivo die Ciaculli)	SP 20	2	0.35	0.01	Whole fruit	7 15	0.21 <u>0.14</u>	RD-00269 96762BO1 Aceta_037
Spain Alfarp, Valencia 1996 (Arrufatina)	SP 20	1+ 1	0.24 0.23	0.01 0.01	Whole fruit	7 15	0.35 <u>0.26</u>	RD-00276 96626V1 Aceta_038
Spain Denia, Alicante 1996 (Clemenvilla)	SP 20	1+ 1	0.24 0.21	0.01 0.01	Whole fruit	7 15	0.26 <u>0.19</u>	RD-00276 96626V2 Aceta_038
Spain Castellon 1996 (Clemenules)	SP 20	2	0.34	0.01	Whole fruit	7 15	0.22 <u>0.25</u>	RD-01012 NTF/1/16S/L Aceta_039
Spain Sueca, Valencia 1996 (Marisol Citrange carrizo)	SP 20	2	0.35	0.01	Whole fruit	7 15	0.52 <u>0.44</u>	RD-01012 NTF/1/15S/L Aceta_039
Spain Alfarp, Valencia 1997 (Arrufatina)	SP 20	1+ 1+ 1	0.2 0.18 0.16	0.01 0.01 0.01	Whole fruit	0 7 14 21	0.22 0.12 <u>0.25</u> 0.19	RD-00727 97749V1 Aceta_040
Spain Denia, Alicante 1997 (Clemenvilla)	SP 20	1+ 2	0.17 0.18	0.01 0.01	Whole fruit	0 7 14 21	0.29 0.15 <u>0.17</u> 0.15	RD-00727 97749V2 Aceta_040

Orange

Results from supervised trials in Italy, Spain on oranges were provided to the Meeting. In these trials, 1-2 applications of acetamiprid (SP formulations) were applied to mature trees, 15 or 44–47 days apart as foliar sprays using backpack sprayers or hand guns, applying 2000–3600 litres water/ha. Plot sizes in these trials ranged from 80-165 square metres or at least 4 trees.

Unreplicated samples of at least 12 fruit (or 3 kg) were taken from each plot, frozen within 24 hours of sampling and stored frozen for up to 10 months before whole fruit analysis for acetamiprid using GC/ECD or GC/NPD (Method EC-521), with an LOQ of 0.01 mg/kg. Recovery rates in untreated samples fortified with 0.01-1.17 mg/kg acetamiprid ranged from 70-118%. At two sites, pulp, peel and juice samples were also analysed.

In one trial conducted in USA, 5 applications of acetamiprid (WP formulation) were made to mature trees 6–7 days apart, as foliar sprays using a commercial airblast sprayer, applying 1550-1630 litres water/ha. Plot sizes in these trials were about 1200 square metres (at least 10 trees).

Unreplicated samples of at least 25 fruit were taken and frozen within 24 hours of sampling and stored frozen for up to 12 months before whole fruit, peel, pulp and oil analysis for acetamiprid using HPLC (Method EC-521-M2), with an LOQ of 0.05 mg/kg. Recovery rates in untreated samples fortified with 0.05-2.5 mg/kg acetamiprid ranged from 91-98% in whole fruit, juice and oil and from 62-89% in dried pulp.

Table 43 Residues in oranges from supervised trials in Italy, Spain and USA involving foliar applications of acetamiprid (SP or WP formulations)

ORANGE Location, Year (variety)	Form	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
Italy Torremuzza Catania 1995 (Tarocco nucellare)	SP 20	1	0.2	0.01	Whole fruit	14	0.83	RD-00127 95785BO1 Aceta_041
Italy Torremuzza, Catania 1995 (Tarocco comune)	SP 20	1	0.25	0.01	Whole fruit	0 7 14 21 28	0.21 0.07 0.06 0.1 0.05	RD-00131 95784BO1 Aceta_042
Italy Torremuzza, Catania 1996 (Tarocco)	SP 20	2	0.35	0.01	Whole fruit	7 15	0.08 <u>0.09</u>	RD-00128 96764BO1 Aceta_043
Italy Bummacaro, Catania 1996 (Navelina)	SP 20	2	0.35	0.01	Whole fruit	7 14	0.31 <u>0.22</u>	RD-01071 NTF/1/4I/O Aceta_044
Spain Valencia 1997 (Navelina)	SP 20	2	0.35	0.01	Whole fruit	0 3 7 14 21	0.33 0.18 0.17 0.08 <u>0.12</u>	RD-00170 NTF/2/1S/O Aceta_045 processing study
Spain Valencia 1997 (Navelina)	SP 20	1+ 1	0.36 0.34	0.01 0.01	Whole fruit	0 3 7 14 21	0.62 0.43 0.27 <u>0.28</u> < 0.01	RD-00170 NTF/2/2S/O Aceta_045 processing study
Spain Torre de la Reina, Sevilla 1996 (Navelina)	SP 20	2	0.35	0.01	Whole fruit	7 15	0.38 <u>0.28</u>	RD-00241 96625SE1 Aceta_046
Spain Alfarp, Valencia 1995 (Navelina)	SP 20	1+ 1	0.25 0.24	0.01 0.01	Whole fruit	0 7 14 21 28	0.39 0.24 <u>0.39</u> 0.34 0.22	RD-00259 95725SE1 Aceta_047
Spain San Jorge, Castellon 1996 (Navelina)	SP 20	1+ 1	0.35 0.32	0.01	Whole fruit	7 15	0.21 <u>0.1</u>	RD-00171 NTF/1/14S/O Aceta_044

ORANGE Location, Year (variety)	Form	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
Spain, Vinaroz, Castellon, 1996 (Navelina)	SP 20	2	0.35	0.01	Whole fruit	7 14	0.65 <u>0.4</u>	RD-00171 NTF/1/13S/O Aceta_044
USA, FL, 1997 (Valencia)	WP 70	1+ 3+ 1	0.43 0.42 1.44	0.03 0.03 0.09	Whole fruit	7	0.31	RD-00753 97512103 Aceta_048 processing study

Pome fruit

Apples, pears

Results from supervised trials in USA on pome fruit were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to mature apple and pear trees, 11-13 days apart as foliar sprays using airblast sprayers, applying either 470-800 litres water/ha (low volume) or 1150-2370 litres/ha (high volume). Plot sizes in these trials involved at least 12 trees.

Duplicate samples of at least 2 kg fruit were taken from each plot, frozen within 2 hours of sampling and stored frozen for up to 8 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in untreated samples fortified with 0.01-1.0 mg/kg acetamiprid ranged from 77-113%.

Table 44 Residues in apples from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

APPLES Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (CA) 1997 (Fuji)	WP 70	4	0.17	0.007	Fruit	7	0.12, 0.12	<u>0.12</u>	RD-00759 12648-12 Aceta_074
USA (CA) 1997 (Granny Smith)	WP 70	4	0.17	0.007	Fruit	7	0.16, 0.11	<u>0.14</u>	RD-00759 12648-11 Aceta_074
USA (CO) 1998 (Golden Delicious)	WP 70	4	0.17	0.02	Fruit	7	0.17, 0.2	<u>0.19</u>	RD-00759 12648-24 Aceta_074
USA (DI) 1998 (Jonathan)	WP 70	4	0.17	0.02	Fruit	7	0.23, 0.23	<u>0.23</u>	RD-00759 12648-27 Aceta_074
USA (IN) 1998 (Gold Rush)	WP 70	4	0.17	0.03	Fruit	7	0.22, 0.39	<u>0.31</u>	RD-00759 12648-23 Aceta_074
USA (MI) 1997 (Golden Delicious)	WP 70	4	0.17	0.03	Fruit	7	0.59, 0.59	<u>0.59</u>	RD-00759 12648-07 Aceta_074

APPLES Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (NC) 1998 (Red Delicious)	WP 70	4	0.17	0.012	Fruit	7	0.15, 0.09	<u>0.12</u>	RD-00759 12648-21 Aceta_074
USA (NY) 1998 (Jonimac)	WP 70	4	0.17	0.018	Fruit	7	0.14, 0.13	<u>0.14</u>	RD-00759 12648-19 Aceta_074
USA (NY) 1998 (McIntosh)	WP 70	4	0.17	0.018	Fruit	7	0.2, 0.23	<u>0.22</u>	RD-00759 12648-18 Aceta_074
USA (OR) 1997 (Red Delicious)	WP 70	4	0.17	0.01	Fruit	7	0.32, 0.24	<u>0.28</u>	RD-00759 12648-15 Aceta_074
USA (OR) 1997 (Red Delicious)	WP 70	2+ 1+ 1	0.17 0.15 0.17	0.01	Fruit	7	0.17, 0.18	<u>0.18</u>	RD-00759 12648-16 Aceta_074
USA (PA) 1997 (Red Fuji)	WP 70	4	0.16	0.03	Fruit	7	0.15, 0.16	<u>0.16</u>	RD-00759 12648-04 Aceta_074
USA (PA) 1998 (Red Delicious)	WP 70	3+ 1	0.17 0.18	0.025	Fruit	7	0.21, 0.28	<u>0.25</u>	RD-00759 12648-20 Aceta_074
USA (VA) 1998 (Red Delicious)	WP 70	4	0.17	0.036	Fruit	7	0.28, 0.24	<u>0.26</u>	RD-00759 12648-22 Aceta_074
USA (WA) 1997 (Red Delicious)	WP 70	4	0.84	0.045	Fruit	7	0.82	0.82	RD-00591 12650-01 Aceta_075 processing study
USA (WA) 1998 (Red Delicious)	WP 70	4	0.17	0.012	Fruit	7	0.45, 0.64	<u>0.55</u>	RD-00759 12648-25 Aceta_074
USA (WA) 1998 (Red Delicious)	WP 70	4	0.17	0.012	Fruit	7	0.28, 0.22	<u>0.25</u>	RD-00759 12648-26 Aceta_074
USA (WI) 1997 (Paula Red)	WP 70	4	0.17	0.036	Fruit	7	0.29, 0.25	<u>0.27</u>	RD-00759 12648-09 Aceta_074

Table 45 Residues in pears from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

PEARS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (CA) 1997 (Bartlett)	WP 70	2+ 1+ 1	0.17 0.16 0.17	0.014 0.013 0.014	Fruit	7	0.23, 0.27	<u>0.25</u>	RD-00760 12649-05 Aceta_076
USA (CA) 1997 (Bosc)	WP 70	4	0.17	0.012	Fruit	7	0.14, 0.16	<u>0.15</u>	RD-00760 12649-03 Aceta_076
USA (CA) 1998 (Yali)	WP 70	4	0.17	0.018	Fruit	7	0.18, 0.21	<u>0.2</u>	RD-00760 12649-11 Aceta_076
USA (NY) 1998 (Clapp’s Favorite)	WP 70	4	0.17	0.018	Fruit	7	0.23, 0.1	<u>0.17</u>	RD-00760 12649-10 Aceta_076
USA (OR) 1997 (Bartlett)	WP 70	2+ 1+ 1	0.17 0.17 0.17	0.013 0.012 0.013	Fruit	1 4 7 10	0.44, 0.48 0.46, 0.36 0.25, 0.29 0.32, 0.31	0.46 0.41 0.27 <u>0.32</u>	RD-00760 12649-08 Aceta_076
USA (OR) 1997 (D’Anjou)	WP 70	4	0.17	0.01	Fruit	7	0.1, 0.08	<u>0.09</u>	RD-00760 12649-09 Aceta_076
USA (PA) 1997 (Bosc)	WP 70	4	0.16	0.02	Fruit	7	0.36, 0.26	<u>0.31</u>	RD-00760 12649-02 Aceta_076
USA (WA) 1997 (Bartlett)	WP 70	4	0.17	0.012	Fruit	7	0.08, 0.1	<u>0.09</u>	RD-00760 12649-06 Aceta_076
USA (WA) 1997 (D’Anjou)	WP 70	4	0.17	0.012	Fruit	7	0.34, 0.3	<u>0.32</u>	RD-00760 12649-07 Aceta_076

*Stone fruit**Cherries, peaches, plums*

Results from supervised trials in USA on stone fruit were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to mature cherry (sweet and sour), peach and plum trees, 6–7 days apart as foliar sprays using airblast sprayers, applying either 1100–3400 litres water/ha (high volume) or 520-870 litres/ha (low volume). Plot sizes in these trials ranged from 60-268 square metres.

Duplicate samples of at least 2 kg fruit were taken from each plot, frozen within 5 hours of sampling and stored frozen for up to 4 months before the stones were removed and the flesh analysed for acetamiprid using Method EC-521 (but with residues measured by HPLC), with an LOQ of 0.01 mg/kg. Recovery rates in untreated samples fortified with 0.01-1.0 mg/kg acetamiprid ranged from 82-118% in cherries, 84-120% in plums and 91-116% in peaches.

Table 46 Residues in cherries from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

CHERRIES Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Tulare (CA) 2003 (Brooks)	WP 70	4	0.17	0.008	Fruit without stones	7	0.53, 0.54	<u>0.54</u>	RD-00451 2003-19-CA-01 Aceta_049
USA Kern (CA) 2003 (Brooks)	WP 70	4	0.17	0.024	Fruit without stones	7	0.44, 0.52	<u>0.48</u>	RD-00451 2003-19-CA-02 Aceta_049
USA Wasco (OR) 2003 (Lapin)	WP 70	4	0.17	0.013	Fruit without stones	7	0.33, 0.39	<u>0.36</u>	RD-00451 2003-19-OR-03 Aceta_049
USA Hood River (OR) 2003 (Lapin)	WP 70	4	0.17	0.015	Fruit without stones	7	0.45, 0.4	<u>0.42</u>	RD-00451 2003-19-OR-04 Aceta_049
USA Ottawa (MI) 2003 (Montmorency)	WP 70	4	0.17	0.013	Fruit without stones	7	0.25, 0.33	<u>0.29</u>	RD-00451 2003-19-MI-05 Aceta_049
USA Kent (MI) 2003 (Montmorency)	WP 70	4	0.17	0.03	Fruit without stones	7	0.12, 0.08	<u>0.1</u>	RD-00451 2003-19-MI-06 Aceta_049
USA Lehigh (PA) 2003 (Montmorency)	WP 70	4	0.17	0.007	Fruit without stones	7	0.68, 0.69	<u>0.68</u>	RD-00451 2003-19-PA-22
		3+ 1	0.17 0.16	0.027	Fruit without stones	7	0.49, 0.5	0.49	Aceta_049
USA Delta (CO) 2003 (Montmorency)	WP 70	2+ 1+ 1	0.17 0.16 0.17	0.009	Fruit without stones	7	0.93, 0.82	<u>0.88</u>	RD-00451 2003-19-CO-23
		4	0.17	0.03	Fruit without stones	7	0.65, 0.77	0.71	Aceta_049

Table 47 Residues in peaches from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

PEACH Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Burks (PA) 2003 (Redskin)	WP 70	4	0.17	0.005	Fruit without stones	8	0.34, 0.3	0.32	RD-00451 2003-19-PA-13
			0.17	0.025	Fruit without stones	8	0.31, 0.57	<u>0.44</u>	Aceta_049
USA Saluda (SC) 2003 (Sureprince)	WP 70	4	0.17	0.008	Fruit without stones	7	0.23, 0.18	<u>0.2</u>	RD-00451 2003-19-SC-14 Aceta_049
USA Aiken (SC) 2003 (Contender)	WP 70	4	0.17	0.02	Fruit without stones	7	0.16, 0.19	<u>0.18</u>	RD-00451 2003-19-SC-15 Aceta_049

Acetamiprid

PEACH Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Saluda (SC) 2003 (Blake)	WP 70	4	0.17	0.008	Fruit without stones	7	0.22, 0.18	<u>0.2</u>	RD-00451 2003-19-SC-16 Aceta_049
USA Attawa (MI) 2003 (Bellaire)	WP 70	4	0.17	0.013	Fruit without stones	7	0.12, 0.14	0.13	RD-00451 2003-19-MI-17
			0.17	0.031	Fruit without stones	7	0.17, 0.15	<u>0.16</u>	Aceta_049
USA Walla (TX) 2003 (Texas Royal)	WP 70	4	0.17	0.005	Fruit without stones	7	0.39, 0.28	<u>0.34</u>	RD-00451 2003-19-TX-18
			0.17	0.025	Fruit without stones	7	0.21, 0.23	0.22	Aceta_049
USA Tulare (CA) 2003 (Snowfall)	WP 70	4	0.17	0.007	Fruit without stones	7	0.19, 0.24	<u>0.22</u>	RD-00451 2003-19-CA-19 Aceta_049
USA Tulare (CA) 2003 (Fay Elberta)	WP 70	4	0.17	0.022	Fruit without stones	7	0.24, 0.21	<u>0.23</u>	RD-00451 2003-19-CA-20 Aceta_049
USA Tulare (CA) 2003 (May Sun)	WP 70	4	0.17	0.008	Fruit without stones	7	0.09, 0.13	<u>0.11</u>	RD-00451 2003-19-CA-21 Aceta_049
USA Limestone (TX) 2003 (Frank)	WP 70	4	0.17	0.005	Fruit without stones	7	0.19, 0.2	<u>0.19</u>	RD-00451 2003-19-TX-24 Aceta_049

Table 48 Residues in plums from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

PLUMS Location, (variety)	Year	Form	Application data			Residues data				Report Trial No Reference
			No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
								sample results	mean	
USA Ottawa (MI) 2003 (Stanley)	WP 70	4	0.17	0.013	Fruit without stones	7	0.11, 0.12	<u>0.11</u>	RD-00451 2003-19-MI-07	
			0.17	0.031	Fruit without stones	7	0.12, 0.07	0.09	Aceta_049	
USA Tulare (CA) 2003 (Angelinas)	WP 70	4	0.17	0.009	Fruit without stones	7	0.01, 0.02	<u>0.02</u>	RD-00451 2003-19-CA-08 Aceta_049	
USA Tulare (CA) 2003 (July Rosa's)	WP 70	4	0.17	0.019	Fruit without stones	7	0.01, 0.02	<u>0.01</u>	RD-00451 2003-19-CA-09 Aceta_049	
USA Tulare (CA) 2003 (French Prune)	WP 70	4	0.17	0.007	Fruit, no stones	7	0.05, 0.06	<u>0.06</u>	RD-00451 2003-19-CA-10 Aceta_049	

PLUMS Location, (variety)	Year	Form	Application data			Residues data				Report Trial No Reference
			No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
								sample results	mean	
USA Tulare (CA) 2003 (French Prune)		WP 70	4	0.17	0.024	Fruit, no stones	7	0.04, 0.04	<u>0.04</u>	RD-00451 2003-19-CA-11 Aceta_049
USA Polk (OR) 2003 (Moyer)		WP 70	4	0.17	0.015	Fruit without stones	7	0.05, 0.03	<u>0.04</u>	RD-00451 2003-19-OR-12
				0.17	0.024	Fruit without stones	7	0.03, 0.04	0.03	Aceta_049

Berries and other small fruits

Grapes

Results from supervised trials in Canada and USA on grapes were provided to the Meeting. In these trials, 2 applications of acetamiprid (WP formulations) were applied 13-15 days apart as foliar sprays, using airblast sprayers to apply about 360-1900 litres water/ha. Plot sizes in these trials ranged from 30-380 square metres.

Duplicate samples of at least 12 bunches (or part bunches) were taken from each plot, frozen within 4 hours and stored frozen for up to 7 months before analysis for acetamiprid using EC-521 or Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-1.0 mg/kg acetamiprid ranged from 71-110%.

Table 49 Residues in grapes from supervised trials in Canada and USA, involving foliar applications of acetamiprid (WP formulations)

GRAPES Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
Canada Jordan Station (ON) 2005 (Concord)	WP 70	2	0.11	0.011	Bunches	3	0.17, 0.15	<u>0.16</u>	RD-01886 ON01 Aceta_087
Canada Jordan Station (ON) 2005 (Riesling)	WP 70	2	0.11	0.011	Bunches	3	0.2, 0.21	<u>0.2</u>	RD-01886 ON04 Aceta_087
Canada Jordan Station (ON) 2005 (Seyval)	WP 70	2	0.11	0.011	Bunches	3	0.08, 0.08	<u>0.08</u>	RD-01886 ON03 Aceta_087
Canada Jordan Station (ON) 2005 (Vidal)	WP 70	2	0.11	0.011	Bunches	3	0.08, 0.09	<u>0.09</u>	RD-01886 ON02 Aceta_087
Canada Summerland (BC) 2005 (Chancellor)	WP 70	2	0.11	0.011	Bunches	3	0.24, 0.22	<u>0.23</u>	RD-01886 BC01 Aceta_087
USA (CA) 1997 (not specified)	WP 70	2	0.28	0.04	Whole grape	7	0.15	0.15	RD-00745 12651-01 Aceta_088 processing study

Acetamiprid

GRAPES Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Bridgeton (NJ) 2005 (Glenora)	WP 70	2	0.11	0.051	Bunches	2	0.03, 0.03	<u>0.03</u>	RD-01886 NJ32 Aceta_087
USA Bridgeton (NJ) 2005 (Glenora)	WP 70	2	0.11	0.031	Bunches	3	0.01, 0.01	<u>0.01</u>	RD-01886 NJ33 Aceta_087
USA Dundee (NY) 2005 (Concord)	WP 70	2	0.11	0.013	Bunches	3	0.05, 0.05	<u>0.05</u>	RD-01886 NY20 Aceta_087
USA Fennville (MI) 2005 (Concord)	WP 70	2	0.11	0.023	Bunches	3	0.08, 0.08	<u>0.08</u>	RD-01886 MI28 Aceta_087
USA Madera (CA) 2005 (Thompson seedless)	WP 70	2	0.11	0.01	Bunches	3	0.15, 0.15	<u>0.15</u>	RD-01886 CA155 Aceta_087
USA Orcutt (CA) 2005 (Chardonnay)	WP 70	2	0.11	0.011	Bunches	3	0.06, 0.06	<u>0.06</u>	RD-01886 CA150 Aceta_087
USA Parlier (CA) 2005 (Princess)	WP 70	2	0.11	0.01	Bunches	3	0.22, 0.22 (c=0.1)	<u>0.22</u>	RD-01886 CA151 Aceta_087
USA Parlier (CA) 2005 (Thompson seedless)	WP 70	2	0.11	0.01	Bunches	2	0.04, 0.04	<u>0.04</u>	RD-01886 CA152 Aceta_087
USA Parlier (CA) 2005 (Thompson seedless)	WP 70	2	0.11	0.01	Bunches	2	0.07, 0.06	<u>0.07</u>	RD-01886 CA153 Aceta_087
USA Paso Robles (CA) 2005 (Cabernet)	WP 70	2	0.11	0.011	Bunches	3	0.1, 0.11	<u>0.11</u>	RD-01886 CA149 Aceta_087
USA Poplar (CA) 2005 (Thompson seedless)	WP, 70	2	0.11	0.023	Bunches	3	0.04, 0.03	<u>0.04</u>	RD-01886 CA154 Aceta_087
USA Prosser (WA) 2005 (Lemberger)	WP 70	2	0.11	0.015	Bunches	3	0.13, 0.13	<u>0.13</u>	RD-01886 WA23 Aceta_087
USA Ukiah (CA) 2005 (Chardonnay)	WP 70	2	0.11	0.015	Bunches	3	0.24, 0.25	<u>0.25</u>	RD-01886 CA148 Aceta_087

Strawberries

Results from supervised trials in Canada and USA on strawberries were provided to the Meeting. In these trials, 2 applications of acetamiprid (WP formulations) were applied 6–9 days apart as foliar sprays, using pressurised backpack sprayers with 2–6 nozzle booms, applying about 200–480 litres water/ha. Plot sizes in these trials ranged from 22–160 square metres.

Duplicate samples of at least 1 kg fruit (with sepals/caps removed) were taken from each plot, frozen within 5 hours and stored frozen for up to 4 months before analysis for acetamiprid using Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.02–1.0 mg/kg acetamiprid ranged from 88–103%. A mean recovery rate of 95% (after correction for 92% procedural recovery) was achieved in samples spiked with 0.1 mg/kg and stored frozen for 5 months.

Table 50 Residues in strawberries from supervised trials in Canada and USA, involving foliar applications of acetamiprid (WP formulations)

STRAWBERRY Location, (variety)	Form Year	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Salinas, (CA) 2004 (Ventana)	WP 70	2	0.15	0.032	Fruit	1	0.23, 0.24	<u>0.24</u>	RD-01112, 09058 09058-CA56 Aceta_086
USA Salinas (CA) 2004 (Camarosa)	WP 70	2	0.15	0.032	Fruit	1 3 5 9	0.22, 0.24 0.19, 0.19 0.15, 0.15 0.09, 0.09	<u>0.23</u> 0.19 0.15 0.09	RD-01112, 09058 09058-CA57 Aceta_086
USA Madera (CA) 2004 (Seascape)	WP 70	2	0.15	0.052	Fruit	1	0.11, 0.11	<u>0.11</u>	RD-01112, 09058 09058-CA58 Aceta_086
USA Dover, (FL) 2004 (Festival)	WP 70	2	0.155	0.052	Fruit	1	0.25, 0.22	<u>0.24</u>	RD-01112, 09058 09058-FL26 Aceta_086
USA Bridgeton, (NJ) 2004 (Avalon)	WP 70	2	0.15	0.069	Fruit	1	0.06, 0.06	<u>0.06</u>	RD-01112, 09058 09058-NJ14 Aceta_086
USA Aurora, (OR) 2004 (Totem)	WP 70	2	0.15	0.031	Fruit	1	0.13, 0.1	<u>0.12</u>	RD-01112, 09058 09058-OR08 Aceta_086
USA Arlington, (WI) 2004 (Honeoye)	WP 70	2	0.14	0.057	Fruit	1	0.03, 0.03	<u>0.03</u>	RD-01112, 09058 09058-WI07 Aceta_086
Canada St Edouard de Kent, (NS) 2004 (Annapolis)	WP 70	2	0.15	0.038	Fruit	1	0.09, 0.09	<u>0.09</u>	RD-01112, 09058 09058-NS04 Aceta_086
Canada Thorndale, (ON) 2004 (Mesiabi)	WP 70	2	0.15	0.058	Fruit	1	0.04, 0.04	<u>0.04</u>	RD-01112, 09058 09058-ON01 Aceta_086

STRAWBERRY Location, (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
Canada St Paul d'Abbotsford (QC) 2004 (Jewel)	WP 70	2	0.15	0.049	Fruit	1	0.05, 0.05	<u>0.05</u>	RD-01112, 09058 09058-QC11 Aceta_086

Blueberries, Blackberries, Raspberries, Boysenberries

Results from supervised trials in USA on blueberries and cane fruit (raspberries/blackberries/boysenberries) were provided to the Meeting. In these trials, 5 applications of acetamiprid (WP or SP formulations) were applied 6-7 days apart as foliar sprays, using airblast sprayers or pressurised backpack sprayers, applying about 175-1000 litres water/ha. Plot sizes in these trials ranged from 19-134 square metres.

Duplicate samples of at least 1 kg fruit were taken from each plot, frozen within 5 hours and stored frozen for up to 8 months before analysis for acetamiprid using Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-5.0 mg/kg acetamiprid ranged from 67-110%.

Table 51 Residues in blueberries from supervised trials in USA, involving foliar applications of acetamiprid (WP formulations)

BLUEBERRY Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (PA) 2004 (Blueray)	WP 70	1+ 4	0.12 0.11	0.022 0.022	Fruit	1	0.08, 0.1	<u>0.09</u>	RD-00904 KP-2004-14-PA-01 Aceta_083
USA (GA) 2004 (Climax)	WP 70	4+ 1	0.11 0.12	0.012 0.013	Fruit	1	0.49, 0.47	<u>0.48</u>	RD-00904 KP-2004-14-GA-02 Aceta_083
USA (GA) 2004 (Brightwell)	WP 70	4+ 1	0.11 0.12	0.024 0.023	Fruit	1	0.69, 0.54	<u>0.62</u>	RD-00904 KP-2004-14-GA-03 Aceta_083
USA (MI) 2004 (Blue Jay)	WP 70	5	0.11	0.016	Fruit	1	0.53, 0.46	<u>0.49</u>	RD-00904 KP-2004-14-MI-05 Aceta_083
USA (WI) 2004 (Elliot)	WP 70	5	0.11	0.02	Fruit	1	0.2, 0.2	<u>0.2</u>	RD-00904 KP-2004-14-WI-06 Aceta_083
USA (OR) 2004 (Rabbiteye)	WP 70	5	0.12	0.024	Fruit	1	0.28, 0.22	<u>0.25</u>	RD-00904 KP-2004-14-OR-09 Aceta_083

Table 52 Residues in blackberries, boysenberries and raspberries from supervised trials in USA, involving foliar applications of acetamiprid (WP or SP formulations)

CANE FRUIT Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Ottawa (MI) 2006 (Nova)	SP 30	1+ 4	0.11 0.11	0.017 0.015	Raspberry fruit	1	1.1, 1.0	<u>1.0</u>	RD-01200 2006-11-MI-01 Aceta_084
USA Washington (OR) 2006 (Boysenberry)	SP 30	1+ 3+ 1	0.11 0.12 0.11	0.04 0.041 0.039	Boysenberry fruit	1	0.54, 0.52	<u>0.53</u>	RD-01200 2006-11-OR-02 Aceta_084
USA Washington (OR) 2006 (Kotata)	SP 30	5	0.11	0.018	Blackberry fruit	1	0.51, 0.61	<u>0.56</u>	RD-01200 2006-11-OR-03 Aceta_084
USA Polk (OR) 2004 (Thornless evergreen)	WP 70	5	0.11	0.024	Blackberry fruit	8	0.38, 0.22	0.3	RD-00904 2004-14-OR-07 Aceta_083
USA Clackamas (OR) 2004 (Thornless evergreen)	WP 70	5	0.11	0.024	Blackberry fruit	7	0.27, 0.33	0.3	RD-00904 2004-14-OR-08 Aceta_083
USA Clackamas (OR) 2004 (Thornless evergreen)	WP 70	1+ 1	0.49 0.12	0.048 0.012	Blackberry fruit	2	2.3, 2.5	2.4	RD-00904 2004-14-OR-10 Aceta_083
USA Benton (OR) 2004 (Caroline)	WP 70	1+ 1+ 1	0.23 0.22 0.12	0.024 0.024 0.012	Raspberry fruit	1	0.82, 0.74	<u>0.78</u>	RD-00904 2004-14-OR-11 Aceta_083
USA Tift (GA) 2004 (Kiowa)	WP 70	1+ 1+ 1+ 2	0.11 0.12 0.11 0.12	0.019 0.019 0.02 0.02	Blackberry fruit	1	0.55, 0.74	<u>0.64</u>	RD-00904 2004-14-GA-04 Aceta_083

*Bulb vegetables**Bulb onions, Spring onions*

Results from supervised trials in USA on bulb and spring (green) onions were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied 6-7 days apart as foliar sprays, using pressurised backpack sprayers or tractor-mounted spray booms, applying about 190-300 litres water/ha. Plot sizes in these trials ranged from 46-124 square metres.

Duplicate samples of at least 2 kg onion bulbs or spring onion tops were taken from each plot, frozen within 4 hours and stored frozen for up to 7 months before analysis for acetamiprid using Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-0.05 mg/kg acetamiprid ranged from 69-102%.

Table 53 Residues in bulb onions from supervised trials in USA, involving foliar applications of acetamiprid (WP formulations)

ONION, BULB Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (PA) 2004 (Stuttgand)	WP 70	1+ 1+ 2	0.17 0.17 0.17	0.062 0.095 0.062	Bulb	6	< 0.01, < 0.01	<u>< 0.01</u>	RD-00905 2004-15-PA-01 Aceta_085
USA (TX) 2004 (Cimarron)	WP 70	3+ 1	0.17 0.16	0.089 0.089	Bulb	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00905 2004-15-TX-03 Aceta_085
USA (TX) 2004 (Yello Spanish)	WP 70	1+ 3	0.16 0.17	0.071 0.071	Bulb	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00905 2004-15-TX-05 Aceta_085
USA (CA) 2004 (Sabrisso)	WP 70	4	0.17	0.059	Bulb	7	< 0.01, 0.018	<u>0.01</u>	RD-00905 2004-15-CA-06 Aceta_085
USA (CA) 2004 (Red Onion)	WP 70	2+ 2	0.17 0.17	0.089 0.058	Bulb	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00905 2004-15-CA-07 Aceta_085
USA (WA) 2004 (Yellow Globe)	WP 70	5	0.17	0.089	Bulb	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00905 2004-15-WA-09 Aceta_085

Table 54 Residues in spring (green) onions from supervised trials in USA, involving foliar applications of acetamiprid (WP formulations)

SPRING ONION Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (WI) 2004 (Feast)	WP 70	4	0.17	0.089	Green onion	7	0.04, 0.06	<u>0.05</u>	RD-00905 2004-15-WI-02 Aceta_085
USA (TX) 2004 (Texas Early Grano 502)	WP 70	4	0.17	0.088	Green onion	7	2.1, 1.8	<u>2.0</u>	RD-00905 2004-15-TX-04 Aceta_085
USA (CA) 2004 (Bunching Onion)	WP 70	1+ 3	0.17 0.17	0.082 0.058	Green onion	7	0.43, 0.34	<u>0.38</u>	RD-00905 2004-15-CA-08 Aceta_085

*Brassica vegetables**Broccoli*

Results from supervised trials in USA on broccoli were provided to the Meeting. In these trials, 5 applications of acetamiprid (WP formulations) were applied to broccoli plants, 6-8 days apart as foliar

sprays using CO₂ backpack or tractor-mounted sprayers with 3-12 nozzle booms, applying about 140-375 litres water/ha. Plot sizes in these trials ranged from 93-220 square metres.

Duplicate samples of at least 2 kg heads and stalks were taken from each plot, frozen within 4 hours and stored frozen for up to 10 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-0.1 mg/kg acetamiprid ranged from 75-92%.

Table 55 Residues in broccoli from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

BROCCOLI Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (TX) 1997 (Legacy)	WP 70	1+ 4	0.08 0.09	0.05 0.06	Head + Stalk	7	0.02, 0.04	<u>0.03</u>	RD-00761 12645-01 Aceta_077
USA (CA) 1997 (Pinnacle F1)	WP 70	5	0.08	0.049	Head + Stalk	7	0.05, 0.04	<u>0.05</u>	RD-00761 12645-02 Aceta_077
USA (CA) 1997 (Big Sur, Hybrid)	WP 70	4+ 1	0.08 0.09	0.022 0.023	Head + Stalk	7	0.01, 0.01	<u>0.01</u>	RD-00761 12645-03 Aceta_077
USA (CA) 1997 (Packman – F1)	WP 70	3+ 1+ 1	0.09 0.08 0.09	0.023 0.023 0.023	Head + Stalk	7	0.02, < 0.01	<u>0.02</u>	RD-00761 12645-04 Aceta_077
USA (CA) 1997 (Green Valiant)	WP 70	1+ 1+ 3	0.08 0.09 0.08	0.03 0.03 0.03	Head + Stalk	7	0.01, 0.02	<u>0.02</u>	RD-00761 12645-05 Aceta_077
USA (CA) 1997 (Marathon)	WP 70	1+ 4	0.08 0.09	0.059 0.061	Head + Stalk	7	0.01, < 0.01	<u>0.01</u>	RD-00761 12645-06 Aceta_077
USA (CA) 1997 (Arcadia)	WP 70	1+ 4	0.08 0.09	0.044 0.061	Head + Stalk	7	0.11, 0.07	<u>0.09</u>	RD-00761 12645-07 Aceta_077
USA (CA) 1998 (DiCicco)	WP 70	1+ 4	0.09 0.08	0.045 0.045	Head + Stalk	7	0.18, 0.25	<u>0.22</u>	RD-00761 12645-10 Aceta_077
USA (OR) 1998 (Arcadia)	WP 70	1+ 1+ 3	0.08 0.09 0.08	0.03 0.03 0.03	Head + Stalk	7	0.02	<u>0.02</u>	RD-00761 12645-11 Aceta_077

Cabbage

Results from supervised trials in USA on cabbages were provided to the Meeting. In these trials, 5 applications of acetamiprid (WP formulations) were applied to cabbage plants, 6-7 days apart as foliar sprays using CO₂ backpack or tractor-mounted sprayers with 4-8 nozzle booms, applying about 93-280 litres water/ha. Plot sizes in these trials ranged from 93-220 square metres.

Duplicate samples of at least 12 heads (with and without wrapper leaves) were taken from each plot, frozen within 3 hours and stored frozen for up to 12 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-0.5 mg/kg acetamiprid ranged from 74-114%.

Table 56 Residues in cabbages from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

CABBAGE Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (NY) 1997 (Heads-up)	WP 70	5	0.09	0.0469	No wrapper leaves With wrapper leaves	7	< 0.01, 0.02 0.13, 0.09	<u>0.02</u> <u>0.11</u>	RD-00770 12646-01 Aceta_078
USA (NY) 1997 (Bravo)	WP 70	5	0.09	0.0469	No wrapper leaves With wrapper leaves	7	< 0.01, 0.01 0.06, 0.06	<u>0.01</u> <u>0.06</u>	RD-00770 12646-02 Aceta_078
USA (PA) 1997 (Market Prize)	WP 70	1+ 1+ 1+ 2	0.09 0.09 0.08 0.09	0.036 0.039 0.035 0.036	No wrapper leaves With wrapper leaves	7	< 0.01, < 0.01 0.07, 0.07	<u>≤ 0.01</u> <u>0.07</u>	RD-00770 12646-03 Aceta_078
USA (NC) 1997 (Jade Pagoda)	WP 70	2+ 3	0.08 0.09	0.09 0.09	No wrapper leaves With wrapper leaves	7	0.03, 0.03 0.03, 0.03	<u>0.03</u> <u>0.03</u>	RD-00770 12646-04 Aceta_078
USA (FL) 1997 (Bravo)	WP 70	5	0.08	0.03	No wrapper leaves With wrapper leaves	7	< 0.01, < 0.01 0.03, 0.03	<u>≤ 0.01</u> <u>0.03</u>	RD-00770 12646-05 Aceta_078
USA (MI) 1997 (Rinda Hybrid)	WP 70	5	0.08	0.053	No wrapper leaves With wrapper leaves	7	0.02, 0.01 0.13, 0.13	<u>0.02</u> <u>0.13</u>	RD-00770 12646-06 Aceta_078
USA (TX) 1997 (Vantage Point)	WP 70	4+ 1	0.08 0.09	0.06 0.06	No wrapper leaves With wrapper leaves	7	0.02, 0.02 0.06, 0.08	<u>0.02</u> <u>0.07</u>	RD-00770 12646-07 Aceta_078
USA (TX) 1997 (Early Jersey Wakefield)	WP 70	1+ 1+ 1+ 1+ 1	0.08 0.09 0.08 0.09 0.08	0.045 0.045 0.045 0.045 0.045	No wrapper leaves With wrapper leaves	7	0.05, 0.05 0.5, 0.49	<u>0.05</u> <u>0.5</u>	RD-00770 12646-08 Aceta_078
USA (CA) 1997 (Market Prize F1)	WP 70	2 1+ 2	0.08 0.09 0.08	0.048 0.047 0.048	No wrapper leaves With wrapper leaves	6	0.03, 0.03 0.11, 0.1	<u>0.03</u> <u>0.11</u>	RD-00770 12646-09 Aceta_078
USA (TX) 1997 (Blue Vantage)	WP 70	1+ 4	0.09 0.08	0.045 0.045	No wrapper leaves With wrapper leaves	7	< 0.01, < 0.01 0.12, 0.09	<u>≤ 0.01</u> <u>0.11</u>	RD-00770 12646-11 Aceta_078

Double-underlined values used for dietary intake estimation

*Fruiting vegetables, Cucurbits**Cucumber*

Results from supervised trials in USA on cucurbits were provided to the Meeting. In these trials, 5 applications of acetamiprid (WP formulations) were applied to cucumber, summer squash and melon (cantaloupe/musk melon) plants, 5 days apart as foliar sprays using CO₂ tractor-mounted or backpack sprayers with 3-8 nozzle booms, applying about 200-500 litres water/ha. Plot sizes in these trials ranged from 74-220 square metres.

Duplicate samples of at least 12 fruit were taken from each plot, frozen and stored for up to 6 months before analysis for acetamiprid using Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-1.0 mg/kg acetamiprid ranged from 75-98% in cucumbers, 90 – 99% in melons and 81-107% in summer squash.

Table 57 Residues in cucumbers from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

CUCUMBER Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Elko (SC) 2003 (Ashley)	WP 70	1+ 4	0.12 0.11	0.029 0.029	Cucumber	0	0.02, 0.04	<u>0.03</u>	RD-00453 2003-23-SC-01 Aceta_051
USA Springfield (SC) 2003 (Ashley)	WP 70	5	0.11	0.029	Cucumber	0	0.02, 0.03	<u>0.02</u>	RD-00453 2003-23-SC-02 Aceta_051
USA Newbury (FL) 2003 (Lightning)	WP 70	5	0.11	0.053	Cucumber	0	0.03, 0.02	<u>0.03</u>	RD-00453 2003-23-FL-03 Aceta_051
USA Conklin (MI) 2003 (Marketmore)	WP 70	5	0.11	0.042	Cucumber	0	0.04, 0.04	<u>0.04</u>	RD-00453 2003-23-MI-04 Aceta_051
USA Comstock Park (MI) 2003 (Marketmore)	WP 70	5	0.11	0.038	Cucumber	0	0.08, 0.09	<u>0.09</u>	RD-00453 2003-23-MI-05 Aceta_051
USA Brookshire (TX) 2003 (Straight Eight)	WP 70	5	0.11	0.04	Cucumber	0	0.02, 0.02	<u>0.02</u>	RD-00453 2003-23-TX-06 Aceta_051

Summer squash

Table 58 Residues in summer squash from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

SUMMER SQUASH Location, Year (variety)	Form	Application data			Residues data				Report
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		Trial No Reference
							sample results	mean	
USA Germansville (PA) 2003 (Black Beauty)	WP 70	1+	0.11	0.036	Squash	0	0.09, 0.09	<u>0.09</u>	RD-00453 2003-23-PA-13 Aceta_051
		1+	0.12	0.037					
		1+	0.11	0.036					
		2	0.12	0.037					

SUMMER SQUASH Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Elko (SC) 2003 (Lemondrop L)	WP 70	5	0.11	0.027	Squash	0	0.14, 0.08	<u>0.11</u>	RD-00453 2003-23-SC-14 Aceta_051
USA Newbury (FL) 2003 (Dixie)	WP 70	5	0.11	0.056	Squash	0	0.07, 0.05	<u>0.06</u>	RD-00453 2003-23-FL-15 Aceta_051
USA Conklin (MI) 2003 (Zucchini Elite)	WP 70	1+ 4	0.11 0.11	0.038 0.037	Squash	0	0.07, 0.05	<u>0.06</u>	RD-00453 2003-23-MI-16 Aceta_051
USA Porterville (CA) 2003 (Caiman)	WP 70	5	0.11	0.037	Squash	0	< 0.01, 0.08	<u>0.05</u>	RD-00453 2003-23-CA-17 Aceta_051

Melons

Table 59 Residues in melons from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

MELON Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Elko (SC) 2003 (Edisto)	WP 70	5	0.11	0.029	Cantaloupe	0	0.09, 0.07	<u>0.08</u>	RD-00453 2003-23-SC-07 Aceta_051
USA Brookshire (TX) 2003 (Hale’s Best)	WP 70	5	0.11	0.04	Cantaloupe	0	0.01, 0.03	<u>0.02</u>	RD-00453 2003-23-TX-09 Aceta_051
USA Porterville (CA) 2003 (Hale’s Best)	WP 70	5	0.11	0.038	Cantaloupe	0	0.02, 0.01	<u>0.02</u>	RD-00453 2003-23-CA-10 Aceta_051
USA Ponterville (CA) 2003 (Hale’s Best)	WP 70	5	0.11	0.022	Cantaloupe	0	0.03, 0.04	<u>0.04</u>	RD-00453 2003-23-CA-11 Aceta_051
USA San Ardo (CA) 2003 (Hale’s Best)	WP 70	1+ 4	0.11 0.11	0.058 0.04	Cantaloupe	0	0.14, 0.06	<u>0.1</u>	RD-00453 2003-23-CA-12 Aceta_051
USA Conklin (MI) 2003 (Star Fire)	WP 70	5	0.11	0.042	Muskmelon	0	0.08, 0.03	<u>0.06</u>	RD-00453 2003-23-MI-08 Aceta_051

*Fruiting vegetables, other than Cucurbits**Tomato*

Results from supervised trials in USA on tomatoes were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to tomato plants, 7 days apart as foliar sprays using CO₂ backpack sprayers with 3-6 nozzle booms or tractor-mounted 6-12 nozzle booms, applying about 100-200 litres water/ha. Plot sizes in these trials ranged from 37-147 square metres.

Duplicate samples of at least 12 fruit or 2 kg were taken from each plot, frozen within 2 hours and stored frozen until analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.⁰¹⁰.5 mg/kg acetamiprid ranged from 80-112%. Sample storage intervals were less than 12 months except in two trials, where samples were stored for up to 14 months before analysis.

Table 60 Residues in tomatoes from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

TOMATO Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (FL) 1997 (XPLT 10,047)	WP 70	1+ 2+ 1	0.08 0.09 0.08	0.045 0.046 0.045	Fruit	7	0.01, 0.01	<u>0.01</u>	RD-00776 12107-01, 13 mon storage Aceta_052
USA (CA) 1997 (Sunpride)	WP 70	4	0.09	0.0489	Fruit	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00776 2107-02, 14 mon storage Aceta_052
USA (MI) 1997 (Peto)	WP 70	4	0.08	0.055	Fruit	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00776 12107-03, Aceta_052
USA (CA) 1997 (Shady Lady)	WP 70	3+ 1	0.08 0.09	0.046 0.046	Fruit	7	0.04, 0.01	<u>0.03</u>	RD-00776 12107-04, Aceta_052
USA (CA) 1997 (#8892)	WP 70	5	0.08	0.059	Fruit	7	0.11, 0.08	<u>0.1</u>	RD-00776 12107-05, Aceta_052
USA (CA) 1997 (Big Beef)	WP 70	4	0.09	0.051	Fruit	7	< 0.01, 0.01	<u>0.01</u>	RD-00776 12107-06, Aceta_052
USA (CA) 1997 (#8892)	WP 70	1+ 2+ 1	0.08 0.08 0.08	0.057 0.054 0.048	Fruit	7	0.08, 0.07	<u>0.08</u>	RD-00776 12107-07, Aceta_052
USA (CA) 1997 (#3155)	WP 70	2 1+ 1	0.08 0.09 0.08	0.057 0.057 0.056	Fruit	7	0.11, 0.06	<u>0.09</u>	RD-00776 12107-08, Aceta_052
USA (CA) 1997 (Shady Lady)	WP 70	3 1	0.08 0.09	0.06 0.061	Fruit	7	0.03, 0.02	<u>0.03</u>	RD-00776 12107-09, Aceta_052
USA (CA) 1997 (APT127)	WP 70	4	0.08	0.084	Fruit	7	0.04, 0.02	<u>0.03</u>	RD-00776 12107-10 Aceta_052

TOMATO Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (CA) 1997 (H9175)	WP 70	4	0.08	0.084	Fruit	7	0.02, 0.02	<u>0.02</u>	RD-00776 12107-11, Aceta_052
USA (CA) 1997 (Heinz 8892)	WP 70	1+ 3	0.09 0.08	0.047 0.047	Fruit	7	0.03, 0.03	<u>0.03</u>	RD-00776 12107-12, Aceta_052
USA (CA) 1997 (Shady Lady)	WP 70	4	0.09	0.051	Fruit	7	0.07, 0.05	<u>0.06</u>	RD-00776 12107-13, Aceta_052
USA (CA) 1997 (Shady Lady)	WP 70	2+ 2	0.09 0.08	0.05 0.05	Fruit	7	0.05, 0.03	<u>0.04</u>	RD-00776 12107-14, Aceta_052
USA (CA) 1997 (Roma cv. Rio Grande)	WP 70	2+ 1+ 1	0.09 0.08 0.09	0.045 0.045 0.045	Fruit	7	0.06, 0.06	<u>0.06</u>	RD-00776 12107-15, Aceta_052
USA (NC) 1997 (Mountain Spring)	WP 70	1+ 3	0.08 0.09	0.058 0.052	Fruit	7	0.03, 0.05	<u>0.04</u>	RD-00776 12107-16 Aceta_052
USA (PA) 1997 (Roma)	WP 70	1+ 3	0.09 0.09	0.087 0.046	Fruit	7	0.03, 0.05	<u>0.04</u>	RD-00776 12107-17, Aceta_052
USA (MI) 1997 (Peto Cherry)	WP 70	4	0.08	0.054	Fruit	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00776 12107-18, Aceta_052
USA (CA) 1997 (Ace)	WP 70	1+ 1+ 2	0.84 0.83 0.84	0.758 0.715 0.72	Fruit	7	0.4, 0.44, 0.52	0.45	RD-00744 12108-02, Aceta_053 processing study

The Meeting also received results from three supervised trials on protected tomatoes in USA and Canada following drip irrigation treatments with acetamiprid. In these trials, 4 applications of acetamiprid (WP formulations) were applied to tomato plants, 7 days apart as simulated drip irrigation treatments, applying 10 ml/plant of a 0.034 kg ai/hL acetamiprid solution to individual tomato plants grown in a glasshouse in rockwool beds. The treatments were applied to 16-24 plant plots just before the daily irrigation.

Duplicate samples of fruit were taken from each plot, frozen within 2 hours and stored frozen for up to 3.3 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-0.1 mg/kg acetamiprid ranged from 91-120%.

Table 61 Residues in protected tomatoes from supervised glasshouse trials in USA involving drip irrigation treatments of acetamiprid (WP formulations)

TOMATO Location, (variety)	Year	Form	Application data			Residues data				Report Trial No Reference
			No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
								sample results	mean	
USA, (TN) Crossville 2003 (Mariachi RZ)		WP 70	4	0.09	0.037	Fruit	1	0.05, 0.05	<u>0.05</u>	RD-01884 TN15 Aceta_091
USA, (TX) Weslaco 2003 (Mariachi RZ)		WP 70	4	0.08	0.034	Fruit	1 3 5 7	0.03, 0.03 0.03, 0.04 0.04, 0.04 0.04, 0.04	0.03 0.03 <u>0.04</u> 0.04	RD-01884 TX26 Aceta_091
Canada, (ON) Simcoe 2003 (Rhapsody)		WP 70	4	0.08	0.034	Fruit	1	< 0.01, < 0.01	<u>< 0.01</u>	RD-01884 ON06 Aceta_091

Peppers (Sweet and Chili)

Results from supervised trials in USA on sweet (bell) and chili peppers were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to pepper plants, 7 days apart as foliar sprays using CO₂ backpack or tractor-mounted sprayers with 4-8 nozzle booms, applying about 190-330 litres water/ha. Plot sizes in these trials ranged from 69-178 square metres.

Duplicate samples of at least 12 fruit (2 kg min) were taken from each plot, frozen and stored for up to 10 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-5.0 mg/kg acetamiprid ranged from 79-119%.

Table 62 Residues in sweet (bell) and chili peppers from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

PEPPERS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (NC) 1997 (California Wonder)	WP 70	1+ 1+ 1+ 1	0.09 0.08 0.09 0.08	0.03 0.028 0.03 0.03	Fruit	7	0.03, 0.04	<u>0.04</u>	RD-00752 12106-01 Aceta_073
USA (SC) 1997 (New Ace)	WP 70	4	0.09	0.043	Fruit	7	0.01, < 0.01	<u>0.01</u>	RD-00752 12106-02 Aceta_073
USA (FL) 1997 (CamelotX3R)	WP 70	4	0.08	0.026	Fruit	0 3 7 14 21	0.07, 0.05 0.05, 0.04 0.02, 0.03 < 0.01, < 0.01 0.01, < 0.01	0.06 0.05 <u>0.03</u> < 0.01 0.01	RD-00752 12106-03 Aceta_073
USA (FL) 1997 (CamelotX3R)	WP 70	2+ 1+ 1	0.08 0.09 0.08	0.025 0.026 0.026	Fruit	7	0.02, 0.02	<u>0.02</u>	RD-00752 12106-04 Aceta_073
USA (OH) 1997 (Galaxy)	WP 70	1+ 1+ 2	0.09 0.09 0.08	0.035 0.039 0.042	Fruit	7	0.08, 0.09	<u>0.09</u>	RD-00752 12106-05 Aceta_073

PEPPERS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (TX) 1997 (Capistrano)	WP 70	4	0.09	0.045	Fruit	7	0.03, 0.03	<u>0.03</u>	RD-00752 12106-06 Aceta_073
USA (CA) 1997 (Jupiter)	WP 70	1+ 1+ 2	0.08 0.08 0.09	0.03 0.03 0.029	Fruit	7	0.07, 0.06	<u>0.07</u>	RD-00752 12106-08, Aceta_073
USA (CA) 1997 (California Wonder)	WP 70	3+ 1	0.08 0.08	0.039 0.032	Fruit	7	0.07, 0.04	<u>0.06</u>	RD-00752 12106-10 Aceta_073
USA (TX) 1997 (Jalapeño, TAM Mild)	WP 70	1+ 1+ 1+ 1	0.08 0.09 0.08 0.09	0.045 0.045 0.045 0.045	Chili pepper	7	0.06, 0.05	<u>0.06</u>	RD-00752 12106-11 Aceta_073
USA (CA) 1997 (Serrano Chili)	WP 70	3+ 1	0.08 0.08	0.039 0.032	Chili pepper	7	0.16, 0.12	<u>0.14</u>	RD-00752 12106-12, Aceta_073
USA (FL) 1997 (Banana Peppers)	WP 70	1+ 3	0.08 0.09	0.03 0.03	Chili pepper	7	0.08, 0.08	<u>0.08</u>	RD-00752 12106-13 Aceta_073

Leafy vegetables

Lettuce

Results from supervised trials in USA on outdoor head and leaf lettuce and on field and protected leaf lettuce in Europe were provided to the Meeting.

In the USA trials, 5 applications of acetamiprid (WP formulations) were applied to lettuce plants, 7 days apart as foliar sprays using CO2 backpack or tractor-mounted sprayers, applying about 140-200 litres water/ha. Plot sizes in these trials ranged from 55-223 square metres.

Duplicate samples of at least 12 plants (with and without wrapper leaves) were taken from each plot, frozen and stored for up to 15 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-1.5 mg/kg acetamiprid ranged from 73-112%.

Table 63 Residues in lettuce from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

LETTUCE Location, Year (variety)	Application data			Residues data				Report Trial No Reference
	No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
						sample results	mean	
USA (CA) 1997 (Great Lakes 659)	1+ 4	0.09 0.08	0.045 0.044	Head, no wrapper leaves	7	0.02, 0.02	0.02	RD-00747 12109-02 Aceta_054
				Head + wrapper leaves	7	0.66, 0.64	<u>0.65</u>	

LETTUCE Location, Year (variety)	Application data			Residues data				Report Trial No Reference
	No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
						sample results	mean	
USA (CA) 1997 (Big valley)	5	0.08	0.057	Head, no wrapper leaves	7	0.06, 0.05	0.05	RD-00747 12109-04 Aceta_054
				Head + wrapper leaves	3	0.55	0.55	
					5	0.19	0.19	
					7	0.15, 0.14	<u>0.14</u>	
					10	0.03	0.03	
					14	0.02	0.02	
USA (CA) 1997 (Diamond)	1+ 4	0.09 0.08	0.057 0.056	Head, no wrapper leaves	7	< 0.01, < 0.01	< 0.01	RD-00747 12109-05 Aceta_054
				Head + wrapper leaves	7	0.36, 0.4	<u>0.38</u>	
USA (CA) 1997 (Premiere)	1+	0.09	0.047	Head, no wrapper leaves	7	0.06, 0.06	0.06	RD-00747 12109-06 Aceta_054
	2+	0.08	0.045					
	1+	0.09	0.048	Head + wrapper leaves	7	0.29, 0.26	<u>0.28</u>	
	1	0.08	0.047					
USA (NY) 1997 (Crispino)	1+	0.08	0.044	Head, no wrapper leaves	7	0.18, 0.16	0.17	RD-00747 12109-07 Aceta_054
	1+	0.09	0.044					
	3	0.08	0.044	Head + wrapper leaves	7	0.45, 0.39	<u>0.42</u>	
USA (FL) 1997 (Boston Esmeralda)	3+	0.08	0.049	Head, no wrapper leaves	7	0.29, 0.25	0.27	RD-00747 12109-08 Aceta_054
	1+	0.09	0.052					
	1	0.08	0.049	Head+ wrapper leaves	7	0.61, 0.74	<u>0.68</u>	
USA (CA) 1997 (Salinas)	2+	0.08	0.05	Head, no wrapper leaves	7	0.01, 0.01	0.01	RD-00747 12109-09 Aceta_054
	2+	0.09	0.05					
	1	0.08	0.05	Head + wrapper leaves	3	0.12	0.12	
					5	0.13	0.13	
					7	0.06, 0.06	<u>0.06</u>	
					10	0.02	0.02	
USA (CA) 1997 (Savannah Green, M.I.)	2+ 3	0.08 0.09	0.06 0.06	Head, no wrapper leaves	7	0.01, 0.01	0.01	RD-00747 12109-10 Aceta_054
					Head + wrapper leaves	7	0.27, 0.25	
USA (CA) 1997 (Waldman´s Green Leaf)	5	0.08	0.045	Leaves	7	0.65, 0.57	<u>0.61</u>	RD-00748 12110-02 Aceta_055
USA (CA) 1997 (Savannah Green)	1+	0.08	0.05	Leaves	7	0.33, 0.27	<u>0.3</u>	RD-00748 12110-03 Aceta_055
	1+	0.09	0.05					
	3	0.08	0.05					
USA (CA) 1997 (Bibb)	3+	0.08	0.054	Leaves	7	0.45, 0.37	<u>0.41</u>	RD-00748 12110-04 Aceta_055
	1+	0.09	0.053					
	1	0.08	0.054					
USA (CA) 1997 (Two Star M.I.)	2+	0.09	0.048	Leaves	7	1.1, 0.85	<u>0.96</u>	RD-00748 12110-06 Aceta_055
	1+	0.08	0.048					
	1+	0.09	0.048					
	1	0.08	0.047					
USA (NY) 1997 (Black Seeded Simpson)	5	0.09	0.046	Leaves	7	0.72, 1.0	<u>0.87</u>	RD-00748 12110-07 Aceta_055

LETTUCE Location, Year (variety)	Application data			Residues data				Report Trial No Reference
	No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
						sample results	mean	
USA (FL) 1997 (Crop-N-Green)	1+ 4	0.08 0.09	0.045 0.045	Leaves	7	0.11, 0.12	<u>0.12</u>	RD-00748 12110-08, Aceta_055
USA (CA) 1997 (Tiara)	1+ 4	0.09 0.08	0.049 0.049	Leaves	7	0.11, 0.1	<u>0.11</u>	RD-00748 12110-09, Aceta_055
USA (CA) 1997 (New Red Fire)	2+ 2+ 1	0.084 0.085 0.084	0.05 0.05 0.048	Leaves	7	0.44, 0.48	<u>0.46</u>	RD-00748 12110-10, Aceta_055

Results from supervised trials in Europe on outdoor and protected leaf lettuce were also provided to the Meeting. In these trials, 2 applications of acetamiprid (WP formulations) were applied to lettuce plants, mostly 13-15 days apart as foliar sprays using CO₂ backpack or hand-held plot sprayers, applying about 350-700 litres water/ha. Five of these trials were reverse decline studies, where separate plots were treated at various intervals before a single harvest date. Plot sizes in these trials ranged from 5-30 square metres.

Samples of at least 12 plants (with decaying leaves removed) were taken from each plot, frozen and stored for up to 14 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-4.0 mg/kg acetamiprid ranged from 70-109%.

Table 64 Residues in field lettuce from supervised trials in Europe involving foliar applications of acetamiprid (SP formulations)

LETTUCE Location, Year (variety)	Form, % ai	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
France (N) Coulomb (Ille et Vilaine) 1998 (Domino)	SP 20	2	0.05 0.05	0.012	Leaves	3 7	0.39 <u>0.28</u>	RD-00765 98519RN1 Aceta_056
France (N) Mézerolles (Somme), 1998 (Titan)	SP 20	2	0.06	0.012	Leaves	0	0.81	RD-00766 98520AM1
		2	0.06	0.012	Leaves	3	0.15	
		<u>2</u>	0.06	0.012	Leaves	<u>8</u>	<u>0.14</u>	Aceta_057
		2	0.06	0.012	Leaves	16	< 0.01	Reverse decline study
France (N) Saint Lambert Des Levés, Pays de Loire 2002 (Versailles)	SP 20	2	0.05	0.008	Leaves	0 3 7	0.64 0.66 <u>0.24</u>	RD-01042 02R600-4 Aceta_058
Germany Dannstadt, RLP 2000 (Batavia Rot)	SP 20	2	0.05	0.008	Leaves	0	0.59	RD-01041
		2	0.05	0.008	Leaves	3	0.19	DEU0201
		<u>2</u>	0.05	0.008	Leaves	<u>7</u>	<u>0.08</u>	Aceta_059 Reverse decline study

LETTUCE Location, Year (variety)	Form, % ai	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
Germany Bornheim, NRW 2000 (Lollo Rosso)	SP 20	2	0.05	0.008	Leaves	0 3 7	1.6 0.58 <u>0.31</u>	RD-01041 DEU0501 Aceta_-59
Germany Lambsheim, RLP 2002 (Anthony)	SP 20	2	0.05	0.008	Leaves	0 3 7	1.1 0.32 <u>0.15</u>	RD-01042 02R600-1 Aceta_058
UK Marsh/Holbeach, Lincolnshire 2000 (Lollo Rosso)	SP 20	2	0.05	0.008	Leaves	0 3 7	1.1 0.63 <u>0.16</u>	RD-01041 GBR0101 Aceta_059
UK Spalding, Lincolnshire 2002 (Lollo Rosso)	SP 20	2	0.05	0.008	Leaves	0 3 7	0.62 0.75 <u>0.25</u>	RD-01042 02R600-3 Aceta_058
France (S) Romegoux, (Charente-Maritimes) 1998 (Nadine)	SP 20	2	0.05	0.013	Leaves	3 7	0.11 <u>0.04</u>	RD-00765 98519BX1 Aceta_056
France (S) Champagne, (Charent-Maritime) 1998 (Rosalie)	SP 20	2	0.05	0.013	Leaves	0 3 7 14	0.74 0.14 <u>0.1</u> < 0.01	RD-00766 98520BX1 Aceta_057
Spain Alginet (Valencia) 1998 (Inverna)	SP 20	2	0.08 0.15	0.008 0.008	Leaves	0 3 7 14	1.3 0.68 0.3 0.04	RD-00105 98628V1 Aceta_060
Italy Bologna 1999 (Canasta)	SP 20	2	0.05	0.007	Leaves	0 3 7 14 21	0.34 0.4 <u>0.14</u> 0.01 < 0.01	RD-00796 99687BO1 Aceta_061
Italy Bologna, Emilia- Romagna 2000 (Laramy)	SP 20	2	0.05	0.008	Leaves	0 3 7	0.39 0.16 <u>0.11</u>	RD-01040 ITA0101 Aceta_062
Italy Andria (Bari), Puglia 2000 (Calgary)	SP 20	2	0.05	0.008	Leaves	0 3 7	0.13 0.07 <u>0.06</u>	RD-01040 ITA0201 Aceta_062
Spain Alginet, Valencia 2000 (Inverna)	SP 20	2	0.05	0.008	Leaves	0 3 7	0.42 0.14 <u>0.06</u>	RD-01040 ESP0101 Aceta_062
Spain Palma-Cartagena 2000 (Almadin)	SP 20	2	0.05	0.01	Leaves	0 3 7	0.55 0.19 <u>0.17</u>	RD-01040 ESP0301 Aceta_062

Table 65 Residues in protected lettuce from supervised trials in Europe involving foliar applications of acetamiprid (SP formulations)

LETTUCE Location, Year (variety)	Form	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
France St Denis en Val (Loiret) 1998 (Colette)	SP 20	2	0.05	0.015	Leaves	3 7	<u>0.5</u> 0.32	RD-00774 98517OR1 Aceta_063
France Ricaud (Aude) 1998 (Sensai)	SP 20	2	0.05	0.01	Leaves	3 7	<u>0.33</u> 0.25	RD-01241 98752TL1 Aceta_064
France Ouvrouer les Champs (Loiret) 1999 (Angie)	SP 20	2	0.06	0.01	Leaves	0	2.0	RD-00782
	SP 20	2	0.06	0.01	Leaves	3	1.1	98754OR1
	SP 20	1+ 1	0.06 0.07	0.01 0.01	Leaves	7	1.1	Aceta_065
	SP 20	1+ 1	0.07 0.06	0.011 0.01	Leaves	14	0.88	Reverse decline study
France Green house 1998 (Sensai)	SP 20	2	0.05	0.01	Leaves	3 7	<u>0.33</u> 0.25	R-00783 98752TL1 Aceta_066
France Lapeyrouse Fossat (Haute Garonne) 1998 (Flandria)	SP 20	2	0.05	0.01	Leaves	0 3 7 14	0.74 <u>0.88</u> 0.81 0.15	RD-00782 98754TL1 Aceta_065
France Entraigues-sur- Sorgues 1999 (Noemie)	SP 20	2	0.05	0.007	Leaves	0	0.94	RD-00800
			0.05	0.007	Leaves	3	0.35	99537AV1
			<u>0.05</u>	<u>0.007</u>	<u>Leaves</u>	<u>7</u>	<u>0.41</u>	Aceta_067
			0.05	0.007	Leaves	14	0.15	Reverse decline study
France St Denis en Val (Loiret) 1999 (Bercie)	SP 20	2	0.05	0.007	Leaves	3 7	<u>0.88</u> 0.8	RD-00805 99536OR1 Aceta_068
France Lucenay, Rhone-Alpes 2001 (Kristo)	SP 20	2	0.05	0.009	Heads	0 7	1.9 1.0	RD-01039 01R602-1 Aceta_069
Germany Bornheim, NRW 2000 (Lello Rosso)	SP 20	2	0.05	0.008	Leaves	0	2.8	RD-01038
	SP 20	2	0.05	0.008	Leaves	4	1.8	DEU0501
	<u>SP 20</u>	<u>2</u>	<u>0.05</u>	<u>0.008</u>	<u>Leaves</u>	<u>7</u>	<u>1.9</u>	Aceta_070
UK Brentwood, Essex 2000 (Vagus)	SP 20	2	0.05	0.008	Leaves	0	1.5	RD-01038
	SP 20	2	0.05	0.008	Leaves	3	0.68	GBR0102
	<u>SP 20</u>	<u>2</u>	<u>0.05</u>	<u>0.008</u>	<u>Leaves</u>	<u>7</u>	<u>0.78</u>	Aceta_070
								Reverse decline study

LETTUCE Location, Year (variety)	Form	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
UK Algakirk / Boston, Lincolnshire 2001 (Emerald)	SP 20	2	0.05	0.008	Leaves	0 7	2.8 1.6	RD-01039 01R602-2 Aceta_069
Italy Pontegradella – Ferrara, Emilia Romagna 2001 (Salad Bowl)	SP 20	2	0.05	0.008	Leaves	0 7	1.8 1.5	RD-01039 01R602-3 Aceta_069

Spinach

Results from supervised trials in USA on spinach were provided to the Meeting. In these trials, 5 applications of acetamiprid (WP formulations) were applied to spinach plants, 7 days apart as foliar sprays using CO₂ backpack or tractor-mounted sprayers and 4-8 nozzle booms, applying about 90-200 litres water/ha. Plot sizes in these trials ranged from 46-223 square metres.

Duplicate samples of at least 2 kg (leaves) were taken from each plot, frozen and stored for up to 16 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-3.0 mg/kg acetamiprid ranged from 84-109%.

Table 66 Residues in spinach from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

SPINACH Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (NY) 1997 (Medania)	WP 70	1+ 2+ 1+ 1	0.09 0.08 0.09 0.08	0.046 0.045 0.046 0.045	Leaf	7	0.03, 0.04	<u>0.04</u>	RD-00749 12111-01, Aceta_071
USA (DE) 1997 (Tye)	WP 70	1+ 3+ 1	0.08 0.09 0.08	0.051 0.051 0.05	Leaf	7	0.24, 0.184	<u>0.21</u>	RD-00749 12111-02 Aceta_071
USA (NC) 1998 (Vienna-F1)	WP 70	5	0.09	0.09	Leaf	7	0.04, 0.03	<u>0.03</u>	RD-00749 12111-03 Aceta_071
USA (TX) 1997 (Bolero)	WP 70	5	0.08	0.06	Leaf	6	2.6, 2.4, 2.5	<u>2.5</u>	RD-00749 12111-05 Aceta_071
USA (CO) 1997 (Melody F1 RS)	WP 70	3+ 2	0.09 0.08	0.06 0.06	Leaf	7	0.59, 0.52	<u>0.55</u>	RD-00749 12111-06 Aceta_071
USA (CO) 1997 (Bolera RS)	WP 70	1+ 1+ 3	0.09 0.08 0.09	0.049 0.048 0.047	Leaf	7	2.2, 2.0	<u>2.1</u>	RD-00749 12111-08 Aceta_071

SPINACH Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (TX) 1998 (Skookum)	WP 70	1+ 4	0.08 0.09	0.044 0.045	Leaf	7	1.2, 1.1	<u>1.1</u>	RD-00749 12111-09, Aceta_071
USA (TX) 1998 (Polka F1 RS)	WP 70	5	0.08	0.06	Leaf	7	0.46, 0.47	<u>0.46</u>	RD-00749 12111-10, Aceta_071

Mustard greens

Results from supervised trials in USA on mustard greens were provided to the Meeting. In these trials, 5 applications of acetamiprid (WP formulations) were applied to mustard greens, 7 days apart as foliar sprays using pressurised backpack or tractor-mounted sprayers with 4-8 nozzle booms, applying about 94-380 litres water/ha. Plot sizes in these trials ranged from 93-250 square metres.

Duplicate leaf samples from at least 12 plants were taken from each plot, frozen within 2 hours and stored frozen for up to 13 months before analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.05-1.5 mg/kg acetamiprid ranged from 78-110%.

Table 67 Residues in mustard greens from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

MUSTARD GREENS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (NC) 1997 (Southern Giant Curled Mustard)	WP 70	1+ 3+ 1	0.08 0.09 0.08	0.09 0.09 0.089	leaves	7	0.18, 0.19	<u>0.19</u>	RD-00771 12647-01 Aceta_079
USA (FL) 1997 (Florida Broadleaf Mustard)	WP 70	1+ 4	0.09 0.08	0.03 0.03	leaves	7	0.09, 0.12	<u>0.11</u>	RD-00771 12647-03 Aceta_079
USA (MS) 1997 (India Mustard Florida Broadleaf)	WP 70	1+ 3+ 1	0.09 0.08 0.09	0.045 0.045 0.045	leaves	7	0.41, 0.44	<u>0.43</u>	RD-00771 12647-04 Aceta_079
USA (MI) 1997 (Southern Giant Curled Mustard)	WP 70	1+ 4	0.09 0.08	0.037 0.039	leaves	7	0.3, 0.29	<u>0.3</u>	RD-00771 12647-05 Aceta_079
USA (TX) 1997 (Southern Dwarf Curled Mustard)	WP 70	3+ 2	0.08 0.09	0.06 0.06	leaves	6	1.1, 1.1, 1.1	<u>1.1</u>	RD-00771 12647-06 Aceta_079

MUSTARD GREENS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (CA) 1997 (Southern Giant Curled Mustard)	WP 70	1+ 2+ 2	0.08 0.09 0.08	0.022 0.022 0.023	leaves	8	0.16, 0.2	<u>0.18</u>	RD-00771 12647-07 Aceta_079
USA (CA) 1997 (Mizuna Leaf Mustard)	WP 70	2+ 3	0.09 0.08	0.023 0.023	leaves	7	0.48, 0.49	<u>0.49</u>	RD-00771 12647-08 Aceta_079
USA (CA) 1997 (Florida Broadleaf Mustard)	WP 70	2+ 1+ 1+ 1	0.09 0.08 0.09 0.08	0.06 0.057 0.058 0.057	leaves	7	0.73, 0.84, 0.66	<u>0.74</u>	RD-00771 12647-09 Aceta_079
USA (SC) 1997 (Florida Broadleaf Mustard)	WP 70	4+ 1	0.09 0.08	0.045 0.044	leaves	8	0.72, 0.63	<u>0.68</u>	RD-00771 12647-10 Aceta_079

Legume vegetables

Beans, Peas

Results from supervised trials in USA on beans and peas (both with and without pods) were provided to the Meeting. In these trials, 3-4 applications of acetamiprid (WP formulations) were applied to beans and peas, 7-8 days apart as foliar sprays, applying about 100-280 litres water/ha. Plot sizes in these trials ranged from 25-200 square metres.

Duplicate samples of at least 1 kg peas (with and without pods), green beans (with pods) and Lima beans (without pods) were taken from each plot, frozen within 6 hours and stored frozen for up to 9 months before analysis for acetamiprid using Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01-0.5 mg/kg acetamiprid ranged from 72-109%.

Table 68 Residues in beans and peas (without pods) from supervised trials on peas and Lima beans in USA, involving foliar applications of acetamiprid (WP formulations)

BEANS & PEAS (no pods) Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Turner (GA) 2004 (Turner)	WP 70	3	0.11	0.079	Lima bean	7	0.09, 0.08	<u>0.08</u>	RD-00903 2004-13-GA-04 Aceta_082
USA Tift (GA) 2004 (Fordhook 242)	WP 70	3	0.11	0.081	Lima bean	7	0.04, 0.03	<u>0.03</u>	RD-00903 2004-13-GA-06 Aceta_082
USA Grady (GA) 2004 (Bridgeston)	WP 70	3	0.11	0.078	Lima bean	7	0.13, 0.09	<u>0.11</u>	RD-00903 2004-13-GA-07 Aceta_082

BEANS & PEAS (no pods) Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Alachua (FL) 2004 (Bridgeston)	WP 70	3	0.11	0.074	Lima bean	7	0.04, 0.04	<u>0.04</u>	RD-00903 2004-13-FL-08 Aceta_082
USA Pepin (WI) 2004 (Henderson bush)	WP 70	1+ 1+ 1	0.11 0.12 0.11	0.061 0.061 0.061	Lima bean	7	0.19, 0.17	<u>0.18</u>	RD-00903 2004-13-WI-13 Aceta_082
USA Tulare (CA) 2004 (Fordhook 242)	WP 70	2+ 1	0.11 0.12	0.048 0.049	Lima bean	7	0.02, 0.02	<u>0.02</u>	RD-00903 2004-13-CA-16 Aceta_082
USA Lehigh (PA) 2004 (Wando)	WP 70	2+ 1	0.12 0.11	0.042 0.043	Shelled pea	7	0.01, 0.01	<u>0.01</u>	RD-00903 2004-13-PA-01 Aceta_082
USA Pepin (WI) 2004 (Lazor)	WP 70	1+ 1+ 1	0.12 0.11 0.12	0.061 0.061 0.061	Shelled pea	7	0.03, 0.03	<u>0.03</u>	RD-00903 2004-13-WI-10 Aceta_082
USA Ottawa (MI) 2004 (Green arrow)	WP 70	3	0.11	0.041	Shelled pea	7	0.03, 0.03	<u>0.03</u>	RD-00903 2004-13-MI-11 Aceta_082
USA Wabasha (WI) 2004 (Lazor)	WP 70	3	0.11	0.061	Green pea	7	0.01, 0.02	<u>0.02</u>	RD-00903 2004-13-WI-12 Aceta_082
USA Jefferson (OR) 2004 (Seedwest)	WP 70	1+ 2	0.12 0.11	0.097 0.11	Green pea	8	< 0.01, < 0.01	<u>< 0.01</u>	RD-00903 2004-13-OR-20 Aceta_082
USA Skagit (WA) 2004 (Snake)	WP 70	3	0.11	0.075	Green pea	7	0.03, 0.02	<u>0.02</u>	RD-00903 2004-13-WA-21 Aceta_082

Table 69 Residues in beans and peas (with pods) from supervised trials on peas and green beans in USA, involving foliar applications of acetamiprid (WP formulations)

COMMON BEAN & PEAS (with pod) Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Lehigh (PA) 2004 (Savannah)	WP 70	1+ 1+ 1	0.12 0.11 0.12	0.044 0.043 0.044	Green bean	7	0.19, 0.17	<u>0.18</u>	RD-00903 2004-13-PA-02 Aceta_082
USA Tift (GA) 2004 (Broncho)	WP 70	3	0.11	0.045	Green bean	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00903 2004-13-GA-05 Aceta_082
USA Ottawa (MI) 2004 (Bush blue lake 274)	WP 70	3	0.11	0.043	Green bean	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00903 2004-13-MI-14 Aceta_082

COMMON BEAN & PEAS (with pod) Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Pepin (WI) 2004 (Bush blue lake 274)	WP 70	1+ 1+ 1	0.12 0.11 0.12	0.061 0.061 0.061	Green bean	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00903 2004-13-WI-09 Aceta_082
USA Jefferson (OR) 2004 (91 GR)	WP 70	3	0.12	0.061	Green bean	7	< 0.01, < 0.01	<u>< 0.01</u>	RD-00903 2004-13-OR-19 Aceta_082
USA Hamilton (FL) 2004 (Bronco)	WP 70	3	0.11	0.075	Green bean	7	0.02, 0.02	<u>0.02</u>	RD-00903 2004-13-FL-22 Aceta_082
USA Huntingdon (NJ) 2004 (Dwarf white blossom)	WP 70	1+ 2	0.11 0.12	0.043 0.042	Sugar pea	7	0.11, 0.15	<u>0.13</u>	RD-00903 2004-13-NJ-03 Aceta_082
USA Hockley (TX) 2004 (Sugarsnap)	WP 70	2+ 1	0.12 0.11	0.048 0.049	Snap pea in pod	8	0.26, 0.28	<u>0.27</u>	RD-00903 2004-13-TX-15 Aceta_082 10, 4 day intervals
USA Tulare (CA) 2004 (Oregon sugar pod II)	WP 70	1+ 2	0.12 0.11	0.04 0.04	Sugar pea	7	0.07, 0.09	<u>0.08</u>	RD-00903 2004-13-CA-17 Aceta_082

Celery

Results from supervised trials in USA on celery were provided to the Meeting. In these trials, 5 applications of acetamiprid (WP formulations) were applied to celery plants, 7 days apart as foliar sprays using CO₂ backpack or tractor-mounted sprayers with 2–8 nozzle booms, applying about 130–200 litres water/ha. Plot sizes in these trials ranged from 53–105 square metres.

Duplicate samples of at least 12 bunches were taken from each plot and stored frozen until analysis for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in samples fortified with 0.01–0.5 mg/kg acetamiprid ranged from 83–102%. Sample storage intervals were less than 12 months except in three trials, where samples were stored for up to 17 months before analysis.

Table 70 Residues in celery from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

CELERY Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (FL) 1997 (Junee Belle)	WP 70	3+ 2	0.08 0.09	0.045 0.045	Stalk + Leaves	7	0.78, 0.78	<u>0.78</u>	RD-00750 12112-01 17 mo storage Aceta_072
USA (FL) 1997 (June Belle)	WP 70	1+ 1+ 2+ 1	0.08 0.09 0.08 0.09	0.051 0.05 0.05 0.05	Stalk + Leaves	7	0.17, 0.18	<u>0.17</u>	RD-00750 12112-02 17 mo storage Aceta_072

CELERY Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (CA) 1997 (Conquistador)	WP 70	1+ 4	0.09 0.08	0.047 0.048	Stalk + Leaves	7	0.35, 0.29	<u>0.32</u>	RD-00750 12112-04 Aceta_072
USA (CA) 1997 (Conquistador)	WP 70	2+ 1+ 1+ 1	0.09 0.08 0.09 0.08	0.049 0.049 0.049 0.049	Stalk + Leaves	7	0.29, 0.25	<u>0.27</u>	RD-00750 12112-05 Aceta_072
USA (CA) 1997 (Conquistador)	WP 70	2+ 2+ 1	0.08 0.09 0.08	0.049 0.05 0.05	Stalk + Leaves	7	0.28, 0.26	<u>0.27</u>	RD-00750 12112-07 17 mo storage Aceta_072
USA (MI) 1998 (Florida 683K)	WP 70	1+ 1+ 1+ 2	0.09 0.08 0.09 0.08	0.047 0.057 0.057 0.058	Stalk + Leaves	7	0.43, 0.4	<u>0.41</u>	RD-00750 12112-09 Aceta_072
USA (CA) 1998 (Sonora)	WP 70	3+ 1+ 1	0.09 0.08 0.09	0.061 0.061 0.06	Stalk + Leaves	7	0.5, 0.52	<u>0.51</u>	RD-00750 12112-10, Aceta_072
USA (CA) 1998 (108)	WP 70	2+ 2+ 1	0.09 0.08 0.09	0.054 0.051 0.054	Stalk + Leaves	7	0.08, 0.07	<u>0.08</u>	RD-00750 12112-11 Aceta_072

Tree nuts

Almonds, Pecans

Results from supervised trials in USA on almonds and pecans were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to mature trees, 14 days apart as foliar sprays using airblast sprayers, applying either 950–2400 litres water/ha (high volume) or 410–930 litres/ha (low volume). Plot sizes in these trials ranged from 134–940 square metres.

Duplicate samples of at least 2 kg fruit were taken from each plot, shelled in the field and the nutmeat frozen within 5 hours of sampling and stored frozen for up to 4 months before analysis for acetamiprid using Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in untreated nutmeat samples fortified with 0.01–0.05 mg/kg acetamiprid ranged from 85–103% in almonds and 73–102% in pecans.

Table 71 Residues in almonds from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

ALMONDS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Kern (CA) 2003 (Non Parcel)	WP 70	4	0.2	0.011	Nutmeat	14	0.02, 0.02	<u>0.02</u>	RD-00452 2003-20-CA-01 Aceta_050
USA Tulare (CA) 2003 (Carmel)	WP 70	2+ 2	0.2 0.2	0.048 0.035	Nutmeat	14	< 0.01, < 0.01	<u>< 0.01</u>	RD-00452 2003-20-CA-02 Aceta_050

ALMONDS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Tulare (CA) 2003 (Butte)	WP 70	4	0.2	0.008	Nutmeat	14	0.01, 0.01	<u>0.01</u>	RD-00452 2003-20-CA-03 Aceta_050
USA King’s (CA) 2003 (Fritz)	WP 70	4	0.2	0.028	Nutmeat	14	< 0.01, < 0.01	<u>≤ 0.01</u>	RD-00452 2003-20-CA-04 Aceta_050
USA Kern (CA) 2003 (Fritz)	WP 70	4	0.2	0.008	Nutmeat	14	< 0.01, 0.01	<u>0.01</u>	RD-00452 2003-20-CA-05 Aceta_050
USA Tulare (CA) 2003 (Fritz)	WP 70	4	0.2	0.039	Nutmeat	14	< 0.01, < 0.01	<u>≤ 0.01</u>	RD-00452 2003-20-CA-11 Aceta_050

Table 72 Residues in pecans from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

PECANS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Tift, (GA) 2003 (Sumner)	WP 70	4	0.2	0.043	Nutmeat	14	< 0.01, < 0.01	<u>< 0.01</u>	RD-00452 2003-20-GA-06 Aceta_050
USA Berrien (GA) 2003 (Stewart)	WP 70	4	0.2	0.014	Nutmeat	14	< 0.01, < 0.01	<u>< 0.01</u>	RD-00452 2003-20-GA-07 Aceta_050
USA Lonoke (AR) 2003 (Stuart)	WP 70	4	0.2	0.021	Nutmeat	14	< 0.01, < 0.01	<u>< 0.01</u>	RD-00452 2003-20-AR-08 Aceta_050
USA Uvalde (TX) 2003 (Stuart)	WP 70	4	0.2	0.037	Nutmeat	14	0.05, 0.04	<u>0.05</u>	RD-00452 2003-20-TX-09 Aceta_050
USA Hockley (TX) 2003 (Western Schluy)	WP 70	4	0.2	0.017	Nutmeat	14	< 0.01, 0.013	<u>0.01</u>	RD-00452 2003-20-TX-10 Aceta_050
USA Brooks (GA) 2003 (Stewart)	WP 70	4	0.2	0.021	Nutmeat	14	< 0.01, < 0.01	<u>< 0.01</u>	RD-00452 2003-20-GA-12 Aceta_050

Cotton seed

Results from supervised trials in USA on cotton seed were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to cotton, 5-7 days apart as foliar sprays using pressurised backpack or tractor-mounted sprayers with 4-9 nozzle booms, applying about 80-190 litres water/ha. Plot sizes in these trials ranged from 120-2600 square metres.

Duplicate seed cotton samples (min 22 kg) were collected from machine-harvested plots, frozen within 20 hours and stored frozen for up to 9 months before ginning to separate the cotton seed from the gin trash. The seed samples were analysed for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg and recovery rates in samples fortified with 0.01-0.5 mg/kg acetamiprid ranged from 70-106%.

Table 73 Residues in cotton seed from supervised trials on cotton in USA, involving foliar applications of acetamiprid (WP formulations)

COTTON SEED Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (NC) 1997 (DP50)	WP 70	4	0.11	0.12	Cotton seed	28	0.03, 0.02	<u>0.02</u>	RD-00793 12104-01 Aceta_080
USA (MS) 1997 (DP50)	WP 70	4	0.11	0.119	Cotton seed	61	< 0.01, < 0.01	< 0.01	RD-00793 12104-02 Aceta_080
USA (AR) 1997 (PM 1215 RR)	WP 70	4	0.11	0.08	Cotton seed	29	0.16, 0.04	<u>0.1</u>	RD-00793 12104-03 Aceta_080
USA (AR) 1997 (DBL 33B)	WP 70	4	0.11	0.129	Cotton seed	29	< 0.01, < 0.01	<u>< 0.01</u>	RD-00793 12104-04 Aceta_080
USA (MS) 1997 (STV 474)	WP 70	2+ 1+ 1	0.11 0.11 0.11	0.097 0.073 0.094	Cotton seed	28	0.07, 0.11	<u>0.09</u>	RD-00793 12104-05-T 2 Aceta_080
USA (MS) 1997 (STV 474)	WP 70	2+ 1+ 1	0.11 0.11 0.11	0.098 0.073 0.094	Cotton seed	20 25 31 35	0.09 0.07 0.09 0.04	0.09 0.07 0.09 0.04	RD-00793 12104-05-T 3 Aceta_080
USA (TX) 1997 (Suregrow 125)	WP 70	4	0.11	0.12	Cotton seed	29	0.05, 0.06	<u>0.06</u>	RD-00793 12104-06 Aceta_080
USA (TX) 1997 (All-Tex Atlas)	WP 70	4	0.11	0.12	Cotton seed	30	0.12, 0.12	<u>0.12</u>	RD-00793 12104-07 Aceta_080
USA (TX) 1997 (PM 2200 RR)	WP 70	4	0.11	0.12	Cotton seed	27	0.12, 0.08	<u>0.1</u>	RD-00793 12104-08 Aceta_080
USA (TX) 1997 (Deltapine 50)	WP 70	4	0.11	0.088	Cotton seed	29	0.02, 0.03	<u>0.02</u>	RD-00793 12104-09 Aceta_080
USA (TX) 1997 (Pay-master HS 26)	WP 70	1+ 1+ 1+ 1	0.11 0.11 0.11 0.11	0.09 0.129 0.124 0.13	Cotton seed	33	0.49, 0.5	<u>0.5</u>	RD-00793 12104-10 Aceta_080
USA (TX) 1997 (Pay-master HS 280)	WP 70	1+ 3	0.11 0.11	0.135 0.124	Cotton seed	27	0.34, 0.37	<u>0.36</u>	RD-00793 12104-11 Aceta_080

COTTON SEED Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (CA) 1997 (Sure-Grow 501)	WP 70	1+ 1+ 2	0.11 0.12 0.11	0.072 0.064 0.069	Cotton seed	28	0.02, 0.04	<u>0.03</u>	RD-00793 12104-12 Aceta_080
USA (CA) 1997 (Maxxa)	WP 70	4	0.11	0.06	Cotton seed	28	0.12, 0.15	<u>0.14</u>	RD-00793 12104-13 Aceta_080 (5-day intervals)
USA (CA) 1997 (Maxxa)	WP 70	4	0.11	0.116	Cotton seed	28	0.05, 0.04	0.05	RD-00793 12104-14 T 2 Aceta_080
USA (CA) 1997 (Maxxa)	WP 70	4	0.11	0.116	Cotton seed	21 24 27 32 35	0.06 0.09 0.05, 0.04 0.04 < 0.01	0.06 0.09 <u>0.05</u> 0.04 < 0.01	RD-00793 12104-14 T 3 Aceta_080

Miscellaneous animal feed commodities

Almond hulls

Results from supervised trials in USA on almond hulls were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to mature trees, 14 days apart as foliar sprays using airblast sprayers, applying either 950-2400 litres water/ha (high volume) or 410-930 litres/ha (low volume). Plot sizes in these trials ranged from 134-940 square metres.

Duplicate samples of at least 2 kg fruit were taken from each plot, shelled in the field and the hulls frozen within 5 hours of sampling and stored frozen for up to 4 months before analysis for acetamiprid using Method KP-216, with an LOQ of 0.01 mg/kg. Recovery rates in untreated hull samples fortified with 0.01-5.0 mg/kg acetamiprid ranged from 70-93%.

Table 74 Residues in almond hulls from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

ALMONDS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Kern (CA) 2003 (Non Parcil)	WP 70	4	0.2	0.011	Hulls	14	2.2, 1.6	<u>1.9</u>	RD-00452 2003-20-CA-01 Aceta_050
USA Tulare (CA) 2003 (Carmel)	WP 70	2+ 2	0.2 0.2	0.048 0.035	Hulls	14	0.84, 0.72	<u>0.78</u>	RD-00452 2003-20-CA-02 Aceta_050
USA Tulare (CA) 2003 (Butte)	WP 70	4	0.2	0.008	Hulls	14	3.6, 4.0	<u>3.8</u>	RD-00452 2003-20-CA-03 Aceta_050
USA King’s (CA) 2003 (Fritz)	WP 70	4	0.2	0.028	Hulls	14	0.18, 0.27	<u>0.22</u>	RD-00452 2003-20-CA-04 Aceta_050

ALMONDS Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA Kern (CA) 2003 (Fritz)	WP 70	4	0.2	0.008	Hulls	14	2.5, 1.5	<u>2.0</u>	RD-00452 2003-20-CA-05 Aceta_050
USA Tulare (CA) 2003 (Fritz)	WP 70	4	0.2	0.039	Hulls	14	0.82, 0.74	<u>0.78</u>	RD-00452 2003-20-CA-11 Aceta_050

Cotton gin trash

Results from supervised trials in USA on cotton seed gin trash were provided to the Meeting. In these trials, 4 applications of acetamiprid (WP formulations) were applied to cotton, 5-7 days apart as foliar sprays using pressurised backpack or tractor-mounted sprayers with 4-9 nozzle booms, applying about 80-190 litres water/ha. Plot sizes in these trials ranged from 120-2600 square metres.

Duplicate seed cotton samples (min 22 kg) were collected from machine-harvested plots, frozen within 20 hours and stored frozen for up to 9 months before ginning to separate the cotton seed from the gin trash. The gin trash samples were analysed for acetamiprid using Method EC-521, with an LOQ of 0.01 mg/kg. Recovery rates in gin trash samples fortified with 0.01-25 mg/kg acetamiprid ranged from 73-116%.

Table 75 Residues in cotton gin trash from supervised trials on cotton in USA, involving foliar applications of acetamiprid (WP formulations)

COTTON SEED Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (NC) 1997 (DP50)	WP 70	4	0.11	0.12	Gin trash	28	2.7, 2.6	<u>2.7</u>	RD-00793 12104-01 Aceta_080
USA (MS) 1997 (DP50)	WP 70	4	0.11	0.119	Gin trash	61	0.15, 0.31 (c = 0.018)	0.23	RD-00793 12104-02 Aceta_080
USA (AR) 1997 (PM 1215 RR)	WP 70	4	0.11	0.08	Gin trash	29	1.9, 1.9	<u>1.9</u>	RD-00793 12104-03 Aceta_080
USA (AR) 1997 (DBL 33B)	WP 70	4	0.11	0.129	Gin trash	29	0.35, 0.42 (c=0.013)	<u>0.39</u>	RD-00793 12104-04 Aceta_080
USA (MS) 1997 (STV 474)	WP 70	2+ 1+ 1	0.11 0.11 0.11	0.097 0.073 0.094	Gin trash	28	3.8, 4.2	<u>4.0</u>	RD-00793 12104-05-T 2 Aceta_080
USA (MS) 1997 (STV 474)	WP 70	2+ 1+ 1	0.11 0.11 0.11	0.098 0.073 0.094	Gin trash	20 25 31 35	7.0 5.2 3.0 1.7	7.0 5.2 <u>3.0</u> <u>1.7</u>	RD-00793 12104-05-T 3 Aceta_080

COTTON SEED Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (TX) 1997 (Suregrow 125)	WP 70	4	0.11	0.12	Gin trash	29	5.3, 6.2	<u>5.8</u>	RD-00793 12104-06 Aceta_080
USA (TX) 1997 (All-Tex Atlas)	WP 70	4	0.11	0.12	Gin trash	30	1.6, 1.4	<u>1.5</u>	RD-00793 12104-07 Aceta_080
USA (TX) 1997 (PM 2200 RR)	WP 70	4	0.11	0.12	Gin trash	27	4.2, 3.5	<u>3.9</u>	RD-00793 12104-08 Aceta_080
USA (TX) 1997 (Deltapine 50)	WP 70	4	0.11	0.088	Gin trash	29	1.6, 2.1 (c=0.025)	<u>1.9</u>	RD-00793 12104-09 Aceta_080
USA (TX) 1997 (Pay-master HS 26)	WP 70	1+ 1+ 1+ 1	0.11 0.11 0.11 0.11	0.09 0.129 0.124 0.13	Gin trash	33	6.4, 6.6	<u>6.5</u>	RD-00793 12104-10 Aceta_080
USA (TX) 1997 (Pay-master HS 280)	WP 70	1+ 3	0.11 0.11	0.135 0.124	Gin trash	27	3.7, 4.0	<u>3.9</u>	RD-00793 12104-11 Aceta_080
USA (CA) 1997 (Sure-Grow 501)	WP 70	1+ 1+ 2	0.11 0.12 0.11	0.072 0.064 0.069	Gin trash	28	6.5, 8.0 (c=0.022)	<u>7.3</u>	RD-00793 12104-12 Aceta_080
USA (CA) 1997 (Maxxa)	WP 70	4	0.11	0.06	Gin trash	28	19, 17 (c=0.015)	<u>18</u>	RD-00793 12104-13 Aceta_080 (5-day intervals)
USA (CA) 1997 (Maxxa)	WP 70	4	0.11	0.116	Gin trash	28	3.2, 4.0 (c=0.085)	3.6	RD-00793 12104-14 T 2 Aceta_080
USA (CA) 1997 (Maxxa)	WP 70	4	0.11	0.116	Gin trash	21 24 27 32 35	4.4 10.0 3.2, 4.0 0.54 0.46	4.4 10.0 <u>3.6</u> 0.54 0.46	RD-00793 12104-14 T 3 Aceta_080

FATE OF RESIDUES IN STORAGE AND PROCESSING

Nature of the Residue in Processing

The hydrolytic behaviour of acetamiprid was investigated in an aqueous hydrolysis radiolabel study reported by Gomyo (1997) [Ref: Aceta_008].

Duplicate samples of standard buffer solutions at pH values of 4, 5, 7 and 9 were fortified with [pyridine-2, 6-¹⁴C]-acetamiprid. The fortified samples (about 10 mg ai/L) were incubated at 22, 35 and 45 °C in the dark, under sterile conditions for various intervals up to 35 days before TLC analysis.

Acetamiprid was stable for at least 35 days in buffer solutions at pH 4, 5 and 7 and over the temperature ranges tested, with more than 95% of the applied radiolabel being identified as the parent

compound and metabolites IM-1-3 and IM-1-4 being found at less than 1% AR, except in the pH 7 samples incubated at 45 °C for more than 5 days, where the IM-1-3 metabolite was measured at 1.4-4.3% AR.

In the pH 9 buffer solutions, acetamiprid degraded to about 92% AR (22 °C), 60% AR (35 °C) and 15% AR (43 °C), with residues of the IM-1-3 and IM-1-4 metabolites increasing to about 64% AR and 21% AR respectively by the end of the 35-day study period.

Magnitude of the residue in processing

The Meeting received information on the fate of acetamiprid residues during simulated commercial processing of oranges, tomato, apple, cotton and grapes.

Oranges

In a field trial conducted in Spain by Fraschini, (1998) [Ref: Aceta_045], orange trees were treated twice with 0.34–0.36 kg ai/ha (0.01 kg ai/hL) and samples of oranges were collected 14 days after the last application and separated into pulp, peel and orange juice.

In a more extensive study in USA reported by Gough, (1999) [Ref: Aceta_048] orange trees were treated four times with about 0.42 kg ai/ha and then with an exaggerated rate of 1.44 kg ai/ha, 7 days before harvest.

Oranges were washed and then scarified to obtain the oil. Oil was separated by passing through a cream separator and centrifuged. The scarified oranges were transferred to a juice separator, which halves the fruit and removes the juice with auger reamers. Juice was extracted and the remaining pulp was shredded and combined with the scarified flavedo, rag and seeds before being adjusted to a pH of 8–10 (by adding lime) and pressed. The pressed wet pulp subsamples were air dried to less than 10% moisture and then milled to obtain dry pulp.

Table 76 Residues in oranges and processed orange fractions from supervised trials in Spain and USA involving foliar applications of acetamiprid (SP or WP formulations)

ORANGE Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	sample results	mean	
Spain Valencia 1997 (Navelina)	SP 20	2	0.35	0.01	Whole fruit	14	0.08	0.08	RD-00170 NTF/2/1S/O Aceta_045
					Pulp	14	0.03	0.03	
					Peel	14	0.23	0.23	
					Juice	14	< 0.01	< 0.01	
Spain Valencia 1997 (Navelina)	SP 20	1+	0.36	0.01	Whole fruit	7	0.27	0.27	RD-00170 NTF/2/2S/O
			0.34	0.01	Juice	7	< 0.01	< 0.01	
					Whole fruit	14	0.28	0.28	Aceta_045
					Pulp	14	0.03	0.03	
					Peel	14	0.78	0.78	
USA FL 1997 (Valencia)	WP 70	1+	0.43	0.027	Fruit	7	0.31	0.31	RD-00753 97512103
		3+	0.42	0.027	Juice	7	< 0.05, < 0.05,	< 0.05	
		1	1.44	0.087	Dry pulp	7	< 0.05	0.88	Aceta_048
					Oil	7	0.95, 0.74, 0.96	< 0.05	
							< 0.05		

Apples

The transfer of acetamiprid residues into apple juice and wet pomace was investigated in a study reported by Kowite, (1999) [Ref: Aceta_075]. In a field trial conducted in USA, apple trees were treated four times with 0.84 kg ai/ha (0.045 kg ai/hL) and fruit were harvested 7 days after the last application. The apples were washed for 5 minutes with cold water and crushed in a hammermill. The pulp was heated to 40–50 °C and 1.5 g of enzyme per kg of apple pulp as added and mixed for about

2 minutes. The enzyme treated apple pulp was transferred to plastic tubs and allowed to sit for about 2 hours before being pressed twice with a fruit press to obtain fresh juice and wet pomace.

Table 77 Residues in apples and processed apple fractions from a supervised trial in USA involving foliar applications of acetamiprid (WP formulations)

APPLE Location, Year (variety)	Form	Application data			Residues data			Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	
USA (WA) 1997 (Red Delicious)	WP 70	4	0.84	0.045	Fruit	7	0.82	RD-00591 12650-01 Aceta_075
					Juice	7	0.72	
					Wet pomace	7	1.1	

Plums

In several field trials conducted in USA, ripe plums (min 22 kg) from plots treated with 4 applications of 0.166 kg ai/ha (1×) and 0.667 kg ai/ha (4×) acetamiprid were sampled and sub-samples were dried in a glasshouse for 7 days before being stored frozen for up to 65 days until analysis using method EC-521 (but with residues measured by HPLC. Recovery rates in spiked samples ranged from 84–120% and the LOQ was 0.01 mg/kg.

Table 78 Residues in plums and prunes from supervised trials in USA involving foliar applications of acetamiprid (WP formulations)

PLUMS Location, Year (variety)	Application data			Residues data				Report Trial No Reference
	No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
						sample residues	mean	
USA Tulare (CA) 2003 (French Prune)	4	0.17	0.007	Fruit, no stones	7	0.05, 0.06	0.06	RD-00451 2003-19-CA-10
				Dried prune	7	0.17, 0.15	0.16	
		0.67	0.029	Fruit, no stones	7	not analysed	-	Aceta_049
				Dried prune	7	0.64, 0.74	0.69	
USA Tulare (CA) 2003 (French Prune)	4	0.17	0.024	Fruit, no stones	7	0.04, 0.04	0.04	RD-00451 2003-19-CA-11
				Dried prune	7	0.12, 0.13	0.13	
		0.67	0.097	Fruit, no stones	7	not analysed	-	Aceta_049
				Dried prune	7	0.55, 0.45	0.5	

Grapes

In a processing study reported by Carringer, (1999) [Ref: Aceta_088], grape vines in a supervised field trial in USA were treated twice at 0.28 kg ai/ha (0.040 kg ai/hL) and grapes were harvested 7 days after the last application. For juice the grapes were crushed and enzyme depectinized at 57–59 °C. The extracted, unclarified juice was heated to 87 °C to inactivate the enzyme. After about five weeks the juice was filtered and then heated to 94–97 °C before being poured into cans and cooled. For raisins the grapes were allowed to sun dry and then mechanical dry at 55–70 °C. The dried grapes were collected in plastic bags and placed in a 21 °C sweat box for 25–40 minutes to adjust the moisture content to about 18.5%.

GRAPE Location, Year (variety)	Form	Application data			Residues data			Report
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]	Trial No Reference
USA (CA) 1997 (not specified)	WP 70	2	0.28 0.28	0.04 0.04	Whole grape Grape juice Raisins	7 7 7	0.15 0.22 0.14	RD-00745 12651-01 Aceta_088

In a study reported by Cappy, (1999) [Ref: Aceta_053], tomatoes from a supervised field trial conducted in USA 1997. Tomato plants were treated with four applications of about 0.84 kg ai/ha and samples collected 7 days after the last application for processing into puree and paste. Tomatoes were triple washed and then crushed. The crush was heated to 95–97 °C in a hot break system and screened to remove the peel and seeds. The resulting juice was concentrated to puree using a vacuum evaporator to achieve natural tomato soluble solids (NTSS) content of 11–13% and canned after heating to 91 °C for 6–8 minutes in a steam jacketed kettle. A portion of the puree was condensed to paste in a vacuum evaporator to an NTSS level of 29–31% and heated to 89 °C for 6–7 minutes in a steam jacketed kettle before canning. Triplicate samples of tomatoes, puree and paste were analysed for acetamiprid residues using Method EC-521.

TOMATO Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (CA) 1997 (Ace)	WP 70	1+	0.84	0.76	Fruit	7	0.4, 0.44, 0.52	0.45	RD-00744 12108-02, Aceta_053
		1+	0.83	0.72	Puree	7	0.66, 0.62, 0.62	0.63	
		2	0.84	0.72	Paste	7	1.3, 1.4, 1.3	1.4	

In a supervised field trial conducted by Macy, (1999) [Ref: Aceta_081], cotton plants were treated four times with an exaggerated rate of 0.56 kg ai/ha. Samples of the seeds were picked 30 days after the last application, saw-ginned to remove most of the lint, mechanically delinted and hulled, producing kernels and hulls. The kernel material was dried in an oven at 54–71 °C to reduce the moisture content to less than 12% and then heated at 82–113 °C, flaked in a roller mill and extruded to produce collets for crude oil extraction. The dried collets were then extracted three times with hexane at 50–60 °C, refined and the residual hexane was removed in a vacuum evaporator. Duplicate samples of cotton seed, meal, hulls and oil were analysed for acetamiprid using Method EC-521.

COTTON SEED Location, Year (variety)	Form	Application data			Residues data				Report Trial No Reference
		No.	kg ai/ha	kg ai/hL	Sample	PHI [days]	Residues [mg/kg]		
							sample results	mean	
USA (TX) 1997 (All-Tex Atlas)	WP 70	4	0.56	0.6	Cotton seed	30	0.22, 0.26	0.24	RD-00792
					Meal		0.072, 0.11	0.09	12105-01
					Hulls		0.15, 0.23	0.19	
					Refined oil		< 0.01, < 0.01	< 0.01	Aceta_081

Table 28 Summary of selected processing factors for acetamiprid

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors	Median or best estimate
Oranges	Juice ^a	< 0.04, <u>< 0.13</u> , < 0.16	< 0.13
	Pulp	<u>0.1</u> , <u>0.3</u>	0.2
	Pulp, dry	2.8	2.8
	Peel	<u>2.8</u> , <u>2.9</u>	2.85
	Oil ^a	< 0.16	< 0.16
Apple	Juice	0.88	0.88
	Wet pomace	1.4	1.4
Plums	Dried prunes	2.8, 3.0	2.9
Grape	Juice	1.5	1.5
	Raisins	0.93	0.93
Tomatoes	Puree	1.4	1.4
	Paste	3	3
Cotton	Meal	0.38	0.38
	Hulls	0.8	0.79
	Refined Oil ^a	< 0.04	< 0.04

^a Residues below LOQ

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Lactating dairy cows

A feeding study with acetamiprid on lactating dairy cows was reported by Williams, (1999) [Ref: Aceta_089]. In this study, lactating Holstein cows (562-688 kg bw) were orally dosed once daily (a.m.) for 28 consecutive days with gelatine-encapsulated acetamiprid at dose rates of 5.77 ppm (1×), 17.4 ppm (3×) and 58.6 ppm (10×), based on a mean feed intake of 21-22 kg dry matter/day. Each dose group involved 3 cows. Milk was collected over whole study period, averaging 24-28 kg/day. Samples of muscle, fat, kidney and liver were obtained at necropsy on study day 28 within 24 hours after the last dose. There were no obvious effects on health, feed consumption or milk production associated with the consumption of acetamiprid at any dose level.

Residues of acetamiprid and its metabolite IM-2-1 were extracted from milk and tissues by blending with acetonitrile (ACN). Solids were filtered from the extract and the filtrate was vacuum-evaporated to 10-20 mL. Following dilution with brine, the samples were successively extracted with hexane, then methylene chloride, dehydrated and vacuum-evaporated to dryness. Subsequent sample clean-up was carried out by chromatography on Florisil and C-18 SPE column. Quantification of acetamiprid and IM-2-1 in the final extracts were performed by HPLC with HPLC/UV at 254 nm (Method AR 149-97). The LOQs of the analytical methods were 0.01 mg/kg residue in milk, muscle and fat; 0.05 mg/kg in kidney and liver. Recovery rates for acetamiprid and IM-2-1 in all animal matrices were between 91% and 101% in samples spiked with 0.01-0.1 mg/kg (0.05-0.5 mg/kg in liver and kidney).

Average residues (3 animals) of acetamiprid in whole milk from treated animals ranged from < 0.01 to 0.21 mg/kg however IM-2-1 was the predominant residue in milk, ranging from 0.03 to 0.95 mg/kg. Residue concentrations of acetamiprid in whole milk increased rapidly, reaching a plateau within 1 day and were dose-related. Residue concentrations of IM-2-1 in whole milk were both dose- and time-dependent, reaching a plateau at about 7–8 days.

Table 83 Residues of acetamiprid and its IM-2-1 metabolite in milk during a 28-day dosing period

Day	Group (Dose level)								
	1× (5.77 ppm)			3× (17.4 ppm)			10× (58.6 ppm)		
	Acetamiprid (mg/kg)	IM-2-1 (mg/kg)	Total (mg/kg)	Acetamiprid (mg/kg)	IM-2-1 (mg/kg)	Total (mg/kg)	Acetamiprid (mg/kg)	IM-2-1 (mg/kg)	Total (mg/kg)
-1	nd	nd	nd	nd	nd	nd	nd	nd	nd
1	0.012	0.031	0.042	0.055	0.13	0.068	0.207	0.54	0.747
4	0.014	0.053	0.067	0.047	0.187	0.234	0.187	0.797	0.984
8	0.013	0.059	0.072	0.048	0.203	0.251	0.193	0.917	1.11
11	0.016	0.051	0.067	0.048	0.173	0.221	0.213	0.873	1.09
15	0.014	0.042	0.056	0.042	0.157	0.199	0.177	0.74	0.917
18	0.014	0.046	0.06	0.044	0.16	0.204	0.177	0.8	0.977
22	0.016	0.05	0.066	0.059	0.17	0.229	0.183	0.747	0.93
25	0.015	0.05	0.065	0.045	0.173	0.218	0.167	0.88	1.05
27	0.015	0.053	0.068	0.053	0.207	0.26	0.193	0.95	1.14
Mean	0.014	0.048	0.063	0.049	0.173	0.209	0.189	0.805	0.993
Max (day) ^a	0.018 (day 11)	0.066 (day 8)		0.079 (day 22)	0.3 (day 27)		0.26 (day 1)	1.1 (day 27)	

Values are mean residues from 3 cows

nd not detected

^a Maximum residues measured in any single sample (at the specified sample day)

Residues of acetamiprid were mostly found in liver and kidney, at low levels (up to 0.25 mg/kg) and only in the higher dose groups. Residues in muscle and fat did not exceed about 0.1 mg/kg and 0.06 mg/kg respectively. The predominant residue in all tissues was the IM-2-1 metabolite, found in all tissues at all dose rates. Highest residues were found in liver and kidney (up to about 2.4 mg/kg) with levels up to 1.0 mg/kg present in muscle and up to 0.65 mg/kg in fat.

Table 84 Residues of acetamiprid and its IM-2-1 metabolite in animal tissues following a 28-day dosing period

Dose level (ppm)	Mean and maximum residues at day 28 (mg/kg)							
	Liver		Kidney		Muscle		Fat	
	mean	max	mean	max	mean	max	mean	max
Acetamiprid								
5.77	< 0.05	< 0.05	< 0.05	< 0.05	< 0.01	< 0.01	< 0.01	< 0.01
17.4	0.053	0.06	< 0.05	< 0.05	0.019	0.029	0.011	0.013
58.6	0.16	0.25	0.094	0.14	0.074	0.11	0.033	0.064
IM-2-1								
5.77	0.1	0.1	0.19	0.2	0.038	0.04	0.027	0.062
17.4	0.39	0.58	0.65	0.81	0.16	0.26	0.064	0.14
58.6	2.1	2.4	2.3	2.4	0.9	1.0	0.33	0.65
IM-2-1 + Acetamiprid								
5.77	< 0.15	< 0.15	0.24	< 0.25	0.048	< 0.05	0.037	< 0.072
17.4	0.443	0.64	0.7	< 0.86	0.179	0.289	0.075	0.153
58.6	2.26	2.65	2.39	2.54	0.974	1.11	0.363	0.714

Values are mean residues from 3 cows and maximum residues in any individual cow in each dose group

nd Not detected

Laying hens

A livestock feeding study with laying hens was conducted by Williams, (1999), [Ref: Aceta_090]. The animals were separated into 4 groups of 10 hens, receiving 28 consecutive daily doses of 0, 1.16 ppm (1×), 3.51 ppm (3×) or 12 ppm (10×) of encapsulated acetamiprid (based on mean feed intakes of 83-89 g dry matter/day). Eggs were collected twice a day over the study period and samples of muscle, fat and liver were obtained at necropsy within 24 hours after the last dose. There were no obvious effects on health, feed consumption or egg production associated with the consumption of acetamiprid at any dose level.

Residues of acetamiprid and IM-2-1 were extracted from eggs and tissues by blending with acetonitrile (ACN). Solids were filtered from the extract and the filtrate was vacuum-evaporated to 10-20 mL. Following dilution with brine, the samples were successively extracted with hexane, then methylene chloride, dehydrated and vacuum-evaporated to dryness. Subsequent sample clean-up was carried out by chromatography on Florisil and C-18 SPE column. Quantification of acetamiprid and IM-2-1 in the final extracts were performed by HPLC/UV at 254 nm. The LOQs of the method (AR 149-97) were 0.01 mg/kg for eggs, muscle and fat and 0.05 mg/kg for liver. Recovery rates for acetamiprid and IM-2-1 in all animal matrices were between 90% and 99% in samples spiked with 0.01-1.0 mg/kg.

Residues of acetamiprid in eggs were not detectable or < 0.01 mg/kg in all dose groups, with the IM-2-1 metabolite being found at up to 0.33 mg/kg in the 10× dose group and were both dose- and time-related, reaching a plateau at about day 8.

Table 85 Residues of acetamiprid and its IM-2-1 metabolite in eggs during a 28-day dosing period

Day	Group (Dose level)								
	1× (1.16 ppm)			3× (3.51 ppm)			10× (12 ppm)		
	Acetamiprid (mg/kg)	IM-2-1 (mg/kg)	Total	Acetamiprid (mg/kg)	IM-2-1 (mg/kg)	Total	Acetamiprid (mg/kg)	IM-2-1 (mg/kg)	Total
-1	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01	< 0.02
1	< 0.01	0.012	< 0.022	< 0.01	0.042	< 0.052	< 0.01	0.12	< 0.13
4	< 0.01	0.025	< 0.035	< 0.01	0.064	< 0.074	< 0.01	0.263	< 0.273
8	nd	0.027	0.027	< 0.01	0.087	< 0.097	< 0.01	0.303	< 0.313
11	nd	0.028	0.028	< 0.01	0.085	< 0.095	< 0.01	0.283	< 0.293
15	nd	0.026	0.026	< 0.01	0.085	< 0.095	< 0.01	0.293	< 0.303
18	< 0.01	0.026	< 0.036	< 0.01	0.08	< 0.09	< 0.01	0.293	< 0.303
22	< 0.01	0.025	< 0.035	< 0.01	0.085	< 0.095	< 0.01	0.313	< 0.323
25	nd	0.026	0.026	< 0.01	0.081	< 0.091	< 0.01	0.293	< 0.303
27	< 0.01	0.028	< 0.038	< 0.01	0.093	< 0.103	< 0.01	0.293	< 0.303
Mean	< 0.01	0.025	0.03	< 0.01	0.078	< 0.088	< 0.01	0.273	< 0.283
Max (day) ^a	< 0.01	0.032 (day 11)		< 0.01	0.1 (day 27)		< 0.01	0.33 (day 8)	

Values are mean residues in eggs from 3 sub-groups of 3-4 hens

nd not detected

^a Maximum residues measured in any single sub-group (at the specified sample day)

In hen tissues, acetamiprid was not detected in any dose group, with IM-2-1 being found at levels of up to 0.5 mg/kg in liver, up to 0.075 mg/kg in muscle and up to 0.012 mg/kg in fat.

Table 86 Residues of acetamiprid and its IM-2-1 metabolite in hen tissues following a 28-day dosing period

Dose level (ppm)	Mean and maximum residues at day 28 (mg/kg)					
	Liver		Muscle		Fat	
	mean	max	mean	max	mean	max
Acetamiprid						
0.0	nd	nd	nd	nd	nd	nd
1.16	nd	nd	nd	nd	nd	nd
3.51	nd	nd	nd	nd	nd	nd
12.0	nd	nd	nd	nd	nd	nd
IM-2-1						
0.0	nd	nd	nd	nd	nd	nd
5.77	0.067	0.092	< 0.01	< 0.01	-- ⁽¹⁾	-- ⁽¹⁾
17.4	0.18	0.21	0.023	0.027	< 0.01	< 0.01
58.6	0.47	0.5	0.069	0.075	0.011	0.012
Acetamiprid + IM-2-1						
0.0	nd	nd	nd	nd	nd	nd
1.16	0.067	0.092	< 0.01	< 0.01	-- ⁽¹⁾	-- ⁽¹⁾
3.51	0.18	0.21	0.023	0.027	< 0.01	< 0.01
12.0	0.47	0.5	0.069	0.075	0.011	0.012

Values are mean residues from 3 cows

Fat from this (1×) dose group not analysed since acetamiprid and IM-2-1 residues in higher (3×) dose group were < 0.01 mg/kg

nd Not detected

NATIONAL RESIDUE DEFINITIONS

The meeting was provided with information on the acetamiprid residue definitions adopted in a number of countries. These are summarised in the following table.

Table 87 National residue definitions for acetamiprid

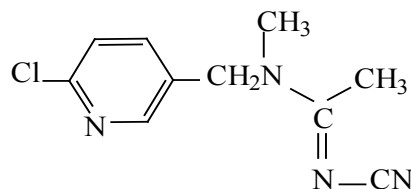
Country	Commodities	Residue definition (dietary intake)	Residue definition (MRL compliance)
Australia (APVMA)	plant commodities	Acetamiprid	Acetamiprid
	animal commodities	Acetamiprid, IM-2-1	Acetamiprid, IM-2-1
Brazil	plant and animal commodities		
European Union	plant commodities	Acetamiprid	Acetamiprid
	animal commodities	Acetamiprid, IM-2-1	Acetamiprid, IM-2-1
Japan	plant commodities	Acetamiprid	Acetamiprid
	animal commodities	Acetamiprid, IM-2-1	Acetamiprid, IM-2-1
USA	plant commodities	Acetamiprid	Acetamiprid
	animal commodities	Acetamiprid, IM-2-1	Acetamiprid, IM-2-1
more...?			

APPRAISAL

Acetamiprid is a neonicotinoid insecticide with contact and stomach action against a range of Hemiptera, Thysanoptera and Lepidoptera plant pests, acting as an agonist of the nicotinic acetylcholine receptor in the insect central nervous system. It exhibits translaminar activity in plants and is authorised for use in North America, Europe and in a number of countries in Asia and the Pacific.

Residue and analytical aspects of acetamiprid were considered for the first time by the present meeting. The manufacturer submitted studies on metabolism, analytical methods, authorised uses, supervised field trials, the effects of processing, freezer storage stability, environmental fate in soil and rotational crop residues.

Acetamiprid, ((E)-N1-[(6-chloro-3-pyridyl)methyl]-N2-cyano-N1-methylacetamidine) is partially soluble in water (3-4 g/litre), stable to hydrolysis and photolysis, has a log POW of 0.8 and is soluble in acetone, methanol, ethanol, dichloromethane, and acetonitrile.



The following abbreviations are used for the metabolites discussed below:

IM-1-2	<i>N</i> ² -carbamoyl- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ¹ -methylacetamidine
IM-1-3	<i>N</i> -[(6-chloro-3-pyridyl)methyl]- <i>N</i> -methylacetamide
IM-1-4	<i>N</i> -methyl(6-chloro-3-pyridyl)methylamine
IM-2-1	<i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]- <i>N</i> ² -cyanoacetamidine
IM-2-2	<i>N</i> ² -carbamoyl- <i>N</i> ¹ -[(6-chloro-3-pyridyl)methyl]-acetamidine
IM-2-3	<i>N</i> -[(6-chloro-3-pyridyl)methyl]acetamide
IM-2-4	(6-chloro-3-pyridyl)methylamine
IM-2-5	<i>N</i> 1-(6-Chloropyridin-3-ylmethyl)-acetamidine
IM-0	(6-chloro-3-pyridyl)methanol
IM-0-Glc	(6-chloro-3-pyridyl)methyl-β-D-glucopyranoside
IC-0	6-chloronicotinic acid

Animal metabolism

The Meeting received acetamiprid metabolism studies on animals (rats, lactating goats and laying hens) using ¹⁴C-acetamiprid (labelled in the 2 and 6 positions of the pyrimidine ring).

In rats, acetamiprid is rapidly and almost completely absorbed and is widely distributed into the tissues, being found at highest concentrations in GI tract, adrenal gland, liver and kidney, following oral administration to the rat. The major route of elimination was via the urine and bile (relevant but not a major route in excreta). The disappearance of radioactivity from the body of the rat was rapid and there was no indication of accumulation in any tissue. Less than 1% of the administered radioactivity was left in the tissues by day four following dosing. The major radioactive compounds in the excreta of rats were acetamiprid (approx. 5-7%); the demethylated compound IM-2-1 (approximately 15-20%), the nicotinic acid derivative IC-O (approximately 8-11%) and the IC-O glycine conjugate IC-O-Gly (approximately 10%). In addition, MeS-IC-O, IM-1-4, IM-2-4, IM-O, IM-1-3 and IM-2-3 were detected, but they were less than 2% of dose. There were several unknown compounds in urine with a maximum abundance of 1%.

The main metabolic pathway of acetamiprid in rats is the transformation to IM-2-1 by demethylation which is further metabolized to IC-O with the release of IS-1-1 and IS-2-1 after the cleavage from the side chains of IN-25 and IM-2-1.

Lactating goats were orally dosed twice daily for 7 days with encapsulated [pyridine-2, 6-¹⁴C]-acetamiprid at dietary equivalent levels of 1.0 ppm or 8.6 ppm per day. At the end of the 7-day dosing period, the goats were sacrificed 22 hours after the last administration.

Most of the administered radioactivity (AR) was excreted via urine or faeces (about 95-99% AR) and less than 1% AR in milk (reaching a plateau after about 3 days). In tissues, radioactivity did not exceed 1.6% AR and in milk, about 94-96% TRR was found in the whey with about 3-5% TRR occurring in milk fat and precipitated milk proteins.

The predominant residue in milk, liver and kidney was the IM-2-1 metabolite (70–89% TRR) and in muscle, the major residue was IM-2-2 (about 50% TRR), with the IM-2-3 and IM-2-4 metabolites also being found at 6% and 13% TRR respectively. Acetamiprid (parent) was only found in milk, at less than 10% TRR and < 0.005 mg/kg.

Laying hens (five hens per dose group) were dosed each morning for 14 days with [pyridine-2, 6-¹⁴C]-acetamiprid at dietary equivalent levels of 1.1 ppm or 12.5 ppm. At the end of the 14-day dosing period, the hens were sacrificed about 24 hours after the last administration.

Most of the applied radioactivity was excreted or found in the cage wash (93-97% AR). Small amounts of radioactivity were detected in edible organs/tissues (0.7-0.8% AR) with about 1.3% AR found in eggs (reaching a plateau after about 8-11 days). In liver and skin, residues were about 0.1% AR and 0.3% AR in muscle.

The IM-2-1 metabolite was the predominant residue, at 83-86% TRR in egg white, about 60% TRR in egg yolk, 65-69% TRR in liver and 53-62% TRR in muscle and skin. The other metabolite found at more than 10% TRR was the IM-2-3 in muscle (17-21% TRR). Metabolite IM-2-5 was the predominant residue in egg yolks (27% TRR) and IC-0 was found in skin at about 13% TRR. Acetamiprid (parent) was not found in any tissues or in eggs.

In summary, acetamiprid metabolism in animals is similar, with more than 95% of the residues being eliminated in excreta and less than 2% remaining in tissues or present in eggs or milk. Residues of the parent acetamiprid were not found (except at low levels in milk), and the predominant residue in most animal products was the IM-2-1 (53-89% TRR) with IM-2-2 occurring in goat muscle at about 50% TRR). The IM-2-4 and IM-2-3 metabolites were also found in muscle at 13-21% TRR, with the IM-2-5 metabolite being found at about 27% TRR in egg yolks.

The proposed metabolic breakdown of acetamiprid in both goats and hens involves degradation to IC-0 or demethylation to IM-2-1 with the IM-1-2 metabolite converting to the amide (IM-2-2) or IM-2-3 and the subsequent formation of the IM-2-4 and IM-2-5 metabolites.

Plant metabolism

The meeting received plant metabolism studies in apples, eggplant, cabbage, cotton and carrot following foliar applications of [pyridine-2, 6-¹⁴C]-acetamiprid and an additional study with cabbages treated with [CN-¹⁴C]-acetamiprid (both as a foliar application and a soil treatment).

In apple fruit, more than 98% of the radioactivity was recovered from the surface wash and extracts of fruit. Residues in surface washes decreased from more than 99% to about 12% TRR after 14 days and to about 6% TRR for fruit sampled 28 and 62 days after treatment. Residues in flesh increased to 48% TRR after 14 days and to about 78% TRR at the end of the 62-day study period. Acetamiprid (parent) was the predominant residue, making up more than 79% TRR. Minor metabolites (IM-2-1 and IM-0-Glc) were found at maximum 3.7%TRR and 1.8%TRR, respectively.

For apple leaves, more than 98% of the radioactivity was recovered from the surface wash and extracts from leaves. Initial residues in surface washes decreased from 99% to about 43% TRR at the end of the 90-day study period and the residues in the leaf extracts increased to about 51% TRR (11.8 mg/kg eq). Translocated radioactivity in untreated leaves was less 0.04 mg/kg.

The majority of the radioactivity was the unchanged acetamiprid, making up 90% or more of the TRR in the first 14 days after treatment, declining to 49% TRR after 90 days. The main metabolite found above 5% TRR was the IM-2-1 metabolite, present at about 10% TRR after 62 days and about 16% TRR after 90 days. The only other metabolite present at more than 5% TRR was IM-0-Glc (max 8.3% TRR at day-90).

For eggplant, most of the radioactivity was found in the surface washes (79-75% TRR for leaves and 84-70% TRR for fruit), with 20-30% TRR present in the extracts from washed fruit and leaves. Translocated radioactivity was negligible. Acetamiprid was the major residue, making up about 85-89% TRR in leaves and 94-95% TRR in fruit. Of the three identified metabolites, IM-2-1

was present in fruit at 0.4% TRR and 1.8% TRR in leaves and the IM-0 metabolite and its glycoside were identified in leaves at 0.6% TRR and 4.6% TRR respectively.

For cabbages following foliar treatments with [pyridine-2, 6-¹⁴C]-acetamiprid, surface residues decreased to 30-50% TRR in the 28 days after treatment and to about 12% at the end of the study period (day-63). Residues in the extracts from washed plants increased accordingly, from 15% TRR (day-0) to 83.5% TRR (day-63). Acetamiprid was the major residue component in leaves, found at about 67-91% TRR with residues of the IM-2-1 metabolite increasing over the study period to a maximum of about 7% TRR (day-63). No parent residues were measured in mature cabbage heads with the wrapper leaves removed, with the major residue being the IC-0 metabolite (about 46% TRR or 0.03 mg/kg).

In cabbages grown in soil treated with [pyridine-2, 6-¹⁴C]-acetamiprid, radioactivity was readily translocated into leaves, reaching levels of about 2-3 × the root concentrations during the 28-day study period. Acetamiprid was also the only major residue in both leaves and roots, initially found at about 90% TRR (leaves) and 78% TRR (roots), decreasing to 60% TRR (leaves) and 50% TRR (roots) after 28 days. The IM-1-4 metabolite was the only other identified metabolite present at more than 5% TRR, being found in roots after 28 days.

An additional study on cabbages treated with a foliar application of [CN-¹⁴C-acetamiprid] reported similar results. Surface residues decreased from an initial 86% TRR down to about 16% TRR after 63 days, with residues in the extracts from washed leaves increasing to about 78% TRR at the end of the study period. The major residue was the unchanged acetamiprid, making up more than 98% TRR (to day-7) and about 65% TRR by day-68.

For carrots treated twice with [pyridine-2, 6-¹⁴C]-acetamiprid as foliar sprays, total radioactivity in carrots (including tops) at harvest was less than 0.1 mg/kg acetamiprid equivalents, mostly in the tops (0.44 mg/kg), with about 0.08 mg/kg in the roots. The main components found at harvest (2 weeks after the second treatment), in the carrot tops were IM-0-Glc (33% TRR), the parent acetamiprid (27% TRR) and IM-1-4 (15% TRR) with no other components exceeding 6% TRR. In the carrot roots the main components were acetamiprid (30-34% TRR and 0.03 mg/kg) and IC-0 (17-31% TRR and 0.02 mg/kg).

In cotton seed and gin trash from plants treated with four foliar applications of [pyridine-2, 6-¹⁴C]-acetamiprid at 7 day intervals, the parent compound was the major residue identified in gin trash, found at 50% TRR (1.4 mg/kg) in the 14-day PHI samples and at 45% TRR (0.71 mg/kg) in the 28-day PHI samples.. The IC-0 metabolite was the predominant residue in cotton seed, found in the 14-day PHI samples at 46% TRR (0.69 mg/kg), decreasing to 24% TRR (0.27 mg/kg) in the 28-day samples.

In summary, the predominant residue in plant part exposed to foliar treatments is the parent compound, with low levels of the IM-2-1 metabolite being a common component in the plants studied, but generally at levels of 10% TRR or less. Acetamiprid is also the predominant residue in cabbage and carrot roots following soil treatments. The other significant metabolite found in plant parts not directly treated was the IC-0 cleavage product, found in cabbage heads (0.03 mg/kg), carrot roots (0.04 mg/kg) and cotton seed (up to 0.69 mg/kg).

The proposed metabolic breakdown of acetamiprid in plants following foliar application involves demethylation to IM-2-1 and further degradation to IC-0-Glc, or conversion of the parent compound to IM-0, with subsequent conjugation with glucose to form the IM-0-Glc. Degradation can also involve formation of the IM-1-2 metabolite which rapidly degrades to IM-1-3 and either IM-2-3 or IM-1-4, both of which degrade to IC-0.

Environmental fate

The Meeting received information the environmental metabolism and behaviour of acetamiprid in soil and rotational crops.

The estimated aerobic soil metabolism half-life for acetamiprid at 25 °C was about 8.2 days with a significant amount of $^{14}\text{CO}_2$ (up to about 19% of the applied dose) being measured during the study. The metabolite IM-1-4 was a major component of the radioactive residue, increasing to about 73% after 120 days and slowly decreasing thereafter. Two minor metabolites, IM-1-3 and IC-0 were also identified but at less than 5% of the applied dose during the study.

In three soils treated with the equivalent of 0.1 kg ai/ha [^{14}C -2, 6-pyridine]-acetamiprid and incubated in the dark for intervals up to 6 months, acetamiprid residues degraded rapidly, with estimated aerobic soil metabolism half-lives of about 1-8 days. The IM-1-4 metabolite was the major residue in soil, present at about 54-72% AR after 6 months.

Residues in succeeding crops

In rotational crop metabolism studies involving radish, lettuce, sorghum and wheat grown in a sandy loam soil treated (bare ground) with [pyridine-2, 6- ^{14}C]-acetamiprid and aged for various intervals (up to 1 year), radioactive residues in samples from all plant-back intervals were less than 0.1 mg/kg parent equivalents except sorghum fodder from the 60-day plant-back interval (0.115 mg/kg). Acetamiprid was not found in any of the matrices and all metabolites were present at less than 0.05 mg/kg in any matrix at any rotation, the highest being IM-1-4 at 0.04 mg/kg in the first plant-back sorghum forage.

The Meeting agreed that residues of acetamiprid would not be expected in rotational crops.

Analytical methods

Several analytical methods have been reported for the analysis of acetamiprid and its IM-2-1 (desmethyl) metabolite in animal and plant matrices. The principle of most methods involves extraction steps using methanol or acetonitrile, liquid/liquid partition (commonly hexane) and further extraction into methylene chloride, column chromatographic clean-up (silica gel, Florisil and C18) and analysis by HPLC (animal and plant matrices) or by GC/ECD or LC-MS/MS (plant matrices).

The methods have been validated for plant and animal matrices with LOQs of 0.05 mg/kg for citrus commodities, liver and kidney and 0.01 mg/kg for other plant and animal commodities.

Based on the results of validation studies and the concurrent recovery rates achieved in the supervised field trials, the available analytical methods are considered suitable for determining residues of acetamiprid and its IM-2-1 metabolite.

Based on an investigation with orange as a test matrix, the US-FDA PAM 1 multi-residue method was shown to be unsuitable for measuring acetamiprid residues in plant commodities.

The multi-residue QuEChERS method using GCMS and/or liquid chromatography coupled with tandem mass spectrum detection (LC-MS/MS) was validated at the LOQ of 0.01 mg/kg for determining acetamiprid residues in dry, high water, acid, oily and high sugar content matrices and in animal matrices.

Stability of pesticide residues in stored analytical samples

Residue stability in stored analytical samples was investigated for a range of representative substrates covering those with a high water content (apple, cabbage, cucumber, grape, lettuce, tomato) a high starch content (potato), a high oil content (cotton seed) and a high acid content (orange) and their processed fractions, stored at ambient temperatures and at freezer temperatures.

In samples fortified with acetamiprid at levels of 0.5 mg/kg or 0.1 mg/kg and stored at either at room temperature for up to 7 days or frozen for up to 12 months (16 months for lettuce and 8 months for potatoes), residues were stable in all samples at the end of the storage periods, both at ambient temperature and under freezer conditions.

In the supervised field trials, frozen storage intervals between sampling and analysis were less than the storage periods in these stability studies, except for one citrus trial (15 months), two tomato

trials (13–14 months) and three celery trials (17 months). The Meeting considered that any residue degradation during these extended storage intervals would be negligible.

Definition of the residue

In livestock metabolism studies (goats, hens), residues of the parent acetamiprid were not found (except at low levels in milk), and the predominant residue in most animal products was the IM-2-1 (53–89% TRR). The IM-2-2 metabolite was the predominant residue (about 50% TRR) in goat muscle, with the IM-2-4 metabolite also present at about 13% TRR. The IM-2-5 metabolite was the predominant residue in eggs (9% TRR). Residues in fat were too low to be characterised.

For MRL compliance, the IM-2-1 metabolite is the predominant residue in milk, liver, kidney poultry muscle and skin and eggs, and is a major residue component in goat muscle. Based on the significance of this metabolite in all animal matrices and because the parent compound was only found in milk (and then only at low levels), the Meeting considered the use of the IM-2-1 metabolite as a marker residue for MRL compliance.

For dietary intake risk assessment, in addition to the IM-2-1 metabolite, other significant residue components above 10% TRR are IM-2-2 (the amide of IM-2-1, found only in goat muscle at up to 0.03 mg/kg), the IM-2-5 metabolite (the imide of IM-2-1, found only in egg yolk at up to 0.24 mg/kg) and the cleavage product IM-2-4, found only in goat muscle at up to 0.008 mg/kg.

Noting that these three metabolites were not found in any other edible animal products; that current analytical methods did not exist to measure these compounds and that at the low levels expected, they are not likely to contribute significantly to the dietary exposure, the Meeting agreed that these metabolites need not be included in the residue definition for dietary risk assessment.

The Meeting recommended that for animal commodities, the residue definition for both dietary intake assessment and MRL compliance should be the sum of acetamiprid and its IM-2-1 metabolite, expressed as acetamiprid.

Based on the results of the cattle feeding study, where residues in muscle and fat were of the same order, and considering the ratio of radioactive residues in milk whey and milk fat/proteins (95:4) in the lactating goat metabolism study, the Meeting agreed that acetamiprid is not fat soluble. The log K_{ow} of acetamiprid (log K_{ow} 0.8) supports this conclusion.

In plants, the metabolism of acetamiprid has been studied in vegetables (cabbage, eggplant, carrots), in fruit (apples) and cotton. In all crops studied, the parent compound is the major residue component following foliar applications, initially as a surface residue and subsequently being taken up into the treated leaf or fruit, with little further translocation. The only significant metabolite identified in the studies was the desmethyl metabolite (IM-2-1), found at less than 10% TRR in edible crop parts.

The other metabolite found in edible plant parts not directly treated (e.g., carrot roots, cabbage heads, cotton seed) was the IC-0 cleavage product, present in carrot roots at 26% TRR and being the predominant residue in cabbage heads and cotton seed (24–46% TRR). This metabolite (6-chloronicotinic acid) was found in significant quantities (24–28 % TRR) in rat metabolism and with an oral $LD_{50} > 5000$, of is of lower acute toxicity than the parent compound and does not exhibit any genotoxic potential. The Meeting noted that IC-0 is also a metabolite of other neonicotinoids and agreed it should be excluded from the residue definition.

The Meeting recommended that for MRL-compliance and dietary intake risk assessment, the residue definition for plant commodities should be acetamiprid.

Definition of the residue for plant commodities (for compliance with the MRL and estimation of dietary intake): *acetamiprid*

Definition of the residue for animal commodities (for compliance with the MRL and estimation of dietary intake): *acetamiprid and N-desmethyl-acetamiprid, expressed as acetamiprid*

The residue is not fat soluble

Results of supervised trials on crops

The Meeting received supervised trial data for foliar applications of acetamiprid (SP and WP formulations) on a range of fruit, nut, vegetable and cotton crops, conducted mainly in Europe and North America.

The OECD MRL calculator was used as a tool to assist in the estimation of maximum residue levels from the selected residue data set obtained from the supervised residue trials. As a first step, the Meeting reviewed all relevant factors related to each data set in arriving at a best estimate of the maximum residue level using expert judgement. Then the OECD calculator was employed. If the statistical calculation spreadsheet suggested a different value from that recommended by the Meeting, a brief explanation of the deviation was supplied.

Citrus fruits

Residue data were provided to the Meeting from trials in Italy and Spain on lemons, mandarins and oranges involving 2–3 applications of 0.01 kg ai/hL.

GAP for citrus fruits in Spain is for foliar applications of up to 0.01 kg ai/hL and a PHI of 14 days, with a maximum of two applications/season.

In citrus trials from Italy and Spain matching this Spanish GAP, acetamiprid residues in lemons were : 0.09, 0.15 and 0.45 mg/kg.

In mandarins, residues were: 0.14, 0.17, 0.19, 0.25, 0.25, 0.26 and 0.44 mg/kg. The Meeting noted that in 2 of the Spanish trials (in bold), the 1st of 3 applications was applied more than 100 days before harvest and agreed to include these results because the contribution from these initial sprays would be negligible.

In oranges, residues were: 0.09, 0.1, 0.12, 0.22, 0.28, 0.28, 0.39 and 0.4 mg/kg.

The Meeting noted that these data sets were similar and agreed to combine them to estimate a group maximum residue level, STMR and HR for citrus fruit.

The combined data set from trials on lemons, oranges and mandarins (whole fruit) matching the GAP in Spain for citrus fruits is: 0.09, 0.09, 0.1, 0.12, 0.14, 0.15, 0.17, 0.19, 0.22, 0.25, 0.25, 0.26, 0.28, 0.28, 0.39, 0.4, 0.44 and 0.45 mg/kg (n = 18)

The Meeting estimated an STMR of 0.25 mg/kg, an HR of 0.45 mg/kg and recommended a maximum residue level of 0.8 mg/kg for acetamiprid in citrus fruit.

Pome fruits

Residue data were provided to the Meeting from trials in the USA on apples and pears. GAP for pome fruit in USA is for a maximum of four foliar applications of up to 0.168 kg ai/ha and a PHI of 7 days.

In trials on apples from the USA matching this GAP, acetamiprid residues were: 0.12, 0.12, 0.14, 0.14, 0.16, 0.18, 0.19, 0.22, 0.23, 0.25, 0.25, 0.26, 0.27, 0.28, 0.31, 0.55 and 0.59 mg/kg.

In trials on pears from USA matching this GAP, acetamiprid residues were: 0.09, 0.09, 0.15, 0.17, 0.2, 0.25, 0.31, 0.32 and 0.32 mg/kg.

The Meeting noted that these data sets were from similar populations and agreed combine them to estimate a group maximum residue level, STMR and HR for pip fruit. The combined data set for apples and pears is: 0.09, 0.09, 0.12, 0.12, 0.14, 0.14, 0.15, 0.16, 0.17, 0.18, 0.19, 0.2, 0.22, 0.23, 0.25, 0.25, 0.25, 0.26, 0.27, 0.28, 0.31, 0.31, 0.32, 0.32, 0.55 and 0.59 mg/kg (n = 26).

The Meeting estimated an STMR of 0.225 mg/kg, an HR of 0.59 mg/kg and recommended a maximum residue level of 0.8 mg/kg for acetamiprid in pome fruits.

Stone fruits

Residue data were provided to the Meeting from trials in USA on cherries, peaches and plums. GAP in USA for stone fruits is for a maximum of four foliar applications of up to 0.168 kg ai/ha and a PHI of 7 days.

In trials on cherries from the USA matching this GAP, acetamiprid residues (in fruit without stones) were: 0.1, 0.29, 0.36, 0.42, 0.48, 0.54, 0.68 and 0.88 mg/kg (n = 8).

The Meeting estimated an STMR of 0.45 mg/kg, an HR of 0.88 mg/kg and recommended a maximum residue level of 1.5 mg/kg for acetamiprid in cherries.

In trials on peaches from the USA matching this GAP, acetamiprid residues (in fruit without stones) were: 0.11, 0.16, 0.18, 0.19, 0.2, 0.2, 0.22, 0.23, 0.34 and 0.44 mg/kg (n = 10).

The Meeting estimated an STMR of 0.2 mg/kg, an HR of 0.44 mg/kg and recommended a maximum residue level of 0.7 mg/kg for acetamiprid for peaches and agreed to extrapolate these recommendations to nectarines.

In trials on plums from the USA matching this GAP, acetamiprid residues (in fruit without stones) were: 0.01, 0.02, 0.04, 0.04, 0.06 and 0.11 mg/kg (n = 6).

The Meeting estimated an STMR of 0.04 mg/kg, an HR of 0.11 mg/kg and recommended a maximum residue level of 0.2 mg/kg for acetamiprid in plums (including prunes).

Berries and other small fruits

Residue data were provided to the Meeting from trials in USA and Canada on grapes, strawberries, blackberries, boysenberries and raspberries.

GAP in USA for grapes and small vine fruits is for a maximum of two foliar applications of up to 0.112 kg ai/ha and a PHI of 3 days.

In trials on grapes from USA matching this GAP, acetamiprid residues in grape bunches were: 0.01, 0.03, 0.04, 0.04, 0.05, 0.06, 0.07, 0.08, 0.08, 0.09, 0.11, 0.13, 0.15, 0.16, 0.20, 0.22, 0.23 and 0.25 mg/kg (n = 18).

The Meeting estimated an STMR of 0.085 mg/kg, an HR of 0.25 mg/kg and recommended a maximum residue level of 0.5 mg/kg for acetamiprid in grapes.

GAP in USA for bush and caneberries (including strawberries and low-bush blueberries) is for a maximum of two foliar applications of up to 0.146 kg ai/ha and a PHI of 1 day.

In trials on strawberries from Canada and the USA matching this GAP, acetamiprid residues in fruit (without sepals) were: 0.03, 0.04, 0.05, 0.06, 0.09, 0.11, 0.12, 0.24, 0.24 and 0.24 mg/kg (n = 10).

The Meeting estimated an STMR of 0.1 mg/kg, an HR of 0.24 mg/kg and recommended a maximum residue level of 0.5 mg/kg for acetamiprid in strawberries.

GAP in USA for bush berries (including low-bush and high-bush blueberries) and cane berries (including blackberries, raspberries and cultivars/hybrids) is for a maximum of five foliar applications of up to 0.112 kg ai/ha and a PHI of 1 day.

In trials on blueberries from USA matching this GAP for bush berries, residues in fruit were: 0.09, 0.2, 0.25, 0.48, 0.49 and 0.62 mg/kg (n = 6).

In trials on blackberries, raspberries and boysenberries from USA matching this GAP for cane berries, residues in fruit were: 0.53, 0.56, 0.64, 0.78 and 1.0 mg/kg (n = 5).

The Meeting agreed to use the data for blackberries, raspberries and boysenberries to propose a group maximum residue level for berries and other small fruit (except grapes and strawberries)

The Meeting estimated an STMR of 0.64 mg/kg, an HR of 1.0 mg/kg and recommended a maximum residue level of 2 mg/kg for acetamiprid in berries and other small fruit except grapes and strawberries

Bulb vegetables

Residue data were provided to the Meeting from trials in USA and Canada on bulb onions and spring onions.

GAP in USA for bulb vegetables (including onions and spring onions) is for a maximum of four foliar applications of up to 0.168 kg ai/ha and a PHI of 7 days.

In trials on onions from USA matching this GAP, acetamiprid residues in onion bulbs were: < 0.01, < 0.01, < 0.01, < 0.01, < 0.01 and 0.01 mg/kg (n = 6).

The Meeting estimated an STMR of 0.01 mg/kg, an HR of 0.01 mg/kg and recommended a maximum residue level of 0.02 mg/kg for acetamiprid for bulb onions and agreed to extrapolate these recommendations to garlic.

The OECD Calculator proposed a maximum residue level of 0.015 mg/kg but as one of the replicate analytical samples contained 0.018 mg/kg, the Meeting recommended a higher level of 0.02 mg/kg.

In trials on spring onions from USA matching this GAP, residues were: 0.05, 0.38 and 2 mg/kg.

The Meeting estimated an STMR of 0.38 mg/kg, an HR of 2 mg/kg and recommended a maximum residue level of 5 mg/kg for acetamiprid in spring onions.

Brassica vegetables

Residue data were provided to the Meeting from trials in USA on broccoli and head cabbage.

GAP in USA for cole crops (including broccoli and head cabbage) is for a maximum of five foliar applications of up to 0.084 kg ai/ha and a PHI of 7 days.

In trials on broccoli from USA matching this GAP, acetamiprid residues in broccoli were: 0.01, 0.01, 0.02, 0.02, 0.02, 0.03, 0.05, 0.09 and 0.22 mg/kg (n = 9).

The Meeting agreed to extrapolate these results to other flower-head brassicas and estimated an STMR of 0.02 mg/kg, an HR of 0.22 mg/kg and recommended a group maximum residue level of 0.4 mg/kg for acetamiprid in flower-head brassicas.

In trials on head cabbage from USA matching this GAP, acetamiprid residues in cabbage heads (with wrapper leaves) were: 0.03, 0.03, 0.06, 0.07, 0.07, 0.11, 0.11, 0.11, 0.13 and 0.5 mg/kg (n = 10).

The Meeting recommended a maximum residue level of 0.7 mg/kg for acetamiprid in head cabbages and for use in calculating the animal dietary burden, estimated a median residue of 0.09 mg/kg, and a highest residue of 0.5 mg/kg.

In the same trials on head cabbage from the USA matching the USA GAP, acetamiprid residues in cabbage heads without wrapper leaves were: < 0.01, < 0.01, < 0.01, 0.01, 0.02, 0.02, 0.02, 0.03, 0.03 and 0.05 mg/kg (n = 10)

The Meeting estimated an STMR of 0.02 mg/kg and an HR of 0.05 mg/kg for acetamiprid in head cabbages (for dietary intake risk assessment).

Fruiting vegetables, Cucurbits

Residue data were provided to the Meeting from trials in USA on cucumber, summer squash and melons.

GAP in USA for cucurbits is for a maximum of five foliar applications of up to 0.112 kg ai/ha and a PHI of 0 days.

In trials on cucumbers from USA matching this GAP, acetamiprid residues were: 0.02, 0.02, 0.03, 0.03, 0.04 and 0.09 mg/kg.

In trials on summer squash from USA matching this GAP, acetamiprid residues were: 0.05, 0.06, 0.06, 0.09 and 0.11 mg/kg.

In trials on melons from USA matching this GAP, acetamiprid residues were: 0.02, 0.02, 0.04, 0.06, 0.08 and 0.1 mg/kg.

The Meeting noted that these data sets for cucumbers and summer squash (representing cucurbits with edible peel) and melons (representing cucurbits with inedible peel) were similar and agreed to combine them to recommend a group maximum residue level, STMR and HR for cucurbits.

The combined data set for cucumbers, summer squash and melons is: 0.02, 0.02, 0.02, 0.02, 0.03, 0.03, 0.04, 0.04, 0.05, 0.06, 0.06, 0.06, 0.08, 0.09, 0.09, 0.1 and 0.11 mg/kg (n = 17).

The Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.11 mg/kg and recommended a maximum residue level of 0.2 mg/kg for acetamiprid in fruiting vegetables, cucurbits.

Fruiting vegetables, other than Cucurbits

Residue data were provided to the Meeting from trials in USA on tomatoes, sweet peppers and chili peppers.

GAP in USA for fruiting vegetables (including tomatoes and peppers) is for a maximum of four foliar applications of up to 0.084 kg ai/ha and a PHI of 7 days. A GAP also exists in USA for indoor tomatoes, applying acetamiprid as a single soil application through drip irrigation systems, using up to 0.084 kg ai/ha (3.4 g ai/1000 plants) and with a PHI of 1 day.

In trials on field tomatoes from USA matching this foliar application GAP, acetamiprid residues in tomatoes were: < 0.01, < 0.01, < 0.01, 0.01, 0.01, 0.02, 0.03, 0.03, 0.03, 0.03, 0.04, 0.04, 0.04, 0.06, 0.06, 0.08, 0.09 and 0.1 mg/kg.

In trials on indoor tomatoes from USA matching the US drip irrigation GAP, acetamiprid residues in tomatoes were: < 0.01, 0.04 and 0.05 mg/kg.

In trials on sweet peppers from USA matching this foliar application GAP, acetamiprid residues were: 0.01, 0.02, 0.03, 0.03, 0.04, 0.06, 0.07 and 0.09 mg/kg.

In trials on chili peppers from USA matching this foliar application GAP, acetamiprid residues were: 0.06, 0.08 and 0.14 mg/kg.

The Meeting noted that the data sets from foliar applications to field tomatoes, sweet peppers and chili peppers were similar and agreed combine them to estimate a group maximum residue level, STMR and HR for fruiting vegetables other than Cucurbits.

The combined data set for tomatoes, sweet peppers and chili peppers is: < 0.01, < 0.01, < 0.01, 0.01, 0.01, 0.01, 0.02, 0.02, 0.03, 0.03, 0.03, 0.03, 0.03, 0.03, 0.04, 0.04, 0.04, 0.04, 0.06, 0.06, 0.06, 0.06, 0.07, 0.08, 0.08, 0.09, 0.09, 0.1 and 0.14 mg/kg (n = 29).

The Meeting estimated an STMR of 0.04 mg/kg, an HR of 0.14 mg/kg and recommended a maximum residue level of 0.2 mg/kg for acetamiprid in fruiting vegetables, other than cucurbits (except sweet corn and mushrooms).

For dried chili peppers, using the combined data set for the fruiting vegetables (except cucurbits) and a dehydration factor of 10, the Meeting estimated an STMR of 0.4 mg/kg, an HR of 1.4 mg/kg and recommended a maximum residue level of 2 mg/kg for acetamiprid in dried chili peppers.

Leafy vegetables

Residue data were provided to the Meeting from trials in USA on head and leaf lettuce, spinach and mustard greens and from trials in Europe on field lettuce and protected lettuce.

GAP in Italy, France and Spain for leafy vegetables (or lettuce and other similar salad vegetables) is for a maximum of two foliar applications of up to 0.05 kg ai/ha and a PHI of 7 days (or 3 days for indoor crops in Italy).

In trials on field lettuce from France, Spain and Italy matching this GAP, acetamiprid residues in lettuce were: 0.04, 0.06, 0.06, 0.1, 0.11, 0.14 and 0.17 mg/kg.

In trials on field lettuce from France, Germany and UK matching this GAP, acetamiprid residues in lettuce were: 0.08, 0.14, 0.15, 0.16, 0.24, 0.25, 0.28 and 0.31 mg/kg.

In trials on protected lettuce from France, Germany, Italy and UK matching the Italian GAP (2×0.05 kg ai/ha, PHI 3 days), residues in lettuce were: 0.33, 0.33, 0.41, 0.5, 0.78, 0.88, 0.88 and 1.9 mg/kg (n = 8).

GAP in USA for leafy vegetables is for a maximum of five foliar applications of up to 0.084 kg ai/ha and a PHI of 7 days.

In trials on field grown head lettuce from USA matching this GAP, acetamiprid residues in head lettuce (with wrapper leaves) were: 0.06, 0.14, 0.26, 0.28, 0.38, 0.42, 0.65 and 0.68 mg/kg.

In trials on field grown leaf lettuce from USA matching this GAP, acetamiprid residues in leaf lettuce were: 0.11, 0.12, 0.3, 0.41, 0.46, 0.61, 0.87 and 0.96 mg/kg.

In trials on spinach from USA matching this GAP, acetamiprid residues were: 0.03, 0.04, 0.21, 0.46, 0.55, 1.1, 2.1 and 2.5 mg/kg (n = 8).

In trials on mustard greens from USA matching this GAP, acetamiprid residues were: 0.11, 0.18, 0.19, 0.3, 0.43, 0.49, 0.68, 0.74 and 1.1 mg/kg.

The Meeting noted that the data sets for head lettuce, leaf lettuce, spinach and mustard greens matching the USA GAP were similar and considered estimating a group maximum residue level, STMR and HR for leafy vegetables based on the spinach data.

However, noting that for spinach the proposed maximum residue level would result in an IESTI that exceeded the ARfD by 180%, the Meeting agreed to estimate a group maximum residue level for leafy vegetables except spinach, based on the data for indoor lettuce matching the Italian GAP.

The Meeting estimated an STMR of 0.64 mg/kg, an HR of 1.9 mg/kg and recommended a maximum residue level of 3 mg/kg for acetamiprid in leafy vegetables except spinach.

For spinach, the Meeting proposed a maximum residue level of 5 mg/kg and estimated an STMR of 0.51 mg/kg and an HR of 2.5 mg/kg, noting that this would result in an exceedance of the ARfD and that an alternative GAP for spinach could not be identified.

Legume vegetables

Residue data were provided to the Meeting from trials in USA on beans and peas (with and without pods).

GAP in USA for legume vegetables is for a maximum of three foliar applications of up to 0.112 kg ai/ha and a PHI of 7 days.

In trials on beans from USA matching this GAP, acetamiprid residues in beans (without pods) were: 0.02, 0.03, 0.04, 0.08, 0.11 and 0.18 mg/kg.

In trials on peas from USA matching this GAP, acetamiprid residues in peas (without pods) were: < 0.01, 0.01, 0.02, 0.02, 0.03, 0.03 mg/kg.

The Meeting estimated an STMR of 0.03 mg/kg, an HR of 0.18 mg/kg and recommended a maximum residue level of 0.3 mg/kg for acetamiprid in peas, shelled and for beans, shelled.

In trials on beans from USA matching the USA GAP for legume vegetables (3× 0.112 kg ai/ha, PHI 7 days), acetamiprid residues in beans (with pods) were: < 0.01, < 0.01, < 0.01, < 0.01, 0.02 and 0.18 mg/kg.

The Meeting estimated an STMR of 0.01 mg/kg, an HR of 0.18 mg/kg and recommended a maximum residue level of 0.4 mg/kg for acetamiprid in beans, except broad bean and soya bean.

In trials on peas in USA matching this GAP, acetamiprid residues in peas (with pods) were: 0.08, 0.13 and 0.27 mg/kg.

The Meeting agreed the data were not sufficient to recommend a maximum residue level for acetamiprid in peas (with pods).

Stalk and stem vegetables

Residue data were provided to the Meeting from trials in USA on celery.

GAP in USA for leafy vegetables (including celery) is for a maximum of five foliar applications of up to 0.084 kg ai/ha and a PHI of 7 days.

In trials on celery from USA matching this GAP, acetamiprid residues in celery stalks and leaves were: 0.08, 0.17, 0.27, 0.27, 0.32, 0.41, 0.51 and 0.78 mg/kg (n = 8).

The Meeting estimated an STMR of 0.3 mg/kg, an HR of 0.78 mg/kg and recommended a maximum residue level of 1.5 mg/kg for acetamiprid in celery.

Tree nuts

Residue data were provided to the Meeting from trials in USA on almonds and pecans.

GAP in USA for tree nuts (including almonds and pecans) is for a maximum of four foliar applications of up to 0.2 kg ai/ha and a PHI of 14 days.

In trials on almonds in USA matching this GAP, acetamiprid residues in nut meat were: < 0.01, < 0.01, < 0.01, 0.01, 0.01 and 0.02 mg/kg.

In trials on pecans in USA matching this GAP, acetamiprid residues in nut meat were: < 0.01, < 0.01, < 0.01, 0.01 and 0.05 mg/kg.

The Meeting noted that these data sets for almonds and pecans were similar and agreed to combine them to support a group maximum residue level. The combined data set is: < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, < 0.01, 0.01, 0.01, 0.01, 0.02 and 0.05 (n = 12).

The Meeting estimated an STMR of 0.01 mg/kg, an HR of 0.05 mg/kg and recommended a maximum residue level of 0.06 mg/kg for acetamiprid in tree nuts.

Oil seeds

Residue data were provided to the Meeting from trials in USA on cotton.

GAP in USA for cotton is for a maximum of four foliar applications of up to 0.112 kg ai/ha and a PHI of 28 days.

In trials on cotton in USA matching this GAP, acetamiprid residues in cotton seed were: < 0.01, 0.02, 0.02, 0.03, 0.05, 0.06, 0.09, 0.09, 0.1, 0.1, 0.12, 0.14, 0.36 and 0.5 mg/kg (n = 14).

The Meeting estimated an STMR of 0.09 mg/kg and recommended a maximum residue level of 0.7 mg/kg for acetamiprid in cotton seed.

Animal feeds***Almond hulls***

In trials on almonds in USA matching the USA GAP, acetamiprid residues in almond hulls (as received) were: 0.22, 0.78, 0.78, 1.9, 2.0 and 3.8 mg/kg.

The Meeting estimated a median residue of 1.34 mg/kg for almond hulls.

Cotton gin trash

In trials on cotton in USA matching this GAP, acetamiprid residues in cotton gin trash were: 0.39, 1.5, 1.9, 1.9, 2.7, 3.0, 3.6, 3.9, 3.9, 4.0, 5.8, 6.5, 7.3 and 18 mg/kg (n = 14).

The Meeting estimated a median residue of 3.6 mg/kg and a highest residue of 18 mg/kg for acetamiprid for cotton gin trash.

Fate of residues during processing

The effect of processing on the nature of residues was investigated in buffer solutions under a range of hydrolysis conditions. Acetamiprid was shown to be stable for at least 35 days in buffer solutions at pH 4, 5, 7 and 9, incubated at up to 45 °C.

The fate of acetamiprid residues has been examined in a number of studies with oranges, tomatoes, apples, cotton seed and plums, reflecting household and simulated commercial processing.

A summary of processing factors (PF) derived from the data on the above commodities is shown below. Based on the estimations made on the raw agricultural commodities, STMR-Ps and HR-Ps were estimated by multiplying the STMR of the raw commodity by the PF. Maximum residue levels were only estimated for commodities with a Codex code and of importance in international trade.

Summary of selected processing factors for acetamiprid

Raw agricultural commodity (RAC)	STMR (mg/kg)	HR (mg/kg)	Processed commodity	Processing factor	STMR-P	HR-P	
Oranges	0.25		Juice ^a	< 0.13	0.03		
			Pulp	0.24	0.05		
			Pulp, dry	2.8	0.7		
			Peel	2.83	0.71		
			Oil ^a	< 0.16	0.04		
Apple	0.23		Juice	0.88	0.2		
			Wet pomace	1.34	0.31		
Plums	0.04	0.11	Dried prunes	2.96	0.12	0.32	
Grape	0.085		Juice	1.5	0.13		
			Raisins	0.93	0.08	0.23	
Tomatoes	0.4		Purée	1.4	0.56		
			Paste	3.1	1.24		
Cotton	0.09		Meal	0.38	0.03		
			Hulls	0.79	0.07		
			Refined Oil ^a	< 0.04	0.004		

^a Residues below LOQ

The Meeting recommended a maximum residue level of 0.6 mg/kg for dried prunes, based on the recommended maximum residue level for plums (0.2 mg/kg) and an average processing factor of 2.9.

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of acetamiprid in farm animals on the basis of the diets listed in Annex 6 of the 2009 JMPR Report (OECD Feedstuffs Derived from Field Crops) and the STMR or highest residue levels estimated at the present Meeting. Dietary burden calculations are provided in Annex 6 and are summarized below.

Animal dietary burden for acetamiprid, ppm of dry matter diet

	Maximum Dietary Burden	Mean Dietary Burden
Beef cattle	1.085	0.435
Dairy cattle	0.836	0.413
Poultry broiler	0.007	0.007
Poultry layer	0.168	0.032

Farm animal feeding studies

Dairy cows

The Meeting received information on the residue levels arising in animal tissues and milk when dairy cows were dosed with acetamiprid for 28 days at 5.77, 17.4 and 58.6 ppm in the diet.

In milk, average residues (3 animals) of acetamiprid in whole milk from treated animals were < 0.02 in the low dose group (5.77 ppm) and were found at up to 0.21 mg/kg in the higher dose groups. Residues of IM-2-1 (the predominant residue) were 0.03–0.06 mg/kg in the low dose group and up to 0.95 mg/kg in the higher dose groups. Residue concentrations of acetamiprid in whole milk increased rapidly, reaching a plateau within 1 day with concentrations of IM-2-1 reaching a plateau at about 7–8 days.

In liver and kidney, acetamiprid residues were < 0.05 mg/kg in the low dose group and up to 0.25 mg/kg in the higher dose groups. Residues of acetamiprid in muscle and fat were < 0.01 mg/kg in the low dose group and did not exceed about 0.1 mg/kg and 0.06 mg/kg respectively in the higher dose groups.

The predominant residue in all tissues was the IM-2-1 metabolite, with highest residues in liver and kidney (0.1–0.2 mg/kg – low dose) and up to about 2.4 mg/kg in higher dose groups. Levels of about 0.05 mg/kg were present in muscle and fat (low dose group); these increasing to 1.0 mg/kg in muscle and 0.65 mg/kg in fat in the higher dose groups.

Residues of total acetamiprid (parent+IM-2-1) in the low dose group averaged 0.24 mg/kg (kidney), < 0.15 mg/kg (liver), 0.048 mg/kg (muscle), 0.037 mg/kg (fat) and 0.063 mg/kg (milk) and generally increased proportionally in the higher dose groups. Maximum residues in individual (low dose) animals were < 0.25 mg/kg (kidney), < 0.15 mg/kg (liver), < 0.05 mg/kg (muscle) and < 0.072 mg/kg (fat).

Laying hens

A feeding study was also conducted with laying hens, fed 1.16 ppm, 3.55 ppm or 12 ppm acetamiprid daily for 28 days.

In eggs, residues of acetamiprid were not detectable or < 0.01 mg/kg in all dose groups, with the IM-2-1 metabolite being found at up to 0.33 mg/kg in the 12 ppm dose group, reaching a plateau at about day 8.

In tissues, acetamiprid was also not detected in any dose group, with IM-2-1 metabolite being found only in liver (< 0.1 mg/kg) in the low dose group and in the high dose group at levels of up to 0.5 mg/kg in liver, up to 0.075 mg/kg in muscle and up to 0.012 mg/kg in fat.

*Animal commodity maximum residue levels**Cattle*

For maximum residue level estimation, the high residues of acetamiprid plus IM-2-1 in tissues were calculated by extrapolating the maximum dietary burden (1.085 ppm) from the lowest feeding level (5.77 ppm) in the dairy cow feeding study and using the highest tissue concentrations of total acetamiprid from individual animals within those feeding groups.

The STMR values for the tissues were calculated by extrapolating the STMR dietary burden (0.435 ppm) from the lowest feeding level in the dairy cow feeding study and using the mean tissue concentrations of total acetamiprid from those feeding groups.

For milk maximum residue level estimation, the high residues in the milk were calculated by extrapolating the maximum dietary burden for dairy cattle (0.836 ppm) from the lowest feeding level (5.77 ppm) in the dairy cow feeding study and using the mean milk concentrations of total acetamiprid from this feeding group.

The STMR value for milk was calculated by extrapolating the mean dietary burden for dairy cows (0.413 ppm) from the lowest feeding level (5.77 ppm) in the dairy cow feeding study and using the mean milk concentrations of total acetamiprid from this feeding group.

	Acetamidrid feed level (ppm) for milk residues	Total acetamidrid residues (mg/kg) in milk	Acetamidrid feed level (ppm) for tissue residues	Total acetamidrid residues (mg/kg) in:			
				Muscle	Liver	Kidney	Fat
Maximum residue level for beef or dairy cattle							
Feeding study ^a	5.77	0.063	5.77	0.05	0.15	0.25	0.07
Dietary burden and residue estimate	0.836	0.009	1.085	0.009	0.028	0.047	0.013
STMR beef or dairy cattle							
Feeding study ^b	5.77	0.063	5.77	0.048	0.15	0.24	0.037
Dietary burden and residue estimate	0.413	0.004	0.435	0.004	0.011	0.018	0.003

^a Highest residues for tissues and mean residues for milk

^b Mean residues for tissues and for milk

The Meeting estimated maximum residue levels of 0.02 mg/kg for acetamiprid in meat (from mammals other than marine mammals), 0.02 mg/kg for mammalian fat, 0.05 mg/kg for edible offal (mammalian) and 0.02 mg/kg for milks.

Estimated STMRs are 0.004 mg/kg for mammalian muscle, 0.003 mg/kg for mammalian fat, 0.011 mg/kg for mammalian liver, 0.018 mg/kg for mammalian kidney and 0.004 mg/kg for milks.

Estimated HRs are 0.01 mg/kg for mammalian fat, 0.007 mg/kg for mammalian muscle, 0.022 mg/kg for mammalian liver, 0.036 mg/kg for mammalian kidney and 0.009 mg/kg for milks.

Poultry

In lowest dose feeding study (1.16 ppm), residues of acetamiprid were not detectable in eggs or any poultry tissues and the combined residues of acetamiprid and IM-2-1 averaged 0.027 mg/kg in eggs, were < 0.01 mg/kg in muscle and fat and was up to 0.09 mg/kg in liver.

Noting that the maximum dietary burden for poultry layers (0.168 ppm) is about 7 times lower than the lowest dose in the feeding study, the Meeting concluded that residues of acetamiprid and its IM-2-1 metabolite would not be expected in eggs.

For poultry liver, muscle and fat, based on a maximum dietary burden of 0.032 ppm (more than 36 times lower than the lowest dose feeding study, the Meeting concluded that residues of acetamiprid and its IM-2-1 metabolite would not be expected in poultry edible tissues above the LOQs of 0.05 mg/kg (liver) and 0.01 mg/kg (muscle and fat).

The Meeting estimated maximum residue levels of 0.01 (*) mg/kg for acetamiprid in poultry meat, poultry fat, and eggs and 0.05 mg/kg for poultry edible offal.

Estimated HRs and STMRs for dietary intake estimation for acetamiprid are 0.0 mg/kg for poultry eggs, meat and fat and for poultry edible offal, the Meeting estimated an HR of 0.01 mg/kg and an STMR of 0.0 mg/kg.

RECOMMENDATIONS

On the basis of the data from supervised trials the Meeting concluded that the residue levels listed below are suitable for establishing maximum residue limits and for IEDI assessment.

Definition of the residue for plant commodities (for compliance with MRL and estimation of dietary intake): *acetamiprid*.

Definition of the residue for animal commodities (for compliance with MRL and estimation of dietary intake): *sum of acetamiprid and desmethyl-acetamiprid, expressed as acetamiprid*.

The residue is not fat-soluble

	Commodity	MRL	mg/kg	STMR or	HR
CCN	Name	New	Prev	STMR-P	
VP 0061	Beans, except broad bean and soya bean	0.4		0.01	0.18
VP 0062	Beans, shelled	0.3		0.03	0.18
FB 0018	Berries and other small fruit (except grapes and strawberries)	2		0.64	1
VB 0041	Cabbages, Head	0.7		0.02 [0.09] ^a	0.05 [0.5] ^a
VX 0624	Celery	1.5		0.3	0.78
FS 0013	Cherries	1.5		0.45	0.88
FC 001	Citrus fruits	0.8		0.25	0.45
SO 0691	Cotton seed	0.7		[0.09] ^a	
HS 0444	Peppers Chili, dried	2		0.4	1.4
DF 0014	Prunes	0.6		0.12	0.32
MO 0105	Edible offal (Mammalian)	0.05		0.011 liver 0.018 kidney	0.03 liver 0.05 kidney
PE 0112	Eggs	0.01 *		0.0	0.0
VB 0042	Flower-head brassicas (includes Broccoli: Broccoli, Chinese and Cauliflower)	0.4		0.02	0.22
VC 0045	Fruiting vegetables, Cucurbits	0.2		0.05	0.11
VO 0050	Fruiting vegetables, other than Cucurbits (except sweetcorn & mushrooms)	0.2		0.04	0.14
VA 0381	Garlic	0.02		0.01	0.01
FB 0269	Grapes	0.5		0.085	0.25
VL 0053	Leafy vegetables (except spinach)	3		0.64	1.9
MF 0100	Mammalian fats (except milk fats)	0.02		0.003	0.01
MM 0093	Meat (from mammals other than marine mammals)	0.02		0.003 fat 0.004 muscle	0.01 fat 0.01 muscle
ML 0106	Milks	0.02		0.004	0.009
FS 0245	Nectarines	0.7		0.2	0.44
VA 0385	Onion, Bulb	0.02		0.01	0.01
FS 0247	Peach	0.7		0.2	0.44
VP 0064	Peas, shelled (succulent seeds)	0.3		0.03	0.18
FS 0014	Plums (including Prunes)	0.2		0.04	0.11
EP 0009	Pome fruits	0.8		0.225	0.59
PM 0110	Poultry meat	0.01 *		0.0	0.0
PO 0111	Poultry, Edible offal of	0.05 *		0.01	0.0
VL 0502	Spinach	5		0.51	2.5
VA 0389	Spring onions	5		0.38	2
FB 0275	Strawberries	0.5		0.1	0.24
TN 0085	Tree nuts	0.06		0.01	0.05

	Commodity	MRL	mg/kg	STMR or	HR
CCN	Name	New	Prev	STMR-P	
AM 0660	Almond hulls			[1.34] ^a	
JF 0226	Apple juice			0.2	
AB 1230	Apple pomace, wet			[0.32] ^a	
JF 0001	Citrus juice			0.03	
	Citrus oil			0.04	
	Citrus peel			0.71	
	Citrus pulp			0.05	
AB 0001	Citrus pulp (dry)			[0.7] ^a	
	Cotton gin trash			[3.6] ^a	[18] ^a
AB 0691	Cotton seed hulls			[0.07] ^a	
AB 1203	Cotton seed meal			[0.03] ^a	
JF 0269	Grape juice			0.13	
DF 0269	Dried grapes (= currants, Raisins and Sultanas)			0.08	0.23
OR 0691	Cotton seed oil, edible			0.004	
VW 0448	Tomato paste			0.09	
	Tomato purée			0.04	

^a [] Used to estimate animal dietary burden

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intake (IEDI) for acetamiprid was calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available. The results are shown in Annex 3 of the 2011 JMPR Report.

The International Estimated Daily Intakes of acetamiprid for the 13 GEMS/Food regional diets, based on estimated STMRs were 0–3% of the maximum ADI of 0.07 mg/kg bw (see Annex 3 of the 2011 JMPR Report). The Meeting concluded that the long-term intake of residues of acetamiprid from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short-term Intake (IESTI) for acetamiprid was calculated for the food commodities for which STMRs or HRs were estimated and for which consumption data were available (see Annex 4 of the 2011 JMPR Report).

For spinach, the IESTI represented 180% of the ARfD of 0.1 mg/kg bw. On the basis of the information provided to the JMPR it was not possible to conclude that the estimate of short-term intake of acetamiprid, from the consumption of spinach, was less than the ARfD. The Meeting noted that an alternative GAP for spinach was not available.

For the other commodities considered by the JMPR, the IESTI represented 0–80% of the ARfD and the Meeting concluded that the short-term intake of residues of acetamiprid, when used in ways that have been considered by the JMPR (other than spinach), is unlikely to present a public health concern.

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Number	Author	Year	Title
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