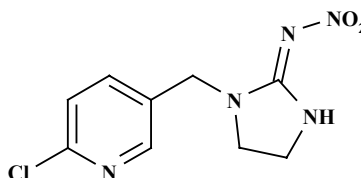


IMIDACLOPRID (206)

First draft prepared by Ursula Banasiak, Federal Biological Research Centre for Agriculture and Forestry (BBA), Kleinmachnow, Germany

IDENTITY

ISO Common name:	imidacloprid
Chemical name:	
IUPAC:	1-(6-chloro-3-pyridylmethyl)- <i>N</i> -nitroimidazolidin-2-ylideneamine
CA:	1-[(6-chloro-3-pyridinyl)methyl]- <i>N</i> -nitro-1 <i>H</i> -2-imidazolidinimine
CAS number:	138261-41-3
CIPAC number:	582
Synonym:	BAY NTN 33893; Confidor, Gaucho, Admire, Provado
Structural formula:	



Molecular formula:	C ₉ H ₁₀ ClN ₅ O ₂
Molecular mass:	255.7 g/mol

Physical and chemical properties

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

References to test materials used:

- 1 BAY NTN 33893 (batch 920717ELB01, purity 99.9%)
- 2 BAY NTN 33893 (batch 880208ELB01, purity 99.5%)
- 3 BAY NTN 33893 (batch 910719ELB02, purity 97.2%)
- 4 BAY NTN 33893 (batch 890315ELB01, purity 99.8%)
- 5 BAY NTN 33893 (batch 910605ELB03, purity 97.2%)
- 6 BAY NTN 33893 technical (batch 17901/88, purity 93.6%)
- 7 BAY NTN 33893 technical (batch 17101/89, purity 92.6%)

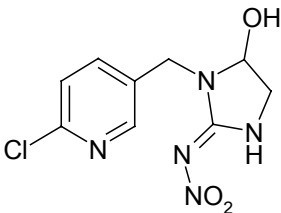
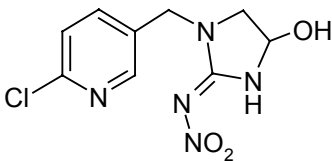
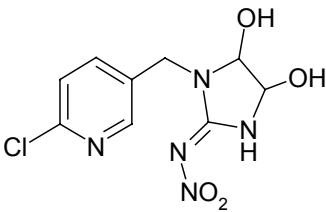
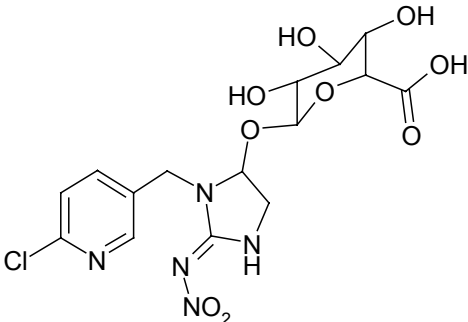
Table 1. Physical and chemical properties of imidacloprid.

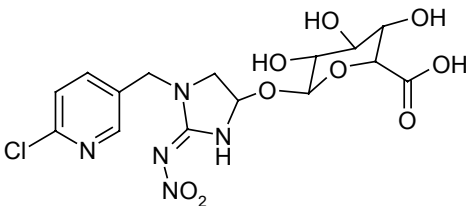
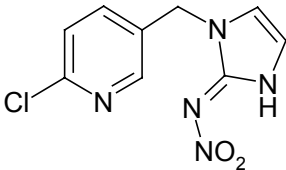
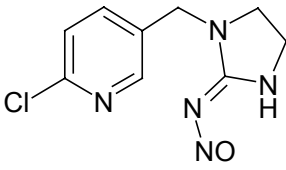
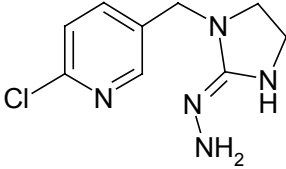
Property	Results	Test material, method	Reference (report)
Physical state, colour	Active substance as manufactured: beige powder		Anon., 2000
Odour	Active substance as manufactured: slight characteristic smell		Anon., 2000

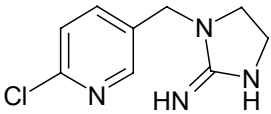
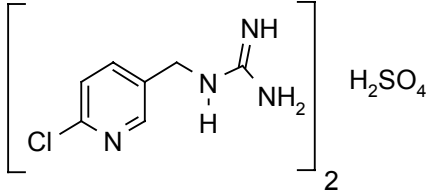
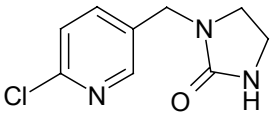
Property	Results	Test material, method	Reference (report)
Vapour pressure	$2.80 \cdot 10^{-8}$ to $3.97 \cdot 10^{-8}$ Pa at 50°C $1.25 \cdot 10^{-7}$ to $1.47 \cdot 10^{-7}$ Pa at 60°C $4.07 \cdot 10^{-7}$ to $4.15 \cdot 10^{-7}$ Pa at 70°C Conclusion: $4 \cdot 10^{-10}$ Pa at 20°C (extrapolated) $9 \cdot 10^{-10}$ Pa at 25°C (extrapolated)	Material 1, OECD 104	Krohn, 1993a (PC313)
Melting point	144°C	Material 1, OECD 102	Krohn, 1993b (PC312)
Partition coefficient n-octanol/water	P_{OW} 3.7 $\log P_{OW}$ 0.57 at 21°C The effect of pH (4-9) was not investigated because there is no influence of pH on the water solubility.	Material 4, OECD 107	Krohn, 1992 (PC337)
Solubility in water	0.61 g/l at 20°C The solubility is not influenced in the range pH 4 to pH 9.	Material 1, CIPAC MT 157	Krohn, 1993c (PC320)
Solubility in organic solvents (at 20°C, in g/l)	n-hexane <0.1 toluene 0.69 2-propanol 2.3 ethyl acetate 6.7 acetonitrile 50 acetone 50 dichloromethane 67 dimethylformamide >200 dimethyl sulfoxide >200	Material 5, CIPAC MT 157, part 2	Krohn, 1993d (PC323)
Relative density	1.41 g/cm ³ at 20°C	Material 3, OECD 109	Krohn, 1995 (PC713)
Hydrolysis rate	Stable in buffered solutions at pHs 5 and 7 at 25°C in the dark, slow degradation at pH 9 with half-life of approx. 1 year		Yoshida, 1989 (NR1276)
Photochemical degradation	Half-life 57 min.		Anderson <i>et al.</i> , 1988 (PF3517)
Dissociation constant	Imidacloprid shows very weak basic properties. Complete protonation only in non-aqueous solutions of very strong acids. Not possible to specify a pK value in pure aqueous systems.	Material 4, OECD 112	Rosenfeldt, 1992 (PC317)
Temperature of decomposition or sublimation	DTA (differential thermoanalysis) measurement: exothermic reaction above 210°C. TGA (thermogravimetric analysis) measurement: weight loss both under nitrogen and air atmospheres above 230°C.	Material 2, OECD 113	Mix and Berg, 1988 (PC339)
Volatility	Henry's law constant at 20°C (calculated) $2 \cdot 10^{-10}$ Pa · m ³ · mol ⁻¹		Krohn, 1993e (PC315)
Storage stability	Weight loss?? negligible during 36 months storage at normal warehouse temperatures. Good chemical stability indicated and shelf-life of at least 3 years.	Material 6, 7, according to OPPTS 830.6313	Swan, 1991

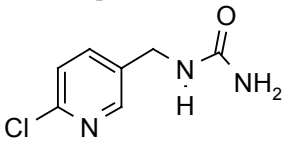
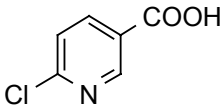
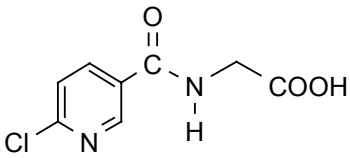
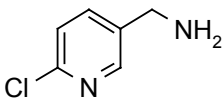
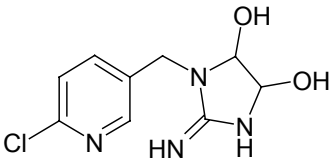
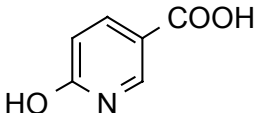
METABOLISM AND ENVIRONMENTAL FATE

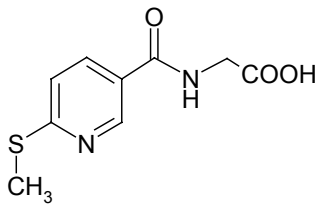
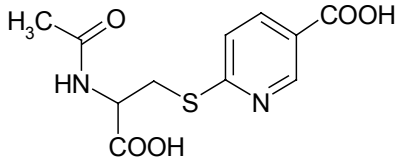
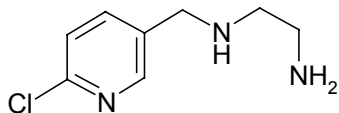
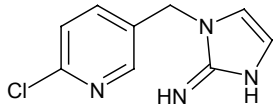
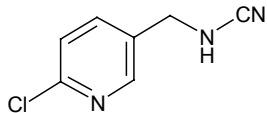
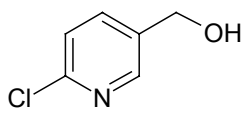
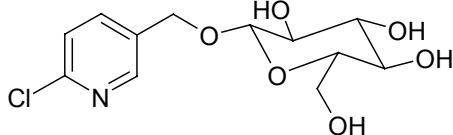
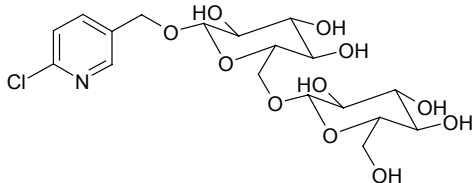
Chemical names, structures and code numbers of metabolites and degradation products of imidacloprid are shown below.

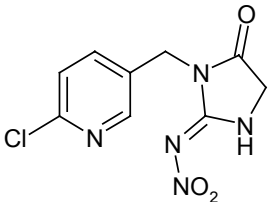
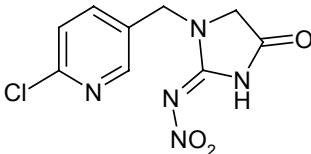
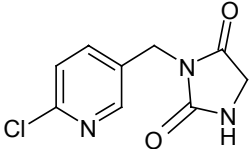
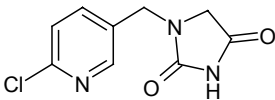
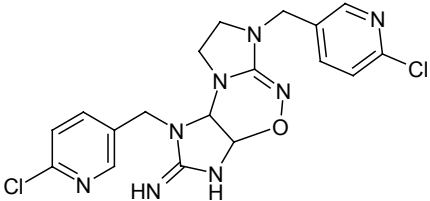
No.	Short name Structure	Molecular formula CAS no. Chemical name Company code	Occurrence
M01	5-hydroxy compound 	Molecular formula: C ₉ H ₁₀ ClN ₅ O ₃ CAS no: 155802-61-2 1-(6-chloro-3-pyridylmethyl)-5-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine BNF 3417 (mixture M01 + M02) BNF 3918Y DIJ 10052 KNO 2041C KWG 4557 Ne 727 d (mixture M01 + M02) WAK 4103	Animals PF3558, PF3759 PF3760, PF3731 Plants M9884, NR1277 NR1284, NR1290 PF3179, PF3257 PF3628, PF3667 PF3673, PF3674 PF3676, PF3678 PF3997 Soil NR1283, PF3438 Water PF3950
M02	4-hydroxy compound 	Molecular formula: C ₉ H ₁₀ ClN ₅ O ₃ CAS-no: 155802-62-3 1-(6-chloro-3-pyridylmethyl)-4-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine BNF 3417 (mixture M01 + M02) BNF 3918Y BNF 5540B KNO 2041A Ne 727 d (mixture M01 + M02) WAK 5839	Animals PF3759, PF3760 PF3731 Plants PO197/1814 PF3179, PF3257 PF3667, PF3673 PF3674, PF3676 PF3678, PF3997 Soil PF3438
M03	dihydroxy compound 	Molecular formula: C ₉ H ₁₀ ClN ₅ O ₄ 1-(6-chloro-3-pyridylmethyl)-4,5-dihydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine BNF 3419 X Ne 727 a WAK 3772	Animals PF3759, PF3760 Plants M9884, PF3628 PF3257, PF3667 PF3673, PF3674 PF3676, PF3678
M04	5-hydroxy glucuronide 	Molecular formula: C ₁₅ H ₁₈ ClN ₅ O ₉ 1-(6-chloro-3-pyridylmethyl)-5-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine glucuronide WAK 4103 glucuronide	Animals PF3760, PF3731

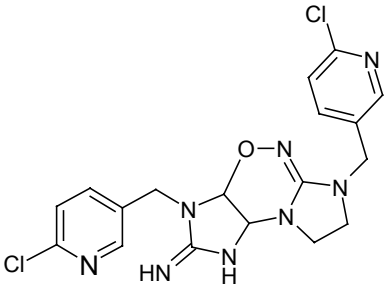
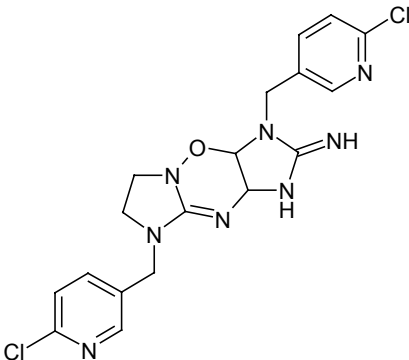
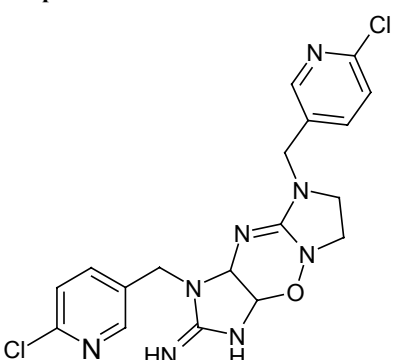
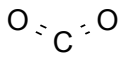
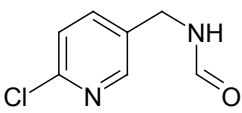
No.	Short name Structure	Molecular formula CAS no. Chemical name Company code	Occurrence
M05	4-hydroxy glucuronide 	Molecular formula: C ₁₅ H ₁₈ ClN ₅ O ₉ 1-(6-chloro-3-pyridylmethyl)-4-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine glucuronide WAK 5839 glucuronide	Animals PF3760
M06	olefin 	Molecular formula: C ₉ H ₈ ClN ₅ O ₂ CAS-no: 115086-54-9 1-(6-chloro-3-pyridylmethyl)- <i>N</i> -nitro-4-imidazolin-2-ylideneamine BNF 3917X GAJ 2269 NTN 35884 Ne 727e WAK 3745 WAK 5868	Animals PF3558, PF3759 PF3760, PF3731 Plants M9884, NR1277 NR1284, R1290 PO197/1814 PF3179, PF3628 PF3257, PF3667 PF3673, PF3674 PF3675, PF3676 PF3678, PF3997 Soil PF3321, PF3433, NR1283
M07	nitrosimine 	Molecular formula: C ₉ H ₁₀ ClN ₅ O 1-(6-chloro-3-pyridylmethyl)- <i>N</i> -nitrosoimidazolidin-2-ylideneamine BNF 5427 E F 4044B KNO 2212 Ne 746 NTN 37571 WAK 3839	Animals PF3558 PF3731 Plants M9884, NR1277 NR1284, R1290 PO197/1814, PF3179, PF3628 PF3257, PF3667 PF3673, PF3674 PF3675, PF3676 PF3678, PF3997 Soil PF3321, PF3433 PF3438, NR1283 Water PF3950, PF4337
M08	amino compound 	Molecular formula: C ₉ H ₁₂ ClN ₅ 1-(6-chloro-3-pyridylmethyl)- <i>N</i> -aminoimidazolidin-2-ylideneamine ECW 8096, NTN 36804, WAK 3877/4	Animals PF3760 Proposed as intermediate Plant Proposed as intermediate

No.	Short name Structure	Molecular formula CAS no. Chemical name Company code	Occurrence
M09	denitro compound 	Molecular formula: C ₉ H ₁₁ ClN ₄ CAS-no: 115970-17-7 1-(6-chloro-3-pyridylmethyl)imidazolidin-2-ylideneamine BEG 5322 BNF 5110B BNF 5427 BNF 5529B NTN 33823 NTN 38014 WAK 4140 WAK 6079	Animals PF3558, PF3760, PF3731 Plants M9884, NR1277 NR1284, NR1290 PO197/1814 PF3628, PF3257 PF3667, PF3673 PF3674, PF3675 PF3676, PF3678 PF3997 Soil BR107819 PF3321, PF3433 PF3438 Water BR107546 BR107547 PF3466, PF3517 PF3524, PF3950, PF4337
M10	guanidine compound (shown as sulfate) 	Molecular formula: C ₁₄ H ₁₈ Cl ₂ N ₈ 1-(6-chloro-3-pyridylmethyl)guanidine sulfate BNF 5127B ECW 8044 WAK 4126 WAK 5756	Animals PF3759, PF3760 PF3731 Plants PF3673 PF3674, PF3997 Water PF3517
M11	nitroguanidine 	Molecular formula: C ₇ H ₈ ClN ₄ O ₂ 1-(6-chloro-3-pyridylmethyl)-2-nitroguanidine DIJ 11324 WAK 4230	Animals PF3759, PF3760 Soil PF3321, PF3433 PF3438
M12	2-ketone 	Molecular formula: C ₉ H ₁₀ ClN ₃ O CAS-no: 120868-66-8 1-(6-chloro-3-pyridylmethyl)imidazolidin-2-one BNF 5104B DIJ 9817 NTN 33519 WAK 4740	Animals PF3760, PF3731 Plants M9884, NR1277 NR1284, NR1290 PO197/1814 PF3257, PF3667 PF3673, PF3676 PF3997 Soil BR107819, NR1283 Water BR107547 NR1276 PF3517, PF3950 PF3524, PF4337

No.	Short name Structure	Molecular formula CAS no. Chemical name Company code	Occurrence
M13	urea compound 	Molecular formula: C ₇ H ₈ ClN ₃ O 1-(6-chloro-3-pyridylmethyl)urea DIJ 10739	Animals PF3759 PF3760
M14	6-CNA 	Molecular formula: C ₆ H ₆ ClNO ₂ 6-chloronicotinic acid BNF 5119B BNF 5518A	Animals PF3731, PF3759 PF3760 Plants M9884, NR1277 NR1284, NR1290 PF3628, PF3257 PF3667, PF3673 PF3674, PF3675 PF3997 Soil NR1283, PF3321, PF3433 PF3438 Water BR107547 PF3466, PF3517 PF3950, PF4337
M15	6-CNA-glycine 	Molecular formula: C ₈ H ₇ ClN ₂ O ₃ N-(6-chloronicotinoyl)glycine WAK 3583	Animals PF3760, PF3731
M16	AMCP 	Molecular formula: C ₆ H ₇ ClN ₂ 6-chloro-3-pyridylmethylamine BNF 5312B GSE 1478	Animals PF3759, PF3760 Water PF3517
M17	dihydroxyimine 	Molecular formula: C ₉ H ₁₁ ClN ₄ O ₂ 1-(6-chloro-3-pyridylmethyl)-4,5-dihydroxyimidazolidin-2-ylideneamine BNF 3975Z BNF 5306A WAK 5031	Plant PF3179 Animals PF3759 PF3760 Water PF3517
M18	6-hydroxynicotinic acid 	Molecular formula: C ₆ H ₅ ClNO ₃ 6-hydroxynicotinic acid BNF 5540A GBH 4315	Soil NR1283 Water PF3950 PF4337

No.	Short name Structure	Molecular formula CAS no. Chemical name Company code	Occurrence
M19	methylthionicotinoyl-glycine 	Molecular formula: C ₉ H ₁₀ N ₂ O ₃ S <i>N</i> -[6-(methylthio)nicotinoyl]glycine	Animals Proposed intermediate
M21	mercapturic acid -NA 	Empirical formula: C ₁₁ H ₁₂ N ₂ O ₅ S 6-[(2-acetamido-2-carboxy)ethylthio]nicotinic acid Mercapturic acid derivate of CNA	Animals Proposed intermediate
M22	PEDA 	Molecular formula: C ₈ H ₁₂ N ₄ <i>N</i> -(6-chloro-3-pyridylmethyl)ethylenediamine DIJ 9646 -2	Animals PF3731 Water PF3466 PF4337
M23	denitro-olefin 	Molecular formula: C ₉ H ₉ ClN ₄ 1-(6-chloro-3-pyridylmethyl)-4-imidazolin-2-ylidenediamine ANC 2126	Soil BR107819 Water PF3517
M24	cyanamide 	Molecular formula: C ₇ H ₆ ClN ₃ <i>N</i> -(6-chloro-3-pyridylmethyl)carbamonitrile WAK 4613/3	Animals PF3558
M28	CHMP 	Molecular formula: C ₆ H ₆ ClNO 6-chloro-3-pyridylmethanol DIJ 9805	Plants M9884, PF3667 PF3673, PF3674 PF3675
M29	CHMP glucoside 	Molecular formula: C ₁₂ H ₁₆ ClNO ₆ 6-chloro-3-pyridylmethanol glucoside BNF 3949Y Ne 761 RBN 1114	Plants M9884, NR1290 PF3628, PF3257 PF3667, PF3674 PF3675, PF3676 PF3678, PF3997
M30	CHMP gentiobioside 	Molecular formula: C ₁₈ H ₂₆ ClNO ₁₁ 6-chloro-3-pyridylmethanol gentiobioside BNA 0358 Gentiobioside NE 770-1	Plants PF3257

No.	Short name Structure	Molecular formula CAS no. Chemical name Company code	Occurrence
M31	5-keto-imidacloprid 	Molecular formula: C ₉ H ₈ ClN ₅ O ₃ 1-(6-chloro-3-pyridylmethyl)-N-nitro-5-oxoimidazolidin-2-ylideneamine BNF 3976X WAK 3738 WAK 4236	Plants PF3179 PF3667
M32	4-keto-imidacloprid 	Molecular formula: C ₉ H ₈ ClN ₅ O ₃ 1-(6-chloro-3-pyridylmethyl)-N-nitro-4-oxoimidazolidin-2-ylideneamine BNF 3976 X	Plants PF3179 PF3667
M33	NTN33893 2,5-diketone 	Molecular formula: C ₉ H ₈ ClN ₃ O ₂ 1-(6-chloro-3-pyridylmethyl)imidazolidine-2,5-dione DIJ 10048 WAK 5060	Soil PF3321 PF3433 Water PF4337
M34	NTN33893 2,4-diketone 	Molecular formula: C ₉ H ₈ ClN ₃ O ₂ 1-(6-chloro-3-pyridylmethyl)imidazolidine-2,4-dione DIJ 10048	Soil PF3321 PF3433
M35 -38	NH photo dimer 	Molecular formula: C ₁₈ H ₁₈ Cl ₂ N ₈ O imidacloprid dimer, 4 possible structures	Water PF3517

No.	Short name Structure	Molecular formula CAS no. Chemical name Company code	Occurrence
M36	NH photo dimer 		
M37	NH photo dimer 		
M38	NH photo dimer 		
M39	carbon dioxide 		Soil PF3129, PF3130 PF3321, PF3433 PF3438 Water PF3466, PF3524 PF3950, PF4337
M40	formyl-AMCP 	Molecular formula: C ₇ H ₇ ClN ₂ O 2-chloro-5-(formylaminomethyl)pyridine BNF 5122B GSE 2712	Animals PF3760 Water PF3517

(NB: metabolites M20, M25, M26 and M27 were found only in rats)

Studies of metabolism and degradation were carried out with [¹⁴C]imidacloprid labelled at the methylene bridge and/or in the imidazolidine ring, as shown below.



* Position of label

It should be noted that some of the short names in Figures 1-5 differ from those listed above. It was not possible to change them in the edited monograph.

Animal metabolism

The metabolism of imidacloprid has been studied in laboratory rats, goats and hens according to GLP. Studies of the metabolism of imidacloprid and the plant metabolite M07 were evaluated by the WHO Core Assessment Group of the 2001 JMPR.

Goats. Two studies on lactating goats were reported (Karl *et al.*, 1991; Klein, 1992).

In the first a suspension consisting of 1 part [methylene-¹⁴C]imidacloprid and 9 parts of unlabelled compound with a specific radioactivity of 3.2 MBq/mg (87 μ Ci/mg) was given orally to a lactating goat (*Capra hircus*) of 31 kg body weight, 10.16 mg/kg bw/day, once a day for three consecutive days, corresponding to a mean dose of 315 mg/day or a total of 945 mg. Assuming a daily food consumption of 5% of body weight, the dose corresponds to an exaggerated concentration of 200 ppm in diet. A second goat was used as a control. The first goat was milked in the morning immediately before each dose and again 8 hours later, and immediately before being slaughtered. About 50 μ l of blood were taken from the ear veins of the goat 0.25, 0.5, 1, 2, 3, 4, 6 and 24 hours after the first dose, and it was slaughtered two hours after the third dose, at the peak plasma level. The urine samples were collected 8 and 24 hours and the faeces 24 hours after each dose (i.e. immediately before the next dose), and the radioactivity was determined. Samples of liver, kidneys, muscle (loin, round, flank) and fat (perirenal, omental, subcutaneous) were assayed for total radioactivity and the remaining samples minced and stored at about -20°C. After extraction the parent compound and the metabolites were isolated and purified by various HPLC methods. Identification was by TLC chromatographic comparison with authentic reference compounds in at least four different solvent systems or by ¹H-NMR and mass spectrometry.

The radioactivity in the plasma reached the highest concentration 2 hours after the first dose with the imidacloprid equivalent concentration of 3.98 μ g/ml, corresponding to about 40% of the equidistribution concentration in the body. The half-life in the plasma was about 4.8 hours 2 to 24 hours after the first dose. About 40% of the administered dose was excreted renally, about one third within 8 hours after both the first and the second doses, and about 10% in the faeces. Only 0.23% of the total dose was in the milk. When the goat was slaughtered 50 hours after first dose, the total residue in the edible organs was estimated to account for 5.5% (Table 2).

Table 2. Distribution of the total radioactivity in the urine, faeces and milk of a lactating goat and recovery of radioactivity after repeated oral doses of 10 mg/kg bw per day (Karl *et al.*, 1991).

Sample	Time after 1st dose (h)	No. of doses	% of total administered radioactivity
Urine (incl. cage rinse)	0	1	-
	8		11.84
	24	2	4.64
	32		14.74

Sample	Time after 1st dose (h)	No. of doses	% of total administered radioactivity
	48	3	8.50
	50	(slaughter)	-
Subtotal			39.72
Faeces	0	1	-
	24	2	2.85
	48	3	6.77
	50	(slaughter)	-
Subtotal			9.62
Milk	0	1	-
No. 1	8		0.078
No. 2	24	2	0.009
No. 3	32		0.079
No. 4	48	3	0.014
No. 5	50	(slaughter)	0.045
Milk	8-50	1-3	0.225
Total excreted radioactivity			49.57
Estimated total residue in edible tissues			5.52
Recovery			55.09

Concentrations of ^{14}C in the milk eight hours after the first and second doses were comparable, and after a further 16 hours were reduced by comparable factors of 12 and 11 respectively. The highest equivalent concentration of 4.1 $\mu\text{g/g}$ was determined 2 hours after the third dose. The results are shown in Table 3.

Table 3. Radioactivity in the milk of a lactating goat after repeated oral doses of 10 mg/kg bw (Karl *et al.*, 1991).

Time after 1st dose (h)	No. of doses	Sample wt. (g)	% of total dose	Concentration as imidacloprid ($\mu\text{g/g}$)
0	1	-	-	-
8		355	0.078	2.09
24 (BA)		475	0.009	0.17
24	2	-	-	-
32		284	0.079	2.62
48 (BA)		565	0.014	0.24
48	3	-	-	-
50		103	0.045	4.1
Total			0.225	

BA immediately before administration

The equivalent concentrations in the three types of muscle ranged from 3.8 to 3.96 $\mu\text{g/g}$, corresponding to 3.45% of the total radioactivity administered, assuming that the muscles accounted for 30% of body weight, and in the fat the mean value was 2.4 $\mu\text{g/g}$, 0.73% of the total administered radioactivity assuming fat accounted for 12% of body weight. The highest equivalent concentration was 15.9 $\mu\text{g/g}$ in the liver followed by 11.6 $\mu\text{g/g}$ in the kidneys. This result reflects the significance of these organs for the metabolism and excretion of the test compound and its labelled biotransformation products. The detailed results are shown in Table 4.

Table 4. Residue levels of ^{14}C as imidacloprid in the edible tissues and organs of a lactating goat after repeated oral doses (3 x 10 mg/kg bw) (Karl *et al.*, 1991).

Sample	Fresh weight (g)	Equivalent concentration ($\mu\text{g/g}$ as imidacloprid)	% of total administered radioactivity
Muscle (round)	4.13 ²	3.96	
Muscle (flank)	3.69 ²	3.82	
Muscle (loin)	3.24 ²	3.8	
Composite muscle	8442	3.86 ³	3.45

Sample	Fresh weight (g)	Equivalent concentration ($\mu\text{g/g}$ as imidacloprid)	% of total administered radioactivity
Fat (perirenal)	0.51 ²	1.81	
Fat (subcutaneous)	0.50 ²	2.1	
Fat (omental)	0.61 ²	2.1	
Composite fat ¹	3377	2.4 ³	0.73
Liver	725.8	15.92	1.22
Kidney	94.8	11.59	0.12

¹ Total weight of muscle and fat were calculated from the body weight assuming 30% muscle and 12% fat to be typical. Weight at slaughter was 28.1 kg.

² Mean weight of three samples.

³ Mean concentration in the three types of muscle or fat.

The radioactive residues were extracted from the milk with acetonitrile, or methanol and acetonitrile. In highly radioactive samples, after separating the fat and the coagulated protein the extraction of the remaining aqueous whey with acetonitrile yielded 85 to 92% of the initial radioactivity, indicating the lack of protein-bound residues. In samples with low levels of radioactivity the whey contained 68 and 72% of the initial radioactivity. The radioactivity in the kidney, liver, muscle and fat is shown in Table 5.

Table 5. Radioactivity in the tissues and organs of a goat (Karl *et al.*, 1991).

Fraction	¹⁴ C, % of total applied radioactivity							
	Kidney	Liver	Muscle			Fat		
			round	flank	loin	perirenal	omental	subcutaneous
Extractable with organic solvents	95.6	94.01	79.7	84.3	89.4	95.3	91.5	96.7
Unextractable with water			13.0	8.4	9.0			
Solids after extraction with acetonitrile	9.7	6.54	3.2	7.5	2.1	11.2	8.4	8.5
Total	105.3	100.55	95.9	100.2	100.5	106.5	99.9	105.1

The following metabolites were identified:

6-chloronicotinic acid	M14	6-CNA
WAK 3583	M15	6-CNA-glycine
NTN 33823/NTN38014	M09	denitro compound
NTN 35884	M06	olefin
WAK 4103	M01	5-hydroxy compound
WAK 5839	M02	4-hydroxy compound
WAK 3839	M07	nitrosimine
NTN 33519	M12	2-ketone
DIJ 9646-2	M22	PEDA
WAK 4126	M10	guanidine compound

In milk samples with high amounts of radioactivity (8 hours after the 1st and 2nd doses and 2 hours after the 3rd dose) about 80% of the TRR was identified (Table 6).

Table 6. Residues of [methylene-¹⁴C]imidacloprid in the milk of a lactating goat after oral doses of 10 mg ai/kg bw (Karl *et al.*, 1991).

Dose no.	1				2				3		mean ²
Time after 1st dose	8 h		24 h (BA)		32 h		48 h (BA)		50 h ¹		pooled milk
	µg/g	% of TRR	µg/g	% of TRR	µg/g	% of TRR	µg/g	% of TRR	µg/g	% of TRR	µg/g
Total ¹⁴ C	2.09	100	0.17	100	2.62	100	0.24	100	4.10	100	
Imidacloprid	0.93	44.6	0.015	8.8	1.08	41.3	0.03	12.5	2.27	55.3	0.50
M01 WAK 4103	0.19	9.0	0.0047	2.7	0.26	9.8	0.010	4.4	0.29	7.0	0.10
M02 WAK 5839	0.24	11.3	0.0056	3.2	0.27	10.2	0.0086	3.6	0.40	9.7	0.12
M06 NTN 35884	0.20	9.5	0.0088	5.1	0.23	8.9	0.014	5.8	0.23	5.6	0.097
M07 WAK 3839	0.073	3.5	0.005	0.3	0.058	2.2	0.0014	0.6	0.012	0.3	0.025
M15 WAK 3583	0.066	3.2	0.011	6.6	0.13	4.8	-	-	0.13	3.1	0.044
Total identified	1.70	81.1	0.046	26.7	2.03	77.2	0.064	26.9	3.33	81.0	0.89

BA: immediately before administration

µg/g: as imidacloprid

¹ at slaughter, 2 h after 3rd administration

² weighted average (calculated)

The main component in the milk was unchanged imidacloprid, accounting for 41 to 55% of the TRR. The monohydroxylated metabolites WAK 4103 and WAK 5839 [M01, M02] and the olefin M06 were minor components. Because of the low level of radioactivity and the complexity of the HPLC chromatograms only 27% of the TRR was identified in milk samples 24 hours after the 1st and 2nd doses which contained low levels of imidacloprid, WAK 5839 [M02], WAK 4103 [M01], NTN 35884 [M06], WAK 3583 [M15] and WAK 3839 [M07].

In kidneys only 37.7% of the TRR was identified. The metabolite WAK 3583 [M15] accounted for about 13% of the TRR. Imidacloprid was of minor importance with about 6%. The minor metabolites 5-hydroxy-imidacloprid [M01], 4-hydroxy-imidacloprid WAK 5839 [M02], the olefin compound NTN 35884 [M06] and the glucuronide of WAK 4103 [M04] were identified, and traces of WAK 3839 [M07].

In liver only 14.38% of the TRR was identified, partly because a large number of other compounds were coextracted with the radioactivity and would probably have reacted with a proportion of the reactive imino- and guanidino-type metabolites. The main metabolites identified were the imino compounds WAK 4126 [M10] and/or NTN 38014 [M09]. The parent compound was a minor component (0.13%) as were WAK 3583 [M15] (1.8%), 6-cloronicotinic acid [M14] (1.5%), the ethylenediamine DIJ 9646-2 [M22] (0.2%) and the urea NTN 33519 [M12] (0.04%). The compounds found in kidney and liver are shown in Table 7.

Table 7. Imidacloprid and its metabolites in the kidney and liver of a lactating goat after repeated oral doses of 10 mg ai/kg bw (Karl *et al.*, 1991).

Compound		Kidney		Liver	
		% of TRR	µg/g ¹	% of TRR	µg/g ¹
Imidacloprid		5.9	0.68	0.79	0.13
M01	WAK 4103	5.6	0.65		
M02	WAK 5839	2.9	0.34		
M04	WAK 4103 glucuronide	5.7	0.66		
M06	NTN 35884	4.3	0.49		
M07	WAK 3839	0.1	0.01		
M09/M10	NTN38014/WAK 4126			10.03	1.59
M12	NTN 33519			0.04	0.01
M14	6-CNA			1.53	0.25
M15	WAK 3583	13.2	1.53	1.78	0.29
M22	DIJ 9646-2			0.21	0.04

Compound	Kidney		Liver	
	% of TRR	µg/g ¹	% of TRR	µg/g ¹
Total, identified	37.7	4.36	14.38	2.31

¹ as imidacloprid

The metabolites in the three muscle types, round, flank and loin, were purified and identified separately. About 78 to 87% of the TRR was identified. Imidacloprid accounted for 64 to 69% and minor metabolites were 5-hydroxy-imidacloprid [M01], 4-hydroxy-imidacloprid [M02] and the olefinic compound NTN 35884 [M06], plus traces of WAK 3839 [M07]. Metabolism in the three muscle types was evidently the same.

The extent of the identification was similar in the perirenal, omental and subcutaneous fat (87 to 91%). Imidacloprid was the main constituent, accounting for 63 to 73.5% of the TRR. The same minor metabolites as in muscle were identified, and traces of WAK 3839 [M07] were detected in perirenal and subcutaneous fat. The results are shown in Table 8.

Table 8. Imidacloprid and its metabolites in the muscle and fat of a lactating goat after repeated oral doses of 10 mg ai/kg bw (Karl *et al.*, 1991).

Compound		Muscle						Fat					
		round		flank		loin		perirenal		omental		subcutaneous	
		% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹
Imidacloprid		64.0	2.54	64.5	2.47	68.9	2.65	67.6	1.22	63.4	1.40	73.5	1.54
M01	WAK 4103	3.4	0.13	3.5	0.13	3.2	0.12	3.5	0.07	4.2	0.09	3.1	0.07
M02	WAK 5839	5.7	0.23	5.8	0.22	7.1	0.27	7.0	0.13	8.2	0.18	5.8	0.12
M06	NTN 35884	4.9	0.19	5.6	0.21	6.1	0.23	7.6	0.14	10.09	0.22	7.9	0.17
M07	WAK 3839	0.25	0.01	0.75	0.03	0.6	0.02	1.0	0.02	-	-	0.6	0.01
Total identified		78.25	3.10	80.15	3.06	86.85	3.29	86.7	1.58	85.8	1.89	90.9	1.91

¹ as imidacloprid

In the study by Klein (1992) a lactating goat (*capra hircus*) weighing 41 kg was given a target dose of 10 mg/kg body weight orally by intubation of [methylene-¹⁴C]imidacloprid three times on 3 consecutive days. 1 part of the labelled compound was diluted with 7 parts of unlabelled compound by weighing (specific radioactivity 14 µCi/mg, 1231 mg of compound, mean dose of 410 mg per day or 10.01 mg/kg bw/day). Assuming a maximum daily feed consumption of 5% bw, this dose is equivalent to the exaggerated amount of 200 ppm in the diet. The goat was milked in the morning immediately before each dose, again 8 hours later, and immediately before slaughter. The milk volumes were recorded, two aliquots were taken from each fraction and measured by liquid scintillation counting. The remaining milk was stored at about -20°C for analysis. Urine samples were collected 8 and 24 hours and faeces 24 hours after each dose, i.e. immediately before the next dose. Some samples were prepared for liquid scintillation counting, and the remainder stored for analysis. The following tissues and organs were dissected: liver without gall-bladder, kidneys, muscle (loin, round, flank), fat (perirenal, omental, subcutaneous).

The metabolites in the liver and kidneys were extracted from these organs with water plus 1% NaCl and 1-2% NaCl in 0.1 M NaOH, then with acetonitrile/methanol mixtures. The kidney extract was purified by partition with methanol and hexane. Identification was by comparative HPLC with authentic reference compounds in at least two independent chromatographic systems. Small amounts of liver and kidney extracts were used for characterization of the residues by oxidation to 6-chloronicotinic acid with KMnO₄.

The results are shown in Table 9. About 58% of the total radioactivity administered was excreted, 46% of which was in the urine and 12% in the faeces. 0.4% was secreted in the milk. At slaughter, 2 hours after the last dose, the total residue in the edible tissues was about 5%, so total

recoveries were about 63%. Because the goat was slaughtered only two hours after the last dose it is understandable that the remaining 40% or so had not been eliminated, and recoveries indicate that most of the final dose remained in the digestive tract.

Table 9. Percentages of the total administered radioactivity in the urine, faeces and milk of a dairy goat after repeated oral doses (3 x 10 mg/kg bw) of [^{14}C]imidacloprid (Klein, 1992).

Sample	Time after 1st dose (h)	Dose no.	% of total dose
Urine (incl. cage rinse)	0	1	-
	24	2	19.76
	48	3	21.19
	50	(slaughter)	5.08
Subtotal			46.03
Faeces	0	1	-
	24	2	2.39
	48	3	6.65
	50	(slaughter)	2.53
Subtotal			11.57
Milk	0	1	-
	8		0.131
	24	2	0.022
	32		0.187
	48	3	0.026
	50	(slaughter)	0.046
Subtotal			0.41
Total excreted radioactivity			58.01
Estimated total residue in edible tissues			5.27
Recovery			63.28

Absorption was immediate, distribution and elimination rapid. Comparable equivalent concentrations were measured in milk eight hours after the first and second doses. Within 16 hours of the first and second doses, concentrations in the milk had decreased by factors of 16 and 14 respectively. The highest equivalent concentration of 3.65 $\mu\text{g/g}$ was determined 2 hours after the third dose. There was no accumulation of radioactivity observable in the milk throughout the trial period (Table 10).

Table 10. Radioactivity in the milk of a lactating goat after oral administration of 3 x 10 mg/kg bw (Klein, 1992).

Time after 1st dose (h)	No. of doses	Milk, (g)	^{14}C as imidacloprid ($\mu\text{g/g}$)	% of total dose
0	1	-	-	-
8		512	3.16	0.131
24 (BA)		1458	0.19	0.022
24	2	-	-	-
32		834	2.77	0.187
48 (BA)		1627	0.20	0.026
48	3		-	-
50		(slaughter)	3.65	0.046
Total				0.413

BA: immediately before administration

Radioactivity levels in the tissues and organs, and respective weights are shown in Table 11. The TRR in muscles accounted for 3.65% of the administered radioactivity, assuming muscles were 30% of the body weight. Lower quantities were determined in the fat. The mean value was 1.07 $\mu\text{g/g}$, 0.73% of the total administered radioactivity. The highest concentration, 17.12 $\mu\text{g/g}$, was in the liver, and the next was 13.54 $\mu\text{g/g}$ in the kidneys, reflecting the importance of these organs for metabolism and excretion.

Table 11. Residue levels of imidacloprid in the edible tissues and organs of a lactating goat after oral administration of 3 x 10 mg/kg bw (Klein, 1992).

Sample	Fresh weight (kg)	% of total radioactivity administered	¹⁴ C as imidacloprid (µg/g)
Liver	0.936	1.30	17.12
Kidney	0.120	0.13	13.54
Muscle (round)	2.555	-	3.33
Muscle (flank)	0.682	-	3.62
Muscle (loin)	0.184	-	3.68
Total¹	11.58	3.44	3.65 ²
Fat (perirenal)	0.225	-	0.92
Fat (subcutaneous)	0.220	-	1.19
Fat (omental)	0.629	-	0.94
Total¹	4.632	0.40	1.07 ²

¹ Total weights based on 30% for muscle and 12% for fat for a typical goat. This goat's slaughtered weight was 38.6 kg.

² Concentrations in the three types of muscle and fat combined in ratio 1:1:1.

Besides the unchanged parent compound the following metabolites were identified together with all the metabolites identified by Karl *et al.* (1991) except M07 and M22.

WAK 3772	M03	dihydroxy
WAK 5839 glucuronide	M05	4-hydroxy glucuronide
WAK 3877/4	M08	amino compound
WAK 4230	M11	nitroguanidine
DIJ 10739	M13	urea compound
GSE 1478	M16	AMCP
WAK 5031	M17	dihydroxyimine
GSE 2712	M40	formyl-AMCP

Extracts of liver and kidney yielded over 99% of the recovered radioactivity, 67.8% of the TRR in the liver and 77.9% in the kidney contained the 6-chloronicotinic acid moiety. In liver 33.9% of the organ radioactivity was identified. The two guanidine compounds M09 and M10 were the main metabolites, accounting for 16.4% and 7.2% respectively. In kidney 71.6% of the total radioactive residue was identified. The 5 main metabolites were the olefin M06 (17.7%), the glycine conjugate M15 of 6-chloronicotinic acid (16.8%), and the glucuronides M04 and M05 of the 4- and 5-monohydroxylated metabolites (14.06%). Other compounds were the guanidine metabolites M09 and M10, accounting for 5.9% and 4.2% respectively, as well as imidacloprid (6.2%). All other metabolites except M16 accounted for less than 1%. The detailed results are shown in Table 12.

Table 12. Residues of imidacloprid and its metabolites in the kidney and liver of a lactating goat after repeated oral doses of 10 mg ai/kg bw (Klein, 1992).

Compound		Kidney		Liver	
		% of TRR	µg/g ¹	% of TRR	µg/g ¹
TRR			13.5		17.11
Imidacloprid		6.19	0.84		
M01	WAK 4103	1.96	0.265		
M02	WAK 5839				
M03	WAK 3772				
M04	WAK 4103 glucuronide	14.06	1.90		
M05	WAK 5839 glucuronide				
M06	NTN 35884	17.71	2.40	3.17	0.54
M08	WAK 3877/4			1.52	0.26
M09	NTN 38014	5.86	0.79	16.39	2.80
M10	WAK 4126	4.19	0.57	7.23	1.24
M11	WAK 4230	0.81	0.11	0.35	0.06

Compound		Kidney		Liver	
		% of TRR	µg/g ¹	% of TRR	µg/g ¹
M12	NTN 33519	0.73	0.10	1.96	0.34
M13	DIJ 10739	0.19	0.026	1.26	0.22
M14	6-CNA	0.32	0.043		
M15	WAK 3583	16.78	2.27	0.96	0.16
M16	GSE 1478	1.84	0.25	0.43	0.07
M17	WAK 5031	0.61	0.083	0.60	0.10
M40	GSE 2712	0.37	0.05		
Total identified		71.62	9.70	33.87	5.8
Residues containing 6-CNA		77.92	10.55	68.67	11.75

¹ as imidacloprid

Summary

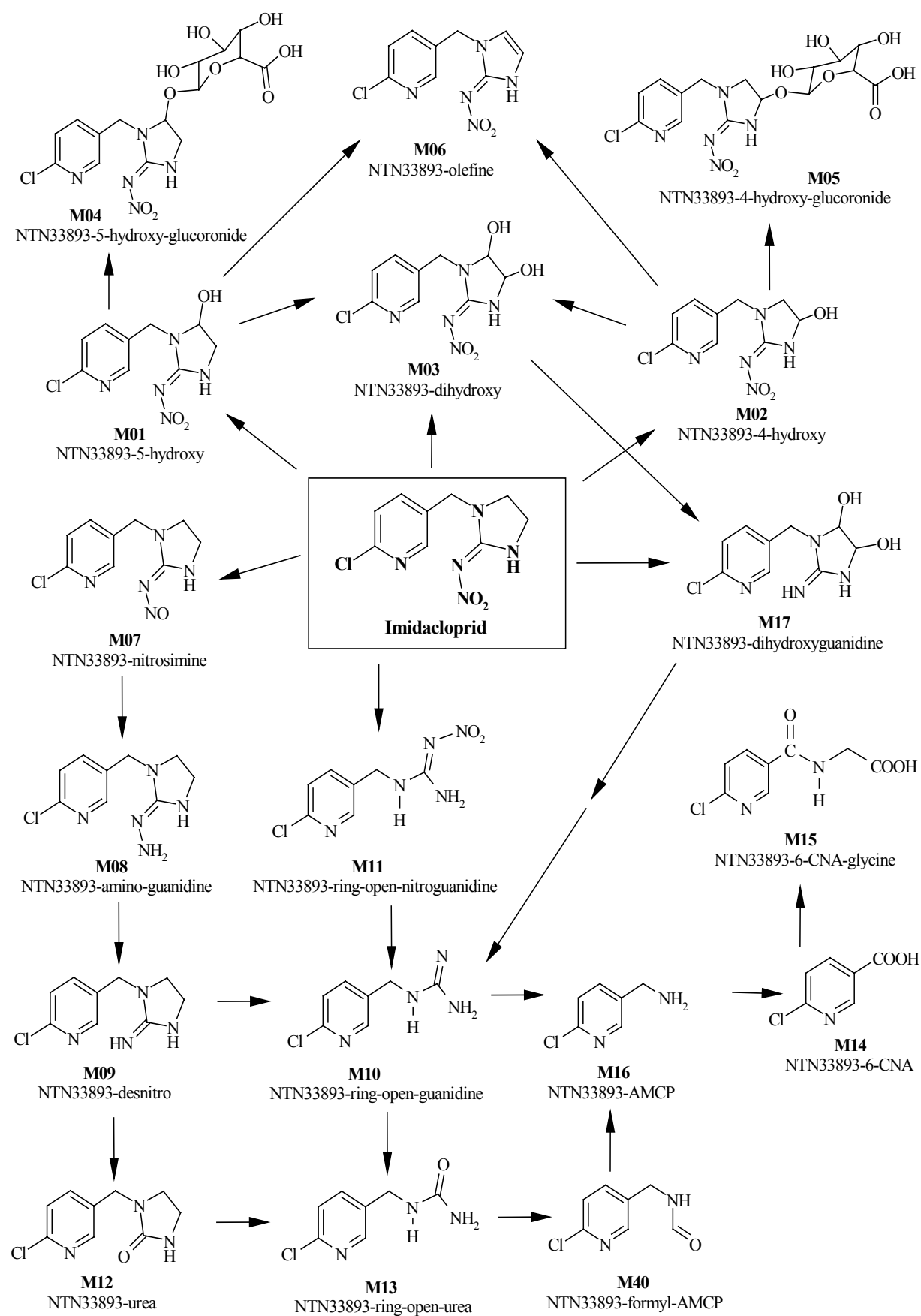
The absorption, distribution and elimination of imidacloprid was rapid in the lactating goat. Within 50 hours of the first dose of [¹⁴C]imidacloprid about 54% of the total administered radioactivity had been excreted, mainly in the urine (about 43%) with about 11% in the faeces. About 0.3% of the total dose was secreted in the milk. At slaughter, 2 hours after the last dose, the total residue in the edible organs was about 5%, and the mean total recovery in the two studies about 60%.

The residues in kidney and liver were mainly metabolites but in muscle and fat about 65% of the residue was imidacloprid. There were three main degradation routes.

- Hydroxylation of the imidazolidine ring of imidacloprid to form 4-hydroxy and 5-hydroxy imidacloprid [M01, M02] plus their glucuronide conjugates [M04, M05], and dihydroxy imidacloprid [M03] followed by the loss of water from M01 and M02 to form the olefin metabolite [M06];
- Reduction and loss of the nitro group on the imidazolidine ring and subsequent oxidation to form the amino compound [M08], then the denitro compound [M09] and finally the 2-ketone [M12];
- Opening of the imidazolidine ring by removal of the ethylene bridge and subsequent oxidation. The first step is the nitroguanidine [M11] followed by the guanidine compound [M10] which can also be formed from both the metabolite M09 and its dihydroxy analogue M17. This metabolite M10 can form the corresponding urea M13 and amine M16 with further degradation to 6-chloronicotinic acid [M14] and its conjugate with glycine [M15].

The proposed metabolic pathways of imidacloprid in the lactating goat are shown in Figure 1.

Figure 1. Proposed metabolic pathways of imidacloprid in the lactating goat.



Laying hens. Klein and Brauner (1990) studied the metabolism of methylene- ^{14}C imidacloprid in four groups of White Leghorn hens (*Gallus domesticus*) dosed orally at 10.6 mg/kg bw/100 ppm in the diet (assuming maximum daily feed consumption of 10% bw) for three consecutive days at 24 h intervals.

Test group no.	No. of animals	Dilution with unlabelled compound	Mean ^{14}C dose per animal (μCi)	Mean imidacloprid dose (mg/kg bw)
1	5	-	-	-
2	5	1:50	252.10	52.9
3	5	1:10	261.59	10.6
4	3	1:10	261.59	10.6

Micro-samples of blood (about 60 μl) were taken from the wing veins of the hens 0.25, 0.5, 1, 2, 3, 4, 6, and 24 h after the third dose from group 2. The eggs of groups 2 and 3 were collected twice daily, once in the morning before dosing and 8 hours after dosing, and eggs were also taken from the oviduct at slaughter, which was 2 hours after the last dose. In group 4 eggs were not taken from the oviduct. Excreta were individually collected at intervals of 24 hours, immediately before each dose. The kidney, liver, heart, gizzard, skin with subcutaneous fat, breast and thigh muscles, and subcutaneous fat of groups 1 and 3 were radioassayed. Extracts were partitioned, fractionated and analysed by HPLC, with identification by ^1H -NMR and mass spectrometry.

Radioactivity in the plasma reached a maximum 2 h after the third oral dose, equivalent concentration 4.9 $\mu\text{g/ml}$ corresponding to about half the equidistribution concentration in the body, demonstrating rapid distribution from the plasma into the peripheral organs and tissues. The half-life of ^{14}C in the plasma was about 14 h during 6 to 24 hours after the last dose, indicating a mean residence time (MRT) of 21.5 h. At the end of the test, 24 h after the third dose, concentration in the plasma had decreased to 2.09 $\mu\text{g/ml}$.

The results are shown Table 13. Until slaughter, 50 hours after the first dose, a mean of 32.9% of the total ^{14}C administered was excreted. About half of the radioactivity administered with the first dose was eliminated within 24 hours. Only 0.06% of the total dose was found in the eggs. At slaughter 2h after the last dose total residues in the organs and tissues were estimated to be 3.4% of the total dose.

Table 13. Percentages of the total radioactivity in the excreta and eggs of hens (Klein and Brauner, 1990).

Sample	Time after 1st dose, h	Mean % of radioactivity in the birds
Excreta	24	47.3 ± 2.3^1
	50	21.3 ± 0.8^2
Subtotal		32.9 ± 0.08^3
Eggs, including those in oviduct	0-50	0.062 ± 0.008^3
Calculated residues in the tissues	50	3.39 ± 1.1^3

¹ % of radioactivity in first dose.

² % of radioactivity in 2nd and 3rd doses incl. unextracted radioactivity from first dose.

³ % of total administered radioactivity.

The residues in the edible tissues and organs at maximum plasma concentration are shown in Table 14.

Table 14. ^{14}C in the tissues and organs of laying hens at slaughter, 50 h after the first dose (Klein and Brauner, 1990).

Sample	^{14}C as imidacloprid, mean $\pm$ SD ¹ ($\mu\text{g/g}$)
Liver	8.16 $\pm$ 1.17
Kidney	11.52 $\pm$ 2.7
Heart	3.18 $\pm$ 1.58
Gizzard without lining and contents	6.49 $\pm$ 3.63
Muscle, thigh	1.48 $\pm$ 0.297
Muscle, breast	2.35 $\pm$ 1.71
Fat, subcutaneous	0.455 $\pm$ 0.15
Skin without fat	1.25 $\pm$ 0.31

¹ mean $\pm$ standard deviation

Identification of radioactive residues by ^1H -NMR and mass spectrometry was not always possible because of the extremely low residues, however an average of 37.1% of the TRR was identified. The results are shown in Table 15.

Table 15. Residues of imidacloprid and its metabolites in the organs, tissues and eggs of laying hens 50 h after three oral doses 10 mg ai/kg bw (Klein and Brauner, 1990).

Compound	Eggs $\mu\text{g/g}^1$	Kidney $\mu\text{g/g}^1$	Breast $\mu\text{g/g}^1$	Heart $\mu\text{g/g}^1$	Gizzard $\mu\text{g/g}^1$	Skin $\mu\text{g/g}^1$	Thigh muscle $\mu\text{g/g}^1$	Fat $\mu\text{g/g}^1$
Imidacloprid	-	-	1.07	0.88	3.43	0.09	0.08	0.49
M06 NTN 35884	0.22	0.69	-	0.64	-	0.35	0.43	-
M09 NTN 38014	-	0.41	-	-	-	-	-	-

¹ as imidacloprid

In a complementary study by Klein and Brauner (1992) using the same test conditions five White Leghorn laying hens were dosed orally three times at intervals of 24h with [methylene- ^{14}C]imidacloprid diluted with the unlabelled compound at a ratio of 1:10. Calibration of this suspension showed an average value of 181.94 μCi , mean dose 11.7 mg/kg bw. Assuming a daily feed consumption of 6.4% bw per day, doses of 10 mg/kg bw were equivalent to 156 ppm in the diet (fresh weight). Eggs were collected twice daily, in the morning before the daily dose and 8 hours after dosing, and the shells discarded. Excreta were individually collected immediately before each dose. Extraction and identification were as before.

The results are shown in Table 16. About 50% of the radioactivity administered was excreted within 24h after the first and the second doses. 0.09% of the total dose was found in the eggs 24 hours after the first dose. At slaughter, 2h after the last dose, total residues in the organs and tissues were about 8% of the radioactivity present in the body at that time (about 0.6% in the skin, 0.9% in fat and 4.5% in muscles, on the basis that skin, fat and muscle account for 4%, 12% and 40% bw respectively).

Table 16. Recovery of radioactivity after three oral doses of 10 mg/kg per day to laying hens (Klein and Brauner, 1992).

Sample	Time after the 1st application hours	Mean values in % of the radioactivity (arithmetic mean $\pm$ standard deviation)
Excreta	24	51.4 $\pm$ 14.9 ¹
	48	47.0 $\pm$ 12.1 ²
	50	5.5 $\pm$ 1.7 ³
Eggs	24	0.087 ¹
	48	0.184 ²
Calculated residues in tissues		7.81 $\pm$ 1.09 ³

¹ % of radioactivity in 1st dose.

² % of radioactivity in 1st, 2nd and 3rd doses including unextracted radioactivity from 1st dose.

³ % of totally administered radioactivity in the body at slaughter.

The ¹⁴C concentration in eggs was low and ranged from 0.043 µg/g as imidacloprid 8 h to 0.803 µg/g 32 h after the first dose. The highest concentrations were in the kidneys and liver (18.9 and 12.75 µg/g respectively), with decreasing concentrations in the skin, gizzard, muscle and subcutaneous fat respectively. The results confirmed the first part of this study and are shown in Table 17.

Table 17. ¹⁴C concentrations in the tissues and organs of laying hens at slaughter, 50 h after the first dose (Klein and Brauner, 1992).

Sample	¹⁴ C, mean (µg/g as imidacloprid) ± CV (%) ¹
Liver	12.75 ± 17.6
Kidney	18.88 ± 18.5
Gizzard	2.36 ± 34.8
Muscle, thigh	2.3 ± 12.8
Muscle, chest	2.1 ± 13.1
Muscle, composite	2.2 ± 11.8
Fat, subcutaneous	1.51 ± 40.3
Skin without fat	2.93 ± 8.1

¹ coefficient of variation

The distribution of radioactivity in the composite egg, tissue and organ samples used for metabolite identification are shown in Table 18.

Table 18. Distribution of radioactivity in eggs and edible tissues (Klein and Brauner, 1992).

Sample	µg parent equivalents/g
Egg	0.49
Liver	12.5
Composite muscle	2.2
Composite fat	1.55

Identification of the residues averaged 67.5% in all samples. The distribution of imidacloprid and metabolites in the eggs and edible tissues is shown in Table 19.

Table 19. Residues of [methylene-¹⁴C]imidacloprid in the eggs, organs and tissues of laying hens 50 h after three oral doses of 10 mg ai/kg bw (Klein and Brauner, 1992).

Parent compound/metabolite		Eggs		Liver		Muscle		Fat	
		µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR
Imidacloprid		0.023	4.83	-	-	0.138	6.26	0.191	12.35
M01	WAK 4103	0.049	10.05			0.190	8.61	0.150	9.68
M02	WAK 5839	0.028	5.7	1.065 ²	8.51 ²	0.102	4.62	0.036	2.34
M03	WAK 3772	0.002	0.47			-	-	-	-
M06	NTN 35884	0.140	28.69	1.914	15.30	0.589	26.74	0.350	22.55
M10	WAK 4126	0.019	3.96	1.994	15.94	0.136	6.16	0.065	4.22
M11	WAK 4230	0.087	17.88	1.123	8.98	0.148	6.71	0.079	5.11
M13	DIJ 10739	0.009	1.81	0.970	7.75	0.081	3.67	0.021	1.38
M14	6-CNA	-	-	0.309	2.47	-	-	0.029	1.86
M16	GSE 1478	0.019	3.90	0.244	1.95	0.079	3.6	0.023	1.49

Parent compound/metabolite		Eggs		Liver		Muscle		Fat	
		µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR	µg/g ¹	% of TRR
M17	WAK 5031	0.004	0.82	0.274	2.19	0.03	1.36	-	-
Total identified		0.38	78.11	7.893	63.09	1.493	67.73	0.944	60.98

¹ As imidacloprid.

² Sum of M01, M02 and M03.

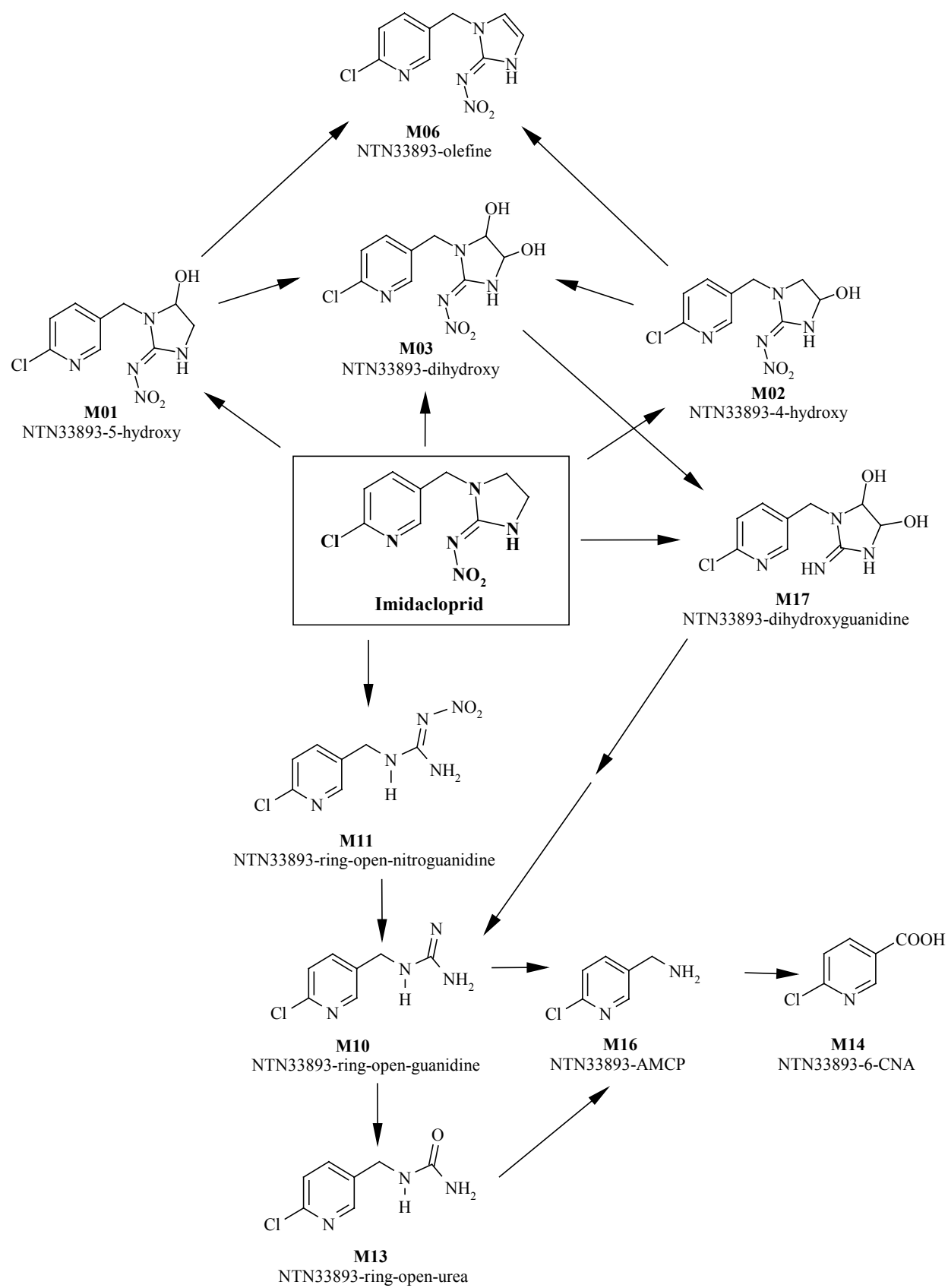
Summary

The metabolism of imidacloprid in hens followed three routes.

- Hydroxylation of the imidazolidine ring to form 4- and 5-hydroxy imidacloprid [M02, M01], followed by loss of water to yield the olefinic compound M06. These three compounds accounted for about 25-38% of the identified radioactivity.
- Loss of the nitro group from dihydroxy-imidacloprid [M03] to yield M17.
- Opening of the imidazolidine ring with loss of the ethylene group and subsequent oxidation. The first step is formation of the nitroguanidine M11 followed by the guanidine M10, which can also be formed from the dihydroxyguanidine-type metabolite M17. M10 can form the urea M13 and amine M16, which is oxidised to 6-chloronicotinic acid [M14].

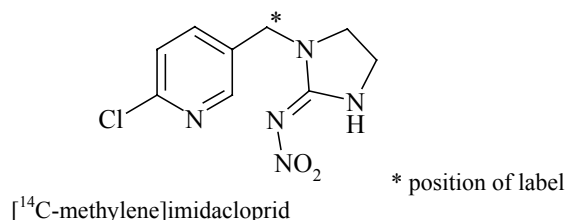
Proposed metabolic pathways of imidacloprid in laying hens are shown in Figure 2.

Figure 2. Proposed pathways of imidacloprid in laying hens.



Plant metabolism

The metabolism of imidacloprid was investigated in tomatoes, apples and potatoes after spray applications, in egg plants, potatoes and rice after granular soil application, in maize and cotton after seed treatment, in rice after nursery box treatment and in tobacco after combined spray and granular applications using [^{14}C -methylene]imidacloprid. The labelling position is shown below. Unless otherwise stated, studies were carried out according to GLP.



Spray application

Tomatoes. Immature fruits of two greenhouse plants were sprayed with 8 ml of 0.2% solution of a 25 WP formulation (Draeger *et al.*, 1989; non-GLP study) corresponding approximately to 0.25 kg ai/ha, 600 l water/ha, a recommended commercial rate, 14 days before harvest. Four of the ripest fruits were picked 4, 7 and 14 days after spraying as well as a post-harvest sample at 21 days. The tomatoes were surface-washed with methanol and extracted by successive maceration in methanol/water, methanol and dichloromethane. In an additional experiment the leaves of 2 plants were sprayed with a 0.12 or 0.15% solution of the 25 WP formulation containing [^{14}C -methylene]imidacloprid, avoiding any contamination of immature fruits. 14 days later mature fruits were harvested and the ^{14}C determined.

The TRR decreased from 1.01 mg/kg 4 days after spraying to 0.84 and 0.85 mg/kg at days 7 and 14 to 0.64 mg/kg in the post-harvest sample at day 21. Lower residues in the later samples may be attributed to growth dilution rather than to degradation. Most of the TRR could be washed off the surfaces by methanol. At harvest, day 14, this still accounted for 76%. However, once radioactivity penetrated the fruit it increased steadily, reaching 40% at day 21, almost all of which was extractable, while only 0.2 to 0.8% (up to 0.005 mg/kg as imidacloprid) remained unextracted in the solids and was not further investigated (Table 20).

Table 20. Distribution of imidacloprid and its metabolites in tomatoes after spray applications of [^{14}C -methyl]imidacloprid (Draeger *et al.*, 1989).

Days after last spray	Radioactive residues, % of TRR			
	4 days	7 days	14 days	21 days
Surface rinse (total)	88.2	77.3	76.4	60.4
Imidacloprid	84.4	71.8	69.7	52.2
Identified metabolites	2.7	3.8	5.1	6.0
Others	1.1	1.7	1.6	2.1
Extractable residues (total)	11.6	22.2	23.2	38.9
Imidacloprid	10.0	18.8	18.3	27.2
Identified metabolites	0.8	2.4	3.4	8.3
Others	0.8	1.0	1.5	3.4
Unextractable residues	0.2	0.5	0.4	0.8

Most of the radioactivity in the surface wash solutions and tomato extracts was unchanged parent compound (Table 21). M01 (0.027 mg/kg) and M09 (0.031 mg/kg) were detected in significant quantities of up to 4.2 and 4.8% of the TRR 21 days after application. Three other metabolites accounted for 1-2% of the TRR: the olefin M06 for 1.1% (0.007 mg/kg) at day 21, the conjugate CHMP gentiobioside M30 for 1.7% (0.011 mg/kg) at day 21 and the urea M12 for 1.9% (0.016 mg/kg) at day 14. In addition the nitrosimine M07 and CHMP glucoside M29 at day 21 accounted for

0.7% (0.004 mg/kg), and 0.3% (0.002 mg/kg). 93.7 to 98.0% of the recovered radioactivity was identified by TLC and $^1\text{H-NMR}$ and mass spectrometry.

The translocation experiment showed no significant transport of radioactivity from treated leaves to untreated fruits. 14 days after spraying the leaves, the recovered radioactivity in the tomatoes accounted for 0.003 and 0.006 mg/kg parent compound equivalents, markedly below the TRR of 0.85 mg/kg detected 14 days after direct application to tomatoes.

Table 21. Metabolism of [methylene- ^{14}C]imidacloprid in tomatoes after spraying at a rate of 0.25 kg ai/ha (Draeger *et al.*, 1989).

Days after last application	Radioactive residues, mg/kg ai equivalents							
	4 days		7 days		14 days		21 days	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	94.4	0.95	90.6	0.76	88.0	0.75	79.4	0.51
M01	1.1	0.011	1.8	0.015	1.8	0.015	4.2	0.027
M06	0.1	0.001	0.3	0.003	0.5	0.004	1.1	0.007
M07	0.3	0.003	0.6	0.005	0.7	0.006	0.7	0.004
M09	1.1	0.011	2.0	0.017	2.6	0.022	4.8	0.031
M12	0.9	0.009	1.0	0.008	1.9	0.016	1.5	0.01
M29	n.d.	n.d.	0.1	0.001	0.1	0.001	0.3	0.002
M30	0.1	0.001	0.3	0.003	0.8	0.007	1.7	0.011
Total identified	98.0	0.99	96.7	0.81	96.4	0.82	93.7	0.6
Others ¹	1.8	0.018	2.8	0.023	3.2	0.027	5.5	0.035
Unextractable residues	0.2	0.002	0.5	0.004	0.4	0.003	0.8	0.005
Total	100	1.01	100	0.84	100	0.85	100	0.64

n.d.: not detected

¹ TLC chromatograms show at least 3 different components.

Apples. A WP 25 formulation of [methylene- ^{14}C]imidacloprid was directly applied to 80 Golden Delicious apples on four trees cultivated in pots in a roofed vegetation area three times with about 100 μg ai at intervals of 28 days using an Eppendorf syringe with a tuft of hair attached to the tip (Vogeler *et al.*, 1992a). The applied amount of 0.3 mg ai/apple was approximately equivalent to the recommended field application rate of 375 g ai/ha. 10 apples were picked on day 0 and 70 apples 14 days after the last application. The apples were surface-washed with methanol and then peeled. Peel and pulp were homogenised separately with liquid nitrogen and stored at approximately -20°C . For extraction, peel and pulp samples were macerated with methanol/water and methanol.

To investigate the transference of imidacloprid from leaves to fruits after spraying, the nearest 5 leaves to each of two apples were treated three times with the test formulation described above at intervals of 28 days (total amount applied to each leaf 0.18 mg ai). Apples and treated leaves from both trials were separately collected 14 days after application and treated as described above.

The TRR in the apples accounted for 1.76 mg/kg parent compound equivalents at day 0 and 1.45 mg/kg at day 14. Most of the TRR (74.2%, 1.3 mg/kg at day 0; 64.9%, 0.94 mg/kg at day 14) was detected in the surface rinse, while 23.7% (0.42 mg/kg; day 0) and 32.1% (0.47 mg/kg; day 14) was detected in extracts of peel and pulp. Only 2.1% (0.036 mg/kg, day 0) and 3.0% (0.044 mg/kg; day 14) of the TRR remained unextracted in the solids and was not further investigated (Table 22).

Table 22. Distribution of imidacloprid and its metabolites as % of total radioactive residue in apples after spray applications of [methylene-¹⁴C]imidacloprid (Vogeler *et al.*, 1992a).

Days after final application	Radioactive residues, % of TRR	
	0 days	14 days
Surface rinse (total)	74.2	64.9
Imidacloprid	66.1	55.8
Metabolites ^{1,2}	7.3	8.3
Others	0.8	0.8
Peel + pulp (extractable) (total) ²	23.7	32.1
Imidacloprid	10.9	13.2
Metabolites ^{1,2}	12.4	18.5
Others	0.4	0.4
Unextractable residues	2.1	3.0

¹ Sum of identified and unidentified metabolites.

² Sum of single values.

The results of the metabolism study are shown in Table 23. 77% (1.36 mg/kg, day 0) and 69% (1.0 mg/kg, day 14) of the radioactivity in the surface wash and the extracts was unchanged parent compound. Seven significant metabolites were identified by TLC with reference substances. 26 other metabolites were at such low levels (maximum 0.01 mg/kg at day 0 and 0.013 mg/kg at day 14) that further identification was not attempted. The main metabolite was the olefin M06, accounting for 4.3% (0.077 mg/kg) at day 0 and 5.7% (0.082 mg/kg) at day 14. At day 14 the 5-hydroxy and dihydroxy metabolites M01 and M03 accounted for 2.7% (0.039 mg/kg) and 1.1% (0.016 mg/kg) respectively. Further metabolites were the denitro M09 (2.2%, 0.031 mg/kg), the 2-ketone M12 (1.7%, 0.024 mg/kg), and the nitrosimine M07 (0.7%, 0.001 mg/kg). The only metabolite formed by cleavage of the two ring systems of imidacloprid was CHMP glucoside, M29 (2.2%, 0.031 mg/kg at day 14). In total 85.3-90.1% of the TRR was identified by TLC and HPLC and ¹H-NMR and mass spectrometry.

Table 23. Metabolism of [methylene-¹⁴C]imidacloprid in apples after spraying at 375 g ai/ha (Vogeler *et al.*, 1992a).

Compound or fraction	Radioactive residues, mg/kg ai equivalents			
	0 days		14 days	
	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	77.0	1.36	69.0	1.0
M01	2.2	0.038	2.7	0.039
M03	0.9	0.014	1.1	0.016
M06	4.3	0.077	5.7	0.082
M07	0.6	0.011	0.7	0.010
M09	2.6	0.021	2.2	0.031
M12	1.3	0.024	1.7	0.024
M29	1.2	0.021	2.2	0.031
Total identified	90.1	1.57	85.3	1.23
Unknown (26 components) ¹	7.4	0.130	11.3	0.160
Others (residue on clean-up columns)	0.4	0.006	0.4	0.005
Unextractable	2.1	0.037	3.0	0.044
Total	100	1.74	100	1.44

¹ Maximum concentration of single components: 0.013 mg/kg.

Less than 0.1% of the radioactivity applied to the leaf surfaces translocated into the apple peel and pulp, so no significant residues in apples due to transport of the parent compound or metabolites from leaves to fruits have to be taken into account.

Potatoes. Draeger *et al.* (1992) planted 4 potatoes in plant boxes (2 per box) for cultivation under simulated field conditions. 64 days before harvest, the 4 plants were sprayed with a 0.2% WP25 formulation containing [methylene-¹⁴C]imidacloprid at a rate equivalent to 134 g ai/ha, 2.5 times the

recommended field application rate of 50 g ai/ha. The tubers and vines of one plant were collected 7 days and of another 28 days after application, and the remaining two plants were harvested after 64 days. The tubers were washed, and tubers and vines macerated separately in methanol/water, methanol and dichloromethane. Soil from the plant boxes was collected on the last sampling date, and separated into layers of 5-10 cm for measurement of radioactivity.

At harvest on day 64, 51% of the applied radioactivity was recovered from the soil, 49% from the vines and only 0.2% was in the tubers. The TRR in the vines decreased from 2.51 mg/kg as imidacloprid after 7 days to 1.35 mg/kg at harvest, mostly in aqueous or organic solvent extracts. Unextractable residues increased from 2.7% (0.073 mg/kg) at day 7 to 14.1% (0.19 mg/kg) at day 64.

In the tubers, total radioactive residues ranged from 0.006 mg/kg to 0.014 mg/kg. In immature potatoes (day 7 and day 28) the TRR consisted mainly of unextractable residues (94.2%, ~0.013 mg/kg at day 7), while in mature potatoes the extractable residues increased to 88.2% (~0.008 mg/kg at day 64). The results are shown in Table 24.

Table 24. Metabolism of [methylene-¹⁴C]imidacloprid in potato vines and tubers after spraying at 134 g ai/ha (Draeger *et al.*, 1992).

Days after final application	Radioactive residues as % of TRR and mg/kg ai equivalents					
	7 days		28 days		64 days	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
<i>Potato vines</i>						
Imidacloprid	71.8	1.80	48.2	0.95	37.9	0.51
M01	7.7	0.19	8.1	0.16	7.0	0.095
M03	0.9	0.022	2.0	0.039	2.7	0.036
M06	1.4	0.035	2.2	0.043	2.5	0.034
M07	1.8	0.045	1.7	0.033	2.2	0.03
M09	4.1	0.10	8.1	0.16	12.6	0.17
M25	n.a.	n.a.	n.a.	n.a.	<0.01	<0.014
M29	1.5	0.038	2.2	0.043	1.9	0.026
Total identified	89.2	2.23	72.5	1.43	66.8	0.90
Unknown	7.9	0.20	18.0	0.35	19.1	0.26
Unextractable	2.9	0.073	9.5	0.19	14.1	0.19
Total	100	2.50	100	1.97	100	1.35
<i>Potato tubers</i>						
Extractable residues ¹	5.8	<0.001	27.0	~0.002	88.2	~0.008
Imidacloprid					~6	~0.0005
M14					~30	~0.003
Others					~56	~0.005
Unextractable	94.2	0.013	73.1	0.005	11.8	0.001
Total	100	0.014	100	0.007	100	0.009

n.a.: not analysed

¹ Sum of single values

The main component in the extracts of potato vines was unchanged imidacloprid. It decreased from 71.8% of the TRR (1.8 mg/kg) at day 7 to 37.9% (0.51 mg/kg) at day 64 with a half-life of about 5 weeks. The two main metabolites were the 5-hydroxy M01 (7.0%, 0.095 at day 64) and denitro M09 (12.6%, 0.17 mg/kg at day 64). Four further metabolites accounting for 2-3% of the TRR at day 64 were the dihydroxy M03 (2.7%, 0.036 mg/kg) and the olefin M06 (2.5%, 0.34 mg/kg), the nitrosimine M07 (2.2%, 0.03 mg/kg), and CHMP glucoside M29 (1.9%, 0.026 mg/kg). Another very minor metabolite identified only in the mature plants extracts was the triazinone M25 which was probably formed by the amino compound M08. Unidentified components amounted to 19.1% (0.26 mg/kg). However, because potato vines are used neither for human nor animal food no further characterization was attempted.

Owing to the high proportion of unextractable residues in immature tubers, single components could only be identified in the extracts of mature potatoes (day 64), but even in these quantification was quite uncertain. Besides the parent compound, detected at about 6% (~0.0005 mg/kg), only 6-chloronicotinic acid (M14, ~0.003 mg/kg) was identified. 66.8-89.2% of the TRR in the vines and about 36% in mature tubers was identified by TLC and HPLC (with different solvent systems) and ¹H-NMR and mass spectrometry.

Tobacco. Clark and Brauner (1994) applied [methylene-¹⁴C]imidacloprid to the soil around 12 tobacco plants grown in a greenhouse and maintained under conditions which assured good plant growth using a pipette, 20 mg per plant soil drench (0.07% solution, c. 400 g ai/ha). 8 leaves of each plant were individually sprayed at 106 g ai/ha 40 days later with a 0.14% solution, three times at intervals of 7 and 6 days (total 28.4 mg ai/plant). The leaves were harvested 14 days after the last application, homogenised in liquid nitrogen and stored at -20°C. Samples were successively extracted with methanol/water, methanol and dichloromethane.

The TRR in the leaves at harvest amounted to 10.2 mg/kg parent equivalents; 97.7% (9.97 mg/kg) was detected in the extracts, while only 2.3% (0.23 mg/kg) remained in the solids and was not further investigated. The results are shown in Table 25.

Table 25. Metabolism of [methylene-¹⁴C]imidacloprid in tobacco leaves 14 days after a granular application and 3 spray applications at 720 g ai/ha (Clark and Brauner, 1994).

Compound or fraction	¹⁴ C	
	% of TRR	mg/kg ai equivalents
Imidacloprid	77.7	7.93
M01	4.0	0.41
M02	1.1	0.11
M06	1.0	0.10
M07	1.1	0.11
M09	5.7	0.58
M10	0.7	0.07
M12	2.1	0.22
M14	0.9	0.09
M29	0.4	0.04
Total identified	94.7	9.66
Unknown (5 components)	3.0	0.31
Unextractable	2.3	0.23
Total	100	10.2

Unchanged imidacloprid accounted for 77.7% of the TRR (7.93 mg/kg). Of nine identified metabolites the two main products were the 5-hydroxy M01 (4.0%, 0.41 mg/kg) and the denitro M09 (5.7%, 0.58 mg/kg). The 4-hydroxy M02, olefin M06, nitrosimine M07, and 2-ketone M12 each accounted for 1-2% (0.1-0.2 mg/kg) and the guanidine M10, 6-CNA M14 and CHMP glucoside M29 for 0.4-0.9% (0.04-0.09 mg/kg). Five unidentified metabolites were separated by thin-layer chromatography. In total, 94.7% of the TRR in the leaves was identified by TLC and HPLC and by mass and ¹H-NMR spectrometry.

Soil application

Egg plants. Yoshida (1991) applied 1% granules containing [methylene-¹⁴C]imidacloprid in the planting holes of 8 young plants at planting in containers in a greenhouse. The application rate of 2 g granules/plant (20 mg ai/plant) corresponded to the currently recommended rate of 1-2 g granules/plant or 0.3 kg ai/ha. The foliage of one plant was sampled after 14 days, that of two plants at day 35 and of 4 plants at day 69. Fruits were sampled at intervals during harvest after 49-67 days and stored in the freezer (-20°C). The foliage was separated into stems, leaves and other parts, and the fruits into edible parts and calyxes. These samples were homogenised separately and extracted with methanol/water and acetonitrile/water.

At harvest 69 days after treatment about 1.6% of the total applied radioactivity was detected in the aerial parts (stems, leaves, fruits, calyxes) of the plants, of which >88% was in the leaves.

The TRR in the foliage decreased from 5.9 mg/kg parent compound equivalents after 14 days to 1.4 mg/kg at harvest after 69 days. Irrespective of sampling dates the extracted residues accounted for 90.7-94.3% of the TRR, and unextracted ^{14}C remaining in the solids for 5.5-9.3%. In the fruits the TRR amounted to 0.043 mg/kg, of which 93.5% (0.04 mg/kg parent compound equivalents) was extracted and 6.5% (0.003 mg/kg) remained unextracted in the solids.

In the plant foliage unchanged parent compound was the main component at day 14 (32.6%, 1.92 mg/kg), but it decreased rapidly to 8.8% (0.3 mg/kg) at day 35 and 10.2% (0.15 mg/kg) at day 69. The main metabolite was the denitro M09, accounting for up to 33.9% (0.97 mg/kg) at day 35 (day 69: 24.6%, 0.28 mg/kg), followed by the 5-hydroxy M01 (3.6%, 0.054 mg/kg at day 69) and CHMP glucoside (M29, 5.6%, 0.096 mg/kg at day 69), then the olefin M06, the nitrosimine M07, 2-ketone M12 and CNA M14 ranging from 0.1% (0.001 mg/kg) to 1.3% (0.019 mg/kg) at day 69. Identification was as before. Up to 44.1% (0.63 mg/kg at day 69) of the TRR remained unidentified. However, as egg plant foliage is not used for human or animal food, no further characterization was attempted.

Except for a very minor metabolite in foliage [M12], the metabolites in the extracts of egg plant fruits and foliage were the same, but the proportions differed. The main component in the fruits was unchanged parent compound at 18.9% (0.008 mg/kg), closely followed by the denitro M09 (14%, 0.005 mg/kg), 6-CNA (M14, 13.4%, 0.004 mg/kg) and CHMP glucoside (M29, 13%, 0.007 mg/kg). The 5-hydroxy M01 accounted for 3.2% (0.002 mg/kg), the olefin M06 for 0.2% (<0.001 mg/kg), and the nitrosime M07 for 0-1% (<0.001 mg/kg). 30.8% of the extractable TRR could not be identified, but as this corresponded to only 0.01 mg/kg parent compound equivalents further characterization was not necessary. Calculation of levels of identified metabolites was based on specific molecular weights. The results are shown in Table 26.

Table 26. Metabolism of [methylene- ^{14}C]imidacloprid in egg plants after granular soil applications at a rate of 0.3 kg ai/ha (Yoshida, 1991).

Compound or fraction	Residues in egg plant foliage and fruit							
	Fruit (edible part)		Foliage					
	harvest (49-67 days)		14 days		35 days		69 days	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	18.9	0.008	32.6	1.92	8.8	0.30	10.2	0.15
M01 ¹	3.2	0.002	8.7	0.55	2.4	0.089	3.6	0.054
M06 ¹	0.2	<0.001	1.5	0.090	1.0	0.035	1.3	0.019
M07 ¹	0.1	<0.001	1.9	0.10	0.8	0.027	0.3	0.003
M09 ¹	14.0	0.005	21.4	1.04	33.9	0.97	24.6	0.28
M12 ¹	n.d.	n.d.	0.2	0.008	0.4	0.01	0.1	0.001
M14 ¹	13.4	0.004	1.2	0.044	0.5	0.011	0.9	0.008
M29 ¹	13.0	0.007	4.2	0.30	5.0	0.21	5.6	0.096
Total identified ^{2,3}	62.8	0.027	71.7	4.22	52.8	1.83	46.6	0.66
Unknown ^{2,3}	30.8	0.013	22.6	1.33	38.5	1.34	44.1	0.63
Unextractable ³	6.5	0.003	5.5	0.32	8.7	0.30	9.3	0.13
Total ³	100.1	0.043	100	5.88	100	3.47	100	1.42

n.d.: not detected

¹ mg/kg values based on specific molecular weights.

² Sum of single % of TRR values.

³ Concentrations in mg/kg as imidacloprid.

Potatoes. Vogeler *et al.* (1992b) planted 6 pre-germinated potatoes into seed furrows (2 potatoes per 80 cm furrow) in a planting box and applied 5% granules containing [methylene- ^{14}C]imidacloprid in the furrows at a rate of 0.05 g ai or 1 g granules per running m at planting, corresponding to the

recommended rate for seed furrow treatment. The study was carried out in the open air under simulated field conditions but the container was protected against rain. Potatoes were harvested 129 days after treatment, as in practice, and the tubers washed with cold water. Tubers and vines were then cut into small pieces and stored at -20°C . The samples were extracted by successive maceration with methanol/water, methanol and dichloromethane.

0.3% of the applied radioactivity was recovered in the tubers, 2.2% in the vines, and almost all (98.4%) was found in the soil.

The TRR in the vines amounted to 5.76 mg/kg parent compound equivalents, including 73.4% (4.23 mg/kg) extractable and 26.4% (1.52 mg/kg) unextractable. As potato vines are not used for human or animal food, no further characterization of the unextractable residues was attempted. The TRR in the tubers after 129 days amounted to 0.091 mg/kg parent compound equivalents: 93.6% (0.085 mg/kg) was detected in the extracts, and the 6.4% (0.006 mg/kg) unextractable in the solids was not further characterized.

In the vines 26.7% of the TRR (1.53 mg/kg) was unchanged imidacloprid. The three main metabolites were the denitro M09 (8.2%, 0.48 mg/kg), 6-CNA (M14, 8.3%, 0.48 mg/kg) and the 5-hydroxy M01 (4.6%, 0.26 mg/kg), followed by the olefin M06 (3.3%, 0.19 mg/kg), the nitrosime M07 (2.6%, 0.15 mg/kg) and CHMP glucoside (M29, 1.4%, 0.08 mg/kg), and small quantities of the dihydroxy M03 (0.3%, 0.02 mg/kg). 14 unknown metabolites accounted for 16.1% (0.93 mg/kg), single components to a maximum of 5.4% (0.3 mg/kg), and further unidentified products were detected in an n-hexane extract (2.1%, 0.12 mg/kg). As potato vines are not used for human or animal food, no further identification or characterization was considered necessary.

In the tubers the main component was unchanged parent compound, amounting to 48.3% (0.044 mg/kg). The three main metabolites were the same as in the vines, accounting for 8.0-11.3% (11.3%, 0.010 mg/kg; 9.4%, 0.009 mg/kg; and 8.0%, 0.007 mg/kg respectively). The olefin M06 (3.1%, 0.003 mg/kg) was also found. Five further unidentified metabolites accounted for 13.1% (0.012 mg/kg). In total, 55.4% of the TRR in vines and 80.1% in tubers was identified. In the tubers, no uncharacterized radioactivity exceeded 0.01 mg/kg parent compound equivalents. Identification was by TLC and HPLC with different solvent systems and by mass and ^1H -NMR spectrometry. The results are shown in Table 27.

Table 27. Metabolism of [methylene- ^{14}C]imidacloprid in potato vines and tubers after granular soil application at a rate of 0.05 g ai per running m 129 days after application (Vogeler *et al.*, 1992b).

Compound or fraction	Residues			
	Vines		Tubers	
	% of TRR	mg/kg (as imidacloprid)	% of TRR	mg/kg (as imidacloprid)
Imidacloprid	26.7	1.53	48.3	0.044
M01	4.6	0.26	8.0	0.007
M03	0.3	0.02	n.d.	n.d.
M06	3.3	0.19	3.1	0.003
M07	2.6	0.15	n.d.	n.d.
M09	8.2	0.48	11.3	0.01
M14	8.3	0.48	9.4	0.009
M29	1.4	0.08	n.d.	n.d.
Total identified	55.4	3.19	80.1	0.073
Unknown ¹	16.1	0.93	13.1	0.012
Others ²	2.1	0.12	0.4	<0.001
Unextractable	26.4	1.52	6.4	0.006
Total	100	5.76	100	0.091

n.d.: not detected

¹ tubers: 5 components; vines: 14 components

² n-hexane phase

Rice. Three rice plants cultivated in plant boxes in a greenhouse under simulated field conditions and maintained in accordance with conventional agricultural practice in a flooded test plot and were treated with a 1% granular formulation containing [methylene-¹⁴C]imidacloprid applied at the middle growth stage 66 days after sowing at 0.5 kg ai/ha (the recommended rate for soil application is 0.27 kg ai/ha) (Kurogochi and Araki, 1989). Mature plants were sampled after 79 days (145 days after sowing) and the samples air-dried for a week before being stored at -20°C. The plant parts were divided into hulled grain, chaff and straw. Some hulled grain was polished and divided into polished rice and bran. All samples were crushed in a mill and extracted several times with methanol/water.

At harvest only 4.5% of the applied radioactivity (AR) had translocated into the plants. The main residues were found in the straw, chaff and roots. In the grain residues amounted to 0.05% AR, and 0.04% AR remained in the polished grain and 0.01% in the bran.

The TRR in the straw accounted for 1.47 mg/kg parent compound equivalents. 56.6% of the TRR (0.83 mg/kg) was in the extracts, 43.4% (0.64 mg/kg) remained unextracted in the plant tissue. In hulled rice grain the TRR amounted to 0.036 mg/kg parent compound equivalents, of which only 19.3% (0.007 mg/kg) was extractable. The results are shown in Table 28.

Table 28. Metabolism of [methylene-¹⁴C]imidacloprid in rice after soil applications at 0.5 kg ai/ha 79 days after application (Kurogochi and Araki, 1989).

Compound or fraction	Residues			
	Grain		Straw	
	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	6.3	0.002	11.5	0.17
M01 ¹	n.d.	n.d.	1.5	0.02
M06 ¹	n.d.	n.d.	0.5	0.008
M07 ¹	n.d.	n.d.	1.1	0.016
M09 ¹	n.d.	n.d.	25.6	0.38
M12 ¹	n.d.	n.d.	0.6	0.007
M14 ¹	2.7	0.001	2.1	0.019
Total identified ²	9.0	0.003	42.9	0.63
Unknown metabolites ^{2,3}	1.5	<0.001	1.8	0.026
Others ⁴	8.8	0.003	11.9	0.17
Unextractable ²	80.7	0.029	43.4	0.64
crude amino acid fraction	2.3	0.001		
crude protein fraction	13.9	0.005		
crude starch fraction	64.5	0.023		
complex substances			6.6	0.097
crude lignin			9.9	0.15
crude cellulose			26.9	0.40
Total	100	0.036	100	1.47

n.d. not detected

¹Concentrations based on specific molecular weights.

²Concentrations in mg/kg parent compound equivalents.

³Grain: 5 components; straw: 4 components.

⁴Grain: 4 fractions; straw: at least 7 fractions or subfractions.

In straw the two main constituents were the denitro M09 (25.6%, 0.38 mg/kg), and the parent compound (11.5%, 0.17 mg/kg), followed by the 5-hydroxy M01, olefin M06, nitrosime M07, 2-ketone M12 and 6-CNA M14, at levels of 0.5-2.1%. Four unidentified metabolites each accounted for up to 0.5% (0.007 mg/kg as imidacloprid). In various fractions of the extract uncharacterized radioactivity amounted to 11.9% of the TRR, only once exceeding 0.05 mg/kg parent compound equivalents (0.1 mg/kg). Four subfractions of this fraction were identified by thin-layer

chromatography, and it can therefore be assumed that no unidentified metabolite exceeded 0.05 mg/kg. Unextractable residues in the straw were fractionated into complex substances, crude lignin and crude cellulose, and most of the ^{14}C was bound either to cellulose (29.6% of the TRR) or to lignin (9.9%). M09 and unchanged parent compound were the main components found after exhaustive extraction of cellulose. Incorporation of radiocarbon into natural constituents such as lignin or cellulose was estimated at approximately 7%. The results are shown in Table 28.

In the hulled rice grain extracts 7 different components were detected, only two of which were significant: unchanged parent compound (6.3%, 0.002 mg/kg) and the acid 6-CNA (M14, 2.7%, 0.001 mg/kg). Unextractable residues were divided into crude amino acid (2.3%, 0.001 mg/kg), protein (13.9%, 0.005 mg/kg) and starch (64.5%, 0.023 mg/kg) fractions. Further analysis showed that about 40% of the TRR, i.e. most of the recovered radioactivity in the starch fraction was attributable to natural constituents derived from $^{14}\text{CO}_2$. Identification was by co-chromatography with TLC and HPLC using different solvent systems as well as by ^1H -NMR spectrometry. The results are shown in Table 28.

Nursery box treatment

Rice. Sakamoto (1991) treated soil in nursery boxes with a 2% granular formulation containing [methylene- ^{14}C]imidacloprid at a rate of 0.32 kg ai/ha (current recommended maximum dose for use in nursery box treatment is 1.6 g ai/box, 200 boxes/ha).

In a further experiment the granular formulation was dissolved in acetone and applied to the soil at a fourfold rate of 1.26 kg ai/ha. The solvent was allowed to evaporate, and after an hour the seedlings were transplanted into the treated soil and grown in a greenhouse according to conventional agricultural practice. Immature shoots were sampled before the heading stage 65 days after treatment, and freeze-crushed by adding liquid nitrogen. 124 days after application the mature plants were divided into hulled rice and straw and milled. Homogenised samples were stored at -20°C , and later extracted with methanol/water and methanol/ethyl acetate or acetonitrile/water and acetonitrile/ethyl acetate.

After the application of 0.32 kg ai/ha about 4% of the applied radioactivity was translocated to immature shoots within 65 days and increased only to 4.4% in mature foliage. Total radioactive residues in mature plants amounted to 1.31 mg/kg parent compound equivalents in the straw (99%) and only 0.014 mg/kg in the grain. In straw about 60% of the TRR was extractable under normal conditions, while about 40% remained unextracted in the plant tissues. In grain about 30% of the TRR was extractable and about 70% unextracted.

The main components in the straw extracts were the denitro M09 (36.2%, 0.48 mg/kg) and unchanged parent compound (8.1%, 0.11 mg/kg). 5 further metabolites were the 5-hydroxy M01, olefin M06, nitrosime M07, 2-ketone M12 and 6-CNA M14 at <1% each. The sum of four unidentified products, unexamined chromatographic fractions and diffuse radioactivity amounted to 13% (0.17 mg/kg), none individually exceeding 0.05 mg/kg. The unextracted residue in straw was characterized by successive extractions under increasingly vigorous conditions. The denitro M09 formed a significant part of the residues (11.7% of TRR), with unchanged parent compound, M14 and M12 up to 7.1%. M12 was considered to be formed by conversion of the parent compound under alkaline conditions. The extent of incorporation of radioactivity into natural constituents as lignin was negligible (3%).

The main component in the grain extracts was unchanged parent compound, 13.6% (0.002 mg/kg) at the normal application. Four metabolites detected at 2-3% of the TRR were the 5-hydroxy M01, olefin M06, denitro M09 and 6-CNA M14, with trace amounts (0.2%) of the nitrosime M07. As most of the TRR in grain was unextracted, this fraction from the plants treated at the exaggerated rate was further investigated. About 16% of the TRR was in the crude protein glutelin fraction, and about 48% in the starch fractions, incorporated into natural constituents.

In total, about 50% of the TRR in shoots and straw and about 20-25% of the TRR in grain were identified. No uncharacterized radioactivity exceeded 0.01 mg/kg parent compound equivalents in grain or 0.05 mg/kg in straw. Identification was by co-chromatography (TLC and HPLC with different solvent systems) and by mass and ^1H -NMR spectrometry. The results are shown in Tables 29 and 30.

Table 29. Metabolism of [methylene- ^{14}C]imidacloprid in rice shoots and straw grown in a nursery box, application at two rates (Sakamoto, 1991).

Compound or fraction	^{14}C in shoots and straw, % of TRR and mg/kg ai equivalent					
	65 days; 0.32 kg ai/ha		124 days; 0.32 kg ai/ha		124 days; 1.26 kg ai/ha	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	7.8	0.029	8.1	0.11	17.6	1.50
M01	1.1	0.004	0.9	0.012	1.4	0.12
M06	0.2	<0.001	0.1	0.001	0.5	0.043
M07	1.1	0.004	0.9	0.012	1.8	0.15
M09	42.9	0.16	36.2	0.48	33.5	2.86
M12	0.4	0.002	0.4	0.005	1.0	0.085
M14	1.9	0.007	0.5	0.007	0.8	0.068
Total identified	55.4	0.21	47.1	0.63	56.6	4.83
Unknown	9.2	0.035 ¹	13.2	0.17 ¹	10.8	0.92 ²
Unextractable	35.9	0.14	39.8	0.52	32.6	2.78
Total	100.5	0.38	100	1.32	100	8.53

¹ Unidentified metabolites and diffuse radioactivity; each fraction <0.05 mg/kg.

² Minimum of 9 components.

Table 30. Metabolism of [methylene- ^{14}C]imidacloprid in rice grain grown in a nursery box, two application rates (Sakamoto, 1991).

Compound or fraction	^{14}C in rice grain, % of TRR and mg/kg ai equivalents			
	124 days; 0.32 kg ai/ha		124 days; 1.26 kg ai/ha	
	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	13.6	0.002	11.9	0.008
M01	3.7	<0.001	3.4	0.002
M06	2.3	<0.001	2.0	0.001
M07	0.2	<0.001	0.2	<0.001
M09	2.2	<0.001	1.2	<0.001
M14	2.6	<0.001	0.8	<0.001
Total identified	24.6	0.003	19.5	0.012
Unknown	6.6	0.001	8.2	0.005
Unextractable	68.9	0.010	72.4	0.046
glutelin fraction	n.a.	n.a.	16.1	0.010
starch fraction	n.a.	n.a.	48.2	0.031
Total	100	0.014	100	0.063

n.a.: not analysed

Seed treatment

Maize. Sixteen seeds treated with a WS 70 formulation containing [methylene- ^{14}C]imidacloprid (Vogeler *et al.*, 1992c) at a rate of 721 g ai/100 kg seed, corresponding to the proposed application rate for seed dressing of 700 g ai/100 kg seed (105 g ai/ha or 15 kg treated maize seed/ha) were planted in pots filled with loamy silt soil and grown in a greenhouse. Immature plants were sampled after 33 days (6–7 leaf stage) and after 61 days (about 9 leaf stage). At harvest, after 134 days, the plants were separated into fodder, husks, cobs and dry grain, which were macerated in liquid nitrogen and stored at -20°C . Extraction under conventional conditions was with methanol/water and methanol, and under exhaustive conditions with boiling methanol, 6N HCl/methanol and 2N NaOH/methanol.

Uptake of the applied radioactivity increased from about 4.2% at day 33 to 10.2% at day 61, and to 20% at day 134. Total residues amounted to 5.84 mg/kg parent compound equivalents in the immature maize at day 33 and 1.52 mg/kg at day 61, and 3.08 mg/kg in fodder, 0.21 mg/kg in the husks, 0.12 mg/kg in the cobs and 0.04 mg/kg in dry grain at day 134. The TRR changed from 93.4% extractable and 7.6% unextractable under conventional conditions in immature plants at day 33 to about 70% extractable and about 30% unextractable in the mature plants (almost the same values were found in all plant parts). In total, about 50% of the TRR in the fodder of mature plants and about 60% of the TRR in ears was identified by co-chromatography (TLC and HPLC with different solvent systems)) and by mass and ^1H -NMR spectrometry. The results are shown in Tables 31 and 32.

Table 31. Metabolism of [methylene- ^{14}C]imidacloprid in maize plants after seed dressing at 721 g ai/100 kg seed. Samples taken 33, 61 and 134 days after sowing (Vogeler *et al.*, 1992c).

Compound or fraction	Residues in maize plants, % of TRR and mg/kg ai equivalents					
	immature: 33 days		immature: 61 days		fodder: 134 days	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	65.2	3.81	47.2	0.72	22.2	0.68
M01	7.0	0.41	5.8	0.09	5.0	0.15
M03	0.5	0.03	0.4	<0.01	0.5	0.02
M06	4.5	0.26	3.0	0.05	2.2	0.07
M07	1.7	0.10	2.9	0.04	1.8	0.06
M09	5.7	0.33	11.2	0.17	10.9	0.34
M10	0.6	0.04	0.9	0.01	1.6	0.05
M12	trace	trace	trace	trace	trace	trace
M14	0.7	0.04	0.4	<0.01	1.3	0.04
M28	0.5	0.03	0.7	0.01	1.1	0.03
M01 conjugate	n.d.	n.d.	n.d.	n.d.	~1	~0.03
Total identified	86.4	5.05	72.5	1.10	47.6	1.47
Unidentified metabolites	3.8	0.22 ¹	7.0	0.11 ²	13.1	0.40 ³
Diffuse radioactivity	2.2	0.13	3.5	0.05	7.2	0.22
Unextractable	7.6	0.44	17.0	0.26	32.1	0.99
Total	100	5.84	100	1.52	100	3.08

n.d.: not detected

¹ 7 unknown metabolites between 0.1 and 1.1% each.

² 9 unknown metabolites between 0.3 and 1.7% each.

³ 12 unknown metabolites between 0.3 and 2.7% each.

Table 32. Metabolism of [methylene- ^{14}C]imidacloprid in maize ears 134 days after planting seed dressed at 721 g ai/100 kg seed (Vogeler *et al.*, 1992c).

Compound or fraction	Residues in mature maize ears, % of TRR and mg/kg ai equivalents					
	grain		husks		cobs	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	25.2	0.01	43.3	0.091	46.6	0.056
M01	9.3	0.004	8.5	0.018	9.1	0.011
M03	4.4	0.002	0.2	<0.001	0.1	<0.001
M06	13.1	0.005	4.3	0.009	6.3	0.008
M07	n.d.	n.d.	1.4	0.003	0.5	<0.001
M09	2.0	<0.001	2.6	0.006	1.0	0.001
M10	n.d.	n.d.	0.4	0.001	0.6	<0.001
M14	trace	trace	0.6	0.001	1.2	0.001
M28	4.4	0.002	0.4	0.001	0.8	0.001
Total identified	58.4	0.023	61.7	0.13	66.2	0.079
Unidentified metabolites	4.6	0.001 ¹	3.0	0.006 ²	1.7	0.002 ³
Diffuse radioactivity	10.8	0.004	3.6	0.007	3.8	0.005
Unextractable	26.2	0.01	31.7	0.067	28.3	0.034
Total	100	0.04	100	0.21	100	0.12

n.d.: not detected

¹ 2 unknown metabolites between 1.7 and 2.9% each.

² 5 unknown metabolites between 0.1 and 1.3% each.

³ 5 unknown metabolites between 0.1 and 0.6%.

The proportion of unchanged parent compound decreased from 65.2% of the TRR (3.81 mg/kg) at day 33 to 22.2% (0.68 mg/kg) in the fodder of mature plants, while unextractable residues increased from 7.6% (0.44 mg/kg) at day 33 to 32.1% (0.99 mg/kg) at day 134. No significant qualitative or quantitative differences in the metabolic profile were found at different samplings. The main metabolites were the denitro M09 (10.9%, 0.34 mg/kg in fodder) and 5-hydroxy (M01, 5.0%, 0.15 mg/kg in fodder), with minor amounts (up to 2.2% in fodder) of the dihydroxy M03, olefin M06, nitrosime M07, 2-ketone M12 and 6-CNA M14 as well as the guanidine M10 and CHMP M28. A conjugate of M01 was found in the fodder of mature plants only. While the former metabolites were all well known from the metabolism studies already described, the last three were not found in the other crops (Table 31). Unextractable residues in the fodder after extraction under conventional conditions were investigated by exhaustive extraction. The components mainly detected in the successive extracts were unchanged parent compound (sum: 4.4%, 0.156 mg/kg), M09 (sum: 2.3%, 0.071 mg/kg), M10 (1.3%) and M06 (0.3%). In alkaline extracts M12 predominated (sum 8.9%, 0.275 mg/kg), probably as a conversion product formed during extraction.

In mature maize ears (grain, cobs, husks), unchanged parent compound was the main component, amounting to 25.2% (0.01 mg/kg) in dry grain. The two main metabolites in all samples were the olefin M06 and 5-hydroxy M01, accounting for 13.1% (0.005 mg/kg) and 9.3% (0.004 mg/kg) in grain respectively, and minor products were the dihydroxy M03, denitro M09, 6-CNA M14 and CHMP M28. The nitrosime M07 and the guanidine M10 were only found in cobs and husks (Table 32). Residues remaining in grain were exhaustively extracted to yield three compounds, each in very low amounts (<0.001 mg/kg): unchanged parent compound, M12 (about 1.2%) and the olefin M06 (1.0%). At least 6% of the TRR in dry grain was incorporated into glucose.

Cotton. Vogeler and Brauner (1992) dressed seed with [methylene-¹⁴C]imidacloprid in a 70 WS formulation applied at a rate equivalent to 460 g ai/100 kg seed. The plants were grown in a greenhouse and harvested at maturity (day 211). Leaves which fell during the experiment were collected and at harvest plants were separated into fruit (cotton bolls, containing lint and seeds) and gin trash. Lint and seed were initially separated manually, then to remove any remaining lint, seeds were treated with sulfuric acid. The leaves, lint, seeds and gin trash were macerated separately in liquid nitrogen and stored at -20°C. Samples were extracted under neutral, acidic and alkaline conditions.

The uptake of the radioactivity from the original treated seeds was 4.88% (seeds 0.067%, gin trash 0.068%, lint 0.02%, and leaves 4.72%). Levels of the TRR in the individual plant parts were very low, amounting 211 days after planting to 0.0049 mg/kg parent compound equivalents in seeds, 0.005 mg/kg in gin trash, 0.0019 mg/kg in the lint and 0.11 mg/kg in the leaves.

Because of the very low residues in seeds, lint and gin trash identification of metabolites was virtually impossible. Only 6-CNA (M14, 23% of the TRR, 0.0012 mg/kg) was identified in the seeds. Further compounds found in methanol/water and methanol/HCl extracts contained the chloropyridylmethyl moiety of imidacloprid. No parent compound was detected in any extract. However the artefacts formed in the extraction process made further identification or quantification impossible. No radioactivity was detected in the seed oil, which was removed by n-hexane. Unextractable residues amounted to 14.4% (<0.001 mg/kg). No radioactive residues could be extracted from the lint (TRR 0.0019 mg/kg), so it can be assumed that radioactivity was incorporated into natural components of the lints, e.g. cellulose. Extracts of gin trash could not be further chromatographed owing to the low levels of radioactivity (0.005 mg/kg), so no metabolites were identified.

In leaves most of the TRR (0.11 mg/kg) was extracted, only 26.8% (0.029 mg/kg) remained in the plant tissues. The composition of metabolites was similar to that in maize. Unchanged parent compound was found in relatively low amounts (2.9%, 0.003 mg/kg as imidacloprid). The three main components in leaf extracts were the denitro M09 (9.8%, 0.011 mg/kg), CHMP glucoside M29 (6.3%, 0.007 mg/kg) and another conjugate CHMP (11.3%, 0.012 mg/kg). In addition eight minor metabolites were detected in amounts ranging from 0.8 to 4.4% (up to 0.005 mg/kg). Four were the olefin M6, nitrosimine M7, 6-CNA M14 and CHMP M28. The others remained unidentified. Some radioactivity (25.5%) could not be separated by TLC and appeared as diffuse radioactivity on the TLC plates. However, as this amounted to only 0.028 mg/kg, no further characterization was attempted. In total, 37.3% of the TRR in leaves was identified, 10.4% was characterized as single but unidentified components. Characterization and identification of metabolites was by TLC, HPLC and mass spectrometry. The results are summarized in Table 33.

Table 33. Metabolism of [methylene-¹⁴C]imidacloprid in cotton 211 days after seed dressing at a rate of 460 g/100 kg seeds (Vogeler and Brauner, 1992).

Compound or fraction	¹⁴ C, % of TRR and mg/kg ai equivalents			
	seeds		leaves	
	% of TRR	mg/kg	% of TRR	mg/kg
Imidacloprid	n.d.	n.d.	2.9	0.003
M06			1.5	0.002
M07			1.4	0.002
M09			9.8	0.011
M14			2.2	0.002
M28	~23	~0.0012	1.9	0.002
M28 conjugate			11.3	0.012
M29			6.3	0.007
Total identified			37.3	0.041
Unknown	~62 ¹	~0.003	10.4 ²	0.012
Diffuse radioactivity			25.5	0.028
Unextractable	14.4	<0.001	26.8	0.029
Total	100	0.0049	100	0.11

n.d.: not detected

¹ Two fractions (methanol/water and methanol/HCl extract, ca. 30% each) containing metabolites determined as the 6-chloronicotinic moiety.

² Sum of single values; 4 unknown compounds between 0.8 and 4.4% each.

Rotational crops

Vogeler *et al.* (1992d) applied [methylene-¹⁴C]imidacloprid at a rate of 454 g ai/ha to the surface of a sandy loam soil in a plant container with a surface area of 1.03 m² and a depth of 60 cm. For the first seven months the container was in an open vegetation area and was then placed in a greenhouse. Wheat, red beet and Swiss chard were planted in rotation after ageing periods of 30 days (1st rotation), 120 days (2nd rotation) and 271 days (3rd rotation), and harvested at maturity. An additional forage sample was taken from immature wheat plants. Before each planting, the soil was mixed and soil samples taken 0, 30, 120, and 271 days after application as well as at harvest times. The TRR in the soil decreased from 0.36 mg/kg parent compound equivalents at day 0 to 0.2 mg/kg at day 412. The residues consisted mainly of parent compound although several metabolites were detected at low concentrations (<5% of the radioactivity in the soil).

The total uptake of radioactivity in the combined crops was 1.5% (1st rotation), 2.4% (2nd rotation) and 1.1% (3rd rotation) based on TRR in the soil at days 30 (0.34 mg/kg), 120 (0.21 mg/kg), and 271 after application (0.28 mg/kg). In each rotation, the lowest residues were in the storage organs wheat grain (0.07, 0.06, and 0.03 mg/kg from the first to the third rotation) and red beet roots (0.07, 0.03, and 0.04 mg/kg). The highest residues (TRR) were in wheat straw (2.5, 2.38, and 0.96 mg/kg) and forage (0.48, 1.0, and 0.26 mg/kg). In Swiss chard the TRR amounted to 0.13, 0.24, and

0.09 mg/kg and in red beet leaves to 0.26, 0.21 and 0.17 mg/kg in the three rotations. A summary of the results is given in Table 34.

Table 34. Total radioactive residues in rotational crops after application of 454g ai/ha of [methylene-¹⁴C]imidacloprid (Vogeler *et al.*, 1992d).

Crop	TRR as imidacloprid equivalents					
	1st rotation		2nd rotation		3rd rotation	
	Sampling day	mg/kg	Sampling day	mg/kg	Sampling day	mg/kg
Red beet roots	120	0.07	271	0.03	384	0.04
Red beet leaves	120	0.26	271	0.21	384	0.17
Swiss chard	91	0.13	201	0.24	345	0.09
Wheat forage	62	0.48	154	1.00	292	0.26
Wheat straw	120	2.50	271	2.38	408	0.96
Wheat grain	120	0.07	271	0.06	408	0.03

The parent compound was metabolised significantly in the crops. The metabolites were essentially the same qualitatively and quantitatively as in crops from plant metabolism studies. The distribution of metabolites (% of recovered radioactivity) in the crops is given in Table 35. The components constituting more than 10% of the radioactivity were:

Parent compound	imidacloprid	(Swiss chard, wheat forage)
denitro	M09	(Swiss chard, wheat straw)
5-hydroxy	M02	(Swiss chard, wheat forage)
6-CNA	M14	(red beet roots)
olefin	M06	(Swiss chard, wheat straw)
Unknown 1	-	(red beet roots)

Table 35. Total radioactive residues (TRR) and distribution of imidacloprid and its metabolites in rotational crops after application of [¹⁴C-methylene]imidacloprid to the surface of soil at 454 g ai/ha (Vogeler *et al.*, 1992d).

Crop	D A T	TRR parent equiv., mg/kg	% of recovered radioactivity													H ₂ O eluate	Un- extr.	Total
			sum from ethyl acetate and water phases															
			ai	M1+ M2	M3	M6	M7	M9	M10	M14	M28	M29	Un- known 1	Others				
Red beet roots	120	0.07	5.9	4.9		1.3	0.3	n.d.	5.4	13.5	0.5*	0.2	18.5	19.4	17.4	12.2	100	
	271	0.03	7.8	5.1	0.7	2.2	n.d.	3.0	5.2	9.6	0.5	n.d.	12.5	12.0	18.5	22.9	100	
	384	0.04	9.2	5.5	n.d.	4.5	n.d.	1.9	n.d.	16.2	n.d.	n.d.	14.5	13.7	8.0	22.0	100	
Red beet leaves	120	0.26	3.7	8.0	1.3	4.0	n.d.	6.2	3.1	1.7	n.d.	7.1	8.5	25.9	15.2	15.3	100	
	271	0.21	3.6	8.2	1.2	3.5	0.6	3.1	2.7	4.9	0.9	5.0	1.5	12.0	22.3	30.5	100	
	384	0.17	4.9	5.9	1.0	3.0	0.5	2.1	3.7	4.7	n.d.	8.5	4.0	18.6	18.5	23.7	100	
Swiss chard	93	0.13	23.5	8.1	1.2	4.6	0.6	11.2	1.3	9.3	n.d.	2.0	2.5	17.0	9.3	9.4	100	
	201	0.24	11.5	10.3		6.4	0.7	3.9	2.3	8.6	1.9*	6.0	9.4	15.3	7.3	16.4	100	
	345	0.09	5.8	17.4	5.7	10.4	n.d.	3.4	3.0	0.3	n.d.	7.1	4.2	20.8	13.2	8.7	100	
Wheat forage	62	0.48	42.1	14.0	0.9	4.0	2.7	11.9	1.5	0.9	n.d.	2.2	n.d.	7.6	2.4	9.8	100	
	154	1.0	40.6	12.6	0.9	5.1	2.4	8.8	1.1	0.9	n.d.	1.4	n.d.	15.3	1.5	9.4	100	
	292	0.26	46.5	16.9	n.d.	6.0	3.4	8.4	1.3	0.7	n.d.	1.5	n.d.	5.6	1.1	8.6	100	
Wheat straw	120	2.5	4.7	6.8	2.6	6.3	1.0	18.5	5.0	0.6	n.d.	4.4	4.0	17.9	5.9	22.3	100	
	271	2.38	8.6	6.9	1.5	6.7	1.4	10.8	3.2	1.0	0.7	3.3	2.6	12.6	17.4	23.3	100	
	408	0.96	9.5	6.3	2.3	6.0	1.6	14.9	4.9	1.1	n.d.	7.0	2.7	17.5	12.2	14.0	100	
Wheat grain	120	0.07	1.9	10.3	3.6	5.3	n.d.	3.7	n.d.	n.d.	n.d.	1.8	3.5	11.5	17.0	41.4	100	
	271	0.06	0.4	0.4	0.3	0.7	n.d.	-	-	0.5	n.d.	n.d.	-	24.9	17.7	55.1	100	
	408	0.03	2.9	8.5	3.1	5.2	0.7	5.0	n.d.	n.d.	n.d.	1.9	5.5	16.6	7.0	43.6	100	

Unknown 1 unknown metabolite containing the 6-chloronicotinic moiety

Others sum of 11 metabolites mostly based on 6-CNA

H₂O eluate eluate from XAD4 clean-up of initial extract

DAT days after soil treatment

* M3 + M28

n.d.: not detected

-: not determined

“Unknown 1” was only found in significant amounts in red beet roots. It contained the 6-chloronicotinic moiety of imidacloprid. Many others, most containing the 6-chloronicotinic moiety, were found. In red beet leaves, CHMP glucoside M29 was found at 0.014 mg/kg, and in wheat straw the olefin M06 (0.157 mg/kg) and the guanidine M10 (0.125 mg/kg). In wheat grain and red beet roots all components were well below 0.01 mg/kg. Low amounts of radioactivity were incorporated into starch of wheat grain.

Summary

In eleven trials, including one confined rotational crop study, 11 different plant species were treated by three different application methods and uptake, translocation and metabolism were similar in all (Table 36). The proposed pathways are shown in Figure 3.

In egg plants, potatoes, rice and cotton uptake of radiolabelled imidacloprid from the soil after granular application or seed treatment was low and ranged from 1.8 to 4.9% of the applied radioactivity in the aerial part of mature plants. In rice and egg plants (in cotton and potatoes this question was not investigated) uptake had been completed after half the growing period and did not increase appreciably in the second half, but in maize plants radioactivity increased continuously and at harvest amounted to 20%.

In all studies translocation in the plants occurred, mainly from the roots to the leaves. After soil application, most of the radioactivity was found in the foliage, with minor amounts in fruits, grain and seed. A trial with spray application in potatoes showed that transport from the sprayed leaves to the tubers was negligible. Acropetal translocation was also demonstrated in translocation experiments in apples and tomatoes. 14 days after the application of imidacloprid to leaves radioactivity in fruits was negligible. The distribution in other plant parts (shoot, stem, untreated leaves) was not further investigated.

In all studies three degradation routes were observed in nearly all plant parts:

- Hydroxylation of the imidazolidine ring leading to the mono- and dihydroxylated compounds (M01, M02, M03) and subsequent removal of water from M03 to form the olefin M06.
- Reduction of the nitro group to form the nitrosimine compound M07 and loss of this group with formation of the guanidine metabolites M09 and M10.
- Oxidative cleavage of the methylene bridge to form 6-chloro-3-pyridylmethanol and its conjugates M28, M29 and M30 with further oxidation to 6-chloronicotinic acid M14.

In rice grain after granular application and in potato tubers after spray application the total amount of recovered radioactivity was very low so that only very few metabolites could be detected.

Analysis of unextracted residues in rice and maize grain showed apparent degradation of imidacloprid to CO₂ and subsequent incorporation into natural constituents such as starch, glutenin or lignin.

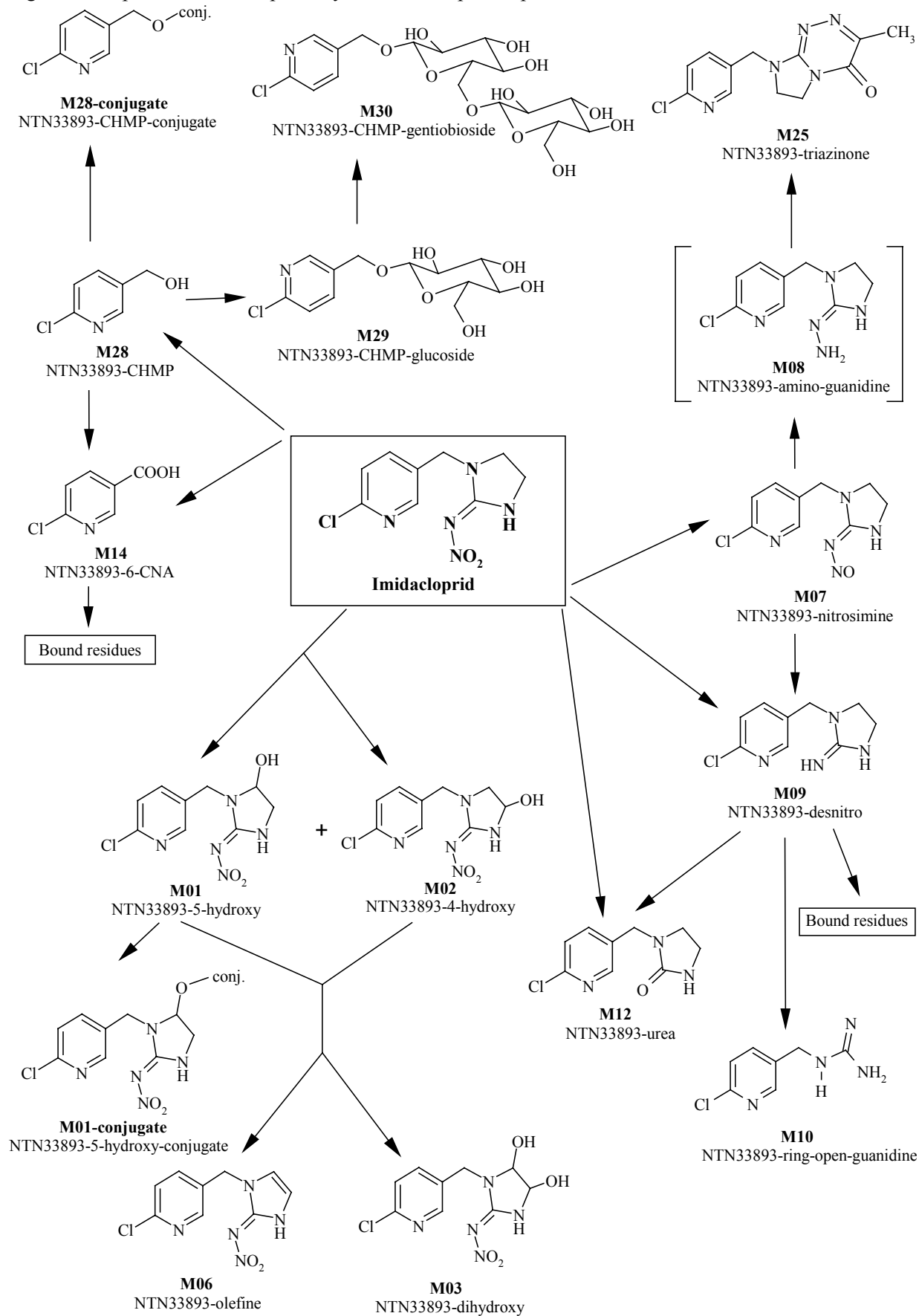
The proportions of unchanged parent compound depended on the application method. After spraying, penetration through the peel into fruits or leaves was slow and so was the degradation of imidacloprid (half-life in potato vines and tomato fruits 5-7 weeks), and the unchanged parent compound represented up to 88% of the TRR. Uptake by roots after soil application led in most cases to more intensive degradation and lower levels of unchanged imidacloprid.

Table 36. Combined results of metabolism studies on various crops after spray, granular or seed applications of [methylene-¹⁴C]imidacloprid.

	¹⁴ C, % of TRR													
	Spray				Granular						Seed			
	toma- toes	apples 14 d	potatoes 64 d	tobac-co 14 d ¹	egg plant 69 d	potatoes 129 d	rice 79 d	rice 124 d	maize 134 d	cotton 211 d				
	fruit	fruit	leaves tubers	leaves	leaves fruit	leaves tubers	straw grain	straw grain	straw grain	leaves				
Ai imidacloprid	88.0	69.0	37.9 ~6	77.7	10.2 18.9	26.7 48.3	11.5 6.3	8.1 13.6	22.2 25.2	2.9				
M01 5-hydroxy	1.8	2.7	7.0	4.0	3.6 3.2	4.6 8.0	1.5	0.9 3.7	5.0 9.3					
M01 conjugate									~1					
M02 4-hydroxy				1.1										
M03 dihydroxy		1.1	2.7			0.3			0.5 4.4					
M06 olefin	0.5	5.7	2.5	1.0	1.3 0.2	3.3 3.1	0.5	0.1 2.3	2.2 13.1	1.5				
M07 nitrosimine	0.7	0.7	2.2	1.1	0.3 0.1	2.6	1.1	0.9 0.2	1.8	1.4				
M09 denitro	2.6	2.2	12.6	5.7	24.6 14.0	8.2 11.3	25.6	36.2 2.2	10.9 2.0	9.8				
M10 guanidine				0.7					1.6					
M12 2-ketone	1.9	1.7		2.1	0.1		0.6	0.4	trace					
M14 6-CNA			~30	0.9	0.9 13.4	8.3 9.4	2.1 2.7	0.5 2.6	1.3 trace	2.2				
M25 triazinone			trace											
M28 CHMP									1.1 4.4	1.9				
M28 conjugate										11.3				
M29 CHMP glucoside	0.1	2.2	1.9	0.4	5.6 13.0	1.4				6.3				
M30 CHMP gentiobioside	0.8													
Extracted radioactivity	99.6	97.0	85.9 82.2	97.7	90.7 97.5	73.6 93.6	56.6 19.3	60.2 31.1	67.9 73.8	73.2				
Total identified	96.4	85.5	66.8 36	94.7	46.6 62.7	55.4 80.1	42.9 9.0	47.1 24.6	47.6 58.4	37.3				
Unextractable	0.4	3.0	14.1 11.8	2.3	9.3 6.5	26.4 6.4	43.4 80.7	39.8 68.9	32.1 26.2	26.8				
Total residue (mg/kg)	0.85	1.45	1.35 0.009	10.20	1.42 0.043	5.76 0.091	1.47 0.036	1.31 0.014	3.08 0.04	0.11				

¹ Two spray applications and one granular application.

Figure 3. Proposed metabolic pathways of imidacloprid in plants.



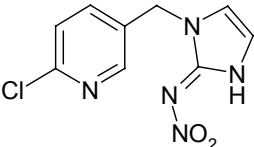
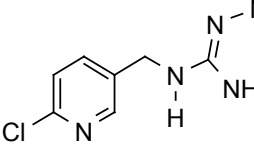
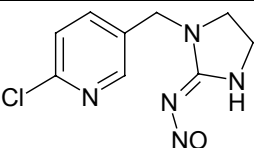
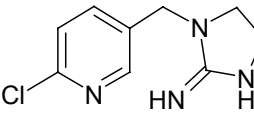
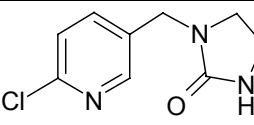
Environmental fate in soil

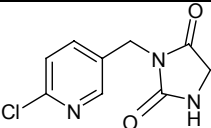
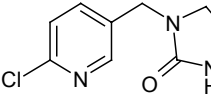
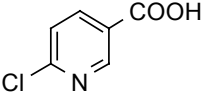
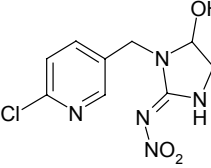
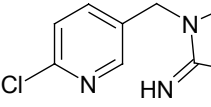
Degradation

In two laboratory studies on aerobic degradation [methylene-¹⁴C]imidacloprid was applied at rates corresponding to 0.2 kg ai/ha (Anderson *et al.*, 1990a,b). Seven metabolites were identified in the soil extracts (Table 36).

The resulting general patterns of degradation were confirmed in two further studies (Anderson and Fritz, 1990a,b). Two further degradation products (see also Table 37) were discovered in a trial with ground cover chosen to counteract the decrease of microbial activity observed in other trials (Scholz, 1992). None of the nine products accounted for more than 10% of the applied radioactivity, the highest in a single component was 3.3% corresponding to concentrations in the soil below 0.01 mg/kg. The sum of all products never exceeded 6%. As the main component in the soil was always the parent compound the first step towards mineralization would seem to be overall rate controlling, in which case most of the intermediates would be degraded faster than imidacloprid and would therefore only be at trace concentrations. In the proposed pathways shown in Figure 4 the denitro-olefin M23 is included although it has only been found in field dissipation studies in northern America (Formella and Cink, 1997; Philpot and Yen, 1998).

Table 37. Degradation products of imidacloprid identified in soil degradation trials.

Product name	Structure	Max. concentration	Reference
M06 olefin		1.8% at day 100 ¹	Anderson <i>et al.</i> , 1990a
		1.1% at day 274 ¹	Anderson <i>et al.</i> , 1990b
M11 nitroguanidine		1.8% at day 100 ²	Anderson <i>et al.</i> , 1990a
		1.6% at day 274 ²	Anderson <i>et al.</i> , 1990b
		1.74 at day 201	Scholz, 1992
M07 nitrosimine		0.8% at day 35	Anderson <i>et al.</i> , 1990a
		1.0% at day 366	Anderson <i>et al.</i> , 1990b
		1.3% at day 56	Scholz, 1992
M09 denitro		1.8% at day 100	Anderson <i>et al.</i> , 1990a
		0.4% at day 100	Anderson <i>et al.</i> , 1990b
		3.3% at day 201	Scholz, 1992
M12 2-ketone		0.3% at day 62	Anderson <i>et al.</i> , 1990a
		0.4% at day 120	Anderson <i>et al.</i> , 1990b

Product name	Structure	Max. concentration	Reference
M33 2,5-diketone		1.8% at day 100 ²	Anderson <i>et al.</i> , 1990a
		1.6% at day 59 ²	Anderson <i>et al.</i> , 1990b
M34 2,4-diketone		1.8% at day 100 ¹	Anderson <i>et al.</i> , 1990a
		1.1% at day 274 ¹	Anderson <i>et al.</i> , 1990b
M14 6-chloronicotinic acid		1.0% at day 56	Scholz, 1992
M01 5-hydroxy		0.28 at day 201	Scholz, 1992
M23 denitro-olefin		Only found in field dissipation studies in northern America	Formella and Cink, 1997
			Philpot and Yen, 1998

¹ Sum of M34 and M06 as not separated analytically

² Sum of M11 and M33 as not separated analytically.

The laboratory studies investigating pathways of degradation of imidacloprid in soil were also used for the calculation of degradation rates. Half-lives ranged from 48 days to more than a year (Table 38).

Table 38. Rates of degradation of imidacloprid in soil calculated from laboratory trials.

Reference; Report	Applic. rate (g ai/ha)	Soil	Organic carbon (%)	DT-50 (best fit statistics) (d)	DT-50 ¹ 1st order (non-linear) (d)
Anderson <i>et al.</i> , 1990a PF3321	200	Loamy sand ("BBA 2.2")	2.15	188 (2nd order)	154 ⁵
Anderson <i>et al.</i> , 1990b PF3433	200	Sandy loam (Kansas ²)	1.40	>1 year ² (sq. root 1st order)	>1 year ²
Anderson and Fritz, 1990a; PF3322	200	Silt soil	1.23	248 (2nd order)	193 ⁵
Anderson and Fritz, 1990b; PF3434	200	Sandy loam ("Monheim 1")	1.31	341 (sq. root 1st order)	186 ⁵
Hellpointner, 1999 MR-389/99	117	Sandy loam ("Laacherhof AXXa")	1.41	77 (sq. root 1st order)	106 ⁵
				geometric mean	156
				arithmetic mean	160
				standard deviation	40
Scholz, 1992 PF3438	200	Loamy sand (BBA 2.2) bare soil	2.15 + organic manure	190 ⁴ 204 (1.5th order)	193 ³
Scholz, 1992 PF3438	200	Loamy sand (BBA 2.2) cropped soil	2.15 + organic manure	48 (1.5th order)	69 ³

¹ Rationale for re-calculation: see Krohn, 2002.

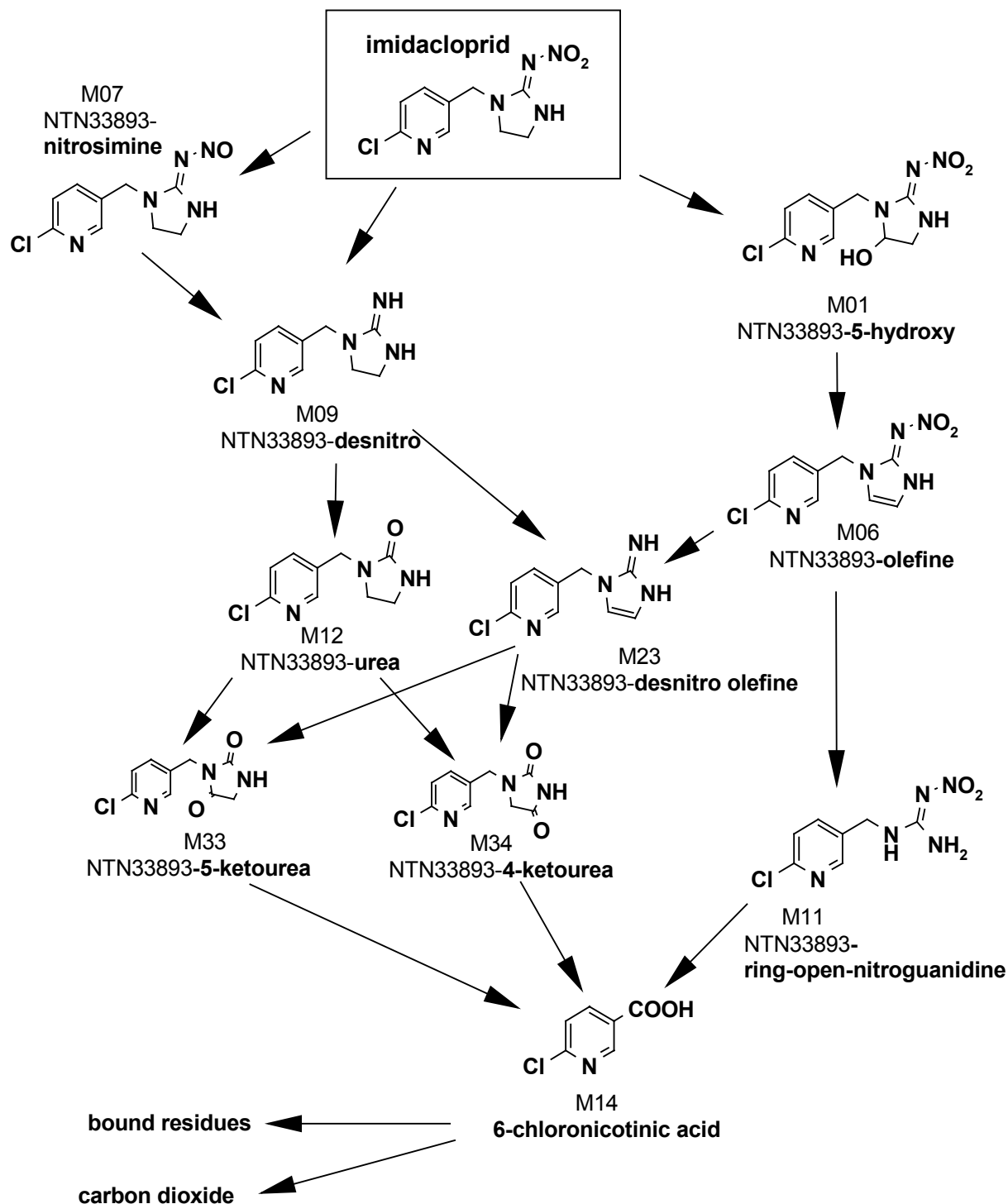
² Microbial activity impaired by improper conditioning during shipment to test facility at Monheim (Germany), value regarded as an outlier by the author.

³ According to first-order kinetics after logarithmic transformation.

⁴ Graphical estimation from three data points according to best-fit (1.5th order) gave negative confidence interval.

⁵ Recalculation by Fahl and Leicht, 2001.

Figure 4. Pathways of degradation of imidacloprid in soil.



Degradation under field conditions

Degradation rates obtained in laboratory studies were confirmed in field trials in northern Europe for 12 months and southern Europe for 24 months (Tables 39 and 40).

Table 39. Rates of degradation of imidacloprid in soil in field trials in Germany.

Reference, Report, Trial	Schedule	Applic. rate (kg ai/ha)	Soil, location	Organic carbon (%)	DT-50 best fit statistics (d)	DT-50 ¹ 1st order, non-linear (d)
Bachlechner, 1992 RA-2082/91 Trial 10490/6	Apr. 24, 1991-Apr. 18, 1992	0.26	Sandy loam Kirchlauter-Pettstadt	0.79	180 sq. root 1st order	216 ²
Bachlechner, 1993a RA-2130/91 Trial 10362/4	Apr. 24, 1991-Apr. 18, 1992	0.12	Sandy loam Kirchlauter-Pettstadt	0.79	142 sq. root 1st order	178 ²
Bachlechner, 1992 RA-2082/91 Trial 10491/4	May 01, 1991-May 31, 1992	0.26	Silt loam Swisttal-Hohn	1.0	140 sq. root 1st order	185 ²
Bachlechner, 1993a RA-2130/91 Trial 10359/4	May 01, 1991-May 31, 1992	0.12	Silt loam Swisttal-Hohn	1.0	173 sq. root 1st order	208 ²
Bachlechner, 1992 RA-2082/91 Trial 10492/2	Apr. 1, 1991 - Apr. 18, 1992	0.26	Sandy loam Maasen	2.22	not evaluated owing to poorly correlated experiment data	
Bachlechner, 1993a RA-2130/91 Trial 10360/8	Apr. 1, 1991 - Apr. 18, 1992	0.12	Sandy loam Maasen	2.22	not evaluated owing to poorly correlated experiment data	
Bachlechner, 1992 RA-2082/91 Trial 10493/0	May 1, 1991 - May 31, 1992	0.26	Silt loam Höfchen	1.11	79 sq. root 1.5th order	131 ²
Bachlechner, 1993a RA-2130/91 Trial 10361/6	May 1, 1991 - May 31, 1992	0.12	Silt loam Höfchen	1.11	62 sq. root 1st order	104 ²
Bachlechner, 1992 RA-2082/91 Trial 10494/9	Apr. 1, 1991 - Apr. 30, 1992	0.26	Loam Worms-Heppenheim	1.56	196 sq. root 1st order	228 ²
Bachlechner, 1993a RA-2130/91 Trial 10363/2	Apr. 1, 1991 - Apr. 30, 1992	0.12	Loam Worms-Heppenheim	1.56	151 sq. root 1.5th order	197 ²
Bachlechner, 1992 RA-2082/91 (Trial 10495/7)	May 1, 1991 - May 31, 1992	0.26	Sandy loam Laacher Hof	1.27	160 sq. root 1st order	186 ²
Bachlechner, 1993a RA-2130/91 Trial 10364/0	May 1, 1991 - May 31, 1992	0.12	Sandy loam Laacher Hof	1.27	119 sq. root 1st order	152 ²
					geometric mean	174
					arithmetic mean	179
					standard deviation	39

¹ Rationale for re-calculation: see Krohn, 2002.² Recalculation by Fahl and Leicht, 1999.

Table 40. Rates of degradation of imidacloprid on bare soil (southern Europe).

Reference Report, Trial	Applic. rate (kg ai/ha)	Soil, location	Organic carbon (%)	DT-50 best fit statistics (d)	DT-50 ¹ 1st order, non-linear (d)
Sommer, 1998a RA-2084/95, Trial R506230	0.15	Clay loam Bagnolo di Naga-role Rocca (Italy)	1.17	183 sq. root 1.5th	288 ²
Sommer, 1998a RA-2084/95, Trial R506249	0.15	Loamy silt St. Etienne du Gres (France)	0.71	63 sq. root 1st	111 ²
Sommer, 1998b RA-2107/96, Trial R607061	0.15	Loamy sand Ca Degli Oppi (Italy)	0.67	28 sq. root 1st	40 ²
Sommer, 1998b RA-2107/96, Trial R607088	0.15	Silty clay loam Castellarnau (Spain)	0.96	77 sq. root 1st	116 ²

Reference Report, Trial	Applic. rate (kg ai/ha)	Soil, location	Organic carbon (%)	DT-50 best fit statistics (d)	DT-50 ¹ 1st order, non-linear (d)
				geometric mean	110
				arithmetic mean	139
				standard deviation	105

¹ Rationale for re-calculation: see Krohn, 2002.

² Recalculation by Fahl and Leicht, 1999.

Microbial activity in the soil and the varying bioavailability of imidacloprid may explain the scattering of dissipation rates. However under normal agricultural conditions imidacloprid will be of medium persistence with half-lives generally less than 180 days. Organic fertilizers may enhance microbial activity and thus stimulate degradation or retard it owing to adsorption and absorption of the ai. Which predominates depends on other factors hard to control.

In laboratory studies by Scholz, 1992 (Table 38) vegetation reduced the half-life considerably. Sugar beet pellets were dressed with imidacloprid and planted in two sites in Germany (Bachlechner, 1993b). Except for the application method and the presence of a crop, the experiment was similar to bare soil experiments run in the area. Half-lives at the two sites were 83 and 124 days whereas they were 185 and 186 days in the corresponding bare soil trials (Table 41).

Table 41. Influence of crops on degradation rates of imidacloprid, comparison of results from bare and cropped soil in Germany (Bachlechner 1992, 1993b).

Reference	Soil Trial location	Organic carbon (%)	Applic. rate (kg ai/ha)	DT-50, days 1st order (non-linear)
<i>Cropped soil</i>				
Bachlechner, 1993b RA-2081/91, Trial 10546/5	Silt loam Swisttal-Hohn	1.72	0.093	83
Bachlechner, 1993b RA-2081/91, Trial 10547/3	Sandy loam Monheim-Laacherhof	2.18	0.085	124
<i>Bare soil</i>				
Bachlechner, 1992a RA-2082/91, Trial 10491/4	Silt loam Swisttal-Hohn	1.72	0.12	185
Bachlechner, 1992a RA-2082/91, Trial 10495/7	Sandy loam Monheim-Laacherhof	2.18	0.12	186

In field dissipation trials when imidacloprid was applied to bare or cropped soil the DT-90 was over a year. Therefore, long-term field trials with repeated single annual applications of imidacloprid to winter barley seed over several years and at two different locations (Bury St Edmunds and Wellesbourne, UK) were conducted by Placke (1998a) at application rates of 0.035 and 0.07 kg ai/100 kg seed. When the dressed seed was analysed it contained about 71-96% of the theoretical amount. Residues in the 0-30 cm soil layer increased gradually over three years of the trial, and after the 4th reached a plateau and remained constant. The maximum residue was 0.044 mg/kg (Wellesbourne) and 0.049 mg/kg (Bury St Edmunds) determined in the 4th year with the high application rate.

Mobility

Three studies were conducted to determine the adsorption/desorption behaviour of imidacloprid in soil as a measure for its mobility and its potential for leaching into ground water (Fritz, 1988, 1993; Williams *et al.*, 1992). From the Freundlich adsorption/desorption isotherms of ten different soils each treated at four concentrations the resulting adsorption constants K_d and adsorption coefficients $1/n$ were calculated. The results are summarised in Table 42. Taking into account the organic carbon content of the respective soil the K_d values were normalised and expressed as K_{oc} values also included in Table 42. With an average K_{oc} value of 242 imidacloprid would be classified as having medium mobility in soil.

Table 42. Adsorption constants for 10 soils.

Soil (Reference)	Organic carbon (%)	Adsorption K_d (ml/g)	Adsorption K_{oc} (ml/g)	Adsorption $1/n$
Sand (Williams <i>et al.</i> , 1992)	0.233	0.956	411	0.781
Loamy sand (Williams <i>et al.</i> , 1992)	0.349	1.02	292	0.877
Loam (Williams <i>et al.</i> , 1992)	1.16	3.45	296	0.758
Silt loam (Williams <i>et al.</i> , 1992)	1.51	4.18	277	0.775
Silty clay (Fritz, 1988)	0.64	1.36	212	0.851
Sand (Fritz, 1988)	0.75	1.17	157	0.777
Silt (Fritz, 1988)	1.80	2.38	132	0.827
Sandy loam (Fritz, 1988)	1.40	3.59	256	0.744
Borstel (Fritz, 1993)	1.20	1.83	153	0.888
Laacher Hof (Fritz, 1993)	1.35	3.17	235	0.782
mean $\pm$ SD (n = 10)			242 $\pm$ 84	0.806

In laboratory column leaching experiments by Koenig (1990a-f) applications of Gaucho 70 WS at 0.17 kg ai/ha and Confidor 70 WG at 0.52 kg ai/ha made onto columns packed with standard soils BBA 2.1, BBA 2.2 and BBA 2.3 did not result in measurable concentrations of imidacloprid in the leachates.

Leaching experiments with aged residues were conducted according to German and Dutch guidelines (Fritz and Brauner, 1988a) and US EPA guidelines (Fritz and Brauner, 1988b). In the former labelled imidacloprid was aged in sand soil BBA 2.1 for 30 and 90 days and in sandy loam Monheim 1 for 90 days, and the soils with aged residues were added to the tops of the corresponding soil columns. Approximately 400 ml of the leachate over 48 hours was collected in fractions. At the end the radioactivity in the leachates and down the columns was measured. The results are shown in Table 43. A maximum of 0.3% of the applied radioactivity was in the leachate and a maximum of 30% had been leached into the middle third of the 2.1 soil column. In the case of Monheim 1 with a higher percentage of organic carbon the respective concentrations were much lower.

Table 43. Column leaching of imidacloprid after ageing on soil (Fritz and Brauner, 1988a).

Fraction	¹⁴ C, % of applied		
	BBA Standard Soil 2.1 sand 88, silt 8.7, clay 3.5%, organic carbon 0.75%		Monheim 1 sand 59, silt 28, clay 13%, organic carbon 1.27%
	30 days	90 days	90 days
Column			
upper third	71	71	98
middle third	29	21	1.2
lower third	0.4	0.5	<0.1
Mineralization	1.8	7.2	2.4
Leachate	0.3	0.2	<0.1
Recovery	102.5	99.9	101.6

In the experiments conducted according to EPA guidelines Monheim 1 was loaded with labelled imidacloprid and incubated for 30 days before it was added to the top of the soil column and eluted with 1 l of a solution containing 0.01% calcium chloride over a 30-day period. Approximately 96% of the radioactivity was found in the upper third of the column, about 0.4% in the bottom third and 0.14% in the entire leachate. The results (Table 44) indicate that imidacloprid and its degradation products are slightly mobile in soil.

Table 44. Leaching of imidacloprid after ageing in Monheim 1 soil. Figures are the mean from two columns (Fritz and Brauner, 1988b).

Sand 59%; silt 28%; clay 13%; organic carbon 1.27%	30 days of ageing, % of applied radioactivity
Aged soil layer	48.5
0-5 cm of column	37.0
5-10 cm of column	10.8
10-15 cm of column	4.2
15-20 cm of column	1.8
20-25 cm of column	0.3
25-30 cm of column	0.1
Leachate	0.14
Recovery	104.7

This mobility was further confirmed by three lysimeter trials conducted under field conditions according to German practice by Hellpointner (1994, 1998, 2001, Table 45).

Table 45. Results of imidacloprid lysimeter trials.

Date	Operation	¹⁴ C in leachate ¹			
		year	volume	TRR ²	ai
<i>Trial PF 3952 (Hellpointner, 1994)</i>					
May 1990	seeding of potatoes treated with 0.52 kg ai/ha				
Nov. 1990	sowing of winter wheat	1st	95 l	0.04 µg/l	<0.01 µg/l
Sep. 1991	sowing of winter barley	2nd	99 l	0.08 µg/l	<0.01 µg/l
<i>Trial PF 4198 (Hellpointner, 1998)</i>					
		year	volume	TRR _{acid} ³	ai
Apr. 1991	sowing of sugar beet treated with 0.12 kg ai/ha				
Nov. 1991	sowing of winter wheat				
Sep. 1992	sowing of winter barley	1st	98 l	0.02 µg/l	<0.01 µg/l
Aug. 1993	sowing of phacelia (green manure)	2nd	225 l	0.06 µg/l	<0.01 µg/l
Apr. 1994	sowing of sugar beet treated with 0.13 kg ai/ha	3rd	301 l	0.07 µg/l	<0.01 µg/l
Oct. 1994	sowing of winter wheat	4th	357 l	0.08 µg/l	<0.01 µg/l
Oct. 1995	sowing of winter barley	5th	102 l	0.08 µg/l	<0.01 µg/l
<i>Trial MR-466/99 (Hellpointner, 2001)</i>					
		year	volume	TRR _{acid} ³	ai
Apr. 1996	sowing of sugar beet treated with 0.14 kg ai/ha				
Sep. 1996	harvest of sugar beet				
Oct. 1996	sowing of winter wheat treated with 0.13 kg ai/ha				
Aug. 1997	harvest of winter wheat	1st	140 l	0.03 µg/l	<10 ⁻³ µg/l
Sep. 1997	sowing of winter barley				
Jul. 1998	harvest of winter barley	2nd	126 l	0.04 µg/l	<10 ⁻³ µg/l
Oct. 1998		3rd	34 l	0.06 µg/l	<10 ⁻³ µg/l

¹ averages of two lysimeter trials.

² as imidacloprid.

³ TRR after acidification to remove CO₂ as imidacloprid. Difference between TRR and TRR_{acid} in all fractions <20% of TRR.

⁴ Organic extracts contained approx. 20% of radioactivity, thus a maximum of 20% was to be expected to have properties similar to parent substance.

⁵ Determined in the mixed leachate from the last year which had the highest TRR_{acid}.

In the first trial which ran for 2 years potatoes were treated at a relatively high rate corresponding to 0.52 kg ai/ha, in the second and third, running over five years and two and a half years respectively the imidacloprid was applied at rates from 0.12 to 0.13 kg ai/ha as a seed dressing each year. The results from the annual leachates showed that the mean acid TRR (acidified, carbon dioxide removed, calculated as imidacloprid) were always below 0.1 µg ai equivalent/l. Neither parent compound nor any known degradation product could be detected by radio-TLC analysis at a detection limit of 0.01 µg/l, nor was the active ingredient or any product translocated significantly into the subsoil for at least two years after the last application. The depth profile of the concentrations of

residues in the soil was in line with the analysis of the leachates. Over 80% of the radioactivity was recovered in the 0–30 cm layer of the soil so the deeper layers had hardly been penetrated.

Photolysis

In a US laboratory study in Kansas [methylene-¹⁴C]imidacloprid was applied to a sandy loam soil and irradiated continuously for 15 days with a xenon lamp (Yoshida, 1990). Samples were analysed at 0 and 6 hours and on days 1, 2, 3, 5, 7, 12 and 15. 91.6% of the applied radioactivity was recovered. Degradation of the parent compound had occurred with an extrapolated half-life of 39 days, considerably shorter than that calculated in laboratory degradation studies without irradiation. Five products, all also occurring in the absence of light, in total accounted for 13.4% of the applied radioactivity. The maximum level of an individual photoproduct was 6.3%.

Summary

Under normal agricultural conditions imidacloprid shows medium persistence to bio-degradation with half-lives generally below 180 days. It is completely mineralized without the occurrence of any degradation product at concentrations above 10%. Degradation on soil surfaces can play an important role in the environmental dissipation of imidacloprid. The compound exhibits a low soil mobility with a negligible leaching potential.

Environmental fate in water-sediment systems

Hydrolysis. Yoshida (1989) found imidacloprid to be stable in buffered solutions at pH 5 and 7 at 25°C when stored in the dark, and that it degraded slowly at pH 9 with a half-life of approximately 1 year.

Photolysis. The quantum yield for imidacloprid in pure water was determined according to the ECETOC method (polychromatic light) from the UV absorption data and kinetic results of two photodegradation experiments to be 0.0142 (Hellpointner, 1989). The half-life for direct photolysis in aqueous solutions was calculated from this to be between 0.17 days in summer and 1.6 days in winter for the 50° latitude. The importance of photodegradation for the overall dissipation of imidacloprid is thus evident.

The calculations were confirmed in a photolysis study in which aqueous solutions of [methylene-¹⁴C]imidacloprid buffered at pH 7 were irradiated with artificial sunlight under sterile conditions (Anderson, 1988). Degradation was rapid with a half-life of 57 min., corresponding to 4.2 hours under environmental conditions when the irradiation spectrum of the lamp and the changing light intensity in the natural night and day cycle were taken into account. Several photoproducts were formed, of which the main three were the denitro M09, denitro-olefin M23 and 2-ketone M12, which together represented more than 10% of the applied radioactivity:

Aquatic microbial metabolism. The degradation of [methylene-¹⁴C]imidacloprid in aquatic micro-ecosystems was investigated in two water-sediment systems in The Netherlands (Wilmes, 1988) and with one in Kansas, USA (Spiteller, 1993), and in a further study the stimulating influence of light was demonstrated (Henneböle, 1998).

In The Netherlands in a system from a recultivated gravel quarry (“Lienden”) the half-life of imidacloprid was 162 days in contrast to 30 days in a system from an orchard drainage ditch (“Ijzendorp”). In both systems the denitro M09, PEDA M22 and CNA M14 were identified at concentrations between 1% and 6.3% of the applied radioactivity (Table 46). Further trace components were detected, but the only one identified was the nitrosime M07.

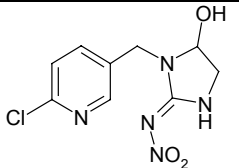
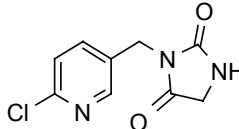
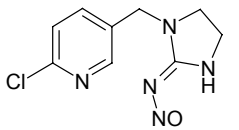
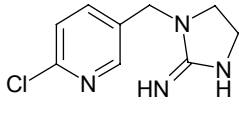
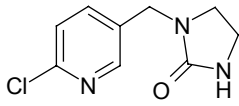
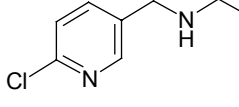
Comparison of the results in the Lienden and Ijzendorp systems shows that the rapid disappearance of radioactivity from the water phase occurring in the latter was a consequence of the

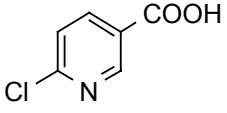
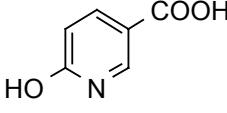
formation of unextractable residues. Complete mineralization by formation of carbon dioxide was observed throughout the test although its proportion amounted only to 1.4% in the former and 2% in the latter at day 92. In both systems the decrease of the concentration of parent compound in the water phase did not produce higher concentrations of parent compound in the sediment or higher concentrations of any degradation product in either phase.

In a pond water-sediment system in an agricultural area in Stilwell, Kansas, USA (Spiteller, 1993) the applied substance decreased in the water phase from an initial 90.7% to 64% after 30 days of incubation and 0.7% of carbon dioxide was formed. The half-life according to pseudo-first order kinetics was 129 days. The maximum concentration was 2.4% of any degradation product, which was the 5-hydroxy M01 in the water phase at day 7. The nitrosime M07, 2-ketone M12 and 6-hydroxynicotinic acid M18 accounted for 0.9% at day 21, 0.9% at day 30 and 0.7% at day 21 in the water phase respectively. The denitro M09, PED A M22 and 6-CNA M14 identified in the previous study could not be detected. These differences may be because all degradation products occurred only as minor or trace components and none was sufficiently persistent to accumulate. Both sets of products are included in Figure 5.

Under the influence of sunlight the degradation of imidacloprid is considerably faster as Henneböle (1998) demonstrated with a water-sediment system similar to that used by Spiteller (1993). Mineralization rates after 21 days were 5.8% with sunlight irradiation and 9.8% with xenon light. An additional product not found in the previous studies was the diketone M33.

Table 46. Metabolites of imidacloprid identified in water-sediment systems.

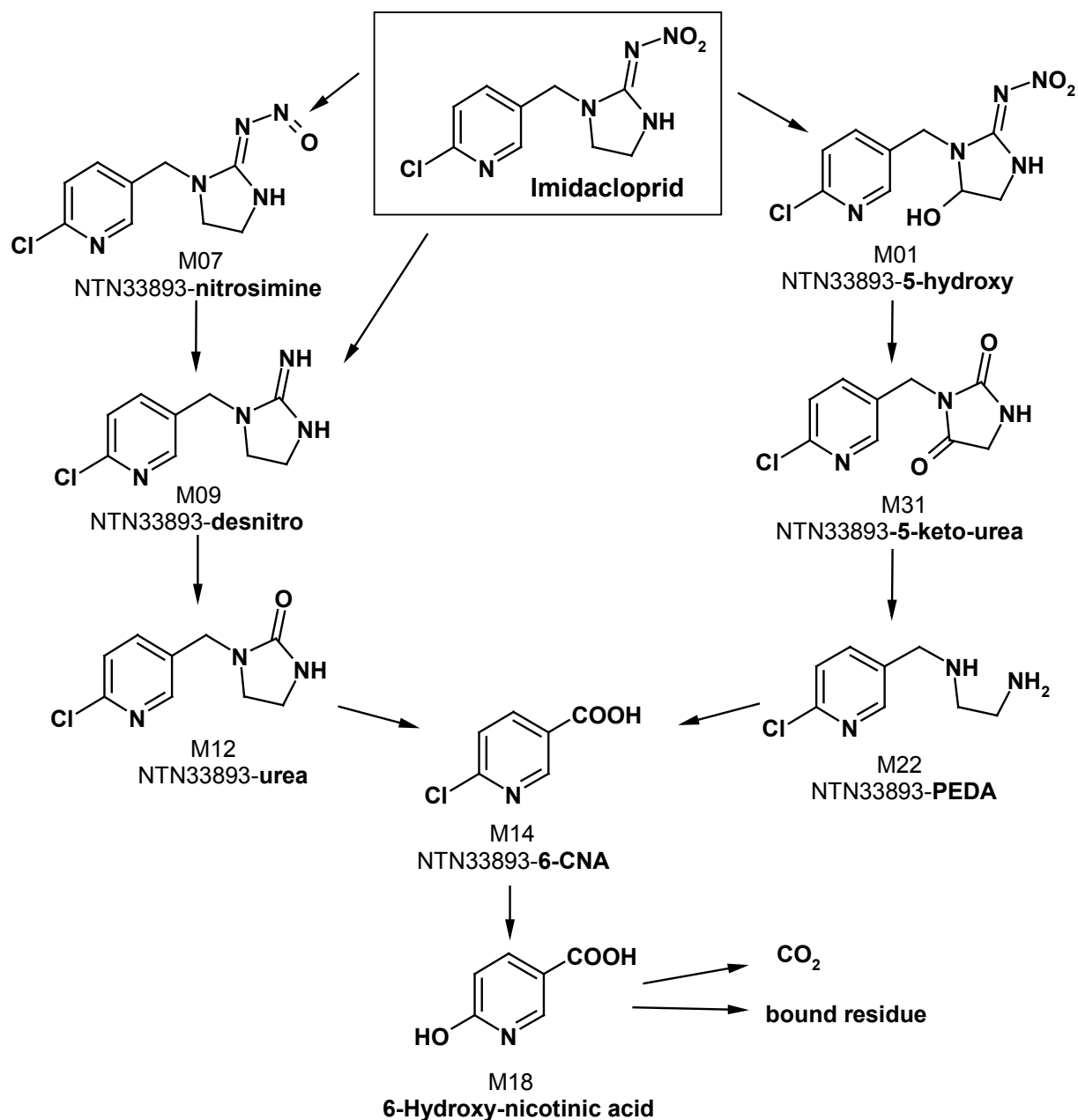
Compound	Structural formula	Maximum concentration (% of applied amount)	Location, system (Reference)
M01 5-hydroxy		2.4 0.4 0.2 0.8	Stilwell, water Stilwell, sediment (Spiteller, 1993) Stilwell (sunlight), water Stilwell (sunlight), sediment (Henneböle, 1998)
M33 2,5-diketone		0.5 0.1	Stilwell (sunlight), water Stilwell (sunlight), sediment (Henneböle, 1998)
M07 nitrosimine		0.9 traces	Stilwell, water (Spiteller, 1993) Lienden, water (Henneböle, 1998)
M09 denitro		4.5 4.3 6.0 6.3 1.3	Lienden, water Lienden, sediment Ijzendorf, water Ijzendorf, sediment (Wilmes, 1988) Stilwell (sunlight), water (Henneböle, 1998)
M12 2-ketone		0.9 3.2 0.8	Stilwell, water Stilwell (sunlight), water Stilwell (sunlight), sediment (Henneböle, 1998)
M22 PEDA		2.3 <0.1 0.4 <0.1	Lienden, water Lienden, sediment Ijzendorf, water Ijzendorf, sediment (Wilmes, 1988)

Compound	Structural formula	Maximum concentration (% of applied amount)	Location, system (Reference)
M14 6-chloronicotinic acid		3.8 0.7 0.4 9.6 0.3	Lienden, water Lienden, sediment Ijzendorp, water Ijzendorp, sediment (Wilmes, 1988) Stilwell (sunlight), water Stilwell (sunlight), sediment (Hennebøle, 1998)
M18 6-hydroxynicotinic acid		0.7 <0.1 1.0 0.1	Stilwell, water Stilwell, sediment Stilwell (sunlight), water Stilwell (sunlight), sediment (Hennebøle, 1998)

Summary of environmental fate in water/sediment systems

Imidacloprid is stable to hydrolysis in aqueous solutions at environmentally relevant pH values. In contrast, photolytic degradation occurs rapidly owing to the nitro chromophore. Although generally the photolytic effect is less important under environmental conditions since light of the relevant wavelengths (>290 nm) will be absorbed by turbidities and impurities to a certain degree, in the case of imidacloprid it must be taken into account. In water-sediment systems the proportion taken up by the sediment and converted into unextracted residues can become large though this is not generally the case. Calculated half-lives in the three systems investigated were 30, 129 and 162 days. Complete mineralization occurs slowly but steadily and there is no tendency of any of the intermediates to accumulate.

Figure 5. Proposed degradation pathways of imidacloprid in aquatic model ecosystems (Wilmes, 1988; Spiteller, 1993; Henneböle, 1998).



RESIDUE ANALYSIS

Analytical methods

A modification of a method describes changes in the analytical procedure. A supplement is written when the method is validated for use with additional sample materials or crops, but without changes to the analytical procedure.

Enforcement methods

Plant materials. Method no. 00300 (Placke and Weber, 1993) can be used for the determination of both the parent compound and the total residue. Residues are extracted with methanol and water and diluted sulfuric acid. The extract or an aliquot is evaporated to the aqueous remainder. Fat- or wax-containing samples are partitioned with hexane. The extract is further cleaned up by column chromatography with XAD 4 (polystyrene resin). The residues containing the 6-chloropyridyl moiety are oxidized to 6-chloronicotinic acid with alkaline KMnO_4 solution. Subsequently the 6-chloronicotinic acid is derivatized with *N*-methyltrimethylsilyltrifluoroacetamide (MSTFA), and determined by gas chromatography with mass selective detection (GC-MS). Mean recoveries of the total residue for each sample material and fortification level ranged from 68 to 113% ($n=152$). The overall mean recovery was 89%, with a relative standard deviation (RSD) of 8.6%. In all samples blank values were normally below 30% of the corresponding LOQ.

For the determination of the parent compound, an aliquot of the extract is evaporated to the aqueous remainder. After clean-up on a ChemElut[®] cartridge and chromatography on Florisil, the residues are determined by HPLC with UV detection. Mean recoveries of the parent compound for each sample material and fortification level were in the range of 72 to 114% ($n=143$). The mean recovery was 91%, with an RSD of 8%. Blank values were normally below 30% of the corresponding LOQ. Recoveries from selected crops are shown in Table 47.

Table 47. Recoveries of imidacloprid and total residues from diverse crops after fortification with imidacloprid and various metabolites (Placke and Weber, 1993).

Crop	No. of samples at level (mg/kg)	Recoveries / RSD (%)				Recoveries (%)			
		Imidacloprid		Total residue		Metabolites as specified in <i>italics</i> in total residue			
		Mean	RSD	Mean	RSD	Mean	Mean	Mean	Mean
Apple, fruit <i>WATERY crop</i>	2 × 0.01 2 × 0.05 3 × 0.1 2 × 0.5	98 86	0.7	109 89		106 [$n=1$] (M12)	106 (M03)	98 83 (M09)	
Pear, fruit <i>WATERY crop</i>	4 × 0.01 4 × 0.05 3 × 0.1 4 × 0.5	91 87	9.2 5.9	87 82	7.6 3.2	91 [$n=2$] (M14)			
Cotton, seed <i>OILY crop</i>	3 × 0.01 3 × 0.02 4 × 0.05 3 × 0.1 3 × 0.5	79 78	4.4 1.5	83 75	3.2 5.1	80 [$n=2$] (M12)	88 [$n=2$] (M03)	79 (M06)	81 (metab. mix ¹)
Barley, grain <i>DRY crop</i>	3 × 0.01 2 × 0.05 3 × 0.1 2 × 0.5	89 90	7.4 4.2	83 ² 82					
Wheat, grain <i>DRY crop</i>	3 × 0.01 2 × 0.05 3 × 0.1	91 86	3.5 3.5	78 ²		86 [$n=2$] (M06)			
Citrus, pulp <i>ACIDIC crop</i>	3 × 0.01 3 × 0.05 3 × 0.1 3 × 0.5	84 86	3.7 6.4	88 85	6.9 6.1				

Crop	No. of samples at level (mg/kg)	Recoveries/RSD (%)				Recoveries (%)			
		Imidacloprid		Total residue		Metabolites <i>as specified in italics</i> in total residue			
		Mean	RSD	Mean	RSD	Mean	Mean	Mean	Mean
Cauliflower head	3 × 0.01	108	1.4			94			
	2 × 0.02					71 [<i>n=1</i>]			
	3 × 0.05	102	20.8	91	2.8		86	81	82
	2 × 0.1								
	3 × 0.5			82	4.9	(M12)	(M06)	(M09)	(M14)

M12 NTN33519: 2-ketone M03 (WAK 3772): dihydroxy compound, M06 (NTN 35884): olefin M09 (WAK 4140): denitro compound M14 (6-CNA): 6-chloronicotinic acid

¹ 6-CNA and WAK 4140, each spiked at specified level.

² Recoveries from wheat and barley grain calculated together because similar: parent: 6 × 0.01, mean recovery 90%, RSD 5.3% total residue: 4 × 0.05, mean recovery 80%, RSD 6.4%

In an independent laboratory study to validate method 00200 and its modification 00200/M002 method 00300 was also validated (Pelz, 1993). Recoveries of parent and total residues from melon peel and pulp, peppers and tomatoes fortified at 0.01 to 1 mg/kg were determined. For the total residue, individual recoveries ranged from 69-112%, with mean recoveries for each sample material and spike level from 72 to 100%, with typical RSDs of approximately 10%. For the parent compound, individual recovery levels were between 68% and 83%. The mean recovery for each sample material and fortification level ranged from 70 to 79%, with typical standard deviations of about 5%. Blank values were below 30% of the corresponding LOQ in all samples.

Table 48. Recoveries by method 00300 in an independent laboratory of imidacloprid and the total residue of imidacloprid, M09 and M14 (determined as 6-chloronicotinic acid) from various commodities fortified with imidacloprid and a metabolite mixture (Pelz, 1993).

Sample	No. of recoveries at specified level (mg/kg)	Recoveries/RSD (%)					
		Imidacloprid		Total residue		Metabolite mixture ¹ (total residue)	
		Mean	RSD	Mean	RSD	Mean	RSD
Melon, pulp	3 × 0.01	81	7.7				
	3 × 0.05			86	9.4	84	7.4
	3 × 0.1	70	2.8				
	3 × 0.5			72	3.7	72	3.8
Melon, peel	3 × 0.01	78	4.6				
	3 × 0.05			86	15	88	9.8
	3 × 0.1	79	6.7				
	3 × 0.5			93	5.0	85	23.2
Bell pepper, fruit	3 × 0.01	75	5.2				
	3 × 0.05			94	14	85	17
	3 × 0.1	74	7.4				
	3 × 0.5			81	7.6	90	6.6
Tomato, fruit	3 × 0.01	75	0.46				
	3 × 0.05			100	12	99	3.4
	3 × 0.1	72	4.3				
	3 × 0.5			93	9.7	95	6.8

¹ M09 (denitro compound) and M14 (6-chloronicotinic acid), each added at the specified level

Since other choropyridyl insecticides are available world-wide Schöning (1999a) has developed method no. 00573 to differentiate between residues resulting from the use of imidacloprid and of thiacloprid. Imidacloprid, thiacloprid, and two metabolites of each of them (M06 imidacloprid olefin, M01 5-hydroxy-imidacloprid, thiacloprid amide and 4-hydroxy-thiacloprid amide) are extracted with a mixture of methanol and water. After filtration, the extract is cleaned up on a Bond ElutTM ENV cartridge; the aqueous remainder is then partitioned against dichloromethane on a ChemElut[®] column. The residues are then taken up in acetonitrile/water and quantified by reverse-phase HPLC with electrospray MS-MS detection. The method was tested with apple fruit, cotton seed,

and potato tubers. The overall LOQ was 0.02 mg/kg with a limit of detection at least ten times lower. Mean recoveries for each sample and spike level were 87-99% for imidacloprid, 84-99% for imidacloprid olefin, and 86-98% for 5-hydroxy-imidacloprid, with overall rates of 96%, 94%, and 94%, and relative standard deviations of 4.8%, 6.6% and 5.3% respectively. In all samples the blank values were below 20% of the corresponding LOQ.

Animal materials. Residues of imidacloprid and related metabolites can be determined by method 00191 (Weber, 1990a). Samples are extracted with a mixture of methanol and water (methanol only for milk samples), filtered, and evaporated to the aqueous remainder. Fat samples are partitioned with hexane. The extracts are further cleaned up by column chromatography with XAD 4 (polystyrene resin); the column is washed with water and residues are eluted with methanol. Subsequently, imidacloprid and its metabolites containing the 6-chloropyridyl moiety are oxidized with alkaline KMnO_4 to 6-chloronicotinic acid, which is extracted from the aqueous phase with *tert*-butyl methyl ether, derivatized with MSTFA, and then determined by gas chromatography with mass-selective detection. Recoveries ranged from 76 to 124% after spiking bovine muscle, kidney, liver, fat and milk, and eggs with imidacloprid at levels of 0.02 and 0.1 mg/kg. The LOQ was 0.02 mg/kg for all samples.

Method 00191 was modified as described in modification 00191/M001 (Weber and Heukamp, 1993), the chief difference being that after extraction milk samples as well as fat are partitioned with *n*-hexane. Control samples were spiked with imidacloprid and several metabolites at levels of 0.02 to 2.5 mg/kg. Mean recoveries ranged from 71 to 111%, with corresponding recoveries for the metabolites of 61-103%. Typical relative standard deviations for each set of recoveries were approximately 10%. The LOQ was 0.02 mg/kg for all samples (Table 49). This method can also be used to determine residues in body fluids, but it has not yet been validated with sample materials such as blood.

Method 00191 was validated in an independent laboratory by Blass (1992). As the extraction procedures for milk included a hexane extraction step, the study also effectively validated method 00191/M001. Recoveries of a mixture of imidacloprid, the denitro compound M09 and 6-chloronicotinic acid at fortification levels of 0.02 and 0.1 mg/kg in milk and eggs and 0.1 and 0.5 mg/kg in chicken livers ranged from 72 to 97%. No blank values from control samples were observed. The results are shown in Table 49.

Table 49. Recoveries of total residues of imidacloprid (determined as 6-chloronicotinic acid) from animal materials after fortification with imidacloprid and various metabolites.

Method	Sample	No. of samples at level (mg/kg)	Recoveries, determined as 6-chloronicotinic acid (%)							
			Imidacloprid		Metabolite mix ¹		Individual metabolites as specified in italics			
			Mean	RSD	Mean	RSD	Mean	Mean	Mean	Mean
00191/ M001 Weber and Heukamp, 1993	Cow, milk	6 × 0.02	71	12	88	8.3	75 [<i>n</i> =9]	77 [<i>n</i> =10]		
		6 × 0.033	81	11			82 [<i>n</i> =7]	89 [<i>n</i> =9]		
		9 × 0.1					(M06)	(M04)		
	Cow, liver	5 × 0.02	71	3.4	81	4.1	75 [<i>n</i> =3]	85 [<i>n</i> =3]	76 [<i>n</i> =3]	72 [<i>n</i> =3]
		3 × 0.2								
		3 × 0.05	77	11						
		3 × 0.25	80	9.6						
		3 × 0.5	80	3.4			89 [<i>n</i> =7]	85 [<i>n</i> =3]	83 [<i>n</i> =3]	81 [<i>n</i> =3]
		3 × 2.52	76	3.8			(M06)	(M04)	(M09)	(M14)
	Cow, kidney	3 × 0.02	84	3.9	70	2.4	71	79	70	
		3 × 0.25								
		3 × 0.5	79	1.3			70	86	66	
		3 × 2.01	78	2.6			(M06)	(M04)	(M14)	

Method	Sample	No. of samples at level (mg/kg)	Recoveries, determined as 6-chloronicotinic acid (%)								
			Imidacloprid		Metabolite mix ¹		Individual metabolites as specified in italics				
			Mean	RSD	Mean	RSD	Mean	Mean	Mean	Mean	
	Cow, fat	5 × 0.02 6 × 0.1 7 × 0.3	96 94	3.8 12	111	8.7	98 [<i>n</i> =6]	94 [<i>n</i> =6]			
							95 [<i>n</i> =6] (<i>M06</i>)	103 [<i>n</i> =6] (<i>M04</i>)			
	Cow, muscle	3 × 0.02 3 × 0.2 3 × 0.6	102 75	1.3 5.2	87	12	84	76			
							72 (<i>M06</i>)	73 (<i>M04</i>)			
	Hen, egg	15 × 0.02 11 × 0.05 5 × 0.1	75 90	15 3.7	74	12	61 [<i>n</i> =8]				
							70 [<i>n</i> =10] (<i>M06</i>)				
	Hen, muscle	6 × 0.02 6 × 0.05 6 × 0.1	74 77	8.8 9.4	68	7.4	73				
							61 (<i>M06</i>)				
	Hen, fat	6 × 0.02 6 × 0.05 5 × 0.1	78 80	12 17	75	7.5	78 [<i>n</i> =5]				
							73 [<i>n</i> =6] (<i>M06</i>)				
	Hen, liver	3 × 0.02 4 × 0.125 3 × 0.313 3 × 0.5 3 × 2.0	111 87 107	7.4 7.0 0.8	93	2.0	85 [<i>n</i> =5]	87	103		
									86		
								87	90		
								(<i>M06</i>)	(<i>M04</i>)	(<i>M14</i>)	
Blass, 1992	Cow, milk	4 × 0.02 4 × 0.1			72 ² 82 ²	2.4 1.8					
	Hen, egg	4 × 0.02 4 × 0.1			73 ² 73 ²	2.7 16					
	Poultry liver	4 × 0.1 4 × 0.5			97 ² 94 ²	5.4 4.0					

M06: imidacloprid olefin, M01: 5-hydroxy-imidacloprid, M14: 6-chloronicotinic acid, M09: denitro compound

¹ Comprises imidacloprid and 1-4 of metabolites, with each compound added at the specified level

² Mixture of imidacloprid, 6-CNA, and M09, each added at the specified level

Soil. In method no. 00577 (Schramel, 1999), a suitable enforcement method for the determination of imidacloprid residues, soil samples are extracted by boiling methanol in “Soxtec” extraction equipment, and subsequently cleaned up on a “Chromabond” SPE silica gel cartridge. After evaporation of the solvent and reconstitution in acetonitrile/water, the residues are quantified by HPLC with UV detection. Two columns of differing selectivity (LiChrospher 60 B and Zorbax SB-CN) are used to avoid interferences. The recoveries were 94-101% (LiChrospher) and 88-89% (Zorbax) at fortifications of 0.01 and 0.1 mg/kg, with respective RSDs of 3.6-6.6% and 3.3-4.3%. The LOQ was 0.01 mg/kg and the limit of detection (LOD) 0.003 mg/kg. Blank values were below 0.004 mg/kg in all samples.

Water. Method no. 00576 (Sommer, 1999) can be used as an enforcement method for the determination of imidacloprid in surface and drinking water. Imidacloprid is concentrated from water samples by solid-phase extraction (C-18 cartridges), after which surface water samples are cleaned up on silica gel cartridges. After evaporation, the residues are reconstituted in milli-Q water and determined by HPLC (stationary phase LiChrospher 60) with UV detection. As a confirmatory procedure, a second column of differing polarity (LiChrospher 100) was also used. Recoveries for drinking water at fortifications of 0.03 and 0.3 µg/l were 93% and 96% respectively, with relative standard deviations of 4.3% and 3.1%. The corresponding values for the LiChrospher 100 column were 100% and 95%, with RSDs of 2.0% and 2.1%. The LOQ was 0.03 µg/l. Imidacloprid was not

detected in any control samples. In surface water, an impurity with the same retention time as imidacloprid was detected by the LiChrospher 60 column corresponding to 0.0085 µg/l imidacloprid equivalents. Recoveries from the LiChrospher 60 column, corrected for blank values, were 82% at 0.03 µg/l and 89% at 0.3 µg/l, with RSDs of 7.3% and 7.9% respectively. No interferences were observed with the confirmatory column and the respective recoveries were 76% (RSD 5.3%) and 87% (RSD 6.9%).

Data collection methods

Plant materials. Residues of imidacloprid and its metabolites (i.e. the total residue of imidacloprid) were determined by method no. 00176 (Weber, 1989) in trials until about 1990. It is essentially the same as method 00300 of Placke and Weber (1993). Residues are extracted with a mixture of methanol and water evaporated to the aqueous remainder. Fat- or wax-containing samples are partitioned with hexane. The extract is cleaned up by column chromatography with XAD 4 (polystyrene resin). The residues containing the 6-chloropyridyl moiety are oxidized to 6-chloronicotinic acid with alkaline KMnO₄ solution, derivatized with MSTFA and then determined by gas chromatography with mass selective detection. Mean recoveries from each sample material ranged from 75 to 113% at fortifications of 0.05 to 0.5 mg/kg, and individual recoveries from 70 to 122%. The metabolites M03, M07, M09 and M12 were also determined at these fortifications and mean recoveries were between 64 and 110%, with individual values ranging from 62 to 112%.

This method was replaced by method no. 00200 (Weber, 1990b). Additional sample materials were included but the analytical procedure remained the same. Mean recoveries from each material ranged from 72% to 113% at 0.05 to 0.5 mg/kg, and individual recoveries from 70% to 122%. The LOQ was 0.05 mg/kg except in hops and tobacco (0.2 mg/kg). The method was tested with numerous additional materials not previously included. Details are documented in supplements 00200/E001, 00200/E003, 00200/E004, 00200/E008, 00200/E010 and 00200/E005 (Placke, 1991a,c,d, 1992b,e,g). Recoveries at the LOQ, which remained at 0.05 mg/kg for the samples tested, were approximately in the same range as in the original method and reported above. Various supplementary commodities were analysed by method 00200 in studies in the USA, some with further minor modifications (Weber, 1994).

Method 00200 was validated in an independent laboratory study by Allmendinger (1991b). Recoveries from apples, sunflowers, and wheat grain and straw ranged from 69% to 112% at fortifications of 0.05 to 0.5 mg/kg. In 35 experiments the mean recovery was 95% with a relative standard deviation (RSD) of 12%. Blank values were below 0.05 mg/kg in all samples.

Until 1990 method no. 00171 (Blass, 1990a) was used to determine the parent compound. Residues were extracted with acetonitrile and evaporated to the aqueous remainder; the aqueous phase was then extracted with n-hexane and hexane/ethyl acetate. After extraction of imidacloprid with dichloromethane and column chromatography on Florisil, the residues were determined by HPLC with UV detection. Individual recoveries from all samples ranged from 77% to 102% at fortifications of 0.01 to 1 mg/kg. The LOQ was 0.01 mg/kg in most materials, and 0.1 mg/kg for straw and cotton seed oil. The method was also tested with tomato paste (method supplement 00171/E003, Blass, 1990d). The recovery at the LOQ (0.01 mg/kg) was 101%.

Method 00171 was also independently validated in a separate laboratory by Allmendinger (1991a). Recoveries from apples, sunflowers, and wheat grain and straw ranged from 73% to 109% at fortifications of 0.01 to 1 mg/kg. The mean recovery was 93%, with a relative standard deviation of 8%, in 36 experiments. Blank values were below 30% of the corresponding LOQ in all samples.

The clean-up procedures in method 00171 were modified (00171/M001) by Blass (1990b). After extraction, the aqueous remainder is transferred to an Extrelut[®] cartridge, and the residues extracted with dichloromethane. Subsequent steps, and the LOQs, are unchanged. Recoveries at fortifications of 0.01 and 0.1 mg/kg ranged from 81 to 106%. This modified method was used with

numerous additional materials, the details of which are given in supplements 00171/M001/E001, 00171/M001/E002, and 00171/M001/E005 (Blass, 1990c,e,f). Recoveries were approximately the same as described above.

From the end of 1990, a modification to method 00200 (00200/M001) was used to determine residues of the parent compound in plant materials (Placke, 1990, 1991b), which facilitated determination of parent and total residues in a single extract, and combined the extraction procedure of method 00200 with the clean-up steps from method 00171 and its modifications. After extraction with methanol/water and XAD 4 column clean-up, the parent compound residues are cleaned up on a ChemElut[®] cartridge eluted with dichloromethane. The residues are then chromatographed on Florisil and determined by HPLC with UV detection.

The method was again modified in 1991 (00200/M002, Placke, 1991e). An aliquot of the extract is used for determination of the parent residues, thus avoiding wastage and the handling of large volumes of extract and solvent, and saving time. For both modifications, mean recoveries for each sample material ranged from 75 to 105% at fortifications of 0.01 to 5 mg/kg, while individual recoveries ranged from 73 to 108%. The LOQ was 0.01 mg/kg for almond seeds, apricots, cabbages, cauliflowers, cotton seed and cotton seed oil, egg plant, lettuce, plums, peaches, citrus fruits and their processed products, tomatoes and their processed products, paprika, onions, nectarines, beans, rape seed and straw, and wheat grain. For rice and wheat straw and sunflower presscake the LOQ was 0.05 mg/kg and for hops and tobacco 0.2 mg/kg (Placke, 1992a,c-f). As a confirmatory method, another HPLC column (LiChrospher RP8) with isocratic separation can be used. The method is specific, since no interferences with other commonly applied plant protection products have been observed in numerous field trials, and blank values have usually been below 30% of the LOQ.

Method 00200 and its modification M002 were validated in an independent laboratory (Pelz, 1993). A slightly modified extraction procedure with the addition of diluted sulfuric acid was used, and was later introduced into method 00300. Individual recoveries of imidacloprid from melon peel and pulp, peppers and tomatoes at fortifications of 0.01 to 1 mg/kg ranged from 69 to 112% and mean recoveries from each type of sample material at each fortification level were 72 to 100%, with typical RSDs of about 10%. Individual recoveries of the parent compound were between 68% and 83%, with mean recoveries from each type of sample at each level of 70 to 79%, with a typical standard deviation of about 5%. Blank values were below 30% of the corresponding LOQ in all samples.

In method no. 00300 Placke and Weber (1993) developed method 00200/M002 by adding diluted sulfuric acid to aid extraction and recovery of polar metabolites, and by modifying the chromatographic separation for shorter run times or better separation. As method 00300 is being recommended for enforcement it is described above. The method has later been validated for numerous additional materials. Details are given in supplements 00300/E001-E003 (Placke, 1994a,b, 1997), and 00300/E004 (Schöning, 1999b).

Recoveries at the same LOQs as 02.00 and 0200/M002 were approximately the same at about 77-100%. Information on recoveries from several important crops is shown in Table 50. The independent laboratory validation of method 00300 is covered by that reported above for method 00200 and its modification 00200/M002 (Pelz, 1993), since this study was with the slightly modified extraction procedures used in 00300.

Table 50. Recoveries by method 00300 of imidacloprid and the total residue of imidacloprid (as 6-chloronicotinic acid) from several important crops and their processed commodities (Placke and Weber, 1993).

Sample	No. of samples at level (mg/kg)	Recoveries and RSD, %			
		Imidacloprid		Total residue	
		Mean	RSD	Mean	RSD
Apple, fruit	2 × 0.01	98			

Sample	No. of samples at level (mg/kg)	Recoveries and RSD, %			
		Imidacloprid		Total residue	
		Mean	RSD	Mean	RSD
	3 × 0.1 2 × 0.05 2 × 0.5	86	0.7	109 89	
Apple sauce	4 × 0.01 2 × 0.1 2 × 0.05	98 89	5.9	79	
Apple juice	4 × 0.01 2 × 0.2 2 × 0.05	91 90	4.1	106	
Apple, dry pomace	3 × 0.01 3 × 0.1 3 × 0.05 3 × 0.5	100 87	8.1 1.3	94 91	8.8 2.9
Apple, dried	3 × 0.01 3 × 0.1 3 × 0.05 3 × 0.5	82 90	8.0 1.1	90 85	1.7 1.8
Pear, fruit	4 × 0.01 3 × 0.1 4 × 0.05 4 × 0.5	91 87	9.2 5.9	87 82	7.6 3.2
Bell peppers	3 × 0.01 3 × 0.05 2 × 0.05 2 × 0.5	92 96	3.5 14	113 89	
Tomatoes	3 × 0.01 3 × 0.05 3 × 0.5 4 × 0.05	99 95 89	2.1 2.8 1.3	84 91	2.1 4.6
Tomato juice	3 × 0.01 3 × 0.05 3 × 0.5	89 89	8.5 11.4	87 83	3.5 3.0
Tomato paste	3 × 0.01 3 × 0.05 3 × 0.5 2 × 0.05	90 85 90	5.9 4.1 2.9	88	
Sugar beet roots	2 × 0.01 2 × 0.1 2 × 0.05 2 × 0.5	102 100		95 95	
Sugar beet leaves	2 × 0.01 2 × 0.1 2 × 0.05 2 × 0.5	95 98		87 94	

Animal materials. Residues of imidacloprid and its metabolites can be determined by methods 00191 (Weber, 1990a) and 00191/M001 (Weber and Heukamp, 1993). Since they are recommended for enforcement they are described above

Soil. Residues of imidacloprid in soil can be determined by method 00267 (Bachlechner, 1992b) using HPLC with UV detection. Soil samples are extracted in “Soxtec” hot-extraction equipment with boiling methanol under reflux. The solvent is evaporated and residues determined without further clean-up by reverse-phase HPLC with UV detection. Mean recoveries at each spiking level from each of the three tested soils ranged from 90% to 110% at fortifications of 0.006 to 0.174 mg/kg. The LOQ was 0.006 mg/kg and the limit of detection 0.002 mg/kg. The overall mean recovery of the method was 99%, with a relative standard deviation of 5.7%.

From 1992 onwards method 00270 (Bachlechner, 1992c) was often used. It uses the same extraction and quantification procedures as method 00267 but includes column chromatography on silica gel with a laboratory robot system to clean up the extracts. Mean recoveries at each spiking level from each of the soils were between 81% and 119% at fortifications of 0.006 to 0.174 mg/kg. The LOQ was 0.006 mg/kg and the limit of detection 0.002 mg/kg. The overall mean recovery was 88%, with a standard deviation of 12.4%.

Stability of residues in stored analytical samples

Data on the frozen storage stability of residues of imidacloprid and some of its metabolites in apples (whole fruit, juice, wet and dry pomace), barley grain, forage and straw, cauliflowers, cotton seed hulls, soapstock and oil, hops (green and dried cones), lettuce, lemons, maize grain and forage, orange (whole fruit, juice, dried pulp and oil), potatoes, sunflower seeds, sugar beet leaves and roots, tomatoes, wheat grain dust, flour, bran and shorts and in eggs, fat, kidney, liver, milk and muscle were reported to the Meeting.

Morishima (1992a,b;1994) fortified samples of head lettuce, maize grain, forage and straw, and lemons with labelled imidacloprid and five of its metabolites (M01, M06, M07, M09, M14) at approximately 1 mg/kg, leading to a final concentration of 6.41 mg/kg imidacloprid equivalents, and stored them in a freezer at an average of -19.2°C (range -4.7° to -23.9° C). Samples were analysed on day 0 and about 3, 6, 9 or 12, and 24 months later. Three samples of each material were analysed on day 0, and thereafter duplicate samples, by TLC, with LSC of the plate scrapings. The results are shown in Table 51.

Table 51. Freezer storage stability of ¹⁴C-imidacloprid and five ¹⁴C-labelled metabolites added to lettuce, lemon, and maize (Morishima, 1992a,b;1994).

Sample	Fortification (mg imidacloprid equivalents/kg)	Storage (months)	Imidacloprid (total residue) in stored samples, % remaining ¹	Report no.
Lettuce, whole plant	6.414 ²	0	94	NR 1291
		3	94	
		6	95	
		12	100	
		24	94	
Lemon, whole fruit	6.414 ²	0	92	NR 1291
		3	93	
		6	95	
		9	96	
		24	91	
Maize, forage	6.414 ²	0	94	NR 1291
		3	93	
		6	99	
		12	103	
		24	93	
Maize, straw	6.414 ²	0	96	NR 1291
		3	95	
		6	98	
		12	103	
		24	92	
Maize, grain	6.414 ²	0	91	NR 1291
		3	86	
		6	95	
		12	99	
		24	95	

¹ Mean value: triplicate values for day 0, duplicates for each of the other samplings

² The fortified concentrations of individual compounds in crops can be calculated from the concentration (6.414 mg/kg) and the ratio of the compounds in the treating solution.

They were as follows:

¹⁴ C-imidacloprid:	1.16 mg/kg
M01 5-hydroxy-imidacloprid:	0.81 mg imidacloprid equivalents/kg
M06 olefin:	1.25 mg imidacloprid equivalents/kg
M07 nitrosimine:	0.94 mg imidacloprid equivalents/kg
M14 6-chloronicotinic acid:	1.19 mg imidacloprid equivalents/kg
M09 denitro-imidacloprid:	1.07 mg imidacloprid equivalents/kg

Samples of sugar beet leaves and roots, barley grain, forage and straw, and sunflower seed were fortified by Ishii and Placke (1992) with imidacloprid, M01, M06 and M09 at 1 mg/kg each, and green and dried hop cones at 10 mg/kg (higher LOQ and typical residue) and stored in a freezer at -20°C or below. Samples were analysed on day 0 and after about 1, 2, 3, 6-8, 12 and 24 months (hops after 1, 3, 6 and 12 months). On day 0 four samples of each material were extracted and analysed and thereafter duplicate samples were analysed together with a stored control and two freshly-fortified samples for the total residue of imidacloprid, determined as 6-chloronicotinic acid. The results are shown in Table 52.

Table 52. Freezer storage stability of imidacloprid and three metabolites added to sugar beet, barley, hops, and sunflowers (Ishii and Placke, 1992).

Sample	Fortification (mg/kg)	Storage (days)	Imidacloprid (total residue)		Report No.
			% remaining ¹	Corrected for analytical recovery ² (%)	
Sugar beet, roots	1.0 ³	0	84	-	RA-428/92
		30	76	97	
		60	83	100	
		94	80	103	
		226	83	109	
		374	86	83	
		742	91	99	
Sugar beet, leaves	1.0 ³	0	83	-	
		30	67	93	
		61	83	100	
		92	79	91	
		223	88	100	
		371	85	94	
		741	87	110	
Barley, grain	1.0 ³	0	74	-	
		30	64	94	
		64	79	95	
		91	71	79	
		231	77	93	
		372	99	97	
		737	81	88	
Barley, forage	1.0 ³	0	86	-	
		30	79	91	
		58	85	97	
		91	90	96	
		226	89	97	
		363	96	94	
		734	96	108	
Barley, straw	1.0 ³	0	78	-	
		30	69	99	
		63	72	97	
		92	78	92	
		231	74	95	
		371	86	98	
		734	91	115	
Sunflower, seeds	1.0 ³	0	80	-	RA-428/92
		30	77	96	
		63	84	93	
		97	71	83	
		222	88	90	

Sample	Fortification (mg/kg)	Storage (days)	Imidacloprid (total residue)		Report No.
			% remaining ¹	Corrected for analytical recovery ² (%)	
		358	87	97	
		727	82	87	
Hops, green cone	10 ⁴	0	86	-	
		28	90	98	
		90	69	86	
		174	102	99	
		359	97	113	
Hops dried cone	10 ⁴	0	85	-	
		28	86	100	
		89	97	93	
		173	85	108	
		361	99	94	

¹ Mean value: 4 replicates for day 0, duplicates for each of the other samplings.

² Corrected with the mean of 2 individual concurrent recoveries at each sampling.

³ Total: 4 mg/kg. Theoretical imidacloprid equivalents calculated to be 3.87 mg/kg.

⁴ Total : 40 mg/kg. Theoretical imidacloprid equivalents calculated to be 38.7 mg/kg.

Fortified with imidacloprid, M01 5-hydroxy-imidacloprid, M06 olefin and M09 denitrop-imidacloprid.



In freezer storage stability studies by Noland (1992, 1993) and Noland and Chickering (1994a,b) on imidacloprid and the four metabolites M01, M06, M09 and M14 in homogenised samples of potatoes, apples (whole fruit, juice, wet and dry pomace), wheat (grain dust, flour, bran, shorts), and cotton seed (hulls, soapstock, oil) were fortified with 0.5 mg/kg (parent equivalents) of each analyte. Control and triplicate fortified samples were analysed immediately, and thereafter duplicate aged samples with single controls and concurrent recoveries stored at -20°C were analysed at intervals by oxidation of the parent and all metabolites to 6-chloronicotinic acid, derivatization, and gas chromatography with mass-selective detection. The results are shown in Table 53.

Table 53. Freezer storage stability of imidacloprid and four metabolites added to various plant samples (Noland 1992, 1993; Noland and Chickering, 1994a,b).

Sample	Fortification (mg/kg)	Storage (days)	% of initial residue remaining ¹	Reference: Report No.
Potato, whole	0.5 ²	0	88	103237
		89	103	
		184	101	103237-1
		376	115	103237-2
		578	96	
		734	94	103237-3
Apple, whole	0.5 ²	0	108	103237
		101	98	
		185	104	103237-1
		398	88	103237-2
		577	96	
		736	84	103237-3
Apple, juice	0.5 ²	0	92	103237
		82	97	
		176	99	103237-1
		371	97	103237-2
		574	92	
		728	92	103237-3
Apple, wet pomace	0.5 ²	0	84	103237
		94	83	
		175	79	103237-1
		370	93	103237-2
		587	101	

Sample	Fortification (mg/kg)	Storage (days)	% of initial residue remaining ¹	Reference: Report No.
		741	90	103237-3
Apple, dry pomace	0.5 ²	0	87	103237
		96	83	
		183	89	103237-1
		372	85	103237-2
		590	90	
		739	87	103237-3
Wheat, grain dust	0.5 ²	0	72	103237
		100	79	
		173	99	103237-1
		365	90	103237-2
		587	86	
		739	64	103237-3
Wheat, flour	0.5 ²	0	96	103237
		115	87	
		195	87	103237-1
		377	71	103237-2
		600	78	
		731	82	103237-3
Wheat, bran	0.5 ²	0	94	103237
		111	72	
		192	75	103237-1
		376	101	103237-2
		598	91	
		731	84	103237-3
Wheat, shorts	0.5 ²	0	102	103237
		102	107	
		193	84	103237-1
		377	99	103237-2
		581	86	
		735	83	103237-3
Cotton seed, hulls	0.5 ²	0	74	103237
		91	86	
		181	86	103237-1
		376	93	103237-2
		588	78	
		745	73	103237-3
Cotton seed, soapstock	0.5 ²	0	80	103237
		116	81	
		199	91	103237-1
		381	95	103237-2
		565	76	
		746	63	103237-3
Cotton seed, oil	0.5 ²	0	103	103237
		91	88	
		175	93	103237-1
		378	95	103237-2
		579	97	
		732	77	103237-3

¹ Mean value: triplicates for day 0, duplicates for the other samplings.

² Samples fortified with a mixed standard containing imidacloprid M06, M09, M01 and M14 (6-chloronicotinic acid) at a total of 2.5 mg/kg.

In another study by Lenz (1992, 1993, 1994a,b) on homogenised samples of wheat grain, forage and straw, tomatoes, cauliflowers, and lettuce were fortified with 0.5 mg/kg parent equivalents

of each analyte. Control and triplicate samples were analysed immediately, and duplicates of the samples stored at -20°C plus single control and recovery samples. The results are shown in Table 54.

Table 54. Freezer storage stability of imidacloprid and its metabolites added to fortified samples of wheat, cotton, tomato, cauliflower and lettuces and their processed products (Lenz, 1992, 1993, 1994a,b).

Sample	Fortification (mg/kg)	Storage (days)	% of initial residue in stored samples ¹	Report No.
Wheat, grain	0.5 ²	0	95	103949
		88	95	
		181	99	
		367	86	103949-2
		556	107	
		759	79	103949-3
Wheat, forage	0.5 ²	0	94	103949
		93	102	
		184	113	
		368	78	103949-2
		546	96	
		749	77	103949-3
Wheat, straw	0.5 ²	0	91	103949
		91	84	
		183	91	
		366	86	103949-2
		551	94	
		754	88	103949-3
Cotton seed	0.5 ²	0	92	103949
		98	86	
		182	111	
		371	89	103949-2
		552	85	
		769	72	103949-3
Tomato	0.5 ²	0	96	103949
		91	113	
		187	110	
		371	96	103949-2
		556	114	
		758	97	103949-3
Cauliflower	0.5 ²	0	94	103949
		92	112	
		185	102	
		366	90	103949-2
		553	110	
		753	102	103949-3
Lettuce	0.5 ²	0	105	103949
		90	106	
		182	86	103949-1
		370	89	103949-2
		543	101	
		741	100	103949-3

¹ Mean value: triplicates for day 0, duplicates for other samplings.

² Samples fortified with M06, M09, M01 and M14 at a total of 2.5 mg/kg.

In additional tests Lenz (1996) fortified the whole fruit, dried pulp, juice and oil of oranges with imidacloprid and the four metabolites listed above at 0.05 mg/kg of each. Samples were stored at or below -5°C. Concurrent recoveries were determined by spiking a control sample with known quantities of a mixture of imidacloprid and denitro-imidacloprid. All of the samples were analysed as before. The results are shown in Table 55.

Table 55. Freezer storage stability of imidacloprid and metabolites added to fortified samples of orange commodities (Lenz, 1996).

Sample	Fortification (mg/kg)	Storage (months)	Imidacloprid (total residue) in stored samples		Report No.
			% remaining ¹	Corrected for analytical recovery ² (%)	
Orange, whole fruit	0.5 ³	0	94	-	103949-4
		3	83	93	
		6	71	84	
		13	104	105	
		21	100	103	
Orange, dried pulp	0.5 ³	0	85	-	
		3	84	94	
		6	97	101	
		13	103	96	
		21	82	99	
Orange, juice	0.5 ³	0	77	-	
		3	99	99	
		6	99	120	
		13	110	95	
		21	105	128	
Orange, oil	0.5 ³	0	77	-	
		3	112	98	
		6	81	86	
		13	99	104	
		21	95	81	

¹ Mean: triplicates for day 0, duplicates for other samplings.

² Corrected with 1 concurrent recovery at each sampling.

³ Samples fortified with M06, M01 and M14 at a total of 2.5 mg/kg.

Concurrent recovery samples fortified with imidacloprid + denitro-imidacloprid at a total of 2.5 mg/kg.

In a study by Placke (1993q), samples of sugar beet (leaf, root), barley (grain, forage, straw), and of sunflower seed were spiked with imidacloprid at 1 mg/kg. Green and dried hop cones were fortified with a mixture of imidacloprid and the three metabolites M01, M06 and M09 (10 mg/kg for imidacloprid and each of the metabolites). Samples were stored in a freezer at -20°C or lower and analysed on day 0 and after about 1, 2, 3, 6-8, 12, and 24 months later. On day 0 four samples of each material were analysed to establish initial recoveries, thereafter duplicate samples plus a control and two freshly fortified samples were analysed. The results are shown in Table 56.

Table 56. Freezer storage stability of imidacloprid added to sugar beet, barley, and sunflowers, and of imidacloprid, M01, M06 and M09 added to hops (Placke, 1993q).

Sample	Fortification (mg/kg)	Storage (days)	Imidacloprid in stored samples		Report No.
			% Remaining ¹	Corrected for analytical recovery ² (%)	
Sugar beet, root	1.0	0	76	-	RA-360/93
		39	93	105	
		75	87	93	
		89	92	101	
		179	85	89	
		376	94	103	
		742	90	107	
Sugar beet, leaf	1.0	0	88	-	
		40	99	103	
		76	84	95	
		90	99	102	
		180	90	94	
		377	94	101	
		747	92	98	

Sample	Fortification (mg/kg)	Storage (days)	Imidacloprid in stored samples		Report No.
			% Remaining ¹	Corrected for analytical recovery ² (%)	
Barley, grain	1.0	0	87	-	
		30	88	100	
		70	82	101	
		88	98	104	
		179	90	88	
		367	95	103	
		735	88	98	
Barley, forage	1.0	0	78	-	
		26	86	98	
		63	90	95	
		91	86	97	
		174	91	96	
		362	94	102	
		720	93	101	
Barley, straw	1.0	0	80	-	
		25	86	101	
		63	116	105	
		92	123	105	
		175	117	105	
		361	103	91	
		729	91	114	
Sunflower, seed	1.0	0	88	-	
		32	86	87	
		59	89	82	
		91	104	99	
		176	108	100	
		359	94	91	
		716	84	93	
Hops, green cone	10 ³	0	89	-	RA-360/93
		28	87	95	
		90	89	103	
		174	86	92	
		359	84	100	
		720	85	88	
Hops dried cone	10 ³	0	89	-	
		28	87	95	
		90	89	103	
		174	86	92	
		359	84	100	
		720	85	88	

¹ Mean value: 4 replicates for day 0, duplicates for other samplings.

² Corrected with the mean of duplicate concurrent recoveries at each sampling.

³ Total added: 40 mg/kg; 10 mg/kg each of imidacloprid, M01, M06 and M09.

To support cattle and poultry feeding studies, Heukamp and Maasfeld (1996) fortified samples of muscle, liver, kidney, fat, milk, and eggs with a mixture of imidacloprid and one to three metabolites. The rates were 0.033 mg/kg of each compound for milk, 0.05 mg/kg for eggs and muscle, 0.1 mg/kg for fat, 0.125 mg/kg for liver, and 0.25 mg/kg for kidneys and samples were then stored at -18°C or lower for analysis on day 0 and after about 1, 2, 3, and 6 months to a maximum of 298-357 days. Triplicate samples were analysed after each interval plus a stored control and two freshly fortified samples for the total residue of imidacloprid, determined as 6-chloronicotinic acid. The results are shown in Table 57.

Table 57. Freezer storage stability of imidacloprid and up to three metabolites added to animal tissues, organs, and products (Heukamp and Maasfeld, 1996).

Sample	Fortification (mg/kg)	Storage interval (days)	Imidacloprid (total residue) in stored samples		Report No.
			% Remaining ¹	Corrected for analytical recovery ² (%)	
Egg	0.05 ³	0	72	-	MR-644/95
		30	90	91	
		65	46	90	
		85	62	89	
		180	65	108	
		357	81	103	
Fat (bovine)	0.1 ³	0	111	-	
		31	73	92	
		67	70	90	
		90	86	91	
		171	93	91	
		307	90	91	
Kidney (bovine)	0.25 ³	0	72	-	
		50	69	96	
		90	70	113	
		183	77	101	
		298	88	104	
Liver (poultry)	0.125 ³	0	95	-	
		30	81	101	
		77	98	105	
		89	88	117	
		181	110	115	
		328	89	120	
Milk	0.033 ³	0	95	-	
		30	78	100	
		59	76	94	
		80	80	113	
		115	78	104	
		178	84	105	
		353	95	109	
Muscle (poultry)	0.05 ³	0	75	-	
		31	72	106	
		79	86	110	
		92	79	107	
		183	68	108	
		325	99	115	

¹ Mean value: triplicate analyses at each sampling² Corrected with the mean of duplicate concurrent recoveries at each sampling³ The spiking mixture consisted of the following compounds:

- *eggs/muscle*: imidacloprid and olefin metabolite (M06), each at 0.05 mg/kg, corresponding to a theoretical equivalent concentration of imidacloprid of 0.1 mg/kg.
- *fat*: imidacloprid, 5-hydroxy metabolite (M01), and olefin metabolite (M06), each at 0.1 mg/kg, corresponding to a theoretical equivalent concentration of imidacloprid of 0.295 mg/kg.
- *kidney*: imidacloprid, 5-hydroxy metabolite (M01), olefin metabolite (M06), and 6-chloronicotinic acid (M14), each at 0.25 mg/kg, corresponding to a theoretical equivalent concentration of imidacloprid of 1.143 mg/kg.
- *liver*: imidacloprid, olefin metabolite (M06) and 6-chloronicotinic acid (M14), each at 0.125 mg/kg and denitro metabolite (M09) at a 0.107 mg/kg, corresponding to a theoretical equivalent concentration of imidacloprid of 0.583 mg/kg.
- *milk*: imidacloprid, 5-hydroxy metabolite (M01), and olefin metabolite (M06), each at 0.033 mg/kg, corresponding to a theoretical equivalent concentration of imidacloprid of 0.098 mg/kg.

Summary

The storage stability of imidacloprid and various important metabolites was tested in various plant and animal materials. Tests on animal samples were carried out to assess the stability of the total

residue. For plants, tests were carried out to assess the stability of the total residue and on plants to assess the stability of residues of the active substance and of the total residue. The results indicate that imidacloprid and the tested metabolites are stable for a minimum of approximately 2 years in plants, and for at least 1 year in animal commodities. The results validate the residue values reported.

Definition of the residue

In the studies on the metabolism of imidacloprid in rats, lactating goats and laying hens imidacloprid and a number of metabolites were detected. The identified metabolites varied qualitatively and quantitatively but all contained the 6-chloronicotinyl moiety of imidacloprid. The definition of the residue in animal products should therefore be defined as “the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid”.

The metabolic pathways of imidacloprid in crops are the same as in animals. All the metabolites were also found in rats and hence covered by the toxicological assessment. All identified transformation products of imidacloprid still contain the 6-chloronicotinic moiety of the parent compound. The residue in plant products should therefore also be defined as “the sum of imidacloprid and its metabolites containing the 6-chloronicotinyl moiety, expressed as imidacloprid”.

USE PATTERN

The systemic chloronicotinyl insecticide imidacloprid has a broad spectrum of activity, particularly against sucking insects such as aphids, leaf hoppers, thrips and white flies. Various species of beetles (e.g. *Atomaria* sp., *Leptinotarsa decemlineata*, *Lissorhoptrus oryzophilus*, *Lema orycea*), some species of flies (e.g. *Oscinella frit* and *Pegomyia* spp.) and leaf miners (e.g. *Lithocolletis* spp., *Phyllocnistis citrella*) are effectively controlled. Imidacloprid is ineffective against spider mites and nematodes.

The acropetal uptake of the active substance by the roots is an important prerequisite for soil treatment, such as drenches, drip irrigation, in-furrow or granular applications. It is also used for stem applications.

The active substance imidacloprid is sold under various trade names; the most common are Confidor® and Gaucho®. The trade name Confidor is used world-wide mainly for foliar and soil application formulations, whereas for use as seed treatment the trade name is Gaucho. In the USA for example it is sold under the trade names Admire® for soil application and Provado® for foliar use. In Japan it is marketed mainly under the trade name Admire®. Certified labels and their English translations were submitted by the manufacturer. Registered uses of imidacloprid reported to the Meeting are shown in the following Tables.

Table 58. Fruits.

Table 59. Bulb vegetables.

Table 60. Brassica vegetables, head cabbage.

Table 61. Fruiting vegetables, cucurbits.

Table 62. Fruiting vegetables, other than cucurbits.

Table 63. Leafy vegetables.

Table 64. Legume vegetables.

Table 65. Root and tuber vegetables.

Table 66. Stalk and stem vegetables.

Table 67. Grasses.

Table 68. Nuts and seeds.

Table 69. Fodder and forage crops.

Table 70. Hops, tea, tobacco.

Table 58. Registered uses of imidacloprid on fruits.

Crop	Country	Form.	Application				PHI, days
			Method, remarks	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Apple	Australia	200 SC	Soil drench	0.6-2.4 g/tree (max. 0.96 kg /ha) ¹	0.06-0.24 ²	1 per 3 years	Note ¹

Crop	Country	Form.	Application				PHI, days
			Method, remarks	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Apple	Austria	70 WG	Foliar spray	0.07-0.11	0.007	1	14
Apple	Belgium	200 SL	Foliar spray	0.07-0.12	0.023-0.025 ¹	1-2	14
Apple	Canada	240 SC	Foliar spray	0.048-0.091	0.0016-0.003	1-2	7
Apple	France	200 SL	Foliar spray	0.035-0.11 ¹	0.007	2 ¹	14
Apple	Germany	70 WG	Foliar spray	0.035 ³	0.007	1	14
Apple	Italy	200 SL	Foliar spray	0.15	0.01	1	28
Apple	Japan	10 WP	Foliar spray	0.1-0.4	0.005-0.01	2	21
Apple	Netherlands	70 WG	Foliar spray	0.07-0.11 ¹	0.007	1-2	14
Apple	Portugal	200 SL	Foliar spray	0.10	0.01	1-2 ¹	14
Apple	South Africa	350 SC	Foliar spray	0.52-0.74	0.021	1 ¹	70
		350 SC	Soil drench treatment	1.1 g/tree	0.11	1	
Apple	South Korea	40 SL	Foliar spray	0.16 ¹	0.004	1-5	30
		10 WP	Foliar spray	0.2 ¹	0.005	1-2	21
		17.5WP	Foliar spray	0.1 ¹	0.0025	1-5	14
Apple	Spain	200 SL	Foliar spray	0.1-0.15 ¹	0.01	1-2 ¹	15
Apple	Turkey	350 SC	Foliar spray	0.11-0.14 ¹	0.007		14
Apple	USA	192 SC	Foliar spray, after flowering, interval 10 days	0.056-0.11	0.0015-0.003	1-2	7
Apricot	Australia	200 SC	Foliar spray	0.1-0.2	0.005	1	21
Apricot	France	200 SL	Foliar spray	0.02-0.07 ¹	0.005-0.007	1-2 ¹	14
Apricot	Italy	200 SL	Foliar spray	0.15	0.01	1	35
Apricot	Japan	10 WP	Foliar spray		0.005	2	21
Apricot	Spain	200 SL	Foliar spray	0.1-0.15 ¹	0.01	1 ¹	15
Banana	Cameroun	200 SL	Undiluted product on the base of the pseudo-trunk and/or diluted product on the base of the pseudo-trunk	max. 0.5 0.25 g/plant (based on 2000 plants/ha)	0.1	1-3	
Banana	Ivory coast	200 SL	Undiluted product on the base of the pseudo-trunk and/or diluted product on the base of the pseudo-trunk	max 0.5 mg/plant (based on 2000 plants/ha)	0.1	1-3	
Banana	Philippines	100 SL	Bud flower injection	max. 16 mg/mat	0.012	1	80 ⁵
Cherry	Australia	200 SC	Foliar spray	0.1-0.2	0.005	1	21
Cherry	Italy	200 SL	Foliar spray	0.15	0.01	1	21
Citrus fruits	Greece	200 SL	Foliar spray, fruits specified on the label: grapefruit, lemon, orange, tangerine	0.3-0.45	0.01-0.015	1-2	21
Citrus fruits	Italy	200 SL	Foliar spray, fruits specified on the label: clementine, lemon, mandarin, orange	0.2-0.3 ¹	0.01-0.015	1	14
Citrus fruits	Philippines	100 SL	Foliar spray, interval 7 days	0.038-0.075 ¹	0.0015-0.003	1-2 ¹	14
Citrus fruits	Portugal	200 SL	Foliar spray, interval 21 days, fruits specified on the label: mandarin, orange	0.1-0.5	0.0067-0.01	1-2	14
Citrus fruits	South Africa	350 SC	Soil drench, bearing trees, excluding lemons	2.1-3.2 g/tree			
Citrus fruits	Spain	200 SL	Foliar spray	0.15-0.75	0.01-0.015	1-2 ¹	30
		200 SL	Stem application, max. 4 l product/ha/application	0.2-1.6 g/tree (depends on age and height of tree)		2-3 ¹	30

Crop	Country	Form.	Application				PHI, days
			Method, remarks	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
		200 SL	Apply in irrigation water, max. 4 l product/ha/ application			1-2	30
Citrus fruits	Thailand	100 SL	Foliar spray	0.075 ¹	0.004	2-3 ¹	14
Citrus fruits	USA ⁴	192 SC	Foliar spray, fruits specified on the label: grapefruit, lemon, orange, calamondin, citron, chironja, tangelo, tangor, kumquat, lime, mandarin, tangerine, pomels, satsuma mandarin	0.14-0.28 (depending on tree size)	0.005-0.007 (for dilute appl.; for concentrate appl. increase the concentration to apply an equivalent rate)	1-2	0
		240 SC	Soil application: surface band, drench to base of the trees, through microsprinkler below the canopy	0.28-0.56			0
Grapes, nursery	Germany	70 WG	Foliar spray, do not use grapes for consumption	0.056-0.11	0.014	2	
Grapes	Japan	10 WP	Foliar spray		0.005-0.01	2	30
Grapes	Portugal	200 SL	Foliar spray	0.07	0.007	1 ¹	14
Grapes	Thailand	100 SL	Foliar spray	0.025-0.05 ¹	0.005	2 ¹	14
Grapes	USA	75 WP	Foliar spray, spray interval 14 days	0.04 -0.052	0.004-0.008 ¹ (leaf hoppers) 0.0053-0.011 ¹ (mealy bugs)	1-2	0
Mango	Japan	10 WP	Foliar spray		0.005	2	14
Mango	Philip- pines	100 SL	Foliar spray	0.02-0.062	0.002-0.0025	1-2 ¹	20
Mango	Thailand	100 SL	Foliar spray	0.02-0.05 ¹	0.0025	1-2 ¹	14
Mango	USA	192 SL	Foliar spray	0.093	0.0046 ¹	max 6 ¹	30
Mango- steen	Thailand	100 SL	Foliar spray		0.005	1-2 ¹	14
Nashi	France	200 SL	Foliar spray	0.035-0.11 ¹	0.007	2 ¹	14
Nashi	USA	192 SC	Foliar spray, after flowering, interval 10 days	0.056-0.11	0.0015-0.003	1-2	7
Nectarine	Australia	200 SC	Foliar spray	0.1-0.2	0.005	1	21
Nectarine	Italy	200 SL	Foliar spray	0.15	0.01	1	21
Nectarine	Spain	200 SL	Foliar spray	0.1-0.15 ¹	0.01	1 ¹	15
Peach	Australia	200 SC	Foliar spray	0.1-0.2	0.005	1	21
Peach	France	200 SL	Foliar spray	0.02-0.07 ¹	0.005-0.007	1-2 ¹	14
Peach	Greece	200 SL	Foliar spray	0.125-0.15 ¹	0.005-0.006	1-2	14
Peach	Italy	200 SL	Foliar spray	0.15	0.01	1	21
Peach	Japan	10 WP	Foliar spray		0.005-0.01	2	30
Peach	Portugal	200 SL	Foliar spray	0.1	0.01	1-2	14
Peach	Spain	200 SL	Foliar spray	0.1-0.15 ¹	0.01	1 ¹	15
Peach	Turkey	350 SC	Foliar spray	0.105-0.14 ¹	0.007		14
Pear	France	200 SL	Foliar spray	0.035-0.11 ¹	0.007	2 ¹	14
Pear	Italy	200 SL	Foliar spray	0.15	0.01	1	50
Pear	Japan	10 WP	Foliar spray		0.01	2	30
Pear	Nether- lands	70 WG	Foliar spray	0.07-0.084 ¹	0.007	1-2	14
Pear	Portugal	200 SL	Foliar spray	0.10	0.01	1-2 ¹	14
Pear	South Korea	10 WP	Foliar spray	0.2 ¹	0.005	1-3	14
Pear	Spain	200 SL	Foliar spray	0.1-0.15 ¹	0.01	1-2 ¹	15

Crop	Country	Form.	Application				PHI, days
			Method, remarks	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Pear	Turkey	350 SC	Foliar spray		0.014		14
Pear	USA	192 SC	Foliar spray, after flowering, interval 10 days	0.28	0.0075	1-2	7
Persim- mon	Japan	10 WP	Foliar spray		0.005-0.01	3	7
Pineapple	Brazil	70 WG	Drench treatment	0.52 ¹	0.021	1	75
Plum	Australia	200 SC	Foliar spray	0.1-0.2	0.005	1	21
Plum	France	200 SL	Foliar spray	0.028-0.07 ¹	0.007	1-2 ¹	56
Plum	Italy	200 SL	Foliar spray	0.15	0.01	1	21
Plum	Japan	10 WP	Foliar spray		0.005	2	21
Quince	USA	192 SC	Foliar spray, after flowering, interval 10 days	0.056-0.11	0.0015-0.003	1-2	7
Quince	France	200 SL	Foliar spray	0.035-0.11 ¹	0.007	2 ¹	14
Rambu- tan	Thailand	100 SL	Foliar spray	0.075-0.15 ¹	0.015	1-2 ¹	14

¹ Not specified on label.

² Chemical control: 0.24 kg ai/tree; beneficial insect plus chemical control: 0.06 g ai/tree

³ 0.035 kg ai/ha/m crown height.

⁴ Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

⁵ Not applicable; timing of application fixed by stage of bud development

Table 59. Registered uses of imidacloprid on bulb vegetables.

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or kg ai/unit	Spray conc., kg ai/hl	No.	
Fennel, Florence	USA ²	F	240 SC	Soil application: narrow band, in-furrow, post-seeding drench, sidedress, drip irrigation	0.18-0.42	0.036-0.084 ¹	1-2 ¹	45
Garlic	Brazil	F	70 WG	Foliar spray	0.07	0.014-0.018	1-2 ¹	30
Garlic	Thailand	F	100 SL	Foliar spray	0.05-0.1 ¹	0.01-0.02	1-2 ¹	7
Leek	Germany	F	70 WS	Seed treatment (minor crop registration, off-label use)	max. 0.09 (0.045 kg/unit) ³		1	-
Onion	Brazil	F	70 WG	Foliar spray	0.07	0.014-0.018	1-2 ¹	21
Onion	Germany	F	70 WS	Seed treatment (of label use), onion except spring-, pearl and silver skin onion	max. 0.18 (0.045 kg/unit) ³		1	-
Onion	Thailand	F	100 SL	Foliar spray	0.05-0.1 ¹	0.01-0.02	1-2 ¹	7
Shallot	Thailand	F	100 SL	Foliar spray	0.05-0.1 ¹	0.01-0.02	1-2 ¹	7

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

³ 1 unit onion or leek seeds = 250,000 seeds. Coated seeds.

Table 60. Registered uses of imidacloprid on brassica vegetables, head cabbage, flowerhead brassicas.

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Broccoli	Australia	F	200 SC	Foliar spray	0.06	0.005	1-2 ¹	7
Broccoli	Brazil	F	70 WG	Drench treatment, after transplanting	0.14-0.21		1	82
Broccoli	South Africa	F/G	350 SC	Drench over seedlings before transplanting	0.1-0.2 ¹ or 4 mg ai/ seedling	0.13	1	76

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
		F/G	70 WS	Drench over seedlings before transplanting	0.1-0.2 ¹ or 4 mg ai/ seedling	0.13	1	76
Broccoli	Spain	F/G	200 SL	Foliar spray	0.1	0.01	1-2	14
		F/G	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1-2	14
Broccoli	USA ²	F	192 SC	Foliar spray, interval 5 days	0.053	0.011 ¹	1-5 ¹	7
		F	240 SC	Soil application: narrow band, in-furrow, post-seeding drench, sidedress, drip irrigation	0.18-0.42			21
Brussels sprouts	Australia	F	200 SC	Foliar spray	0.06	0.005	1-2 ¹	7
Brussels sprouts	South Africa	F/G	350 SC	Drench over seedlings before transplanting	0.1-0.2 ¹ or 4 mg ai/ seedling	0.13	1	91
		F/G	70 WS	Drench over seedlings before transplanting	0.1-0.2 ¹ or 4 mg ai/ seedling	0.13	1	91
Brussels sprouts	USA ²	F	192 SC	Foliar spray, interval 5 days	0.052	0.011 ¹	1-5 ¹	7
		F	240 SC	Soil application: narrow band, in-furrow, post-seeding drench, side drench, drip irrigation	0.18-0.42			21
Cabbage	Brazil	F	70 WG	Drench treatment, after transplant	0.14-0.21		1	82
Cabbage	Philippines	F	100 SL	Foliar spray, interval 7 days	0.025-0.03 ¹	0.0025-0.003	1-2	28
Cabbage	Spain	F	200 SL	Foliar spray	0.06-0.1 ¹	0.01-0.015	2	28
Cabbage	USA ²	F	192 SC	Foliar spray, interval 5 -7 days	0.052	0.011 ¹	1-5	7
		F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42			21
Cabbage, Mustard (Bok Choi, Napa) Cabbage, Chinese (Gai Choi)	USA ²	F	192 SC	Foliar spray, interval 5 days	0.052	0.011 ¹	1-5	7
		F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42			21
Cabbage, head	Australia	F	200 SC	Foliar spray	0.06	0.005	1-2 ¹	7
Cabbage, head	South Africa	F/G	350 SC	Drench over seedlings before transplanting	0.1-0.2 ¹ or 4 mg ai/ seedling	0.13	1	93
Cabbage, head	Spain	F	200 SL	Foliar spray	0.1	0.01	1-2	28
		F	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1-2	28
Cauli-flower	Australia	F	200 SC	Foliar spray	0.06	0.005	1-2 ¹	7
Cauli-flower	Brazil	F	70 WG	Drench treatment, after transplant	0.14-0.21		1	82
Cauli-flower	South Africa	F/G	350 SC	Drench over seedlings before transplanting	0.1-0.2 ¹	0.13	1	136
		F/G	70 WS	Drench over seedlings before transplanting	0.1-0.2 ¹ or 4 mg ai/ seedling	0.13	1	136
Cauli-flower	Spain	F	200 SL	Foliar spray	0.1	0.01	1-2	14
		F	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1-2	14

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Cauli- flower	USA ²	F	192 SC	Foliar spray, interval 5 days	0.052	0.011 ¹	1-5	7
		F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42			21
Kohlrabi	USA ²	F	192 SC	Foliar spray, interval 5 days	0.052	0.011 ¹	5	7
		F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42			21

F: Field; G: greenhouse.

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

Table 61. Registered uses of imidacloprid on cucurbits.

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Bitter gourd	Japan	F/G	10 WP	Foliar spray	0.075 -0.15	0.005	2	1
Cucurbit vegetables	USA ²	F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.28-0.42			21
Cucumber	Australia	F	200 SC	Foliar spray	0.05	0.005	1-2 ¹	1
Cucumber	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	40
Cucumber	Denmark	G	70 WG	Foliar spray or drip irrigation	0.24 (0.01 g/plant)	0.024-0.019	1	3
Cucumber	Greece	F/G	200 SL	Apply in irrigation water	0.15-0.25 (0.01 g/plant)		2	7
Cucumber	Japan	F/G	10 WP	Foliar spray, 3 applications after planting	0.075 -0.15	0.005	1-4	1
		G	10 WP	Cold fogging, 3 applications after planting	0.1	0.2	4	1
Cucumber	Nether- lands	G	70 WG	Foliar spray	0.07	0.0046-0.023 ¹	1	
		G	70 WG	Apply in nutrient solution in rock wool (substrate)	2.45-9.8 g/1000 plants			1
Cucumber	South Korea	F/G	10 WP	Foliar spray	0.075 ¹	0.005	1-4	2
Cucumber	Spain	F/G	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		F/G	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1 ¹	3
Cucurbits	Australia	F	200 SC	Foliar spray	0.06	0.005	1-2 ¹	1
Cucurbits	South Africa	F	350 SC	Drench in planting hole	0.02 g/plant	0.07	1-2	100
Gherkin	Nether- lands	G	70 WG	Foliar spray	0.07	0.0046-0.023 ¹	1	
		G	70 WG	Apply in nutrient solution in rock wool (substrate)	2.45-9.8 g/1000 plants			1
Gherkin	Spain	F/G	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		F/G	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1 ¹	3
Melons	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	40
Melons	Italy	F/G	100 EC	Foliar spray	0.072-0.09	0.009-0.011	1-2 ¹	7
		F/G	200 SL	Foliar spray	0.1-0.15 ¹	0.01-0.015	1	7
Melons	Japan	F/G	10 WP	Foliar spray (3 applications after transplanting)	0.075-0.15	0.005	4	3

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or g ai/plant	Spray conc., kg ai/hl	No.	
Melons	Portugal	F	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
Melons	South Africa	F	350 SC	Soil application in planting hole or after planting	0.21-0.28 ¹ (0.02 g/plant)	0.07	1	100
Melons	Spain	F	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		F	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1-2 ¹	3
Oriental melon	South Korea	F	2 GR	Soil incorporation, at transplanting	0.6		1	
Pumpkin	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	40
Squash, Summer	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	40
Squash, summer	Netherlands	G	70 WG	Apply in nutrient solution in rock wool (substrate)	9.8 g/1000 plants			1
		G	70 WG	Foliar spray, plant treatment	0.07			
Squash, Summer	Spain	F ¹ G	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		F ¹ G	200 SL	Applied in irrigation water	0.10-0.15 (0.01 g/plant)		1 ¹	3
Water-melon	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	40
Water-melon	Italy	F/G	100 EC	Foliar spray	0.09	0.011	1-2 ¹	7
		F/G	200 SL	Foliar spray	0.1-0.15 ¹	0.01-0.015	1	7
Water-melon	Japan	F/G	10 WP	Foliar spray, 3 applications after transplanting	0.075-0.15	0.005	4	3
Water-melon	Philippines	F	100 SL	Foliar spray, interval 7 days	0.015-0.02	0.0038-0.005	1-2	7
Water-melon	South Korea	F	2 GR	Spreading and incorporation, before planting	0.06 g/plant		2	45
Water-melon	Spain	F/G	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		F/G	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1 ¹	3
Water-melon	Thailand		100 SL	Foliar spray	0.025-0.05 ¹	0.005-0.01	1-2 ¹	7

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

Table 62. Registered uses of imidacloprid on fruiting vegetables other than cucurbits.

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or as stated	Spray conc., kg ai/hl	No.	
Egg plant	Australia	F	200 SC	Foliar spray	0.05-0.06	0.005	1-2 ¹	7
Egg plant	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	7
Egg plant	Italy	F/G	200 SL	Foliar spray	0.1-0.15 ¹	0.01-0.015	1	7
Egg plant	Japan	F	10 WP	Foliar spray, 2 applications after transplanting	0.075-0.15	0.005	3	1
		G	10 WP	Cold fogging, 2 applications after transplanting	0.1	0.2	3	1
Egg plant	Netherlands	G	70 WG	Apply in nutrient solution in rock wool (substrate)	2.45-9.8 g/1000 plants			1
		G	70 WG	Foliar spray	0.07			
Egg plant	Spain	F/G	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or as stated	Spray conc., kg ai/hl	No.	
		F/G	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1-2 ¹	3
Egg plant	Turkey	F	350 SC	Foliar spray	0.042-0.07 ¹	0.007		7
Egg plant	USA ²	F	192 SC	Foliar spray	0.053	0.011	1-5 ¹	0
		F	240 SC	Soil application: narrow band or in-furrow or post- seeding drench or sidedress or drip irrigation	0.28-0.42 (0.011 -0.017 g/plant)			21
Peppers	Denmark	G	70 WG	Foliar spray or drip irrigation	0.24 (0.01 g/plant)	0.024-0.019	1	3
Peppers	Italy	F/G	200 SL	Foliar spray	0.10-0.15 ¹	0.01-0.015	1	7
		F/G	100 EC	Foliar spray	0.09	0.011	1-2 ¹	3 G 7 F
Peppers	Nether- lands	G	70 WG	Foliar spray	0.07	0.0175 ¹	1	
		G	70 WG	Applied in nutrient solution in rock wool (substrate)	2.45-9.8 g/1000 plants		1-3 ¹	1
Peppers	Portugal	F	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		G	100 EC	Foliar spray	0.075	0.0075 -0.0093	1-2 ¹	3
Peppers	Spain	F/G	200 SL	Foliar spray	0.1	0.01	1 ¹	3
		F/G	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1-2 ¹	3
Peppers	USA ²	F	192 SC	Foliar spray	0.053	0.011	5	0
		F	240 SC	Soil application: narrow band or in-furrow or post- seeding drench or sidedress or drip irrigation	0.28-0.56 (0.011-0.023 g/plant)			21
Pepper, Green	Japan	F	10 WP	Foliar spray, 2 applications after transplanting	0.075-0.15	0.005	3	1
Pepper, Hot (Chilli)	Thailand	F	100 SL	Foliar spray	0.05-0.1 ¹	0.01-0.02	1-2 ¹	7
Peppers, Sweet	Australia	F	200 SC	Foliar spray	0.06	0.005	1-2 ¹	7
Peppers, Sweet	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	7
Pepper, Red	South Korea	F	10 WP	Foliar spray		0.005	1-4	3
		F	2 GR	Soil incorporation, before planting	0.6		2	30
Sweet corn	Australia	F	600 FS	Seed treatment	2.6 kg/t		1	28
Sweet corn	Germany	F	600 FS	Seed treatment	max. 0.12 ¹ (54 g/unit) ³		1	100
Tomato	Australia	F	200 SC	Foliar spray	0.06	0.005	1-2 ¹	3
Tomato	Brazil	F	70 WG	Drench treatment	0.14-0.21		1	7
Tomato	Canada	F	240 SC	Soil application, band in-furrow	0.11-0.31 (1.7-2.4 g/ 100 m row)		1	
		F	240 SC	Foliar spray; interval 5 days	0.048	0.0048 ¹	1-2	7
Tomato	Denmark	G	70 WG	Foliar spray or drip irrigation	0.24 (0.01 g/plant)	0.024-0.019	1	3
Tomato	Greece	F	200 SL	Foliar spray	0.1 ¹	0.01	2	7
		G	200 SL	Apply in irrigation water	0.15-0.25 (0.01 g/plant)		2	7
Tomato	Italy	F/G	200 SL	Foliar spray	0.1-0.15 ¹	0.01-0.015	1	7
		F/G	100 EC	Foliar spray	0.09	0.011	1-2 ¹	3 G 7 F

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or as stated	Spray conc., kg ai/hl	No.	
Tomato	Japan	F	10 WP	Foliar spray, 2 applications after transplanting	0.075-0.15	0.005	3	1
Tomato	Nether- lands	G	70 WG	Foliar spray	0.07	0.0047-0.023 ¹	1	
		G	70 WG	Applied in nutrient solution in rock wool (substrate)	2.4-9.8 g /1000 plants		1-3 ¹	1
Tomato	Portugal	F	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		G	100 EC	Foliar spray	0.075	0.0075 -0.0094	1-2 ¹	3
Tomato	South Africa	G	350 SC	Soil drench, 1 day after transplanting, interval 28 days	0.018 g/plant	0.018	1-2	23
Tomato	Spain	G/F	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		G/F	200 SL	Applied in irrigation water	0.1-0.15 (0.01 g/plant)		1-2 ¹	3
Tomato	Thailand	F	100 SL	Foliar spray	0.1 ¹	0.02	1-2 ¹	14
Tomato	Turkey	F	350 SC	Foliar spray	0.042-0.07 ¹	0.007	1-2 ¹	7
Tomato, incl. Ground Cherry, Tomatillo	USA ²	F	192 SC	Foliar spray	0.05	0.011	5	0
		F	240 SC	Soil application: narrow band or in-furrow or post- seeding drench or sidedress or drip irrigation	0.28-0.42 (0.011 -0.017 g/plant) (based on 10.000 plants/acre)			21

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

³ 1 unit = 50,000 seeds. Coated seeds.

Table 63. Registered uses of imidacloprid on leafy vegetables (including brassica leafy vegetables).

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha/g ai/100 m row/g ai/plant	Spray conc., kg ai/hl	No.	
Leafy vegetables (Amarant, Arrugula, Chervil, Chrysanthemum edible leafed, Chrysanthemum garland, Collards, Corn salad, Cress, Dandelion, Dock, Endive, Lettuce head, Lettuce leaf, Orach, Parsley, Purslane, Radicchio, Spinach)	USA ²	F	192 SC	Foliar spray, interval 5-7 days	0.053	0.011 ¹	5	7
		F	240 SC	Soil application at planting or up to 14 days before planting or as post-seeding drench	0.18-0.42 (1.4-2.1 g/100m row)			21
Kale	Brazil		70 WG	Drench treatment, after transplanting	0.14-0.21		1	82
Kale	USA ²		192 SC	Foliar spray, interval 5 days	0.053	0.011 ¹	5	7
			240 SC	Soil application at planting or up to 14 days before planting or as post-seeding drench	0.18-0.42			21
Lettuce	Brazil		70 WG	Foliar spray	0.21	0.017-0.035	1-2 ¹	14
Lettuce	South Korea		10 WP	Foliar spray	0.075 ¹	0.005	1-3	5
Lettuce	Spain		200 SL	Foliar spray	0.1	0.01	1-2 ¹	3

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha/g ai/100 m row/g ai/plant	Spray conc., kg ai/hl	No.	
			200 SL	Applied in irrigation water	0.1 - 0.14 (0.01 g/plant)		1-2 ¹	3
Mustard greens	USA ²	F	192 SC	Foliar spray, interval 5-7 days	0.053	0.011 ¹	5	7
		F	240 SC	Soil application: at planting or up to 14 days before planting or as post-seeding drench	0.18-0.42			21
Rape greens	USA ²	F	192 SC	Foliar spray, interval 5-7 days	0.053	0.011 ¹	5	7
		F	240 SC	Soil application: at planting or up to 14 days before planting or as post-seeding drench	0.18-0.42			21
Swiss chard	USA ²		240 SC	Soil application: at or after planting	0.18-0.42			45

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

Table 64. Registered uses of imidacloprid on legume vegetables.

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha or kg ai/t seed or g ai/plant	Spray conc., kg ai/hl	No.	
Bean	Brazil	F	70 WS	Seed treatment	0.084 ¹ (1.4 kg/t seed)		1	
		F	70 WG	Foliar spray	0.11-0.18	0.021-0.044 ¹	1-2 ¹	21
Bean, green	Spain	G	200 SL	Foliar spray	0.1	0.01	1-2 ¹	3
		G	200 SL	Applied in irrigation water	0.1 - 0.14 (0.01 g/plant)		1-2 ¹	3
Bean, edible podded (incl. Runner bean, Snap bean, Wax bean, Asparagus bean, Chinese long bean, Moth bean, Yard-long bean, Jackbean)	USA ² (except CA)	F	240 SC	Soil application: narrow band or in-furrow spray or post seeding drench	0.28-0.42			21
		F	192 SC	Foliar spray, interval 6-7 days	0.049	0.01	max. 3 ¹	7
Bean and peas, succulent shelled (incl. Lima bean green, Broad bean succulent, Blackeyed pea, Cow pea, Southern pea)	USA ² (except CA)	F	240 SC	Soil application: narrow band or in-furrow spray or post seeding drench	0.28-0.42			21
		F	192 SC	Foliar spray	0.049	0.01	max. 3 ¹	7

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

Table 65. Registered uses of imidacloprid on root and tuber vegetables.

Crop	Country	Form.	Application				PHI, days
			Method	kg ai/ha, or kg ai/t seed or g ai/100 m row	Spray conc., kg ai/hl	No.	
Root and tuber vegetables (Chinese artichoke and others) ³	USA ²	240 SC	Soil treatment, at planting and no more than 45 days after planting	1.4-3.8 g/100 m row		1-2	3 leaves 125 corms
Potato	Australia	200 SC	Foliar spray	0.06	0.005	1-2 ¹	7
Potato	Austria	600 FS	Seed treatment	0.09 kg/t		1	
		600 FS	Spray at planting	0.18	0.23-0.3	1	
Potato	Brazil	70 WG	Foliar spray	0.07-0.25	0.007-0.05	1-2 ¹	21
Potato	Canada	240 SC	Soil application, band in-furrow	0.2-0.31		1	
		240 SC	Foliar spray	0.048	0.0096	1-2	7
Potato	Germany	600 FS	Seed treatment	0.18 ¹ (0.072 kg/t)	0.23-0.3 ¹	1	
Potato	Greece	200 SL	Foliar spray	0.1	0.01 ¹	1-2 ¹	14
Potato	Italy	200 SL	Foliar spray	0.1 ¹ 0.15 ¹	0.01-0.015	1	14
		100 EC	Foliar spray	0.072	0.009	1-2	14
		350 FS	Seed treatment	0.25-0.44 ¹ (0.14-0.25 kg/t)		1	
Potato	Japan	10 WP	Foliar spray, 2 applications after planting	0.15-0.3 ¹	0.0067-0.01	3	14
Potato	Nether- lands	350 SC	Soil treatment in- furrow at planting	0.18 (0.08-0.11 kg/t) ¹		1	
Potato	Portugal	100 EC	Foliar spray	0.075	0.0075- 0.009	1-2	21
		200 SL	Foliar spray	0.11-0.12 ¹	0.015	1-2 ¹	21
Potato	South Africa	350 SC	Soil treatment	0.14-0.21 ¹	0.039-0.052 ¹	1	120
Potato	South Korea	10 WP	Foliar spray	0.075 ¹	0.005	1-4	2
		2 GR	Spreading and soil incorporation, before planting	0.6		1	30
Potato	Spain	200 SL	Foliar spray	0.1-0.15	0.01-0.015 ¹	1 ¹	30
		200 SL	Applied in irrigation water	0.15	0.015 ¹	1 ¹	30
		350 FS	Seed treatment	0.14 kg/t		1	
Potato	Turkey	600 FS	Seed treatment	0.21 kg/t		1	
		70 WS	Seed treatment	0.11-0.21 kg/t		1	
		350 SC	Foliar spray	0.053			14
Potato	USA ² (except CA)	240 SC	Soil application: narrow band, in- furrow, sidedress	0.35 (2-3 g/100 m row)			
		240 SC	seed-piece treatment	0.28 (0.13 kg/t)		1	
		192 SC	Foliar spray, interval 7 days	0.052	0.018 ¹	2	7
Sugar beet	Austria	70 WS	Seed treatment	0.017 ¹ (0.09 kg/unit) ⁴		1	
		600 FS	Seed treatment	0.017 ¹ (0.09 kg/unit) ⁴		1	
Sugar beet	France	70 WS	Seed treatment	0.091 ¹ (0.091 kg/unit) ⁴		1	
Sugar beet	Germany	70 WS	Seed treatment	max. 0.12 (0.091 kg/unit) ⁴		1	
		600 FS	Seed treatment	max. 0.12 (0.09 kg/unit) ⁴		1	
		190 FS	Seed treatment	max. 0.02 (0.015 kg/unit) ⁴		1	

Crop	Country	Form.	Application				PHI, days
			Method	kg ai/ha, or kg ai/t seed or g ai/100 m row	Spray conc., kg ai/hl	No.	
Sugar beet	Japan	10 WP	Drench at planting		0.17	1	
Sugar beet	UK	70 WS	Seed treatment	0.12 ¹ (0.091 kg/unit) ⁴		1	
		600 FS	Seed treatment	0.12 ¹ (0.09 kg/unit) ⁴		1	
Sugar beet	Nether- lands	70 WS	Seed treatment	0.12 ¹ (0.091 kg/unit) ⁴		1	
Sugar beet	Sweden	70 WS	Seed treatment	0.11 ¹ (0.09 kg/unit) ⁴		1	
Sugar beet	Greece	350 FS	Seed treatment	0.12-0.18 ¹ (0.06-0.09 kg/unit) ⁴		1	
Sugar beet	Italy	100 EC	Foliar spray	0.072	0.009	1-2 ¹	30
		70 WS	Seed treatment	0.08-0.16 ¹ (0.046-0.09 kg/unit) ⁴		1	
Sugar beet	Spain	70 WS	Seed treatment	0.091 kg/unit ⁴		1	
Sugar beet	Turkey	70 WS	Seed treatment	5.3 kg/t		1	
		600 FS	Seed treatment	9 kg/t		1	
Sweet potato	Australia	200 SC	Foliar spray	0.05	0.005	1-2 ¹	7

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than 0.35 kg ai/ha per season.

³ Root and tuber vegetables include arracacha, arrow-root, chinese artichoke, jerusalem artichoke, canna edible, cassava bitter and sweet, chayote root, chufa, dasheen, ginger, leren, sweet potato, tanier, turmeric, yam bean, yam true.

⁴ 1 unit sugar beet seeds = 100,000 seeds. Pelleted seeds.

Table 66. Registered uses of imidacloprid on stalk and stem vegetables in the USA, except California, PHI 45 days.¹

Crop	F/G	Form.	Application			
			Method	kg ai/ha	Spray conc., kg ai/hl	No.
Cardoon	F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42	Not stated on label	Not stated on label
Celery	F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42	Not stated on label	Not stated on label
Celery, Chinese	F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42	Not stated on label	Not stated on label
Celtuce	F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42	Not stated on label	Not stated on label
Rhubarb	F	240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42	Not stated on label	Not stated on label

¹ Regardless of formulation or method of application, apply not more than 0.56 kg ai/ha per season, including seed treatment, soil and foliar uses, max. 6 field applications per season. Do not graze treated fields after any application of "Admire".

Table 67. Registered uses of imidacloprid on grasses (cereals and sugar cane).

Crop	Country	Form.	Application				PHI, days
			Method	kg ai/ha, kg ai/t seed	Spray conc., kg ai/hl	No.	
Barley	Australia	600 FS	Seed treatment	0.72-1.4 kg/t		1	note ³
Barley, winter	Belgium	375 FS	Seed treatment	0.7 kg/t		1	
Barley	France	375 FS	Seed treatment	0.7 kg/t		1	
Barley	Germany	350 FS	Seed treatment	0.35 kg/t		1	
		145 FS	Seed treatment	0.06 (0.35 kg/t)		1	
Maize	Australia	600 FS	Seed treatment	2.6 kg/t		1	note ⁴

Crop	Country	Form.	Application				PHI, days
			Method	kg ai/ha, kg ai/t seed	Spray conc., kg ai/hl	No.	
Maize	Austria	600 FS	Seed treatment	8.6-9.2 kg/t		1	
Maize	Brazil	70 WS	Seed treatment	0.098-0.14 ¹ (4.9-7 kg/t)		1	
Maize	France	70 WS	Seed treatment	0.1 (49 g/unit) ⁵		1	
Maize	Germany	600 FS	Seed treatment	0.11 ¹ (54 g/unit) ⁵		1	-
Maize	Greece	350 FS	Seed treatment	0.12-0.18 ¹ (4.9-7 kg/t)		1	
Maize	Italy	350 FS	Seed treatment	3.5-7 kg/t		1	
Maize	Nether-lands	70 WS	Seed treatment	60 g/unit		1	
Maize	South Africa	600 FS	Seed treatment	0.7- 3.5 kg/t		1	65 ²
		70 WS	Seed treatment	0.7-3.5 kg/t		1	65 ²
Maize	Turkey	600 FS	Seed treatment	3.6 kg/t		1	
		70 WS	Seed treatment	3.5 kg/t		1	
Oat	Australia	600 FS	Seed treatment	0.72-1.4 kg/t		1	note ³
Oat	France	337.5 F	Seed treatment	0.7 kg/t		1	
Oat	Germany	350 FS	Seed treatment	0.35 kg/t		1	-
Rice	Brazil	70 WS	Seed treatment	0.084-0.13 ¹ (1.4-2.1 kg/t)		1	
Rice	Japan	10 WP	Foliar spray, 2 applications in paddy	0.03-0.075	0.005	3	30
		10 WP	Seed treatment, before sowing direct in flooded paddy field	0.15-0.2		1	
		10 WP	Drench to nursery box, 0-2 days before transplanting	0.1 ⁷	0.1	1	
Rice	South Korea	10 WP	Foliar spray	0.03 ¹	0.002	1-3	45
		40 SL	Foliar spray	0.03 ¹	0.002	1-3	15
		2 GR	Nursery box, at transplanting	1.5 g/box		2	
Rice	Thailand	100 SL	Foliar spray	0.038 ¹	0.0075	1-2 ¹	21
Rye	France	337.5 FS	Seed treatment	0.7 kg/t		1	
Rye	Germany	350 FS	Seed treatment	0.35 kg/t		1	-
Sorghum	Australia	600 FS	Seed treatment	2.6 kg/t		1	note ⁴
Sorghum	South Africa	70 WS	Seed treatment	1.8 kg/t		1	55 ²
Sugar cane	Brazil	70 WG	Soil treatment, in-furrow	0.28		1	
Triticale	Australia	600 FS	Seed treatment	0.72-1.4 kg/t		1	note ³
Triticale	France	337.5 FS	Seed treatment	0.7 kg/t		1	
Triticale	Germany	350 FS	Seed treatment	0.35 kg/t		1	
Wheat	Australia	600 FS	Seed treatment	0.72-1.4 kg/t		1	note ³
Wheat	Belgium	337.5 FS	Seed treatment	0.7 kg/t		1	
Wheat	Brazil	70 WS	Seed treatment	0.056 ¹ (0.35 kg/t)		1	
		350 FS	Seed treatment	0.36 kg/t		1	
Wheat	France	337.5 FS	Seed treatment	0.7 kg/t		1	
Wheat	Germany	350 FS	Seed treatment	0.35 kg/t		1	
Wheat	Turkey	70 WS	Seed treatment	1.4 kg/t		1	
Wheat	South Africa	70 WS	Seed treatment	1.4 kg/t		1	100 ²

¹ Not specified on label.

² South Africa: days between planting and feeding.

³ Australia: do not graze cereal plants grown from treated seeds, or cut for stock food, within nine weeks of sowing.

⁴ Australia: do not graze maize, sorghum or sweet corn plants grown from treated seed, or cut for stock food, within four weeks of sowing.

⁵ Germany: 1 unit maize seeds = 50,000 seeds. Coated seeds.

⁶ Japan: applied as seed dressing together with calcium peroxide to enhance germination.

⁷ Japan: 500 mg/box, 200 boxes/ha.

Table 68. Registered uses of imidacloprid on nuts and seeds (tree nuts, oilseeds, coffee beans).

Crop	Country	Form.	Application				PHI, days
			Method	kg ai/ha, or g ai/plant or kg ai/t seed	Spray conc., kg ai/hl	No.	
Coffee	Brazil	70 WG	Drench treatment	0.7-0.91 ² (0.035 g/plant) ³	0.07-0.23	1	45
Cotton	Australia	600 FS	Seed treatment	0.07-0.14 ⁴ (3.5-0.7 kg/t)		1	
		200 SC	Foliar spray: ground or aerial application	0.05	0.05 ¹ (aerial 0.2 ¹)	1-2 ¹	91
Cotton	Brazil	70 WG	Foliar spray	0.049-0.07	0.01-0.018	1-2 ¹	30
		70 WS	Seed treatment	0.042-0.052 ¹ (2.8-3.5 kg/t)		1	
Cotton	Egypt	200 SL	Foliar spray	0.048-0.096 ¹	0.01	1 ¹	7
		350 SC	Foliar spray	0.13-0.25 ¹	0.026	1-2 ¹	7
		70 WS	Seed treatment	0.29 (4.9 kg/t)		1	
Cotton	Greece	350 FS	Seed treatment	0.11-0.18 ¹ (5.3-7 kg/t)		1	
		200 SL	Foliar spray	0.1	0.01 ¹	1-2	28
Cotton	South Africa	350 SC	Foliar spray (ground or aerial)	0.042	0.021 (aerial 0.105)	1-3 ¹	7
Cotton	Thailand	100 SL	Foliar spray	0.075-0.15 ¹	0.015	1-2 ¹	14
Cotton	Turkey	350 SC	Foliar spray	0.12-0.3	0.031-0.05 ¹	1-2 ¹	14
		600 FS	Seed treatment	0.14 ⁴ (4.2 kg/t)		1	
Cotton	USA ⁴	240 SC	Soil application: 7 or fewer days before planting or in-furrow at planting	0.37 (0.003 kg/100 m row)			
		192 SC	Foliar spray, interval 7-10 days	0.028-0.052	0.06-0.11	max. 5	14
Hazel-nuts	Spain	200 SL	Foliar spray	0.1	0.01 ¹		
Pecan	USA ⁴ (except CA)	192 SC	Foliar spray	0.049-0.2	0.0033 - 0.013 ¹	2	
		240 SC	Soil application: irrigation, spot treatment, shanked-in treatment	0.28-0.56		1-2 ¹	
Pistachio nut	Turkey	350 SC	Foliar spray	0.17-0.22 ¹	0.011		14
Rape	Australia	600 FS	Seed treatment	0.01 kg/t (2.4)		1	
Rape	Germany	200 FS	Seed treatment	0.01 kg/t (2)		1	-
Rape, winter	UK	200 FS	Seed treatment	0.01 ¹ kg/t (2)		1	
Sun-flower	Australia	600 FS	Seed treatment	2.6 kg/t		1	
Sun-flower	Italy	350 FS	Seed treatment	7-11 kg/t		1	
Sun-flower	South Africa	70 WS	Seed treatment	3.5 kg/t		1	63 ⁵

¹ Not specified on label.

² For coffee plants older than 2 years.

³ For coffee plants up to 2 years.

⁴ Regardless of formulation or method of application, apply not more than 0.56 kg ai/ha per season, including seed treatment, soil and foliar uses. Do not apply more than total of 6 field applications per season. Do not graze treated fields after any application of "Admire".

⁵ South Africa: days between planting and feeding.

Table 69. Registered uses of imidacloprid on miscellaneous fodder and forage crops.

Crop	Country	Form.	Application				PHI, days
			Method	kg ai/ha or kg ai/unit	Spray conc., kg ai/hl	No.	
Fodder beet	France	70 WS	Seed treatment	0.091 ¹ (0.091 kg/unit)		1	
Fodder beet	Germany	70 WS	Seed treatment	max. 0.12 (0.091 kg/unit)		1	
		600 FS	Seed treatment	max. 0.12 (0.09 kg/unit)		1	
		190 FS	Seed treatment	max. 0.02 (0.015 kg/unit)		1	
Fodder beet	UK	600 FS	Seed treatment	0.12 ¹ (0.09 kg/unit)		1	
Fodder beet	Netherlands	70 WS	Seed treatment	0.091 kg/unit		1	
Turnip tops (leaves)	USA ²	192 SC	Foliar spray, interval 5-7 days	0.052	0.011 ¹	5	7
		240 SC	Soil application: narrow band or in-furrow or post-seeding drench or sidedress or drip irrigation	0.18-0.42			21

¹ Not specified on label.

² Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

Table 70. Registered uses of imidacloprid on hops, tea and tobacco.

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha, or g ai/plant	Spray conc., kg ai/hl	No.	
Hops, dry	Austria	F	70 WG	Foliar spray	0.12	0.0035	1	35
		F	70 WG	Stem painting	0.12	1.9	1	35
Hops, dry	Germany	F	70 WG	Foliar spray	0.12	0.0035	1	35
		F	70 WG	Stem painting	0.12	1.9-3.9	1	35
Hops, dry	UK	F	70 WG	Directed stem base spray	0.125	0.024-0.034	1	Note ²
Hops, dry	Spain	F	200 SL	Foliar spray	0.1	0.01 ¹	1	35
Hops, dry	USA	F	192 SC	Foliar spray	0.11	0.0037 ¹	1-3	28
Tea	Japan	F	10 WP	Foliar spray	0.1-0.4	0.005-0.01	1	14
Tobacco (bed and field)	Brazil	F	70 WG	Foliar spray	0.25		1	
Tobacco (nursery plants)	Germany	G	70 WG	Addition to water for swimming plants, stages 12-16: until 40 days after seeding	2.8 g/1000 plants		1	
		G		Foliar spray or watering: until 60 days after seeding	0.35 g/m ²		1	
Tobacco	Greece	F	200 SL	Foliar spray	0.05-0.06	0.005-0.006 ¹	1-2 ¹	7
		F		Apply to irrigation water	0.1-0.2		1 ¹	7
Tobacco	Italy	F	100 EC	Foliar spray	0.072	0.009	1-2 ¹	30
Tobacco	Japan	F	10 WP	Foliar spray, 1 application after planting		0.005	2	10
Tobacco	Portugal	F	200 SL	Foliar spray	0.1	0.01	1-2 ¹	14

Crop	Country	F/G	Form.	Application				PHI, days
				Method	kg ai/ha, or g ai/plant	Spray conc., kg ai/hl	No.	
Tobacco	South Africa	F	350 SC	Drench at transplanting	0.23 ¹	0.025	1	70
Tobacco	South Korea	F	2 GR	Soil spreading and incorporation at transplanting	0.4 g/plant		1	
Tobacco	Spain	F	200 SL	Foliar spray	0.1	0.01 ¹	1-2 ¹	15
Tobacco	Turkey	F	350 SC	Foliar spray	0.049	0.007-0.12 ¹	1-2 ¹	14
Tobacco	USA ³	F	192 SC	Foliar spray	0.028-0.056			14
		F/G	240 SC	Drench at transplanting	0.007-0.02 g/plant			14

¹ Not specified on label.

² UK: latest permitted timing of application before vines reach 2 m in length or before the end of the first week in June.

³ Regardless of formulation or type of application (soil or foliar), do not apply more than a total of 0.56 kg ai/ha per season.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on imidacloprid supervised residue trials for

Fruits	Tables	71-78	Citrus fruits
	Tables	79-80	Apple and pear
	Tables	81-83	Stone fruits
	Table	84	Grapes
	Table	85	Banana
	Table	86	Mango
Vegetables	Table	87	Leek
	Table	88	Onions, Bulb
	Table	89	Broccoli
	Table	90	Cauliflower
	Table	91	Brussels sprouts
	Table	92	Cabbages, Head
	Table	93	Cucumber
	Table	94	Squash, Summer
	Table	95	Melons, except watermelon
	Table	96	Watermelon
	Table	97	Egg plant
	Tables	98-99	Peppers
	Tables	100-102	Tomatoes
	Table	103	Sweet corn (corn-on-the-cob)
	Tables	104-107	Endive and lettuce
Cereals	Tables	108-109	Beans
	Tables	110-112	Potato
	Tables	113-114	Sugar beet
	Table	115	Celery
	Tables	116-123	Barley, maize, oats, rice, triticale, wheat
Nuts and Seeds	Table	124	Pecans
	Tables	125-126	Cotton seed
	Table	127	Rape seed

Further crops	Tables 128-129	Hops
	Table 130	Coffee beans
	Table 131	Tea

According to the definition of the residue, the residue to be determined in plant and products of animal origin should be the “*sum of imidacloprid and its metabolites containing the 6-chloropyridyl moiety, all expressed as imidacloprid*”. This is referred to as the “*total residue*” in the Tables below.

However, the definition of the residue differs in various countries. In Japan and Korea, it is imidacloprid itself, and in Brazil, South Africa, Australia and some other countries it was changed from “imidacloprid” to the “*total residue of imidacloprid*”. In the early residue trials therefore the parent compound only was determined in some countries, and only later the total residue. In the Tables below both imidacloprid and total residues are reported from these countries. In the USA and European countries the residue definition is the “*sum of imidacloprid and its metabolites containing the 6-chloropyridyl moiety, all expressed as imidacloprid*” (total residue). In the US trials, only the “total residue” was determined. In the European trials the total residue and the residues of the parent compound were determined in parallel at day 0 and at the envisaged PHIs. Both values are reported in the Tables, but only the total residue values are discussed in the appraisal.

Where residues were not detected, they are reported as below the LOQ, e.g. <0.05 mg/kg. Residue data, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Although trials included control plots, no control data are given except when these residues exceeded the LOQ. Residues are recorded unadjusted for procedural recoveries if not otherwise stated.

Most trials were carried out on single plots. Where replicate residues are shown they represent samples from split plots. Periods of freezer storage between sampling and analysis were recorded for all trials and were within the acceptable determined stability period. Double underlined residue values are from treatments according to maximum GAP and were used for estimating maximum residue levels, STMRs and HRs.

Citrus fruits (foliar spray treatment (Table 71-75). Field trials were conducted on clementine, grapefruit, lemon, mandarin and orange trees in Europe and USA.

In Italy, in eight field trials, two foliar spray applications of a 200 SL formulation were made to clementine trees at an interval of 30 days at a concentration of 0.01 kg ai/hl, 1200-1600 l/ha (Table 71).

In the USA six field trials were carried out on the foliage of grapefruit trees. A 240 SC formulation was sprayed twice at a 10 ($\pm$ 2) day interval at about 0.28 kg ai/ha and 0.011-0.015 kg ai/hl or 0.04-0.043 kg ai/hl (Table 72).

In Italy in two residue field trials lemon trees were sprayed twice (interval 30 days) with a 200 SL formulation at 0.01 kg ai/hl, 4100 and 1500 l/ha, 0.41 and 0.15 kg ai/ha respectively. In the USA in five field trials the trees were again sprayed twice (interval 9–11 days) with a 240 SC formulation. The trees in each trial were treated with imidacloprid at 0.28 kg ai/ha (Table 73).

In five field trials on mandarin trees in Italy, Portugal and Spain the trees were sprayed twice (interval Italy 30, Portugal 34, Spain 118 days). In the trial in Italy the concentration was 0.01 kg ai/hl, corresponding to 0.12 kg ai/ha, and in Portugal 0.015 mg/kg corresponding to 0.2 kg ai/ha. In the three trials in Spain after the first treatment at 0.3 kg ai/ha the second was at 0.75 kg ai/ha (Table 74).

In southern European countries 11 field trials on oranges, 9 according to GAP, were with two spray applications of a 200 SL formulation. In three trials in Italy the trees were sprayed at an interval of 30 days at 0.01 kg ai/hl, corresponding to 0.12 kg ai/ha. In Spain five trials were conducted with concentrations of 0.01 kg ai/hl for the first treatment and 0.015 kg ai/hl for the second (interval 101-130 days), corresponding to 0.3 kg ai/ha and 0.45-0.75 kg ai/ha. Two trials in Greece and one in

Portugal were in accordance with Greek GAP: two sprays (interval Greece 9-10, Portugal 31 days) were made each at 0.015 kg ai/hl, corresponding to about 0.3 kg ai/ha.

In the USA in 12 field trials orange trees were sprayed twice at an interval of 3.13 days with a 240 SC formulation at 0.28 kg ai/ha (Table 75).

Table 71. Residues in clementines after spraying in Italy.

Year	Application				Residues, mg/kg				Report Study No.	no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI, days	Imidacloprid	Total residue		
1991	200 SL	2	0.12	0.01	peel	0	0.24	0.52	RA-2018/91 0279-91	
						14		0.40		
						21		0.28		
						28		0.33		
					pulp	0	<0.01	0.07		
						14		<0.05		
						21		<0.05		
						28		<0.05		
					whole fruit, calculated	0	0.07	0.18		
						14		0.12		
						21		0.09		
						28		0.11		
1992	200 SL	2	0.12	0.01	pulp	0 ¹	0.01	0.06	RA-2050/92 0252-92	
						0		<0.05		
						7		<0.05		
						14		<0.05		
						21		<0.05		
						28		<0.05		
					peel	0 ¹	0.32	0.49		
						0		0.18		
						7		0.41		
						14		0.72		
						21		0.40		
						28		0.43		
					whole fruit, calculated	0 ¹	0.1	0.16 ²		
						0		0.07 ²		
						7		0.12		
						14		0.21		
						21		0.13		
						28		0.13		
1992	200 SL	2	0.12	0.01	pulp	0 ¹	0.01	<0.05	RA-2050/92 0253-92	
						0		0.09		
						7		<0.05		
						14		<0.05		
						21		<0.05		
						28		<0.05		
					peel	0 ¹	0.16	0.12		
						0		0.32		
						7		0.34		
						14		0.23		
						21		0.24		
						28		0.26		

Year	Application				Residues, mg/kg				Report Study No.	no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI, days	Imidacloprid	Total residue		
					whole fruit, calculated	0 ¹ 0 7 14 21 28	0.04	<0.05 0.13 0.1 <u>0.07</u> 0.07 0.07		
1993	200 SL	2	0.16	0.01	pulp	0 ¹ 0 7 14 21 28	0.02	0.05 0.07 0.05 <u>0.05</u> 0.05	RA-2020/93 0012-93	
						0 ¹ 0 7 14 21 28	0.01	0.05 0.05 0.05		
					peel	0 ¹ 0 7 14 21 28	0.16	0.38 1.1 0.57 0.43		
						0 ¹ 0 7 14 21 28	0.2	0.53 0.42		
						0 ¹ 0 7 14 21 28	0.05	0.13 0.32 0.17 0.13		
					whole fruit, calculated	0 ¹ 0 7 14 21 28	0.05	<u>0.16</u> 0.13		
						0 ¹ 0 7 14 21 28	0.05	0.13 0.32 0.17 0.13		
					pulp	0 ¹ 0 7 16 21 28	<0.01	<0.05 0.05 <0.05 <u><0.05</u> <0.05		
						0 ¹ 0 7 16 21 28	<0.01	<0.05 0.05 0.05 0.05		
						0 ¹ 0 7 16 21 28	0.07	0.12 0.36 0.19 0.16		
1993	200 SL	2	0.12	0.01	peel	0 ¹ 0 7 16 21 28	0.06	0.1 0.17	RA-2020/93 0383-93	
						0 ¹ 0 7 16 21 28	0.06	0.1 0.17		
						0 ¹ 0 7 16 21 28	0.06	0.1 0.17		
						0 ¹ 0 7 16 21 28	0.06	0.1 0.17		
						0 ¹ 0 7 16 21 28	0.06	0.1 0.17		
					whole fruit, calculated	0 ¹ 0 7 16 21 28	0.02	<0.05 0.13 0.06 <u>0.06</u> <0.05		
						0 ¹ 0 7 16 21 28	0.02	<0.05 0.13 0.06 <u>0.06</u> <0.05		
						0 ¹ 0 7 16 21 28	0.02	<0.05 0.13 0.06 <u>0.06</u> <0.05		
						0 ¹ 0 7 16 21 28	0.02	<0.05 0.13 0.06 <u>0.06</u> <0.05		
1994	200 SL	2	0.16	0.01	pulp	0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06	RA-2057/94 0216-94	
						0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		
						0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		
						0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		
						0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		
					whole fruit, calculated	0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		
						0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		
						0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		
						0 ¹ 0 14 21 28	0.02	<0.05 0.05 0.05 <u>0.06</u> 0.06		

Year	Application				Residues, mg/kg				Report Study No.	no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI, days	Imidacloprid	Total residue		
					peel	0 ¹ 0 14 21 28	0.57 0.52	0.7 1.7 1.0 0.73 0.58		
					whole fruit, calculated	0 ¹ 0 14 21 28	0.16 0.16	0.19 0.41 <u>0.29</u> 0.21 0.19		
					pulp	0 ¹ 0 14 21 28	0.01 0.01	<0.05 <0.05 <u>0.05</u> <0.05 0.05		
					peel	0 ¹ 0 14 21 28	0.58 0.80	0.78 1.6 1.3 0.91 1.1		
					whole fruit, calculated	0 ¹ 0 14 21 28	0.15 0.24	0.21 0.43 <u>0.38</u> 0.26 0.35		
1994	200 SL	2	0.16	0.01	pulp	0 ¹ 0 14 21 28	0.01 0.02	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2057/94 0217-94	
					peel	0 ¹ 0 14 21 28	0.37 0.76	0.58 1.9 1.6 1.1 1.3		
					whole fruit, calculated	0 ¹ 0 14 21 28	0.11 0.23	0.17 0.54 <u>0.44</u> 0.32 0.4		

¹ Before last treatment.² Comment by manufacturer: day-0 samples may be have been accidentally exchanged.

Table 72. Residues in grapefruit after spray applications, USA.

Location, Year	Application				Residues, mg/kg				Report Study No. <i>no.</i>
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
TX 1993	240 SC	2	0.28	0.012	whole fruit	0 7 14 21		<u>0.3</u> 0.21 0.23 0.19	106437 352-CO006-93D
CA 1993	240 SC	2	0.28	0.011	whole fruit	0 7 14 21		0.14 <u>0.17</u> 0.15 0.1	106437 FCA-CO009-93D
CA 1993	240 SC	2	0.28	0.043	whole fruit	0 7 14 21		<u>0.32</u> 0.3 0.12 0.12	106437 458-CO007-93D
FL 1993	240 SC	2	0.28	0.043	whole fruit	0 7 14 21		<u>0.17</u> 0.16 0.05 <0.05	106437 753-CO008-93D
FL 1993	240 SC	2	0.28	0.04	whole fruit	0 7 14 21		<u>0.18</u> 0.14 0.06 <0.05	106437 VBL-CO010-93D
FL 1995	240 SC	2	0.29	0.015	whole fruit	0 0		0.13 <u>0.14</u>	106437-1 VBL-AD014-95H

Table 73. Residues in lemons after foliar spray applications.

Country Year	Application				Residues, mg/kg				Report Study No. <i>no.</i>
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI days	Imidacloprid	Total residue	
Italy 1991	200 SL	2	0.41	0.01	pulp	0	0.03	<0.05	RA-2018/91 0281-91
						14		<0.05	
						21		0.06	
						28		<u>0.07</u>	
					peel	0	0.38	0.62	
						14		0.57	
						21		0.54	
						28		0.43	
					whole fruit	0	0.17	0.26	
						14		0.25	
						21		<u>0.26</u>	
						28		0.22	

Country Year	Application				Residues, mg/kg				Report Study No.	no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI days	Imidacloprid	Total residue		
Italy 1992	200 SL	2	0.15	0.01	pulp	0 ¹		0.08	RA-2050/92 0261-92	
						0		0.16		
						7		0.09		
						14		0.09		
						21	0.04	0.09		
						28	0.06	0.11		
					peel	0 ¹		0.61		
						0		1.4		
						7		1.3		
						14		1.2		
						21	0.7	1.3		
						28	0.53	0.89		
					whole fruit	0 ¹		0.29		
						0		0.64		
						7		0.56		
						14		0.54		
						21	0.3	0.57		
						28	0.26	0.44		
USA, CA 1993	240 SC	2	0.28	0.043	whole fruit	0		0.51	106437 458-CO011-93D	
						7		0.62		
						14		0.55		
						21		0.38		
USA, CA 1993	240 SC	2	0.28	0.01	whole fruit	0		0.31	106437 FCA-CO012-93D	
						7		0.27		
						14		0.25		
						21		0.23		
USA, CA 1996	240 SC	2	0.28	0.041 0.042	whole fruit	0		0.28	106437-1 458-AD015-95H	
						0		0.38		
USA, CA 1996	240 SC	2	0.28	0.042 0.041	whole fruit	0		0.17	106437-1 FCA-AD016-95H	
						0		0.21		
USA, FL 1996	240 SC	2	0.28	0.014 0.013	whole fruit	0		0.23	106437-1 353-AD017-95H	
						0		0.3		

¹ Before last treatment

Table 74. Residues in mandarins after spray applications.

Country Year	Application				Residues, mg/kg				Report Study No.	no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue		
Italy 1991	200 SL	2	0.12	0.01	peel	0		0.42	RA-2018/91 0282-91	
						14		0.52		
						21	0.44	0.5		
						28		0.52		

Country Year	Application				Residues, mg/kg				Report no. Study No.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
					pulp	0 14 21 28	<0.01	<0.05 <u><0.05</u> <0.05 <0.05	
					whole fruit, calculated	0 14 21 28	0.13	0.12 0.15 0.16 <u>0.17</u>	
Portugal 1993	200 SL	2	0.21 0.21	0.015	fruit	0 ¹ 0 7 14 21 28	0.09 0.15	0.16 0.44 0.29 <u>0.29</u> 0.23 0.27	RA-2020/93 0411-93
Spain 1991	200 SL	2	0.3 0.75	0.01 0.015	pulp	0 14 21 28	0.01	<0.05 <u><0.05</u> <0.05 <0.05	RA-2026/91 0196-91
					peel	0 14 21 28	0.45	1.4 1.0 0.77 0.54	
					whole fruit, calculated	0 14 21 28	0.13	0.41 <u>0.28</u> 0.23 0.16	
Spain 1994	200 SL	2	0.3 0.75	0.01 0.015	pulp	0 14 28	0.01	<0.05 <0.05 <u>0.05</u>	RA-2043/94 0350-94
					peel	0 14 28	0.15	1.1 0.57 0.4	
					whole fruit, calculated	0 14 28	0.05	0.3 <u>0.16</u> 0.15	
Spain 1994	200 SL	2	0.3 0.75	0.01 0.015	pulp	0 14 28	0.01	<0.05 <0.05 <u>0.06</u>	RA-2043/94 0349-94
					peel	0 14 28	0.17	1.1 0.55 0.49	
					whole fruit, calculated	0 14 28	0.05	0.27 0.14 <u>0.16</u>	

¹ Before last treatment.

Table 75. Residues in oranges after spray applications.

Country Year	Application				Residues, mg/kg				Report Study No. <i>no.</i>
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
Italy 1991	200 SL	2	0.12	0.01	pulp	0 14 21 28	<0.01	<0.05 <u><0.05</u> <0.05 <0.05	<i>RA-2018/91</i> 0280-91
					peel	0 14 21 28	0.26	0.44 0.39 0.36 0.37	
					whole fruit, calculated	0 14 21 28	0.07	0.15 <u>0.12</u> 0.12 0.1	
					pulp	0 ¹ 0 7 14 21 28	<0.01	<0.05 <0.05 <0.05 <u><0.05</u> <0.05 <0.05	<i>RA-2050/92</i> 0248-92
					peel	0 ¹ 0 7 14 21 28	0.21	0.2 0.4 0.36 0.39 0.34 0.47	
					whole fruit, calculated	0 ¹ 0 7 14 21 28	0.06	0.07 0.11 0.12 0.13 0.1 <u>0.16</u>	
					pulp	0 21 28	<0.01	0.05 <0.05 <0.05	
					peel	0 21 28	0.12	0.32 0.27 0.29	
					whole fruit, calculated	0 21 28	0.03	0.12 0.08 0.1	
Spain 1991	200 SL	2	0.3 0.58	0.01 0.015	pulp	14 21 28	0.01	< <u>0.05</u> <0.05 <0.05	<i>RA-2019/91</i> 0195-1

Country Year	Application				Residues, mg/kg				Report Study No. no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
					peel	14 21 28	0.39	0.73 0.65 0.67	
					fruit	0	0.27	0.27	
					whole fruit, calculated	14 21 28	0.12	<u>0.24</u> 0.2 0.22	
Spain 1991	200 SL	2	0.3 0.75	0.01 0.015	pulp	0 14 21 28	0.02	<0.05 <u><0.05</u> <0.05 <0.05	RA-2019/91 0483-91
					peel	0 14 21 28	0.5	1.6 1.3 1.1 0.31	
					whole fruit, calculated	0 14 21 28	0.14	0.42 <u>0.35</u> 0.3 0.09	
Spain 1991	200 SL	2	0.29 0.45	0.01 0.015	pulp	14 21 28	<0.01	<u><0.05</u> <0.05 <0.05	RA-2019/91 0484-91
					peel	14 21 28	0.1	0.29 0.24 0.26	
					fruit	0	0.28	0.3	
					whole fruit, calculated	14 21 28	0.04	<u>0.11</u> 0.1 0.1	
Spain 1993	200 SL	2	0.3 0.75	0.01 0.015	pulp	0 ¹ 0 7 14 21 29	0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2020/93 0412-93
					peel	0 ¹ 0 7 14 21 29	0.06 0.07	0.22 0.76 0.37 0.36 0.26 0.23	

Country Year	Application				Residues, mg/kg				Report Study No. no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
					whole fruit, calculated	0 0 7 14 21 29	0.02 0.02	0.08 0.22 0.12 <u>0.12</u> 0.09 0.09	
Spain 1993	200 SL	2	0.3 0.75	0.01 0.015	pulp	0 29	0.01 <0.01	0.05 <0.05	RA-2020/93 0413-93
					peel	0 29	1.5 0.13	1.8 0.56	
					whole fruit, calculated	0 29	0.4 0.04	0.51 0.81	
Portugal 1993	200 SL	2	0.28 0.24	0.015	fruit	0 ¹ 0 8 15 22 33	0.36 0.61 0.28	0.5 1.1 1.1 <u>0.88</u> 0.84 0.87	RA-2020/93 0410-93
Greece 1998	200 SL	2	0.3	0.015	pulp	14	0.02	<0.05	RA-2154/98 1353-98
					peel	14	0.93	1.2	
					fruit	0 7 14 21	0.69 0.35	0.62 0.65 0.34 <u>0.44</u>	
					whole fruit, calculated	14	0.26	0.33	
Greece 1999	200 SL	2	0.3	0.015	pulp	15	0.03	<u>0.05</u>	RA-2040/99 0136-99
					peel	15	0.93	1.5	
					fruit	0 7 15 21	0.25 0.5 0.23 0.28	0.35 0.72 <u>0.53</u> 0.44	
USA, CA 1993	240 SC	2	0.28	0.04	fruit	0 7 14 21		<u>0.61</u> 0.41 0.4 0.53	106437 457-CO001-93D
USA, CA 1993	240 SC	2	0.28	0.043	fruit	0 7 14 21		0.26 0.24 <u>0.28</u> 0.24	106437 458-CO002-93D

Country Year	Application				Residues, mg/kg				Report Study No.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
USA, CA 1993	240 SC	2	0.28	0.011	fruit	0 7 14 21		0.11 0.16 <u>0.18</u> 0.16	106437 FCA-CO003-93D
USA, FL 1993	240 SC	2	0.28	0.015	fruit	0 7 14 21		<u>0.26</u> 0.24 0.09 0.15	106437 VBL-CO004-93D
USA, FL 1993	240 SC	2	0.28	0.04	fruit	0 7 14 21		<u>0.29</u> 0.27 0.12 0.19	106437 VBL-CO005-93D
USA, FL 1995	240 SC	2	0.28	0.015	fruit	0 0		0.25 <u>0.26</u>	106437-1 353-AD007-95H
USA, FL 1995	240 SC	2	0.28	0.04	fruit	0 0		<u>0.34</u> 0.34	106437-1 353-AD008-95H
USA, FL 1995	240 SC	2	0.28	0.04	fruit	0 0		0.2 <u>0.21</u>	106437-1 353-AD009-95H
USA, FL 1995	240 SC	2	0.28	0.04	fruit	0 0		0.34 <u>0.36</u>	106437-1 353-AD010-95H
USA, FL 1995	240 SC	2	0.28	0.014	fruit	0 0		0.3 <u>0.37</u>	106437-1 VBL-AD011-95H
USA, FL 1995	240 SC	2	0.28	0.04	fruit	0 0		0.14 <u>0.15</u>	106437-1 VBL-AD012-95H
USA, TX 1995	240 SC	2	0.28	0.012	fruit	0 0		0.27 <u>0.36</u>	106437-1 459-AD013-95H

¹ Before last treatment

Citrus fruits (soil drench application, Tables 76-78). In the USA twenty trials were conducted according to GAP. In 1993 (Report no. 106662) in field trials on grapefruit (6 trials, Table 76) and orange trees (6 trials, Table 77) a 240 SC formulation was applied once to the soil at 0.56 kg ai/ha as a 1.2-1.8 m band, under the dripline on each side of the tree rows, a minimum of about 0.3 to 0.45 m from the trunk, followed by shallow incorporation either in late spring or in the autumn. After treatment in the spring, grapefruit and oranges were harvested after 120, 150, 180, 210, 240, 270, and 365 days, but only one sample at day 365 was analysed, and after treatment in the autumn harvest was after 0, 7, 15, 30, 60, 90, 120, and 150 days.

In a further three trials on grapefruit, three on lemon, and two on orange trees in 1994 (Report no. 106662-1) application was as in 106662, incorporated into the soil with either sprinkler or drip irrigation, rototilling, or hand raking. A reverse decline procedure was used to ensure marketable size fruit at all intervals. Mature grapefruit, oranges, and lemons were harvested 0, 4, 7, 15, 30, 56 to 62, 90 (± 1), 119 to 120, 149 to 153, 208 to 215, 240 to 244, 270 to 274, and 365 (± 1) days after treatment.

In three field trials in South Africa a soil drench application of a 200 SC formulation was made to orange trees after each site had been treated around the trees with either a single label rate of 2 g ai/tree, or at double or triple this rate, in 10 l water/tree, corresponding to 4000 l/ha. Oranges were sampled after 179 and 212 days. Only the parent compound imidacloprid was determined. Three further trials were conducted with a 200 SL formulation.

Table 76. Results of trials in the USA with imidacloprid in grapefruit after soil applications.

Location, Year, Season	Application				PHI (days)	Total residue (mg/kg)	Report no./Study no.
	Form.	No.	kg ai/ha	kg ai/hl			
FL 1993 autumn	240 SC	1	0.56	0.66	0 7 15 30 60 90 120 150	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 353-CO040-92/93D
FL 1993 autumn	240 SC	1	0.56	0.71	0 7 15 30 60 90 121 150	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 VBL-CO041-92/93D
FL 1993 autumn	240 SC	1	0.56	0.72	0 7 15 30 60 88 121 150	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 VBL-CO042-92/93D
FL 1993 spring	240 SC	1	0.56	0.6	120 150 180 210 240 270	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 353-CO046-92/93D
FL 1993 spring	240 SC	1	0.56	0.75	120 150 180 210 240 270	0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 VBL-CO047-92/93D
FL 1993 spring	240 SC	1	0.56	0.6	120 150 180 210 240 270	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 VBL-CO048-92/93D

Location, Year, Season	Application				PHI (days)	Total residue (mg/kg)	Report no./Study no.
	Form.	No.	kg ai/ha	kg ai/hl			
CA 1994	240 SC	1	0.56	0.42	0	<0.05	106662-1 458-AD022-94/95D
					7	<0.05	
					15	<0.05	
					30	<0.05	
					60	<0.05	
					91	<0.05	
					120	<0.05	
					150	<0.05	
					181	<0.05	
					210	<0.05	
					240	<0.05	
					274	<0.05	
					366	<0.05	
TX 1994	240 SC	1	0.56	0.61	0	<0.05	106662-1 459-AD023- 94/95D-B A, B: Reverse decline study
					7	<0.05	
					15	<0.05	
					30	<0.05	
					62	<0.05	
					90	<0.05	
					120	<0.05	
	240 SC	1	0.56	0.68	153	<0.05	106662-1 459-AD023- 94/95D-A
					183	<0.05	
					215	<0.05	
					243	<0.05	
					273	<0.05	
CA 1994	240 SC	1	0.56	0.62	0	<0.05	106662-1 FCA-AD024-94/95D-A A, B, C: Reverse decline study
					7	<0.05	
					15	<0.05	
					30	<0.05	
					60	<0.05	
					91	<0.05	
					120	<0.05	
	240 SC	1	0.56	0.64	150	<0.05	106662-1 FCA-AD024-94/95D-B
					180	<0.05	
					210	<0.05	
					241	<0.05	
					270	<0.05	
	240 SC	1	0.56	0.62	365	<0.05	106662-1 FCA-AD024-94/95D-C

Table 77. Residues in lemons after soil application, California, USA, 1994.

Application				PHI (days)	Total residue (mg/kg)	Report No. Study no.
Form.	No.	kg ai/ha	kg ai/hl			
240 SC	1	0.56	0.42	0 7 15 30 56 91 120 150 181 210 240 274 366	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662-1 458-AD017-94/95D
240 SC	1	0.56	0.62	0 4 15 30 60 91 120	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662-1 FCA-AD018-94/95D-A A, B, C: Reverse decline study
240 SC	1	0.56	0.64	150 180 210 241 270	<0.05 <0.05 <0.05 <0.05 <0.05	106662-1 FCA-AD018-94/95D-B
240 SC	1	0.56	0.62	365	<0.05	106662-1 FCA-AD018-94/95D-C
240 SC	1	0.56	0.6	0 7 15 30 56 89 119 149 175 208 244 274 364	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662-1 457-AD019-94/95D

Table 78. Residues in oranges after soil application.

Country, Year, application	Application				Residues, mg/kg				Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	

Country, Year, application	Application				Residues, mg/kg				Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
USA, FL 1993 fall	240 SC	1	0.56	0.61	fruit	0 7 15 30 60 90 120 150		< <u>0.05</u> <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 353-CO037-92/93D
USA, FL 1993 fall	240 SC	1	0.56	0.71	fruit	0 7 15 30 60 90 121 150		<0.05 <0.05 <0.05 <0.05 <0.05 <u>0.08</u> <0.05 <0.05	106662 VBL-CO038- 92/93D
USA, FL 1993 fall	240 SC	1	0.56	0.71	fruit	0 7 15 30 60 90 121 150		< <u>0.05</u> <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662 VBL-CO039- 92/93D
USA, FL 1993 spring	240 SC	1	0.56	0.6	fruit	120 150 180 210 240 270		< <u>0.05</u> <0.05 <0.05 <0.05 <0.05 <0.05	106662 353-CO043-92/93D
USA, FL 1993 spring	240 SC	1	0.56	0.75	fruit	120 150 180 210 240 270		<u>0.06</u> 0.06 <0.05 <0.05 <0.05 <0.05	106662 VBL-CO044- 92/93D
USA, FL 1993 spring	240 SC	1	0.56	0.75	fruit	120 150 180 210 240 270		0.10 <u>0.12</u> 0.08 <0.05 0.08 0.09	106662 VBL-CO045- 92/93D

Country, Year, application	Application				Residues, mg/kg				Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
USA, 1994	240 SC	1	0.56	0.41	fruit	0 7 15 30 60 91 120 150 181 210 240 274 366		<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662-1 458-AD020-94/95D
USA, CA 1995	240 SC	1	0.56	0.63	fruit	0 7 15 30 60 90 120		<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	106662-1 FCA-AD021-94/95D-A A, B, C: Reverse decline study
	240 SC	1	0.56	0.71	fruit	150 180 210 241 270		<0.05 <0.05 <0.05 <0.05 <0.05	106662-1 FCA-AD021-94/95D-B
	240 SC	1	0.56	0.62	fruit	365		<0.05	106662-1 FCA-AD021-94/95D-C
South Africa 1989	200 SC	1	2.4 ¹	0.06	peel	179 212	0.33 0.27		311/88879/G312 88879-G312-A
					pulp	179 212	0.15 0.14		
					whole fruit	179 212	0.2 0.18		
South Africa 1989	200 SC	1		0.04	peel	179 212	0.11 0.09		311/88879/G312 88879-G312-B
					pulp	179 212	0.05 0.05		
					whole fruit	179 212	0.06 0.06		
South Africa 1989	200 SC	1	0.8 ³	0.02	peel	179 212	0.06 0.02		311/88879/G312 88879-G312-C
					pulp	179 212	0.02 <0.02		

Country, Year, application	Application				Residues, mg/kg				Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total residue	
					whole fruit	179 212	0.03 <0.02		
South Africa 1990	200 SL	1	0.57 ³	0.07	peel	294	0.02	0.08	311/88048/H113
					pulp	294	0.01	<0.05	311-88048-H113-A
					whole fruit	294	0.01	0.02	
South Africa 1990	200 SL	1	1.1 ²	0.13	fruit	94		0.13	311/88048/H113
					peel	154 210 266 294	0.03	0.15 0.14 0.14 0.15	311-88048-H113-B
					pulp	154 210 266 294	0.02	0.11 0.1 0.11 0.07	
					whole fruit	154 210 266 294	0.02	0.12 0.11 0.12 0.08	
South Africa 1990	200 SL	1	2.3 ⁴	0.27	fruit	94		0.44	311/88048/H113
					peel	154 210 266 294	0.09	0.91 0.42 0.33 0.8	311-88048-H113-C
					pulp	154 210 266 294	0.04	0.33 0.27 0.19 0.28	
					whole fruit	154 210 266 294	0.05	0.55 0.33 0.23 0.41	

¹ 6 g ai/tree² 4 g ai/tree³ 2 g ai/tree⁴ 8 g ai/tree

Apples (foliar spray treatment, Table 78). In eleven trials in Germany (70 WG and 25 WP formulations) one or two high- or low-volume sprays were applied (interval 14–21 days). At a rate of 1500 l/ha the concentration was 0.007-0.13 kg ai/hl, corresponding to 0.11 to 0.19 kg ai/ha. At a water rate of 200 or 250 l/ha, the concentration ranged between 0.052 and 0.063 kg ai/hl, corresponding to 0.11 to 0.16 kg ai/ha.

In Southern Europe, 13 trials were conducted in Italy, Spain and France according to GAP with the 200 SL or 200 SC formulation. In all except one trial one or two spray applications were made at a concentration ranging from 0.008 to 0.01 kg ai/hl, corresponding to 0.08 to 0.15 kg ai/ha. In the remaining trial a pre-blossom application at 0.02 kg ai/hl and 0.3 kg ai/ha was followed by a spray at 0.01 kg ai/hl (0.15 kg/ha).

In Italy 4 further trials were conducted with a 004 SC formulation containing a mineral oil. In 3 of the 4 a pre-blossom spray was applied at 0.01 kg ai/hl, corresponding to 0.15 kg ai/ha. In the fourth the concentration was 0.008 kg ai/hl, corresponding to 0.12 kg ai/ha. Samples were harvested 147 to 205 days after the last treatment.

In the USA and Canada in 14 trials five applications of a 240 SC formulation spray were made. In nine of the trials a low-volume spray was used and in the remaining 5 a high-volume spray at intervals of 12-26, 63-120, 8-11, and 10-14 days. At water rates ranging from 1870 to 3741 l/ha, the concentration was 0.005 to 0.01 kg ai/hl corresponding to 0.12-0.19 kg ai/ha. At water rates between 234 and 500 l/ha, the concentration ranged between 0.015 and 0.082 kg ai/hl, except in 2 trials with low application rates. The amount of imidacloprid applied per hectare ranged between 0.07 and 0.19 kg.

In South Korea 5 trials were conducted with a 10 WP formulation spray, concentration from 0.005 kg ai/hl, with two to 6 treatments at 0.25 kg ai/ha.

In South Africa in 6 trials with a 350 SC formulation the foliage of the trees was sprayed once at either 0.021 or 0.042 kg ai/hl. The application rate was about 0.53-0.59 kg ai/ha or 1.1-1.2 kg ai/ha.

Table 78. Residues in apples after foliar spray treatment.

Country Year, Location	Application				PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Germany 1990 Freinsheim	25 WP SRU	2	0.13	0.063	0 5 7 14 21	0.08 0.1	0.14 0.11 0.11 <u>0.11</u> 0.1	PF-3741 0186-90
Germany 1990 Burscheid	25 WP SPI	2	0.19	0.013	0 5 7 14 21	0.11 0.09	0.31 0.24 0.28 0.2 0.16	PF-3741 0187-90
Germany 1990 Burscheid	25 WP SPI	2	0.19	0.013	0 5 7 13 21	0.07 0.07	0.42 0.14 0.13 0.11 0.11	PF-3741 0188-90
Germany 1990 Heidesheim	25 WP SRU	2	0.16	0.063	0 5 7 14 21	0.04 0.03	0.12 0.06 0.06 0.06 0.07	PF-3741 0189-90
Germany 1990 Freinsheim	70 WG SRU	2	0.11	0.052	0 5 7 14 21	0.07 0.04	0.06 0.06 0.06 <0.05 <u>0.07</u>	PF-3741 0520-90

Country Year, Location	Application				PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Germany 1990 Heidesheim	70 WG SRU	2	0.13	0.052	0 5 7 14 21	0.02 0.02	0.06 <0.05 n.a. < <u>0.05</u> <0.05	PF-3741 0521-90
Germany 1990 Burscheid	70 WG SPI	2	0.16	0.011	0 5 7 14 21	0.13 0.09	0.34 0.26 0.22 0.18 0.14	PF-3741 0522-90
Germany 1990 Freinsheim	70 WG SPI	1	0.11	0.007	0 5 7 14 21	0.04	0.1 0.08 0.08 <u>0.08</u> 0.07	RA-2066/92 0101-92
Germany 1990 Burscheid	70 WG SPI	1	0.11	0.007	0 5 7 14 21	0.03	0.19 0.09 0.06 <u>0.06</u> <0.05	RA-2066/92 0102-92
Germany 1990 Heidesheim	70 WG SPI	1	0.11	0.007	0 5 7 14 21	0.02	0.05 <0.05 0.06 < <u>0.05</u> <0.05	RA-2066/92 0103-92
Germany 1990 Monheim	70 WG SPI	1	0.11	0.007	0 5 7 14 21	0.02	0.3 0.07 0.06 < <u>0.05</u> <0.05	RA-2066/92 0104-92
France, South 1996 Les Roche	200 SC SPI	2	0.15	0.01	0 14	0.03	0.2 <u>0.08</u>	RA-2041/96 0490-96
France, South 1996 Cloue	200 SC SPI	2	0.15	0.01	0 14	0.02	0.15 <u>0.06</u>	RA-2041/96 0491-96
France, South 1992 Pratmirail	200 SL SPI	2	0.08	0.01	0 ¹ 0 7 14 28	0.03	0.1 0.26 0.23 <u>0.18</u> 0.18	RA-2040/92 0455-92
France, South 1992 Montfavet	SPI	2	0.15	0.01	0 ¹ 0 7 14 28	0.08	<0.05 0.24 0.23 <u>0.2</u> 0.07	RA-2040/92 0456-92
Italy 1998 Ravenna	004 SC	1	0.15 ²	0.01	180	<0.01	<0.05	RA-2027/98 1229-98

Country Year, Location	Application				PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Italy 1998 Montemarzino	004SC	1	0.15 ²	0.01	205	<0.01	<0.05	RA-2027/98 1349-98
Italy 1998 Ravenna	004 SC	1	0.12 ²	0.008	180	<0.01	<0.05	RA-2037/99 0298-99
Italy 1998 Ravenna	004 SC	1	0.15 ²	0.01	172	<0.01	<0.05	RA-2037/99 0655-99
Italy 1992 Ravenna	200 SL SPI	2	0.15	0.01	0 ¹ 0 7 14 28	0.12	0.06 0.32 0.21 <u>0.23</u> 0.19	RA-2049/92 0277-92
Italy 1993 Ravenna	200 SL SPI	2	0.15	0.01	0 ¹ 0 3 7 10 14	0.02 0.05	0.05 0.17 0.17 0.2 0.18 <u>0.17</u>	RA-2019/93 0015-93
Italy 1993 Fondi		2	0.15	0.01	0 ¹ 0 3 7 10 14	0.03 0.03	0.07 0.13 0.13 0.06 0.06 <u>0.06</u>	RA-2019/93 0375-93
Italy 1994 Ravenna	200 SL SPI	2	0.3 ² 0.15	0.02 0.01	-1 ¹ 0 5 7 14 21	<0.01 0.02	<0.05 0.09 0.05 0.05 <u><0.05</u> <u><0.05</u>	RA-2059/94 0241-94
Italy 1996 Pineta di Laives	200 SC SPI	2	0.15	0.01	0 14	0.02	0.14 <u>0.07</u>	RA-2041/96 0330-96
Spain 1990 San Sadurni	200 SL SPI	1	0.08	0.008	0 19	0.01	0.05 <0.05	0220-90
Spain 1992 Castellidans	200 SL SPI	1	0.1	0.01	0 14 21	0.05	0.1 <u>0.13</u> 0.07	RA-2057/92 0123-92
Spain 1992 La Fortesa	200 SL SPI	1	0.1	0.01	0 7 14 21	0.04	0.33 0.23 <u>0.17</u> 0.13	RA-2057/92 0124-92
Spain 1992 Viladamat	200 SL SPI	1	0.1	0.01	0 14 21	0.03	0.11 <u>0.06</u> 0.06	RA-2057/92 0125-92

Country Year, Location	Application				PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Spain 1996 Prescador	200 SC SPI	2	0.15	0.01	0 14	0.04	0.15 <u>0.08</u>	RA-2041/96 0492-96
USA, WA 1991	240 SC SPI	5	0.19	0.005	6 13 20 29		0.17 0.13 0.11 0.09	103234 454-N3001-91D
USA, CA 1991	240 SC SRU	5	0.19	0.041	7 14 21 30		0.14 0.12 0.09 0.09	103234 455-N3002-91D
USA, PA 1991	240 SC SRU	5	0.19	0.082	7 14 21 30		0.10 0.05 <0.05 <0.05	103234 757-N3003-91D
USA, NY 1991	240 SC SPI	5	0.19	0.01	7 14 21 30		0.18 0.14 0.09 0.14	103234 758-N3004-91D
USA, MI 1991	240 SC SRU	5	0.14	0.03	7 14 21 30		<0.05 <0.05 <0.05 <0.05	103234 855-N3005-91D
USA, IN 1991	240 SC SRU	5	0.07	0.015	7 14 21 30		<0.05 <0.05 <0.05 <0.05	103234 HIN-N3006-91D
USA, WS 1991	240 SC SRU	5	0.023	0.005	7 14 21 30		<0.05 <0.05 <0.05 <0.05	103234 454-N3083-91D
USA, CA 1991	240 SC SPI	5	0.12	0.005	7 14 21 30		0.13 0.10 0.07 0.05	103234 FCA-N3084-91D
USA, VA 1991	240 SC SPI	5	0.19	0.005	7 14 21 30		0.1 0.09 0.08 <0.05	103234 757-N3085-91D
USA, WA 1992	240 SC SRU	5	0.019 0.19	0.005 0.049	7 14 21 30		0.74 0.46 0.23 0.24	103234-1 454-N3199-92D
USA, IN 1992	240 SC SRU	5	0.11	0.024	7 14 21 30		0.05 <0.05 <0.05 <0.05	103234-1 HIN-N3200-92D

Country Year, Location	Application				PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
USA, CA 1992	240 SC SPI	5	0.15	0.005	7 14 21 30		0.08 0.07 <0.05 <0.05	103234-1 FCA-N3201-92D
Canada, Oranorio 1993	240 SC SRU	5	0.11	0.02	7 14 21 30		0.12 0.13 0.14 0.12	106985 510-CO049-93D
Canada, Aylesfood 1993	240 SC SRU	5	0.11	0.02	7 14 21 28		0.22 0.25 0.14 0.19	106985 533-CO050-93D
South Korea 1993	10 WP SPI	2	0.25	0.005	45	0.02		R2301-93
South Korea 1993	10 WP SPI	3	0.25	0.005	7 14 21 30	0.09 0.08 0.05 0.06		R2305-93 R2304-93 R2303-93 R2302-93
South Korea 1993	10 WP SPI	4	0.25	0.005	7 14 21	0.11 0.08 0.1		R2308-93 R2307-93 R2306-93
South Korea 1993	10 WP SPI	5	0.25	0.005	7 14	0.2 0.08		R2310-93 R2309-93
South Korea 1993	10 WP SPI	6	0.25	0.005	7	0.21		R2311-93
South Africa 1995 Vyeboom	350 SC SPI	1	0.53	0.021	0 16 33 49 65		1.6 0.3 0.26 0.13 <u>0.12</u>	311-88061-N66 88061-N66-A Variety Golden Delicious
South Africa 1995 Vyeboom	350 SC SPI	1	1.1	0.042	0 65		1.5 0.13	311-88061-N66 88061-N66-B Variety Golden Delicious
South Africa 1995 Vyeboom	350 SC SPI	1	0.53	0.021	0 26 51 77 103		1.3 0.18 0.1 <u>0.07</u> 0.04	311-88063-N68 88063-N68-A Variety Granny Smith
South Africa 1995 Vyeboom	350 SC SPI	1	1.1	0.042	0 26 51 77 103		1.6 0.16 0.13 0.12 <0.02	311-88063-N68 88063-N68-B Variety Granny Smith
South Africa 1995	350 SC SPI	1	0.59	0.021	0 15		0.6 0.18	311-88065-N70 88065-N70-A

Country Year, Location	Application				PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Villiersdorp					30 47 79		0.13 0.13 <u>0.08</u>	
South Africa 1995 Villiersdorp	350 SC SPI	1	1.2	0.042	0 15 30 47 79		2.0 0.42 0.24 0.21 0.12	311-88065-N70 88065-N70-B

SPI: spray application

SRU: low volume spray application

¹ Before last application

² Pre-blossom application

Apple (soil drench application, Table 79). In 6 trials in South Africa the concentrations were 0.05 and 0.1 kg ai/hl corresponding respectively to 1 and 2 g ai/tree and in 19 trials in Australia application rates were 0.3-4.8 g ai/tree.

Table 79. Residues in apples after soil drench applications.

Country Year	Application				PHI (days)	Total residue, mg/kg	Report No. Study no.
	Form.	No.	g ai/tree	kg ai/hl			
South Africa 1995	350 SC	1	1	0.05	69 154	< <u>0.01</u> <0.01	311-88062-N67 88062-N67-A
		1	2	0.1	69 154	<0.01 <0.01	311-88062-N67 88062-N67-B
South Africa 1995	350 SC	1	1	0.05	69 86 104 121 140	< <u>0.03</u> <0.03 <0.03 <0.03 <0.03	311-88064-N69 88064-N69-A
		1	2	0.1	69 86 140	<0.03 <0.03 <0.03	311-88064-N69 88064-N69-B
South Africa 1995	350 SC	1	1	0.05	80 154	< <u>0.02</u> <0.02	311-88066-N71 88066-N71-A
		1	2	0.1	80 154	0.06 <0.02	311-88066-N71 88066-N71-B
Australia 1996	350 SC	1	0.3	0.03	97	0.02	RTL 397/96 AUS-RTL397-96-A
		1	0.6	0.06	97	<0.01	RTL 397/96 AUS-RTL397-96-B
		1	1.2	0.12	97	0.01	RTL 397/96 AUS-RTL397-96-C

Country Year	Application				PHI (days)	Total residue, mg/kg	Report No. Study no.
	Form.	No.	g ai/tree	kg ai/hl			
		1	2.4	0.24	97	<u>0.03</u>	RTL 397/96 AUS-RTL397-96-D
		1	4.8	0.48	97	0.06	RTL 397/96 AUS-RTL397-96-E
Australia 1996	350 SC	1	0.6	0.06	91	0.05	EMH 337/96 AUS-EMH337-96-A
		1	1.2	0.12	91	0.1	EMH 337/96 AUS-EMH337-96-B
		1	2.4	0.24	91	<u>0.16</u>	EMH 337/96 AUS-EMH337-96-C
		1	4.8	0.48	91	0.09	EMH 337/96 AUS-EMH337-96-D
		1	2.4	0.24	463	0.11	EMH 337/96 AUS-EMH337-96-E
Australia 1996	350 SC	1	0.3	0.03	97	<0.01	TAB 176/96 AUS-TAB176-96-A
		1	0.6	0.06	97	<0.01	TAB 176/96 AUS-TAB176-96-B
		1	1.2	0.12	97	<0.01	TAB 176/96 AUS-TAB176-96-C
		1	2.4	0.24	97	<u>0.02</u>	TAB 176/96 AUS-TAB176-96-D
		1	4.8	0.48	97	0.02	TAB 176/96 AUS-TAB176-96-E
Australia 1999	350 SC	1	2.4	0.24	482	<0.02 <0.02	EMH-419/99 AUS-EMH419-99-A
		2	2.4	0.24	114	< <u>0.05</u> <0.05	EMH-419/99 AUS-EMH419-99-B
Australia 1999	350 SC	1	2.4	0.24	474	0.06 <0.05	RTL-506/99 AUS-RTL506-99-A
		2	2.4	0.24	110	0.08 <u>0.14</u>	RTL-506/99 AUS-RTL506-99-B

Pears (Table 80). Two trials with single foliar sprays of a 70 WG formulation were conducted in France (1 North, 1 South) at a concentration of 0.021 kg ai/hl, corresponding to 0.21 kg ai/ha.

Eight trials were conducted using foliar sprays in Southern Europe: one in Greece, four in Italy and three in Spain, with a 200 SL formulation. One application was used in the trials in Greece and Spain and two in Italy at intervals of 21 to 139 days. In two of the trials in Italy, the first spray

was pre-blossom at 0.02 kg ai/hl, 0.3 kg ai/ha. The spray concentrations in the other trials were 0.01-0.012 kg ai/hl, corresponding to 0.1-0.18 kg ai/ha.

In Italy three pre-blossom trials were conducted with the 004 SC formulation containing a mineral oil. In two the rate was 0.01 kg ai/hl, corresponding to 0.15 kg ai/ha, and in the third 0.008 kg ai/hl, corresponding to 0.12 kg ai/ha. Samples were harvested 132 to 147 days after the last treatment. The total residues were below the limit of determination of 0.05 mg/kg.

In the USA and Canada trials were carried out using two foliar spray methods, with two treatments in each trial. In five trials a concentrated spray (0.06 to 0.063 kg ai/hl), and in four a diluted spray (0.01 to 0.015 kg ai/hl) was used corresponding to 0.28-0.31 kg ai/ha.

Table 80. Residues in pears after spray applications.

Country Year	Application				PHI (days)	Residues, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
France, North 1990	70 WG	1	0.21	0.021	0 15 29	0.05	0.25 0.12 0.1	0565-90
France, South 1990	70 WG	1	0.21	0.021	0 15 30	<0.01	0.19 <0.05 <0.05	0689-90
Greece 1999	200 SL	1	0.18	0.012	0 3 7 10 14	0.15 0.04 0.04 0.03 0.01	0.15 0.1 0.09 0.09 <u>0.05</u>	RA-2066/99 0428-99
Italy 1992	200 SL	2	0.17	0.01	0 ¹ 0 7 14 21	0.14	0.1 0.39 0.35 <u>0.26</u> 0.2	RA-2049/92 0262-92
Italy 1992	200 SL	2	0.17	0.01	0 14 28	0.06	0.22 <u>0.1</u> 0.1	RA-2049/92 0263-92
Italy 1994	200 SL	2	0.3 ² 0.15	0.02 0.01	0 ¹ 0 5 7 14 21	<0.01 0.02	<0.05 0.14 0.1 <0.05 <u><0.05</u> <0.05	RA-2059/94 0242-94
Italy 1994	200 SL	2	0.3 ² 0.15	0.02 0.01	0 ¹ 0 5 7 14	<0.01 0.03	<0.05 0.12 0.1 0.07 <u>0.06</u>	RA-2059/94 0243-94
Italy 1998	004 SC	1	0.15 ²	0.01	147	<0.01	<0.05	RA-2027/98 1230-98
Italy 1998	004 SC	1	0.116 ²	0.008	140	<0.01	<0.05	RA-2037/99 0299-99

Country Year	Application				PHI (days)	Residues, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Italy 1999	004 SC	1	0.15 ²	0.01	132	<0.01	<0.05	RA-2037/99 0656-99
Spain 1992	200 SL	1	0.1	0.01	0 7 14 20	0.05	0.13 0.11 <u>0.08</u> <0.05	RA-2057/92 0127-92
Spain 1992	200 SL	1	0.1	0.01	0 14 21	0.04	0.12 <u>0.07</u> 0.07	RA-2057/92 0128-92
Spain 1992	200 SL	1	0.1	0.01	0 14 21	0.07	0.29 <u>0.23</u> 0.09	RA-2057/92 0129-92
USA, OR 1993	240 SC	2	0.28	0.06 ⁴	0 7 14 21		0.48 0.29 0.26 <u>0.33</u>	106789 451-CO015-93D
USA, WA 1993	240 SC	2	0.28 ³	0.06 ⁴	0 7 14 21		0.49 0.38 <u>0.53</u> 0.18	106789 454-CO016-93D
USA, CA 1993	240 SC	2	0.28 ³	0.015	0 7 14 21		0.53 0.36 <u>0.4</u> 0.22	106789 454-CO017-93D
USA, CA 1993	240 SC	2	0.28 ³	0.01	0 7 14 21		0.32 <u>0.33</u> 0.28 0.22	106789 455-CO018-93D
USA, CA 1993	240 SC	2	0.28 ³	0.06 ⁴	0 7 14 21		0.66 0.22 0.22 <u>0.25</u>	106789 455-CO019-93D
USA, NY 1993	240 SC	2	0.28 ³	0.015	0 7 14 21		0.42 <u>0.5</u> 0.34 0.32	106789 758-CO020-93D
USA, MI 1993	240 SC	2	0.28 ³	0.06 ⁴	0 7 14 21		0.44 <u>0.27</u> 0.21 0.16	106789 855-CO021-93D
Canada, Ontario 1995	240 SC	2	0.28	0.015	0 7 14 21		0.88 <u>0.71</u> 0.42 0.32	107717 510-N3002- 95D-A

Country Year	Application				PHI (days)	Residues, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Canada, Ontario 1995	240 SC	2	0.31	0.062 0.063 ⁴	0 7 14 21		0.67 <u>0.38</u> 0.25 0.16	107717 510-N3002- 5D-B

¹ Day before last application

² Pre-blossom application

³ Foliar spray in combination with spray oil

⁴ Concentrate spray (all unmarked entries are dilute sprays)

Apricots (Table 81). In a single residue trial in France a single spray application was made at 0.0035 kg ai/hl, corresponding to 0.035 kg ai/ha.

Nectarines (Table 81). In three trials in Italy with the 200 SL formulation two spray applications (interval 30 or 142 days) were made at 0.01 kg ai/hl (except in one trial in which the first treatment was pre-blossom at 0.02 kg ai/hl, 0.24 kg ai/ha). Application rates ranged from 0.12 to 0.15 kg ai/ha.

Peaches (Table 81). 22 trials were conducted in Southern Europe: one in Greece, 5 in Italy, 7 in Spain and 9 in France. In six of the trials in France a 70 WG formulation was used conforming with French GAP, at 0.007 kg ai/hl, 0.07 kg ai/ha. In two more of the trials in France a 200 SL formulation at a spray concentration of 0.0035 kg ai/hl, corresponding to 0.03 to 0.035 kg ai/ha, was used. In the remaining 14 trials two spray applications of a 200 SC or a 200 SL formulation (interval 19–30 days) were used at 0.01 kg ai/hl (0.008 kg ai/hl in 2 trials). The application rates ranged between 0.08 and 0.15 kg ai/ha.

In four trials in Australia with a 200 SL formulation 3 applications (intervals 16, 22 -70 days) were made. In two trials the lowest label concentration of 0.005 kg ai/hl was used and in the other two the highest label concentration of 0.01 kg ai/hl, corresponding to 0.075 kg ai/ha and 0.15 kg ai/ha respectively. Only the parent compound was determined.

Table 81. Residues in apricots, nectarines and peaches after foliar spray treatment.

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report Study	No. no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total		
Apricot										
France, North 1991	200 SL	1	0.035	0.0035	fruit whole fruit calculated	0 15 21 0 15 21	0.03 0.03	0.09 <0.05 <0.05 0.08 <0.05 <0.05	0442-91	
Nectarine										
Italy 1991	200 SL	2	0.15	0.01	fruit fruit without stone	0 14 21	0.09	0.22 <u>0.13</u> 0.11	RA-2029/91 0278-91	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report Study	No. no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total		
					whole fruit calculated	14 21	0.08	<u>0.12</u> 0.1		
Italy 1992	200 SL	2	0.15	0.01	fruit	0 ¹ 0	0.06	0.06 0.25	RA-2054/92 0279-92	
					fruit without stone	7 14 21		0.09 <u>0.13</u> 0.1		
					whole fruit calculated	7 14 21		0.06		
Italy 1994	200 SL	1 1	0.24 ² 0.12	0.02 0.01	fruit	0 ¹ 0 5 7	<0.01	<0.05 <0.05 <0.05	RA-2044/94 0234-94	
					fruit without stone	14 21		<0.05 <0.05		
					whole fruit calculated	14 21	<0.01	<0.05 <0.05		
Peach										
France, South 1990	70 WG	1	0.07	0.007	fruit fruit without stone	0 7 15	0.02	0.15 <0.05 <u>0.07</u>	0566-90	
					whole fruit calculated	7 15		<0.05 <u>0.06</u>		
France, South 1990	70 WG	1	0.07	0.007	fruit	0 14 28	<0.01 <0.01	0.12 <u>0.06</u> <0.05	0567-90	
					whole fruit calculated	28		<0.05		
France, South 1990	70 WG	1	0.07	0.007	fruit	0 14 42	<0.01 <0.01	0.06 <u>0.06</u> <0.05	0568-90	
					whole fruit calculated	42		<0.05		
France, South 1990	70 WG	1	0.07	0.007	fruit without stone	0 7 15	0.02	0.12 0.07 <u><0.05</u>	0690-90	
					whole fruit calculated	0 7 15		0.11 0.07 <u><0.05</u>		
France, South 1990	70 WG	1	0.07	0.007	fruit without stone	0 15 30	0.01	0.17 <u>0.11</u> <0.05	0691-90	
					whole fruit calculated	0 15 30		0.15 <u>0.1</u> <0.05		

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report Study	No. no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total		
France, South 1990	70 WG	1	0.07	0.007	fruit without stone whole fruit calculated	0 15 45 0 15 45	0.02 0.02	0.15 <u>0.07</u> <0.05 0.12 <u>0.06</u> <0.05	0692-90	
France, South 1991	200 SL	1	0.03	0.0035	fruit without stone whole fruit calculated	0 14 21 0 14 21	0.03 0.03	0.16 0.07 <0.05 0.14 0.07 <0.05	0439-91	
France, South 1991	200 SL	1	0.035	0.0035	fruit without stone whole fruit calculated	0 15 21 0 15 21	<0.01 <0.01	0.28 <0.05 <0.05 0.24 <0.05 <0.05	0441-91	
Greece 1991	200 SL	2	0.15	0.01	fruit without stone whole fruit calculated	0 14 21 0 14 21	0.12 0.09	0.33 <u>0.26</u> 0.19 0.22 <u>0.19</u> 0.15	0126-91	
Italy 1992	200 SL	1 1	0.12 0.15	0.01 0.01	fruit fruit without stone whole fruit calculated	0 ¹ 0 7 14 21 7 14 21	0.04 0.04	0.05 0.08 0.09 <u>0.07</u> 0.06 0.08 <u>0.06</u> 0.06	RA-2054/92 0264-92	
Italy 1992	200 SL	2	0.15	0.01	Fruit fruit without stone whole fruit calculated	0 14 21 14 21	0.03 0.03	0.18 <u>0.07</u> 0.06 <u>0.06</u> 0.06	RA-2054/92 0265-92	
Italy 1993	200 SL	2	0.15	0.01	fruit fruit without stone whole fruit calculated	0 ¹ 0 5 7 14 21 14 21	<0.01 0.06 0.06	0.21 0.56 0.24 0.15 <u>0.16</u> 0.15 <u>0.15</u> 0.14	RA-2027/93 0018-93	
Italy 1996	200 SC	2	0.15	0.01	fruit	0 14	0.01	0.11 <u><0.05</u>	RA-2043/96 0497-96	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report Study	No. no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total		
Italy 1996	200 SC	2	0.15	0.01	fruit	0 14	0.06	0.15 <u>0.12</u>	RA-2043/96 0498-96	
France, South 1996	200 SC	2	0.15	0.01	fruit whole fruit calculated	0 14 14	0.07 0.06	0.14 <u>0.11</u> 0.1	RA-2043/96 0329-96	
Spain 1996	200 SC	2	0.15	0.01	fruit	0 14	0.07	0.21 <u>0.15</u>	RA-2043/96 0499-96	
Spain 1990	200 SL	2	0.08	0.008	fruit without stone whole fruit calculated	0 15 0 15	0.07 0.06	0.14 <u>0.13</u> 0.12 <u>0.11</u>	0222-90	
Spain 1990	200 SL	2	0.08	0.008	fruit without stone whole fruit calculated	0 15 0 15	0.02 0.02	0.31 <u><0.05</u> 0.28 <u><0.05</u>	0223-90	
Spain 1991	200 SL	2	0.1	0.01	fruit	0 14 24	0.07	0.61 <u>0.35</u> 0.19	RA-2035/91 0512-91	
Spain 1991	200 SL	2	0.1	0.01	fruit whole fruit calculated	0 14 21 21	0.05 0.05	0.38 <u>0.2</u> 0.13 0.12	RA-2035/91 0513-91	
Spain 1992	200 SL	2	0.1	0.01	fruit fruit without stone whole fruit calculated	0 ¹ 0 8 14 21 14 21	0.10 0.09	0.18 0.41 0.37 <u>0.22</u> 0.18 <u>0.2</u> 0.17	RA-2060/92 0121-92	
Spain 1992	200 SL	2	0.1	0.01	fruit fruit without stone whole fruit calculated	0 14 21 14 21	0.10 0.09	0.86 <u>0.32</u> 0.25 <u>0.29</u> 0.23	RA-2060/92 0122-92	
Australia 1991	200 SL	3	0.075	0.005	fruit	0 7 14 21	0.40 0.01 0.02 0.01		17/91 AUS-17-91-A	
Australia 1991	200 SL	3	0.15	0.01	fruit	0 7 14 21	0.80 0.05 0.13 0.04		17/91 AUS-17-91-B	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report Study	No. no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total		
Australia 1991	200 SL	3	0.075	0.005	fruit	0 7 14 21	0.38 0.12 0.05 0.04		B18/91 AUS-18-91-A	
Australia 1991	200 SL	3	0.15	0.01	fruit	0 7 14 21	0.86 0.39 0.22 0.09		B18/91 AUS-18-91-B	

¹ Before last treatment² Pre-blossom application

Cherries, sweet (Table 82). Of nine field trials on sweet cherries in Southern Europe with a 200 SL formulation, 6 were in Italy and 3 in Spain. In five of the trials in Italy there were two foliar spray applications (interval 30 days) at 0.01 kg ai/hl (about 0.1 kg ai/ha), except in one in which the first application was at 0.02 kg ai/hl (0.2 kg ai/ha, interval 67 days). In the 3 trials in Spain and the sixth in Italy only one spray was applied at 0.01 kg ai/hl, 0.1 kg ai/ha.

Two trials in Australia were with a 200 SL and a further 2 with a 350 SC spray formulation. Two applications were made at an interval of 35 days. For each formulation, one trial was at the label rate and the other at twice this rate, corresponding to 0.005 kg ai/hl (0.063 to 0.079 kg ai/ha) and 0.01 kg ai/hl (0.13 to 0.16 kg ai/ha) respectively.

Table 82. Residues in sweet cherries after spray applications.

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Italy 1991	200 SL	2	0.1	0.01	fruit fruit without stone	0 14 21	0.12	0.57 0.29 0.3	RA-2025/91 0273-91
					whole fruit calculated	14 21	0.11	0.26 <u>0.28</u>	
Italy 1992	200 SL	2	0.1	0.01	fruit without stone	0 14 21	0.09	0.38 0.19 0.17	RA-2054/92 0251-92
					whole fruit calculated	0 14 21	0.07	0.3 0.16 <u>0.14</u>	
Italy 1993	200 SL	2	0.1	0.01	fruit	0 ¹ 0 5 7 14 21	0.02 0.09	0.06 0.12 0.19 0.17 0.22 <u>0.15</u>	RA-2030/93 0010-93

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Italy 1993	200 SL	2	0.1	0.01	fruit	0 ¹ 0 5 7 14 21	0.01 0.05	<0.05 0.14 0.11 0.11 0.08 <u>0.11</u>	RA-2030/93 0390-93
Italy 1994	200 SL	1 1	0.2 0.1	0.02 0.01	fruit	0 ¹ 0 5 7 14 21	<0.01 0.1	<0.05 0.25 0.19 0.14 0.16 <u>0.15</u>	RA-2056/94 0214-94
Italy 1999	200 SL	1	0.1	0.01	fruit	21 28 35	0.02 0.03 0.02	0.06 <u>0.08</u> <0.05	RA-2047/99 0270-99
Spain 1998	200 SL	1	0.1	0.01	fruit	20 28	 0.07	<u>0.16</u> 0.14	RA-2165/98 1293-98
Spain 1998	200 SL	1	0.1	0.01	fruit	21 28 35	 0.02	<u>0.07</u> 0.05 0.06	RA-2165/98 1638-98
Spain 1999	200 SL	1	0.1	0.01	fruit	20 28 34	0.05 0.03 0.02	<u>0.12</u> 0.09 0.08	RA-2047/99 0269-99
Australia 1990	200 SL	2	0.063- 0.075	0.005	fruit	24	0.02		4/91 AUS-4-91-A1
Australia 1990	200 SL	2	0.13- 0.15	0.01	fruit	24	0.05		4/91 AUS-4-91-A2
Australia 1990	350 SC	2	0.066- 0.079	0.005	fruit	24	0.06		4/91 AUS-4-91-B1
Australia 1990	350 SC	2	0.13- 0.16	0.01	fruit	24	0.09		4/91 AUS-4-91-B2

¹ Before last treatment

Plums (Table 83). Six trials with foliar sprays in northern Europe were with single applications at 0.007 kg ai/hl, corresponding to 0.088 and 0.11 kg ai/ha, two in France, two in Germany and two in the UK. In four a 70 WG formulation and in the remaining two a 200 SL were used.

In 18 foliar spray trials in southern Europe, nine in France, seven in Italy and two in Spain, a 200 SL formulation was used. In eight of the trials in 1994/1995 in France single applications were made at the French GAP concentration of 0.007 kg ai/hl, corresponding to 0.036-0.11 kg ai/ha. All except one of the remaining ten trials were conducted according to Italian GAP with spray concentrations of 0.01 kg ai/hl, with two treatments at an interval of 30 days made at 0.1 or 0.15 kg ai/ha. In the exception the first application was at 0.02 kg ai/hl and 0.3 kg ai/ha, interval 144 days.

Table 83. Residues in plums after foliar spray application.

Country Year	Application				PHI (days)	Residues, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
France, North 1996	200 SL	1	0.11	0.007	0 56	<0.01	<0.05 <u><0.05</u>	RA-2045/96 0505-96
UK 1996	200 SL	1	0.11	0.007	0 ¹ 0 21 35 42 56	<0.01	<0.05 <0.05 <0.05 <0.05 <0.05 <u><0.05</u>	RA-2045/96 0504-96
France, North 1997	70 WG	1	0.11	0.007	0 3 7 14 21	0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <u><0.05</u>	RA-2092/97 0586-97
Germany 1997	70 WG	1	0.088	0.007	0 3 7 14 21	0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <u><0.05</u>	RA-2092/97 0273-97
Germany 1997	70 WG	1	0.088	0.007	0 3 7 14	<0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u>	RA-2092/97 0584-97
UK 1997	70 WG	1	0.11	0.007	0 3 7 14 21	<0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <u><0.05</u>	RA-2092/97 0585-97
France, South 1994	200 SL	1	0.068	0.007	0 21 35 42 56	<0.01	<0.05 <0.05 <0.05 <0.05 <u><0.05</u>	RA-2031/94 0127-94
France, South 1994	200 SL	1	0.065	0.007	0 21 35 42 56	<0.01	<0.05 <0.05 <0.05 <0.05 <u><0.05</u>	RA-2031/94 0128-94
France, South 1994	200 SL	1	0.086	0.007	0 56		<0.05 <u><0.05</u>	RA-2031/94 0129-94
France, South 1994	200 SL	1	0.036	0.007	0 56		<0.05 <u><0.05</u>	RA-2031/94 0130-94
France, South 1995	200 SL	1	0.11	0.007	0 57	<0.01	<0.05 <u><0.05</u>	RA-2119/95 0139-95

Country Year	Application				PHI (days)	Residues, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
France, South 1995	200 SL	1	0.11	0.007	0 56	0.01	0.07 <0.05	RA-2119/95 0140-95
France, South 1995	200 SL	1	0.11	0.007	0 57	<0.01	<0.05 <0.05	RA-2119/95 0641-95
France, South 1995	200 SL	1	0.11	0.007	0 56	<0.01	<0.05 <0.05	RA-2119/95 0642-95
Italy 1991	200 SL	2	0.15	0.01	0 14 21	0.02	0.19 0.05 0.05	RA-2036/91 0276-91
Italy 1992	200 SL	2	0.15	0.01	0 ¹ 0 7 14 21	0.01	<0.05 0.07 <0.05 <0.05 <0.05	RA-2054/92 0268-92
Italy 1992	200 SL	2	0.15	0.01	0 14 21	0.01	0.06 <0.05 <0.05	RA-2054/92 0270-92
Italy 1993	200 SL	2	0.15	0.01	0 ¹ 0 5 7 14 21	0.01 0.03 0.01	<0.05 0.06 <0.05 <0.05 <0.05 <0.05	RA-2030/93 0020-93
Italy 1993	200 SL	2	0.15	0.01	0 ¹ 0 5 7 14 21	0.02 0.05 0.03	0.06 0.11 0.14 0.16 0.13 0.09	RA-2030/93 0401-93
Italy 1994	200 SL	1 1	0.3 0.15	0.02 0.01	0 ¹ 0 7 14 21	<0.01 <0.01	<0.05 0.11 0.06 <0.05 <0.05	RA-2056/94 0237-94
Italy 1999	200 SL	2	0.1	0.01	14 21 28	<0.01 <0.01 <0.01	<0.05 <0.05 <0.05	RA-2046/99 0267-99
France, South 1999	200 SL	2	0.1	0.01	14 21 28	<0.01 <0.01 <0.01	<0.05 <0.05 <0.05	RA-2046/99 0268-99
Spain 1998	200 SL	2	0.1	0.01	14 21	0.03	0.12 0.12	RA-2164/98 1292-98
Spain 1998	200 SL	2	0.1	0.01	14 21 28	0.01	<0.05 <0.05 <0.05	RA-2164/98 1636-98

¹ Before last treatment

Grapes (Table 84). In trials in southern Europe (one in Italy, one in Spain and 7 in Portugal) in countries with comparable climates the foliage of grapes was sprayed once with the 200 SL formulation. In 5 of the trials the concentration was 0.01 kg ai/hl, 0.1 kg ai/ha, and in the other 4 a low-volume 0.02 kg ai/hl, 0.10 to 0.11 kg ai/ha.

In 16 trials according to GAP in the USA in 1991/92 two applications at intervals of 11-16 days were made, 11 with the 240 SC formulation and 6 the 75 WP, all approximately at the highest label rate (0.053 kg ai/ha). With a concentrated spray at 374-477 l/ha, the concentration ranged between 0.011 and 0.014 kg ai/hl, and with a dilute spray at 935-1189 l/ha between 0.0045 and 0.0057 kg ai/hl.

Table 84. Residues in grapes after foliar spray treatment.

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Portugal 1997	200 SL	1	0.11	0.02	bunch of grapes	0	0.04	0.08	<i>RA-4000/97</i> 0691-97
						7		0.06	
					berries	14	0.01	0.05	
						21	0.01	0.05	
Portugal 1997	200 SL	1	0.1	0.02	bunch of grapes	0	0.06	0.11	<i>RA-4000/97</i> 0692-97
						7		0.05	
					berries	14	0.01	0.06	
						21		0.06	
Portugal 1998	200 SL	1	0.1	0.02	bunch of grapes	0	0.10	0.11	<i>RA-4000/98</i> 1354-98
						7		0.07	
					berries	14	0.04	0.07	
						21		0.07	
Portugal 1998	200 SL	1	0.1	0.02	bunch of grapes	0	0.12	0.12	<i>RA-4000/98</i> 1355-98
						7		0.12	
					berries	14	0.04	0.06	
						21		0.06	
Portugal 1999	200 SL	1	0.1	0.01	bunch of grapes	0 ¹	<0.01	<0.05	<i>RA-2030/99</i> 0307-99
						0	0.29	0.28	
					berries	7		0.2	
						14	0.14	<u>0.2</u>	
Portugal 1999	200 SL	1	0.1	0.01	bunch of grapes	21		0.05	<i>RA-2030/99</i> 0308-99
						28	0.03	0.07	
					berries	14	0.03	0.07	
Portugal 1999	200 SL	1	0.1	0.01	bunch of grapes	0	0.11	0.12	<i>RA-2030/99</i> 0308-99
						14	0.02	< <u>0.05</u>	
					berries	14	0.01	<0.05	

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Portugal 1999	200 SL	1	0.1	0.01	bunch of grapes	0	0.25	0.22	RA-3030/99 0716-99
						14	0.07	0.09	
					berries	14	0.09	<u>0.12</u>	
						14	0.07	0.06	
Spain 1999	200 SL	1	0.1	0.01	bunch of grapes	0	0.02	<0.05	RA-2030/99 0310-99
						14	<0.01	<u><0.05</u>	
					berries	14	<0.01	<0.05	
						14	<0.01	<0.05	
Italy 1999	200 SL	1	0.1	0.01	bunch of grapes	0 ¹	<0.01	<0.05	RA-2030/99 0311-99
						0	0.03	<0.05	
						7		<0.05	
						14	<0.01	<u><0.05</u>	
						14	0.01	<0.05	
						21		<0.05	
						28	<0.01	<0.05	
					berries	14	0.01	<0.05	
USA, OR 1991	240 SC	2	0.053	0.011	bunch of grapes	0		<u>0.16</u>	103245 451-N3035-91D
						1		0.12	
						3		0.08	
						7		0.07	
						14		0.07	
						21		0.06	
USA, WA 1991	240 SC	2	0.053	0.011	bunch of grapes	0		<u>0.2</u>	103245 454-N3036-91D
						1		0.18	
						3		0.1	
						7		0.1	
						14		0.07	
						21		<0.05	
USA, CA 1991	240 SC	2	0.053	0.0057	bunch of grapes	0		<u>0.21</u>	103245 455-N3037-91D
						1		0.16	
						3		0.15	
						7		0.18	
						14		0.12	
						21		0.11	
USA, NC 1991	240 SC	2	0.053	0.0057	bunch of grapes	0		<u>0.61</u>	103245 751-N3086-91D
						1		0.54	
						3		0.51	
						7		0.51	
						14		0.36	
						21		0.21	
USA, NY 1991	240 SC	2	0.053	0.011	bunch of grapes	0		<u><0.05</u>	103245 758-N3039-91D
						1		<0.05	
						3		<0.05	
						7		<0.05	
						14		<0.05	
						21		<0.05	

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, CA 1991	240 SC	2	0.053	0.011	bunch of grapes	0 1 3 7 14 21		<0.05 <u>0.05</u> <0.05 <0.05 <0.05 <0.05	103245 FCA-N3041-91D
USA, IN 1991	240 SC	2	0.053	0.0057	bunch of grapes	0 1 3 7 14 21		<u>0.19</u> 0.17 0.14 0.12 0.11 0.11	103245 HIN-N3040-91D
USA, WA 1992	75 WP	2	0.053	0.005	bunch of grapes	0 1 3 7		0.08 <u>0.12</u> 0.09 0.05	103245-1 454-N3203-92D
USA, CA 1992	75 WP	2	0.053	0.014	bunch of grapes	0 1 3 7 14 21		0.11 <u>0.12</u> 0.11 0.11 0.11 0.11	103245-1 455-N3204-92D
USA, NY 1992	75 WP	2	0.053	0.013	bunch of grapes	0 1 3 7 14 21		<u>0.11</u> 0.08 0.06 0.06 0.05 <0.05	103245-1 758-N3206-92D
USA, CA 1992	75 WP	2	0.053	0.011	bunch of grapes	0 1 3 7 14 21		<u>0.06</u> <0.05 <0.05 <0.05 <0.05 <0.05	103245-1 FCA-N3207-92D
USA, IN 1992	240 SC	2	0.053	0.0045	bunch of grapes	0 1 3 7 14 21		<u>0.11</u> 0.1 0.09 0.08 0.05 <0.05	103245-1 HIN-N3211-92D
USA, IN 1992	75 WP	2	0.053	0.0045	bunch of grapes	0 1 3 7 14 21		<u>0.17</u> 0.12 0.12 0.09 0.07 0.06	103245-1 HIN-N3208-92D

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report No. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, CA 1992	240 SC	2	0.053	0.011	bunch of grapes	0 1 3 7 14 21		<0.05 <0.05 <u>0.06</u> 0.05 <0.05 <0.05	103245-1 FCA-N3210-92D
USA, MI 1992	75 WP	2	0.053	0.0057	bunch of grapes	0 1 3 7 14 21		<u>0.06</u> <0.05 <0.05 <0.05 <0.05 <0.05	103245-1 855-N3205-92D
USA, CA 1992	240 SC	2	0.053	0.014	bunch of grapes	0 1 3 7 14 21		0.1 0.08 <u>0.11</u> 0.07 0.07 0.09	103245-1 455-N3209-92D

¹ Before last treatment

Bananas (Table 85). In Martinique in four reversed decline trials 1.25 ml of undiluted 200 SL formulation was applied with a special dispensing syringe to the base of the mother plants, corresponding to 1.25 g ai/plant. Each application was to a separate plot with a separate study number 0, 3, 7, 14, 28 or 60 days before harvest. On days three and 28 additional bananas were picked and ripened for separation into peel and pulp. The total residue was below the LOQ of 0.05 mg/kg in all samples at all sampling intervals.

In twelve field trials according to GAP in Central and South America at eight sites in the Caribbean coastal area of Central America and in the Pacific coastal area of Ecuador single drench applications of a 350 SC formulation were made to the base of the trees at 0.35 to 0.37 kg ai/ha, 0.19-0.29 g ai/plant, diluted in 200-300 ml water/plant. The total residues were below the LOQ of 0.01 mg/kg in all samples.

Table 85. Residues in bananas.

Country, Year	Application				Sample	PHI (days)	Residue, mg/kg		Reference Report no. Study no.
	Form.	No.	g ai/plant	kg ai/hl			imida- cloprid	Total	
Reversed decline study (location 1)									
Martinique 1999	200 SL	1	0.25		fruit	0		< <u>0.05</u>	RA-2166/99 0685-99
		1	0.25		fruit	3	<0.01	<0.05	RA-2166/99 0684-99
					pulp	3	<0.01	<0.05	
					peel	3	<0.01	<0.05	

Country, Year	Application				Sample	PHI (days)	Residue, mg/kg		Reference Report no. Study no.
	Form.	No.	g ai/plant	kg ai/hl			imida- cloprid	Total	
					fruit, ripened	3	<0.01	<0.05	
		1	0.25		fruit	7		<0.05	RA-2166/99 0683-99
		1	0.25		fruit	14		<0.05	RA-2166/99 0681-99
		1	0.25		fruit	28	<0.01	<0.05	RA-2166/99 0680-99
					pulp	28	<0.01	<0.05	
					peel	28	<0.01	<0.05	
					fruit, ripened	28	<0.01	<0.05	
1	0.25		fruit	60		<0.05	RA-2166/99 0678-99		
Reversed decline study (location 2)									
Martinique 1999	200 SL	1	0.25		fruit	0		<0.05	RA-2166/99 0691-99
		1	0.25		fruit	3	<0.01	<0.05	RA-2166/99 0690-99
					pulp	3	<0.01	<0.05	
					peel	3	<0.01	<0.05	
					fruit, ripened	3	<0.01	<0.05	
		1	0.25		fruit	7		<0.05	RA-2166/99 0689-99
1	0.25		fruit	14		<0.05	RA-2166/99 0688-99		
		1	0.25		fruit	28	<0.01	<0.05	RA-2166/99 0687-99
					pulp	28	<0.01	<0.05	
					peel	28	<0.01	<0.05	
					fruit, ripened	28	<0.01	<0.05	
		1	0.25		fruit	60		<0.05	RA-2166/99 0686-99
Reversed decline study (location 3)									
Martinique 1999	200 SL	1	0.25		fruit	0		<0.05	RA-2166/99 0698-99
		1	0.25		fruit	3	<0.01	<0.05	RA-2166/99 0697-99

Country, Year	Application				Sample	PHI (days)	Residue, mg/kg		Reference Report no. Study no.
	Form.	No.	g ai/plant	kg ai/hl			imida- cloprid	Total	
					pulp	3	<0.01	<0.05	
					peel	3	<0.01	<0.05	
					fruit, ripened	3	<0.01	<0.05	
		1	0.25		fruit	7		<0.05	RA-2166/99 0695-99
		1	0.25		fruit	14		<0.05	RA-2166/99 0694-99
		1	0.25		fruit	28	<0.01	<0.05	RA-2166/99 0693-99
					pulp	28	<0.01	<0.05	
					peel	28	<0.01	<0.05	
					fruit, ripened	28	<0.01	<0.05	
		1	0.25		fruit	60		<0.05	RA-2166/99 0692-99
<i>Reversed decline study (location 4)</i>									
Martinique 1999	200 SL	1	0.25		fruit	0		<0.05	RA-2166/99 0705-99
		1	0.25		fruit	3	<0.01	<0.05	RA-2166/99 0704-99
					pulp	3	<0.01	<0.05	
					peel	3	<0.01	<0.05	
					fruit, ripened	3	<0.01	<0.05	
		1	0.25		fruit	7		<0.05	RA-2166/99 0703-99
		1	0.25		fruit	14		<0.05	RA-2166/99 0702-99
		1	0.25		fruit	28	<0.01	<0.05	RA-2166/99 0700-99
					pulp	28	<0.01	<0.05	
					peel	28	<0.01	<0.05	
					fruit, ripened	28	<0.01	<0.05	
		1	0.25		fruit	60		<0.05	RA-2166/99 0699-99

Country, Year	Application				Sample	PHI (days)	Residue, mg/kg		Reference Report no. Study no.
	Form.	No.	g ai/plant	kg ai/hl			imida- cloprid	Total	
Ecuador 1998	350 SC	1	0.35 (0.25)	0.08	fruit	0 0		<0.01 <0.01	108731 ECU-N3001-98D-B
Ecuador 1998	350 SC	1	0.35 (0.25)	0.08	fruit	7 7 14 14 19 19 33 33		<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	108731 ECU-N3001-98D-A
Ecuador 1998	350 SC	1	0.35 (0.25)	0.08	fruit	7 7		<0.01 <0.01	108731 ECU-N3002-98H
Ecuador 1998	350 SC	1	0.35 (0.27)	0.09	fruit	6 6		<0.01 <0.01	108731 ECU-N3003-98H
Ecuador 1998	350 SC	1	0.35 (0.29)	0.1	fruit	6 6		<0.01 <0.01	108731 ECU-N3004-98H
Costa Rica 1998	350 SC	1	0.35 (0.22)	0.11	fruit	0 0 7 7 14 14 20 20 35 35		<0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01 <0.01	108731 CRI-N3005-98D
Costa Rica 1998	350 SC	1	0.35 (0.19)	0.1	fruit	8 8		<0.01 0.01	108731 CRI-N3006-98H
Costa Rica 1998	350 SC	1	0.35 (0.21)	0.1	fruit	7 7		0.01 0.01	108731 CRI-N3007-98H
Costa Rica 1998	350 SC	1	0.35 (0.21)	0.1	fruit	7 7		<0.01 <0.01	108731 CRI-N3008-98H
Honduras 1998	350 SC	1	0.35 (0.20)	0.1	fruit	7 7		<0.01 <0.01	108731 HON-N3009-98H
Honduras 1998	350 SC	1	0.35 (0.21)	0.1	fruit	7 7		<0.01 <0.01	108731 HON-N3010-98H-A
Guatemala 1998	350 SC	1	0.35 (0.29)	0.15	fruit	7 7		<0.01 <0.01	108731 GUA-N3011-98H
Guatemala 1998	350 SC	1	0.35 (0.29)	0.15	fruit	7 7		<0.01 <0.01	108731 GUA-N3012-98H

Mangoes (Table 86). In four trials in the Philippines two, three, four and five foliar sprays were applied at intervals of 11-30 days at 0.0025 kg ai/hl, 0.02-0.063 kg ai/ha, 200 SL formulation.

In six trials in the USA using a 240 SC formulation 6 treatments (interval 6-8 days) were applied. In the three in Hawaii sprays were dilute (0.004 kg ai/hl) and in the three in Florida concentrated (0.016 kg ai/hl), with application rates from 0.072 to 0.097 kg ai/ha.

Table 86. Residues in mangoes after foliar spray treatment.

Country Year Location	Application				Sample	PHI (days)	Residue, mg/kg		Report No. Study no. Remark
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Philippines 1996	200 SL	2	0.02- 0.063	0.0025	<i>Unripe fruit</i>				<i>CROP/F16/96</i> Total residue 6-CNA ³
					peel ¹	92	<0.02	0.02	
					peel ²	92	<0.02	0.02	
					pulp	92	<0.02	0.01	
					<i>Ripe fruit</i>				
					peel	92	<0.02	0.02	
Philippines 1996	200 SL	3	0.02- 0.063	0.0025	<i>Unripe fruit</i>				<i>CROP/F16/96</i> Total residue 6-CNA ³
					peel ¹	75	0.03	0.05	
					peel ²	75	0.02	0.03	
					pulp	75	<0.02	0.02	
					<i>Ripe fruit</i>				
					peel	75	0.02	0.05	
Philippines 1996	200 SL	4	0.02- 0.063	0.0025	<i>Unripe fruit</i>				<i>CROP/F16/96</i> Total residue 6-CNA ³
					peel ¹	60	0.06	0.14	
					peel ²	60	0.04	0.01	
					pulp	60	<0.02	0.01	
					<i>Ripe fruit</i>				
					peel	60	0.02	0.02	
Philippines 1996	200 SL	5	0.02- 0.063	0.0025	<i>Unripe fruit</i>				<i>CROP/F16/96</i> Total residue 6-CNA ³
					peel ¹	30	0.23	0.05	
					peel ²	30	0.19	0.04	
					pulp	30	<0.02	0.03	
					<i>Ripe fruit</i>				
					peel	30	0.08	0.05	
USA, HI 1992 Waianae	240 SC	6	0.086- 0.097	0.004	fruit without stone	29		<u>0.11</u>	105011 USA-HI-S1 dilute spray
						29		<0.05	
						29		<0.05	
						39		0.10	
						39		<0.05	
						39		<0.05	
USA, HI 1992 Pahala	240 SC	6	0.081	0.004	fruit without stone	50		0.07	105011 USA-HI-S2 dilute spray
						50		<0.05	
						50		<0.05	
						31		<0.05	
						31		<0.05	
						31		<0.05	
USA, HI 1992 Pahala	240 SC	6	0.081	0.004	fruit without stone	39		<0.05	105011 USA-HI-S2 dilute spray
						39		<0.05	
						39		<0.05	
						52		<0.05	
						52		<0.05	
						52		<0.05	

Country Year Location	Application				Sample	PHI (days)	Residue, mg/kg		Report No. Study no. Remark
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, HI 1992 Waimanalo	240 SC	6	0.093	0.004	fruit without stone	30 30 30 40 40 40 50 50 50		0.14 <0.05 <0.05 0.14 <0.05 <0.05 <u>0.15</u> <0.05 <0.05	105011 USA-HI-S3 dilute spray
USA, FL 1992 Homestead	240 SC	6	0.072- 0.078	0.016	fruit without stone	41 41 41 52 52 52		<0.05 <0.05 <0.05 <0.05 <0.05 <0.05	105011 USA-FL-S4 concentrate spray
USA, FL 1992 Homestead	240 SC	6	0.072- 0.078	0.016	fruit without stone	31 31 31 41 41 41 52 52 52		<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	105011 USA-FL-S5 concentrate spray
USA, FL 1992 Homestead	240 SC	6	0.072- 0.078	0.016	fruit without stone	31 31 31 41 41 41 52 52 52		<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	105011 USA-FL-S6 concentrate spray

¹ Unwashed² Washed³ Total residue was 6-chloronicotinic acid only (determined by HPLC)

Leeks (Table 87). Imidacloprid has an authorized minor use for seed dressing in Germany with application rates of 45 g ai/unit (250,000 seeds), with a maximum equivalent rate of 0.09 kg ai/ha. In four trials in northern European countries a seed dressing rate of 60 g ai/unit, corresponding to 0.06 to 0.072 kg ai/ha, was used.

Table 87. Residues in leeks after seed treatment, 1998.

Country	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha			Imidacloprid	Total	
Belgium	70 WS	1	0.06 ¹	treated seed shoot	0 190	62000 <0.01	<0.05	RA-2178/98 1675-98

Country	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha			Imidacloprid	Total	
Germany	70 WS	1	0.072 ¹	treated seed shoot	1 158	61000 <0.01	<0.05	RA-2178/98 1672-98
Germany	70 WS	1	0.072 ¹	treated seed shoot	1 158	61000 <0.01	<0.05	RA-2178/98 1673-98
UK	70 WS	1	0.072 ¹	treated seed shoot	0 160	62000 <0.01	<0.05	RA-2178/98 1674-98

¹ 60 g ai/unit (1 unit 250000 seeds)

Onions, Bulb (Table 88). In northern Europe eight trials were conducted with a seed treatment rate of 45 g ai/unit, corresponding to 0.18 kg ai/ha.

Table 88. Residues in onions after seed treatment.

Country	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha			Imidacloprid	Total	
Germany 1996	70 WS	1	0.18 ¹	treated seed bulb	0 199	59000 <0.01	<0.05	RA-2047/96 0242-96
Germany 1996	70 WS	1	0.18 ¹	treated seed bulb	0 179	59000 <0.01	<0.05	RA-2047/96 0507-96
UK 1998	70 WS	1	0.18 ¹	treated seed bulb	0 180	55000 <0.01	<0.05	RA-2047/96 0508-96
France, North 1998	70 WS	1	0.18 ¹	treated seed bulb	0 183	58000 0.03	0.06	RA-2047/96 0509-96
Germany 1997	70 WS	1	0.18 ¹	treated seed bulb	0 182	46110 <0.01	<0.05	RA-2158/97 0003-97
Benelux 1997	70 WS	1	0.18 ¹	treated seed bulb	0 187	45730 <0.01	<0.05	RA-2158/97 0350-97
Germany 1997	70 WS	1	0.18 ¹	treated seed bulb	0 181	46110 <0.01	<0.05	RA-2158/97 0349-97
UK 1997	70 WS	1	0.18 ¹	treated seed bulb	0 181	45550 <0.01	<0.05	RA-2158/97 0351-97

¹ 45 g ai/unit (1 unit = 1 kg = 250,000 seeds)

Broccoli (Table 89). In three trials in Italy and one in Spain 2 foliar spray applications (interval 14-15 days) of a 200 SL formulation were made at 0.01 kg ai/hl, 0.1 kg ai/ha (water 1000 l/ha) .

In Australia two field trials with four spray applications (interval 19-28 days) of a 350 SC formulation, the first 2 weeks after transplanting, at the recommended rate or a double rate were applied (0.05-0.06 and 0.011-0.12 kg ai/ha, 800–1000 l/ha).

In twelve US trials a 240 SC formulation of imidacloprid was applied three times, the first as a soil drench to the base of the plants 14 days after transplanting at 0.01 g ai/plant, followed by two foliar sprays each at 0.12 kg ai/ha.

In a further ten US trials the results from various types of soil applications were compared.

- Soil drench applications 14 days after transplanting (4 trials in parallel with the above soil drench plus spray applications, at 0.01 g ai/plant. Two field samples were taken at harvest.
- In-furrow soil applications at planting, 0.03 g ai/m row, equivalent to 0.57- 0.6 kg ai/ha (three trials).
- Sidedress applications two weeks after transplanting, 0.03 g ai/m row, 0.57-0.6 kg ai/ha (three trials).

In two field trials in South Africa imidacloprid was applied once as a drench application to seedling trays a day before transplanting (70 WS formulation, 0.27 and 0.53 kg ai/ha). Leaves were sampled on days 48 and 62 and heads on days 76 and 90.

Table 89. Residues in broccoli.

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Foliar spray treatment									
Italy 1995	200 SL	2	0.1	0.01	head	0 ¹ 0 7 10 14 21	0.04 0.07	0.16 0.64 0.42 0.35 <u>0.31</u> 0.27	RA-2117/95 0148-95
Italy 1995	200 SL	2	0.1	0.01	head	0 14	0.02	0.37 <u>0.1</u>	RA-2117/95 0637-95
Italy 1995	200 SL	2	0.1	0.01	head	0 14	0.03	0.64 <u>0.29</u>	RA-2117/95 0638-95
Spain 2000	200 SL	2	0.1	0.01	head	0 ¹ 0 7 9 14 21	0.11 0.28 0.02	0.23 0.47 0.11 0.09 <u>0.08</u> 0.07	RA-2083/00 0584-00
Australia 1994	350 SC	4	0.048-0.061	0.006	head	0 3 7 14		0.70 (0.92) ² 0.52 (0.63) ² <u>0.19</u> (0.20) ² 0.17 (0.17) ²	RTL-23-94 AUS-RTL23- 94-A
Australia 1994	350 SC	4	0.097-0.12	0.012	head	0 3 7 14		1.3 (1.7) ² 1.1 (1.3) ² 0.83 (1.04) ² 0.29 (0.33) ²	RTL-23-94 AUS-RTL23- 94-B

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Soil drench and spray application									
USA, TX 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.008 0.15- 0.16	head with stalk	0 7 14		0.39 0.18 0.19	105019 352-N3001-92D
USA, OR 1992	240 SC	1 2	0.21 ³ 0.12 ⁴	0.008 0.13	head with stalk	0 7 14		0.47 0.18 0.12	105019 451-N3002-92D
USA, CA 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.008 0.13	head with stalk	0 7 14		0.32 0.25 0.25	105019 457-N3003-92D
USA, CA 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.008 0.13	head with stalk	0 7 14		2.5 0.33 0.11	105019 457-N3004-92D
USA, CA 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.008 0.044	head with stalk	0 8 15		1.3 0.62 0.31	105019 458-N3005-92D
USA, AZ 1992	240 SC	1 2	1.0 ³ 0.12 ⁴	0.008 0.13	head with stalk	0 7 14		0.4 0.3 0.32	105019 458-N3006-92D
USA, NJ 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.008 0.13	head with stalk	0 7 15		3.3 2.3 1.4	105019 756-N3007-92D
USA, MS 1992	240 SC	1 2	0.42 ³ 0.12 ⁴	0.007 0.14- 0.15	head with stalk	0 7 14		0.7 0.24 0.49	105019 BMS-N3008-92D
USA, CA 1992	240 SC	1 2	0.51 ³ 0.12 ⁴	0.008 0.14	head with stalk	0 7 14		0.54 0.12 0.38	105019 FCA-N3009-92D
USA, IN 1992	240 SC	1 2	0.62 ³ 0.12 ⁴	0.008 0.15	head with stalk	0		1.1	105019 HIN-N3010-92D
					head without stalk	7 14		0.63 0.35	
					stalk	7 14		0.23 0.18	
USA, KS 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.011 0.13	head with stalk	0 7 14		0.47 0.1 0.07	105019 STF-N3011-92D
USA, GA 1992	240 SC	1 2	0.21 ³ 0.12 ⁴	0.008 0.05- 0.06	head with stalk	0 7 14		1.1 0.41 0.13	105019 TGA-N3224-92D
Soil drench application									
USA, CA 1992	240 SC	1	0.56 ³	0.008	head with stalk	32 32		0.2 0.19	105019 FCA-N3013-92H

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, IN 1992	240 SC	1	0.6 ³	0.008	head with stalk	37 40		0.21 0.18	105019 HIN-N3014-92H
USA, KS 1992	240 SC	1	0.56 ³	0.01	head with stalk	36		<0.05	105019 STF-N3015-92H
USA, GA 1992	240 SC	1	0.19 ³	0.01	head with stalk	50 50		<0.05 <0.05	105019 TGA-N3225-92H
<i>In-furrow application (0.03 g ai/m row)</i>									
USA, CA 1992	240 SC	1	0.6	1.2	head with stalk	92 92		0.26 0.21	105019 FCA-N3020-92H
USA, IN 1992	240 SC	1	0.57	1.2	head with stalk	85		<0.05	105019 HIN-N3021-92H
USA, GA 1992	240 SC	1	0.6	1.3	head with stalk	78		<0.05	105019 TGA-N3227-92H
<i>Sidedress application (0.03 g ai/m row)</i>									
USA, IN 1992	240 SC	1	0.57	1.2	head with stalk	37 42		0.29 0.36	105019 HIN-N3027-92H
USA, CA 1992	240 SC	1	0.6	1.3	head with stalk	56 56		0.23 0.2	105019 FCA-N3026-92H
USA, GA 1992	240 SC	1	0.6	1.1	head with stalk	17		<0.05	105019 TGA-N3253-92H
<i>Soil drench application</i>									
South Africa 1994	70 WS	1	0.27	0.27	leaf	48 62		0.95 2.3	311-88980-M200 88980-M200-B
					head	76		<0.05	
	70 WS	1	0.53	0.53	head	90		0.19	311-88980-M200 88980-M200-C

¹ Before last treatment² () Residue data Corrected for analytical recovery and residue in untreated control (0.04 mg/kg).³ Drench application: 0.01 g ai/plant⁴ Foliar spray: Silwet L-77 was used as a spray adjuvant.

Cauliflower (Table 90). In five field trials in Italy there were two foliar spray applications of a 200 SL formulation (interval 15–18 days), 0.01 kg ai/hl, 0.1 kg ai/ha (1000 l water/ha).

In Australia in two field trials two spray applications of a 350 SC formulation were made, the first application 2 weeks after transplanting, and the second 21 days later, at 0.06 and 0.12 kg ai/ha (390 l/ha).

In the USA 22 field trials were conducted using with a 240 SC formulation. In twelve three applications of imidacloprid were made, the first as a soil drench to the base of each plant 14 days after transplanting, 0.02 g ai/plant, 0.56 kg ai/ha, and the other two as foliar sprays, 0.12 kg ai/ha.

The other ten were bridging trials to compare residues from the various types of soil application.

- Soil drench applications 14 days after transplanting, 0.02 g ai/plant, 0.51-0.66 kg ai/ha (four trials run in parallel to those described above).
- In-furrow soil applications at planting 0.03 g ai/m row, 0.57- 0.60 kg ai/ha (three trials).
- Sidedress applications two weeks after transplanting, 0.03 g ai/m row, 0.57-0.6 kg ai/ha (three trials).

In South Africa in a single field trial a 70 WS formulation was applied as a drench to seedling trays a day before transplanting, 0.27 kg ai/ha.

Table 90. Residues in cauliflowers.

Country Year Location	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Foliar spray application									
Italy 1993 Borgo Piave	200 SL	2	0.1	0.01	head	0 ¹ 0 7 14 21 28	0.02 0.02	0.05 0.19 0.1 0.09 <u>0.11</u> 0.07	RA-2023/93 0009-93
Italy 1993 Cerig- nola	200 SL	2	0.1	0.01	head	0 ¹ 0 7 14 21 28	<0.01 <0.01	<0.05 <0.05 0.06 <u>0.08</u> 0.07 0.07	RA-2023/93 0382-93
Italy 1995 Foggia	200 SL	2	0.1	0.01	head	0 ¹ 0 7 10 14 21	0.01 <0.01	<0.05 0.08 0.07 0.08 <0.05 <u>0.07</u>	RA-2116/95 0147-95
Italy 1995 Latina	200 SL	2	0.1	0.01	head	0 14	0.01	0.08 <u>0.06</u>	RA-2116/95 0635-95
Italy 1995 Bari	200 SL	2	0.1	0.01	head	0 14	0.01	0.12 <u>0.09</u>	RA-2116/95 0636-95
Australia 1996 WA	350 SC	2	0.06 ²	0.015	head	0 3 7 14 21		<0.01 <0.01 <0.01 <u>0.01</u> <0.01	MWS 362/96 AUS-MWS362- 96-A

Country Year Location	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Australia 1996	350 SC	2	0.12 ²	0.031	head	0 3 7 14 21		0.06 0.02 0.03 0.03 <0.01	MWS 362/96 AUS-MWS362- 96-B
<i>Soil drench and spray application</i>									
USA, TX 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.16	head	0 7 14		0.2 0.1 0.11	105022 352-N3058-92D
USA, OR 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.13	head	0 7 14		0.06 <0.05 <0.05	105022 451-N3059-92D
USA, CA 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.13	head	0 7 14		0.68 0.27 0.25	105022 457-N3060-92D
USA, CA 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.13	head	0 7 14		0.44 0.14 0.12	105022 457-N3061-92D
USA, CA 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.13	head	0 7 14		0.33 0.1 0.16	105022 458-N3062-92D
USA, AR 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.13	head	0 7 14		0.19 0.13 0.11	105022 458-N3063-92D
USA, NY 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.14	head	0 7 14		0.21 0.09 0.08	105022 758-N3064-92D
USA, MS 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.013 0.16	head	0 7 14		0.2 0.11 0.1	105022 BMS-N3065- 92D
USA, CA 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.14	head	0 7 14		0.33 0.34 0.28	105022 FCA-N3066-92D
USA, IN 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.15	head	0 7 14		0.2 0.08 0.09	105022 HIN-N3067-92D
USA, KS 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.022 0.13	head	0 7 14		0.46 0.23 0.13	105022 STF-N3068-92D
USA, FL 1992	240 SC	1 2	0.56 ³ 0.12 ⁴	0.02 0.13	head	0 7 11		0.88 0.39 0.6	105022 VBL-N3069-92D

Country Year Location	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Soil drench application									
South Africa 1995	70 WS	1	0.27		head	99 112 125 136		4.0 0.23 0.13 <0.05	311-88977-M196 88977-M196-B drench
USA, CA 1992	240 SC	1	0.62 ³	0.017	head	151 151		0.22 0.25	105022 FCA-N3070-92H
USA, IN 1992	240 SC	1	0.66 ³	0.017	head	47 47		0.14 0.15	105022 HIN-N3071-92H
USA, KS 1992	240 SC	1	0.51 ³	0.022	head	38 38		<u>0.21</u> 0.18	105022 STF-N3072-92H
USA, GA 1992	240 SC	1	0.56 ³	0.02	head	50 50		0.15 0.1	105022 VBL-N3073-92H
In-furrow application									
USA, CA 1992	240 SC	1	note 5		head	152		0.05	105022 FCA-N3077-92H
USA, IN 1992	240 SC	1	0.57	1.2	head	91		<0.05	105022 HIN-N3078-92H
USA, GA 1992	240 SC	1	0.6	1.1	head	82		0.06	105022 VBL-N3079-92H
Sidedress application									
USA, IN 1992	240 SC	1	note 5		head	116 116		0.11 0.09	105022 FCA-N3083-92H
USA, CA 1992	240 SC	1	0.57	1.2	head	47 47		0.16 0.08	105022 HIN-N3084-92H
USA, GA 1992	240 SC	1	0.6	1.1	head	50		0.07	105022 VBL-N3085-92H

¹ Before last treatment

² Treatment applied with Agral 600 adjuvant at 0.01%.

³ Drench application: 0.02 g ai/plant

⁴ Foliar spray: Silwet L-77 was used as a spray adjuvant

⁵ In-furrow or sidedress application: 0.03 g ai m/row

Brussels sprouts (Table 91). In Australia there were four field trials with two or three spray applications of a 350 SC formulation (interval 19–22 days). Either the recommended rate of 0.06 or a double rate of 0.12 kg ai/ha was applied.

In South Africa in two field trials a 70 WS formulation was applied as a drench on the seedling trays a day before transplanting, at rates corresponding to 0.27 and 0.53 kg ai/ha respectively.

Only the total residue was determined in all the trials.

Table 91. Residues in Brussels sprouts.

Country Year	Application				PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			
Foliar spray application							
Australia 1994	350 SC	3	0.06	0.006 (water 1000 l/ha)	control 0 3 7 14	0.1 0.47 0.34 <u>0.32</u> 0.22	RTL 24/94 AUS-RTL24-94-A
Australia 1994	350 SC	3	0.12	0.012 (water 1000 l/ha)	control 0 3 7 14	0.1 0.56 0.4 0.4 0.27	RTL 24/94 AUS-RTL24-94-B
Australia 1996	350 SC	2	0.06	0.021 (water 283 l/ha)	0 3 7 14	<0.01 0.02 0.01 <u>0.03</u>	DJR 104/96 AUS-DJR104-96-A
Australia 1996	350 SC	2	0.12	0.042 (water 283 l/ha)	0 3 7 14	0.04 0.03 0.04 0.06	DJR 104/96 AUS-DJR104-96-B
Soil drench application							
South Africa 1995	70 WS	1	0.27	0.27 (water 100 l/ha)	91	< <u>0.05</u>	311-88979-M199 88979-M199-B
South Africa 1995	70 WS	1	0.53	0.53 (water 100 l/ha)	135	< <u>0.05</u>	311-88979-M199 88979-M199-C

Cabbages, Head (Table 92). In Australia four field trials were with three or five spray applications (interval 17–28 days). Either the recommended rate of 0.06 kg ai/ha or a double rate of 0.12 kg ai/ha was applied.

In the USA 22 trials were conducted with a 240 SC formulation. Cabbages were collected with wrapper leaves (head) and without wrapper leaves (cleaned head). Twelve trials were with three applications. The first was a soil drench at the base of each plant fourteen days after transplanting, when 0.02 g ai/plant was applied. The remaining applications were two foliar sprays at rates of 0.12 kg ai/ha.

The remaining ten trials were bridging studies to compare the residues from three types of soil application.

- Soil drenches were applied 14 days after transplanting in 4 trials which were run in parallel to the above trials with soil drench plus spray applications. The rate was 0.02 g ai/plant.
- In-furrow applications at planting at 0.03 g ai/m row, corresponding to 0.57-0.6 kg ai/ha (3 trials).
- Sidedress applications two weeks after transplanting. Three trials conducted at 0.03 g ai/m row. This rate corresponded to 0.57-0.6 kg ai/ha.

Only the total residue was determined in all trials.

Table 92. Residues in head cabbages.

Country Year	Application				Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl				
Foliar spray application								
Australia 1994	350 SC	5	0.061	0.006	heart and wrapper leaves	0 2 7 13	0.30 (0.36) ³ 0.47 (0.59) ³ 0.22 (0.26) ³ 0.22 (0.26) ³	RTL 25/94 AUS-RTL25-94-A
Australia 1994	350 SC	5	0.12	0.012	heart and wrapper leaves	0 2 7 13	0.71 (0.9) ³ 0.95 (1.2) ³ 0.27 (0.32) ³ 0.19 (0.22) ³	RTL 25/94 AUS-RTL25-94-B
Australia 1996	350 SC	3	0.06	0.006- 0.012	head without wrapper leaves	0 4 7 14 21	0.15 0.01 <u>0.02</u> 0.02 0.01	ADM 040/97 AUS-ADM040- 97-A
Australia 1996	350 SC	3	0.12	0.012- 0.024	head without wrapper leaves	0 4 7 14 21	0.27 0.07 0.03 0.02 0.02	ADM 040/97 AUS-ADM040- 97-B
Soil drench plus spray application								
USA, TX 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.16	head with wrapper leaves	0 7 14	0.61 0.47 0.3	105040 352-N3029-92D
					head, cleaned	0 7 14	0.07 0.09 <0.05	
USA, TX 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.13	head with wrapper leaves	0 7 14	3.0 1.0 1.3	105040 353-N3030-92D
					head, cleaned	0 7 14	0.5 0.21 0.27	
USA, CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.13	head with wrapper leaves	0 7 14	4.1 1.8 3.3	105040 457-N3031-92D
					head, cleaned	0 7 14	1.2 0.6 0.94	
USA, NC 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.13	head with wrapper leaves	0 7 14	2.1 1.6 1.3	105040 751-N3032-92D

Country Year	Application				Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl				
					head, cleaned	0 7 14	0.24 0.16 0.15	
USA, FL 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.13	head with wrapper leaves	0 7 14	1.9 0.94 0.94	105040 753-N3033-92D
					head, cleaned	0 7 14	0.46 0.13 0.16	
USA, NJ 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.13	head with wrapper leaves	0 8 14	0.7 0.28 0.13	105040 756-N3034-92D
					head, cleaned	0 8 14	0.15 0.11 0.11	
USA, NY 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.14	head with wrapper leaves	0 7 14	0.17 0.22 0.49	105040 758-N3035-92D
					head, cleaned	0 7 14	0.11 0.07 <0.05	
USA, WI 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.13	head with wrapper leaves	0 7 14	0.3 0.27 0.13	105040 851-N3036-92D
					head, cleaned	0 7 14	<0.05 <0.05 <0.05	
USA, MS 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.15	head with wrapper leaves	0 7 14	0.91 0.34 0.29	105040 BMS-N3037-92D
					head, cleaned	0 7 14	0.2 0.17 0.14	
USA, CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.16 0.16	head with wrapper leaves	0 7 14	0.78 0.76 0.28	105040 FCA-N3038-92D
					head, cleaned	0 7 14	0.08 0.16 0.13	
USA, IN 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.15 0.15	head with wrapper leaves	0 7 14	0.84 0.45 0.28	105040 HIN-N3039-92D

Country Year	Application				Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl				
					head, cleaned	0 7 14	0.2 0.14 0.17	
USA, KS 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.132	head with wrapper leaves	0 7 15	0.08 0.05 <0.05	105040 STF-N3040-92D
					head, cleaned	0 7 15	<0.05 <0.05 <0.05	
USA, FL 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.021 0.132	head with wrapper leaves	0 7 14	0.85 0.27 0.2	105040 VBL-N3041-92D
					head, cleaned	0 7 14	0.14 0.11 0.12	
Soil drench application								
USA, CA 1992	240 SC	1	0.56 ¹	0.02	head with wrapper leaves	47 47	0.19 0.33	105040 FCA-N3042-92H
					head, cleaned	47	0.08	
USA, IN 1992	240 SC	1	0.56 ¹	0.02	head with wrapper leaves	40 40	0.17 0.21	105040 HIN-N3043-92H
					head, cleaned	40 40	0.13 0.1	
USA, KS 1992	240 SC	1	0.56 ¹	0.025	head with wrapper leaves	64	<0.05	105040 STF-N3044-92H
					head, cleaned	64	0.06	
USA, FL 1992	240 SC	1	0.56 ¹	0.02	head with wrapper leaves	51 51	0.28 0.32	105040 VBL-N3045-92H
					head, cleaned	51 51	0.27 0.23	
In-furrow application (0.03 g ai/m row)								
USA, FL 1992	240 SC	1	0.6	1.1	head with wrapper leaves	104	0.07	105040 VBL-N3051-92H
					head, cleaned	104	0.05	
USA, IN 1992	240 SC	1	0.57	1.2	head with wrapper leaves	86	0.05	105040 HIN-N3050-92H
					head, cleaned	86	0.09	
USA, CA 1992	240 SC	1	0.59	1.2	head with wrapper leaves	92 92	0.18 0.22	105040 FCA-N3049-92H

Country Year	Application				Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl				
					head, cleaned	92	0.06	
Sidedress application (0.03 g ai/m row)								
USA, FL 1992	240 SC	1	0.6	1.1	head with wrapper leaves	72	<0.05	105040 VBL-N3057-92H
					head, cleaned	72	<0.05	
USA, IN 1992	240 SC	1	0.57	1.2	head with wrapper leaves	40 40	0.2 0.21	105040 HIN-N3056-92H
					head, cleaned	40	<0.05	
USA, CA 1992	240 SC	1	0.6	1.3	head with wrapper leaves	56 56	0.35 0.33	105040 FCA-N3055-92H
					head, cleaned	56 56	0.15 0.13	

¹ Drench application: 0.02 g ai/plant

² Foliar spray: Silwet L-77 was used as a spray adjuvant

³ () Residue data corrected for analytical recovery and residue in control (0.02 mg/kg).

Cucumbers (Table 93). In Italy in an indoor trial the crop was sprayed twice at 0.15 kg ai/ha (interval 7 days), which was in accordance with the Spanish GAP.

In Spain three outdoor spray trials were with application rates of 0.1 kg ai/ha (2 treatments, interval 15 days). The spray concentration was 0.01 kg ai/hl.

In Australia 2 outdoor trials were carried out with four spray applications (intervals 5, 26 and 14 days). The application rates were 0.06 and 0.12 kg ai/ha (water 164-168 l/ha).

Six indoor trials were conducted with drip irrigation by automatic irrigators in The Netherlands with a 70 WG formulation. The plants were grown on rock wool. In two trials 2.5 mg imidacloprid was applied to the base of each plant in 10 ml water, equal to a rate of 0.024-0.034 kg ai/ha, in accordance with the lowest rate registered in The Netherlands. The plants in the other four trials received 10 mg imidacloprid per plant, which corresponds to an application rate of about 0.12-0.15 kg ai/ha. The rate of 10 mg ai/plant is in accordance with the label rate in The Netherlands, Denmark, Greece, and Spain.

Eight further indoor trials were in southern France and two in Spain with drip irrigation at 25 mg ai/plant. This represented 2.5 times the label rate in Greece and Spain. In 3 trials the crop was cultivated on rock wool and in the other 7 trials in soil.

Table 93. Residues in cucumbers.

Country Year	Application				PHI (days)	Residues, mg/kg		Report no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total residue	Study no. Remark
Foliar spray application								
Italy 1994 Greenhouse	200 SL	2	0.15	0.015	0 ¹ 0 3 5 7 10	<0.01 <0.01	0.06 0.12 0.1 0.1 0.15 0.11	RA-2050/94 0210-94
Spain 1990 Field	200 SL	2	0.1	0.01	0 7 14 21 28	 <0.01	0.11 0.06 <0.05 <0.05 <0.05	0508-90
Spain 1990 Field	200 SL	2	0.1	0.01	0 7 14 21 28	 <0.01	0.1 <0.05 <0.05 <0.05 <0.05	0509-90
Spain 1990 Field	200 SL	2	0.1	0.01	0 7 14 21 29	 <0.01	0.11 0.07 0.05 <0.05 <0.05	0511-90
Australia 1993 Field	350 SC	4	0.06	0.036	0 1 3 7	 	0.03 0.03 <u>0.04</u> <0.02	67/93 A AUS-67-93-A
Australia 1993 Field	350 SC	4	0.12	0.07	0 1 3 7	 	0.1 0.09 0.08 0.06	67/93 B AUS-67-93-B
Drip irrigation application								
Netherlands 1993 Greenhouse	70 WG	1	0.034 ²	0.025	1 3 5 7 14 21	0.02 0.02 0.02 0.01 <0.01 <0.01	<0.05 0.11 <u>0.15</u> 0.13 0.07 <0.05	RA-2096/93 0492-93 Artificial substrate (rock wool)
Netherlands 1993 Greenhouse	70 WG	1	0.025 ²	0.025	1 3 5 7 14 21	0.04 0.06 0.05 0.05 0.02 0.01	<0.05 0.08 0.08 <u>0.1</u> 0.06 <0.05	RA-2096/93 0494-93 Artificial substrate (rock wool)

Country Year	Application				PHI (days)	Residues, mg/kg		Report no. Study no. Remark
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total residue	
Netherlands 1994 Greenhouse	70 WG	1	0.14 ³	0.098	1	0.07	0.07	RA-2096/94 0495-93 Artificial substrate (rock wool)
					3	0.14	0.29	
					5	0.09	<u>0.31</u>	
					7	0.06	0.3	
					14	0.02	0.17	
					21	<0.01	0.1	
Netherlands 1994 Greenhouse	70 WG	1	0.13 ³	0.098	1	0.04	0.07	RA-2096/94 0287-94 Artificial substrate (rock wool)
					3	0.07	<u>0.25</u>	
					5	0.05	0.21	
					7	0.02	0.2	
					14	<0.01	0.08	
					21	<0.01	<0.05	
Netherlands 1995 Greenhouse	70 WG	1	0.15 ³	0.098	7	0.07	<u>0.39</u>	RA-2146/95 0203-95 Artificial substrate (rock wool)
					14	0.01	0.2	
					21	<0.01	0.08	
					28	<0.01	0.07	
					35	<0.01	0.05	
Netherlands 1995 Greenhouse	70 WG	1	0.12 ³	0.098	7	<0.01	<u>0.39</u>	RA-2146/95 0687-95 Artificial substrate (rock wool)
					14	<0.01	0.12	
					21	<0.01	0.05	
					28	<0.01	<0.05	
					35	<0.01	<0.05	
France 1992 Greenhouse	200 SL	1	0.29 ⁴	0.12	0		<0.05	RA-2043/92 0445-92 Artificial substrate (rock wool)
					3		0.39	
					7	<0.01	0.42	
France 1992 Greenhouse	200 SL	1	0.29 ⁴	0.12	0		<0.05	RA-2043/92 0446-92 Artificial substrate (rock wool)
					3		0.61	
					7	0.03	0.61	
France 1992 Greenhouse	200 SL	1	0.29 ⁴	0.12	0		<0.05	RA-2043/92 0447-92 Artificial substrate (rock wool)
					3		0.5	
					7	0.03	0.53	
France 1992 Greenhouse	200 SL	1	0.29 ⁴	0.12	0		<0.05	RA-2043/92 0449-92 Soil
					3		<0.05	
					7	<0.01	<0.05	
France 1994 Greenhouse	200 SL	1	0.4 ⁴		3	0.01	<0.05	RA-2030/94 0123-94 Soil
					5		<0.05	
					7	0.02	<0.05	
					14		<0.05	
France 1994 Greenhouse	200 SL	1	0.4 ⁴		3	0.01	<0.05	RA-2030/94 0124-94 Soil
					5		0.05	
					7	0.02	<0.05	
					14		<0.05	
France 1994 Greenhouse	200 SL	1	0.4 ⁴		3	<0.01	<0.05	RA-2030/94 0125-94 Soil
					5		<0.05	
					7	<0.01	<0.05	
					14		<0.05	

Country Year	Application				PHI (days)	Residues, mg/kg		Report no. Study no. Remark
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total residue	
France 1994 Greenhouse	200 SL	1	0.4 ⁴		3	<0.01	<0.05	RA-2030/94 0126-94 Soil
					5		<0.05	
					7	<0.01	<0.05	
					14		<0.05	
Spain 1993 Greenhouse	200 SL	1	0.28 ⁴		0		<0.05	RA-2021/93 0456-93 Soil
					3	0.01	<0.05	
					7	0.02	0.05	
					14		0.05	
Spain 1993 Greenhouse	200 SL	1	0.66 ⁴		0	<0.01	<0.05	RA-2021/93 0457-93 Soil
					3	0.01	0.26	
					7	<0.01	0.29	
					14	<0.01	0.26	

¹ Before final treatment² 2.5 mg ai/plant³ 10 mg ai/plant⁴ 25 mg ai/plant

Squash, Summer (zucchini, Table 94). In Italy two trials were according to GAP in Spain with two foliar spray applications at 0.15 kg ai/ha (interval 7 days).

Table 94. Residues in zucchini after foliar spray applications in the greenhouse, Italy 1944.

Application				PHI (days)	Residue, mg/kg		Report no. Study no.
Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
200 SL	2	0.15	0.015	0 ¹	0.07	0.08	RA-2050/94 0219-94
				0		0.31	
				3		0.23	
				5	0.13	0.21	
				7		0.17	
				10		0.08	
200 SL	2	0.15	0.015	0 ¹	0.01	0.05	RA-2050/94 0220-94
				0		0.14	
				3		0.05	
				5	0.02	<0.05	
				7		<0.05	
				10		<0.05	

¹ Before final treatment

Melons, except watermelon (Table 95). In eight field trials in southern Europe (Italy and Spain) two foliar sprays of 200 SL formulation (interval 15 days) were at a concentration of 0.01 kg ai/hl. This corresponded to 0.1 kg ai/ha in seven trials, and 0.05-0.075 kg ai/ha in the eighth. These trials were according to GAP in Italy, Portugal and Spain.

Four trials were reported from Italy in which 2 foliar sprays (interval 20 days) with 300 EC formulation were applied at 0.009 kg ai/hl, corresponding to a rate of 0.09 kg ai/ha.

In Australia four field trials were conducted with four foliar sprays of a 350 SC formulation (interval 7-17 days) were made. Two trials were with the recommended rate of 0.06 kg ai/ha and two with twice that rate.

Two trials were conducted with drip irrigation in the greenhouse in Spain, with two applications at 0.1 kg ai/ha (interval 14 days). The spray volume was 6000 l/ha, corresponding to a concentration of 0.0016 kg ai/hl.

In South Africa two field trials with drench application were at either the label rate of 21 mg ai/plant hole or 42 mg ai/plant hole. The water volume was 30 ml/plant hole.

Table 95. Residues in melons.

Country Year type	Application				Residue, mg/kg				Report no. Study no.													
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total														
Foliar spray application																						
Italy 1991 Field	200 SL	2	0.05 0.075	0.01	pulp	0 3 7 10	<0.01	0.05 <u><0.05</u> <0.05 <0.05	RA-2028/91 0269-91													
					peel	0 3 7 10		<0.01		0.07 <0.05 <0.05 <0.05												
					whole fruit	0 3 7 10				<0.01	0.05 <u><0.05</u> <0.05 <0.05											
					Italy 1992 Field	200 SL	2				0.1	0.01	pulp	0 ¹ 0 3 7 10	<0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2053/92 0278-92					
								peel					0 ¹ 0 3 7 10	0.04		<0.05 0.09 0.06 0.11 0.07						
								whole fruit		0 ¹ 0 3 7 10			0.02			<0.05 0.06 <u><0.05</u> <0.05 <0.05						
								Italy 1993 Field		200 SL					2	0.1		0.01	pulp	0 ¹ 0 3 7 10	<0.01 <0.01	0.07 0.08 0.08 0.07 0.11

Country Year type	Application				Residue, mg/kg				Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total	
					peel	0 ¹ 0 3 7 10	<0.01 0.01	0.07 0.25 0.17 0.11 0.14	
					whole fruit	0 ¹ 0 3 7 10	<0.01 <0.01	0.07 0.17 <u>0.13</u> 0.09 0.13	
Italy 1993 Field	200 SL	2	0.1	0.01	pulp	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2025/93 0396-93
						peel	0 ¹ 0 3 7 10	<0.01 0.01	
					whole fruit	0 ¹ 0 3 7 10	<0.01 0.01	<0.05 0.05 0.09 0.05 0.05	
						0 ¹ 0 3 7 10	<0.01 0.01	<0.05 <0.05 <u>0.05</u> <0.05 <0.05	
						0 ¹ 0 3 7 10	<0.01 0.01	<0.05 <0.05 <u>0.05</u> <0.05 <0.05	
						0 ¹ 0 3 7 10	<0.01 0.01	<0.05 <0.05 <u>0.05</u> <0.05 <0.05	
Spain 1991 Field	200 SL	2	0.1	0.01	peel	0 3 7 10	 0.02	0.44 0.26 0.1 0.06	RA-2028/91 0197-91
						pulp	0 3 7 10	<0.05 <u>0.05</u> 0.05 0.05	
					whole fruit	0 3 7 10	 0.01	0.1 <u>0.07</u> 0.06 0.05	
						0 3 7 10	 0.01	0.1 <u>0.07</u> 0.06 0.05	
						0 3 7 10	 0.01	0.1 <u>0.07</u> 0.06 0.05	
						0 3 7 10	 0.01	0.1 <u>0.07</u> 0.06 0.05	
Spain 1992 Field	200 SL	2	0.1	0.01	peel	0 ¹ 0 3 7 9	 0.01	<0.05 0.12 0.07 0.07 0.06	RA-2059/92 0114-92
						pulp	0 ¹ 0 3 7 9	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
					whole fruit	0 ¹ 0 3 7 9	 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
						0 ¹ 0 3 7 9	 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
						0 ¹ 0 3 7 9	 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
						0 ¹ 0 3 7 9	 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	

Country Year type	Application				Residue, mg/kg				Report no. Study no.							
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total								
					whole fruit	0 ¹ 0 3 7 9	<0.01	<0.05 0.05 <u><0.05</u> <0.05 <0.05								
Spain 1993 Field	200 SL	2	0.1	0.01	pulp	0 ¹ 0 3 5 7	<0.01 <0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2026/93 0418-93							
						peel	0 ¹ 0 3 5 7	<0.01 0.02 0.01		0.10 0.24 0.18 0.17 0.17						
							whole fruit	0 ¹ 0 3 5 7		<0.01 <0.01 <0.01	<0.05 0.11 <u>0.08</u> 0.07 0.07					
					Spain 1994	200 SL	2	0.1		0.01	pulp	0 3 7	<0.01 <0.01	<0.05 <u><0.05</u> <0.05	RA-2055/94 0684-94	
												peel	0 3 7	0.01 0.01		0.25 0.09 0.08
													whole fruit	0 3 7		<0.01 <0.01
					Italy 1996 Field	300 EC	2	0.09		0.009	pulp	7	<0.01	<0.05	RA-2049/96 0040-96	
peel	7	0.01	0.07													
fruit	0 ¹ 0 3 10		<0.05 0.05 0.05 <u>0.06</u>													
	whole fruit	7	<0.01	<0.05												
Italy 1996 Field	300 EC	2	0.09	0.009	fruit	0 7	0.03	0.08 0.08	RA-2049/96 0317-96							

Country Year type	Application				Residue, mg/kg				Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total	
Italy 1997 Field	300 EC	2	0.09	0.009	fruit	0 ¹ 0 3 7 10	<0.01 <0.01	0.08 0.13 <u>0.15</u> 0.12 0.12	RA-2140/97 0056-97
					pulp	7	<0.01	0.14	
					peel	7	0.02	0.16	
Italy 1997 Field	300 EC	2	0.09	0.009	fruit	0 7	 0.01	0.10 <0.05	RA-2140/97 0057-97
Australia 1993 Field	350 SC	4	0.06	0.036	fruit	0 1 3 7		0.02 <u>0.03</u> 0.01 0.02	65/93 AUS-65-93-A
Australia 1993 Field	350 SC	4	0.12	0.071– 0.073	fruit	0 1 3 7		0.04 0.06 0.06 0.05	65/93 AUS-65-93-B
Australia 1995 Field	350 SC	4	0.06	0.043	fruit	0 1 3 7		0.09 (0.12) ³ <0.05) <u>0.07</u> (0.09) ³ <0.05	KGW 164/96 AUS-KGW164- 96-A
Australia 1995 Field	350 SC	4	0.12	0.086	fruit	0 1 3 7		0.09 (0.12) ³ <0.05 0.16 (0.21) ³ 0.17 (0.22) ³	KGW 164/96 AUS-KGW164- 96-B
Soil drip irrigation treatment									
Spain 1993 Green- house	200 SL	2	0.1	0.0016	pulp	0 ¹ 0 2 7 14	<0.01 <0.01 <0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2026/93 0416-93
					peel	0 ¹ 0 2 7 14	<0.01 <0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <0.05	
					whole fruit calcu- lated	0 ¹ 0 2 7 14	<0.01 <0.01 <0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
Spain 1993 Green- house	200 SL	2	0.1	0.0016	pulp	-14 ² 0 2 7 14	 <0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2026/93 0417-93

Country Year type	Application				Residue, mg/kg				Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total	
					peel	-14 ² 0 2 7 14	 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <0.05	
					whole fruit calcu- lated	-14 ² 0 2 7 14	 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <0.05	
Soil drench application									
South Africa 1995	350 SC	1	0.021 g ai/plant		fruit	120		<0.01	311/88987/M208A 88987-M208A-A
South Africa 1995	350 SC	1	0.042 g ai/plant		fruit	120		<0.01	311/88987/M208A 88987-M208A-B

¹ Sampling before last treatment

² Sampling before first treatment

³ () Residue data corrected for analytical recovery.

Watermelons (Table 96). Nine field trials in southern Europe (Greece, Italy and Spain) were with 2 spray applications (interval 7–20 days) of a 200 SL formulation at 0.01 kg ai/hl, corresponding to 0.1 kg ai/ha (water 1000 l/ha) in eight trials and 0.05 kg ai/ha (water 500 l/ha) in the ninth.

In Italy in four field trials there were 2 spray applications (interval 20 days) of a EC 300 formulation at 0.009 kg ai/ha, 0.09 kg ai/ha.

Table 96. Residues in watermelons after spray applications in field trials.

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Spain 1991	200 SL	2	0.1	0.01	peel	0		0.09	RA-2028/91 0198-91
						3		0.08	
						7	<0.01	0.05	
						10		<0.05	
					pulp	0		<0.05	
						3		<0.05	
						7	<0.01	<0.05	
						10		<0.05	
					whole fruit	0		<0.05	
						3		<0.05	
						7	<0.01	<0.05	
						10		<0.05	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Italy 1991	200 SL	2	0.05	0.01	peel	0	<0.01	<0.05	RA-2028/91 0283-91
						3		0.05	
						7		<0.05	
						10		0.06	
					pulp	0	<0.01	<0.05	
						3		<0.05	
						7		<0.05	
						10		<0.05	
					whole fruit	0	<0.01	<0.05	
						3		<0.05	
						7		<0.05	
						10		<0.05	
Italy 1992	200 SL	2	0.1	0.01	pulp	0 ¹	<0.01	<0.05	RA-2053/92 0254-92
						0		<0.05	
						3		<0.05	
						7		0.05	
						10		<u>0.06</u>	
					peel	0 ¹	0.01	<0.05	
						0		0.12	
						3		0.15	
						7		0.11	
						10		0.18	
					whole fruit	0 ¹	<0.01	<0.05	
						0		0.06	
						3		0.07	
						7		0.07	
						10		<u>0.1</u>	
Italy 1992	200 SL	2	0.1	0.01	pulp	0	<0.01	<u><0.05</u>	RA-2053/92 0256-92
						7		<0.05	
						10		<0.05	
					peel	0	<0.01	<0.05	
						7		<0.05	
						10		<0.05	
					whole fruit	0	<0.01	<u><0.05</u>	
						7		<0.05	
						10		<0.05	
Spain 1993	200 SL	2	0.1	0.01	pulp	0 ¹	<0.01	<0.05	RA-2059/92 0115-92
						0		<0.05	
						3		<u><0.05</u>	
						7		<0.05	
						10		<0.05	
					peel	0 ¹	<0.01	0.05	
						0		0.14	
						3		0.07	
						7		0.08	
						10		0.05	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
					whole fruit	0 ¹ 0 3 7 10	<0.01	<0.05 0.06 <u><0.05</u> <0.05 <0.05	
Italy 1993	200 SL	2	0.1	0.01	pulp	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	RA-2032/93 0014-93
					peel	0 ¹ 0 3 7 10	<0.01 0.01	<0.05 <0.05 <0.05 0.05 <0.05	
					whole fruit	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
					pulp	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
					peel	0 ¹ 0 3 7 10	<0.01 0.01	<0.05 0.06 <0.05 0.08 0.06	
					whole fruit	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
					pulp	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05	
					peel	0 ¹ 0 3 7 10	<0.01 0.01	0.05 0.17 0.14 0.12 0.12	
					whole fruit	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 0.11 <u>0.09</u> 0.07 0.07	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Greece 2000	200 SL	2	0.1	0.01	fruit	0 1 3 7 10	0.02 ≤0.01 	<0.05 <0.05 <0.05 <u>0.05</u> <0.05	RA-2087/00 0071-00
					pulp	3	≤0.01	<0.05	
					peel	3	≤0.01	0.06	
Italy 1996	300 EC	2	0.09	0.009	pulp	7	≤0.01	<0.05	RA-2049/96 0039-96
					peel	7	≤0.01	<0.05	
					fruit	0 ¹ 0 3 10		<0.05 <0.05 <u><0.05</u> <0.05	
					whole fruit	7	≤0.01	<0.05	
Italy 1996	300 EC	2	0.09	0.009	fruit	0 7	 ≤0.01	<0.05 <0.05	RA-2049/96 0316-96
Italy 1997	300 EC	2	0.09	0.009	fruit	0 ¹ 0 3 7 10	<0.01 ≤0.01 	<0.05 0.06 <u>0.07</u> 0.05 0.07	RA-2140/97 0053-97
					pulp	7	≤0.01	0.05	
					peel	7	0.01	0.1	
Italy 1997	300 EC	2	0.09	0.009	fruit	0 7	<0.01	0.05 <0.05	RA-2140/97 0054-97

¹ Before last treatment

Egg plant (Table 97). Trials were only in southern Europe and Brazil. The uses in The Netherlands, the USA and Australia are not supported by residue data.

In Italy four indoor trials were with two foliar sprays (interval 7–14 days) at a concentration of 0.015 kg ai/hl, corresponding to a rate of 0.15 kg ai/ha.

In two field trials in southern France and four in Italy one or two spray applications were made. Four trials were with application rates of 0.1 kg ai/ha and two with 0.15 kg ai/ha.

In Brazil in two trials 3 spray applications of a 70 WG formulation (interval 14 days) were made at either the recommended rate or a double rate (0.14 or 0.28 kg ai/ha).

In a single Italian indoor trial the equivalent of 0.2 kg ai/ha imidacloprid was applied in the irrigation water. The soil was covered with a black plastic foil and drip irrigation simulated by flooding water under the foil. Three further field trials in Italy were at rates of 0.15 or 0.3 kg ai/ha administered by drip irrigation.

Table 97. Residues in egg plants.

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Foliar spray treatment								
Italy 1994 Green-house	200 SL	2	0.15	0.015	0 ¹ 0 3 7 14	<0.01 0.02	<0.05 <0.05 0.06 <u>0.08</u> 0.07	RA-2046/94 0224-94
Italy 1994 Green-house	200 SL	2	0.15	0.015	0 ¹ 0 3 7 14	0.01 0.01	<0.05 0.05 0.05 <u>0.06</u> <0.05	RA-2046/94 0225-94
Italy 1995 Green-house	200 SL	2	0.15	0.015	0 ¹ 0 1 3 7 14	<0.01 0.01	<0.05 <0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2114/95 0141-95
Italy 1995 Green-house	200 SL	2	0.15	0.015	0 ¹ 0 1 3 7 14	0.02 0.08	0.05 0.19 0.16 0.14 <u>0.14</u> 0.06	RA-2114/95 0633-95
France, South 1992 Field	200 SL	1	0.1	0.02	0 5 7 14	 0.01	<u><0.05</u> <0.05 <0.05 <0.05	RA-2041/92 0436-92
France, South 1992 Field	200 SL	1	0.1	0.02	0 5 7 14	 0.01	<u><0.05</u> <0.05 <0.05 <0.05	RA-2041/92 0495-92
Italy 1993 Field	200 SL	2	0.1	0.01	0 ¹ 0 3 5 7 14	<0.01 0.01 0.02	<0.05 <0.05 <u><0.05</u> <0.05 <0.05 <0.05	RA-2021/93 0013-93

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Italy 1993 Field	200 SL	2	0.1	0.01	0 ¹ 0 3 5 7 14	<0.01 0.01 <0.01	<0.05 <0.05 <u><0.05</u> <0.05 <0.05 <0.05	RA-2021/93 0376-93
Italy 1994 Field	200 SL	2	0.15	0.015	0 ¹ 0 3 7 10	<0.01 <0.01	<0.05 <0.05 <0.05 <u>0.06</u> 0.06	RA-2046/94 0221-94
Italy 1994 Field	200 SL	2	0.15	0.015	0 ¹ 0 3 5 7 10	<0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2046/94 0222-94
Brazil 1995 Field	70 WG	3	0.14	0.028	7	<0.01	<0.01	BRA I-D1-607/95 BRA-I-D1-607-95-A
Brazil 1995 Field	70 WG	3	0.28	0.056	7	0.01	<0.01	BRA I-D1-607/95 BRA-I-D1-607-95-B
<i>Drip irrigation treatment</i>								
Italy 1998 Green- house	200 SL	1	0.2		3 10 20 30 40 50 60	0.02 0.02 0.03 0.01 0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	RA-2083/98 1158-98
Italy 1994 Field	200 SL	1	0.3		60 67 70	<0.01 <0.01	<0.05 <0.05 <0.05	RA-2046/94 0223-94
Italy 1998 Field	200 SL	1	0.15		20 31 40 60	<0.01	<0.05 <0.05 <0.05 <0.05	RA-2081/98 1156-98
Italy 1999 Field	200 SL	1	0.15		20 30 40 60	<0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05	RA-2041/99 0384-99

¹ Before last treatment

Sweet peppers (foliar spray applications, (Table 98). In two greenhouse trials in Spain sprays were 0.01 kg ai/hl, 0.1 kg ai/ha, and in three in Italy 0.015 kg ai/hl, 0.15 kg ai/ha. All were with 2 applications (interval 7-14 days) of a 200 SL formulation.

In 1996 and 1997 in six greenhouse trials in Italy a 300 EC formulation of imidacloprid and cyfluthrin was sprayed twice at 0.011 kg ai/hl (interval 20 days), corresponding to 0.11 kg imidacloprid/ha.

In 1998 two bridging greenhouse trials were conducted in Italy, one with a 300 EC formulation and other with 100 EC, both at 0.14 kg imidacloprid/ha (2 applications, interval 20 days).

In Australia three protected trials were conducted with application rates at the recommended label rate of 0.05 kg ai/ha as well as at double and fourfold rates. The foliage was sprayed eight times at intervals of 14-16 days. Only imidacloprid was determined.

In Italy and Spain six field trials were conducted with 2 applications (interval 7-15 days) of a 200 SL formulation. In one trial the concentration was 0.01 kg ai/hl, corresponding to 0.15 kg ai/ha, and in the other five 0.01 kg ai/hl, 0.1 kg ai/ha.

In Brazil two trials were conducted with 3 applications (interval 14-15 days) of a 70 WG formulation at either the recommended or a double rate (0.14 or 0.28 kg ai/ha).

Table 98. Residues in sweet peppers after spray applications.

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Foliar spray								
Italy 1994 G	200 SL	2	0.15	0.015	0 ¹ 0 3 7 10 14	0.06 0.11	0.09 0.13 0.16 <u>0.15</u> 0.1 0.14	RA-2051/94 0228-94
Spain 1993 G	200 SL	2	0.1	0.01	0 3 7	 0.12	0.24 0.21 <u>0.26</u>	RA-2059/92 0111-92
Spain 1993 G	200 SL	2	0.1	0.01	0 3 7	 0.21	0.51 <u>0.48</u> 0.33	RA-2059/92 0113-92
Italy 1995 G	200 SL	2	0.15	0.015	0 ¹ 0 1 3 7 14	0.04 0.15	0.09 0.32 0.32 0.23 <u>0.22</u> 0.2	RA-2118/95 0152-95

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Italy 1995 G	200 SL	2	0.15	0.015	0 ¹ 0 1 3 7 14	0.09 0.16	0.21 0.31 0.34 0.33 <u>0.24</u> 0.23	RA-2118/95 0640-95
Italy 1996 G	300 EC	2	0.11	0.011	0 ¹ 0 1 3 7	0.02	0.05 0.13 0.14 0.09 <u>0.1</u>	RA-2053/96 0041-96
Italy 1996 G	300 EC	2	0.11	0.011	0 3	0.06	0.11 <u>0.1</u>	RA-2053/96 0319-96
Italy 1996 G	300 EC	2	0.11	0.011	0 ¹ 0 1 3 7	0.08	0.06 0.19 0.09 0.11 <u>0.12</u>	RA-2053/96 0520-96
Italy 1996 G	300 EC	2	0.11	0.011	0 3	0.13	0.14 <u>0.21</u>	RA-2053/96 0521-96
Italy 1997 G	300 EC	2	0.11	0.011	0 ¹ 0 1 3 7	0.04 0.08	0.09 0.17 0.18 0.13 <u>0.15</u>	RA-2058/97 0058-97
Italy 1997 G	300 EC	2	0.11	0.011	0 ¹ 3	0.06	0.09 <u>0.11</u>	RA-2058/97 0059-97
Italy 1998 G	300 EC	2	0.14	0.011	0 3 7	0.11 0.08	0.15 0.14 <u>0.11</u>	RA-2002/98 1214-98
Italy 1998 G	100 EC	2	0.14	0.011	0 3 7	0.12 0.1	0.17 0.15 <u>0.17</u>	RA-2002/98 1215-98
Australia 1990 UP	200 SL	8	0.05	0.027- 0.03	0 1 3 5 7 10 14	0.19 0.06 0.06 0.04 0.01 0.04 0.01		12/90 AUS-12-90-A

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Australia 1990 UP	200 SL	8	0.1	0.053- 0.06	0 1 3 5 7 10 14	0.69 0.2 0.13 0.06 0.07 0.05 0.03		12/90 AUS-12-90-B
Australia 1990 UP	200 SL	8	0.2	0.11-0.12	0 1 3 5 7 10 14	1.3 0.53 0.57 0.23 0.09 0.09 0.17		12/90 AUS-12-90-C
Spain 1991 F	200 SL	2	0.15	0.01	control 0 3 7 10	0.06 0.39 0.23 0.11 0.11	0.08 0.42 0.37 <u>0.27</u> 0.23	RA-2031/91 0487-91
Italy 1991 F	200 SL	2	0.1	0.01	0 3 7 10	0.01	0.1 <u><0.05</u> <u><0.05</u> <u><0.05</u>	RA-2030/91 0277-91
Italy 1992 F	200 SL	2	0.1	0.01	0 ¹ 0 3 7 10	0.01	<0.05 0.1 <u>0.07</u> <u><0.05</u> <u><0.05</u>	RA-2053/92 0281-92
Spain 1989 F	200 SL	2	0.1	0.01	0 3 7 14	0.10 0.06 0.06 0.05	0.12 <u>0.09</u> 0.07 0.09	0128-89
Spain 1989 F	200 SL	2	0.1	0.01	control 0 3 7 14	0.01 0.07 0.05 0.03 0.03	0.07 <u>0.07</u> <u><0.05</u> <u><0.05</u>	0129-89
Spain 1992 F	200 SL	2	0.1	0.01	0 ¹ 0 3 7 9	0.02	0.06 0.43 <u>0.15</u> 0.12 0.09	RA-2059/92 0110-92
Brazil 1995 F	70 WG	3	0.14	0.028	7	<0.01	<0.05	BRA I-D1-611/95 BRA-I-D1-611-95-A
Brazil 1995 F	70 WG	3	0.28	0.056	7	0.02	<0.05	BRA I-D1-611/95 BRA-I-D1-611-95-B

F/G/UP: Field/Greenhouse/Under plastic

¹ Before last treatment

Peppers (various application methods, Table 99). In four trials with drip irrigation application in greenhouses in The Netherlands a 70 WG formulation was applied to the base of each plant with automatic drip irrigators (10 mg imidacloprid/10 ml water, 0.2-0.32 kg ai/ha, in accordance with GAP in The Netherlands). The crop was grown on rock wool.

In two southern European greenhouse trials (Italy and Portugal) 0.2 kg ai/ha imidacloprid was applied with irrigation water and the plants were grown in soil.

In the USA twenty-five field trials were conducted on sweet and hot peppers. In nine trials a soil drench to the base of the plants about two weeks after transplanting (0.025 g ai/plant, 0.41-0.67 kg ai/ha) was followed by two foliar sprays of 0.12 kg ai/ha. The other sixteen trials were bridging trials for comparison of residues from various soil applications and formulations. In four parallel trials soil drench applications were made about two weeks after planting using a 240 SC formulation (0.025 g ai/plant, 0.41-0.57 kg ai/ha).

The other US bridging trials were to compare the residues from the 240 SC and 2.5 GR formulations in sweet and hot peppers. Sidedress applications were made in six trials two weeks after transplanting (0.03 g ai/m row, equivalent to 0.56-0.59 kg ai/ha), and six in-furrow at planting at the same rates. At harvest the total residue in sweet peppers (4 trials) was below the LOD of 0.05 mg/kg for both formulations.

Table 99. Residues in sweet and hot peppers after various applications.

Country Year F/GF/G	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Drip irrigation, Sweet pepper									
Nether- lands 1994 G	70 WG	1	0.2 ¹	0.1	fruit	1	<0.01	<0.05	RA-2096/94 0299-94
						3	<0.01	<0.05	
						5	0.04	0.07	
						7	0.06	0.08	
						14	0.09	<u>0.17</u>	
						21	0.07	0.11	
Nether- lands 1994 G	70 WG	1	0.31 ¹	0.1	fruit	1	<0.01	<0.05	RA-2096/94 0300-94
						3	0.03	<0.05	
						5	0.03	<0.05	
						7	0.07	0.09	
						14	0.08	0.11	
						21	0.10	<u>0.16</u>	
Nether- lands 1996 G	70 WG	1	0.32 ²	0.098	fruit	14	0.21	<u>0.27</u>	RA-2170/96 0813-96
						21	0.17	0.26	
						28	0.08	0.15	
						35	0.02	0.05	
						42	<0.01	<0.05	
						49	<0.01	<0.05	
						56	<0.01	<0.05	
						63	<0.01	<0.05	

Country Year F/GF/G	Application				Sample	PHI (days)	Residue, mg/kg		Report no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	Study no.
Nether- lands 1996 G	70 WG	1	0.31 ²	0.098	fruit	14 21 28 35 42 49 56 63	0.19 0.17 0.13 0.05 0.04 0.02 0.01 0.01	<u>0.24</u> 0.24 0.2 0.1 0.08 0.05 0.05 <0.05	RA-2170/96 0814-96
Italy 1995 G	200 SL	1	0.2	0.015	fruit	3 10 20 30 40 50 60	<0.01 <0.01 <0.01 <0.01 0.01 0.01 0.01	<u><0.05</u> <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	RA-2084/98 1228-98
Portugal 1995 G	200 SL	1	0.2	0.015	fruit	3 10 20 30 40 50 60	0.01 0.02 0.02 0.02 0.02 0.01 0.01	<u><0.05</u> <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	RA-2084/98 1448-98
Soil drench and spray application, Sweet pepper									
USA, CA 1992 F	240 SC	1 2	0.56 ³ 0.12 ⁴	0.021 0.13	fruit	0 3 7		0.25 0.22 0.13	105016 457-N3169-92D
USA, IN 1992 F	240 SC	1 2	0.49 ³ 0.12 ⁴	0.021 0.15	fruit	0 3 7		0.19 0.18 0.13	105016 HIN-N3173-92D
USA, GA 1992 F	240 SC	1 2	0.46 ³ 0.12 ⁴	0.021 0.053	fruit	0 3 7		0.34 0.19 0.19	105016 TGA-N3218-92D
Soil drench and spray application, Hot pepper									
USA, NM 1992 F	240 SC	1 1 1	0.41 ³ 0.12 ⁴ 0.12 ⁴	0.021 0.69 0.11	fruit	0 3 7		0.76 0.32 0.32	105016 353-N3168-92D
USA, NJ 1992 F	240 SC	1 2	0.54 ³ 0.12 ⁴	0.021 0.13	fruit	0 3 7		0.44 0.2 0.16	105016 756-N3170-92D
USA, MS 1992 F	240 SC	1 1 1	0.65 ³ 0.12 ⁴ 0.12 ⁴	0.018 0.15 0.16	fruit	0 7		0.36 0.25	105016 BMS-N3171- 92D
USA, CA 1992 F	240 SC	1 2	0.46 ³ 0.12 ⁴	0.024 0.14	fruit	0 3 7		0.17 0.17 0.17	105016 FCA-N3172-92D
USA, KS 1992 F	240 SC	1 2	0.6 ³ 0.12 ⁴	0.021 0.13	fruit	0 3 7		0.96 0.29 0.3	105016 STF-N3174-92D

Country Year F/GF/G	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, FL 1992 F	240 SC	1 2	0.67 ³ 0.12 ⁴	0.024 0.13	fruit	0 3 7		0.37 0.39 0.24	105016 VBL-N3260-92D
<i>Soil drench application, Sweet pepper</i>									
USA, IN 1992 F	240 SC	1	0.49 ³	0.021	fruit	54		<u>0.24</u>	105016 HIN-N3177-92H
USA, GA 1992 F	240 SC	1	0.45 ³	0.021	fruit	60		< <u>0.05</u>	105016 TGA-N3219-92H
<i>Soil drench application, Hot pepper</i>									
USA, CA 1992 F	240 SC	1	0.41 ³	0.024	fruit	55		<u>0.06</u>	105016 FCA-N3176-92H
USA, KS 1992 F	240 SC	1	0.57 ³	0.021	fruit	82		0.55	105016 STF-N3178-92H
<i>Sidedress application (0.03 g ai/m row), Sweet pepper</i>									
USA, IN 1992 F	240 SC	1	0.56	1.2	fruit	54		0.1	105016 HIN-N3190-92H
USA, IN 1992 F	2.5 GR	1	0.56		fruit	55		0.1	105016 HIN-N3187-92H
USA, GA 1992 F	240 SC	1	0.59	0.98	fruit	59		<0.05	105016 TGA-N3223-92H
USA, GA 1992 F	2.5 GR	1	0.59		fruit	59		<0.05	105016 TGA-N3222-92H
<i>Sidedress application (0.03 g ai/m row), Hot pepper</i>									
USA, CA 1992 F	240 SC	1	0.59	1.2	fruit	60		0.35	105016 FCA-N3189-92H
USA, CA 1992 F	2.5 GR	1	0.59		fruit	60		0.27	105016 FCA-N3186-92H
<i>In-furrow application (0.03 g ai/m row), Sweet pepper</i>									
USA, IN 1994 F	240 SC	1	0.56	1.2	fruit	123		<0.05	105016 HIN-N3184-92H
USA, IN 1992 F	2.5 GR	1	0.56		fruit	123		<0.05	105016 HIN-N3181-92H

Country Year F/GF/G	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, GA 1992 F	240 SC	1	0.59	1.1	fruit	92		<u><0.05</u>	105016 TGA-N3221-92H
USA, GA 1992 F	2.5 GR	1	0.59		fruit	92		<u><0.05</u>	105016 TGA-N3220-92H
<i>In-furrow application (0.03 g ai/m row), Hot pepper</i>									
USA, CA 1992	240 SC	1	0.59	1.2	fruit	123		<u>0.2</u>	105016 FCA-N3183-92H
USA, CA 1992	2.5 GR	1	0.59		fruit	123		<u>0.13</u>	105016 FCA-N3180-92H

F/G: Field/Greenhouse

¹10 mg ai/plant

²9.8 mg ai/plant

³ Soil drench application: 0.025 g ai/plant

⁴Foliar spray

Tomatoes (foliar spray treatment, Table 100). Eight greenhouse trials were conducted in northern and southern Europe with 2 applications (interval 7–14 days) of a 200 SL formulation. The spray concentrations were 0.01-0.015 kg ai/hl, corresponding to 0.15–0.16 kg ai/ha. Eight further greenhouse trials were with a 300 EC formulation of imidacloprid and cyfluthrin (2 x 0.11 kg ai/ha, interval 20 days, 0.011 kg ai/hl).

In Italy and Spain nine field trials were with two spray applications (interval 7–15 days) of a 200 SL formulation at a concentration of 0.01 kg ai/hl, corresponding to 0.1-0.15 kg ai/ha.

In Brazil two field trials were conducted with 3 applications (interval 14 days) of a 70 WG formulation at the recommended rate or a double rate (0.14 or 0.28 kg ai/ha).

Table 100. Residues in tomatoes after spray applications.

Country Year F/G	Application					Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	PHI (days)	Imidacloprid	Total	
Italy 1994 G	200 SL	2	0.15	0.015	0 ¹ 0 3 5 7 10	<0.01 0.05	0.09 0.1 0.08 0.1 <u>0.08</u> 0.08	RA-2046/94 0226-94
Italy 1995 G	200 SL	2	0.15	0.015	0 ¹ 0 1 3 7 14	0.03 0.06	0.05 0.11 0.09 0.08 0.07 <u>0.1</u>	RA-2121/95 0153-95
Italy 1995 G	200 SL	2	0.16	0.015	0 ¹ 0 1 3 7 14	0.13 0.2	0.12 0.27 0.22 0.2 0.25 <u>0.29</u>	RA-2121/95 0650-95
Nether- lands 2001 G	200 SL	2	0.15	0.01	0 ¹ 0 3 7 10	 0.06 0.05 0.05	<0.05 0.11 0.08 <u>0.08</u> 0.08	RA-2079/01 0185-01
France 2001 G	200 SL	2	0.15	0.01	0 3 7	0.06 0.06 0.04	0.13 0.07 <u>0.07</u>	RA-2079/01 0186-01
Italy 2001 G	200 SL	2	0.15	0.01	0 3 7	0.11 0.09 0.08	0.16 0.14 <u>0.13</u>	RA-2079/01 0346-01
Spain 2001 G	200 SL	2	0.15	0.01	0 3 7	0.12 0.1 0.1	0.21 0.15 <u>0.07</u>	RA-2079/01 0347-01
Portugal 2001 G	200 SL	2	0.13 0.15	0.01	0 3 7	0.05 0.06 0.03	0.07 0.06 <u>0.06</u>	RA-2079/01 0348-01
Italy 1995 G	300 EC	2	0.11	0.011	0 ¹ 0 1 3 7	 0.02 0.04	<0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2133/95 0196-95
Italy 1995 G	300 EC	2	0.11	0.011	0 ¹ 0 1 3 7	 0.02 0.07	0.05 0.07 0.11 <u>0.1</u> 0.09	RA-2133/95 0659-95

Country Year F/G	Application					Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	PHI (days)	Imidacloprid	Total	
Italy 1995 G	300 EC	2	0.11	0.011	0 3	0.03	<0.05 <u><0.05</u>	RA-2133/95 0660-95
Italy 1995 G	300 EC	2	0.11	0.011	0 1 3	0.1 0.07	0.08 0.08 <u>0.06</u>	RA-2133/95 0661-95
Italy 1996 G	300 EC	2	0.11	0.011	0 1 3 7	0.09	0.24 0.21 0.17 <u>0.18</u>	RA-2054/96 0522-96
Italy 1996 G	300 EC	2	0.11	0.011	0 3	0.05	0.08 <u>0.09</u>	RA-2054/96 0523-96
Italy 1997 G	300 EC	2	0.11	0.011	0 ¹ 0 1 3 7	0.02 0.05	0.07 0.13 0.12 <u>0.12</u> 0.1	RA-2012/97 0114-97
Germany 1997 G	300 EC	2	0.11	0.011	0 3	0.04	0.13 <u>0.08</u>	RA-2012/97 0115-97
Italy 1992 F	200 SL	2	0.1	0.01	0 ¹ 0 3 7 10	<0.01	<0.05 0.07 <u><0.05</u> <0.05 <0.05	RA-2053/92 0266-92
Italy 1992 F	200 SL	2	0.1	0.01	0 ¹ 0 3 7 10	<0.01	<0.05 0.07 <u><0.05</u> <0.05 <0.05	RA-2053/92 0267-92
Italy 1993 F	200 SL	2	0.1	0.01	0 ¹ 0 3 5 7 10	<0.01 <0.01	<0.05 0.08 <0.05 <u>0.05</u> <0.05 <0.05	RA-2032/93 0019-93
Spain 1990 F	200 SL	2	0.1	0.01	0 3 7 14	0.02	0.06 <u>0.07</u> 0.05 0.06	0218-90
Spain 1990 F	200 SL	2	0.1	0.01	0 3 7 10	0.02	0.12 <u><0.05</u> <0.05 <0.05	0219-90

Country Year F/G	Application					Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl	PHI (days)	Imidacloprid	Total	
Spain 1991 F	200 SL	2	0.15	0.01	0 3 7 10	0.04	0.29 0.23 0.11 <u>0.17</u>	RA-2037/91 0488-91
Spain 1991 F	200 SL	2	0.1– 0.15	0.01	0 3 7 10	<0.01	0.08 <0.05 <u><0.05</u> <0.05	RA-2037/91 0489-91
Spain 1992 F	200 SL	2	0.15	0.01	0 ¹ 0 3 7	0.03	<0.05 0.1 0.08 <u>0.09</u>	RA-2059/92 0107-92
Spain 1992 F	200 SL	2	0.15	0.01	0 3 7	0.04	0.15 0.11 <u>0.14</u>	RA-2059/92 0108-92
Brazil 1995	70 WG	3	0.14	0.014	7	<0.01	<0.05	BRA I-D1-602/95 BRA-I-D1-607-95-A
Brazil 1995	70 WG	3	0.28	0.028	7	<0.01	<0.05	BRA I-D1-602/95 BRA-I-D1-607-95-B

F/G: Field/Greenhouse

¹ Before last treatment

Tomatoes (drip irrigation, Table 101). In six indoor trials in The Netherlands automatic drip applications of a 70 WG formulation were made to the base of each plant (10 mg imidacloprid in 10 ml water, equivalent to 0.2-0.29 kg ai/ha) in accordance with GAP in The Netherlands. The tomatoes were grown on rock wool.

In two greenhouse trials in Italy and Portugal 0.2 kg ai/ha imidacloprid was applied with the irrigation water to tomato plants grown in soil.

Table 101. Residues in tomatoes after drip irrigation (Europe).

Country Year	Application				PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Nether- lands 1994	70 WG	1	0.24 ¹	0.098	1 3 5 7 14 21	<0.01 0.02 0.03 0.04 0.05 0.05	<0.05 <0.05 <0.05 0.05 0.07 <u>0.08</u>	RA-2096/94 0297-94
	70 WG	1	0.28 ¹	0.098	1 3 5 7 14 21	<0.01 <0.01 0.02 0.02 0.04 0.04	<0.05 <0.05 <0.05 <0.05 0.05 <u>0.09</u>	RA-2096/94 0298-94
Nether- lands 1995	70 WG	1	0.23 ¹	0.098	7 14 21 28 35	0.01 0.02 0.02 0.02 0.01	<0.05 <0.05 <0.05 <0.05 <u>0.05</u>	RA-2148/95 0204-95
	70 WG	1	0.25 ¹	0.098	7 14 21 28 35	0.04 0.06 0.09 0.09 0.09	0.05 0.09 0.12 <u>0.15</u> 0.14	RA-2148/95 0688-95
	70 WG	1	0.29 ¹	0.098	7 14 21 28 35	0.03 0.07 0.09 0.09 0.08	<0.05 0.08 0.12 <u>0.16</u> 0.14	RA-2148/95 0689-95
	70 WG	1	0.23 ¹	0.098	7 14 21 28 35	0.03 0.07 0.08 <0.01 0.06	<0.05 0.06 0.11 <0.05 <u>0.14</u>	RA-2148/95 0690-95
Italy 1998	200 SL	1	0.2		3 10 20 30 40 50 60	<0.01 <0.01 <0.01 <0.01 <0.01 0.01 0.01	< <u>0.05</u> <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	RA-2083/98 1157-98
Portugal 1998	200 SL	1	0.2		3 10 20 30 40 50 60	<0.01 <0.01 <0.01 0.01 0.01 <0.01 <0.01	< <u>0.05</u> <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	RA-2083/98 1449-98

¹10 mg ai/plant

Tomatoes (US and Canadian trials, Table 102). In the USA thirty field trials were conducted to determine the total residue of imidacloprid on tomatoes and cherry tomatoes to support the maximum soil and foliar application patterns.

In nine of the trials there were three applications: the first a soil drench to the base of the plants about two weeks after transplanting, 0.025 g ai/plant (0.56 kg ai/ha) and two foliar sprays at 5 ($\pm$ 2) day intervals at 0.12 kg ai/ha, 0.13-0.15 kg ai/hl and 77-94 l/ha.

The other twenty one trials were with soil applications. In five drench applications were made about two weeks after planting with a 240 SC formulation at 0.025 g ai/m, 0.56 kg ai/ha.

The remaining 16 were bridging trials to compare total residues from the 240 SC and 2.5 GR formulations. In-furrow applications were made at planting (0.03 g ai/m/row, 0.56 kg ai/ha). Sidedress applications were made two weeks later or when the plants reached transplant size if grown from seed.

In Canada two field trials were conducted on tomatoes and two on cherry tomatoes. In all trials the plants were drenched two weeks after transplanting with a 240 SC formulation at 0.025 g ai/plant, 0.5 kg ai/ha. This was followed in two trials by two foliar sprayings at 0.12 kg ai/ha.

Table 102. Residues in tomatoes from field applications in the USA and Canada.

Crop	Location Year		Application				PHI (days)	Total residue, mg/kg	Report no. Study no.
			Form.	No.	kg ai/ha	kg ai/hl			
Soil drench and spray applications									
Tomato	USA, 1992	OH	240 SC	1 2	0.56 ¹ 0.12 ²	0.018 0.15	0 3 7	0.18 0.19 0.18	105015 854-N3143-92D
Tomato	USA, 1992	MI	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.15	0 3 7	0.06 <0.05 <0.05	105015 855-N3144-92D
Tomato	USA, 1992	MS	240 SC	1 2	0.56 ¹ 0.12 ²	0.016 0.16	0 3 7	0.26 0.19 0.22	105015 BMS-N3145-92D
Tomato	USA, 1992	CA	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.14	0 3 7	0.17 0.13 0.09	105015 FCA-N3146-92D
Tomato	USA, 1992	IN	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.15	0 3 7	0.14 0.06 0.06	105015 HIN-N3147-92D
Tomato	USA, 1992	KS	240 SC	1 2	0.56 ¹ 0.12 ²	0.03 0.13	0 3 7	<0.05 <0.05 <0.05	105015 STF-N3148-92D

Crop	Location Year	Application				PHI (days)	Total residue, mg/kg	Report no. Study no.
		Form.	No.	kg ai/ha	kg ai/hl			
Tomato	Canada 1995	240 SC	1 2	0.5 ¹ 0.12 ²	0.02 0.025	0 0 0 0	<0.1 <0.1 <0.1 0.1	107320 510-N3004-95H-B
Cherry tomato	USA, NJ 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.02 0.13	0 3 7	0.21 0.16 0.13	105015 756-N3142-92D
Cherry tomato	USA, GA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.025 0.05	0 3 7	0.11 0.15 0.1	105015 TGA-N3212-92D
Cherry tomato	USA, FL 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.025 0.13	0 3 7	0.45 0.48 0.35	105015 VBL-N3254-92D
Cherry tomato	Canada 1995	240 SC	1 2	0.5 ¹ 0.12 ²	0.02 0.025	0 0 0 0 3 3 3 3 8 8 8 8 15 15 15 15	0.33 0.28 0.28 0.19 0.28 0.2 0.17 0.16 0.18 0.16 0.15 0.14 0.2 0.21 0.18 0.18	107320 510-N3003-95D-B (4 replicate field samples)
Soil drench application (0.025 g ai/plant)								
Tomato	USA, CA 1992	240 SC	1	0.56	0.02	56 56	0.06 <0.05	105015 FCA-N3150-92H
Tomato	USA, IN 1992	240 SC	1	0.56	0.02	78 78	<0.05 <0.05	105015 HIN-N3151-92
Tomato	USA, KS 1992	240 SC	1	0.56	0.03	98 98	<0.05 <0.05	105015 STF-N3152-92H
Tomato	Canada 1995	240 SC	1	0.5	0.02	83 83 83 83	<0.1 <0.1 <0.1 <0.1	107320 510-N3004-95H-A
Cherry tomato	USA, GA 1992	240 SC	1	0.56	0.025	64 64	<0.05 <0.05	105015 TGA-N3213-92H
Cherry tomato	USA, FL 1992	240 SC	1	0.56	0.025	58 58	0.26 0.19	105015 VBL-N3255-92H

Crop	Location Year	Application				PHI (days)	Total residue, mg/kg	Report no. Study no.
		Form.	No.	kg ai/ha	kg ai/hl			
Cherry tomato	Canada 1995	240 SC	1	0.5	0.02	54 57 62 69	<0.1 <0.1 <0.1 <0.1	107320 510-N3003-95D-A
In-furrow application (0.03 g ai/m row)								
Tomato	USA, CA 1992	240 SC	1	0.56	1.1	91 91	0.13 0.18	105015 FCA-N3157-92H
Tomato	USA, CA 1992	2.5 GR	1	0.56		91 91	0.2 0.11	105015 FCA-N3154-92H
Tomato	USA, IN 1992	240 SC	1	0.56	1.1	112 112	<0.05 <0.05	105015 HIN-N3158-92H
Tomato	USA, IN 1992	2.5 GR	1	0.56		112 112	<0.05 <0.05	105015 HIN-N3155-92H
Cherry tomato	USA, GA 1992	240 SC	1	0.56	1.0	92 92	<0.05 <0.05	105015 TGA-N3215-92H
Cherry tomato	USA, GA 1992	2.5 GR	1	0.56		92 92	<0.05 <0.05	105015 TGA-N3214-92H
Cherry tomato	USA, FL 1992	240 SC	1	0.56	1.0	84 84	0.08 0.11	105015 VBL-N3257-92H
Cherry tomato	USA, FL 1992	2.5 GR	1	0.56		84 84	0.1 0.07	105015 VBL-N3256-92H
Sidedress application (0.03 g ai/m row)								
Tomato	USA, CA 1992	240 SC	1	0.56	1.0	44 44	0.22 0.19	105015 FCA-N3163-92H
Tomato	USA, CA 1992	2.5 GR	1	0.56		44 44	0.14 0.19	105015 FCA-N3160-92H
Tomato	USA, IN 1992	240 SC	1	0.56	1.1	78 78	0.06 0.05	105015 HIN-N3164-92H
Tomato	USA, IN 1992	2.5 GR	1	0.56		79 79	<0.05 <0.05	105015 HIN-N3161-92H
Cherry tomato	USA, GA 1992	240 SC	1	0.56	0.93	59 59	<0.05 <0.05	105015 TGA-N3217-92H
Cherry tomato	USA, GA 1992	2.5 GR	1	0.56		59 59	<0.05 <0.05	105015 TGA-N3216-92H
Cherry tomato	USA, FL 1992	240 SC	1	0.56	1.0	58 58	0.19 0.1	105015 VBL-N3259-92H
Cherry tomato	USA, FL 1992	2.5 GR	1	0.56		58 58	0.16 0.26	105015 VBL-N3258-92H

¹ Soil drench application: 0.025 g ai/plant

² Foliar spray (Silwet L-77 was added as a spray adjuvant)

Sweet corn (corn-on-the-cob, Table 103). In eight trials in Australia, six were bridging trials to compare residues from seed treatments with the 350 FS and 600 FS formulations. In three trials the recommended rate was applied (0.26 kg ai/100 kg seed) and in the other three seed was treated at 0.35 kg ai/100 kg. In the remaining two twice the label rate was applied.

Table 103. Residues in sweet corn after seed treatment in Australia (immature and mature cob, husks left intact).

Location Year	Application			Sample	PHI (days)	Total residue (mg/kg)	Report Study no. <i>no.</i>
	Form.	No.	kg ai/100 kg seed				
NSW 1994	600 FS	1	0.26	forage	28	0.34	TAB71-94 AUS-TAB71-94-A1
				cob, immature cob, mature	69 79	<0.01 <0.01	
				forage ¹	28	2.9	
NSW 1994	600 FS	1	0.35	forage	28	1.01	TAB71-94 AUS-TAB71-94-A2
				cob, immature cob, mature	69 79	<0.01 <0.01	
				forage ¹	28	8.5	
NSW 1994	600 FS	1	0.52	forage	28	1.21	TAB71-94 AUS-TAB71-94-A3
				cob, immature cob, mature	69 79	<0.01 <0.01	
				forage ¹	28	10.1	
NSW 1994	350 FS	1	0.26	forage	28	0.54	TAB71-94 AUS-TAB71-94-B1
				cob, immature cob, mature	69 79	<0.01 <0.01	
				forage ¹	28	4.5	
NSW 1994	350 FS	1	0.35	forage	28	0.23	TAB71-94 AUS-TAB71-94-B2
				cob, immature cob, mature	69 79	<0.01 <0.01	
				forage ¹	28	1.9	
NSW 1994	350 FS	1	0.52	forage	28	0.62	TAB71-94 AUS-TAB71-94-B3
				cob, immature cob, mature	69 79	<0.01 <0.01	
				forage ¹	28	5.2	

Location Year	Application			Sample	PHI (days)	Total residue (mg/kg)	Report Study no.
	Form.	No.	kg ai/100 kg seed				
QLD 1998	600 FS	1	0.26	cob without husks	82	<0.02 <0.02	AMD 087/98 AUS -AMD087-A
QLD 1998	600 FS	1	0.35	cob without husks	82	<0.02 <0.02	AMD 087/98 AUS -AMD087-B

¹ Calculated on dry weight basis, 11.9% moisture content.

Lettuce and endive (European trials, Tables 104 and 105). In seven field trials, 1989–1995, in southern Europe, one on endive and six on lettuce, (two trials in Italy and five in Spain) crop leaves were sprayed twice with a 200 SL formulation (interval 7–14 days) at 0.01 kg ai/hl, 0.1 kg ai/ha, except in one trial in Spain with only one spray.

In 2001 two field trials on lettuce in Greece and Italy were carried out using 2 spray applications of a 200 SL formulation (0.013 kg ai/hl, 0.1 kg ai/ha). Heads were sampled immediately before and 0, 3, 7 and 14 days after the second spray.

In eight greenhouse trials in France (6) and Germany (2) a 200 SL formulation was applied as a single drench at 2.4 mg ai/plant, corresponding in four of the trials to 0.012 kg ai/hl, at a water rate of 20 ml/plant.

Nine field trials on lettuce and one on endive were conducted with a 200 SL formulation in northern France. In the trials on lettuce a single drench was applied at 2.4 mg ai/plant, 20 ml water/plant, corresponding in seven of the trials to 0.012 kg ai/hl. No information on the concentration was given for the two remaining trials. A single application was made in the endive trial at 0.32 kg ai/ha, 4.8 mg ai/plant.

Of ten field trials in southern Europe, two on endive and eight on lettuce, nine were carried out in France and one in Italy. In all trials single applications of the 200 SL formulation were made either at planting or a few weeks later. In the two endive trials the drench was at 0.216 kg ai/ha, 4.8 mg ai/plant. In seven of the lettuce trials in France a rate of 2.4 mg ai/plant was used, corresponding in two trials to 0.11 kg ai/ha, and in five to 0.012 kg ai/hl. In the trial in Italy a single drip irrigation application was made at 0.3 kg ai/ha.

Table 104. Residues in endive heads, European field trials.

Country Year	Application				PHI (days)	Residue, mg/kg		Report Study	no. no.
	Form.	No	kg ai/ha	kg ai/hl		Imidacloprid	Total		
Foliar spray treatment									
Italy 1995	200 SL	2	0.1	0.01	0 ¹	0.03	0.09	RA-2058/94 0238-94	
					0		1.9		
					3		0.7		
					7		0.55		
					10		0.38		
					0.28				

Country Year	Application				PHI (days)	Residue, mg/kg		Report Study	no. no.
	Form.	No	kg ai/ha	kg ai/hl		Imidacloprid	Total		
<i>Drench application</i>									
France, North 1993	200 SL	1	0.32 ²		56	0.01	<0.05	RA-2028/93 0387-93	
France, South 1993	200 SL	1	0.22 ²		56	0.09	0.36	RA-2028/93 0388-93	
France, South 1993	200 SL	1	0.22 ²		56	0.07	0.21	RA-2028/93 0389-93	

¹ Before last treatment² Drench application: 4.8 mg ai/plant

Table 105. Residues in lettuce, European trials.

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Foliar spray treatment									
Italy 1994 F	200 SL	2	0.1	0.01	head	0 ¹ 0 3 7 10 20	<0.01 0.04	0.09 1.4 <u>0.99</u> 0.4 0.23 0.07	RA-2058/94 0239-94
Spain 1989 F	200 SL	1	0.1	0.01	leaf	0 3 7 14	3.8 0.74 0.17 0.05	3.4 1.5 0.65 0.35	0131-89
Spain 1992 F	200 SL	2	0.1	0.01	head	0 ¹ 0 3 7 14	 0.04	0.06 0.27 <u>0.87</u> 0.21 0.17	RA-2013/92 0118-92
Spain 1992 F	200 SL	2	0.1	0.01	head	-7 ¹ 0 3 7 14	 0.16	1.5 1.4 0.62 <u>0.69</u> 0.64	RA-2013/92 0119-92
Spain 1992 F	200 SL	2	0.1	0.01	head	0 3 7	0.38 0.09	1.4 <u>0.98</u> 0.3	RA-2013/92 0120-92
Spain 1994 F	200 SL	2	0.1	0.01	head	0 ¹ 0 3 5 7	0.02 0.39	0.1 1.7 <u>0.88</u> 0.69 0.52	RA-2058/94 0685-94

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Greece 2001 F	200 SL	2	0.1	0.013	head	0 ¹ 0 3 7 14	1.8 0.11	0.26 2.4 <u>1.2</u> 0.26 0.05	RA-2045/01 0115-01
Italy 2001 F	200 SL	2	0.1	0.013	head	0 ¹ 0 3 7 14	0.51 0.08	0.17 1.0 <u>0.9</u> 0.72 0.34	RA-2045/01 0349-01
<i>Drench application</i>									
France, North 1996, G	200 SL	1	2.4 mg ai/plant	0.012	head	21 35 42	 0.09	1.1 0.4 0.27	RA-2039/96 0320-96
France, South 1996, G	200 SL	1	2.4 mg ai/plant		head	21 35 42	 0.04	0.5 0.19 0.13	RA-2040/96 0322-96
France, North 1996, G	200 SL	1	2.4 mg ai/plant	0.012	head	21 35 42 59	 0.08 0.05	0.79 0.31 0.28 0.15	RA-2039/96 0483-96
France, South 1996, G	200 SL	1	2.4 mg ai/plant		head	21 35 42	 0.05	0.48 0.2 0.14	RA-2040/96 0486-96
France, South 1996, G	200 SL	1	2.4 mg ai/plant		head	21 35 42	 0.04	0.33 0.16 0.09	RA-2040/96 0487-96
France, South 1996, G	200 SL	1	2.4 mg ai/plant		head	21 35 42	 0.11	1.5 0.63 0.28	RA-2040/96 0488-96
Germany 1996, G	200 SL	1	2.4 mg ai/plant	0.012	whole plant ²	25 39 46 67	 0.04	0.63 0.59 0.29 0.12	RA-2039/96 0484-96
Germany 1996, G	200 SL	1	2.4 mg ai/plant	0.012	whole plant ² head	21 35 44 54	 0.09 0.05	1.3 0.42 0.22 0.13	RA-2039/96 0485-96
France, North 1992, F	200 SL	1	0.26 ⁴	0.012	head	21 42	 0.02	0.96 0.05	RA-2045/92 0450-92
France, North 1992, F	200 SL	1	0.26 ⁴	0.012	head	21 42	 0.03	0.95 0.19	RA-2045/92 0452-92

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
France, North 1992, F	200 SL	1	0.26 ⁴	0.012	head	21 42	0.02	0.25 0.06	RA-2045/92 0489-92
France, North 1992, F	200 SL	1	0.26 ⁴	0.012	head	21 42	0.01	0.16 <0.05	RA-2045/92 0454-92
France, North 1993, F	200 SL	1	0.28 ⁴		head	42	0.03	0.06	RA-2028/93 0096-93
France, North 1993, F	200 SL	1	0.28 ⁴		head	42	<0.01	0.09	RA-2028/93 0384-93
France, North 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42 49	0.39 0.03 0.02	2.4 0.25 0.12	RA-2120/95 0120-95
France, North 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42	0.06 0.01	1.3 <0.05	RA-2120/95 0643-95
France, North 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42	0.21 0.03	2.0 0.15	RA-2120/95 0649-95
France, South 1993, F	200 SL	1	0.11 ⁴		head	42	0.05	1.0	RA-2028/93 0385-93
France, South 1993, F	200 SL	1	0.11 ⁴		head	42	0.09	0.39	RA-2028/93 0386-93
France, South 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42	0.04 0.02	0.35 0.05	RA-2120/95 0119-95
France, South 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42	0.06 0.02	0.71 0.11	RA-2120/95 0644-95
France, South 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42	0.12 0.03	0.84 0.11	RA-2120/95 0645-95
France, South 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42	0.05 0.01	0.26 <0.05	RA-2120/95 0647-95
France, South 1995, F	200 SL	1	2.4 mg ai/plant	0.012	head	21 42	0.08 0.02	0.36 0.08	RA-2120/95 0648-95

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Italy 1994, F	200 SL	1	0.3 ⁵		head	30	0.01	0.05	RA-2058/94 0240-94
						40		<0.05	
						50		<0.05	

¹ Before last treatment² Whole plant without root³ Drench application: 4.8 mg ai/plant⁴ Drench application: 2.4 mg ai/plant⁵ Drip irrigation

F/G: Field/Greenhouse

Lettuce (US trials, Tables 106 and 107). A total of 43 field trials in the USA were on head and leaf lettuce using 240 SC formulations. In 26 a soil drench was applied to the base of the plant at 0.01 g ai/plant, 56040 plants/ha, 0.56 kg ai/ha, 0.0085 kg ai/hl, followed by two foliar sprayings with adjuvant "Silwet L-77" at 0.12 kg ai/ha, equivalent to 0.05-0.16 kg ai/hl. In seven trials only single soil drenches were applied at the same rate as before, concentrations were 0.008 or 0.01 kg ai/hl. The remaining 10 trials were in-furrow and sidedress applications at 0.03 g ai/m row, 0.56 kg ai/ha. Concentrations were 1.0 or 1.1 kg ai/hl.

Table 106. Residues in head lettuce, US field trials.

Location Year	Application				Sample	PHI (days)	Total residue, mg/kg	Report Study	no. no.
	Form.	No	kg ai/ha	kg ai/hl					
Soil drench and spray application									
TX 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	0 7 14	2.1 1.7 0.8	105164 352-N3086-92D	
					head, cleaned	0 7 14	1.1 0.72 0.29		
WA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.16	head, cleaned	0 7 14	0.21 <0.05 0.15	105164 454-N3087-92D	
					head	0 7 14	0.44 0.59 0.46		
CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	0 7 14	1.4 0.67 0.26	105164 457-N3088-92D	
					head, cleaned	0 7 14	0.28 0.15 0.09		
CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	0 7 14	5.1 2.1 0.99	105164 457-N3089-92D	

Location Year	Application				Sample	PHI (days)	Total residue, mg/kg	Report Study	no. no.
	Form.	No	kg ai/ha	kg ai/hl					
					head, cleaned	0 7 14	0.68 0.58 0.28		
AZ 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	0 7 14	0.96 0.67 0.33	105164 458-N3090-92D	
					head, cleaned	0 7 14	0.11 0.06 0.08		
NJ 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	0 7 15	0.91 0.32 0.12	105164 756-N3091-92D	
					head, cleaned	0 7 15	0.11 0.07 0.06		
CO 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.14	head	0 7 14	2.6 0.6 0.27	105164 858-N3092-92D	
					head, cleaned	0 7 14	0.54 0.12 <0.05		
MS 1992	240 SC	1 1 1	0.56 ¹ 0.12 ² 0.12 ²	0.007 0.15 0.14	head	0 7	2.5 1.3	105164 BMS-N3093-92D	
					head, cleaned	0 7	1.37 0.38		
CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.14	head	0 7 14	1.2 0.57 0.52	105164 FCA-N3094-92D	
					head, cleaned	0 7 14	0.22 0.17 0.2		
IN 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	0	2.8	105164 HIN-N3095-92D-A	
					head, cleaned	0	0.49		
IN 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	7	0.73	105164 HIN-N3095-92D-B	
					head, cleaned	7	0.16		
IN 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	14	0.34	105164 HIN-N3095-92D-C	
					head, cleaned	14	0.09		
KS 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	head	0 7 14	0.69 0.31 0.12	105164 STF-N3096-92D	

Location Year	Application				Sample	PHI (days)	Total residue, mg/kg	Report Study	no. no.
	Form.	No	kg ai/ha	kg ai/hl					
					head, cleaned	0 7 14	<0.05 0.1 0.09		
GA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.052	head	0 7 14	1.2 0.85 0.99	105164 TGA-N3241-92D	
					head, cleaned	0 7 14	0.34 0.12 0.06		
Soil drench application									
IN 1992	240 SC	1	0.56 ¹	0.008	head	29 29	0.39 0.37	105164 HIN-N3099-92H	
					head, cleaned	29	0.09		
KS 1992	240 SC	1	0.56 ¹	0.008	head	40	<0.05	105164 STF-N3100-92H	
					head, cleaned	40	<0.05		
GA 1992	240 SC	1	0.56 ¹	0.008	head	94	<0.05	105164 TGA-N3242-92H	
					head, cleaned	94	<0.05		
In-furrow application (0.03 g ai/m row)									
IN 1992	240 SC	1	0.56	1.1	head	77 77	0.15 0.13	105164 HIN-N3106-92H	
					head, cleaned	77	<0.05		
IN 1992	240 SC	1	0.56	1.1	head	27 27	0.13 0.1	105164 HIN-N3112-92H	
					head, cleaned	27	0.05		
GA 1992	240 SC	1	0.56	1.0	head	133	<0.05	105164 TGA-N3244-92H	
					head, cleaned	133	<0.05		
GA 1992	240 SC	1	0.56	1.0	head	91	<0.05	105164 TGA-N3246-92H	
					head, cleaned	91	<0.05		

¹ Soil drench: 0.01 g ai/plant.

² Foliar spray: Silwet L-77 was used as a spray adjuvant.

Head, cleaned: head without wrapper leaves.

Table 107. Residues in leaf lettuce, US field trials.

Location Year	Application		Sample	PHI (days)	Total residue, mg/kg	Report no. Study no.

	Form.	No	kg ai/ha	kg ai/hl				
<i>Soil drench and spray application</i>								
TX 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	leaf	0 7 14	3.4 2.5 1.0	105164 352-N3114-92D
WA 1992	240 SC	1 1 1	0.56 ¹ 0.12 ² 0.12 ²	0.008 0.16 0.15	leaf	0 7 14	1.3 1.5 0.63	105164 454-N3115-92D
CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	leaf	0 7 14	4.0 2.3 <0.05	105164 457-N3116-92D
CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	leaf	0 7 14	8.6 2.2 1.4	105164 457-N3117-92D
AZ 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	leaf	0 7 14	1.3 0.09 0.1	105164 458-N3118-92D
NJ 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.13	leaf	0 7 14	5.4 1.2 0.38	105164 756-N3119-92D
CO 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.14	leaf	0 7 14	2.5 1.5 0.97	105164 858-N3120-92D
MS 1992	240 SC	1 1 1	0.56 ¹ 0.12 ² 0.12 ²	0.007 0.13 0.15	leaf	0 7 14	6.0 0.92 0.79	105164 BMS-N3121-92D
CA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.14	leaf	0 7 14	1.5 0.95 0.75	105164 FCA-N3122-92D
IN 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.14	leaf	0 7 14	11 0.54 0.27	105164 HIN-N3123-92D
KS 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.01 0.13	leaf	0 7 14	1.3 1.5 2.6	105164 STF-N3124-92D
GA 1992	240 SC	1 2	0.56 ¹ 0.12 ²	0.008 0.05	leaf	0 7 14	4.8 2.4 1.2	105164 TGA-N3247-92D
<i>Soil drench application</i>								
CA 1992	240 SC	1	0.56 ¹	0.008	leaf	36 36	0.57 0.68	105164 FCA-N3126-92H
IN 1992	240 SC	1	0.56 ¹	0.008	leaf	13 13	2.1 2.0	105164 HIN-N3127-92H
KS 1992	240 SC	1	0.56 ¹	0.01	leaf	43 43	0.44 0.56	105164 STF-N3128-92H

Location Year	Application				Sample	PHI (days)	Total residue, mg/kg	Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl				
GA 1992	240 SC	1	0.56 ¹	0.008	leaf	39 39	0.18 0.1	105164 TGA-N3248-92H
<i>In-furrow application (0.03 g ai/m row)</i>								
CA 1992	240 SC	1	0.56	1.1	leaf	68 68	0.6 0.8	105164 FCA-N3133-92H
IN 1992	240 SC	1	0.56	1.1	leaf	63 63	0.31 0.27	105164 HIN-N3134-92H
GA 1992	240 SC	1	0.56	1.1	leaf	85 85	0.29 0.35	105164 TGA-N3250-92H
<i>Sidedress application (0.03 g ai/m row)</i>								
CA 1992	240 SC	1	0.56	1.1	leaf	27 27	0.74 0.93	105164 FCA-N3139-92H
IN 1992	240 SC	1	0.56	1.1	leaf	12 12	0.68 0.71	105164 HIN-N3140-92H
GA 1992	240 SC	1	0.56	1.1	leaf	43	0.05	105164 TGA-N3252-92H

¹ Soil drench: 0.01 g ai/plant.

² Foliar spray: Silwet L-77 was used as a spray adjuvant.

Common bean (pods and/or immature seeds, Table 108). In Kenya two seed treatment trials were conducted (0.28 kg ai/100 kg seed) and beans with pods were harvested 50–79 days after treatment.

In 11 trials in Europe (two in France, three in Italy and six in Spain) crops were sprayed twice (interval 7-15 days) at 0.01 kg ai/hl, 0.1 kg ai/ha, and beans were harvested 0–10 days later.

In Brazil, four trials were with five foliar spray applications (intervals 9-10 days). In two the rate was 0.058 kg ai/hl, 0.18 kg ai/ha, and in the other two 0.12 kg ai/hl, 0.35 kg ai/ha).

13 trials were conducted on common and Lima beans in the USA. In the six trials on common beans the first application was a seed treatment at 0.25 kg ai/100 kg seed, the second an in-furrow spray at planting (0.022 to 1.0 kg ai/hl, 0.042 to 0.42 kg ai/ha) followed by three foliar sprays (0.006 to 0.03 kg ai/hl, 0.048 to 0.083 kg ai/ha).

In six of the seven trials on Lima beans there were four applications (five in the seventh trial) the first was an in-furrow spray at planting, 0.022 to 0.3 kg ai/hl, 0.42 kg ai/ha. The three or four further treatments were all foliar sprays with spray concentrations from 0.005 to 0.027 kg ai/hl, 0.05-0.083 kg ai/ha (intervals 6-7 days).

Table 108. Residues in beans (pods and/or immature seeds).

Country Year	Application	Sample	PHI (days)	Residue, mg/kg	Report no. Study no.

	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
<i>Common bean, seed treatment</i>									
Kenya 1997	350 FS	1	0.28 kg ai/100 kg seed		with pod	50 61 71	<0.01 <0.01 <0.01	<0.05 <0.05 <0.05	0799-97
Kenya 1997	350 FS	1	0.28 kg ai/100 kg seed		with pod	66 72 79	<0.01 <0.01 <0.01	0.13 0.10 0.07	0800-97
<i>Common bean, foliar spray application</i>									
Italy 1991	200 SL	2	0.1	0.01	with pod	0 3 7 10	<0.01	0.32 <u>0.44</u> 0.32 0.23	RA-2022/91 0272-91
Spain 1991	200 SL	2	0.1	0.01	with pod	0 3 7 10	0.01	0.49 <u>0.32</u> 0.19 0.22	RA-2023/91 0199-91
Spain 1991	200 SL	2	0.1	0.01	with pod	0 3 7 10	0.02	0.47 0.34 <u>0.39</u> 0.14	RA-2023/91 0200-91
Spain 1991	200 SL	2	0.1	0.01	with pod	0 ¹ 0 3 5	0.01 0.16 0.05	0.09 0.28 <u>0.61</u> 0.12	RA-2111/93 0415-93
Spain 1992	200 SL	2	0.1	0.01	with pod	0 ¹ 0 3 7 10	<0.01 0.26 0.07 <0.01 <0.01	0.18 0.53 <u>0.41</u> 0.32 0.21	RA-2058/92 0116-92
Spain 1992	200 SL	2	0.1	0.01	with pod	0 3 7	0.24 0.15 0.03	0.6 <u>0.66</u> 0.62	RA-2058/92 0117-92
Italy 1992	200 SL	2	0.1	0.01	pod	0 ¹ 0 3 7 10	0.01 0.26 0.06 0.03 0.01	0.13 0.49 <u>0.38</u> 0.29 0.28	RA-2095/92 0257-92
France 1996	200 SC	2	0.1	0.01	with pod	0 3 7	0.02 <0.01	0.41 <u>0.24</u> 0.24	RA-2042/96 0339-96
France 1996	200 SC	2	0.1	0.01	with pod	0 3 7	0.05 0.05	0.24 0.12 <u>0.16</u>	RA-2042/96 0493-96
Italy 1996	200 SC	2	0.1	0.01	with pod	0 3 7	0.05 <0.01	0.43 <u>0.33</u> 0.24	RA-2042/96 0494-96

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Spain 1996	200 SC	2	0.1	0.01	with pod	0 3 7	0.11 <0.01	1.0 <u>0.55</u> <0.05	RA-2042/96 0496-96
Brazil 1996	70 WG	5	0.18	0.058	without pod	21	<0.01	<u>0.01</u>	BRA I-D3-601/96 BRA-I-D3-601-96-A
Brazil 1996	70 WG	5	0.35	0.12	without pod	21	0.01	0.02	BRA I-D3-601/96 BRA-I-D3-601-96-B
Brazil 1996	70 WG	5	0.18	0.058	without pod	21	<0.01		4219 BRA-4219-A
Brazil 1996	70 WG	5	0.35	0.12	without pod	21	<0.01		4219 BRA-4219-B
<i>Common bean, various application methods</i>									
USA 1995	480 FS 240 SC 192 SC	1 1 3	0.25 ² 0.42 ³ 0.083 ⁴	0.3 0.03	with pod	6 6		<0.05 <0.05	109599 USA-95-SC-10
USA 1995	480 FS 240 SC 192 SC	1 1 3	0.25 ² 0.42 ³ 0.048- 0.053 ⁴	0.022 0.006- 0.007	with pod	7 7		<u>0.38</u> 0.35	109599 USA-95-OH-19
USA 1995	480 FS 240 SC 192 SC	1 1 3	0.25 ² 0.042 ³ 0.049 ⁴	0.023 0.013	with pod	0 3 7 13		0.51 0.48 <u>0.61</u> 0.52	109599 USA-95-WA-28
USA 1995	480 FS 240 SC 192 SC	1 1 3	0.25 ² 0.042 ³ 0.048 ⁴	0.14 0.017	with pod	7 7		<u>0.88</u> 0.72	109599 USA-95-FL-44
USA 1995	480 FS 240 SC 192 SC	1 1 3	0.25 ² 0.042 ³ 0.049 ⁴	0.57 0.022	with pod	6 6		<u>0.23</u> 0.23	109599 USA-95-WI-06
USA 1995	480 FS 240 SC 192 SC	1 1 3	0.25 ² 0.042 ³ 0.05 ⁴	1.0 0.018	with pod	7 7		<u>0.52</u> 0.37	109599 USA-96-NY-03
<i>Lima bean, various application methods</i>									
USA 1995	240 SC 192 SC	1 3	0.42 ³ 0.05 ⁴	0.18 0.006	without pod	7 7		<u>0.25</u> 0.24	109670 06201.95-MD03
USA 1995	240 SC 192 SC	1 3	0.42 ³ 0.083 ⁴	0.3 0.027	without pod	8 8		0.65 0.65	109670 06201.95-SC06
USA 1995	240 SC 192 SC	1 3	0.42 ³ 0.083 ⁴	0.3 0.027	without pod	8 8		0.44 0.67	109670 06201.95-SC07
USA 1995	240 SC 192 SC	1 3	0.42 ³ 0.05 ⁴	0.22 0.013	without pod	8 8		<u>0.12</u> 0.12	109670 06201.95-GA11

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA 1995	240 SC 192 SC	1 4	0.42 ³ 0.05 ⁴	0.022 0.006- 0.007	without pod	8 8		<0.05 <0.05	109670 06201.95-OH11
USA 1995	240 SC 192 SC	1 3	0.42 ³ 0.05 ⁴	0.029 0.005	without pod	7 7		<0.05 <0.05	109670 06201.95-CA23
USA 1995	240 SC 192 SC	1 3	0.42 ³ 0.05 ⁴	0.23 0.013	without pod	0 2 7 14		0.19 0.18 <u>0.17</u> 0.13	109670 06201.95-WA19

¹ Before last treatment

² Seed treatment (kg ai/100 kg seed)

³ In-furrow spray application at planting

⁴ Foliar spray: kinetic spray adjuvant (0.125%) included in tank mixes

Beans (dry, Table 109). In three trials in Brazil seeds were treated at 0.42 or 0.84 kg ai/100 kg seed (3 to 6 times GAP). The beans were harvested 95 and 98 days after treatment.

Table 109. Residues in dry beans, Brazil, 1992.

Application			PHI (days)	Residue, mg/kg		Report no. Study no.
Form.	No	kg ai/100 kg seed		Imidacloprid	Total	
70 WS	1	0.42	98	<0.01	0.51	0694-92
70 WS	1	0.42	95	0.03		055/93 BRA-055-93-A
70 WS	1	0.84	95	0.07		055/93 BRA-055-93-B

Potatoes (seed treatment and in-furrow application, Table 110). In northern Europe in eight trials in Germany the potatoes themselves were sprayed and then the furrow during planting (Dutch method) or an in-furrow band spray was applied to seed potatoes and soil. The application rate was 12 g ai/100 kg seed potatoes. A further six trials were with seed treatment at 7.2 or 14 g ai/100 kg in northern France, Germany and the UK. In southern Europe there were twelve trials, of which three were according to the maximum rate of 24.5 g ai/100 kg seed potatoes registered in Italy and five according to GAP in Spain.

Table 110. Residues in potatoes after seed treatment and/or in-furrow applications.

Country Year	Application			Sample ¹	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	kg ai/ha	g ai/ 100 kg seed			Imidacloprid	Total residue	

Country Year	Application			Sample ¹	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	kg ai/ha	g ai/ 100 kg seed			Imidacloprid	Total residue	
Spray during planting and in-furrow spray								
Germany 1991	350 FS	0.25	12	tuber	89	0.02	< <u>0.05</u>	RA-2079/91 0066-91
Germany 1991	350 FS	0.3	12	tuber	142	0.02	< <u>0.05</u>	RA-2079/91 0067-91
Germany 1991	350 FS		12	tuber, treated tuber	0 145	2.1 <0.01	< <u>0.05</u>	RA-2079/92 0394-92
Germany 1991	350 FS		12	tuber, treated tuber	0 97	3.0 0.01	< <u>0.05</u>	RA-2079/92 0396-92
In-furrow spray on seed potatoes and soil (band spray)								
Germany 1991	350 FS	0.36	12	tuber	79	0.02	< <u>0.05</u>	RA-2079/91 0069-91
Germany 1991	350 FS	0.35	12	tuber	139	<0.01	< <u>0.05</u>	RA-2079/91 0070-91
Germany 1991	350 FS	0.35	12	tuber, treated tuber	0 109	4.5 0.01	< <u>0.05</u>	RA-2079/92 0392-92
Germany 1991	350 FS	0.26	12	tuber, treated tuber	0 128	4.2 <0.01	< <u>0.05</u>	RA-2079/92 0393-92
Seed treatment								
France N 1999	600 FS		14	tuber, treated tuber	0 137	49 0.01	< <u>0.05</u>	RA-2114/99 0523-99
France N 1999	600 FS		7.2	tuber, treated tuber	0 137	25 <0.01	< <u>0.05</u>	RA-2114/99 0527-99
Germany 1999	600 FS		7.2	tuber, treated tuber	1 152	48 0.02	< <u>0.05</u>	RA-2114/99 0514-99
Germany 1999	600 FS		7.2	tuber, treated tuber	1 144	49 0.01	<u>0.05</u>	RA-2114/99 0515-99
Germany 1999	600 FS		14	tuber, treated tuber	1 144	97 0.04	<u>0.12</u>	RA-2114/99 0517-99
UK 1999	600 FS	0.25	7.2	tuber, treated tuber	0 124	21 0.02	< <u>0.05</u>	RA-2114/99 0526-99
Spain 1991	350 FS		14	tuber	103	<0.01	< <u>0.05</u>	RA-2078/91 0192-91

Country Year	Application			Sample ¹	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	kg ai/ha	g ai/ 100 kg seed			Imidacloprid	Total residue	
Spain 1991	350 FS		14	tuber	161	0.01	<0.05	RA-2078/91 0193-91
Italy 1992	350 FS		14	tuber	123	0.09	0.12	RA-2078/92 0134-92
Italy 1997	350 FS	0.27	25	tuber, treated tuber	0 137	150 0.06	0.2	RA-2104/97 0468-97
Italy 1997	600 FS	0.27	25	tuber, treated tuber	0 137	140 0.05	0.15	RA-2104/97 0469-97
Italy 1997	600 FS	0.26	25	tuber, treated tuber	0 103	140 0.02	0.06	RA-2104/97 0786-97
France 1999	S 600 FS	0.34	14	tuber, treated tuber	0 119	46 0.04	0.1	RA-2114/99 0524-99
Italy 1999	600 FS	0.19	14	tuber, treated tuber	0 136	112 0.03	0.09	RA-2114/99 0525-99
France 1999	S 600 FS	0.16	7.2	tuber, treated tuber	0 119	28 0.02	<0.05	RA-2114/99 0516-99
Italy 1999	600 FS	0.25	7.2	tuber, treated tuber	0 136	50 0.01	<0.05	RA-2114/99 0528-99
Greece 1999	600 FS	0.22	7.2	tuber, treated tuber	0 90	57 0.01	<0.05	RA-2114/99 0529-99
Spain 1999	600 FS	0.12	7.2	tuber, treated tuber	1 94	55 0.01	<0.05	RA-2114/99 0530-99

N: North, S: South

¹ "Treated tuber" is the seed tuber. "Tuber", without qualification, is the harvested tuber

Potatoes (foliar spray application, Table 111). In southern Europe seven trials were with the 200 SL formulation and eight with the 300 EC formulation at rates of 0.1 kg ai/ha (200 SL) and 0.09 kg ai/ha (300 EC). In Australia five trials were conducted with rates of 0.05 to 0.2 kg ai/ha.

Table 111. Residues in potato tubers after spray applications.

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Italy 1991	200 SL	2	0.1	0.01	14	<0.01	<0.05	RA-2024/91 0270-91
					21	<0.01	<0.05	

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Italy 1992	200 SL	2	0.1	0.01	7 14 21	<0.01	<0.05 <u><0.05</u> <0.05	RA-2051/92 0280-92
Italy 1993	200 SL	2	0.1	0.01	7 14 21	<0.01	<0.05 <u><0.05</u> <0.05	RA-2024/93 0016-93
Italy 1994	200 SL	2	0.1	0.01	0 7 10 14	<0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u>	RA-2062/94 0249-94
Italy 1994	200 SL	2	0.1	0.01	0 7 10 14	<0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u>	RA-2062/94 0250-94
Italy 1994	200 SL	2	0.1	0.01	0 14	<0.01 <0.01	<0.05 <u><0.05</u>	RA-2062/94 0251-94
Italy 1994	200 SL	2	0.1	0.01	0 14	0.05 <0.01	0.05 <u><0.05</u>	RA-2062/94 0252-94
Italy 1995	300 EC	2	0.09	0.009	0 ¹ 0 6 14 20	<0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2131/95 0202-95
Italy 1995	300 EC	2	0.09	0.009	0 ¹ 0 7 15 21	<0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2131/95 0656-95
Italy 1995	300 EC	2	0.09	0.009	0 15	<0.01	<0.05 <u><0.05</u>	RA-2131/95 0657-95
Italy 1995	300 EC	2	0.09	0.009	0 15	<0.01	<0.05 <u><0.05</u>	RA-2131/95 0658-95
Italy 1997	300 EC	2	0.09	0.009	0 14	<0.01 <0.01	<0.05 <u><0.05</u>	RA-2147/97 0624-97
Italy 1997	300 EC	2	0.09	0.009	0 14	<0.01 <0.01	<0.05 <u><0.05</u>	RA-2147/97 0625-97
Spain 1997	300 EC	2	0.09	0.009	0 14	<0.01 <0.01	<0.05 <u><0.05</u>	RA-2147/97 0621-97
Spain 1997	300 EC	2	0.09	0.009	0 14	<0.01 <0.01	<0.05 <u><0.05</u>	RA-2147/97 0622-97
Australia 1989	200 SL	4	0.05	0.0083	7	<0.01		43/89 AUS-43-89-A

Country Year	Application				PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total	
Australia 1989	200 SL	4	0.1	0.017	7	<0.01		43/89 AUS-43-89-B
Australia 1989	200 SL	4	0.2	0.033	7	<0.01		43/89 AUS-43-89-C
Australia 1992	200 SL	4	0.05	0.016	0 7 14	<0.01 <0.01 <0.01		23/92 AUS-23-92-A
Australia 1989	200 SL	4	0.1	0.031	0 7 14	<0.01 <0.01 <0.01		23/92 AUS-23-92-B

¹ Before last application

Potatoes (various application methods, Table 112). In Canada imidacloprid is registered for soil and foliar applications at rates of 0.2-0.31 kg ai/ha and 0.048 kg ai/ha respectively. In the USA imidacloprid is registered for use in soil at rates of 0.02-0.03 g ai/m, about 0.28 to 0.35 kg ai/ha, and as a foliar spray at about 0.05 kg ai/ha with a PHI of 7 days. In general in the USA the 240 SC formulation is registered for use in soil and the 192 SC for spray applications. Regardless of formulation or type of application it is forbidden to use more than 0.56 kg ai/ha per season.

In Canada in three trials in-furrow applications (0.03 g ai/m row) at planting were followed by four sprayings at 0.053 kg ai/ha (2.5 GR formulation for in-furrow and 240 SC formulation for sprays).

In 19 field trials in the USA both in-furrow and foliar spray applications were used. In 14 of the trials a 2.5 GR formulation was applied in-furrow and a 240 SC formulation for the sprays. Use of the 2.5 GR formulation is not registered in the USA. In five of the trials the 240 SC formulation was used for soil and foliar treatment (0.03 g ai/m row, 0.29-0.40 kg ai/ha, in-furrow spray, was followed by four sprayings at 0.053 kg ai/ha). In three more of the trials only in-furrow applications were made, at 0.33-0.34 kg ai/ha.

In South Africa imidacloprid is registered for use on potatoes at rates of 1.1 to 1.6 g ai/100 m row length, 0.14-0.21 kg ai/ha. Three trials were reported with in-furrow applications of 0.1, 0.2 and 0.3 kg ai/ha.

In South Korea the 2 GR formulation is registered for soil application at 0.06 kg ai/ha and a PHI of 30 days. In four trials in South Korea 1 to 4 applications were used. After spreading at 0.06 kg ai/ha, the GR formulation was incorporated into the soil. Only the parent compound was determined.

Table 112. Residues in potato tubers after various application methods.

Country, Location Year	Application				PHI (days) ²	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total residue	
In-furrow plus foliar spray application								
Canada, Ontario 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.028	7 14 21 28		< <u>0.1</u> <0.1 <0.1 <0.1	106554 510-N3117-91D
Canada, Prince 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.011	8 13 20 27		<u>0.12</u> <0.1 <0.1 <0.1	106554 533-N3118-91D
Canada, Manitoba 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.04	7 14 21 28		< <u>0.1</u> <0.1 <0.1 <0.1	106554 PMA-N3121-91D
USA, KS 1991	240 SC	1 4	0.33 ¹ 0.053 ²	0.22 0.028	7 14 21 28		< <u>0.05</u> <0.05 <0.05 <0.05	103235 STF-N3051-91D
USA, FL 1991	240 SC	1 4	0.33 ¹ 0.053 ²	0.4 0.028	7 14 21 29		<u>0.16</u> 0.14 0.14 0.14	103235 VBL-N3052-91D
USA, MI 1991	240 SC	1 4	0.33 ¹ 0.053 ²	0.7 0.028	7 14 21 28		<0.05 <u>0.05</u> <0.05 <0.05	103235 855-N3056-91D
USA, CA 1991	240 SC	1 4	0.33 ¹ 0.053 ²	0.64 0.028	7 14 21 28		<0.05 <u>0.05</u> <0.05 <0.05	103235 FCA-N3057-91D
USA, IN 1991	240 SC	1 4	0.33 ¹ 0.053 ²	0.7 0.028	7 14 21 28		< <u>0.05</u> <0.05 <0.05 <0.05	103235 HIN-N3058-91D
USA, ND 1991	2.5 GR 240 SC	1 4	0.32 ¹ 0.053 ²	0.028	7 14 21 28		< <u>0.05</u> <0.05 <0.05 <0.05	103235 251-N3053-91D
USA, ID 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.011	7 14 21 28		< <u>0.05</u> <0.05 <0.05 <0.05	103235 451-N3054-91D
USA, ME 1991	2.5 GR 240 SC	1 4	0.35 ¹ 0.053 ²	0.011	7 14 21 28		<u>0.07</u> 0.06 0.05 0.05	103235 758-N3055-91D

Country, Location Year	Application				PHI (days) ²	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total residue	
USA, MS 1991	2.5 GR 240 SC	1 4	0.4 ¹ 0.053 ²	0.028	7 14 21 28		<0.05 <0.05 <0.05 <0.05	103235 251-N3059-91D
USA, CO 1991	2.5 GR 240 SC	1 4	0.35 ¹ 0.053 ²	0.011	7 14 21 28		0.12 0.28 0.15 0.15	103235 253-N3060-91D
USA, ID 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.011	8 14 22 28		<0.05 <0.05 <0.05 <0.05	103235 452-N3061-91D
USA, WA 1991	2.5 GR 240 SC	1 4	0.35 ¹ 0.053 ²	0.012- 0.013	7 14 21 28		<0.05 <0.05 <0.05 <0.05	103235 454-N3062-91D
USA, ME 1991	2.5 GR 240 SC	1 4	0.35 ¹ 0.053 ²	0.011	7 14 21 28		<0.05 <0.05 <0.05 <0.05	103235 758-N3063-91D
USA, MI 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.028	7 14 21 28		<0.05 <0.05 <0.05 <0.05	103235 855-N3064-91D
USA, CA 1991	2.5 GR 240 SC	1 4	0.29 ¹ 0.053 ²	0.028	7 14 21 28		0.05 <0.05 <0.05 <0.05	103235 FCA-N3065-91D
USA, IN 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.028	7 14 21 28		<0.05 <0.05 <0.05 <0.05	103235 HIN-N3066-91D
USA, KS 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.028	7 14 21 28		<0.05 <0.05 <0.05 <0.05	103235 STF-N3087-91D
USA, FL 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.028	7 14 21 29		0.13 0.13 0.13 0.11	103235 VBL-N3088-91D
USA; WI 1991	2.5 GR 240 SC	1 4	0.33 ¹ 0.053 ²	0.024	7 14 21 28		<0.05 <0.05 <0.05 <0.05	103235 851-N3089-91D
<i>In-furrow application at planting</i>								
USA, FL 1997	240 SC	1	0.33	0.54	71		0.07 0.04	108847 VBL-AD001-97H-A

Country, Location Year	Application				PHI (days) ²	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl		Imidacloprid	Total residue	
USA, KS 1997	240 SC	1	0.34	0.5	113		<u>0.02</u> 0.02	108847 STF-AD002-97H-A
USA, IN 1997	240 SC	1	0.34	1.8	111		0.1 <u>0.18</u>	108847 HIN-AD003-97H-A
South Africa 1994	200 SL	1	0.1	0.02	99 120		< <u>0.04</u> <0.04	311-88838-M39 88838-M39-A
South Africa 1994	200 SL	1	0.2	0.04	99 120		<u>0.04</u> <0.04	311-88838-M39 88838-M39-B
South Africa 1994	200 SL	1	0.3	0.06	99 120		0.07 0.05	311-88838-M39 88838-M39-C
<i>Spreading and incorporation into the soil</i>								
South Korea 1992	2 GR	1	0.6		147 147	<0.01 <0.01		R2601-93
South Korea 1992	2 GR	2	0.6		30 30 60 60 90 90	0.04 0.04 0.03 0.03 <0.01 <0.01		R2602-93 R2603-93 R2604-93
South Korea 1992	2 GR	3	0.6		30 30 60 60	0.05 0.05 0.02 0.02		R2605-93 R2606-93
South Korea 1992	2 GR	4	0.6		30 30	0.08 0.08		R2607-93

¹ In-furrow application: 0.03 g ai/m row² After spray application

Sugar beet (seed treatment, Table 113). 21 trials were conducted on sugar beet in Europe with application rates of 90-110 g ai/100,000 seeds in 20 of them.

Table 113. Residues in sugar beet after seed treatment.

Country Year	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	g ai/unit			Imidacloprid	Total	
France, North 1989	70 WS	1	150	root or beet	199	<0.01	<0.05	0108-89
				leaf	199	<0.01	0.1	
France, North 1989	70 WS	1	90	root or beet	199	<0.01	< <u>0.05</u>	0472-89
				leaf	199	<0.01	< <u>0.05</u>	

Country Year	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	g ai/unit			Imidacloprid	Total	
Germany 1989	70 WS	1	110	leaf	23	1.1	6.9	<i>PF-3711/89</i> 0176-89
					58	0.03	0.22	
					120	<0.01	0.06	
					128	<0.01	<0.05	
					159	<0.01	<0.05	
				root or beet	58	0.06	0.16	
					120	<0.01	<0.05	
					128	<0.01	<0.05	
					159	<0.01	<0.05	
Germany 1989	70 WS	1	110	leaf	49	0.22	0.86	<i>PF-3711/89</i> 0177-89
					73	0.06	0.24	
					155	<0.01	<0.05	
					188	<0.01	<0.05	
					210	<0.01	<0.05	
				root or beet	73	0.15	0.25	
					155	<0.01	<0.05	
					188	<0.01	<0.05	
					210	<0.01	<0.05	
Germany 1989	70 WS	1	110	leaf	49	2.1	9.2	<i>PF-3711/89</i> 0178-89
					90	<0.01	0.2	
					168	<0.01	0.09	
					190	<0.01	<0.05	
				root or beet	90	0.04	0.13	
					168	<0.01	<0.05	
					190	<0.01	<0.05	
Germany 1989	70 WS	1	110	leaf	49	0.16	7.6	<i>PF-3711/89</i> 0179-89
					69	0.05	0.16	
					162	<0.01	0.05	
					188	<0.01	<0.05	
					210	<0.01	<0.05	
				root or beet	69	0.05	0.71	
					162	<0.01	<0.05	
					188	<0.01	<0.05	
					210	<0.01	<0.05	
Germany 1990	70 WS	1	110	leaf	28		22	<i>PF-3462/90</i> 0190-90
					33		12	
					73		0.09	
					147		<0.05	
					185	<0.01	<0.05	
				root or beet	73		0.06	
					147		<0.05	
					185	<0.01	<0.05	
Germany 1990	70 WS	1	110	leaf	51		1.6	<i>PF-3462/90</i> 0191-90
					62		0.56	
					83		0.1	
					161		0.07	
					196	<0.01	<0.05	

Country Year	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	g ai/unit			Imidacloprid	Total	
				root or beet	83 161 196	<0.01	0.22 <u><0.05</u> <u><0.05</u>	
Germany 1990	70 WS	1	110	leaf	48 64 92 157 191	<0.01	10 1.9 0.08 <u><0.05</u> <u><0.05</u>	PF-3462/90 0192-90
				root or beet	92 157 191	<0.01	0.06 <u><0.05</u> <u><0.05</u>	
Germany 1990	70 WS	1	110	leaf	37 44 79 142 184	<0.01	8.0 3.0 0.06 <u><0.05</u> <u><0.05</u>	PF-3462/90 0193-90
				root or beet	79 142 184	<0.01	0.07 <u><0.05</u> <u><0.05</u>	
UK 1990	70 WS	1	90	leaf	89 158	<0.01	0.12 <u>0.07</u>	0055-90
				root or beet	158	<0.01	<u><0.05</u>	
UK 1990	70 WS	1	90	leaf	89 158	<0.01	0.26 <u>0.14</u>	0407-90
				root or beet	158	<0.01	<u><0.05</u>	
UK 1991	70 WS	1	90	whole plant with root	90		0.14	RA-2084/91 0157-91
				leaf	141	<0.01	<u>0.11</u>	
				root or beet	141	<0.01	<u><0.05</u>	
UK 1991	70 WS	1	90	whole plant with root	83		0.43	RA-2084/91 0158-91
				leaf	144	<0.01	<u>0.11</u>	
				root or beet	144	<0.01	<u><0.05</u>	
Sweden 1990	70 WS	1	90	leaf	173	<0.01	<u>0.06</u>	0391-90
				root or beet	173	<0.01	<u><0.05</u>	
Sweden 1990	70 WS	1	90	leaf	191	<0.01	<u><0.05</u>	0397-90

Country Year	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	g ai/unit			Imidacloprid	Total	
				root or beet	191	<0.01	<0.05	
Italy 1992	70 WS	1	90 ¹	leaf	143	<0.01	<0.05	RA-2080/92 0313-92
				root or beet	143	<0.01	<0.05	
Italy 1997	600 FS	1	103	treated seed	0	39590		RA-2163/97 0271-97
				root or beet	153	<0.01	<0.05	
				leaf	153	<0.01	0.06	
Italy 1997	70 WS	1	106	treated seed	0	40500		RA-2163/97 0748-97
				root or beet	153	<0.01	<0.05	
				leaf	153	<0.01	0.06	
Italy 1998	600 FS	1	90.6	treated seed	0	26260		RA-2094/98 1163-98
				root or beet	148	<0.01	0.05	
				leaf	148	<0.01	<0.05	
Italy 1998	70 WS	1	95.5	treated seed	0	31520		RA-2094/98 1164-98
				root or beet	148	<0.01	<0.05	
				leaf	148	<0.01	0.07	

¹ 0.17 kg ai/ha

Sugar beet (foliar spray application, Table 114). In Italy in eight field trials crops were sprayed twice (0.09 kg ai/ha, water 600 l/ha).

Table 114. Residues in sugar beet after foliar spray treatments in Italy.

Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
1995	300 EC	2	0.09	0.015	root or beet	0 30	<0.01	<0.05 <0.05	RA-2132/95 0199-95
					leaf	0 30	<0.01	0.64 0.4	
1995	300 EC	2	0.09	0.015	root or beet	0 30	<0.01	<0.05 <0.05	RA-2132/95 0653-95

Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
					leaf	0 30	<0.01	1.8 <u>0.47</u>	
1995	300 EC	2	0.09	0.015	root or beet	01 0 15 30 40	<0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2132/95 0654-95
					leaf	01 0 15 30 40	<0.01 <0.01	0.48 2.5 0.91 <u>0.61</u> 0.13	
1995	300 EC	2	0.09	0.015	root or beet	01 0 15 30 40	<0.01 <0.01	<0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2132/95 0655-95
					leaf	01 0 15 30 40	<0.01 0.01	0.1 0.74 0.52 <u>0.33</u> 0.22	
1996	300 EC	2	0.09	0.015	root or beet	0 30	<0.01	<0.05 <u><0.05</u>	RA-2048/96 0065-96
					leaf	0 30	0.01	1.9 <u>0.67</u>	
1996	300 EC	2	0.09	0.015	root or beet	0 30	<0.01	0.06 <u><0.05</u>	RA-2048/96 0066-96
					leaf	0 30 40	0.01	1.9 <u>0.45</u> 0.06	
1996	300 EC	2	0.09	0.015	root or beet	0 32	<0.01	<0.05 <u><0.05</u>	RA-2048/96 0518-96
					leaf	0 32	0.01	1.5 <u>0.23</u>	
1996	300 EC	2	0.09	0.015	root or beet	01 0 15 30 40	<0.01	<0.05 <0.05 <0.05 <u><0.05</u> <0.05	RA-2048/96 0519-96

Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.	
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total		
					leaf	01 0 15 30 40	<0.01	0.45 2.2 0.59 <u>0.31</u> 0.12		

¹ Before last application

Celery (Table 115). In the USA twelve field trials were conducted to determine residues in untrimmed celery stalks with foliage after treatment of plants with a 240 SC formulation. Stalks without leaves were not analysed.

In six of the trials a plant drench was used at about 0.54 to 0.59 kg ai/ha 43-46 days before harvest. The other six were bridging trials. In three of them in-furrow applications were carried out at transplanting with application rates of 0.56 to 0.6 kg ai/ha, and in the other three sidedress applications 43-46 days before harvest at rates of 0.56-0.59 kg ai/ha.

Table 115. Residues in celery after various soil applications in trials in the USA.

Year	Application				Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl				
Plant drench application								
1994	240 SC	1	0.56	0.006	stalk with foliage	46	1.0	107126 454-AD028-94D
1994	240 SC	1	0.57	0.005	stalk with foliage	45	0.13	107126 457-AD029-94D
1994	240 SC	1	0.54	0.004	stalk with foliage	43	0.13	107126 459-AD030-94D
1994	240 SC	1	0.56	0.007	stalk with foliage	45	0.57	107126 FCA-AD031-94D
1994	240 SC	1	0.59	0.006	stalk with foliage	43	0.42	107126 HIN-AD032-94D
1995	240 SC	1	0.56	0.007	stalk with foliage	46	4.3	107126 VBL-AD033-94D
Sidedress application								
1994	240 SC	1	0.56	0.43	stalk with foliage	45	5.6	107126 FCA-AD034-94D
1994	240 SC	1	0.56	1.1	stalk with foliage	3	0.78	107126 HIN-AD035-94D
1995	240 SC	1	0.56	0.98	stalk with foliage	45	2.8	107126 VBL-AD036-94D

Year	Application				Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl				
In-furrow application								
1994	240 SC	1	0.56	0.43	stalk with foliage	103	1.4	107126 FCA-AD037-94D
1994	240 SC	1	0.56	1.2	stalk with foliage	81	0.38	107126 HIN-AD038-94D
1995	240 SC	1	0.6	1.1	stalk with foliage	97	1.2	107126 VBL-AD039-94D

Barley (seed treatment, Tables 116 and 117). In nine European field trials imidacloprid was applied in a 70 WS or a 350 FS formulation to winter and spring barley at 0.07 kg ai/100 kg seed. In another German trial seeds were treated with 0.035 kg ai/100 kg seed, and in a UK trial a 350 FS formulation was applied at 0.11 kg ai/100 kg seed, 50% higher than the recommended rate in France and Belgium.

In Australia in 11 trials either a 350 FS or a 600 FS formulation was used. In four of the trials seeds were treated at 0.07 or 0.014 kg ai/100 kg in accordance with Australian GAP, and in three others at higher rates of 0.28-0.29 kg ai/100 kg. Forage was sampled 28-77 days after treatment and drilling, and straw and grain collected at harvest (i.e. after 154 to 210 days).

Table 116. Residues in spring and winter barley after seed treatments in Europe.

Country Year	Application					Sample	PHI (days)	Residue, mg/kg		Reference Study no.
	Crop	Form.	No.	kg ai/100 kg seed	kg ai/ha			Imidacloprid	Total	
France 1990	winter	70 WS	1	0.07	0.074	grain straw	287 287	<0.01 <0.1	<u><0.05</u> <u><0.05</u>	0553-90
France 1990	winter	350 FS	1	0.07	0.074	grain straw	287 287	<0.01 <0.1	<u><0.05</u> <u><0.05</u>	0558-90
France 1990	winter	70 WS	1	0.07	0.053	grain straw	270 270	<0.01 <0.1	<u><0.05</u> <u><0.05</u>	0678-90
France 1990	winter	350 FS	1	0.07	0.053	grain straw	270 270	<0.01 <0.1	<u><0.05</u> <u><0.05</u>	0681-90
UK 1991	spring	350 FS	1	0.11		treated seed forage grain straw control straw	0 70 155 155	920 <0.01 0.06	<u>0.24</u> <u><0.05</u> <u>0.32</u> 0.06	RA-2129/91 0164-91
UK 1991	spring	350 FS	1	0.07		treated seed forage grain straw	0 70 155 155	600 <0.01 <0.05	<u>0.12</u> <u><0.05</u> <u>0.16</u>	RA-2129/91 0165-91

Country Year	Application					Sample	PHI (days)	Residue, mg/kg		Reference Study no.
	Crop	Form.	No.	kg ai/100 kg seed	kg ai/ha			Imidacloprid	Total	
Germany 1990	spring	350 FS	1	0.07	0.095	forage	39 48 58		0.17 0.08 <u>0.05</u>	<i>PF3736</i> 0082-90
						grain	108	<0.01	< <u>0.05</u>	
						straw	108	<0.1	<u>0.09</u>	
Germany 1990	spring	350 FS	1	0.07	0.095	forage	40 49 61		0.33 0.24 <u>0.13</u>	<i>PF3736</i> 0083-90
						grain	106	<0.01	< <u>0.05</u>	
						straw	106	<0.1	<u>0.09</u>	
Germany 1991	spring	350 FS	1	0.07	0.109	forage	47 57 68		0.44 <0.05 <u>0.09</u>	<i>RA-2080/91</i> 0071-91
						grain	146	<0.01	< <u>0.05</u>	
						straw	146	<0.05	<u>0.11</u>	
Germany 1991	spring	350 FS	1	0.07	0.095	forage	61 70 79		0.31 0.14 <u>0.15</u>	<i>RA-2080/91</i> 0072-91
						grain	118	<0.01	< <u>0.05</u>	
						straw	118	<0.05	<u>0.28</u>	
Germany 1997	spring	341.25 FS	1	0.035		treated seed	-13	340		<i>RA-2078/97</i> 0284-97
						forage	50	0.01	<u>0.12</u>	
						grain	128	<0.01	< <u>0.05</u>	
						straw	128	<0.05	<u>0.05</u>	

Table 117. Results of Australian trials with imidacloprid in barley after seed treatment in Australia.

Year	Application			Sample	PHI (days)	Total residue (mg/kg)	Reference Study no.
	Form.	No.	kg ai/100 kg seed				
1993	350 FS	1	0.07	forage	63	<u>0.52</u>	29/93 AUS-29-93-A LOQ 0.05 mg/kg
				forage, residue calculated on dry weight basis	63	2.8	
				grain	210	< <u>0.05</u>	
				straw	210	<u>0.06</u>	
1993	350 FS	1	0.14	forage	63	<u>0.67</u>	29/93 AUS-29-93-B LOQ 0.05 mg/kg
				forage, residue calculated on dry weight basis	63	3.6	
				grain	210	< <u>0.05</u>	

Year	Application			Sample	PHI (days)	Total residue (mg/kg)	Reference Study no.
	Form.	No.	kg ai/100 kg seed				
				straw	210	<u>0.12</u>	
1993	350 FS	1	0.28	forage	63	0.95	29/93 AUS-29-93-C LOQ 0.05 mg/kg
				forage, residue calculated on dry weight basis	63	5.1	
				grain	210	<0.05	
				straw	210	0.15	
1993	350 FS	1	0.07	forage	28 42 56 63	0.31 0.1 0.05 <u>0.03</u>	59/93 AUS-59-93-A LOQ 0.05 mg/kg
				forage, residue calculated on dry weight basis	28 42 56 63	3.1 1 0.5 0.3	
				grain	154	< <u>0.05</u>	
				straw	154	< <u>0.05</u>	
1993	350 FS	1	0.14	forage	28 42 56 63	0.43 0.18 0.06 <u>0.06</u>	59/93 AUS-59-93-B LOQ 0.05 mg/kg
				forage, residue calculated on dry weight basis	28 42 56 63	4.3 1.8 0.6 0.6	
				grain	154	< <u>0.05</u>	
				straw	154	< <u>0.05</u>	
1994	600 FS	1	0.072	forage	28 42 56 63	0.97 0.16 0.05 <u>0.07</u>	19/94 AUS-19-94-A LOQ 0.02 mg/kg
				forage, residue calculated on dry weight basis	28 42 56 63	2.0 1.0 0.32 0.45	
				grain	181	< <u>0.02</u>	
				straw	181	<u>0.05</u>	

Year	Application			Sample	PHI (days)	Total residue (mg/kg)	Reference Study no.
	Form.	No.	kg ai/100 kg seed				
1994	600 FS	1	0.14	forage	28 42 56 63	3.0 0.28 0.14 <u>0.19</u>	19/94 AUS-19-94-B LOQ 0.02 mg/kg
				forage, residue calculated on dry weight basis	28 42 56 63	6.0 1.8 0.89 1.2	
				grain	181	< <u>0.02</u>	
				straw	181	<u>0.11</u>	
				forage	28 42 56 63	6.5 0.5 0.24 0.2	
				forage, residue calculated on dry weight basis	28 42 56 63	13.1 3.2 1.5 1.3	
				grain	181	<0.02	
				straw	181	0.29	
1994	600 FS	1	0.072	forage	28 42 56 63 77	0.11 <0.02 0.03 < <u>0.02</u> <0.02	28-94 AUS-28-94-A LOQ 0.02 mg/kg
				forage, residue calculated on dry weight basis	28 42 56 63 77	0.8 <0.02 0.19 <0.02 <0.02	
				forage	28 42 56 63 77	0.29 <0.02 0.04 <u>0.03</u> <0.02	28-94 AUS-28-94-B LOQ 0.02 mg/kg
				forage, residue calculated on dry weight basis	28 42 56 63 77	2.1 <0.02 0.25 0.19 <0.02	
				forage	28 42 56 63 77	0.18 0.04 0.05 <u>0.04</u> <0.02	28-94 AUS-28-94-C
				forage, residue calculated on dry weight basis	28 42 56 63 77	0.18 0.04 0.05 <u>0.04</u> <0.02	
				forage	28 42 56 63 77	0.18 0.04 0.05 <u>0.04</u> <0.02	
				forage, residue calculated on dry weight basis	28 42 56 63 77	0.18 0.04 0.05 <u>0.04</u> <0.02	

Year	Application			Sample	PHI (days)	Total residue (mg/kg)	Reference Study no.
	Form.	No.	kg ai/100 kg seed				
				forage, residue calculated on dry weight basis	28 42 56 63 77	1.3 0.29 0.32 0.25 <0.02	LOQ 0.02 mg/kg

Maize (Table 118). In Europe (Germany, France, Italy and Spain) 14 field trials were conducted in accordance with GAP of various countries to determine the total residue of imidacloprid in maize kernels, cobs and plants after seed treatment. In the four in Germany (600 FS formulation, 54 g ai/unit, corresponding to 0.47 kg ai/100 kg seed, 0.11 kg ai/ha) plants without roots were sampled when 10 cm tall (BBCH code 13), and at BBCH stages 33 and 53. In one trial forage and cobs were sampled when ripe enough for silage (BBCH code 85: dough stage).

The four trials in France were bridging trials, in two of which seeds were treated with a 70 WS and two with a 350 FS formulation, at 0.7 kg ai/100 kg seed. Plants without roots were sampled at the ripening stage for silage (dough stage), 133-178 days after seed treatment and sowing, and kernels were collected on the day of harvest, after 165-198 days.

The four bridging trials in Italy were in accordance with Italian GAP, two with a 350 FS formulation and the other two with 600 FS, 0.7 kg ai/100 kg seed, 0.13-0.15 kg ai/ha. Plants and cobs without husk were sampled at the dough stage, i.e. 127-129 days after treatment and sowing, and kernels were collected at harvest.

In two trials in Spain a 70 WS formulation was used at 0.7 kg ai/100 kg seed. Only kernels were collected at harvest.

In South Africa a 70 WS formulation was used in four trials, two were treated at the recommended label rate of 0.35 kg ai/100 kg seed and two at twice that rate. Forage samples were collected after 42 days and thereafter four times at intervals of about 20 days, so that the last sampling was 127 or 129 days after treatment and sowing. Cobs were sampled on day 97, and kernels and straw at harvest, 165 or 198 days after sowing.

Table 118. Residues in maize after seed treatment.

Country Year	Application				Sample BBCH Code	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	mg ai/ seed	kg ai/100 kg seed	kg ai/ ha			imida- cloprid	Total	
Germany 1993	600 FS	1.1 ²	0.47	0.11	treated seed	0	3460		RA-2072/93 0058-93
					forage	13 ⁵	19	35	
						33	40	0.04	
						53	69	<0.01	
					forage	85	151	<0.01	
					cob	151	<0.01	<0.05	

Country Year	Application				Sample BBCH Code	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	mg ai/ seed	kg ai/100 kg seed	kg ai/ ha			imida- cloprid	Total	
Germany 1993	600 FS	1.1 ²	0.47	0.11	treated seed	0	3090		RA-2072/93 0179-93
					forage	13 ⁵ 16	3.1	23	
						33	0.02	0.15	
						53	<0.01	<0.05	
					kernel	89	<0.01	<0.05	
Germany 1993	600 FS	1.1 ²	0.47	0.11	straw	167	<0.05	<0.05	RA-2072/93 0180-93
					treated seed	0	4340		
					forage	13 ⁵ 22	1.9	14	
						33	<0.01	0.06	
						53	<0.01	<0.05	
Germany 1993	600 FS	1.1 ²	0.47	0.11	kernel	89	<0.01	<0.05	RA-2072/93 0181-93
					straw	149	<0.05	0.1	
					treated seed	0	4300		
					forage	13 ⁵ 17	0.7	10	
						33	0.01	0.11	
France, N 1990	70 WS	n.s.	0.7	n.s.		53	<0.01	0.08	0679-90
					kernel	85	<0.01	<0.05	
					plant without roots	85		0.06	
					kernel	165	<0.01	<0.05	
					kernel	165	<0.01	<0.05	
France, N 1990	350 FS	n.s.	0.7	n.s.	plant without roots	85		0.1	0685-90
					kernel	133	<0.01	<0.05	
					kernel	165	<0.01	<0.05	
					plant without roots	85		0.05	
					kernel	198	<0.01	<0.05	
France, S 1990	70 WS	1.7 ¹	0.7	0.13	plant without roots	85		0.05	0556-90
					kernel	178	<0.01	<0.05	
					kernel	198	<0.01	<0.05	
					plant without roots	85		<0.05	
					kernel	198	<0.01	<0.05	
Italy 1997	350 FS	1.86 ³	0.7	0.15	plant without roots	85		<0.05	RA-2105/97 0470-97
					kernel	129	<0.01	<0.05	
					kernel	160	<0.01	<0.05	
					treated seed	0	5930		
					plant without roots	85		<0.05	
Italy 1997	600 FS	1.86 ³	0.7	0.15	kernel	129	<0.01	<0.05	RA-2105/97 0471-97
					kernel	160	<0.01	<0.05	
					treated seed	0	4470		
					plant without roots	85		<0.05	
					kernel	160	<0.01	<0.05	
Italy 1998	350 FS	1.84 ⁴	0.7	0.13	kernel	-4 ⁶	6300		RA-2096/98 1162-98
					plant without roots	85	<0.01	<0.05	
					cob w/o husks	85	<0.01	<0.05	
					kernel	150	<0.01	<0.05	
					kernel	150	<0.01	<0.05	
Italy 1998	600 FS	1.84 ⁴	0.7	0.13	plant without roots	85	<0.01	<0.05	RA-2096/98 1161-98
					cob without husks	85	<0.01	<0.05	
					kernel	150	<0.01	<0.05	
					kernel	150	<0.01	<0.05	
					kernel	150	<0.01	<0.05	
Spain 1990	70 WS	n.s.	0.7	n.s.	kernel	155	<0.01	<0.05	0224-90
					kernel	155	<0.01	<0.05	
					kernel	155	<0.01	<0.05	
					kernel	155	<0.01	<0.05	
					kernel	155	<0.01	<0.05	

Country Year	Application				Sample BBCH Code	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	mg ai/ seed	kg ai/100 kg seed	kg ai/ ha			imida- cloprid	Total	
Spain 1990	70 WS	n.s.	0.7	n.s.	kernel	152	<0.01	< <u>0.05</u>	0230-90
South Africa 1990	70 WS		0.7		forage	43 64 84 107 129	<0.02 <0.02 <0.02 <0.02 <0.02		311-88075- H175 88075-H175- A
					cob	97	<0.02		
					straw	169	0.03		
					kernel	169	<0.02		
South Africa 1990	70 WS		0.35		forage	43 64	<0.02 <0.02		311-88075- H175 88075-H175- B
					cob	97	<0.02		
					straw	169	<0.02		
					kernel	169	<0.02		
South Africa 1989	70 WS		0.35		forage	42 63 84		0.45 0.16 < <u>0.02</u>	311-88809- G151 88809-G151- A
					straw	196		< <u>0.02</u>	
					kernel	196		< <u>0.02</u>	
South Africa 1989	70 WS		0.56		forage	42 63 84 105		0.88 0.32 0.13 <0.02	311-88809- G151 88809-G151- B
					straw	196		<0.02	
					kernel	196		<0.02	

¹ seed rate: 18 kg seed/ha (75 000 seeds/ha)

² seed rate: 23 kg seed/ha (100 000 seeds); 54 g ai/unit

³ seed rate: 82 000 seeds/ha

⁴ seed rate: 70 000 seeds/ha

⁵ height of maize plants: 10 cm

⁶ directly after seed dressing

n.s.: not specified

N: North, S: South

Oats (Table 119). In Sweden two trials were conducted in which seeds were treated at 0.11 kg ai/100 kg with a 350 FS formulation. Grain and straw was sampled at harvest.

In Australia in two trials using a 350 FS or a 600 FS formulation seeds were treated at rates of 0.07 or 0.14 kg ai/100 kg in accordance with Australian GAP. Forage was sampled after 28-63 days, and straw and grain collected at harvest after 154 days.

In a single trial in Germany seed was treated at the GAP rate of 0.035 kg ai/100 kg seed.

Table 119. Residues in oats after seed treatment.

Country Year	Application			Sample	PHI (days)	Residues, mg/kg		Report Study	no. no.
	Form.	No.	kg ai/100 kg seed			Imidacloprid	Total		
Sweden 1990	350 FS	1	0.11	grain straw	128 128	<0.01 <0.1	< <u>0.05</u> <u>0.08</u>	0395-90	
Sweden 1990	350 FS	1	0.11	grain straw	142 142	<0.01 <0.1	< <u>0.05</u> <u>0.05</u>	0396-90	
Australia 1993	350 FS	1	0.07	forage	28 42 56 63		0.36 0.09 <0.02 < <u>0.02</u>	57/93 AUS-57-93-A	
				forage, residue calculated on dry weight basis	28 42 56 63		3.3 0.8 <0.02 <0.02		
				grain	154		< <u>0.02</u>		
				straw	154		< <u>0.02</u>		
Australia 1993	350 FS	1	0.14	forage	28 42 56 63		0.71 0.21 0.11 <u>0.06</u>	57/93 AUS-57-93-B	
				forage, residue calculated on dry weight basis	28 42 56 63		6.6 1.9 1.0 0.5		
				grain	154		< <u>0.02</u>		
				straw	154		< <u>0.02</u>		
Germany 1997	87.5 FS	1	0.035	treated seed forage kernel straw	0 50 138 138	380 <0.01 <0.01 <0.05	0.09 < <u>0.05</u> < <u>0.05</u>	RA-2078/97 0283-97	

Rice (Table 120). In six trials with foliar spray treatments in Thailand, two were with a 100 SL formulation and the other four with a 25 WP. In all trials there were two applications with an interval of 20-21 days, four at 0.005 kg ai/hl and two at 0.01 kg ai/hl (0.015-0.025 kg ai/ha and 0.05 kg ai/ha respectively).

In four trials in South Korea a 40 SL formulation was used, 0.004 kg ai/hl, 0.064 kg ai/ha (3-5 applications, intervals 8-29 days).

Table 120. Residues in rice after spraying.

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Thailand 1991	100 SL	2	0.015	0.005	control forage	0 22	0.05 <0.01 ¹	0.45 0.06	RA-2016/91 0223-91
					straw	29	<0.05	<0.05	
					grain, polished	29	<0.01	<0.05	
					grain, unpolished	29	<0.01	<0.05	
Thailand 1991	100 SL	2	0.024	0.005	forage	0 22	<0.01	0.62 0.07	RA-2016/91 0482-91
					straw	33	<0.05	0.58	
					grain, polished	33	<0.01	<0.05	
					grain, unpolished	33	<0.01	<0.05	
					panicle	22	<0.01	<0.05	
Thailand 1992	25 WP	2	0.025	0.005	forage	0	0.81	0.74	RA-2067/92 0218-92
					straw	56	<0.05	<0.05	
					grain, polished	56	<0.01	<0.05	
					glume	56	<0.05	<0.05	
					bran	56	<0.01	<0.05	
Thailand 1992	25 WP	2	0.05	0.01	forage	0	1.3	1.2	RA-2067/92 0219-92
					straw	56	<0.05	<0.05	
					grain, polished	56	<0.01	<0.05	
					glume	56	<0.05	<0.05	
					bran	56	<0.01	<0.05	

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Thailand 1993	25 WP	2	0.025	0.005	forage	0	0.63	0.69	RA-2067/92 0698-92
					straw	48	<0.05	<0.05	
					grain, polished	48	<0.01	<0.05	
					glume	48	<0.05	<0.05	
					bran	48	<0.01	<0.05	
Thailand 1993	25 WP	2	0.05	0.01	forage	0	1.3	1.5	RA-2067/92 0699-92
					straw	48	<0.05	<0.05	
					grain, polished	48	<0.01	<0.05	
					glume	48	<0.05	<0.05	
					bran	48	<0.01	0.08	
South Korea 1991	40 SL	3	0.064	0.004	grain, hulled	46	<0.025		R-2021/92 KOR-R2021-92
					straw	46	0.29		
South Korea 1991	40 SL	4	0.064	0.004	grain, hulled	8	0.07		Summary of R-2022/92 R-2023/92 R-2024/92 KOR-R2022-92 KOR-R2023-92 KOR-R2024-92
						16	0.04		
						31	<0.025		
					straw	8	0.76		
						16	0.71		
						31	0.64		
South Korea 1991	40 SL	5	0.064	0.004	grain, hulled	8	0.08		Summary of R-2025/92 R-2026/92 KOR-R2025-92 KOR-R2026-92
						16	0.07		
					straw	8	1.29		
						16	0.64		
South Korea 1991	40 SL	6	0.064	0.004	grain, hulled	8	0.06		R-2027/92 KOR-R2027-92
					straw	8	1.95		

¹ 0.05 mg/kg in the control sample. It is possible that the control and the treated sample were exchanged (manufacturer's comment).

Triticale (Table 121). In Australia in four trials in accordance with GAP seeds were treated at 0.07 or 0.14 kg ai/100 kg with a 350 FS formulation. Forage was sampled 4, 6, 8, and 9 weeks after sowing, and grain and straw at harvest.

Table 121. Residues in triticale after seed treatment.

Country Year	Application			Sample	PHI (days)	Total residues, mg/kg	Report no. Study no.
	Form.	No.	kg ai/100 kg seed				
Australia 1993	350 FS	1	0.07	forage	28 42 56 63	0.25 0.07 <0.05 <u><0.05</u>	58/93 AUS-58-93-A
				forage, residue calculated on dry weight basis	28 42 56 63	2.2 0.6 0.3 0.2	
				grain	154	<u><0.05</u>	
				straw	154	<u><0.05</u>	
				forage	28 42 56 63	0.52 0.06 0.04 <u>0.04</u>	
				forage, residue calculated on dry weight basis	28 42 56 63	4.6 0.5 0.4 0.4	
				grain	154	<u><0.05</u>	
				straw	154	<u><0.05</u>	
Australia 1993	350 FS	1	0.14	forage	28 42 56 63	0.52 0.06 0.04 <u>0.04</u>	58/93 AUS-58-93-B
				forage, residue calculated on dry weight basis	28 42 56 63	4.6 0.5 0.4 0.4	
				grain	154	<u><0.05</u>	
				straw	154	<u><0.05</u>	

Wheat (Tables 122 and 123). In ten field trials in France, the UK and Germany imidacloprid was applied as seed treatment in accordance with GAP in Belgium and France. A 70 WS or a 350 FS formulation was used. Winter and spring wheat (5 trials each) were treated with 0.07 kg ai/100 kg seed. In a further trial a 350 FS formulation was applied at 0.11 kg ai/100 kg seed, 50% higher than the recommended rate. In two other German trials the seed was treated with 0.035 kg ai/100 kg seed in accordance with the UK label rate.

In Australia in 15 trials either a 350 FS or a 600 FS formulation was used. Of ten trials, in five seeds were treated at 0.07 and in the other five at 0.14 kg ai/100 kg, all in accordance with Australian GAP. In two further trials higher rates of 0.28-0.29 kg ai/100 kg were used, and in another two low rates of 0.018 and 0.035 kg ai/100 kg seed. Forage was sampled after 28-77 days and grain and straw at harvest after 154 to 189 days.

In Brazil two field trials were conducted on wheat (seed treatment at 0.049 and 0.096 kg ai/100 kg seed).

Table 122. Residues in wheat after single seed treatments in Europe.

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Crop	Form.	kg ai/100 kg seed	kg ai/ha			Imidacloprid	Total	
UK 1990	winter	350 FS	0.07		forage	152		0.12	0058-90
						195		<0.05	
					grain	262	<0.01	<0.05	
					straw	262	<0.05	<u>0.11</u>	
France 1990	winter	70 WS	0.07	0.088	grain	301	<0.01	<0.05	0552-90
					straw	301	<0.1	<0.05	
France 1990	winter	350 FS	0.07	0.088	grain	301	<0.01	<0.05	0557-90
					straw	301	<0.1	<0.05	
France 1990	winter	70 WS	0.07	0.07	grain	274	<0.01	<0.05	0677-90
					straw	274	<0.05	<u>0.09</u>	
France 1990	winter	350 FS	0.07	0.11	grain	274	<0.01	<0.05	0680-90
					straw	274	<0.1	<0.05	
Germany 1990	spring	350 FS	0.07	0.19	forage	41		0.29	PF3736 0080-90
						46		0.18	
						62		<u>0.05</u>	
					grain	136	<0.01	<0.05	
Germany 1990	spring	350 FS	0.07	0.17	straw	136	<0.1	<0.05	PF3736 0081-90
					forage	54		0.12	
						60		0.07	
						66		<u>0.07</u>	
Germany 1991	spring	350 FS	0.07	0.21	grain	135	<0.01	<0.05	RA-2080/91 0073-91
					straw	135	<0.1	<u>0.13</u>	
					forage	56		0.5	
						66		0.18	
Germany 1991	spring	350 FS	0.07	0.16		77		<u>0.12</u>	RA-2080/91 0074-91
					grain	163	<0.01	<0.05	
					straw	163	0.05	<u>0.11</u>	
					forage	46		0.59	
UK 1991	spring	350 FS	0.11	0.23		64		0.15	RA-2129/91 0161-91
					grain	72		<u>0.1</u>	
					straw	150	<0.01	<0.05	
						150	<0.05	<u>0.08</u>	
UK 1991	spring	350 FS	0.07	0.15	treated seed	0			RA-2129/91 0162-91
					forage	63		<u>0.39</u>	
					grain	159	<0.01	<0.05	
					straw	159	<0.05	<u>0.23</u>	
UK 1991	spring	350 FS	0.07	0.15	forage	0		<u>0.19</u>	RA-2129/91 0162-91
					grain	64		<0.05	
					straw	160	<0.01	<0.05	
						160	0.05	<u>0.21</u>	

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Crop	Form.	kg ai/100 kg seed	kg ai/ha			Imidacloprid	Total	
Germany 1997	spring	87.5 FS	0.035		treated seed forage grain straw	-13 51 139 139	390 0.01 <0.01 <0.05	<u>0.1</u> <0.05 <0.05	RA-2078/97 0281-97
Germany 1997	spring	87.5 FS	0.035		treated seed forage grain straw	-13 43 133 133	390 <0.01 <0.01 <0.05	<0.05 <0.05 <u>0.09</u>	RA-2078/97 0347-97

Table 123. Residues in wheat after single seed treatments in Australia and Brazil.

Country, Year	Application		Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	kg ai/ 100 kg seed				
Australia 1993	350 FS	0.07	forage	28 42 56 63	0.88 0.09 0.03 <u>0.02</u>	60/93 AUS-60-93-A
			forage, residue calculated on dry weight basis	28 42 56 63	7.3 0.7 0.3 0.2	
			grain	154	<0.05	
			straw	154	<0.05	
			forage	28 42 56 63	0.9 0.07 0.05 <u>0.03</u>	
			forage, residue calculated on dry weight basis	28 42 56 63	7.4 0.6 0.4 0.3	
			grain	154	<0.05	
			straw	154	<u>0.06</u>	
Australia 1993	350 FS	0.07	forage	42	0.59	74/93 AUS-74-93-A
			forage, residue calculated on dry weight basis	42	3.0	
			grain	189	<0.05	

Country, Year	Application		Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	kg ai/ 100 kg seed				
			straw	189	<u>0.05</u>	
Australia 1993	350 FS	0.14	forage	42	1.0	74/93 AUS-74-93-B
			forage, residue calculated on dry weight basis	42	5.2	
			grain	189	< <u>0.05</u>	
			straw	189	<u>0.06</u>	
Australia 1993	350 FS	0.28	forage	42	1.0	74/93 AUS-74-93-C
			forage, residue calculated on dry weight basis	42	5.1	
			grain	189	<0.05	
			straw	189	0.21	
Australia 1993	350 FS	0.018	forage	control 29	0.03 0.18	KGW47/93 AUS-KGW-47-93- A
			forage, residue calculated on dry weight basis	control 29	0.2 1.1	
			grain	171	<0.05	
			straw	171	0.05	
Australia 1993	350 FS	0.035	forage	control 29	0.03 0.36	KGW47/93 AUS-KGW-47-93- B
			forage, residue calculated on dry weight basis	control 29	0.2 2.2	
			grain	171	<0.05	
			straw	171	<0.05	
Australia 1993	350 FS	0.07	forage	control 29	0.03 0.81	KGW47/93 AUS-KGW-47-93- C
			forage, residue calculated on dry weight basis	control 29	0.2 4.9	
			grain	171	< <u>0.05</u>	
			straw	171	<u>0.05</u>	
Australia 1993	350 FS	0.14	forage	control 29	0.03 0.85	KGW47/93 AUS-KGW-47-93- D
			forage, residue calculated on dry weight basis	control 29	0.2 5.2	

Country, Year	Application		Sample	PHI (days)	Total residue (mg/kg)	<i>Report no.</i> <i>Study no.</i>
	Form.	kg ai/ 100 kg seed				
			grain	171	< <u>0.05</u>	
			straw	171	<u>0.05</u>	
Australia 1994	600 FS	0.072	forage	28	0.84	18/94 AUS-18-94-A
				42	0.3	
				56	0.08	
				63	<u>0.11</u>	
			forage, residue calculated on dry weight basis	28	1.8	
				42	1.9	
				56	0.52	
				63	0.71	
grain	181	< <u>0.05</u>				
	181	<u>0.24</u>				
Australia 1994	600 FS	0.14	forage	28	1.8	18/94 AUS-18-94-B
				42	0.52	
				56	0.13	
				63	<u>0.19</u>	
			forage, residue calculated on dry weight basis	28	3.6	
				42	3.4	
				56	0.84	
				63	1.2	
grain	181	< <u>0.05</u>				
	181	<u>0.45</u>				
Australia 1994	600 FS	0.29	forage	28	8.3	18/94 AUS-18-94-C
				42	1.1	
				56	0.4	
				63	0.49	
			forage, residue calculated on dry weight basis	28	17	
				42	7.0	
				56	2.5	
				63	3.2	
grain	181	<0.05				
	181	0.22				
Australia 1994	600 FS	0.072	forage	28	0.42	27/94 AUS-27-94-A
				42	0.17	
				56	0.13	
				63	<u>0.1</u>	
				77	0.02	

Country, Year	Application		Sample	PHI (days)	Total residue (mg/kg)	Report no. Study no.
	Form.	kg ai/ 100 kg seed				
			forage, residue calculated on dry weight basis	28 42 56 63 77	1.1 0.93 0.71 0.55 0.12	
Australia 1994	600 FS	0.14	forage	28 42 56 63 77	0.89 0.2 0.08 <u>0.09</u> 0.02	27/94 AUS-27-94-B
			forage, residue calculated on dry weight basis	28 42 56 63 77	2.3 1.1 0.43 0.48 0.12	
Australia 1994	600 FS	0.29	forage	28 42 56 63 77	0.92 0.61 0.44 <u>0.27</u> 0.03	27/94 AUS-27-94-C
			forage, residue calculated on dry weight basis	28 42 56 63 77	2.4 3.4 2.4 1.5 0.18	
Brazil 1992	70 WS	0.05	grain	124	<u>0.04</u>	056/93 BRA-056-93-A
Brazil 1992	70 WS	0.1	grain	124	<u>0.05</u>	056/93 BRA-056-93-B

Pecans (Table 124). In 15 US trials the 192 SC and 240 SC formulations were sprayed twice (interval 6-10 days) and in a 16th once. Five trials were with dilute sprays at concentrations of 0.0056-0.007 kg ai/hl, corresponding to 0.19-0.2 kg ai/ha. Five more were with concentrate sprays at concentration rates of 0.024-0.041 kg ai/hl, corresponding to 0.2 kg ai/ha. The remaining six trials were with concentrations from 0.02 to 0.026 kg ai/hl, 0.19-0.2 kg ai/ha. The LOQ was 0.05 mg/kg in report 106777 and 0.01 mg/kg in report 109712 (lowest fortification level).

In seven soil treatment US trials conducted according to GAP the 240 SC formulation was applied as a soil band under the dripline on both sides of the trees, at concentrations of 0.61-0.69 kg ai/hl, corresponding to 0.56-0.58 kg ai/ha.

Table 124. Residues in pecan kernels after spray and soil applications in the USA.

Location Year	Application				PHI (days)	Total residue (mg/kg)	Report no. Study no. Remarks
	Form.	No.	kg ai/ha	kg ai/hl			
Foliar spray application							
TX 1993	240 SC	1	0.19	0.026	31	< <u>0.05</u>	106777 353-CO022-93D
NM 1993	240 SC	2	0.19 ¹	0.026	20	< <u>0.05</u>	106777 353-CO023-93D
OK 1993	240 SC	2	0.2 ¹	0.023	4	< <u>0.05</u>	106777 353-CO024-93D
AL 1993	240 SC	2	0.19 ¹	0.02	21	< <u>0.05</u>	106777 754-CO025-93D
GA 1993	240 SC	2	0.19 ¹	0.02	17	< <u>0.05</u>	106777 755-CO026-93D
GA 1993	240 SC	2	0.19 ¹	0.02	8	< <u>0.05</u>	106777 755-CO027-93D
GA 1998	192 SC	1 1	0.2 0.19	0.007 0.006	0 7 14 21	< <u>0.01</u> < <u>0.01</u> < <u>0.01</u> < <u>0.01</u>	109712 BAY-PO021-98D-A dilute spray
GA 1998	192 SC	1 1	0.2 0.2	0.04 0.04	0 7 14 21	< <u>0.01</u> < <u>0.01</u> < <u>0.01</u> < <u>0.01</u>	109712 BAY-PO021-98D-B concentrate spray
GA 1998	192 SC	1 1	0.2 0.2	0.006 0.006	7	< <u>0.01</u>	109712 BAY-PO022-98H-A dilute spray
GA 1998	192 SC	1 1	0.2 0.2	0.024 0.024	7	< <u>0.01</u>	109712 BAY-PO022-98H-B concentrate spray
LA 1998	192 SC	1 1	0.2 0.19	0.007 0.007	7	< <u>0.01</u>	109712 BAY-PO023-98H-A dilute spray
LA 1998	192 SC	1 1	0.2 0.2	0.034 0.035	7	< <u>0.01</u>	109712 BAY-PO023-98H-B concentrate spray
OK 1998	192 SC	1 1	0.2 0.2	0.007 0.007	7	<u>0.011</u>	109712 BAY-PO024-98H-A dilute spray
OK 1998	192 SC	1 1	0.2 0.2	0.032 0.031	7	< <u>0.01</u>	109712 BAY-PO024-98H-B concentrate spray

Location Year	Application				PHI (days)	Total residue (mg/kg)	Report no. Study no. Remarks
	Form.	No.	kg ai/ha	kg ai/hl			
OK 1998	192 SC	1 1	0.2 0.19	0.006 0.006	7	< <u>0.01</u>	109712 BAY-PO025-98H-A <i>dilute spray</i>
OK 1998	192 SC	1 1	0.2 0.2	0.032 0.032	7	< <u>0.01</u>	109712 BAY-PO025-98H-B <i>concentrate spray</i>
<i>Soil application</i>							
TX 1993	240 SC	1	0.57	0.69	150	< <u>0.05</u>	106777 353-CO028-93H
NM 1993	240 SC	1	0.57	0.69	150	< <u>0.05</u>	106777 353-CO029-93H
OK 1993	240 SC	1	0.58	0.62	109	< <u>0.05</u>	106777 353-CO030-93H
LA 1993	240 SC	1	0.56	0.65	127	< <u>0.05</u>	106777 355-CO031-93H
AL 1993	240 SC	1	0.57	0.61	115	< <u>0.05</u>	106777 754-CO032-93H
GA 1993	240 SC	1	0.58	0.65	102	< <u>0.05</u>	106777 755-CO033-93H
GA 1993	240 SC	1	0.58	0.65	99	< <u>0.05</u>	106777 755-CO034-93H

¹ Silwet L-77 silicone spray adjuvant was tank-mixed with each foliar application

Cotton (seed treatment, Table 125). One trial in Greece was with 350 FS formulation at 0.7 kg ai/100 kg seed.

In four trials in Brazil using the 70 WS formulation, two were according to GAP and the other two at double the maximum GAP rate of 0.35 kg ai/100 kg seed.

Two trials in Egypt with the 70 WS formulation were according to GAP in Egypt, 0.49 kg ai/100 kg seed.

In ten trials in Australia the 70 WS and 350 FS formulations were used at rates of 0.7-2.8 kg ai/100 kg seed.

Table 125. Residues in cotton after seed treatment.

Country Year	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/100 kg seed			Imidacloprid	Total	

Country Year	Application			Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No.	kg ai/100 kg seed			Imidacloprid	Total	
Greece 1992	350 FS	1	0.7	seed	157	<0.01	< <u>0.05</u>	0106-92
Brazil 1991	70 WS	1	0.35	seed	161	0.03		054/93 BRA-054-93-A
Brazil 1991	70 WS	1	0.7	seed	161	0.04		054/93 BRA-054-93-B
Brazil 1992	70 WS	1	0.35	seed	183	<0.01	< <u>0.05</u>	0691-92
Brazil 1992	70 WS	1	0.7	seed	183	<0.01	< <u>0.05</u>	0693-92
Egypt 1992	70 WS	1	0.49	seed	198	<0.01	<u>0.09</u>	0001-92
Egypt 1992	70 WS	1	0.49	seed	195	<0.01	<u>0.06</u>	0105-92
Australia 1989	70 WS	1	0.12	seed	202	<0.01		29-90 AUS-29-90-A
Australia 1989	70 WS	1	0.25	seed	202	<0.01		29-90 AUS-29-90-B
Australia 1989	70 WS	1	0.49	seed	202	<0.01		29-90 AUS-29-90-C
Australia 1992	350 FS	1	1.4	fuzzy seed	154	<0.01		B31/92 AUS-31-92-B
Australia 1992	350 FS	1	2.8	fuzzy seed	154	<0.01		B31/92 AUS-31-92-C
Australia 1992	350 FS	1	0.7	fuzzy seed	154	<0.01		B31/92 AUS-31-92-A
Australia 1992	350 FS	1	0.7	seed	158	<0.01		B14/93 AUS-14-93-A
Australia 1992	350 FS	1	1.4	seed	158	<0.01		B14/93 AUS-14-93-B
Australia 1993	350 FS	1	0.7	seed	138	<0.01		KGW045/93 AUS-KGW045-93-A
Australia 1993	350 FS	1	1.4	seed	138	<0.01		KGW045-93 AUS-KGW045-93-B

Cotton (various application methods, Table 126). Two trials in Spain were conducted with two foliar sprays of the 200 SL formulation at 0.022 kg ai/hl for the first and 0.033 kg ai/hl for the second, 0.1 and 0.15 kg ai/ha respectively.

In a trial in South Africa four foliar spray applications were made at 0.04 kg ai/hl, 0.08 kg ai/ha.

In 26 trials in the USA the 2.5 GR and/or the 240 SC formulation were used. Four applications were made in 23 trials and six in the other three, the first at 0.25 kg ai/100 kg seed, the second in-furrow to soil at planting (0.03 g ai/m row) and the rest as foliar sprays with or without the adjuvant Silwet. One trial, 752-N3077-91D, did not contain any documentation about the seed treatment. Seed treatment applications of imidacloprid in cotton are not currently registered in the USA but residue levels should not be affected, since historically seed treatment applications have resulted in negligible levels of residues when compared to in-furrow or foliar applications.

Fourteen of the trials were conducted with both 2.5 GR (in furrow) and 240 SC (seed and foliar) formulations. Foliar applications without spray adjuvant were at 0.26-1.39 kg ai/hl, 0.26 kg ai/ha, and with Silwet 0.3-0.35 kg ai/hl, 0.26 kg ai/ha. The other 12 trials were conducted with only 240 SC formulation for seed, foliar and in-furrow treatments. Foliar applications without spray adjuvant were at 0.22-1.4 kg ai/hl, 0.26 kg ai/ha, and with adjuvant 0.14 kg ai/hl for the three trials with four foliar sprays and 0.35 kg ai/hl for the others, corresponding to 0.13 kg ai/ha and 0.26 kg ai/ha.

Table 126. Residues in cotton from various application methods.

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Foliar spray application									
Spain 1994	200 SL	2	0.1 0.15	0.02 0.03	seed	30 44	<0.01 <0.01	0.95 0.6	RA-2085/94 0347-94
Spain 1994	200 SL	2	0.1 0.15	0.02 0.03	seed	32	<0.01	0.49	RA-2085/94 0348-94
South Africa 1992	200 SL	4	0.08	0.04	forage seed	0 7 14 21 28 70	0.06 <0.02 <0.02 <0.02 <0.02 <0.02		311/88333/J123 88333-J123-A1
Various application methods, dual formulation trials									
USA, TX 1991	240 SC	1	0.25 ¹	1.4	seed	7		0.87	103824 352-N3075-91D
	2.5 GR	1	0.33 ²			14		1.1	
	240 SC	2	0.26 ³			21		2.3	
					forage	7 14 21		8.9 4.2 3.4	
USA, AR 1991	240 SC	1	0.25 ¹	0.28 0.26	seed	7		0.38	103824 354-N3076-91D
	2.5 GR	1	0.33 ²			14		0.2	
	240 SC	1	0.26 ³			20		0.19	
	240 SC	1	0.26 ³		forage	7 14 20		15 9.2 5.9	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, SC 1991	240 SC	1	0.25 ¹	0.28	forage	7		26	103824 752-N3077-91D
	2.5 GR	1	0.31 ²			14		10	
	240 SC	2	0.26 ³			21		13	
					seed	7		0.38	
						14		1.3	
						21		2.5	
USA, MS 1991	240 SC	1	0.25 ¹	1.4	forage	7		6.8	103824 BMS-N3078-91D
	2.5 GR	1	0.31 ²			14		2.6	
	240 SC	2	0.26 ³			21		3.9	
					seed	7		0.11	
						14		0.15	
						21		0.18	
USA, CA 1991	240 SC	1	0.25 ¹	0.28	forage	7		11	103824 FCA-N3079-91D
	2.5 GR	1	0.3 ²			14		6.8	
	240 SC	2	0.26 ³			21		2.2	
					seed	7		0.3	
						14		0.36	
						21		0.23	
USA, GA 1991	240 SC	1	0.25 ¹	0.33 0.32	forage	7		3.4	103824 TGA-N3080-91D
	2.5 GR	1	0.33 ²			14		1.3	
	240 SC	1	0.26 ³			21		0.92	
	240 SC	1	0.26 ³		seed	7		0.2	
						14		0.16	
						21		0.17	
USA, TX 1992	240 SC	1	0.25 ¹	0.31	seed	8		1.3	103824-1 352-N3192-92D-A
	2.5 GR	1	0.31 ²			15		1.5	
	240 SC	2	0.26 ³			22		2.0	
					forage	8		5.8	
						15		2.9	
						22		0.71	
USA, MS 1992	240 SC	1	0.25 ¹	0.31 0.3	forage	7		25	103824-1 BMS-N3193-92D-A
	2.5 GR	1	0.31 ²			14		16	
	240 SC	1	0.26 ³			21		8.6	
	240 SC	1	0.26 ³		seed	7		0.15	
						14		0.14	
						21		0.19	
USA, CA 1992	240 SC	1	0.25 ¹	0.31	forage	7		22	103824-1 FCA-N3194-92D-A
	2.5 GR	1	0.3 ²			14		4.0	
	240 SC	2	0.26 ³			21		3.3	
					seed	7		0.54	
						14		0.44	
						21		0.33	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, GA 1992	240 SC	1	0.25 ¹	0.35 0.34	seed	7		0.53	103824-1 TGA-N3195-92D-A
	2.5 GR	1	0.33 ²			13		0.55	
	240 SC	1	0.26 ³		forage	21	0.44		
	240 SC	1	0.26 ³			7	6.7		
					13	2.7			
					21	2.0			
Various application methods, dual formulation trials with spray adjuvant									
USA, TX 1992	240 SC	1	0.25 ¹	0.31	forage	8		6.4	103824-1 352-N3192-92D-B
	2.5 GR	1	0.31 ²			15		3.9	
	240 SC	2	0.26 ⁴		seed	22	1.4		
						8	3.3		
					15	4.3			
					22	5.1			
USA, MS 1992	240 SC	1	0.25 ¹	0.31 0.3	seed	7		0.22	103824-1 BMS-N3193-92D-B
	2.5 GR	1	0.31 ²			14		0.26	
	240 SC	1	0.26 ⁴		forage	21	0.24		
	240 SC	1	0.26 ⁴			7	27		
					14	25			
					21	5.0			
USA, CA 1992	240 SC	1	0.25 ¹	0.31	seed	7		0.43	103824-1 FCA-N3194-92D-B
	2.5 GR	1	0.3 ²			14		0.51	
	240 SC	2	0.26 ⁴		forage	21	0.32		
						7	34		
					14	2.6			
					21	2.3			
USA, GA 1992	240 SC	1	0.25 ¹	0.35 0.34	forage	7		4.3	103824-1 TGA-N3195-92D-B
	2.5 GR	1	0.33 ²			13		3.7	
	240 SC	1	0.26 ⁴		seed	21	2.4		
	240 SC	1	0.26 ⁴			7	1.5		
					13	0.7			
					21	0.93			
Various application methods, single formulation trials									
USA, GA 1992	240 SC	1	0.25 ¹	0.65 0.35 0.34	forage	7		9.0	103824-1 TGA-N3198-92D-A
		1	0.33 ²			13		5.5	
		1	0.26 ³		seed	21	5.7		
		1	0.26 ³			7	0.78		
					13	0.79			
					21	0.83			
USA, MS 1991	240 SC	1	0.25 ¹	0.54 1.4	seed	7		0.15	103824 BMS-N3072-91D
		1	0.31 ²			14		0.2	
		2	0.26 ³		forage	21	0.22		
						7	6.5		
					14	2.7			
					21	3.5			

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, CA 1991	240 SC	1	0.25 ¹	0.64	forage	7		18	103824 FCA-N3073-91D
		1	0.3 ²			14		5.2	
		2	0.26 ³	0.28		21		4.6	
					seed	7		0.41	
						14		0.38	
						21		0.41	
USA, GA 1991	240 SC	1	0.25 ¹	0.72	forage	7		2.5	103824 TGA-N3074-91D
		1	0.33 ²			14		1.2	
		1	0.26 ³	0.33		21		1.1	
		1	0.26 ³	0.22	seed	7		0.25	
						14		0.19	
						21		0.2	
USA, CA 1992	240 SC	1	0.25 ¹	0.64	seed	7		0.45	103824-1 FCA-N3197-92D-A
		1	0.3 ²			14		0.55	
		2	0.26 ³	0.31		21		0.21	
					forage	7		19	
						14		3.2	
						21		2.0	
USA, MS 1992	240 SC	1	0.25 ¹	0.6	forage	7		38	103824-1 BMS-N3196-92D-A
		1	0.31 ²			14		16	
		2	0.26 ³	0.31		21		11	
					seed	7		0.12	
						14		0.16	
						21		0.21	
<i>Various application methods, single formulation trials with spray adjuvant</i>									
USA, CA 1992	240 SC	1	0.25 ¹	0.64	seed	7		0.53	103824-1 FCA-N3197-92D-B
		1	0.3 ²			14		0.48	
		2	0.26 ⁴	0.31		21		0.26	
					forage	7		19	
						14		3.2	
						21		4.3	
USA, MS 1992	240 SC	1	0.25 ¹	0.6	seed	7		0.19	103824-1 BMS-N3196-92D-B
		1	0.31 ²			14		0.24	
		2	0.26 ⁴	0.31		21		0.23	
					forage	7		54	
						14		20	
						21		9.2	
USA, GA 1992	240 SC	1	0.25 ¹	0.65	forage	7		5.63	103824-1 TGA-N3198-92D-B
		1	0.33 ²			13		4.95	
		1	0.26 ⁴	0.35		21		11.37	
		1	0.26 ⁴	0.34	seed	7		1.02	
						13		0.65	
						21		0.69	

Country Year	Application				Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			Imidacloprid	Total	
USA, OK 1994	240 SC	1	0.25 ¹	0.66 0.14	seed	20		<1	103824-2 456-AD025-94D
		1	0.31 ²		gintrash	20		2.5	
		4	0.13 ⁴		gin by-products	20		2.4	
USA, TX 1994	240 SC	1	0.25 ¹	0.66 0.14	seed	13		<1.0	103824-2 456-AD026-94D
		1	0.31 ²		gintrash	13		4.0	
		4	0.13 ⁴		gin by-products	13		3.4	
USA, TX 1994	240 SC	1	0.25 ¹	0.63 0.14	seed	14		<1.0	103824-2 456-AD058-94D
		4	0.3 ² 0.13 ⁴		gin by-products	14		3.5	

¹ kg ai/dt seed (seed treatment)

² soil in-furrow application: 0.03 g ai/m row

³ foliar spray

⁴ foliar spray using Silwet L-77 as a spray adjuvant

Rape (Table 127). The use of imidacloprid is authorized as seed treatment in Australia, Germany and the UK.

In northern Europe four trials were conducted in Sweden in 1990, two on summer and two on winter rape using the 70 WS formulation as a dressing at 1.4 kg ai/100 kg seed, and three trials in 1993 on winter rape in France with the 70 WS formulation at 0.011 kg ai/ha, 1.05 kg ai/100 kg seed. In 1996 or 1997 in four trials in Germany, two in the UK, two in northern France and four in southern France the 500 FS was used in half the trials and the 600 FS formulation in the other half at 0.053 kg ai/ha, 1.05 kg ai/100 kg seed.

Three trials performed in 1993 in Australia were with the 350 FS formulation at 0.12, 0.25 and 0.5 kg ai/100 kg seed.

Table 127. Residues in rape after seed treatments.

Country Year	Crop	Application			Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
		Form.	No.	kg ai/ha			Imidacloprid	Total	
Sweden 1990	summer	70 WS	1	1.4 ¹	seed straw	152 152	<0.01 <0.1	<0.05 0.14	0392-90
Sweden 1990	summer	70 WS	1	1.4 ¹	seed straw	132 132	<0.01 <0.1	<0.05 0.1	0398-90

Country Year	Crop	Application			Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
		Form.	No.	kg ai/ha			Imidacloprid	Total	
Sweden 1990	winter	70 WS	1	1.4 ¹	seed straw	338 338	<0.01 <0.1	<0.05 0.05	0394-90
Sweden 1990	winter	70 WS	1	1.4 ¹	seed straw	337 337	<0.01 <0.1	<0.05 <0.05	0399-90
France, North 1993	winter	70 WS	1	0.011 ²	seed	304	<0.01	<0.05	RA-2073/93 0092-93
France, North 1993	winter	70 WS	1	0.011 ²	treated seed seed	0 ³ 311	8510 <0.01	<0.05	RA-2073/93 0405-93
France, North 1993	winter	70 WS	1	0.011 ²	treated seed seed	0 ³ 311	8410 <0.01	<0.05	RA-2073/93 0454-93
France, North 1996	winter	500 FS	1	0.053 ²	treated seed forage seed	-58 ³ 81 179 210 312	8850 0.01 <0.01 <0.01 <0.01	0.08 <0.05 <0.05 <0.05	RA-2173/96 0830-96
France, North 1996	winter	600 FS	1	0.053 ²	treated seed forage seed	-58 ³ 81 312	11730 0.01 <0.01	0.09 <0.05	RA-2174/96 0835-96
Germany 1997	winter	500 FS	1	0.053 ²	treated seed forage seed	-54 ³ 59 192 226 310	8850 0.02 <0.01 <0.01 <0.01	0.06 <0.05 <0.05 <0.05	RA-2173/96 0825-96
Germany 1996	winter	500 FS	1	0.053 ²	treated seed forage seed	-48 ³ 59 198 232 336	8850 <0.01 <0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05	RA-2173/96 0828-96
Germany 1996	winter	600 FS	1	0.053 ²	treated seed forage seed	-54 ³ 59 310	11730 0.02 <0.01	0.09 <0.05	RA-2174/96 0826-96
Germany 1996	winter	600 FS	1	0.053 ²	treated seed forage seed	-48 ³ 59 336	11730 <0.01 <0.01	<0.05 <0.05	RA-2174/96 0833-96
UK 1996	winter	500 FS	1	0.053 ²	treated seed forage seed	-69 ³ 154 185 197 321	8850 <0.01 <0.01 <0.01 <0.01	<0.05 <0.05 <0.05 <0.05	RA-2173/96 0829-96
UK 1996	winter	600 FS	1	0.053 ²	treated seed forage seed	-69 ³ 154 321	11730 <0.01 <0.01	<0.05 <0.05	RA-2174/96 0834-96

Country Year	Crop	Application			Sample	PHI (days)	Residues, mg/kg		Report no. Study no.
		Form.	No.	kg ai/ha			Imidacloprid	Total	
France, South 1996	winter	500 FS	1	0.053 ²	treated seed	-51 ³	8850		RA-2173/96 0831-96
					forage	94	<0.01	<0.05	
					seed	189	0.01	<0.05	
						220	<0.01	<0.05	
France, South 1996	winter	500 FS	1	0.053 ²	treated seed	-61 ³	8850		RA-2173/96 0832-96
					forage	93	<0.01	<0.05	
					seed	179	0.01	<0.05	
						210	<0.01	<0.05	
France, South 1996	winter	600 FS	1	0.053 ²	treated seed	-51 ³	11730		RA-2174/96 0837-96
					forage	94	<0.01	<0.05	
					seed	312	<0.01	<0.05	
France, South 1996	winter	600 FS	1	0.053 ²	treated seed	-61 ³	11730		RA-2174/96 0838-96
					forage	93	0.01	<0.05	
					seed	302	<0.01	<0.05	
Australia, 1993	Rape	350 FS	1	0.12 ¹	forage calculated on dry weight basis	42 56 63		0.6 <0.05 0.2	52/93 AUS-52-93-A
					forage	42 56 63		0.06 <0.05 <0.05	
					straw	154		<0.05	
					seed	154		<0.05	
Australia, 1993	Rape	350 FS	1	0.25 ¹	forage calculated on dry weight basis	42 56 63		0.3 <0.05 0.2	52/93 AUS-52-93-B
					forage	42 56 63		<0.05 <0.05 <0.05	
					straw	154		<0.05	
					seed	154		<0.05	
Australia, 1993	Rape	350 FS	1	0.5 ¹	forage calculated on dry weight basis	42 56 63		0.5 0.2 0.5	52/93 AUS-52-93-C
					forage	42 56 63		0.05 <0.05 0.05	
					straw	154		0.06	
					seed	154		<0.05	

¹ kg ai/100 kg seed² 1.05 kg ai/100 kg seed³ Days between treatment and sowing

Hops (Tables 128 and 129). The use of imidacloprid as 70 WG, 200 SL and 192 SC formulations is registered in Germany, Spain, the UK and the USA for foliar spray and stem applications to hops.

In Germany in eight trials according to GAP using the 70 WG formulation leaves were sprayed at rates of 0.0035 and 0.004 kg/hl, 0.12 and 0.13 kg ai/ha. Cones were sampled on days 0, 14, 28, 35 and 42. The green cones were dried according to local practice for 7-8 hours at 60°C until the water content of the dried cones was 10-12%.

In eight trials in the UK single basal sprays of a 70 WG formulation at 0.13 kg ai/ha were used: in two in 1992 the rate was 0.13 kg ai/ha, 0.03 kg ai/hl, and in the two in 1993 again 0.13 kg ai/ha, as a spot treatment to the base of the plants at 0.025-0.03 kg ai/hl. Depending on the density of the plants, the treatments corresponded to 0.025-0.03 g ai/plant. In the four trials in 2000 rates of 0.13 kg ai/ha were used and each plant was treated with 0.029-0.055 g ai in 100 ml water. The treated and untreated trial plots from trial 0066-00 were treated erroneously with 0.18 kg "Admire"/ha 16 days before the start of the trial. Cone samples were taken at harvest 103 to 120 days after treatment. In 6 trials, the green cones were dried overnight in kilns. No information was available on the drying process for trials 0175-92 and 0176-92.

Three US trials were conducted on hops in Washington, Oregon and Idaho because these states include nearly all of the total commercial US production. Three foliar applications were made at approximately 21-day intervals of a 240 SC formulation tank mixed with an organosilicone-based surfactant at 0.11 kg ai/ha each.

In four trials in Germany (Table 129) single brush applications were made at 2.3 kg ai/hl, 0.14 kg ai/ha.

Table 128. Residues in hops after foliar or basal sprays.

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Germany 1991	70 WG	1	0.13	0.004	cone, green	0	<0.2	9.1	RA-2131/91 0075-91
						14		1.9	
						28		0.78	
						35		<0.2	
						42		0.2	
					cone, kiln-dried	35	<0.2	<0.2 ¹	
						42		<u>0.48</u>	
Germany 1991	70 WG	1	0.13	0.004	cone, green	0	<0.2	8.3	RA-2131/91 0076-91
						14		1.1	
						28		0.31	
						35		<0.2	
						42		<0.2	
					cone, kiln-dried	35	<0.2	0.48	
						42		<u>0.59</u>	

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
Germany 1991	70 WG	1	0.13	0.004	cone, green	0 14 28 35 42	<0.2	5.4 0.78 0.2 0.43 <0.2	RA-2131/91 0077-91
					cone, kiln-dried	35 42		<u>1.2</u> 0.51	
Germany 1991	70 WG	1	0.13	0.004	cone, green	0 14 28 35 42	<0.2	11 1.7 0.5 0.24 <0.2	RA-2131/91 0078-91
					cone, kiln-dried	35 42		<u>0.73</u> 0.65	
Germany 1992	70 WG	1	0.12	0.0035	whole plant without roots	0		1.1	RA-2063/92 0049-92
					cone, kiln-dried	35 42	<0.2	<u>1.3</u> 1.0	
					cone, green	14 28 35 42		0.83 0.49 0.26 0.19	
Germany 1992	70 WG	1	0.12	0.0035	whole plant without roots	0		2.3	RA-2063/92 0050-92
					cone, kiln-dried	35 42	<0.2	<u>0.81</u> 0.7	
					cone, green	14 28 35 42		0.89 0.27 0.17 0.18	
Germany 1992	70 WG	1	0.12	0.0035	cone, green	14 28 35 42	<0.05	0.64 0.43 0.21 0.12	RA-2063/92 0051-92
					cone, kiln-dried	35 42		<u>0.73</u> 0.55	
					whole plant without roots	0		4.2	
Germany 1992	70 WG	1	0.12	0.0035	cone, green	14 28 35 42	<0.05	0.7 0.74 0.25 0.27	RA-2063/92 0052-92
					cone, kiln-dried	35 42		<u>1.2</u> <u>1.6</u>	

Country Year	Application				Sample	PHI (days)	Residue, mg/kg		Report no. Study no.
	Form.	No	kg ai/ha	kg ai/hl			Imidacloprid	Total	
					whole plant without roots	0		4.1	
UK 1992	70 WG	1	0.13	0.03	cone, green	103	<0.05	0.06	0175-92
					cone, kiln-dried	103	<0.2	<u>0.25</u>	
UK 1992	70 WG	1	0.13	0.03	cone, green	107	<0.05	0.07	0176-92
					cone, kiln-dried	107	<0.2	<u>0.29</u>	
UK 1993	70 WG	1	0.13	0.03	cone, green	119	<0.05	<0.05	RA-2035/93 0002-93
					cone, kiln-dried	119	<0.2	< <u>0.2</u>	
UK 1993	70 WG	1	0.13	0.025	cone, green	113	<0.05	<0.05	RA-2035/93 0402-93
					cone, kiln-dried	113	<0.2	< <u>0.2</u>	
UK 2000	70 WG	1	0.13	0.034	cone, kiln-dried	118	0.21	<u>0.7</u>	RA-2085/00 0066-00
UK 2000	70 WG	1	0.13	0.029	cone, kiln-dried	103	<0.2	< <u>0.2</u>	RA-2085/00 0412-00
UK 2000	70 WG	1	0.13	0.053	cone, kiln-dried	107	<0.2	< <u>0.2</u>	RA-2085/00 0413-00
UK 2000	70 WG	1	0.13	0.055	cone, kiln-dried	120	<0.2	< <u>0.2</u>	RA-2085/00 0414-00
USA 1993	240 SC	3	0.11	0.012	cone, dried ²	28 28		<u>5.8</u> 3.4	106683, USA- 5369.93-ID02
USA 1993	240 SC	3	0.11	0.015	cone, dried ²	27 27		0.82 <u>1.3</u>	106683, USA- 5369.93-OR27
USA 1993	240 SC	1	0.11	0.008	cone, dried ³	28		<u>5.5</u>	106683, USA- 5369.93-WA29
		2	0.11	0.005		28		4.0	

¹ The treated sample seems to have been exchanged with the control (0.27 mg/kg).

² Hand harvested

³ Mechanically picked

Table 129. Residues in hop cones after brush application, Germany, 1992.

Application				Residues				Report no. Study no.
Form.	No	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total	
70 WG	1	0.14 ¹	2.3	green	35 42	<0.05	0.17 0.19	RA-2063/92 0053-92
				kiln-dried	35 42	<0.2	<u>0.83</u> 0.8	

Application				Residues				Report no. Study no.
Form.	No	kg ai/ha	kg ai/hl	Sample	PHI (days)	Imidacloprid	Total	
70 WG	1	0.14 ¹	2.3	green	35 42	<0.05	0.2 0.12	RA-2063/92 0054-92
				kiln-dried	35 42	<0.2	<u>0.75</u> 0.47	
70 WG	1	0.14 ¹	2.3	green	35 42	<0.05	0.15 0.16	RA-2063/92 0055-92
				kiln-dried	35 42	<0.2	0.47 <u>0.52</u>	
70 WG	1	0.14 ¹	2.3	green	35 42	<0.05	0.09 0.07	RA-2063/92 0056-92
				kiln-dried	35 42	<0.2	<u>0.43</u> 0.23	

¹ 0.14 kg ai/6 l water per hectare for a plant density of 3906 to 4608 plants/ha.

Coffee (Table 130). The use of imidacloprid on coffee is registered in Brazil for drench applications of the 70 WG formulation. In six trials single applications were made at 0.035 to 0.36 kg ai/hl, 0.7 to 1.8 kg ai/ha.

Table 130. Residues in coffee beans after drench application at the base of the plant in trials in Brazil.

Year	Application				Residues, mg/kg			Report no.
	Form.	No.	kg ai/ha	kg ai/hl	PHI (days)	Imidacloprid	Total	Study no.
1998	70 WG	1	0.7	0.035	45	<0.02		4784 BRA-4784-A
1998	70 WG	1	1.4	0.07	45	<0.02		4784 BRA-4784-B
1999	70 WG	1	0.7	0.035	45		< <u>0.05</u>	BRA-C-E1-604/99-S2 BRA-C-E1-604/99-S2-A
1999	70 WG	1	1.4	0.07	45		<0.05	BRA-C-E1-604/99-S2 BRA-C-E1-604/99-S2-B
1999	70 WG	1	0.91	0.18	45		< <u>0.05</u>	I-E1-605/99 BRA-I-E1-605-99-A
1999	70 WG	1	1.8	0.36	45		0.05	I-E1-605/99 BRA-I-E1-605-99-B

Tea (Table 131). The use of imidacloprid on tea is registered in Japan as a foliar spray with the 10 WP formulation. In two trials in Japan according to GAP leaves were sprayed once at 0.01 kg ai/hl, 0.2 kg ai/ha, and in two other trials in 1998 plants were sprayed once with a 50 WG formulation.

Table 131. Residues in dried tea leaves after spray application in Japan.

Year, location	Application				PHI (days)	Imidacloprid, mg/kg	Report no. Study no.
	Form.	No.	kg ai/ha	kg ai/hl			
1990 Shizu- oka	10 WP	1	0.2	0.01	13 13 20 20 27 27	1.8 2.3 ¹ 0.54 0.78 ¹ 0.1 0.11 ¹	NR91-47 JAP-NR91-47-A
1990 Miya- zaki	10 WP	1	0.2	0.01	14 14 21 21 28 28	1.4 1.9 ¹ 0.6 0.8 ¹ 0.2 0.17 ¹	NR91-47 JAP-NR91-47-B
1998 Shizu- oka	50 WG	1	0.2	0.01	7 7 14 14 21 21	3 3.8 ¹ 2.3 3.0 ¹ 0.34 0.55 ¹	NR98035 JAP-NR98035-A Saku 109 JAP-Saku 109-A
1998 Miya- zaki	50 WG	1	0.2	0.01	7 7 14 14 21 21	4.0 3.3 ¹ 1.8 1.7 ¹ 1.0 0.97 ¹	NR98035 JAP-NR98035-B Saku 109 JAP-Saku 109-B

¹ Analysed in a second laboratory

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

Imidacloprid is not used on stored products.

In processing

Qualitative effects

Sneikus (2000) studied the hydrolysis of imidacloprid in unsterilized buffered drinking water at pH 4, 5 and 6 with [methylene-¹⁴C]imidacloprid at about 0.2 mg/l, 308 kBq/l, incubated at 90°C (pH 4 for 20 min), 100°C (pH 5 for 60 min) or 120°C (pH 6 for 120 min) to approximate commercial processing of raw agricultural commodities (RACs). Samples were taken before and after heating for the determination of total ¹⁴C by digital autoradiography and the nature of the residues. Analyses were by TLC with confirmation by TLC on RP-18 thin-layer plates. The limit of detection was 0.4% of the applied radioactivity. The identity of detected components was confirmed by co-chromatography.

The recovery of ^{14}C was 100-101% of the applied radioactivity, showing that the radioactivity did not dissipate during incubation. The initial content of imidacloprid was in the range of 98.4-99.8% of the ^{14}C with a mean of 99.1%, and the final values were 98%-99.3% (average 98.8%). The results are shown in Table 132. Imidacloprid was stable under the conditions tested, so it is not expected that hydrolysis will contribute to the degradation of imidacloprid or affect the nature of residues during processing.

Table 132. Degradation of [methylene- ^{14}C]imidacloprid in buffered drinking water (Sneikus, 2000).

Conditions	Time	Recovery %	% of applied radioactivity
pH 4, 90°C, 20 min	zero	100 ¹	98.37
	termination	100.26	97.96
pH 5, 100°C, 60 min	zero	100 ¹	99.13
	termination	100.13	99.31
pH 6, 120°C, 20 min	zero	100 ¹	99.80
	termination	101.02	99.11

¹ Measured values set to 100% representing the radioactivity at zero time

Quantitative effects

Processing trials were conducted on citrus fruits, apples, cherries, grapes, tomatoes, lettuce, green beans, potatoes, rice, wheat, cotton seed, hops and tea. In the US trials only the total residue was determined whereas in the European trials the parent compound was determined as well as the total residue.

Citrus fruits. Four trials were carried out on citrus fruits, three on oranges and one on lemons, the results of which are given in Table 133.

In two field trials on oranges in Italy and Spain, two applications of imidacloprid were made at 0.12 kg ai/ha (Italy) and 0.3-0.6 kg ai/ha (Spain). Oranges were sampled 21 days after the second application for processing into juice and marmalade. Simulated industrial processes on a laboratory scale were used for juice. Thawed and weighed oranges were washed and separated into pulp and peel (ratio 1/9 for treated samples). Subsequently the pulp was pressed at high pressure into pomace and juice. The juice was pasteurized at about 86°C for about 1 min, and samples were stored frozen until analysis (Figure 6).

Marmalade was prepared according to household practices. Thawed and weighed oranges were washed and separated into pulp and peel. The pulp/peel ratio was 1.8 for the treated samples. The pulp was macerated and passed through a sieve. The peel was cut into fine strips and added to the pulp together with sugar and gelling agent. After cooking for 5 min the marmalade was cooled and stored frozen until analysis (Figure 7).

In the Italian lemon study, two foliar sprays at 0.41 kg ai/ha were applied to the trees, and the lemons were picked on day 21 for processing into juice as described above for oranges.

In a single field study in the USA the trees were sprayed twice at 1.4 kg ai/ha, five times the maximum recommended label rate. Oranges were picked 6 days later. Processing into juice, oil, molasses and dried pulp simulated typical commercial practice as closely as possible. A flow chart is given in Figure 8.

Table 133. Effects of processing on residues in citrus fruits treated with imidacloprid.

Crop Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
Orange Spain 1991	fruit ¹	21	0.12		0.2		RA-2019/91
	marmalade		0.10	0.83	0.15	0.75	0195-91
	juice		0.02	0.17	0.05	0.25	
Orange Italy 1991	fruit ¹	21	0.07		0.12		RA-2018/91
	marmalade		0.05	0.71	0.06	0.5	0280-81
	juice		<0.01	<0.14	<0.05	<0.42	
Orange USA 1993	fruit	6			0.19		106771
	pulp, dry				1.42	7.47	VBL-CO014-93P
	juice				<0.05	<0.26	
	oil				<0.05	<0.26	
	molasses				1.23	6.47	
Lemon Italy 1991	fruit ¹	21	0.17		0.26		RA-2018/91
	juice		0.02	0.12	<0.05	<0.19	0281-91

¹ Whole fruit, calculated

Figure 6. Flow diagram for the preparation of juice (trials no. 0195-91, 0280-81, 0281-91).

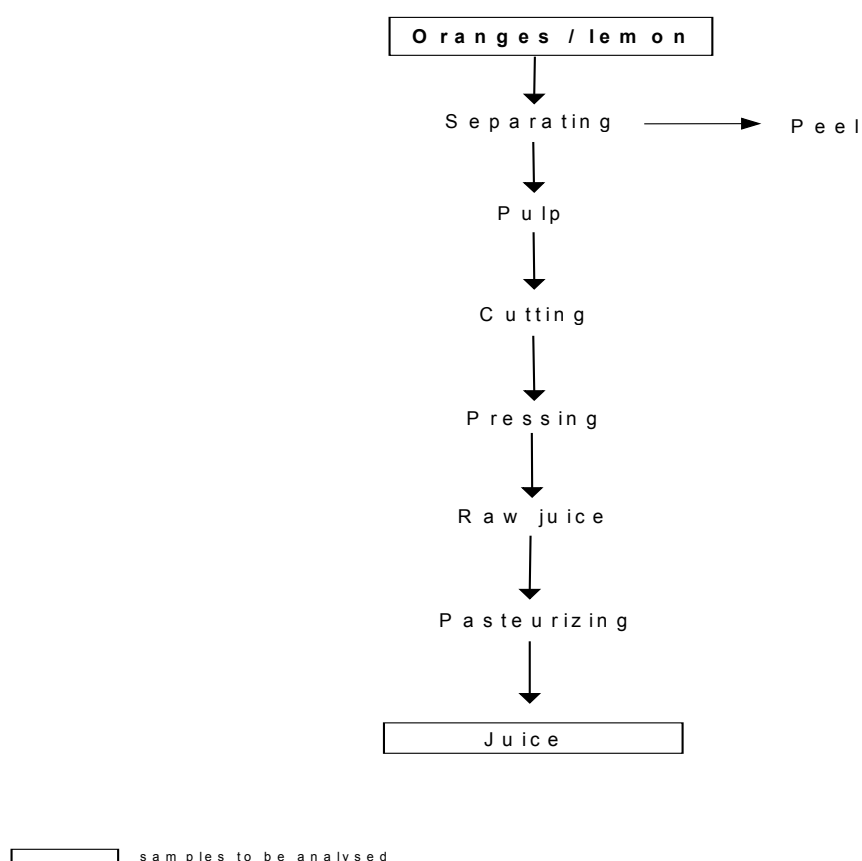


Figure 7. Flow diagram for the preparation of marmalade (trials no. 0195-91, 0280-81).

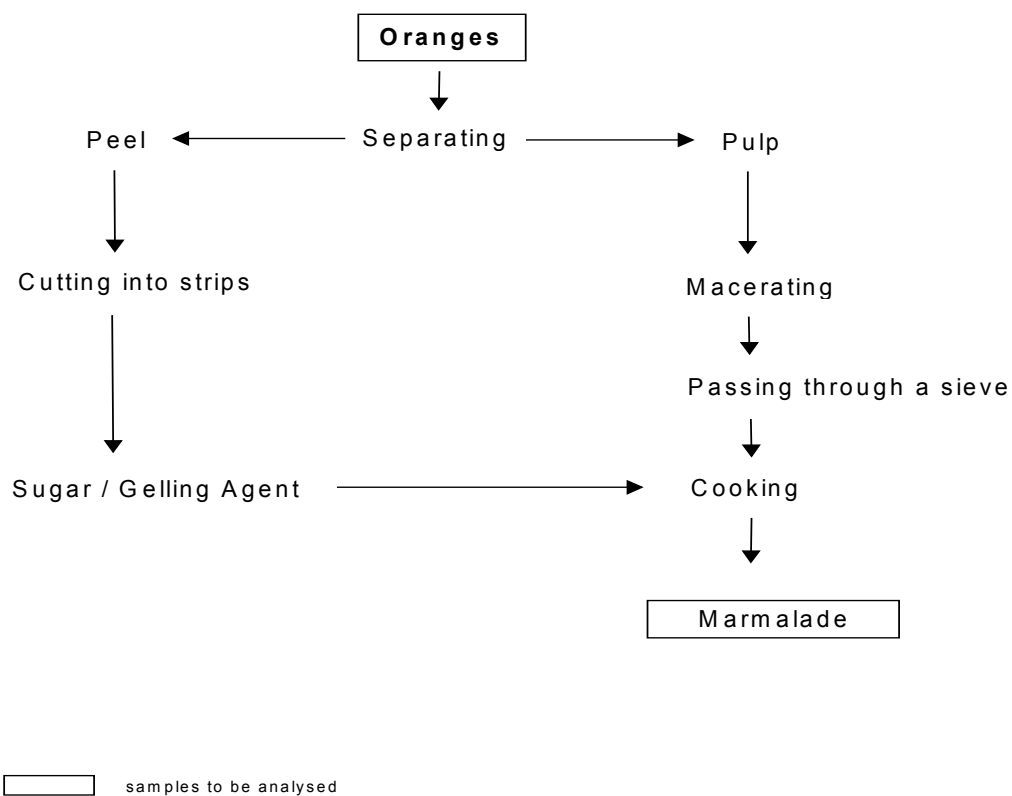
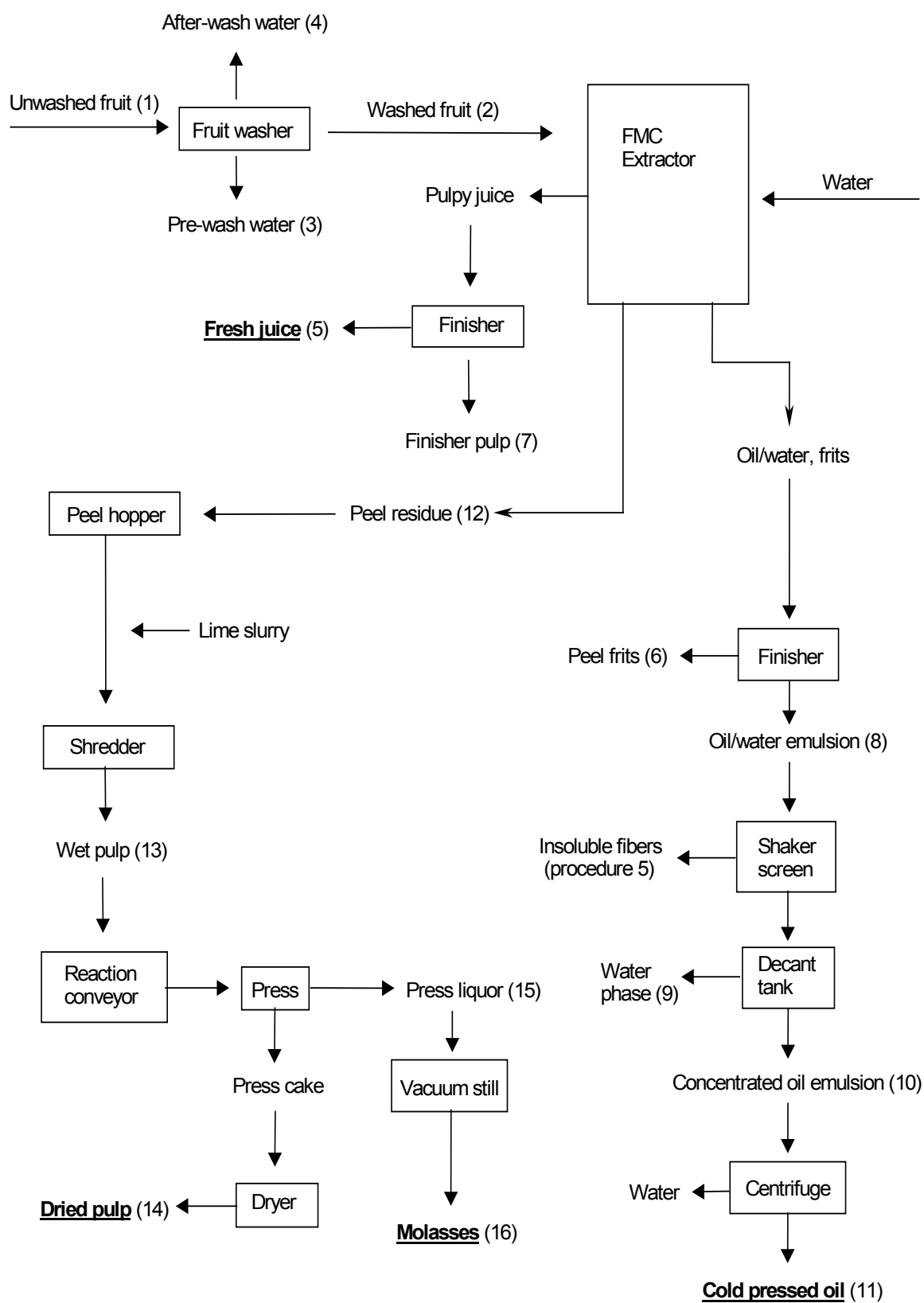


Figure 8. Flow diagram for the processing of oranges (USA study no. VBL-CO014-93P).



Samples analysed

FRACTIONATION STUDY PRODUCT/SAMPLE LIST with brief descriptions (Figure 8)

- (1) UNWASHED FRUIT whole fruit as delivered to pilot plant
- (2) WASHED FRUIT whole fruit washed in packing house
- (3) PRE-WASH WATER initial water rinse of unwashed fruit
- (4) AFTER-WASH WATER water rinse after fruit-cleaning detergent applied
- (5) FRESH JUICE single strength finished juice from washed fruit extracted in FMC 291 B-100 extractor
- (6) PEEL FRITS small (1 mm) bits of peel recovered by finishing during oil extraction
- (7) FINISHER PULP ruptured juice sacs removed from pulpy juice stream by FMC Model 35 juice finisher
- (8) OIL/WATER EMULSION peel oil and aqueous phase of the liquid stream from the peel oil extraction process
- (9) WATER PHASE lower aqueous phase drained from tank in which oil/water emulsion is allowed to separate by standing
- (10) CONCENTRATED OIL EMULSION emulsion of liquid stream from peel oil extraction process after water-phase of emulsion drained off
- (11) COLD-PRESSED OIL citrus oil produced by centrifuging and filtering solids and water from concentrated peel oil emulsion
- (12) PEEL RESIDUE solid fraction from juice extraction that contains peel, membrane, rag, seeds
- (13) WET PULP (chopped peel residue) peel, membrane, rag and seeds uniformly chopped by shredder before drying
- (14) DRIED PULP (dried peel residue) wet pulp limed, reacted, and pressed, then put through feed mill dryer to result in product of 8-10% moisture
- (15) PRESS LIQUOR liquid that has been pressed from the limed reacted wet peel residue
- (16) MOLASSES press liquor concentrated to approximately 50° Brix.

Apples. The results of eight processing trials are shown in Table 134. In six field trials in Germany and one in Italy two foliar sprays were applied at 0.11-0.15 kg ai/ha and apples harvested 14 days later for processing into washed fruit, juice, sauce, dried fruit and dried pomace (see Figure 6). The processing procedures were not reported for the eighth trial (report no. PF-3741) although household practices were used to produce sauce whereas for juice industrial practices were simulated in a laboratory.

In study RA-2066/92 apples were washed and in one trial sauce was prepared by household practices, whereas in two others sauce, juice, and wet and dry pomace were produced by simulated industrial practices in the laboratory.

Apples were washed in slowly agitated standing water, cut into small pieces and stored frozen until analysis. For sauce the apples were blanched at 70°C for 20-30 min, passed through a sieve, put into preserving cans, sugar added and the sauce pasteurised at about 80-90°C before being frozen. In household preparation the cans with apple sauce were heated in a boiling water bath for 20 minutes.

For juice and wet and dry pomace, the apples were washed as described above, cut into small pieces, shredded in a cutter and put into a high-pressure press for processing into juice and wet pomace. The raw juice was pasteurized in a plate heat-exchanger at a temperature up to about 84°C, and stored deep frozen. The pomace was dried at a temperature of 70°C to a water content of 10–20%. In one trial the wet pomace was heated too long resulting in a water content of only 0.5%. The wet and dry pomace were shredded and stored deep frozen until analysis.

For dried apples, washed and peeled fruits were cut into slices, dipped in potassium sulfite (0.01%), then in a citric acid solution (0.01%), washed with water and dried for about 4 hours at about 65-70°C to a water content of 24-28% for frozen storage.

In a field study in the USA trees were sprayed five times at 0.35 kg ai/ha with a 240 SC formulation (five times the recommended rate) and apples were picked 14 days after the last spray. Processing into juice and wet and dry pomace simulated typical commercial practices as closely as possible.

Table 134. Effects of processing on residues in apples treated with imidacloprid, 14-day PHI.

Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report Study no. no.
		mg/kg	Processing factor	mg/kg	Processing factor	
Germany 1990	fruit	0.1		0.11		<i>PF-3741</i> 0186-90
	juice	0.03	0.3	0.05	0.45	
	sauce	0.03	0.3	0.08	0.73	
Germany 1990	fruit	0.03		0.06		<i>PF-3741</i> 0189-90
	juice	0.02	0.67	<0.05	<0.83	
	sauce	0.02	0.67	0.05	0.83	
Germany 1990	fruit	0.04		<0.05		<i>PF-3741</i> 0520-90
	juice	0.02	0.5	<0.05		
	sauce	0.03	0.75	<0.05		
Germany 1990	fruit	0.02		<0.05		<i>PF-3741</i> 0521-90
	juice	<0.01	<0.5	<0.05		
	sauce	0.01	0.5	<0.05		
Germany 1992	fruit	0.03		0.06		<i>RA-2066/92</i> 0102-92
	fruit, washed	0.03	1.0	0.06	1	
	sauce	0.02	0.7	<0.05	<0.83	
	juice	0.01	0.3	<0.05	<0.83	
	fruit, dried	0.03	1.0	<0.05	<0.83	
	pomace, dried	0.13	4.3	0.22	3.7	
Germany 1992	fruit	0.02		<0.05		<i>RA-2066/92</i> 0103-92
	fruit, washed	<0.01	<0.5	<0.05		
	sauce	0.01	0.5	<0.05		
	sauce, heated	0.01	0.5	<0.05		
	juice	0.01	0.5	<0.05		

Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
	pomace, dried	0.05	2.5	0.1	>2	
	fruit, dried	0.04	2.0	<0.05		
Italy 1992	fruit	0.12		0.23		RA-2049/92 0277-92
	fruit, washed	0.07	0.6	0.16	0.7	
	sauce	0.03	0.3	0.14	0.6	
	juice	0.03	0.3	0.1	0.4	
	pomace, dried	0.41	3.4	1.3	5.7	
	fruit, dried	0.09	0.8	0.21	0.9	
USA 1991	fruit			0.13		103236 HIN-N3009-91P
	juice			0.1	0.77	
	pomace, wet			0.21	1.6	
	pomace, dry			0.82	6.3	

Cherry, sweet. In a field trial in Italy and another in Spain trees were sprayed once at 0.01 kg ai/hl, 0.1 kg ai/ha, and samples of mature cherries were collected 14 days later. Samples of the RAC cherry, washed cherries, washing water, cherries without stone, and preserves (canned fruit) were analysed (Table 135). Cherries were washed according to normal household practices and processed into preserves by simulating industrial processes on a laboratory scale.

Cherries were washed in standing water under slow movement, cut into small pieces and stored frozen. For the preserves fruits were washed and depitted with a cherry pitter: some were then stored frozen and the rest put into preserving cans to which a sugar solution was then added before pasteurization (93°C maximum). The preserves were macerated before being stored frozen (Figure 10).

Figure 9. Flow diagram for the preparation of apple juice and wet and dry pomace.

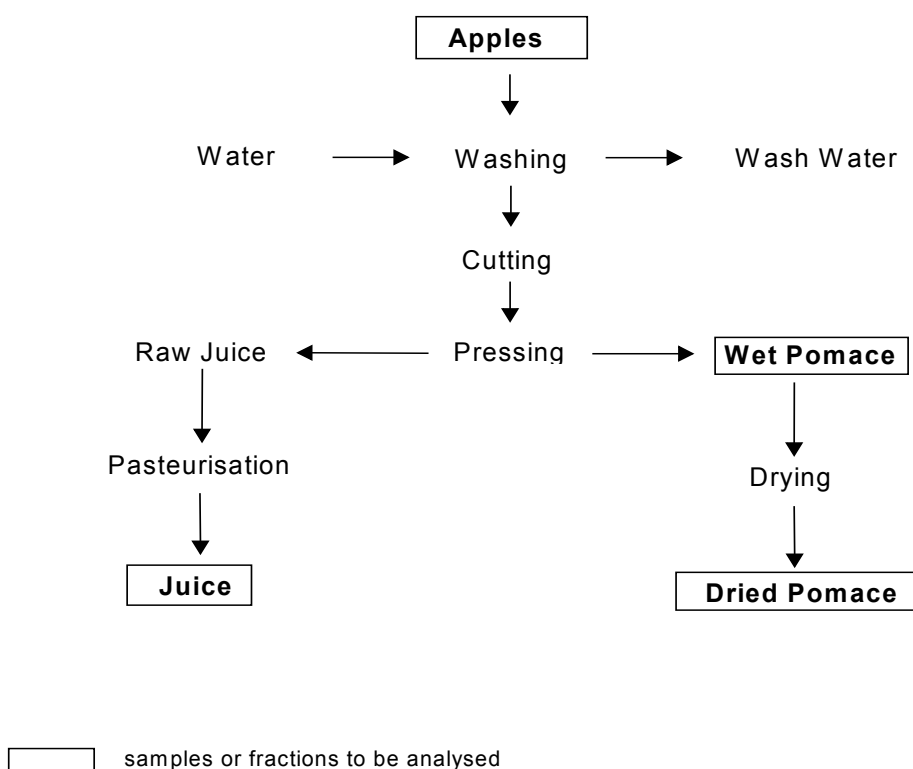


Table 135. Effects of processing on residues in cherries treated with imidacloprid, 1999.

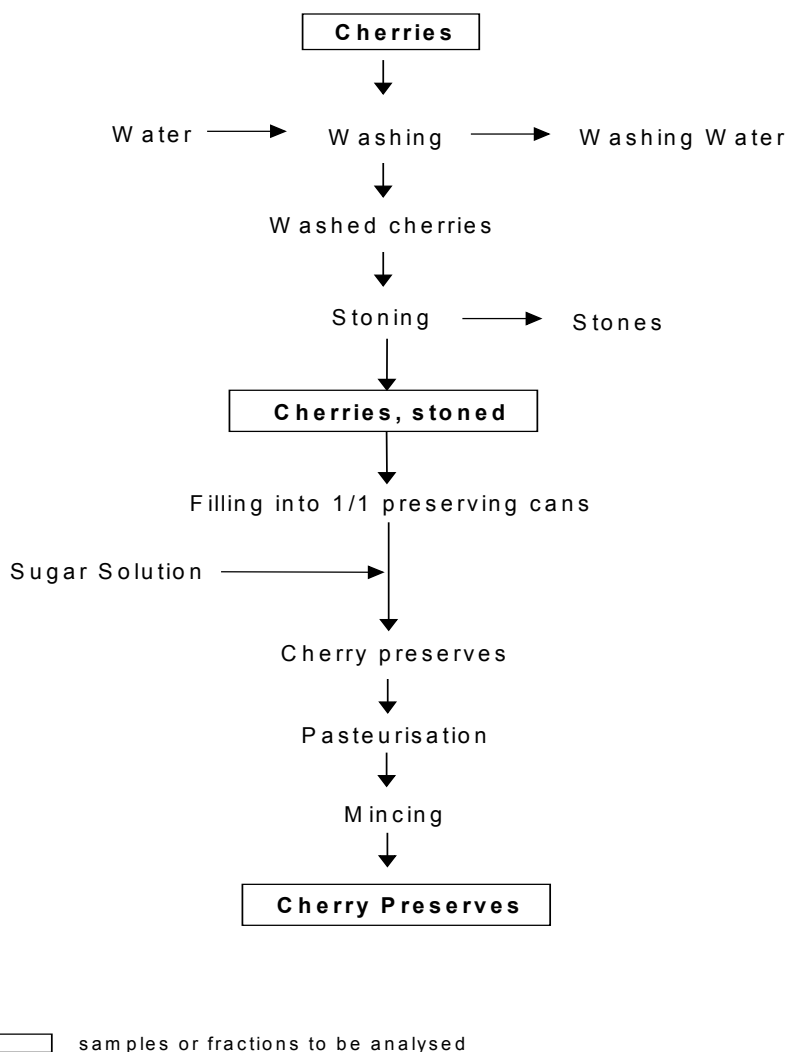
Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
Spain	fruit	0.03		0.09		RA-3047/99 0269-99 (A)
	fruit without stone	0.03	1	0.06	0.67	
	washed fruit	0.03	1	0.07	0.78	
	canned fruit	0.01	0.3	<0.05	<0.56	
	washings	<0.01	<0.3	<0.05	<0.56	
Spain	fruit	0.03		0.09		RA-3047/99 0269-99 (B)
	fruit without stone	0.03	1	0.06	0.67	
	washed fruit	0.03	1	0.07	0.78	
	canned fruit	0.02	0.67	<0.05	<0.56	
	washings	<0.01	<0.3	<0.05	<0.56	

Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
Italy	fruit	0.03		0.08		RA-3047/99 0270-99 (A)
	fruit without stone	0.01	0.3	<0.05	<0.63	
	washed fruit	0.02	0.67	0.05	0.63	
	canned fruit	<0.01	<0.3	<0.05	<0.63	
	washings	<0.01	<0.3	<0.05	<0.63	
Italy	fruit	0.03		0.08		RA-3047/99 0270-99 (B)
	fruit without stone	0.02	0.67	0.06	0.75	
	washed fruit	0.02	0.67	0.06	0.75	
	canned fruit	<0.01	<0.3	<0.05	<0.63	
	washings	<0.01	<0.3	<0.05	<0.63	

(A): first of two processing trials from single field trial.

(B): second of two processing trials from single field trial.

Figure 10. Flow diagram for the preparation of sweet cherry preserves (canned fruit).



Peaches. In a field trial in Spain peaches were sprayed twice at a rate of 0.08 kg ai/ha, and harvested 15 days after the second application for processing into preserve (canned fruit) and jam using simulated industrial practices on a laboratory scale. The results are shown in Table 136.

Table 136. Effects of processing on residues in peaches treated with imidacloprid, Spain, 1990.

Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
fruit	15	0.07		0.13		0222-90
canned fruit		0.04	0.57	<0.05	<0.38	
jam		0.03	0.43	<0.05	<0.38	

Grapes. To determine whether residues of imidacloprid are concentrated during processing four trials were conducted on wine production and four on juice production using grapes from European trials. A

further processing study in the USA was conducted for juice and raisins. The results are given in Table 137.

In four field trials in Portugal grapes were sprayed once at 0.02 kg ai/hl, 0.1 kg ai/ha. Two trials were on red and two on white varieties. Bunches were sampled 14 days after treatment plus control samples. Vinification of red wine and white wine was carried out in Portugal.

Red wine - mash fermentation (*curtimento*). Bunches of grapes were destemmed and then crushed, at which stage part of the must was taken for analysis. A solution of hyposulfite (80 mg/l) was then added and the resulting mash used to fill a demijohn for fermentation to be started. The temperature of the alcoholic fermentation, which took 5-8 days, was monitored each working day. After about 50 days the wine was transferred to another demijohn (first racking) and hyposulfite solution (30 mg/l) added. Four months later, after the second racking, the wine was bottled. The samples of must and wine were stored frozen (Figure 11).

White wine - must fermentation (*bica-aberta*). Bunches of grapes were immediately crushed and the resulting mash pressed into must, at which stage part was taken for analysis. After the remaining must had been stored for 48 hours at 4°C for clarification, a solution of 120 mg hyposulfite/l was added and fermentation started. Alcoholic fermentation was monitored as before and took about 10 days. About 38 days later, the first racking was carried out and, as before, hyposulfite was then added. Four months later, after the second racking, the wine was bottled (Figure 12).

Two field trials were conducted in Italy and Portugal on red and white grapes with a spray concentration of 0.01 kg ai/hl, 0.1 kg ai/ha, and two samples of mature grapes were collected 14 days after treatment from each trial for processing into juice.

Processing to juice. After destemming, the grapes were washed according to household practices, but processing simulated industrial practices. Washed berries were crushed in a punctured disk mill, and the resulting mash was pressed into raw juice and wet pomace. The raw juice was heated to 80-85°C, cooled to 50-55°C, treated with enzyme, stored for about 17 hours at 4°C, coarsely clarified by decanting and centrifugation, and ultra-centrifugated to obtain retentate and juice. The filtered juice was pasteurised at about 85°C for about 0.7-0.85 min (Figure 13).

In a US field trial two foliar spray applications of imidacloprid were made to grapes at 0.27 kg ai/ha (five times the label rate). Processing into canned juice and sun- and oven-dried raisins simulated typical commercial practices as closely as possible. Bunches of grapes, juice, wet and dry pomace, sun- and oven-dried raisins and their waste were analysed.

Table 137. Effects of processing on residues in grapes treated with imidacloprid.

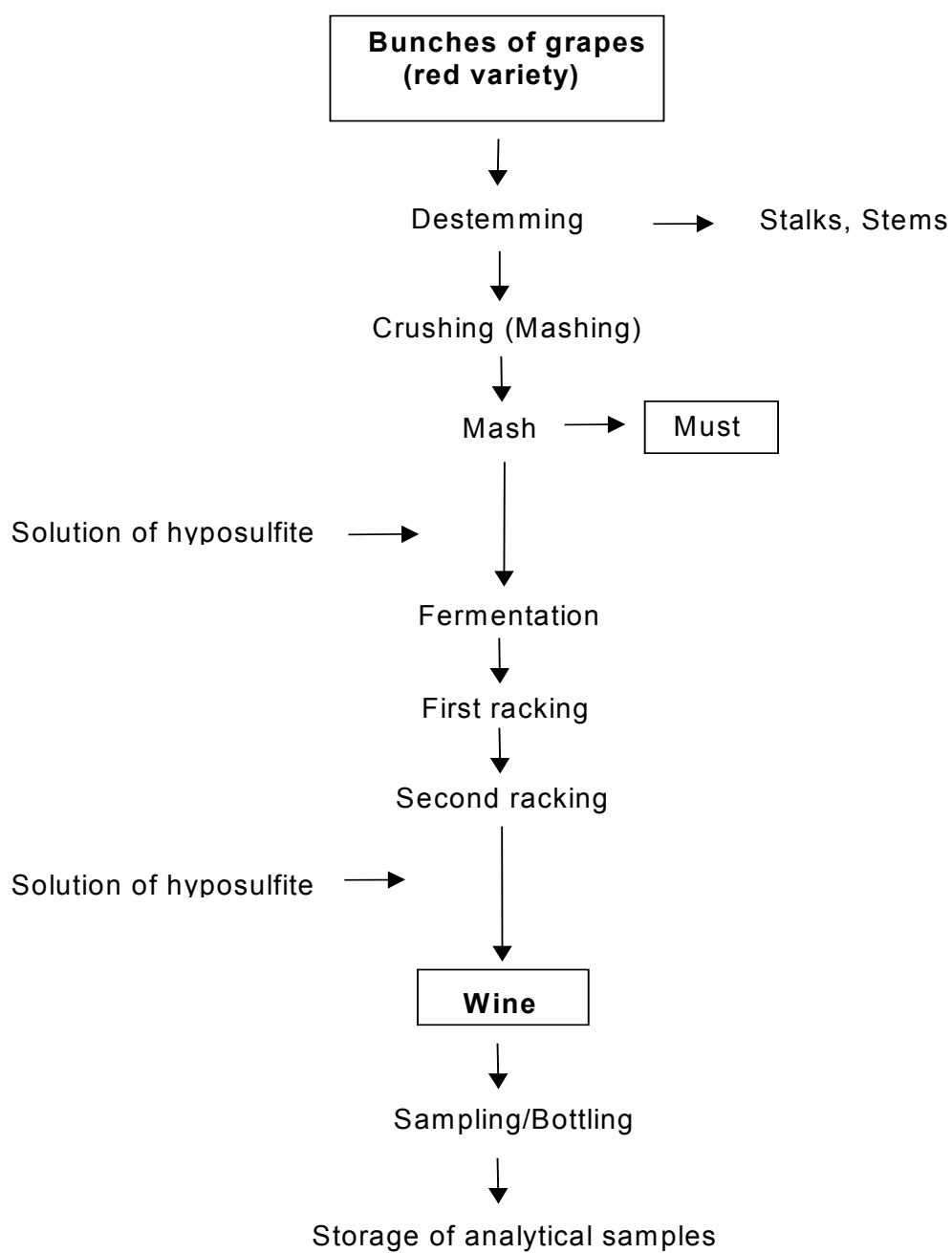
Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
Portugal 1997	grape bunch	0.01		0.05		RA-5000/97
	must	0.01	1	0.06	1.2	0691-97
	wine	<0.01	<1	0.06	1.2	red grape
Portugal 1997	grape bunch	0.01		0.06		RA-5000/97
	must	0.02	2	0.08	1.3	0692-97
	wine	0.02	2	0.08	1.3	white grape
Portugal 1998	grape bunch	0.04		0.07		RA-6000/98
	must	0.07	1.75	0.1	1.43	1354-98
	wine	0.04	1	0.06	0.86	red grape
Portugal 1998	grape bunch	0.04		0.06		RA-6000/98
	must	0.09	2.25	0.13	2.17	1355-98
	wine	0.07	1.75	0.08	1.33	white grape
Portugal 1999	grape bunch	0.08		0.1		RA-3030/99
	washed berries	0.02	0.25	<0.05	<0.5	0716-99 (A)
	washing water	<0.01	<0.125	<0.05	<0.5	white grape
	pomace, wet	0.24	3	0.29	2.9	
	retentate	<0.01	<0.125	<0.05	<0.5	
	juice	<0.01	<0.125	<0.05	<0.5	
Portugal 1999	grape bunch	0.08		0.1		RA-3030/99
	washed berries	0.02	0.3	<0.05	<0.5	0716-99 (B)
	washing water	<0.01	<0.1	<0.05	<0.5	white grape
	pomace, wet	0.23	2.9	0.29	2.9	
	retentate	<0.01	<0.1	<0.05	<0.5	
	juice	<0.01	<0.1	<0.05	<0.5	
Italy 1999	grape bunch	0.01		<0.05		RA-3030/99
	washed berries	<0.01	<1	<0.05		0311-99 (A)

Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
	washing water	<0.01	<1	<0.05		<i>red grape</i>
	pomace, wet	0.04	4	0.08	1.6	
	retentate	<0.01	<1	<0.05		
	juice	<0.01	<1	<0.05		
Italy 1999	grape bunch	0.01		<0.05		<i>RA-3030/99</i> 0311-99 (B) <i>red grape</i>
	washed berries	<0.01	<1	<0.05		
	washing water	<0.01	<1	<0.05		
	pomace, wet	0.04	4	0.09	1.8	
	retentate	<0.01	<1	<0.05		
	juice	<0.01	<1	<0.05		
USA 1991	grape bunch			0.2		<i>103839</i> FCA- N3050-91P
	pomace, wet			0.39	1.95	
	pomace, dry			0.86	4.3	
	raisin waste ¹			2.29	11.45	
	raisin waste ²			2.02	10.1	
	raisin ¹			0.2	1	
	raisin ²			0.21	1.1	
	juice			0.23	1.2	

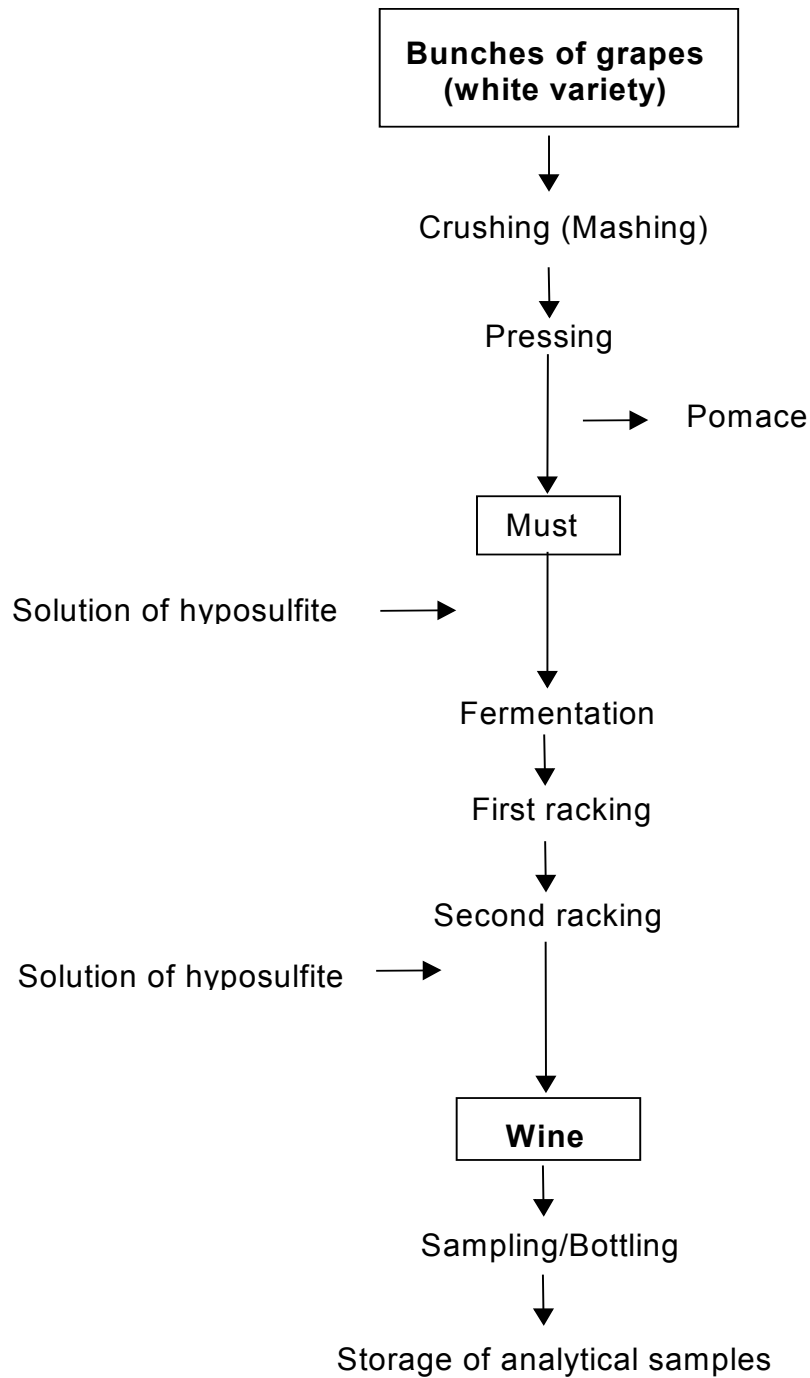
¹ sun-dried² oven-dried

(A): first of two processing trials from single field trial.

(B): second of two processing trials from single field trial.

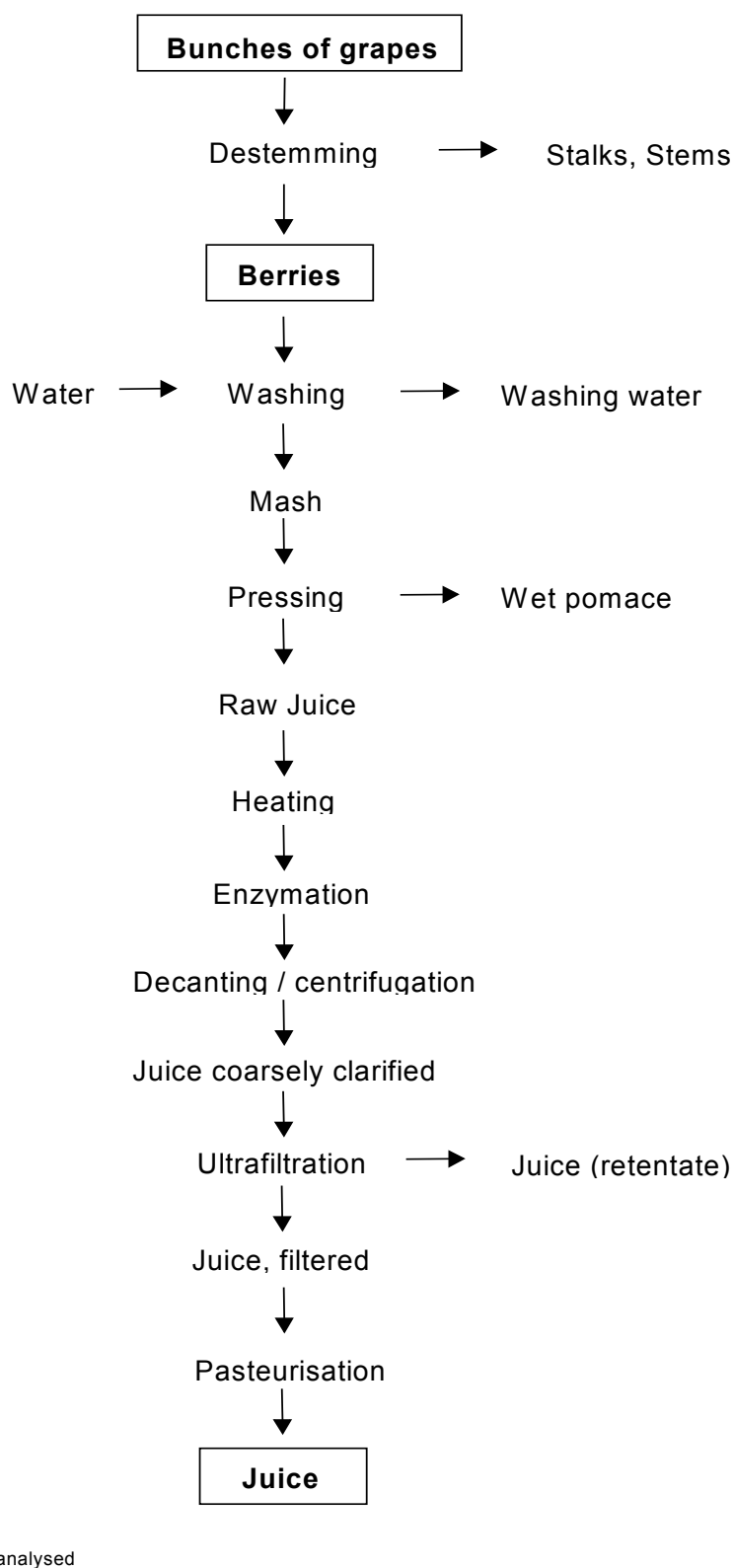
Figure 11. Flow diagram for vinification of red wine; mash fermentation (*Curtimenta*).

samples to be analysed

Figure 12. Flow diagram for the vinification of white wine; Must fermentation (*Bica aberta*).

samples to be analysed

Figure 13. Flow diagram for vinification of grape juice.



Tomatoes. Six processing trials were carried out on tomatoes (four in Spain and two in the USA). The results are given in Table 138.

In the four field trials in Spain two spray applications of imidacloprid at 0.01 kg ai/hl, 0.1-0.15 kg ai/ha were made and tomatoes harvested 7 days later. In trials 0218-90 and 0219-90 tomatoes were processed into paste and ketchup simulating industrial practice on a laboratory scale. In the other two trials tomatoes, washed and peeled according to household practices, were then processed into preserves (canned fruit), paste, purée and juice by simulated industrial practice on a laboratory scale. To prepare preserves the tomatoes were washed and the cups removed, their skins were perforated before the preserving cans were filled. Pickling liquor (NaCl solution) was added (tomatoes/liquor 1:0.8). After pasteurization the tomatoes and the liquor were macerated to give analytical samples.

To prepare juice and paste, tomatoes were washed and the cups removed, then cut into small pieces and heated after the addition of water to prevent enzymatic reactions. After this blanching process, the pulp was strained to separate juice and paste. NaCl was added to some of the juice before it was canned and pasteurized in an autoclave. The remainder was concentrated to 36-39% dry weight to give three-fold concentrated paste, then canned and pasteurized as before.

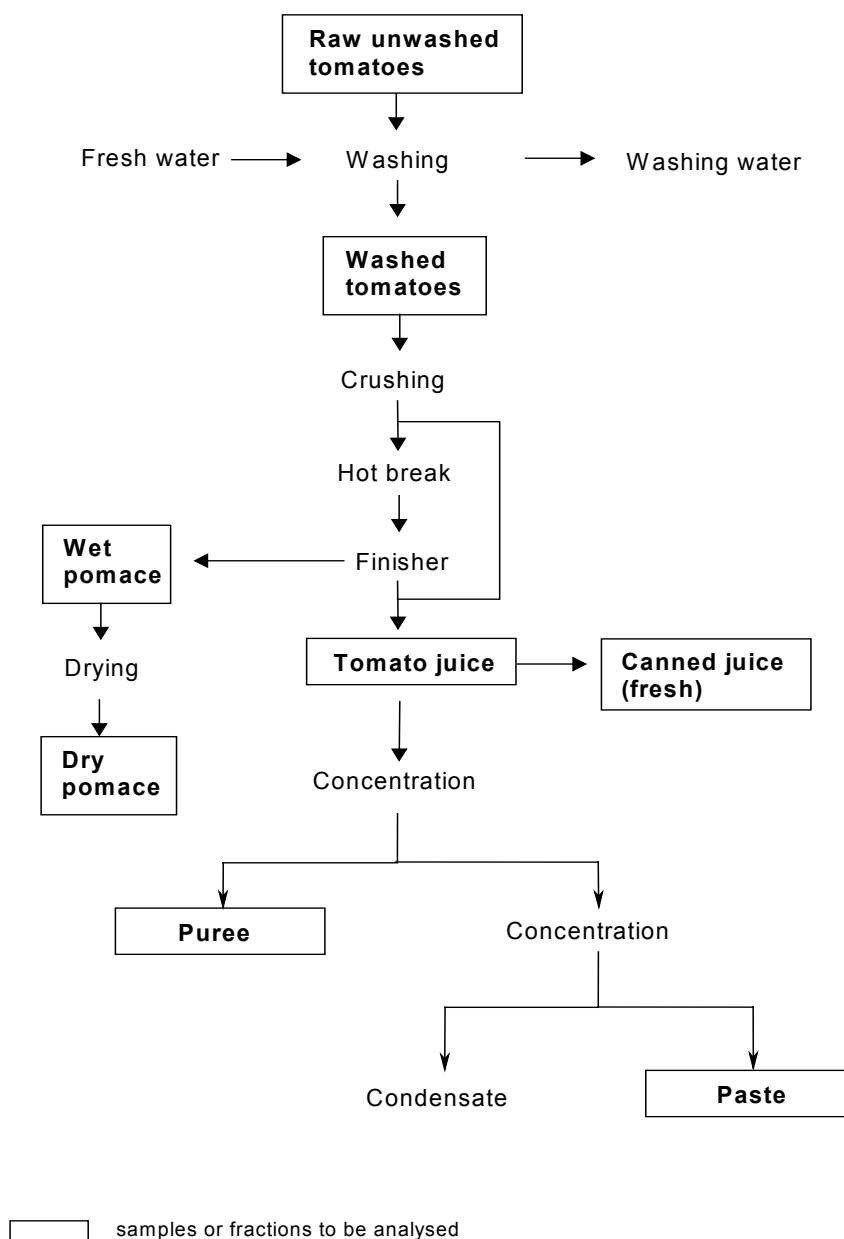
In the USA in two field trials a soil drench of 2.8 kg ai/ha was followed by two foliar sprays at 0.62 kg ai/ha resulting in a rate seven times the maximum recommended. Mature tomatoes were harvested 14 days after the last treatment, and processed into juice, purée, paste, and wet and dry pomace. Processing simulated typical commercial practices as closely as possible (Figure 14).

Table 138. Effects of processing on residues in tomato treated with imidacloprid.

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
Spain 1990	fruit	7	0.02		0.05		0218-90
	paste		0.05	2.5	0.17	3.4	
	ketchup		0.03	1.5	0.1	2.0	
Spain 1990	fruit	7	0.02		<0.05		0219-90
	paste		0.03	1.5	0.09		
	ketchup		0.04	2	0.08		
Spain 1991	fruit	7	0.04		0.11		RA-2037/91 0488-91
	washed fruit		0.07	1.75	0.23	2.1	
	canned fruit		0.04	1	0.1	0.91	
	paste		0.29	7.25	0.96	8.7	
	juice		0.06	1.5	0.2	1.8	
Spain 1991	fruit	7	<0.01		<0.05		RA-2037/91 0489-91
	washed fruit		<0.01		<0.05		
	canned fruit		<0.01		<0.05		

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
	paste		0.02	2	<0.05		
	juice		<0.01		<0.05		
USA 1992	fruit	14			0.44		105024 FCA-N3167-92P
	pomace, wet				0.69	1.57	
	pomace, dried				2.2	5.0	
	purée				0.83	1.89	
	juice				0.44	1.0	
USA 1994	fruit	14			0.16		106651 FCA-AD027-94P
	juice				0.21	1.3	
	pomace, wet				0.24	1.5	
	pomace, dried				0.57	3.6	
	paste				0.82	5.1	
	purée				0.43	2.7	

Figure 14. Flow diagram for the preparation of tomato purée, paste and juice.



Lettuce. In two field trials in Greece and Italy crops were sprayed twice with a 200 SL formulation at 0.013 kg ai/hl, 0.1 kg ai/ha. Two samples of head lettuce from each trial were collected 7 days later for analysis of washed and unwashed samples. The heads were washed according to household practices. About 60% of the total residue was removed by washing (Table 139).

In 19 US field trials a 240 SC formulation of imidacloprid was applied to head lettuce by various methods (Table 105). After soil drench plus foliar sprays, heads were sampled 0, 7, and 14 days after the last treatment. After drench, in-furrow or sidedress applications heads were sampled at harvest, 27 to 133 days after treatment. The total residue was determined in lettuce heads with wrapper leaves (head) and in heads without wrapper leaves (head, cleaned). When wrapper leaves were removed the total residue decreased to a mean of about one third (Table 139).

Table 139. Effects of processing on residues in lettuce treated with imidacloprid.

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
Greece 2001	head	7	0.11		0.26		<i>RA-2045/01</i> 0115-01 (A)
	head, washed		0.02	0.18	0.08	0.31	
Greece 2001	head	7	0.11		0.26		<i>RA-2045/01</i> 0115-01 (B)
	head, washed		0.03	0.27	0.1	0.38	
Italy 2001	head	7	0.08		0.72		<i>RA-2045/01</i> 0115-01 (A)
	head, washed		0.04	0.5	0.43	0.6	
Italy 2001	head	7	0.08		0.72		<i>RA-2045/01</i> 0115-01 (B)
	head, washed		0.05	0.63	0.58	0.81	
USA 1992	head	0			2.1		<i>105164</i> 352-N3086-92D
	head, cleaned				1.1	0.52	
	head	7			1.7		
	head, cleaned				0.72	0.42	
	head	14			0.8		
	head, cleaned				0.29	0.36	
USA 1992	head	0			0.44		<i>105164</i> 454-N3087-92D
	head, cleaned				0.21	0.48	
	head	7			0.59		
	head, cleaned				<0.05	<0.08	
	head	14			0.46		
	head, cleaned				0.15	0.33	
USA 1992	head	0			1.35		<i>105164</i> 457-N3088-92D
	head, cleaned				0.28	0.21	
	head	7			0.67		
	head, cleaned				0.15	0.22	
	head	14			0.26		
	head, cleaned				0.09	0.35	

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
USA 1992	head	0			5.1		105164 457-N3089-92D
	head, cleaned				0.68	0.13	
	head	7			2.1		
	head, cleaned				0.58	0.28	
	head	14			0.99		
	head, cleaned				0.28	0.28	
USA 1992	head	0			0.96		105164 458-N3090-92D
	head, cleaned				0.11	0.11	
	head	7			0.67		
	head, cleaned				0.06	0.09	
	head	14			0.33		
	head, cleaned				0.08	0.24	
USA 1992	head	0			0.91		105164 756-N3091-92D
	head, cleaned				0.11	0.12	
	head	7			0.32		
	head, cleaned				0.07	0.22	
	head	14			0.12		
	head, cleaned				0.06	0.5	
USA 1992	head	0			2.6		105164 858-N3092-92D
	head, cleaned				0.54	0.21	
	head	7			0.6		
	head, cleaned				0.12	0.2	
	head	14			0.27		
	head, cleaned				<0.05	<0.19	
USA 1992	head	0			2.5		105164 BMS-N3093-92D
	head, cleaned				1.4	0.56	
	head	7			1.3		

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		<i>Report no.</i> Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
	head, cleaned				0.38	0.29	
USA 1992	head	0			1.2		<i>105164</i> FCA-N3094-92D
	head, cleaned				0.22	0.18	
	head	7			0.57		
	head, cleaned				0.17	0.3	
	head	14			0.52		
	head, cleaned				0.2	0.38	
USA 1992	head	0			2.8		<i>105164</i> FCA-N3095-92D A + B + C
	head, cleaned				0.49	0.18	
	head	7			0.73		
	head, cleaned				0.16	0.22	
	head	14			0.34		
	head, cleaned				0.09	0.26	
USA 1992	head	0			0.69		<i>105164</i> STF-N3096-92D
	head, cleaned				<0.05	<0.07	
	head	7			0.31		
	head, cleaned				0.1	0.32	
	head	14			0.12		
	head, cleaned				0.09	0.75	
USA 1992	head	0			1.2		<i>105164</i> TGA-N3241-92D
	head, cleaned				0.34	0.28	
	head	7			0.85		
	head, cleaned				0.12	0.14	
	head	14			0.99		
	head, cleaned				0.06	0.06	
USA 1992	head	29			0.38		<i>105164</i> HIN-N3099-92H
	head, cleaned				0.09	0.24	

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
USA	head	40			<0.05		105164
1992	head, cleaned				<0.05		STF-N3100-92H
USA	head	94			<0.05		105164
1992	head, cleaned				<0.05		TGA-N3242-92H
USA	head	77			0.14		105164
1992	head, cleaned				<0.05	<0.36	HIN-N3106-92H
USA	head	27			0.12		105164
1992	head, cleaned				0.05	0.42	HIN-N3112-92H
USA	head	133			<0.05		105164
1992	head, cleaned				<0.05		TGA-N3244-92H
USA	head	91			<0.05		105164
1992	head, cleaned				<0.05		TGA-N3246-92H

(A): first of two processing trials from single field trial.

(B): second of two processing trials from single field trial.

Beans, green. The field parts for two processing trials was carried out in Italy: two sprays were applied at 0.1 kg ai/ha. Preparing preserves (canned fruit) from beans with pod was according to simulated industrial preparation on a laboratory scale. After being thawed and weighed pods were washed gently in a container of water. Once the stems were removed the pods were cut into small pieces, blanched at 95-100°C for 3 min, transferred to cans together with NaCl solution, and sterilized in an autoclave. The preserved beans with pods plus the cover brine (ratio 5:3) were macerated to give an analytical sample. The results are shown in Table 140.

Table 140. Effects of processing on residues in beans treated with imidacloprid.

Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
Italy	bean with pod	<0.01		0.32		RA-2022/91
1991	bean with pod, cooked	<0.01		0.26	0.81	0272-91
	cooking water	<0.01		0.13	0.41	
	canned beans with pod	<0.01		0.12	0.375	
Italy	bean with pod	0.03		0.29		RA-2095/92

Country Year	Sample	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
1992	bean with pod, cooked	0.01	0.3	0.33	1.14	0257-92
	cooking water	0.01	0.3	<0.05	<0.17	
	preserve with pod	<0.01	<0.3	0.14	0.48	

Potatoes. Five processing trials were carried out, four in Germany and one in the USA. The results are given in Table 141.

In the trials in Germany, tubers were treated at rates of 12 and 25 g ai/100 kg seed respectively. Tubers were sampled at normal harvest, 79-135 days after planting. In two trials the tubers were peeled and cooked, and in the other two also fried, according to household procedures. In these two dried potatoes were also produced by simulated industrial processes.

In a single field trial in the USA after an in-furrow application of 1.5 kg ai/ha and four foliar sprays at an exaggerated rate of about 0.27 kg ai/ha, potatoes were sampled 14 days after the last treatment. Processing procedures simulated typical commercial practice as closely as possible. Potatoes were washed and separated or processed into wet and dry peel, chips and granules.

Table 141. Effects of processing on residues in potatoes treated with imidacloprid.

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
Germany 1990	tuber	135	<0.01		<0.05		PF-3737 0076-90
	peeled tuber		<0.01		<0.05		
	peel		0.04	>4	<0.05		
	cooked potato		<0.01		<0.05		
Germany 1990	tuber	107	0.02		<0.05		PF-3737 0077-90
	peeled tuber		0.01	0.5	<0.05		
	peel		0.04	2	0.07	1.4	
	cooked potato		0.01	0.5	<0.05		
Germany 1991	tuber	79	0.02		<0.05		RA-2079/91 0069-91
	peeled tuber		0.01	0.5	<0.05		
	peel		0.02	1	<0.05		
	cooked potato		0.02	1	<0.05		

Country Year	Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
			mg/kg	Processing factor	mg/kg	Processing factor	
	cooking, water		<0.01	<0.5	<0.05		
	fried potato		0.01	0.5	<0.05		
	dried potato		<0.05	<2.5	<0.05		
Germany 1991	tuber	91	<0.01		<0.05		RA-2079/91 0070-91
	peeled tuber		<0.01		<0.05		
	peel		0.03	3	<0.05		
	cooked potato		<0.01		<0.05		
	cooking, water		<0.01		<0.05		
	fried potato		<0.01		<0.05		
	dried potato		<0.05		<0.05		
USA 1991	tuber	14			0.26		103238 FCA-N3068- 91P
	washed tuber				0.21	0.81	
	granules				0.24	0.92	
	chips				0.35	1.35	
	peel, wet				0.17	0.65	
	peel, dried				0.76	2.92	

Rice. In six trials in Thailand rice was sprayed at 0.005 kg ai/hl in four and at 0.01 kg ai/hl in two, 0.015-0.025 kg ai/ha and 0.05 kg ai/ha respectively, and unpolished grain, sampled after 33-56 days, was processed to polished grain, glume and bran. Processing factors could not be calculated (Table 142).

Table 142. Effects of processing on residues in rice and its processed products treated with imidacloprid.

Country Year	Sample	PHI (days)	Imidacloprid (mg/kg)	Total residue as imidacloprid (mg/kg)	Report no. Study no.
Thailand 1991	grain, unpolished	33	<0.01	<0.05	0482-91
	grain, polished		<0.01	<0.05	
Thailand ¹	grain, polished	56	<0.01	<0.05	0218-92

Country Year	Sample	PHI (days)	Imidacloprid (mg/kg)	Total residue as imidacloprid (mg/kg)	Report no. Study no.
1992	bran		<0.01	<0.05	
	glume		<0.05	<0.05	
Thailand ¹	grain, polished	56	<0.01	<0.05	0219-92
1992	bran		<0.01	<0.05	
	glume		<0.05	<0.05	
Thailand ¹	grain, polished	48	<0.01	<0.05	0698-92
1992	bran		<0.01	<0.05	
	glume		<0.05	<0.05	
Thailand ¹	grain, polished	48	<0.05	<0.05	0699-92
1992	bran		<0.01	<0.05	
	glume		<0.05	0.08	

¹ Unpolished grain was not analysed.

Wheat. A single field study was conducted in the USA to determine whether residues of imidacloprid from a single seed treatment of 0.63 kg ai/100 kg seed (5 times the application rate) would be concentrated in processed products. Wheat samples were collected at earliest harvest, and the grain was processed into bran, flour, middlings and shorts. The processing procedure simulated commercial practice as closely as possible. Dried wheat samples were cleaned by aspiration and screening, adjusted for moisture and passed four times through corrugated roller mills. After separation of bran, the sample was reduced to flour with a smooth roller mill (Figure 15). Additional validation data have been generated which reduced the LOQ for the analytical method used from 0.1 to 0.05 mg/kg. On the basis of the LOQ of 0.05 mg/kg and the MDL (minimum detection limit) of 0.01 mg/kg the residue data from report 105007 were re-analysed (report 105007-1). The results from both reports are given in Table 143.

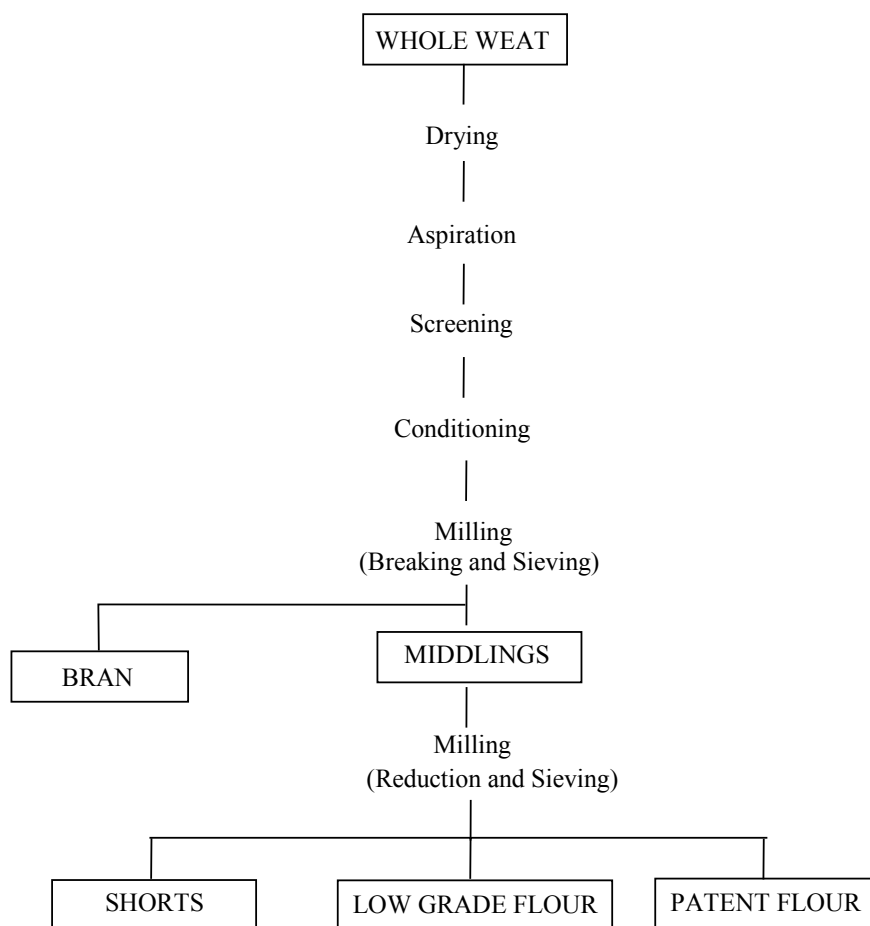
Table 143. Effects of processing on residues in wheat treated with imidacloprid, USA, 1991.

Sample	PHI (days)	Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	
grain	110	<0.05		105007
bran		0.07		HIN-N3024-91P
flour		<0.05		LOQ:
middlings		<0.05		0.1 mg/kg

Sample	PHI (days)	Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	
shorts		<0.05		105007-1 HIN-N3024-91P LOQ: 0.05 mg/kg
grain	110	0.02 ¹		
bran		0.07 ¹	3.5	
flour		0.01 ¹	0.5	
middlings		0.01 ¹	0.5	
shorts		0.04 ¹	2	

¹ Based on MDL (minimum detection limit) of 0.01 mg/kg.

Figure 15. Flow diagram for the processing of wheat.



Samples to be analyzed

Cotton seed. Four processing trials were conducted, one in Greece, two in the Turkey and one in the USA (Table 144). In the two trials in Turkey the crop was sprayed twice at 0.53 and 0.35 kg ai/ha, and the seed processed into oil (no further information was given).

In the trial in Greece 3 spray applications of a 200 SL formulation at 0.012 kg ai/hl, 0.12 kg ai/ha, were made and the seed processed into crude and refined oil (no further information was given on processing).

In the trial in the USA an in-furrow application at planting at 1.6 kg ai/ha was followed by two foliar sprays with a seven-day interval, each at 1.3 kg ai/ha, the second 14 days before harvest. This is five times the recommended yearly rate. Cotton seed was processed into crude and refined oil, hulls, meal and soapstock according to simulated typical commercial practices as far as possible. The seed was first delinted, then mechanically cracked and screened to separate the hulls from the kernels. The kernels, which still included some hulls, were flaked, expanded into collets, heated and extracted with hexane to remove crude oil from the collets. The spent collets (meal) were dried with warm forced air. After the crude oil and hexane mixture was adjusted to the proper ratio, the crude oil was refined (Figure 16).

Table 144. Effects of processing on residues in cotton seed treated with imidacloprid.

Country Year	Sample	PHI (days)	Imidacloprid (mg/kg)	Total residue as imidacloprid		Report no. Study no.
				mg/kg	Processing factor	
Greece 1991	seed	58	<0.01	2.7		0127-91
	oil, crude		<0.01	<0.05	<0.019	
	oil, refined		<0.01	<0.05	<0.019	
Turkey 1989	seed	28	<0.01	0.66		0043-89
	oil, crude		<0.1	<0.05	<0.076	
Turkey 1989	seed	28	<0.01	0.54		0571-89
	oil, crude		<0.1	<0.05	<0.093	
USA 1991	seed	14		2.9		103246
	oil, crude			<0.5	<0.17	
	oil, refined			<0.5	<0.17	
	hull			1.1	0.38	
	meal			4.2	1.45	
	soapstock			<0.5	<0.17	

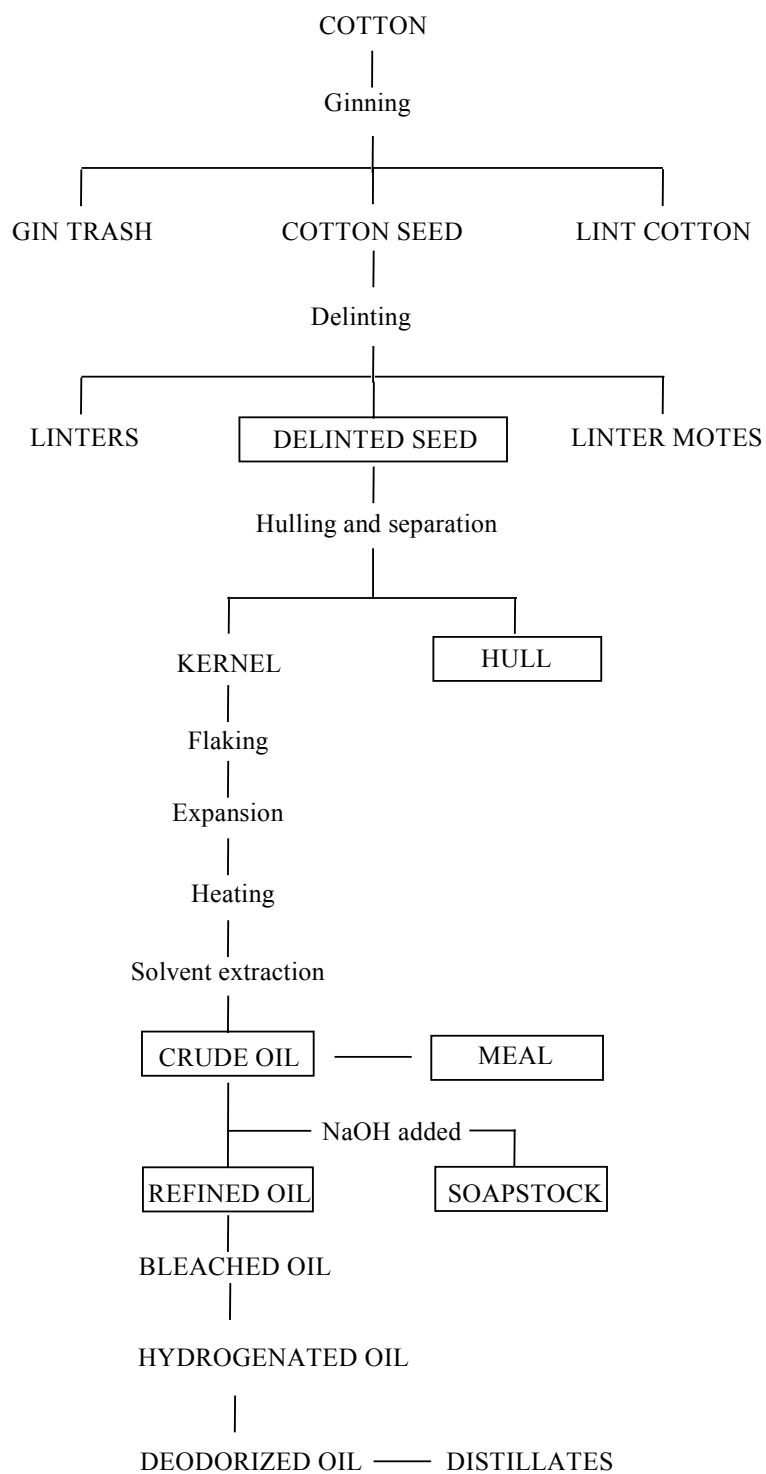
Hops. In two German trials hop cones were harvested 35 days following one spray application of 0.52 kg ai/ha. Green cones were kiln-dried according to local practice and the dried cones used to brew beer. The results are given in Table 145.

Table 145. Effects of processing on residues in hops treated with imidacloprid, Germany, 1990.

Sample	PHI (days)	Imidacloprid		Total residue as imidacloprid		Report no. Study no.
		mg/kg	Processing factor	mg/kg	Processing factor	
cone, green	35	0.46		1.4		PF3739 0184-90
cone, kiln-dried (RAC)		1.8		5.8		
brewer's grains		<0.05	0.028	0.02	0.003	
beer		<0.01	0.006	0.01	0.002	
cone, green	35	0.41		1.3		PF3739 0185-90
cone, kiln-dried (RAC)		1.7		6.4		
brewers grains		<0.05	0.029	0.04	0.006	
beer		0.01	0.006	0.03	0.005	

Tea. In two field trials in Japan according to GAP with a 10 WP formulation and two more trials in 1998 with a 50 WG formulation, the crop was sprayed once at 0.01 kg ai/hl, corresponding to 0.2 kg ai/ha. Leaves were harvested after 13-27 days and 7-21 days, twisted and dried in a tea-making machine, then infused. Only the parent compound was determined in the dried leaves and the infusion. The samples from report no. NR91-47 were analysed by two different laboratories, at Nihon Bayer Agrochem (NBA) and the official Japanese laboratories (JFRL), with comparable results (Table 146).

Figure 16. Processing of cotton to seed and oil.



Samples to be analyzed

Table 146. Effects of processing on residues in tea treated with imidacloprid.

Year, Location	Sample	PHI (days)	Imidacloprid		Report no. Study no.
			mg/kg	Processing factor	
1990 Shizuoka	leaf, dried ¹	13	1.8		NR91-47
	infusion ¹		1.9	1.06	JAP-NR91-47-A1
	leaf, dried ²		2.3		
	infusion ²		1.8	0.78	
1990 Shizuoka	leaf, dried ¹	20	0.54		NR91-47
	infusion ¹		0.61	1.13	JAP-NR91-47-A2
	leaf, dried ²		0.78		
	infusion ²		0.64	0.82	
1990 Shizuoka	leaf, dried ¹	27	0.1		NR91-47
	infusion ¹		0.08	0.8	JAP-NR91-47-A3
	leaf, dried ²		0.11		
	infusion ²		0.06	0.55	
1990 Miyazaki	leaf, dried ¹	14	1.4		NR91-47
	infusion ¹		1.1	0.79	JAP-NR91-47-B1
	leaf, dried ²		1.9		
	infusion ²		1.9	1	
1990 Miyazaki	leaf, dried ¹	21	0.6		NR91-47
	infusion ¹		0.59	0.98	JAP-NR91-47-B2
	leaf, dried ²		0.8		
	infusion ²		0.64	0.98	
1990 Miyazaki	leaf, dried ¹	28	0.17		NR91-47
	infusion ¹		0.14	0.82	JAP-NR91-47-B3
	leaf, dried ²		0.2		
	infusion ²		0.15	0.75	
1998	leaf, dried ¹	7	3.0		NR98035
	infusion ¹		2.5	0.83	JAP-NR98035-A1

Year, Location	Sample	PHI (days)	Imidacloprid		Report no. Study no.
			mg/kg	Processing factor	
Shizuoka	leaf, dried ¹	14	2.3		NR98035
	infusion ¹		1.8	0.78	JAP-NR98035-A2
	leaf, dried ¹	21	0.34		NR98035
	infusion ¹		0.26	0.76	JAP-NR98035-A3
1998 Miyazaki	leaf, dried ¹	7	4.0		NR98035
	infusion ¹		3.3	0.83	JAP-NR98035-B1
	leaf, dried ¹	14	1.8		NR98035
	infusion ¹		1.9	1.06	JAP-NR98035-B2
	leaf, dried ¹	21	1.0		NR98035
	infusion ¹		0.88	0.88	JAP-NR98035-B3

¹ Analysed at Nihon Bayer Agrochem K.K. (NBA)

² Analysed at Japan Food Research Laboratories (JFRL)

Leaf, dried: extraction by acetonitrile and high-speed homogenizer after swelling with water

Infusion: extraction by hot water

Residues in the edible portion of food commodities

Information is available on citrus pulp (Tables 70, 72, 73 74, 77), melons (Table 95) and watermelons (Table 96), recorded in the section on supervised trials on crops.

RESIDUES IN ANIMAL COMMODITIES

Direct animal treatments

Imidacloprid is not used for direct animal treatment so no information is available.

Farm animal feeding trials

Dairy cattle. In a study by Heukamp (1992b) three groups of cows three plus one control in each) were given bolus capsules at levels equivalent to 5, 15, and 50 ppm in the diet (0.15, 0.45 and 1.5 mg/kg bw for 28 days). The levels were based on a dietary burden of 4.4 ppm in dry feed matter, calculated by the manufacturer. Feed consumption, milk production and body weights of the cows were not affected by the daily doses.

The cows were milked in the morning and in the evening and the milk from each cow was mixed thoroughly and two 250 ml samples taken. Milk samples from different cows and different days were not combined, but just before analysis the respective morning and evening milk samples were pooled in the ratio of milk production. The cows were slaughtered on day 29 immediately after

the morning milking (i.e. 14 to 18 hours after the last dose) and samples of liver without gall bladder (whole organ), both kidneys (whole organs), round, flank and loin muscle, and subcutaneous, mesenteric and renal fat were stored frozen (milk at -18 to -20°C and tissues at -20 to -24°C).

The total residue in milk was below the LOQ of 0.02 mg/kg in the 5 ppm dose group in all samples. In the 15 ppm group, having reached a plateau directly after the first dose it remained relatively constant over the first 13 days then slowly decreased. At the 50 ppm dose the total residues reached a plateau 3 days after the first dose and were nearly constant until day 13, then decreased to about 0.1 mg/kg after 28 days (Table 147).

Table 147. Residues in milk of treated cows (Heukamp, 1992b).

Days after treatment	Total residues of imidacloprid as imidacloprid (mg/kg)							
	Control		5 ppm		15 ppm		50 ppm	
	Single values	Mean	Single values	Mean	Single values	Mean	Single values	Mean
0	<0.02 (3)	<0.02	<0.02 (3)	<0.02	<0.02 (3)	<0.02	<0.02 (3)	<0.02
1	<0.02 (3)	<0.02	<0.02 (3)	<0.02	0.054, 0.035, 0.035	0.041	0.123, 0.121, 0.174	0.14
2							0.108	0.11
3							0.154	0.15
4							0.123, 0.163, 0.16	0.15
5					0.051, 0.024, 0.028	0.034		
7							0.152, 0.148, 0.159	0.15
10							0.126, 0.118, 0.178	0.14
13	<0.02 (3)	<0.02	<0.02 (3)	<0.02	0.048, 0.037, 0.031	0.039	0.131, 0.157, 0.169	0.15
16							0.123, 0.147, 0.093	0.12
19							0.118, 0.148, 0.138	0.13
22					0.033, 0.025, 0.025	0.028	0.099, 0.131, 0.119	0.12
25							0.1, 0.113, 0.138	0.12
28	<0.02 (3)	<0.02	<0.02 (3)	<0.02	0.032, 0.03, 0.022	0.028	0.099, 0.101, 0.102	0.1

In the muscles and fat of the 5 ppm group the total residue was below the LOQ of 0.02 mg/kg in all samples. In liver the mean residue was 0.05 mg/kg and in kidney 0.03 mg/kg. The residues increased at higher dose levels. At the 50 ppm dose the highest residues were found in liver (mean 0.49 mg/kg) and kidney about 0.3 mg/kg (mean); in muscle the mean was 0.12 mg/kg and in fat only 0.06 mg/kg (mean). The results are given in Table 148.

Table 148. Residues in animal tissues from treated cows after doses of imidacloprid on 28 consecutive days (Heukamp, 1992b).

Dose in ppm	Total residues of imidacloprid as imidacloprid (mg/kg)							
	Muscle		Fat		Liver		Kidney	
	Single values	Mean	Single values	Mean	Single values	Mean	Single values	Mean
0	<0.02 (3)	<0.02	<0.02 (3)	<0.02	<0.02 (3)	<0.02	<0.02 (3)	<0.02
5	<0.02 (3)	<0.02	<0.02 (3)	<0.02	0.054, 0.049, 0.047	0.05	0.032, 0.028, 0.024	0.03
15	0.033, 0.029, <0.02	0.03	<0.02 (3)	<0.02	0.166, 0.136, 0.096	0.13	0.1, 0.101, 0.054	0.09
50	0.114, 0.15, 0.1	0.12	0.062, 0.078, 0.051	0.06	0.537, 0.406, 0.526	0.49	0.365, 0.269, 0.225	0.29

The results show that there is only a minimum transfer of the total residue into milk and tissues. Preferential transfer was found only in liver, in which the residues were generally highest, and kidney.

Poultry. Four groups of twelve laying hens were fed daily rations of feed fortified with imidacloprid at levels of 0, 2, 6, and 20 ppm (equivalent to mean daily doses of 0.18, 0.52, and 1.8 mg/kg bw respectively) for 30-32 consecutive days (Heukamp, 1992a). The dose levels were based on a dietary burden of 1.6 ppm in dry feed matter, calculated by the manufacturer. Commercial poultry feed was fortified with technical grade imidacloprid, dissolved in peanut oil and was mixed with the feed. The amount of imidacloprid, homogeneity and storage stability were determined before and during the dosing period in some samples.

Feed and water were available *ad libitum* throughout the study. Feed consumption, egg production and body weights were not affected by the daily dose. Eggs were collected twice daily. For each sample eggs from three hens of the same dose group collected on the same day were combined without their shells as shown in Table 149. After 30 days (dose levels 0 and 2 ppm) and 32 days (dose levels 6 and 20 ppm) the hens were killed. No morphological deviations or pathological findings were detected. Liver, composite thigh, leg and breast muscle and abdominal fat samples were taken. Tissue samples from the same hens were combined as reported for eggs. The samples were analysed for the total residue of imidacloprid by gas chromatographic determination.

Residues were not corrected for recoveries. In the control egg samples the total residue was undetectable, in eggs of the 2 ppm group the total residue was below the LOQ of 0.02 mg/kg, and in the higher dose groups it reached a plateau after some days (at 6 ppm approximately 0.05 mg/kg and at 20 ppm approximately 0.13 mg/kg). The results are shown in Table 150.

Table 149. Combining scheme for samples before analysis (Heukamp, 1992b).

Dose	Hen No.	Mean daily feed consumption (g/kg bw) (mean value from all hens)
0 mg/kg		
	Sample 1 4, 7, 34	84.3
	Sample 2 22, 23, 43	86.6

Dose	Hen No.	Mean daily feed consumption (g/kg bw) (mean value from all hens)
Sample 3	19, 24, 25	97.7
2 mg/kg		
Sample 1	26, 28, 33	87.9
Sample 2	20, 49, 50	95.5
Sample 3	15	114.2
6 mg/kg		
Sample 1	16, 30, 47	87.2
Sample 2	3, 12, 44	89.9
Sample 3	9, 14	95.0
20 mg/kg		
Sample 1	10, 41, 45	91.5
Sample 2	1, 29, 42	97.1
Sample 3	48	108.1

Table 150. Residues in eggs of treated laying hens (Heukamp, 1992a).

Days after treatment	Total residues of imidacloprid as imidacloprid [mg/kg]							
	Control		2 ppm dose		6 ppm dose		20 ppm dose	
	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean
0					<0.02 (3)	<0.02	<0.02 (3)	<0.02
1					0.023, 0.021, 0.024	0.023	0.051, 0.045, 0.05	0.049
2					0.029, 0.029, 0.029	0.029	0.082, 0.091, 0.041	0.071
3							0.079, 0.085, 0.078	0.081
5							0.098	0.098
6							0.095, 0.0107	0.1
7					0.035, 0.042	0.039		
8							0.102	0.1
9							0.112, 0.112	0.11
12							0.105, 0.118, 0.099	0.11
13					0.049, 0.041, 0.039	0.043		
15							0.111, 0.137, 0.092	0.11
17	<0.02 (3)	<0.02	<0.02 (3)	<0.02				
18							0.118, 0.097, 0.092	0.1
19					0.041, 0.036, 0.039	0.039		
21							0.128, 0.143, 0.113	0.13
24							0.126, 0.132, 0.124	0.13
25					0.044, 0.038, 0.04	0.041		

Days after treatment	Total residues of imidacloprid as imidacloprid [mg/kg]							
	Control		2 ppm dose		6 ppm dose		20 ppm dose	
	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean
27							0.108, 0.116, 0.14	0.12
29	<0.02 (3)	<0.02	<0.02 (3)	<0.02				
31					0.043, 0.051, 0.052	0.049	0.119, 0.133, 0.137	0.13

The residues in muscle were very low. In the lowest dose group the total residue was below the LOQ of 0.02 mg/kg. In the 6 and 20 ppm groups the mean was at the limit of quantification and about 0.05 mg/kg respectively. In fat residues were below the LOQ of 0.02 mg/kg in all samples. The residues in the liver increased from 0.04 mg/kg at the lowest dose to about 0.35 mg/kg at the highest dose (Table 151). The results show that there is only a minimum of transfer of imidacloprid residues into eggs and tissues.

Table 151. Residues in liver and tissues of laying hens (Heukamp, 1992a).

Dose, ppm	Total residues of imidacloprid as imidacloprid [mg/kg]					
	Muscle		Fat		Liver	
	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean	Sample 1, 2, 3	Mean
0	<0.02 (3)	<0.02	<0.02 (3)	<0.02	<0.02 (3)	<0.02
2	<0.02 (3)	<0.02	<0.02 (3)	<0.02	0.036, 0.042, 0.041	0.04
6	<0.02, 0.021, 0.021	0.02	<0.02 (3)	<0.02	0.127, 0.136, 0.159	0.14
20	0.039, 0.072, 0.034	0.048	<0.02 (3)	<0.02	0.287, 0.431, 0.321	0.35

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No data were received.

NATIONAL MAXIMUM RESIDUE LIMITS

The following MRLs were reported.

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
Argentina	Artichoke	0.1	Imidacloprid
	Aubergine	0.3	
	Cabbage, head	0.1	
	Cherry	0.1 T	
	Cucumber	0.1 T	
	Lettuce	0.1	
	Peach	0.1	
	Pepper, sweet	0.1	
	Plum	0.1 T	
	Potato	0.01	
	Tomato	0.3	
Australia	Apple	0.3	Sum of imidacloprid and metabolites containing the 6-chloropyridinyl methylene moiety, expressed as imidacloprid
	Bergamot	5 T	
	Apple pomace, dry	0.05 *	
	Brassica (cole or cabbage) vegetables	0.5	
	Burnet, Salad	5 T	
	Cabbage, head	0.5	

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Celery	0.05 *	
	Chervil	5 T	
	Citrus fruit	0.5 T	
	Coriander, leaves, stems, roots	5 T	
	Coriander, seed	5 T	
	Cotton, fodder, dry	0.1	
	Cotton seed meal, hulls and trash	0.02	
	Cotton, seed	0.02 *	
	Cucurbits	0.2	
	Dill seed	5 T	
	Eggs	0.02 *	
	Fennel seed	5 T	
	Fennel bulb	0.1 T	
	Flowerhead brassicas	0.5	
	Fruiting vegetables except cucurbits	0.5	
	Galangal, greater	0.05 T	
	Herbs	5 T	
	Kaffir lime leaves	5 T	
	Lemon grass	5 T	
	Lemon verbena (fresh weight)	5 T	
	Lupin, dry	0.05 *	
	Maize	0.05	
	Maize fodder	0.05 *	
	Mammalian meat	0.05	
	Mammalian meat by-products	0.2	
	Milks	0.05	
	Mixed pasture (legume/grasses)	1	
	Mizuna	5 T	
	Other cereals, straw and fodder, dry	0.2	
	Other cereals, forage, green	10	
	Other cereals, grain	0.05 *	
	Other cereals, straw	0.2	
	Potato	0.5 T	
	Poultry meat	0.02 *	
	Poultry, Edible offal of	0.02 *	
	Rape (canola) seed	0.05 *	
	Rape seed fodder and forage	0.05 *	
	Rose and dianthus (edible flowers)	5 T	
	Rucola (rocket)	5 T	
	Sorghum	0.02 *	
	Sorghum straw and fodder, dry	0.05 *	
	Sorghum forage, green	2	
	Sorghum straw	0.05 *	
	Stone fruits	0.5	
	Sugar cane	0.05 *T	
	Sugar cane fodder, dry	1 T	
	Sunflower seed	0.02 *	
	Sweet corn (corn-on-the-cob)	0.02 *	
	Sweet corn forage	10	
	Sweet potato	0.05 *T	
	Turmeric root (fresh)	0.05 *T	
Austria	Beet, sugar	0.1	Imidacloprid
	Hops	2 T	
	Maize/corn	0.1	
	Other plant commodities	0.05	
	Pome fruit	0.5 T	
	Potato	0.1	
	Rape	0.1	
	Sunflower seed	0.1	
Belgium	Apple	0.2	Imidacloprid and its metabolites, containing the 6-chloropicolyl moiety, expressed as imidacloprid
	Other plant commodities	0.05 *	
Brazil	Aubergine	0.5	Imidacloprid
	Bean	0.5	
	Broccoli	0.5	

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Cabbage, white	0.5	
	Cauliflower	0.5	
	Chicory, witloof	0.5	
	Citrus fruit	1	
	Collard	0.5	
	Cotton	0.5	
	Cucumber	0.5	
	Endive	0.5	
	Garlic	0.5	
	Gilo	0.5	
	Gourd	0.5	
	Lettuce, head	0.5	
	Maize/Corn	0.5	
	Melon	0.5	
	Onion	0.5	
	Pepper, sweet	0.5	
	Pineapple	0.5	
	Potato	0.5	
	Pumpkin	0.5	
	Rice	0.05	
	Sugar cane	0.5	
	Tomato	0.5	
	Wheat	0.5	
	Watermelon	0.5	
Canada	Apple	0.5	Imidacloprid, including metabolites, containing the 6-chloropicolyl moiety, expressed as imidacloprid
	Brassica vegetables	3.5	
	Citrus fruit	1	
	Cucumber	0.5	
	Cotton seed oil	0.05	
	Grape	1.5	
	Lettuce	3.5	
	Mango	0.2	
	Mustard seed	0.05	
	Other food	0.1	
	Pear	0.6	
	Pecans	0.05	
	Peppers	1	
	Potato	0.3	
	Rapeseed (canola)	0.05	
	Tomato	1	
	Tomato juice	1	
	Tomato paste	6	
	Tomato purée	3	
Croatia	Aubergine	0.2 T	Imidacloprid
	Bean	0.2	
	Beet, sugar	0.05	
	Cabbage	0.2 T	
	Cauliflower	0.2 T	
	Cereals	0.05	
	Citrus fruit	0.5 T	
	Cucumber	0.2 T	
	Forage crops	10 T	
	Fruit	0.5	
	Hop	3	
	Lettuce	1 T	
	Meat	0.05	
	Meat, preparations of	0.05	
	Melon	0.2	
	Milk	0.02	
	Milk products	0.02	
	Pea, garden	0.2 T	
	Pepper, sweet	0.5 T	
	Potato	0.2	
	Tobacco, dry	10 T	
	Tomato	0.2 T	

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Watermelon	0.2	
Cuba	Bean, dry	0.5	Imidacloprid
	Pepper, sweet	0.3	
	Potato	0.5	
	Tomato	0.3	
Denmark	Beets (<i>Beta vulgaris</i>)	0.05 *	Sum of Imidacloprid and its metabolites containing the 6-chloropyridinyl methylene moiety, expressed as imidacloprid
	Cauliflower	0.2	
	Chinese cabbage	0.2	
	Cucumber	0.5	
	Lettuce	0.2	
	Pepper, sweet	0.5	
	Tomato	0.2	
France	Apple	0.3	Imidacloprid
	Apricot	0.3	
	Barley	0.1	
	Beets (<i>Beta vulgaris</i>)	0.1 T	
	Buckwheat, common	0.1	
	Maize/corn	0.1	
	Melon	0.1	
	Oat	0.1	
	Peach	0.3	
	Pear	0.3	
	Rye	0.1	
	Sunflower	0.1	
	Triticale	0.1	
	Wheat	0.1	
Germany	Apricots	0.3	Imidacloprid incl. the degradation and reaction products, determined as 6-chloronicotinic acid; calculated as imidacloprid
	Aubergine	0.3	
	Citrus fruit	0.5	
	Cucurbits with inedible peel	0.1	
	Endive	1	
	Hops	2	
	Lettuce	1	
	Other plant commodities	0.05 *	
	Peaches including nectarines	0.3	
	Peppers	1	
	Pome fruit	0.5	
	Potatoes	0.2	
	Tomatoes	0.3	
Greece	Citrus fruit	0.26 T	Imidacloprid
	Cotton seed	1 T	
	Peach	0.5 T	
	Tobacco, fermented	5 T	
	Tobacco, green	2 T	
Hungary	Beet, sugar	0.1	Imidacloprid
	Maize/corn	0.1	
	Peach	0.1	
	Pepper, sweet	0.2	
	Potato	0.05	
	Sunflower	0.1	
	Tobacco	1	
Israel	Tomato	0.2	Imidacloprid
	Almond	0.3	
	Apple	0.3	
	Citrus fruit	0.5	
	Cucumber	0.1	
	Melon	0.1	
	Peach	0.3	
	Pecan nut	0.3	
	Squash, summer	0.1	
	Tomato	0.3	
Italy	Watermelon	0.1	Imidacloprid
	Apple	0.5	
	Apricot	0.3	
	Aubergine	0.3	

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Beet, sugar	0.05	
	Cherry	0.3	
	Clementine	1	
	Lemon	1	
	Maize/Corn	0.05	
	Mandarin	1	
	Melon	0.1	
	Orange	1	
	Peach	0.3	
	Pear	0.5	
	Pepper, sweet	1	
	Plum	0.3	
	Potato	0.05	
	Sunflower seed	0.05	
	Tobacco leaf, dried	50	
	Tobacco leaf, green	10	
	Tomato	0.3	
	Watermelon	0.1	
Japan	Apple	1	Imidacloprid
	Aubergine	1	
	Bean (pods and/or immature seeds)	0.5	
	Beet, sugar	0.1	
	Berries and small fruit	1	
	Cabbage	0.5	
	Cereals except wheat	0.1	
	Cherry	1	
	Cucumber	1	
	Mango	1	
	Melon	1	
	Other citrus fruit	1	
	Pear, oriental	1	
	Pepper, sweet	5	
	Plum	1	
	Potato	0.1	
	Pulses	0.1	
	Radish, Japanese	2	
	Rice	0.2	
	Satsuma	0.3	
	Soya	0.1	
	Spinach and similar	2	
	Tea	10	
	Tomato	1	
	Watermelon	0.5	
Malaysia	Aubergine	0.2	Imidacloprid
	Pepper, sweet	0.2	
	Rice	0.2	
Netherlands	Pome fruit	0.2	Imidacloprid and its metabolites,
	Cucurbits with edible peel	0.5	containing the 6-chloropicolyl
	Solanacea	0.5	moiety, expressed as imidacloprid
	Other plant commodities	0.05 *	
Philippines	Mango	0.4 T	Imidacloprid
Portugal	Apple	0.5	Imidacloprid, incl. the degradation
	Grape	0.2 T	and reaction products, determined as
	Melon	0.1	6-chloronicotinic acid; calculated as
	Orange	0.5	imidacloprid
	Other citrus fruit	0.5	
	Peach	0.3	
	Pear	0.5	
	Pepper, sweet	0.5	
	Potato	0.05 *	
	Tomato	0.5	
South Africa	Broccoli	0.05 T	Imidacloprid
	Brussels sprouts	0.05 T	
	Cauliflower	0.05 T	
	Citrus fruit	0.5	

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Cotton seed	0.05	
	Cucurbits	0.05	
	Maize/corn green	0.05	
	Sorghum	0.02	
	Sunflower seed	0.02	
	Tobacco, dry	0.5	
	Tobacco, fresh	0.2	
	Tomato	0.1 T	
	Wheat	0.02	
South Korea	Apple	0.5	Imidacloprid
	Aubergine	1	
	Cabbage	3.5	
	Cabbage, Chinese	3.5	
	Citrus fruit	0.5	
	Collard	3.5	
	Cotton seed	6	
	Cucumber	0.5	
	Grape	1	
	Hop	3	
	Mango	0.2	
	Pe-tsai	3.5	
	Pepper, cayenne-	1	
	Pepper, sweet	1	
	Potato	0.3	
	Rice	0.05	
	Sorghum grain	0.05	
	Tomato	1	
	Watermelon	0.1	
Spain	Apricot	0.5	Imidacloprid, incl. the degradation and reaction products, determined as 6-chloronicotinic acid; calculated as imidacloprid
	Aubergine	0.1	
	Bean with pods	0.5	
	Beet, sugar	0.05 *	
	Beets (<i>Beta vulgaris</i>) tops or leaves	0.2	
	Berries and small fruit	0.05 *	
	Bulb vegetables	0.05 *	
	Cacao	0.05 *	
	Cereals	0.05 *	
	Cereal straw	0.5	
	Cherry	0.2	
	Citrus fruit	1	
	Coffee	0.05 *	
	Cola	0.05 *	
	Corn, sweet	0.05 *	
	Cucumber	0.1	
	Cucurbits with inedible peel	0.05 *	
	Flowering brassicas	0.3	
	Fruit and vegetables, dried	0.05 *	
	Gherkin	0.1	
	Herbs	0.05 *	
	Hop	2	
	Lettuce	3	
	Mushroom	0.05 *	
	Nectarine	0.5	
	Nuts	0.05 *	
	Oil plants, seed	0.05 *	
	Other Brassica vegetables	0.05 *	
	Other cucurbits with edible peel	0.05 *	
	Other forage crops	0.05 *	
	Other leafy vegetables	0.05 *	
	Other legume vegetables	0.05 *	
	Other solanacea	0.05 *	
	Other stone fruit	0.05 *	
	Peach	0.5	
	Pepper, sweet	0.5	
	Plum	0.1	

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Pome fruit	0.5	
	Potato	0.05 *	
	Pulses	0.05 *	
	Root and tuber vegetables	0.05 *	
	Spices	0.05 *	
	Stem vegetables	0.05 *	
	Sugar cane	0.05 *	
	Tea	0.05 *	
	Tobacco	5	
	Tomato	0.1	
	Tropical and subtropical fruit	0.05 *	
	Zucchini	0.1	
Sweden	Rape seed	0.05 *	Imidacloprid
Switzerland	Beet, sugar	0.01	Imidacloprid
	Maize/corn	0.01	
	Pome fruit	0.05	
	Rape seed	0.01	
Taiwan	Melon, netted (musk)	0.5	Imidacloprid
	Rice	0.5	
Turkey	Pear	0.05	Imidacloprid
	Tobacco	10	
	Tomato	0.5	
Uruguay	Fruit	1	Imidacloprid
	Potato	0.5	
	Rice	0.2	
	Vegetables	0.5	
USA			Imidacloprid and its metabolites, containing the 6-chloropyridinyl moiety, expressed as imidacloprid
	Almond	0.05 T	expires 31.12.2003
	Almond, hulls	4.0 T	expires 31.12.2003
	Apple pomace, wet	3 F	
	Aubergine	1	
	Barley grain	0.05	
	Barley hay	0.5	
	Barley straw	0.5	
	Bean with pod	1	
	Bean without pod	1	
	Beet, sugar molasses	0.3	
	Beet, sugar root	0.05	
	Beet, sugar top or leaves	0.5	
	Beetroot root	0.3 R,T	expires 30.06.2002
	Beetroot tops	3.5 R,T	expires 30.06.2002
	Blueberry	1 R,T	expires 31.12.2003
	Broccoli	3.5	
	Brussels sprouts	3.5	
	Cabbage	3.5	
	Cardoon	6	
	Cattle fat	0.3	
	Cattle meat	0.3	
	Cattle meat by-products	0.3	
	Cauliflower	3.5	
	Celery	6	
	Citrus fruit	0.7	
	Citrus fruit pulp, dry	5	
	Chard, Swiss	6	
	Coriander, common (Cilantro)	3.5	
	Corn, sweet (corn-on-the-cob)	0.05	
	Corn, sweet stover	0.2	
	Corn, sweet forage	0.1	
	Corn, sweet grain	0.05	
	Cotton gin by-products	4	
	Cotton meal	8 F	
	Cotton seed	6	
	Cranberry	0.5 R,T	expires 31.12.2003

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Cucurbits	0.5	
	Dasheen leaf	3.5	
	Eggs	0.02	
	Fennel, Florence	6	
	Goat fat	0.3	
	Goat meat	0.3	
	Goat meat by-products	0.3	
	Grape	1	
	Grape juice	1.5 F	
	Grape raisin	1.5 F	
	Grape raisin waste	15 D	
	Grape pomace, wet/dry	5 F	
	Hop dry	6	
	Horse fat	0.3	
	Horse meat	0.3	
	Horse meat by-products	0.3	
	Legume vegetables	1 T	expires 30.06.2002
	Legume vegetables foliage	2.5	
	Legume vegetables seed	0.3	
	Lettuce	3.5	
	Maize/corn forage	0.1	
	Maize/corn grain	0.05	
	Maize/corn fodder	0.2	
	Mango	0.2	
	Milk	0.1	
	Other cereals fodder	2	
	Other cereals forage	2	
	Other cereals grain	0.05	
	Other cereals hay	6	
	Other cereals stover	0.3	
	Other cereals straw	3	
	Other leafy vegetables	3.5	
	Pecan nut	0.05	
	Pepper, sweet	1	
	Pig fat	0.3	
	Pig meat	0.3	
	Pig meat by-products	0.3	
	Plum, prune	10 T	expires 31.12.2003
	Pome fruit	0.6	
	Potato	0.3	
	Potato chips	0.4 F	
	Potato waste	0.9 D	
	Poultry fat	0.05	
	Poultry meat	0.05	
	Poultry meat by-products	0.05	
	Rape canola seed	0.05	
	Rhubarb	6	
	Safflower meal	0.5	
	Safflower seed	0.05	
	Sheep fat	0.3	
	Sheep meat	0.3	
	Sheep meat by-products	0.3	
	Sorghum forage	0.1	
	Sorghum grain	0.05	
	Sorghum stover	0.1	
	Soya meal	0.5	
	Stone fruit	3.0 T	expires 31.12.2003
	Strawberry	0.1 R,T	expires 31.12.2004
	Tomato	1.0	
	Tomato paste	6.0 F	
	Tomato pomace wet/dry	4.0 D	
	Tomato purée	3.0 F	
	Tuberous and corm vegetables	0.3	
	Turnip, edible root	0.3 R,T	expires 30.06.2002
	Turnip, edible leaf	3.5	

Country	Commodity	MRL (mg/kg)	Definition of the residue, Remarks
	Watercress	3.5	
	Wheat forage	7.0	
	Wheat grain	0.05	
	Wheat hay	0.5	
	Wheat straw	0.5	
Yugoslavia	Potato	0.5	Imidacloprid (definition of the residue not given, MRL from certificate)

T temporary tolerance

R regional tolerance

E export tolerance

F food-additive tolerance

D feed-additive tolerance

* MRL set at or about the limit of analytical quantification

APPRAISAL

Residue and analytical aspects of the new insecticide imidacloprid [1-(6-chloro-3-pyridylmethyl)-*N*-nitroimidazolidin-2-ylideneamine], which acts as an agonist at postsynaptic nicotinic acetylcholine receptors of insects, were considered for the first time by the present Meeting.

The 2001 JMPR established an ADI of 0.06 and an acute RfD of 0.4 mg/kg bw.

The manufacturer sent the Meeting information on metabolism in animals and plants, environmental fate in soil and water, methods of residue analysis and stability of residues in stored analytical samples, uses, residue supervised trials and processing data as well as national MRLs. Information on national GAP data and MRLs were provided by the governments of Australia, Germany and The Netherlands.

Pure imidacloprid is a beige powder with a melting point of 144°C and low volatility. It has low solubility in water and medium to high solubility in certain organic solvents. The log P_{OW} of 0.57 suggests that the compound is not fat soluble.

The parent, metabolites and degradation products are identified by code numbers as shown below.

Code	Chemical name	Short name
	<u>1-(6-chloro-3-pyridylmethyl)-<i>N</i>-nitroimidazolidin-2-ylideneamine imidacloprid</u>	
<u>M01</u>	1-(6-chloro-3-pyridylmethyl)-5-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine	5-hydroxy compound
<u>M02</u>	1-(6-chloro-3-pyridylmethyl)-4-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine	4-hydroxy compound
<u>M03</u>	1-(6-chloro-3-pyridylmethyl)-4,5-dihydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine	dihydroxy compound
<u>M04</u>	1-(6-chloro-3-pyridylmethyl)-5-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine glucuronide 5-hydroxy glucuronide	glucuronide
<u>M05</u>	1-(6-chloro-3-pyridylmethyl)-4-hydroxy- <i>N</i> -nitroimidazolidin-2-ylideneamine glucuronide 4-hydroxy glucuronide	4-hydroxy glucuronide
<u>M06</u>	1-(6-chloro-3-pyridylmethyl)- <i>N</i> -nitro-4-imidazolin-2-ylideneamine	olefin
<u>M07</u>	1-(6-chloro-3-pyridylmethyl)- <i>N</i> -nitrosoimidazolidin-2-ylideneamine	nitrosimine
<u>M08</u>	1-(6-chloro-3-pyridylmethyl)- <i>N</i> -aminoimidazolidin-2-ylideneamine	amino compound
<u>M09</u>	1-(6-chloro-3-pyridylmethyl)imidazolidin-2-ylideneamine	denitro compound
<u>M10</u>	1-(6-chloro-3-pyridylmethyl)guanidine sulfate	guanidine sulfate
<u>M11</u>	1-(6-chloro-3-pyridylmethyl)-2-nitroguanidine	nitroguanidine
<u>M12</u>	1-(6-chloro-3-pyridylmethyl)imidazolidin-2-one	2-ketone

M13	1-(6-chloro-3-pyridylmethyl)urea	urea compound
M14	6-chloronicotinic acid	6-CNA
M15	<i>N</i> -(6-chloronicotinoyl)glycine	
M16	6-chloro-3-pyridylmethylamine	
M17	1-(6-chloro-3-pyridylmethyl)-4,5-dihydroxyimidazolidin-2-ylideneamine	dihydroxyimine
M28	6-chloro-3-pyridylmethanol	CHMP
M29	6-chloro-3-pyridylmethanol glucoside	CHMP glucoside
M30	6-chloro-3-pyridylmethanol gentiobioside	

Animals metabolism

The rat metabolism was reviewed by the 2001 JMPR. Metabolites identified in urine and faeces as well as in kidney and liver are 6-chloronicotinic acid [M14] and its glycine conjugate [M15], further M01, M02, M06 and M09.

Absorption, distribution and elimination of imidacloprid was a rather fast process in lactating goat and laying hen after administration of 3 oral doses of 10 mg/kg bw on 3 consecutive days. Within 50 hours after the first administration the excretion amounted to about 54 % (goat) and 50 % (hen) of the radioactivity totally administered until sacrifice. Excretion with urine was the predominant route of elimination in goat, accounting for about 43 % of the dose. Faecal excretion was low with about 11 % of the total dose. Although the excrete of birds represents a mixture of urine and faeces, it can be concluded from the high concentration in the kidneys that the bulk of the radioactivity was excreted with the urinary fraction of the excreta. An amount of 0.3 % of the total dose was secreted with the milk of goat. At sacrifice, 2 hours after the final administration, the total residue in the edible organs was estimated to be about 5 % in goat. 7.8 % were found in tissues of hens.

The extent of metabolism of imidacloprid in kidney of goat and in liver of goat and poultry was very high. In muscle and fat tissues of goat about 65 % of total radioactive residues (TRR) were identified as imidacloprid. In milk about 10 % of TRR was identified as imidacloprid, 24 h after each application. In laying hens imidacloprid amounted to about 5 % of TRR in eggs, 6 % in muscle and 12 % in fat.

The metabolism of imidacloprid in goat and laying hen followed three similar, but not identical degradation routes with different metabolites as follows:

Goats

The first step of metabolism the hydroxylation of the imidazolidine ring of imidacloprid took place to form 5-hydroxy and 4-hydroxy imidacloprid [M01, M02] plus the glucuronide conjugate of the monohydroxy metabolites [M04, M05], and the dihydroxy imidacloprid [M03] followed by the loss of water to form the olefin metabolite [M06].

After reduction and loss of the nitro group on the imidazolidine ring the amino metabolite [M08] was formed, followed by the denitro compound [M09] and finally the 2-ketone [M12].

The third route followed opening of the imidazolidine ring by removal of the ethylene bridge and subsequent oxidation. The first step is forming the nitroguanidine compound [M11] followed by guanidine sulfate [M10] which can also be formed from both the denitro metabolite [M09] and the dihydroxyimine metabolite [M17]. This metabolite M10 can form the urea compound [M13] and [M16]. A further degradation to 6-chloronicotinic acid [M14] took place which conjugated with glycine [M15].

Hens

The first important biodegradation step starts with the hydroxylation of the imidazolidine ring to form the 5- and 4-hydroxy imidacloprid [M01, M02]. The loss of water yields the olefinic compound [M06]. These three metabolites accounted for about 25 - 38% of the identified radioactivity.

The reduction and loss of the nitro group on the imidazolidine ring yielded dihydroxyimine [M17].

A third route of degradation follows opening of the imidazolidine ring with loss of the ethyl group and subsequent oxidation. The first step is forming the nitroguanidine [M11] followed by guanidine sulfate [M10] that can also be formed by the dihydroxyimine metabolite [M17]. This metabolite M10 can form the urea compound [M13] and [M16] which is oxidized to 6-chloronicotinic acid [M14].

Plants metabolism

The fate of imidacloprid in plants was investigated with [pyridinyl-¹⁴C-methyl]-imidacloprid in 11 different plant species using three different application forms. Ten metabolism studies and one confined rotational crop study were performed:

foliar spray treatment	apple, tomato, potato
soil granular application	eggplant, potato, rice
soil granular plus foliar spray application	tobacco
seed treatment	maize, cotton
nursery box treatment	rice

In most crops (eggplant, potatoes, rice, cotton) uptake of imidacloprid from the soil after granular application or seed treatment was low, ranging from 1.8 to 4.9 % of the applied radioactivity in the aerial part of mature plants. In rice and eggplants (in cotton and potatoes this question was not investigated) uptake was completed after half a growing period and did not increase appreciably in the second half. In maize plants radioactivity increased continuously to the end of the growing period and amounted to finally 20 % of the applied radioactivity in mature plants.

In all studies it was found that translocation in the plants goes off obviously by acropetal transport mainly from the roots to the leaves. After soil application, the main part of the radioactivity was found in the foliage, while only minor amounts were detected in fruits, grain or seed. A trial with spray application in potatoes showed that transport from the top (sprayed leaves) to the bottom (tubers) was negligible. Acropetal translocation was also demonstrated in special translocation experiments in apples and tomatoes. 14 days after application of imidacloprid to leaves radioactivity in fruits was negligible while the distribution in other plant parts (shoot, stem, untreated leaves) was not further investigated.

After translocation in the plants imidacloprid was significantly metabolised to a number of metabolites. In all studies and in nearly all plant parts three different routes of metabolic degradation were established:

Hydroxylation of the imidazolidine ring leading to the mono- and dihydroxylated compounds [M01, M02, M03] and subsequent removal of water to form the olefin metabolite [M06].

Reduction of the nitro group to form the nitrosimine compound [M07] and loss of this group with formation of the metabolites M09, M10 and M12.

Oxidative cleavage of the methylene bridge to form 6-chloro-3-pyridylmethanol (and conjugates) [M28, M29, M30] and further oxidation to 6-chloronicotinic acid [M14].

The only exceptions were residues in rice grains after granular application and in potato tubers after spray application. In these cases the total amount of recovered radioactivity was very low so that only very few metabolites could be detected.

Analysis of non-extracted residues in rice and maize grains showed furthermore that degradation of imidacloprid to CO₂ and subsequent incorporation into natural constituents as starch, glutelin or lignin seemed to be possible.

Amounts of unchanged parent compound depended on the application form. After spray application, penetration through the peel into fruits or leaves occurred relatively slow. Consequently, the metabolic degradation of imidacloprid was slow (half-life of imidacloprid in potato vines and tomato fruits: 5 to 7 weeks), and unchanged parent compound was found as the major component up to 88 % of the TRR. Uptake via roots after soil application led in most cases to more intensive biotransformation and to smaller amounts of unchanged imidacloprid.

Environmental fate

The DT50 values of imidacloprid will be generally below 180 days. The parent compound is completely mineralized without the occurrence of any metabolite at concentrations greater than 10 % of the applied radioactivity. Due to its spectral characteristics degradation on soil surfaces can play an important role in the environmental dissipation of imidacloprid. The compound exhibits a low soil mobility with a negligible leaching potential.

The nature of metabolites in the rotational crops was essentially the same as in crops from plant metabolism studies. The following compounds were identified: the denitro compound [M09], 5- and 4-hydroxy compounds [M01 and M02], 6-CNA [M14], olefin compound [M06], CHMP glucoside [M29], dihydroxy compound [M03], guanidine sulfate [M10], nitrosimine compound [M07] and CHMP [M28]. The sum of uptake of radioactivity in all rotational crops together was in the range from 1.1 to 2.4 % of TRR in the soil at the planting dates.

Imidacloprid is stable with regard to hydrolysis in aqueous solutions at environmentally relevant pH-values. In contrast, photolytic degradation occurs rapidly due to the nitro-chromophore. Though generally the photolytic effect is less important under environmental conditions since light of the relevant wavelengths (> 290 nm) will be absorbed by turbidities and impurities to a certain degree, in the case of imidacloprid it must be taken into account. In the water-sediment system the portion translocated to the sediment and converted into bound residues can become large though it is not generally the case. Calculated half-lives for three different water-sediment systems investigated were 30, 129 and 169 days. Complete mineralization occurs slowly but steadily and there is no tendency for accumulation of any of the intermediates.

Methods of analysis

In metabolism studies in plants, all metabolites identified in plants after treatment with imidacloprid contained the 6-chloropicolyl moiety. Therefore, an analytical method was developed for the determination of imidacloprid and the total residues in plants including all compounds containing the 6-chloropicolyl moiety. After extraction with methanol/water and sulphuric acid, hexane partitioning is performed. The extract is further cleaned up via column chromatography with XAD 4 (polystyrene resin). Then imidacloprid and its metabolites containing the 6-chloropyridine moiety are oxidized to 6-chloronicotinic acid with alkaline KMnO₄ solution. Subsequently, the 6-chloronicotinic acid is derivatized with N-methyltrimethylsilyltrifluoro-acetamide (MSTFA), and detected by gas chromatography with mass selective detection (GC-MS). Mean recoveries per sample material and fortification level (0.5 mg/kg and 0.05 mg/kg = LOQ) for the total residue were in the range of 68-113% (n=152). Blank values normally were below 30% of the LOQ.

For the determination of parent compound residues, an aliquot of the extract is evaporated to the aqueous remainder. After partitioning with dichloromethane on a ChemElut[®] cartridge and chromatography on Florisil[®], the residues are detected via HPLC with UV detection. Mean recoveries per sample material and fortification level (0.1 mg/kg and 0.01 mg/kg = LOQ) for the parent compound residues were in the range of 72-114% (n=143). Blank values normally were below 30% of the LOQ.

The method described above was validated in an independent laboratory (ILV). Recoveries were determined with representative sample materials (melon peel and pulp, peppers, tomato) for the total residue and also for parent compound residues at fortification levels of 0.01 to 1 mg/kg. For the total residue, the individual recoveries obtained ranged from 69-112%; the mean recovery per sample material and fortification level ranged from 72-100%, with typical RSDs of approx. 10%. For the parent compound, individual recovery levels were between 68% and 83%; the mean recovery per sample material and fortification level ranged from 70-79%, with typical standard deviations of about 5%. Blank values were below 30% of the corresponding LOQ (0.05 mg/kg for the total residue and 0.01 mg/kg for the parent) in all samples.

Residues of imidacloprid and related metabolites in animal matrices can be determined in a similar manner. Samples are extracted with a mixture of methanol and water (methanol only for milk samples), filtered, and evaporated to the aqueous remainder. For fat samples, partitioning against n-hexane is performed. The extracts are further cleaned up via column chromatography with XAD 4 (polystyrene resin); the column is washed with water, after which the residues are eluted with methanol. Subsequently, imidacloprid and its metabolites containing the 6-chloropyridinyl moiety are oxidized with alkaline KMnO₄ to 6-chloronicotinic acid. The 6-chloronicotinic acid is extracted from the aqueous phase with t-butylmethyl ether and derivatized with N-methyltrimethylsilyltrifluoroacetamide (MSTFA), and then determined by gas chromatography with mass-selective detection (GC-MS). Recovery rates were in the range from 76-124% after spiking animal materials (bovine muscle, kidney, liver, fat, milk; eggs) with imidacloprid at levels of 0.02 and 0.1 mg/kg. The LOQ was 0.02 mg/kg for all materials.

The method for animal matrices was validated in an independent laboratory (ILV). Recoveries determined with representative sample materials (milk, egg, poultry liver) ranged from 72 to 97% at fortification levels of 0.02 and 0.1 mg/kg (milk, egg), and 0.1 and 0.5 mg/kg (liver). Each sample was fortified with a mixture of imidacloprid and two metabolites (M09 denitro compound and M14 6-chloronicotinic acid). No "blank values" from control samples were observed.

Soil samples are extracted with boiling methanol in Soxtec extraction equipment, and subsequently cleaned up over a Chromabond SPE silica gel cartridge. After evaporation of the solvent and reconstitution in acetonitrile/water, the residues are quantified by HPLC with UV detection. Two columns of differing selectivity (LiChrospher 60 B and Zorbax SB-CN) were tested so as to avoid interferences. The recovery rates per spiking level were in a range between 94-101% (LiChrospher) and 88-89% (Zorbax) at fortification levels of 0.01 and 0.1 mg/kg, with respective RSDs of 3.6-6.6% and 3.3-4.3%. The LOQ was 0.01 mg/kg and the limit of detection (LOD) was 0.003 mg/kg. Blank values were below 0.004 mg/kg in all samples.

Imidacloprid is concentrated from water samples by solid phase extraction (C₁₈ cartridges), after which surface water samples are further cleaned up over silica gel cartridges. After evaporation, the residues are determined by HPLC with UV detection. Recoveries for drinking water at fortification levels of 0.03 and 0.3 µg/l were 93% and 96%, respectively, with relative standard deviations of 4.3% and 3.1%. For surface water, the recovery rates were 76% (RSD 5.3%) and 87% (RSD 6.9%).

Stability of residues in stored analytical samples

The storage stability of imidacloprid and various important metabolites (M01, M06, M07, M09, M14) was tested in multiple plant materials and animal tissues, organs, and products. Tests on animal matrices were carried out to assess the stability of the total residue. For plants, tests were carried out to assess the stability both of residues of the active substance itself as well as of the total residue; additional tests were also conducted with radio-labelled substances. The results all of the studies indicate that the compounds are stable in frozen storage in the tested plant commodities for a minimum period of approximately 2 years, and in animal commodities for at least 1 year. Hence, the results of the storage stability studies validate the residue values obtained from the trials presented in this evaluation.

Definition of residue

In the studies on the metabolism of imidacloprid in lactating goat and laying hen imidacloprid and a number of metabolites were detected. The qualitative and quantitative composition of the metabolic spectrum varied among the animal species and tissues. However, all metabolites identified contain the 6-chloropyridyl moiety of imidacloprid.

A rather consistent picture of uptake, translocation and metabolism of imidacloprid in plants was observed. In all crops the metabolic pathway runs via the same routes of degradation and results in qualitatively and quantitatively similar composition of the metabolic spectrum. All identified transformation products of imidacloprid still contain the 6-chloropyridinyl moiety of the parent compound.

Therefore, the relevant residue to be analyzed in products of animal and plant origin can be defined as the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid.

This definition applies for both compliance with MRLs and estimation of dietary intake.

Residues resulting from supervised trials on crops

Citrus fruits. Residue field trials on citrus fruits were performed with spray or soil drench applications of imidacloprid on clementine, grapefruit, lemon, mandarin and orange trees in Europe and USA.

The GAP in Greece, Italy and Spain ranges from 1 - 2 foliar sprays of 0.01 – 0.015 kg ai/hl (Portugal 0.007 – 0.01 kg ai/hl) and PHIs of 14 – 30 days. In Italy, a total of eight residue field trials were conducted with two foliar spray applications of a 200 SL formulation on clementine trees at an interval of 15 - 30 days. In all trials a spray concentration of 0.01 kg ai/hl was applied. At the shortest PHI of 14 days, registered in Italy and Portugal, the total residues in whole fruit were 0.06, 0.07, 0.12, 0.16, 0.21, 0.29, 0.38 and 0.44 mg/kg.

In Italy two residue field trials were conducted with 2 spray applications (interval 30 days) of 0.01 kg ai/hl on lemon trees. Considering the PHI of 14 days, the residues were 0.07 and 0.11 mg/kg in pulp and 0.26 and 0.57 mg/kg in whole fruit.

A total of five residue field trials were performed on mandarin trees with two applications (interval Italy 30, Portugal 34, Spain 118 days) of 0.01 – 0.015 kg ai/hl. In one Italian trial the spray concentration was 0.01 kg ai/hl, corresponding to 0.12 kg ai/ha. In one Portuguese trial a spray concentration of 0.015 kg ai/hl corresponded to 0.2 kg ai/ha. Three further trials (2 x 0.015 kg ai/hl) were performed in Spain, with a first rate of 0.3 kg ai/ha a second rate of 0.75 kg ai/ha was applied. The residues in the edible portion and whole fruit were <0.05, <0.05, 0.05, 0.06 mg/kg and 0.16, 0.16, 0.17, 0.28, 0.29 mg/kg with a 14-day PHI.

In southern European countries a total of 9 residue field trials were performed in orange with two foliar applications of 0.01 – 0.015 kg ai/hl and a 14-day PHI. In two Italian trials orange trees

received two foliar spray applications (interval 30 days), each of a spray concentration of 0.01 kg ai/hl, corresponding to an application rate of 0.12 kg ai/ha. In Spain four trials were performed with 0.01 kg ai/hl at the first and 0.015 kg ai/hl at the second application (interval 101- 130 days), corresponding to rates of 0.3 kg ai/ha and 0.45 - 0.75 kg ai/ha. Three further trials were performed in Greece (2) and Portugal (1). Two spray applications (interval Greece 9 – 10 days, Portugal 31 days) were made each with a spray concentration of 0.015 kg ai/hl, corresponding to a rate of about 0.3 kg ai/ha. The residues in the edible portion were <0.05 (7), 0.05 and in whole fruit 0.11, 0.12, 0.12, 0.16, 0.24, 0.35, 0.44, 0.53 and 0.88 mg/kg with a 14-day PHI.

The combined residues in the European foliar sprayed trials (14-day PHI) in whole clementine, mandarin, lemon and oranges in rank order were: 0.06, 0.07, 0.11, 0.12 (3), 0.16 (4), 0.17, 0.21, 0.24, 0.26, 0.28, 0.29, 0.29, 0.35, 0.38, 0.44, 0.44, 0.53, 0.57, 0.88 mg/kg. The residues in the corresponding edible portion samples were: <0.05 (14), 0.05 (4), 0.06 (2) 0.07 and 0.11 mg/kg.

GAP in USA includes 1- 2 foliar sprays of 0.005 – 0.007 kg ai/hl, 0.14 – 0.28 kg ai/ha and a 0-day PHI. In the USA five residue field trials were performed in lemon with 2 spray applications (interval 9 – 11 days) of imidacloprid at a rate of 0.28 kg ai/ha. The spray concentration was 0.01 – 0.043 kg ai/hl. The residues in whole fruit were 0.21, 0.3, 0.31, 0.38 and 0.62 mg/kg with a 0-day PHI.

A total of six residue field trials were performed in grapefruit, also in the USA. The trees received two foliar spray applications, each at a rate of about 0.28 kg ai/ha. The interval between applications was 10 ($\pm$ 2) days. Spray concentrations were about 0.011 - 0.015 kg ai/hl, resulting in whole fruit residues of 0.14, 0.17, 0.3 mg/kg, and 0.04 - 0.043 kg ai/hl, resulting in whole fruit residues of 0.17, 0.18, 0.32 mg/kg with a 0-day PHI. No difference was seen in the order of magnitude of the residues resulting from the two spray concentrations. Residues in rank order were 0.14, 0.17, 0.17, 0.18, 0.3 and 0.32 mg/kg.

Twelve other US field trials were with 2 spray applications (interval 3 – 13 days) of a 240 SC formulation. trees of each trial were treated with imidacloprid at a rate of 0.28 kg ai/ha. Spray The orange concentrations were 0.011 - 0.015 kg ai/hl, resulting in whole fruit residues of 0.18, 0.26, 0.26, 0.36, 0.37 mg/kg, and 0.04 - 0.043 kg ai/hl, resulting in whole fruit residues of 0.15, 0.21, 0.28, 0.29, 0.34, 0.36, 0.61 mg/kg with a 0-day PHI. No difference was seen in the order of magnitude of residues resulting from both spray concentrations. Residues in rank order were 0.15, 0.18, 0.21, 0.26, 0.26, 0.28, 0.29, 0.34, 0.36, 0.36, 0.37 and 0.61 mg/kg.

The combined residues of the USA foliar sprayed trials (0-day PHI) of whole lemon, grapefruit and oranges were, in rank order: 0.14, 0.15, 0.17, 0.17, 0.18, 0.18, 0.21, 0.21, 0.26, 0.26, 0.28, 0.29, 0.3, 0.3, 0.31, 0.32, 0.34, 0.36, 0.36, 0.37, 0.38, 0.61, 0.62 mg/kg.

Three residue field trials were conducted in South Africa with soil drench application in oranges. The trees of each test plot had been treated around the trunks with a single label use rate of 2 – 8 g ai/tree. Oranges were sampled 179 and 212 days after treatment. Because only the parent compound imidacloprid was analyzed, the results were not used for estimation of maximum residue levels.

In the USA a total of twenty residue trials on citrus fruits were performed with soil application according to GAP (max. 0.56 kg ai/ha). In 1993 a total of twelve residue field trials were performed on grapefruit trees (6 trials) and orange trees (6 trials). The trees of each field trial received one application to the soil at a rate of 0.56 kg ai/ha. The application was made either late in spring or in fall. After late spring treatment, grapefruit and oranges were harvested after 120, 150, 180, 210, 240, 270, and 365 days. With treatment in fall, grapefruit and oranges were harvested after 0, 7, 15, 30, 60, 90, 120, and 150 days. The highest residues in each trial were <0.05 (5) and 0.05 mg/kg in whole grapefruit and <0.05 (3), 0.06, 0.08, 0.12 mg/kg in whole oranges, from samples at all sampling dates.

An additional three grapefruit, three lemon, and two orange residue trials with soil treatment were performed in 1994 - 1995. The trees of each field trial also received one application to the soil at a rate of 0.56 kg ai/ha. Mature grapefruits, oranges, and lemons were harvested 0, 4, 7, 15, 30, 56 to 62, about 90, 119 to 120, 149 to 153, 208 to 215, 240 to 244, 270 to 274, and about 365 days after treatment. The residues were <0.05 (3) mg/kg in whole grapefruit, <0.05 (3) in whole lemon, and <0.05 (2) mg/kg in whole orange, at all sampling dates.

The combined residues of the USA soil treatment trials of whole lemon, grapefruit and oranges in rank order were: <0.05 (16), 0.05, 0.06, 0.08, 0.12 mg/kg. These residues were considered to belong to a different population from those resulting from foliar spray use and were excluded from the evaluation.

The Meeting noted that the data obtained by the USA and Europe for whole fruits of clementine, mandarin, lemon, grapefruit and orange with foliar treatment, constituted one population. The combined residues for whole fruits of the two data sets from the USA and Europe were: 0.06, 0.07, 0.11, 0.12 (3), 0.14, 0.15, 0.16 (4), 0.17 (3), 0.18, 0.18, 0.21 (3), 0.24, 0.26 (3), 0.28, 0.28, 0.29 (3), 0.3, 0.3, 0.31, 0.32, 0.34, 0.35, 0.36, 0.36, 0.37, 0.38, 0.38, 0.44, 0.44, 0.53, 0.57, 0.61, 0.62 and 0.88 mg/kg. The Meeting estimated a maximum residue level of 1 mg/kg for citrus fruits.

The residue concentrations in the edible portion samples of the European trials were: <0.05 (14), 0.05 (4), 0.06 (2), 0.07 and 0.11 mg/kg. The Meeting estimated an STMR of 0.05 mg/kg and an HR of 0.11 mg/kg for citrus fruits on the basis of foliar spray use.

Pome fruits. Residue field trials with imidacloprid on apples were performed with foliar spray treatment in Canada, Europe, Korea, South Africa and the USA, and with soil drench applications in Australia and South Africa.

The GAP for apples in northern Europe (Austria, northern France, Germany, the Netherlands) includes 1 – 2 foliar spray treatments of 0.007 kg ai/hl (0.07 – 0.11 kg ai/ha), with a PHI of 14 days. Seven residue trials were carried out according to GAP in Germany. In these trials, one or two spray or low-volume spray applications were performed (interval 14 – 21 days). With a water application rate of 1500 l/ha, the spray concentration was 0.007 kg ai/hl, corresponding to 0.11 kg ai/ha. With a water rate of 200 or 250 l/ha, the concentration ranged from 0.052 to 0.063 kg ai/hl, corresponding to 0.11 to 0.13 kg ai/ha. After a 14-day PHI, the total residue concentrations were <0.05 (3), 0.06, 0.07, 0.08 and 0.11 mg/kg.

The GAP in southern Europe (Italy, Portugal, Spain) for apples includes 1 – 2 foliar spray treatments with 0.01 kg ai/hl (0.1 - 0.15 kg ai/ha) and PHIs from 14 – 28 days. 13 residue trials were performed in Italy, Spain and France. In all except one trial, one or two applications were made at spray concentrations ranging from 0.008 to 0.01 kg ai/hl, corresponding to 0.08 to 0.15 kg ai/ha. The remaining trial was performed with 2 applications including one pre-blossom application at a concentration of 0.02 kg ai/hl (0.3 kg ai/ha). With a 14-day PHI, the total residue concentrations were <0.05, 0.06, 0.06, 0.06, 0.07, 0.08, 0.08, 0.13, 0.17, 0.17, 0.18, 0.2 and 0.23 mg/kg.

In Canada and the USA, the GAP for apples includes 1 – 2 foliar spray treatments with about 0.05 – 0.1 kg ai/ha (0.0015 – 0.003 kg ai/hl) and a 7-day PHI. A total of 14 residue trials were performed with 5 x of 0.07 – 0.19 kg ai/ha. With a 7-day PHI, the total residues ranged from <0.05 to 0.74 mg/kg. The Meeting noted that the trials were inadequate because they did not reflect the GAP.

In South Korea, 5 apple trials were performed with a foliar spray concentration of 0.005 kg ai/hl. Two to 6 treatments were made at an application rate of 0.25 kg ai/ha. Because the parent imidacloprid was determined instead of the total residue, the trials could not be used for evaluation.

Imidacloprid is registered in South Africa for apples with one foliar spray treatment of 0.021 kg ai/hl (0.51 – 0.74 kg ai/ha) and a 70-day PHI or one soil drench treatment with 1.1 g ai/tree and no fixed PHI. Three residue trials were performed according to GAP by foliar spray (1 x 0.021 kg ai/hl, 0.53 kg ai/ha, 65 – 79 days PHI) and showed residues of 0.07, 0.08 and 0.12 mg/kg. In three trials with one soil drench application of 1 g ai/tree, no residue higher than the LOQ could be determined at PHIs of 69 – 154 days (<0.01, <0.02, <0.03 mg/kg).

In Australia, soil drench application of imidacloprid in apples is registered with 0.6 – 2.4 g ai/tree without a fixed PHI. Five residue trials were conducted according to GAP (1 x 2.4 g ai/tree, PHIs 91 – 110 days) and resulted in residue concentrations of <0.05, 0.02, 0.03, 0.14 and 0.16 mg/kg.

The combined apple residue results of the 20 European trials with foliar spray and the eight trials from South Africa and Australia with soil drench application were, in rank order: <0.01, <0.02, 0.02, <0.03, 0.03, <0.05 (5), 0.06, 0.06, 0.06, 0.06, 0.07 (3), 0.08 (4), 0.11, 0.12, 0.13, 0.14, 0.16, 0.17, 0.17, 0.18, 0.2 and 0.23 mg/kg. The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in apples of 0.5, 0.07 and 0.23 mg/kg, respectively.

Residue field trials with imidacloprid as foliar spray treatment on pears were performed in Europe, Canada and the USA. The GAP in northern Europe (northern France, the Netherlands) for pears is the same as for apples and includes 1 – 2 foliar spray treatments of 0.007 kg ai/hl (0.07 – 0.11 kg ai/ha) and a PHI of 14 days. The GAP in southern Europe (Italy, Portugal, Spain) for pears is the same as for apples and includes 1 – 2 foliar spray treatments with 0.01 kg ai/hl (0.1 – 0.15 kg ai/ha) and a PHI of 14 days (Italy 50 days). A total of 8 GAP residue trials were performed with foliar spray application in southern Europe, one in Greece, four in Italy and three in Spain. Only one application was carried out in the Greek and Spanish studies; two applications were carried out in Italy (interval 21 or 139 days). In 2 of the 4 Italian studies, the first treatment was a pre-blossom application at a rate of 0.3 kg ai/ha. The spray concentration was 0.01–0.012 kg ai/hl, corresponding to 0.1–0.18 kg ai/ha. The residue concentrations were <0.05, 0.05, 0.06, 0.07, 0.08, 0.1, 0.23 and 0.26 mg/kg after a 14-day PHI.

In the USA, imidacloprid is registered in pears with 1- 2 foliar spray applications of 0.28 kg ai/ha, 0.0075 kg ai/hl and a 7-day PHI, and cannot be compared with the GAP for apples (0.05 – 0.1 kg ai/ha, 0.0015 – 0.003 kg ai/hl, 7-day PHI). Residue trials in pears were carried out in Canada and the USA with 2 methods of foliar spray application. Two treatments were made in each trial. Five studies were performed with a concentrated spray volume, and 4 with a diluted spray volume. The spray concentration ranged from 0.06 to 0.063 kg ai/hl for the “concentrate” sprays, and from 0.01 to 0.015 kg ai/hl for the “dilute” ones. This corresponded to an application rate of 0.28–0.31 kg ai/ha. With a 7-day PHI, the residues were 0.25, 0.27, 0.33, 0.33, 0.38, 0.4, 0.5, 0.53 and 0.71 mg/kg.

The Meeting compared both pear data sets from Europe and the USA by the Mann-Whitney U-test (see FAO Manual, p. 73) and decided that they belonged to different populations and could not be combined. Based on the US data set, the Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in pears of 1, 0.38 and 0.71 mg/kg, respectively.

Stone fruits. Imidacloprid is registered in southern France and Greece in peach with 1 - 2 foliar spray treatments of 0.005 – 0.007 kg ai/hl and in Italy, Portugal and Spain with 0.01 kg ai/hl. The PHI is 14/15 days with exception of Italy with 21 days. An identical GAP like for peach exists in Spain for nectarine, in France for apricot, in Spain for apricot and nectarine and, apart from the PHI of 35 days, in Italy for apricot. No trials according to GAP were carried out in apricots.

A total of 20 peach residue trials were performed in southern Europe according to the registered uses. Some of samples were separated in flesh and stones and the residue in whole fruit was calculated, in other cases whole fruits were analysed. The use patterns in 6 French studies (1 x 0.007 kg ai/hl, 0.07 kg ai/ha) were according to the French GAP resulting, after a 14-day PHI, in residue concentrations of <0.05, 0.07, 0.07, 0.11 mg/kg in fruits without stone and of <0.05, 0.06, 0.06, 0.06,

0.06 and 0.1 mg/kg in whole fruits. In the remaining 14 trials from Greece (1 trial), Italy (5 trials) and Spain (7 trials), 2 applications (interval 19 – 30 days) were carried out at a spray concentration of 0.01 kg ai/hl (0.008 kg ai/hl in 2 trials). The application rates ranged between 0.08 and 0.15 kg ai/ha. The trials matched the GAP from Italy, Portugal and Spain and showed, after a 14-day PHI, residues of <0.05, 0.07, 0.07, 0.13, 0.16, 0.22, 0.26, 0.32 mg/kg in fruits without stone and of <0.05, <0.05, 0.06, 0.06, 0.11, 0.11, 0.12, 0.15, 0.15, 0.19, 0.2, 0.2, 0.29, 0.35 mg/kg in whole fruits. Four further trials from Australia could not be used for evaluation because only the parent compound imidacloprid was determined.

Three nectarine residue trials were performed in Italy. In each trial, two applications (interval 30 or 142 days) were carried out at a spray concentration of 0.01 kg ai/hl (except for one trial in which the first treatment was a pre-blossom application at a concentration of 0.02 kg ai/hl). The application rates ranged from 0.12 to 0.15 kg ai/ha (0.24 kg ai/ha for the pre-blossom application). The last trial showed no residue higher than the LOQ at any sampling time, including the initial residue after treatment, and was therefore excluded from evaluation. The residues in the two remaining trials were 0.13 (2) mg/kg in fruits without stone and 0.12 (2) mg/kg in whole fruits after the shortest southern European PHI of 14 days.

The Meeting noted that the residue data sets for peaches and nectarines can be combined and were in whole fruits <0.05 (3), 0.06 (6), 0.1, 0.11, 0.11, 0.12 (3), 0.15, 0.15, 0.19, 0.2, 0.2, 0.29 and 0.35 mg/kg. Based on identical GAPs in southern Europe, the residue levels estimated for peaches and nectarines should be extrapolated for apricots. The Meeting estimated a maximum residue level for imidacloprid in peaches, nectarines and apricots of 0.5 mg/kg.

The combined residues in the edible portion of peaches and nectarines were <0.05, <0.05, 0.07 (4), 0.11, 0.13 (3), 0.16, 0.22, 0.26, 0.32 mg/kg. The Meeting estimated an STMR value and an HR value for imidacloprid in peaches, nectarines and apricots of 0.12 and 0.32 mg/kg, respectively.

Imidacloprid is registered for use in sweet cherries in Italy with one foliar spray treatment, 0.15 kg ai/ha, 0.01 kg ai/hl and a 21-day PHI. Nine field studies were performed on sweet cherry in southern Europe with a 200 SL formulation: 6 in Italy and 3 in Spain. Five trials made in Italy were performed with 2 foliar applications (interval 30 days) at a spray concentration of 0.01 kg ai/hl, except for one in which the first application was carried out at a concentration of 0.02 kg ai/hl (interval 67 days). With a 21-day PHI, residues in whole fruits (or fruits without stone) were 0.11, 0.14 (0.17), 0.15, 0.15, 0.28 (0.3) mg/kg. In the remaining 4 trials performed in Spain and Italy, only one application was carried out at a spray concentration of 0.01 kg ai/hl, resulting in residue concentrations in whole fruits of 0.07, 0.08, 0.12, 0.16 mg/kg after a 21-day PHI. Four further trials from Australia could not be used for evaluation because only the parent compound imidacloprid was determined.

The Meeting noted that in the case of two applications with intervals of 30 – 67 days only the last one is of importance for the concentration of residues, and therefore the results from trials with one and two applications were combined: 0.07, 0.08, 0.11, 0.12, 0.14, 0.15, 0.15, 0.16, 0.28 mg/kg. As for two of nine trials only results for the edible portion were available, the STMR and HR was derived from the whole fruit data set. The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in cherries, sweet, of 0.5, 0.14 and 0.28 mg/kg, respectively.

The current French label indicates imidacloprid may be applied on plums 1 – 2 times at 0.007 kg ai/hl with a PHI of 56 days. A total of 14 residue trials with foliar spray application on plums were performed in Europe with 1 x 0.007 kg ai/hl; 10 in France, 2 in Germany and 2 in UK. The total residues were <0.05 (14) mg/kg in whole fruits after a 21- or 56-day PHI.

Imidacloprid is registered in Italy in plums with one foliar spray treatment of 0.01 kg ai/hl and 21-day PHI. Ten trials were performed in France, Italy and Spain according to the Italian GAP spray concentration of 0.01 kg ai/hl. In each trial, 2 treatments (interval 30 days) were made at application

rates of 0.1 or 0.15 kg ai/ha (except for one trial in which the first application had a rate of 0.3 kg ai/ha, interval 144 days). The whole fruit residue concentrations were <0.05 (7), 0.05, 0.09, 0.12 mg/kg after a 21-day PHI.

The Meeting decided to combine the values. The ranked order of concentrations of residues was: <0.05 (21), 0.05, 0.09 and 0.12 mg/kg. The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in plums of 0.2, 0.05 and 0.12 mg/kg, respectively.

Grapes. Imidacloprid is registered for foliar spraying in grapes in Portugal (1 x 0.007 kg ai/hl, 0.07 kg ai/ha, PHI 14 days) and in the USA (1 – 2 x 0.04 – 0.052 kg ai/ha, 0-day PHI). Three residue trials were performed in Portugal and one each in Italy and Spain and complied approximately with the GAP (1 x 0.01 kg ai/hl). The residue concentrations were <0.05 (3), 0.12 and 0.2 mg/kg after a 14-day PHI.

A total of 16 residue trials were performed according to GAP in the USA in 1991/92. In each trial, 2 applications (interval 11-16 days) were made. All applications were carried out approximately at the highest label application rate (0.053 kg ai/ha). Based on a concentrated spray volume of 374-477 l/ha, the spray concentration ranged between 0.011 and 0.014 kg ai/hl. Based on a diluted spray volume of 935-1189 l/ha, the spray concentration ranged between 0.0045 and 0.0057 kg ai/hl. At the 0-day PHI, the concentrations of residues were: <0.05 , 0.05, 0.06, 0.06, 0.06, 0.11, 0.11, 0.11, 0.12, 0.12, 0.16, 0.17, 0.19, 0.2, 0.21 and 0.61 mg/kg.

The Meeting decided to combine the values from European and US trials. The ranked order of concentrations of residues was: <0.05 (4), 0.05, 0.06 (3), 0.11 (3), 0.12 (3), 0.16, 0.17, 0.19, 0.2, 0.2, 0.21, 0.61 mg/kg. The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in grapes of 1, 0.11 and 0.61 mg/kg, respectively.

Tropical fruits. Imidacloprid is registered for banana in Cameroon and Ivory Coast with application of 0.25 g ai/plant of the non-diluted product to the base of the pseudo-trunk and a 1-day PHI. A further use is bud flower (bell) injection with 0.012 kg ai/hl in the Philippines.

Four residue trials from Martinique with application of 0.25 g ai/plant to the base of pseudo-trunk and twelve trials with single basal drench application of 0.21 – 0.29 g ai/plant in Costa Rica, Ecuador, Guatemala and Honduras complied with GAP in Cameroon and in Ivory Coast. In the Martinique trials, the total residue was below the LOQ of 0.05 mg/kg in all samples (pulp, peel, whole fruit) and at all sampling dates. In the Central and South America trials, the total residues were below or at the LOQ of 0.01 mg/kg in all banana whole fruit samples (PHIs 0 – 35 days).

The residues in whole banana in rank order were: <0.01 (10), 0.01, 0.01, <0.05 (4) mg/kg. The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in banana of 0.05, 0.01 and 0.05 mg/kg, respectively.

Imidacloprid is registered for mango in the Philippines with 1 – 2 x 0.002 – 0.0025 kg ai/hl, 0.02 – 0.062 kg ai/ha, PHI 20 days and in the USA with 6 x 0.093 kg ai/ha., PHI 30 days. Four residue trials were conducted in the Philippines with 2 – 5 foliar spray applications at a spray concentration of 0.0025 kg ai/hl. The trials could not be used for evaluation because the PHIs were 30 – 92 instead of 20 days. Six further trials were performed in the USA. In each trial, six treatments were made. Three trials were performed with diluted sprays at a concentration of 0.004 kg ai/hl and the remaining 3 were made with concentrated sprays at a concentration of 0.16 kg ai/hl. The application rates ranged from 0.072 to 0.097 kg ai/ha. With a PHI of 30 days, the total residues in depitted fruits were <0.05 (3), 0.11, 0.15 mg/kg.

The Meeting noted that no data were received for whole mango fruits. Taking into account the stone weight of about 20% of the fruit, a maximum residue level of 0.2 mg/kg was estimated for mango.

The Meeting estimated an STMR and HR for imidacloprid in mango of 0.05 and 0.15 mg/kg, on the basis of the data for fruits without stone.

Bulb vegetables. Imidacloprid is an authorised minor use for dressing of leek seed in Germany with an application rate of 45 g ai/unit (250 000 seeds) and a maximum rate of 0.09 kg ai/ha. Four trials were performed in northern European countries with a seed dressing rate of 60g ai/unit, which corresponds to 0.06 to 0.072 kg ai/ha. The total residues in shoots were <0.05 mg/kg (4) with PHIs of 158 – 190 days.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in leek of 0.05*, 0.05 and 0.05 mg/kg.

Imidacloprid is an authorized minor use for dressing of onion seed in Germany with an application rate of 45 g ai/unit (250 000 seeds) and a maximum rate of 0.18 kg ai/ha. Further use is foliar spray in Brazil and Thailand, but no adequate residue data were submitted. In northern Europe a total of eight residue trials were performed on onions with a seed treatment rate of 45 g ai/unit according to German GAP. The total residues in bulb were <0.05 (7), 0.06 mg/kg at PHIs of 179 – 199 days.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in onions of 0.1, 0.05 and 0.06 mg/kg, respectively.

Brassica vegetables, head cabbage, flowerhead brassicas. The residue trials for broccoli, cauliflower, Brussels sprouts and head cabbage were evaluated together for mutual support.

Imidacloprid is registered world-wide in broccoli for foliar spray, drench or soil application. Spanish GAP allows 2 foliar sprays with 0.1 kg ai/ha and a 14-day PHI. Four residue field trials performed in Italy (3 trials) and Spain (1 trial) on broccoli complied with Spanish GAP. The residues were 0.08, 0.1, 0.29, 0.31 mg/kg. Australian broccoli GAP allows foliar spray at 0.06 kg ai/ha and a 7-day PHI. The concentration of residues in broccoli in one trial that complied with GAP (4 x 0.05 – 0.06 kg ai/ha) was 0.19 mg/kg. The current USA labels allow soil application with 0.18 – 0.42 kg ai/ha with a 21-day PHI and 1 – 5 foliar spray applications of 0.053 kg ai/ha with a 7-day PHI. Twelve field studies were conducted using three applications of imidacloprid. The first was a soil drench application, localised at the base of the plants, fourteen days after transplanting, at a rate of 0.01 g ai/plant (0.56 kg ai/ha). The remaining applications were two foliar spray applications at a rate of 0.12 kg ai/ha. These overdosed trials could not be used for evaluation.

In South Africa drench application over seedlings prior to transplanting with 0.1 – 0.2 kg ai/ha and a 76-day PHI is registered. One trial complied with GAP and did not show residues in curds higher than the LOQ of 0.05 mg/kg at 76 days after treatment.

The combined residues from broccoli trials according to GAP were <0.05, 0.08, 0.1, 0.19, 0.29, 0.31 mg/kg.

Imidacloprid is registered world-wide in cauliflower for foliar spray, drench or soil application. Spanish GAP allows 2 foliar sprays with 0.1 kg ai/ha and a 14-day PHI. Five residue field trials performed in Italy complied with Spanish GAP. The residues were 0.06, 0.07, 0.08, 0.09 and 0.11 mg/kg.

Australian GAP allows foliar spray at 0.06 kg ai/ha and a 7-day PHI. The concentration of residues in cauliflower in one trial that complied with GAP (2 x 0.06 kg ai/ha) was 0.01 mg/kg. The current USA labels allow soil application with 0.18 – 0.42 kg ai/ha with a 21-day PHI and 1 – 5 foliar spray applications of 0.052 kg ai/ha with a 7-day PHI. Twelve cauliflower field studies were conducted using three applications of imidacloprid. The first application was a soil drench application,

localized at the base of the plants. Fourteen days after transplanting, a rate of 0.01 g ai/plant was applied (0.56 kg ai/ha). The remaining applications were two foliar spray applications at rates of 0.12 kg ai/ha. These overdosed trials could not be used for evaluation.

In South Africa drench application over seedlings prior to transplanting with 0.1 – 0.2 kg ai/ha and a 136-day PHI is registered. One trial complied with GAP and residues in curds were below than the LOQ of 0.05 mg/kg at 136 days after treatment.

The combined residues from trials according to GAP in cauliflower were 0.01, <0.05, 0.06, 0.07, 0.08, 0.09 and 0.11 mg/kg.

Australian GAP for Brussels sprouts allows foliar spray at 0.06 kg ai/ha and a 7-day PHI. The concentration of residues in two trials that complied with GAP (2 – 3 x 0.06 kg ai/ha) was 0.03 and 0.32 mg/kg.

In South Africa drench application over Brussels sprouts seedlings prior to transplanting with 0.1 – 0.2 kg ai/ha and a 91-day PHI is registered. One trial complied with GAP, another was overdosed with residues below the LOQ of 0.05 mg/kg at 91 or 136 days after treatment.

The concentration of residues in Brussels sprouts were in rank order: 0.03, <0.05, <0.05, 0.32 mg/kg.

Australian GAP for head cabbage allows foliar spray at 0.06 kg ai/ha and a 7-day PHI. The concentration of residues in heads in two trials that complied with GAP (3 - 5 x 0.06 kg ai/ha) were 0.02 mg/kg and 0.22 mg/kg. The value of 0.22 mg/kg was not included in evaluation because in this trial 'heart and wrapper leaves' was analyzed.

The current USA labels allow soil application in head cabbage with 0.18 – 0.42 kg ai/ha with a 21-day PHI and 1 – 5 foliar spray applications of 0.053 kg ai/ha with a 7-day PHI. Thirteen field studies were conducted using three applications of imidacloprid. The first application was a soil drench application, localized at the base of the plants. Fourteen days after transplanting, a rate of 0.01 g ai/plant was applied (0.56 kg ai/ha). The remaining applications were two foliar spray applications at a rate of 0.12 kg ai/ha. These overdosed trials could not be used for evaluation.

Thirty bridging studies to compare the residues from the various types of soil application patterns were carried out in the USA with 0.19 - 0.6 kg ai/ha in broccoli, cauliflower and head cabbage. Treatments were made as soil drench, in-furrow or sidedress applications at the time of planting, or 14 days after planting at the latest. On the one hand, some trials treated with application rates of 0.19 or 0.27 kg ai/ha did not match the maximum GAP of 0.42 kg ai/ha, on the other hand the trials applied with 0.56 and 0.6 kg ai/ha exceeded the maximum GAP for 33 – 42% and were outside of the tolerance. Only one trial on cauliflower treated with 0.51 kg ai/ha approximately matched the GAP and showed residues of 0.21 mg/kg at a 38-day PHI. As the Meeting was informed that the waiting period of 21 days ('do not apply a soil application within 21 days of harvest'), prescribed in the US label of the 240 SC formulation for cabbages and flowerhead brassicas is not a normal residue-related PHI, the result was used for evaluation.

The Meeting noted that the data on broccoli, cauliflower, Brussels sprouts and head cabbage (without wrapper leaves) were similar and could be combined for mutual support. The combined residues were, in rank order: 0.01, 0.02, 0.03, <0.05 (4), 0.06, 0.07, 0.08, 0.08, 0.09, 0.1, 0.11, 0.19, 0.21, 0.29, 0.31, 0.32 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in broccoli, cauliflower, Brussels sprouts and head cabbages of 0.5, 0.08 and 0.32 mg/kg, respectively.

Cucurbits. Imidacloprid is registered in cucumber in Europe (indoor Denmark, Netherlands; in- and outdoor Spain, Greece) as foliar spray, treatment with the irrigation water or treatment in nutrition solution in rock wool.

A total of six indoor residue trials were conducted in cucumber with drip irrigation application in the Netherlands. The plants were grown on rock wool. In two of the trials, 2.5 mg imidacloprid was applied in 10 ml water to the base of each plant. This amount is equal to a rate of 0.024 - 0.034 kg ai/ha, which is in accordance with the lowest rate, registered in The Netherlands. The plants in the four further trials received 10 mg imidacloprid per plant, which corresponds to an application rate of about 0.12 - 0.15 kg ai/ha. The use rate of 10 mg ai/plant is in accordance with the maximum label rate in The Netherlands, Denmark, Greece, and Spain. The registered PHI is 1 day but the highest residues were found after about 5 – 7 days. The residues were in rank order: 0.25, 0.31, 0.39 and 0.39 mg/kg.

Ten further indoor trials were performed in cucumber in southern France (8 trials) and Spain (2 trials) with drip irrigation application. Each plant received 25 mg ai/plant. This rate represented 2.5 times the recommended label use rate in Greece and Spain and could not be used for evaluation.

In Spain, imidacloprid is registered in cucumber with 1 – 2 foliar spray treatments of 0.1 kg ai/ha, 0.01 kg ai/hl and a 3-day PHI in glasshouse or in the field. In Italy one indoor trial in cucumber was conducted with foliar spray application of 0.15 kg ai/ha (0.015 kg ai/hl) and was not in accordance with the Spanish GAP. In Spain three residue outdoor field trials were performed according to GAP with application rates of 0.1 kg ai/ha (2 treatments, interval 15 days, 0.01 kg ai/hl) but samples were not taken at the registered PHI of 3 days.

The current Australian label indicates imidacloprid may be applied as foliar spray with a rate of 0.05 kg ai/ha in the field to cucumber. The concentration of residues in cucumbers in one trial that complied with GAP (4 x 0.06 kg ai/ha) was 0.04 mg/kg.

The residues from trials according to maximum GAP from the Netherlands and Australia were in rank order: 0.04, 0.25, 0.31, 0.39, 0.39 mg/kg. The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in cucumber of 1, 0.31 and 0.39 mg/kg, respectively.

The use patterns of imidacloprid for summer squash and cucumber in the Netherlands and in Spain are identical. In Italy two summer squash trials were conducted with foliar spray application of 2 x 0.15 kg ai/ha, which is not in accordance with the Spanish GAP.

The Meeting agreed to extrapolate the data on residues in cucumber to summer squash and estimated a maximum residue level, an STMR value and an HR value for imidacloprid in summer squash of 1, 0.31 and 0.39 mg/kg, respectively.

Imidacloprid is registered in melons in Spain and Portugal with 1 – 2 foliar spray treatments of 0.1 kg ai/ha, 0.01 kg ai/hl and 3-day PHI. A total of ten residue field trials were performed in southern Europe (Italy and Spain) according to Spanish GAP. The residue concentrations in whole fruit were in rank order: <0.05 (4), 0.05, 0.06, 0.07, 0.08, 0.13, 0.15 mg/kg and in pulp <0.05 (6), 0.05, 0.11 mg/kg. In Spain, application of 0.1 – 0.15 kg ai/ha in the irrigation water is registered. Two indoor residue trials were conducted in melon using drip irrigation application of 0.1 kg ai/ha and did not match the maximum GAP.

The current Australian label indicates foliar spray treatments with 0.06 kg ai/ha and a 1-day PHI. Two trials were conducted with four foliar spray applications each (interval 7-17 days) of 0.06 kg ai/ha and showed residues of 0.03 and 0.07 mg/kg in whole fruits.

In South Africa one residue field trial in melons was performed according to GAP using drench application of 0.02 g ai/plant at planting. The residue was <0.01 mg/kg in whole fruits 100 days after planting.

The residues from trials according to GAP from Italy, Spain, Australia and South Africa were <0.01, 0.03, <0.05 (4), 0.05, 0.06, 0.07, 0.07, 0.08, 0.13, 0.15 mg/kg in whole fruit. The Meeting estimated a maximum residue level of 0.2 mg/kg for imidacloprid in melons.

The residues were <0.05 (6), 0.05, 0.11 mg/kg in the edible portion. The Meeting estimated an STMR and an HR for imidacloprid in melons of 0.05 and 0.11 mg/kg.

Imidacloprid is registered in watermelons in Spain with 1 – 2 foliar spray treatments of 0.1 kg ai/ha, 0.01 kg ai/hl and 3-day PHI. A total of ten residue field trials were conducted in southern Europe (Greece, Italy and Spain) with 2 applications (interval 7 – 20 days) of 0.1 kg ai/ha according to Spanish GAP. The residues were <0.05 (6), 0.05, 0.07, 0.09, 0.1 mg/kg in whole fruit. The Meeting estimated a maximum residue level of 0.2 mg/kg for imidacloprid in watermelons.

The residues were <0.05 (7), 0.05, 0.06 mg/kg in the edible portion. The Meeting estimated an STMR value and an HR value for imidacloprid in watermelons of 0.05 and 0.06 mg/kg.

Fruiting vegetables, other than cucurbits. Imidacloprid is registered for indoor and outdoor use with foliar spray treatment in egg plants in Italy (1 x 0.1 - 0.15 kg ai/ha, 0.01 - 0.015 kg ai/hl, 7-day PHI) and in Spain (1 – 2 x 0.1 kg ai/ha, 0.01 kg ai/hl, 3-day PHI). The residue concentrations from trials according to GAP were <0.05 (6), 0.06, 0.06, 0.08, 0.14 mg/kg. Four further trials from Italy and two from Brazil did not match the GAP.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in egg plant of 0.2, 0.05 and 0.14 mg/kg, respectively.

Imidacloprid is registered in peppers world-wide as foliar spray, treatment with the irrigation water or treatment in nutrition solution on rock wool.

Imidacloprid is registered for indoor and outdoor use with foliar spray treatment in peppers in Italy (1 x 0.1 - 0.15 kg ai/ha, 0.01 - 0.015 kg ai/hl, 7-day PHI) and in Spain (1 – 2 x 0.1 kg ai/ha, 0.01 kg ai/hl, 3-day PHI). The southern European trials (13 indoor, 6 outdoor) treated with 0.15 kg ai/ha complied with Italian GAP (PHI 7 days) and those with 0.1 kg ai/ha with Spanish GAP (PHI 3 days). The residue concentrations from trials according to GAP were <0.05, 0.07, 0.07, 0.09, 0.1, 0.1, 0.11, 0.11, 0.12, 0.15, 0.15, 0.15, 0.17, 0.21, 0.22, 0.24, 0.26, 0.27 and 0.48 mg/kg.

In Australia three indoor pepper residue trials were performed with application rates at the recommended label use rate of 0.05 kg ai/ha as well as at the double and four fold rates of 0.1 kg ai/ha and 0.2 kg ai/ha. Eight applications were made in each trial (interval 14-16 days). Because only imidacloprid was analysed in the pepper fruits, the data could not be used for evaluation. Two further foliar spray trials from Brazil did not match the GAP.

A total of four residue trials were conducted in sweet pepper simulating drip irrigation application in greenhouses in the Netherlands. The pepper crop was grown on rock wool. 10 mg imidacloprid was applied in 10 ml water at the base of each plant. This quantity corresponds to an application rate of 0.2 - 0.32 kg ai/ha, which is in accordance with GAP (9.8 g/1000 plants). The residue concentrations were 0.16, 0.17, 0.24 and 0.27 mg/kg.

In two pepper greenhouse residue trials (Italy, Portugal) a rate of 0.2 kg ai/ha imidacloprid was applied with the irrigation water to the soil. The trials were in accordance with Danish GAP. Residues below the LOQ were found at all sampling dates (3 – 60 days). The residues were <0.05 (2) mg/kg.

The current USA labels allow soil application with 0.28 – 0.56 kg ai/ha with a 21-day PHI and 5 foliar spray applications of 0.053 kg ai/ha with a 0-day PHI. Nine pepper field studies were conducted with three applications of imidacloprid. The first application was a soil drench application, localised at the base of the plants. Fourteen days after transplanting, a rate of 0.025 g ai/plant was applied (0.41 – 0.67 kg ai/ha). The remaining applications were two foliar spray applications at rates of 0.12 kg ai/ha. These overdosed trials could not be used for evaluation.

The remaining sixteen US pepper residue trials were bridging studies to compare the residues from the various types of soil applications and formulations. Treatments were made at the time of planting, or two weeks after planting at the latest. Only two trials for sweet pepper and one for hot pepper with soil drench application of 0.41-0.49 kg ai/ha matched the GAP resulting in concentrations of residues of <0.05, 0.06 and 0.24 mg/kg at PHIs of 54 – 60 days. As the Meeting was informed that the waiting period of 21 days ('do not apply a soil application within 21 days of harvest'), prescribed in the US label of the 240 SC formulation for fruiting vegetables is not a normal residue related PHI, the results were used for evaluation..

The Meeting considered that the data from indoor and outdoor trials as well as from the different treatments are from the same pool and combined them, resulting in a ranked order as follows: <0.05 (4), 0.06, 0.07, 0.07, 0.09, 0.1, 0.1, 0.11, 0.11, 0.12, 0.15 (3), 0.16, 0.17, 0.17, 0.21, 0.22, 0.24 (3), 0.26, 0.27, 0.27 and 0.48 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in peppers of 1, 0.15 and 0.48 mg/kg, respectively.

Imidacloprid is registered in tomatoes world-wide as foliar spray, application with the irrigation water or treatment in nutrition solution in rock wool.

Imidacloprid is registered for indoor and outdoor use with foliar spray treatment in tomatoes in Italy (200 SL: 1 x 0.1 - 0.15 kg ai/ha, 0.01 - 0.015 kg ai/hl, 7-day PHI; 100 EC: 0.09 kg ai/ha, 0.011 kg ai/hl, 3-day PHI greenhouse, 7-day PHI field) and in Spain/Portugal (1 – 2 x 0.1 kg ai/ha, 0.01 kg ai/hl, 3-day PHI). The southern European trials (9 indoor, 9 outdoor) treated with 0.015 kg ai/ha complied with Italian GAP (PHI 7 or 3 days) and those with 0.1 kg ai/ha with Spanish GAP (PHI 3 days). The residue concentrations from trials according to GAP were <0.05 (6), 0.05, 0.06, 0.06, 0.07 (3), 0.08 (3), 0.09, 0.09, 0.1, 0.1, 0.12, 0.13, 0.14, 0.17, 0.18 and 0.29 mg/kg. Two further foliar spray trials from Brazil did not match the GAP.

A total of six residue trials were conducted in tomatoes simulating drip irrigation application in the greenhouse in the Netherlands. The crop was grown on rock wool. 10 mg imidacloprid was applied in 10 ml water at the base of each plant,. This quantity corresponds to an application rate of 0.23 - 0.29 kg ai/ha, which is in accordance with GAP (9.8 g/1000 plants). The residue concentrations were 0.05, 0.08, 0.09, 0.14, 0.15 and 0.16 mg/kg.

In two greenhouse residue trials (Italy, Portugal) a rate of 0.2 kg ai/ha imidacloprid was applied with the irrigation water to the soil. The trials were in accordance to Danish GAP. Residues were below the LOQ at all sampling dates (3 – 60 days). The residues were <0.05 (2) mg/kg.

The current USA labels for tomato allow soil application with 0.28 – 0.42 kg ai/ha with a 21-day PHI and 5 foliar spray applications of 0.05 kg ai/ha with a 0-day PHI. Eleven field studies (9 in the USA, 2 in Canada) were conducted utilising three applications of imidacloprid. The first application was a soil drench application, localized at the base of the plants. Fourteen days after transplanting, a rate of 0.025 g ai/plant was applied (0.5 – 0.56 kg ai/ha). The remaining applications were two foliar spray applications at rates of 0.12 kg ai/ha. These overdosed trials could not be used for evaluation.

The remaining US tomato residue trials were bridging studies to compare the residues from the various types of soil applications and formulations. Treatments were made at the time of planting, or two weeks after planting at the latest. As the application rate of 0.56 kg ai/ha exceeded the maximum GAP rate of 0.42 mg/kg for more than 30%, the trials were not used for evaluation.

The Meeting considered that the data from indoor and outdoor trials as well as from the different treatments are from the same pool and combined them, resulting in the following ranked order of concentrations of 33 residue values: <0.05 (8), 0.05, 0.05, 0.06, 0.06, 0.07 (3), 0.08 (4), 0.09 (3), 0.1, 0.1, 0.12, 0.13, 0.14, 0.14, 0.15, 0.16, 0.17, 0.18 and 0.29 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in tomatoes of 0.5, 0.08 and 0.29 mg/kg, respectively.

The Australian label indicates imidacloprid may be applied as seed treatment in sweet corn with 0.26 kg ai/100 kg seed. Three trials each were carried out with 0.26 or 0.35 kg ai/100 kg seed and two trials with 0.52 kg ai/100 kg seed. In all samples, the residues in cobs were lower than the LOQs: <0.01 (6), <0.02 (2) mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in sweet corn (corn-on-the-cob) of 0.02*, 0.01 and 0.02 mg/kg, respectively.

Leafy vegetables. Imidacloprid is registered in Spain for use in lettuce as foliar spray treatment (1 – 2 x 0.1 kg ai/ha, 0.01 kg ai/hl, 3-day PHI). A total of seven field residue trials on head lettuce were performed according to Spanish GAP in southern Europe (1 Greece, 2 Italy, 4 Spain) in 1989 - 2001. Residues in rank order were: 0.69, 0.87, 0.88, 0.9, 0.98, 0.99, 1.2 mg/kg. One further trial on leaf lettuce was carried out in Spain according to GAP and showed residues of 1.5 mg/kg.

Another Spanish lettuce use pattern is application in irrigation water with 0.01 g ai/plant. Twenty four indoor and outdoor residue trials in France and Germany carried out with drench application of 0.0024 g ai/plant and one trial from Italy with 0.3 kg ai/ha did not match Spanish GAP and could not be used for evaluation.

The current USA labels allow soil application in lettuce with 0.18 – 0.42 kg ai/ha with a 21-day PHI and 5 foliar spray applications of 0.05 kg ai/ha with a 7-day PHI. Fourteen field studies on head lettuce and twelve on leaf lettuce were conducted utilizing three applications of imidacloprid. The first application was a soil drench application, localized at the base of the plants. Fourteen days after transplanting, a rate of 0.01 g ai/plant was applied (0.56 kg ai/ha). The remaining applications were two foliar spray applications at rates of 0.12 kg ai/ha. These overdosed trials could not be used for evaluation.

The remaining US lettuce residue trials (10 leaf lettuce, 7 head lettuce) were bridging studies to compare the residues from the various types of soil applications. Treatments on head and leaf lettuce were made at the time of planting, or two weeks after planting at the latest. As the application rate of 0.56 kg ai/ha exceeded the maximum GAP rate of 0.42 kg ai/ha for more than 30%, the trials were not used for evaluation.

Based on the southern European head lettuce residue data, the Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in head lettuce of 2, 0.9 and 1.2 mg/kg, respectively.

Legume vegetables. Imidacloprid is registered in Spain for use in green beans with foliar spray treatment (1 – 2 x 0.1 kg ai/ha, 0.01 kg ai/hl, 3-day PHI). A total of 11 field residue trials were performed in 1991 – 1996 in Europe (2 France, 3 Italy, 6 Spain) according to Spanish GAP. Residues in beans with pods were, in rank order: 0.16, 0.24, 0.32, 0.33, 0.38, 0.39, 0.41, 0.44, 0.55, 0.61 and 0.66 mg/kg.

Four Brazilian trials were carried out with 5 x 0.18 or 5 x 0.35 kg ai/ha by foliar spraying. One of them complied with Brazilian GAP (0.18 kg ai/ha, PHI 21 days) and showed in beans without pods a residue of 0.01 mg/kg at a PHI of 21 days.

The current USA labels allow soil application with 0.28 – 0.42 kg ai/ha with a 21-day PHI and 3 foliar spray applications of 0.049 kg ai/ha with a 7-day PHI. Trials with different treatment scenarios were made in the USA.

Five field studies in common bean were conducted using five applications of imidacloprid. The first application was seed treatment with 0.25 kg ai/100 kg seed, one in-furrow spray application at planting with 0.42 kg ai/ha and three foliar spray applications of about 0.05 kg ai/ha. At a 7-day PHI, the residues were in beans with pods: 0.23, 0.38, 0.52, 0.61, 0.88 mg/kg.

Five field studies in lima bean were conducted using four applications of imidacloprid. One in-furrow spray application at planting with 0.42 kg ai/ha followed by three foliar spray applications of about 0.05 kg ai/ha. At a 7-day PHI, residues in beans without pods were: <0.05, <0.05, 0.12, 0.17, 0.25 mg/kg.

The combined residues for beans with pods in rank order were: 0.16, 0.23, 0.24, 0.32, 0.33, 0.38, 0.38, 0.39, 0.41, 0.44, 0.52, 0.55, 0.61, 0.61, 0.66 and 0.88 mg/kg. The combined residues for beans without pods in rank order were: 0.01, <0.05, <0.05, 0.12, 0.17 and 0.25 mg/kg. The Meeting considered the two data sets to be from different populations and agreed to use those for beans with pods (higher values) for making estimates.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in beans, except broad bean and soya bean, of 2, 0.4 and 0.88 mg/kg, respectively.

Pulses. Three residue trials with dry beans were conducted in Brazil, none of them complied with the Brazilian GAP for seed treatment with 0.14 kg ai/100 kg seed. The Meeting considered that the data were inadequate to allow assessment of residues of imidacloprid in dry beans.

Potatoes. Imidacloprid is registered world-wide in potatoes as seed treatment, soil treatment at planting and foliar spraying.

Eight residue trials were carried out in Germany with two procedures: direct spray on potatoes and in-furrow spray during planting or in-furrow spray on seed potatoes and soil band spray. The application rate was 12 g ai/100 kg seed potatoes according to the Netherlands' GAP (soil treatment in-furrow at planting with 11 g ai/100 kg seed). The residues were <0.05 mg/kg (8).

Eight further trials were performed in France (2 trials), Greece (1), Germany (2), Italy (1), Spain (1) and UK (1) with seed treatment of 7.2 g ai/100 kg according to German GAP. The residues were <0.05 (7) and 0.05 mg/kg.

Seven trials were performed in France (2 trials), Germany (1), Italy (2) and Spain (2) with seed treatment of 14 g ai/100 kg according to Spanish GAP. The residues were <0.05 (3), 0.09, 0.1, 0.12 and 0.12 mg/kg.

Three trials were performed in Italy at seed treatment of 25 g ai/100 kg seed according to maximum Italian GAP. The residues were 0.06, 0.15 and 0.2 mg/kg.

Foliar spray treatment is registered in Europe with 1 - 2 x 0.1 kg ai/ha in Greece, 1 x 0.1 – 0.15 kg ai/ha in Spain or 1 – 2 x 0.072 – 0.15 kg ai/ha in Italy/Portugal. The PHI is 14 days in Greece and Italy, 21 days in Portugal and 30 days in Spain. Fifteen trials were performed with foliar spraying

of 2 x 0.09 - 0.1 kg ai/ha in Italy (13) and Spain (2). Residues in samples taken after 7, 14 or 21 days were <0.05 mg/kg (15).

In Canada imidacloprid is registered for use in soil and for foliar application on potatoes. The use rates for soil application are 0.2 - 0.31 kg ai/ha, and for spray application 0.048 kg ai/ha. In the USA imidacloprid is also registered for use in soil at rates of 0.02 - 0.03 g ai/m, corresponding to between 0.28 and 0.35 kg ai/ha, and as a foliar spray with an application rate of about 0.05 kg ai/ha and a PHI of 7 days. Regardless of formulation or type of application (soil or foliar) it is not allowed to apply more than a total of 0.56 kg ai/ha per season.

In Canada three trials were conducted with in-furrow application (0.03 g ai/m row) at planting, followed by four spray applications at rates of 0.053 kg ai/ha. The residues were <0.1, <0.1 and 0.12 mg/kg.

Three residue field trials were performed in the USA with in-furrow application of 0.33 – 0.34 kg ai/ha only. The residues were 0.02, 0.07 and 0.18 mg/kg.

A total of nineteen residue field trials were performed in the USA with both in-furrow application and foliar spray application. A rate of 0.03 g ai/m row was applied as an in-furrow spray, which corresponds to 0.29-0.4 kg ai/ha. Four foliar sprays at rates of 0.053 kg ai/ha followed. The residues were <0.05 (12), 0.05, 0.05, 0.05, 0.07, 0.13, 0.16 and 0.28 mg/kg.

In South Africa, use of imidacloprid on potatoes is registered for soil treatment with application rates of 1.1 to 1.6 g ai/100 m row, corresponding to 0.14 - 0.21 kg ai/ha. Three trials were received with in-furrow application of 0.1, 0.2 and 0.3 kg ai/ha. The residues in the two trials according to GAP were <0.04 and 0.04 mg/kg.

In South Korea, imidacloprid is registered for soil application with 0.06 kg ai/ha and a 30-day PHI. Three residue trials were performed in South Korea with 1 to 4 applications of 0.06 kg ai/ha and incorporation into the soil. Only the parent compound imidacloprid was determined. The trials could not be used for evaluation.

In total, the following three data sets according to GAP were available (i) in-furrow treatment and in-furrow treatment followed by foliar spraying: 0.02, <0.04, 0.04, <0.05 (20), 0.05, 0.05, 0.05, 0.07, 0.07, <0.1, <0.1, 0.12, 0.13, 0.16, 0.18, 0.28 mg/kg, (ii) seed treatment <0.05 (10), 0.05, 0.06, 0.09, 0.1, 0.12, 0.12, 0.15, 0.2 mg/kg, and (iii) foliar spray only <0.05 mg/kg (15).

Because a residues were below the LOQ in tubers after foliar spraying, the Meeting noted that these data are a different population and agreed to combine only the data sets for seed dressing and in-furrow treatment/in-furrow treatment followed by foliar spray for making estimations. The combined 53 residue concentrations were in rank order: 0.02, <0.04, 0.04, <0.05 (30), 0.05 (4), 0.06, 0.07, 0.07, 0.09, <0.1, <0.1, 0.1, 0.12 (3), 0.13, 0.15, 0.16, 0.18, 0.2, 0.28 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in potatoes of 0.5, 0.05 and 0.28 mg/kg, respectively.

Sugar beets. Imidacloprid is registered in European countries in sugar beet for seed treatment with 0.09 kg ai/unit (100 000 seeds). 20 residue trials were performed in France (1), Germany (8), Italy (5), Sweden (2) and UK (4) with application rates of 0.09 – 0.11 kg ai/unit. Residues in sugar beet at harvest were <0.05 mg/kg (20).

Further use is foliar spray with 1 – 2 x 0.072 kg ai/ha and a 30-day PHI in Italy. In Italy a total of 8 residue field trials were performed in sugar beet with 2 spray applications of 0.09 kg ai/ha. With the PHI of 30 days, residues in sugar beet were <0.05 mg/kg (8).

Based on the combined residues of <0.05 mg/kg (28), the Meeting estimated a maximum residue level and an STMR value for imidacloprid in sugar beet of 0.05* and 0.05 mg/kg.

Celery. The current US labels for celery allow soil application with 0.18 – 0.42 kg ai/ha with a 45-day PHI. 12 trials with different treatment scenarios were made in the USA: Six residue field trials were conducted with plant drench application. Application rates of 0.54 kg ai/ha (1 trial) and 0.56 to 0.59 kg ai/ha (5 trials) were applied, 43 - 46 days prior to harvest. The remaining six other residue trials were bridging studies to compare the residues from the various types of soil applications. As the application rate of 0.56 – 0.6 kg ai/ha exceeded the maximum GAP rate of 0.42 mg/kg for more than 30%, the trials were not used for evaluation.

The Meeting concluded that there were insufficient data to estimate a maximum residue level for celery.

Cereal grains. Imidacloprid is registered for seed treatment in barley, oat, rye, triticale and wheat with 0.35 kg ai/100 kg seed in Germany, 0.07 kg ai/100 kg seed in Belgium or France and with 0.07 – 0.14 kg ai/100 kg seed in Australia.

Barley. In one German and one UK trial the seed treatment was performed with 0.035 and 0.11 kg ai/100 kg seed. In 13 residue field trials conducted in different European countries and Australia imidacloprid was applied as seed treatment with 0.07 kg ai/100 kg seed. In 3 Australian trials the seed treatment was performed with 0.14 kg ai/100 kg seed. Residues in barley grains were: <0.02 , <0.02 , <0.05 (15) mg/kg.

Oat. In one German, two Swedish and two Australian trials seed treatment was performed with 0.035, 0.11 and 0.07 kg ai/100 kg seed. Residues in oat grains were: <0.02 , <0.02 , <0.05 (3) mg/kg.

Triticale. In two Australian trials seed treatment was performed with 0.07 or 0.14 kg ai/100 kg seed. Residues in triticale grain were: <0.05 (2) mg/kg.

Wheat. In eight Australian, two Brazilian, six German, four French and three UK trials, seed treatment was performed with 0.035, 0.05, 0.07, 0.1, 0.11, 0.14 kg ai/100 kg seed. Residues in wheat grains were: 0.04, <0.05 (21), 0.05 mg/kg. All residue values of barley, oat, triticale and wheat were in rank order: <0.02 (4), 0.04, <0.05 (41), 0.05 mg/kg.

Imidacloprid is registered for seed treatment in maize with 0.35 kg ai/100 kg seed in South Africa, with 54 g/unit = 0.47 kg ai/100 kg seed in Germany and 0.7 kg ai/100 kg seed in Italy. In four German trials the seed treatment was performed with 0.47 kg ai/100 kg seed. In 10 residue field trials conducted in different European countries imidacloprid was applied as seed treatment with 0.7 kg ai/100 kg seed. In one South African trial the seed treatment was performed with 0.35 kg ai/100 kg seed. The residues were in maize grains <0.02 and <0.05 (14) mg/kg.

Imidacloprid is registered for seed treatment in rice in Brazil and Japan and/or for foliar spray in Japan, South Korea and Thailand. The use pattern allows foliar spray treatments in Thailand 1 – 2 x 0.038 kg ai/ha, in South Korea 1-3 x 0.03 kg ai/ha and in Japan 3 x 0.03 – 0.075 kg ai/ha. Six residue trials were received from Thailand, four of them were treated with 2 x 0.015 – 0.024 kg ai/ha and could not be used for evaluation. Two further trials treated with 2 x 0.05 kg ai/ha complied approximately with Thailand's GAP. At PHIs of 48 or 56 days, no residues higher than the LOQ of 0.05 mg/kg were analysed. Four trials received from South Korea (3 – 6 x 0.064 kg ai/ha) could not be used for evaluation because only parent compound imidacloprid was determined.

The Meeting concluded to combine the seed treatment residue data on barley, oats, maize, triticale, rice and wheat which were in rank order <0.02 (5), 0.04, <0.05 (57) and 0.05 mg/kg.

The Meeting estimated a maximum residue level, an STMR and an HR for imidacloprid in cereal grains each of 0.05 mg/kg.

Tree nuts. Imidacloprid is registered in pecan in USA for foliar spray treatment with 2 x 0.2 kg ai/ha and soil application with maximum 0.56 kg ai/ha (no PHI). Sixteen trials with foliar treatment and seven with soil treatment according to US GAP were received. Residues in nuts without shell were at each sampling date: <0.01 (9), 0.011 and <0.05 (13) mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in pecan of 0.05 mg/kg.

Oilseed. The use of imidacloprid in cotton is authorized as seed dressing, foliar spray and soil application before or at planting. Seed treatment trials were conducted in Greece, Brazil, Egypt and Australia.

One residue trial each was performed in Greece and Brazil at a rate of 0.7 kg ai/100 kg seed according to Spanish GAP and one further trial according to Brazilian GAP (0.35 kg ai/100 kg seed). Residues in cotton seed were <0.05 (3) mg/kg. Two trials from Egypt complied with GAP (0.49 kg ai/100 kg seed) and showed residues of 0.06, 0.09 mg/kg. The ten Australian trials could not be used for evaluation because only parent compound imidacloprid was determined. Altogether, the residue data in cotton seed from seed treatment use were <0.05 (3), 0.06 and 0.09 mg/kg.

Two foliar spray trials were conducted in Spain on cotton (0.1 + 0.15 kg ai/ha) resulting in residues of 0.49, 0.95 mg/kg in seed but were overdosed in comparison with Greek GAP (2 x 0.1 kg ai/ha). Also one South African trial (0.08 kg ai/ha) and 26 US trials with different treatment scenarios were not made according to the respective GAP.

The Meeting concluded that five seed treatment trials only were insufficient to estimate a maximum residue level or STMR value for imidacloprid in cotton seed.

Imidacloprid is registered for rape seed treatment in Australia, Germany and the UK with 0.2 – 0.24 kg ai/100 kg seed. Four residue trials were conducted in Sweden (1.4 kg ai/100 kg seed), 9 in France, 4 in Germany and 2 in UK (1.05 kg ai/100 kg seed) which were 4-to-5fold overdosed. The residues from these trials in rape seed were <0.05 (19) mg/kg. Two trials from Australia were carried out with 0.25 and 0.5 kg ai/100 kg seed. Residues in rape seed were <0.05 (2) mg/kg. Altogether, the data set is <0.05 mg/kg (21).

The Meeting estimated a maximum residue level, an STMR value and an HR value for imidacloprid in rape seed of 0.05*, 0.05 and 0.05 mg/kg.

Coffee beans. The current Brazilian label allows drench treatment with 0.7 – 0.91 kg ai/ha and a 45-day PHI. Three trials according to Brazilian GAP were submitted. In one of them only parent imidacloprid was determined. With a 45-day PHI, the residue data were: <0.05 (2) mg/kg for total residues and <0.02 mg/kg for imidacloprid.

The Meeting concluded that three trials were insufficient to estimate a maximum residue level or STMR value for imidacloprid in coffee beans.

Hops. Imidacloprid is registered in Europe (Austria, Germany, Spain, UK) and the USA in hops for foliar spray, stem painting or spray directed at stem base. Eight German foliar spray trials according to German GAP (1 x 0.13 kg ai/ha, 0.004 kg ai/hl, 35-day PHI) showed residues in kiln-dried cones of 0.48, 0.59, 0.73, 0.73, 0.81, 1.2, 1.3, 1.6 mg/kg. Brush application was carried out in four German trials and complied with German GAP (1 x 0.14 kg ai/ha, 2.3 kg ai/hl, PHI 35 days). The residues were in kiln-dried cones 0.43, 0.52, 0.75, 0.83 mg/kg. All residue data in rank order were 0.43, 0.48, 0.52, 0.59, 0.73, 0.73, 0.75, 0.81, 0.83, 1.2, 1.3 and 1.6 mg/kg.

Three US trials complied with US GAP (foliar spray 3 x 0.11 kg ai/ha, PHI 28 days). The residues were 1.3, 5.5 and 5.8 mg/kg in dried cones.

Eight UK trials complied with UK GAP (foliar spray 1 x 0.13 kg ai/ha, 0.03 – 0.055 kg ai/hl, PHI 103 – 120 days). The residues were <0.2 (5), 0.25, 0.29, 0.7 mg/kg in kiln-dried cones.

All data in rank order were <0.2 (5), 0.25, 0.29, 0.43, 0.48, 0.52, 0.59, 0.7, 0.73, 0.73, 0.75, 0.81, 0.83, 1.2, 1.3, 1.3, 1.6, 5.5 and 5.8 mg/kg.

The Meeting estimated a maximum residue level and an STMR value for imidacloprid in dried hops of 10 and 0.7 mg/kg.

Tea. The use of imidacloprid in tea is registered in Japan as a foliar spray with 0.01 kg ai/hl, 0.1 - 0.4 kg ai/ha and a 14-days PHI. Two residue trials each were performed in Japan in 1990 and 1998 according to GAP with 0.01 kg ai/hl, 0.2 kg ai/ha. With a 14-day PHI, residues of imidacloprid *per se* in dried leaves were 1.8, 1.9, 2.3, 3 mg/kg. Because only parent compound was analysed, these trials could not be used for evaluation.

Sugar beet leaves and tops. Imidacloprid is registered in Europe in sugar beets for seed treatment with 0.09 kg ai/unit (100 000 seeds). Altogether, 20 residue trials were performed in sugar beet in France (1), Germany (8), Italy (5), Sweden (2) and UK (4) with application rates of 90 -110 g ai/unit. Residues in sugar beet leaves at harvest (PHI >140 days) were <0.05 (9), 0.05, 0.06 (3), 0.07 (3), 0.09, 0.11, 0.11 and 0.14 mg/kg.

Further use is foliar spray with 1 – 2 x 0.072 kg ai/ha and a 30-day PHI in Italy. In Italy a total of 8 residue field trials were performed in sugar beet after 2 spray applications of 0.09 kg ai/ha. With a PHI of 30 days, residues in sugar beet leaves were 0.23, 0.31, 0.33, 0.4, 0.45, 0.47, 0.61 and 0.67 mg/kg.

The Meeting considered the two data sets to be from different populations and agreed to use those from foliar spray treatment (higher values) for making estimations. Allowing for the standard 23 % dry matter (*FAO Manual*), the Meeting estimated a maximum residue level and an STMR value (dry weight) for imidacloprid in sugar beet leaves and tops of 5 mg/kg and 1.8 mg/kg.

Cereals, forage and fodder. Imidacloprid is registered for seed treatment in barley, oat, rye, triticale and wheat with 0.35 kg ai/100 kg seed in Germany, 0.07 kg ai/100 kg seed in Belgium or France and with 0.07 – 0.14 kg ai/100 kg seed in Australia. The residues from trials according to GAP were as follows:

Barley. With PHIs from 50 - 79 days, residues in forage were: <0.02, 0.03, 0.03, 0.05, 0.06, 0.07, 0.09, 0.12, 0.12, 0.13, 0.15, 0.19, 0.24, 0.52, 0.67 mg/kg on fresh weight basis. The residues in straw were at harvest: <0.05 (6), 0.05, 0.05, 0.06, 0.09, 0.09, 0.11, 0.11, 0.12, 0.16, 0.28, 0.32 mg/kg.

Oat. With a 63-day PHI, residues in oat forage were: <0.02, 0.06, 0.09 mg/kg (fresh weight). The residues in straw were at harvest: <0.02, <0.02, <0.05, 0.05, 0.08 mg/kg (fresh weight). Triticale. With a 50 - 63-day PHI, residues in triticale forage were: 0.04, <0.05 mg/kg (fresh weight). The residues in straw were at harvest: <0.05, <0.05 mg/kg (fresh weight).

Wheat. With PHIs from 62 to 77 days, residues in wheat forage were: 0.02, 0.03, <0.05, 0.05, 0.07, 0.09, 0.1, 0.1, 0.1, 0.11, 0.12, 0.19, 0.19, 0.27, 0.39 mg/kg on fresh weight basis. The residues in straw were at harvest: <0.05 (6), 0.05 (3), 0.06, 0.06, 0.08, 0.09, 0.09, 0.11, 0.11, 0.13, 0.21, 0.23, 0.24, 0.45 mg/kg (fresh weight).

All residue values in barley, oats, triticale and wheat forages were in rank order: <0.02, <0.02, 0.02, 0.03 (3), 0.04, <0.05, <0.05, 0.05, 0.05, 0.06, 0.06, 0.07, 0.07, 0.09 (3), 0.1 (3), 0.11, 0.12 (3), 0.13, 0.15, 0.19 (3), 0.24, 0.27, 0.39, 0.52, 0.67 mg/kg on fresh weight basis. Allowing for the standard 28 % dry matter (average of wheat, rye and oat forage, *FAO Manual*), the Meeting estimated the following residue levels for cereal forage commodities listed as animal feed item:

A maximum residue level and an STMR of 5 mg/kg and 0.32 mg/kg for rye and oat forage. An highest residue level and an STMR of 2.4 mg/kg and 0.32 mg/kg for triticale and wheat forage.

All residue values in straw of barley, oats, triticale and wheat were in rank order: <0.02, <0.02, <0.05 (15), 0.05 (6), 0.06 (3), 0.08, 0.08, 0.09 (4), 0.11 (4), 0.12, 0.13, 0.16, 0.21, 0.23, 0.24, 0.28, 0.32, 0.45 mg/kg (fresh weight).

Allowing for the standard 89 % dry matter (average of barley, wheat, rye and oat straw, *FAO Manual*), the Meeting estimated a maximum residue level and an STMR value for imidacloprid in straw and fodder (dry) of barley, oats, rye, triticale and wheat of 1 mg/kg and 0.056 mg/kg.

Imidacloprid is registered for seed treatment in maize with 0.35 kg ai/100 kg seed in South Africa, with 54 g/unit = 0.47 kg ai/100 kg seed in Germany and 0.7 kg ai/100 kg seed in Italy. In four German trials the seed treatment was performed with 0.47 kg ai/100 kg seed. In 10 residue field trials conducted in different European countries imidacloprid was applied as seed treatment with 0.7 kg ai/100 kg seed. In one South African trial the seed treatment was performed with 0.35 kg ai/100 kg seed. At the ripening stage of maize for silage [BBCH code 85: Dough stage] the residues were in maize forage <0.02, <0.05 (8), 0.05, 0.06, 0.1 mg/kg on a fresh weight basis. The residues were in maize straw <0.02, <0.05 (2), 0.1 mg/kg.

Allowing for the standard 83 % dry matter for maize stover (*FAO Manual*, p. 147), the Meeting estimated a maximum residue level and an STMR value for imidacloprid in maize fodder of 0.2 and 0.06 mg/kg.

Allowing for the standard 40% dry matter, the Meeting estimated a maximum residue level and an STMR value (dry weight) for imidacloprid in maize forage of 0.5 mg/kg and 0.125 mg/kg.

Fate of residues during storage and processing

One hydrolysis study to determine the effects of processing on the nature of residues shows that imidacloprid was stable after simulated pasteurisation, baking/boiling and sterilisation. Considering the hydrolytic stability under the conditions tested, it is not expected that hydrolysis will contribute to the degradation of imidacloprid or affect the nature of imidacloprid residues during processing.

The effect of processing on the concentrations of residues of imidacloprid has been studied in oranges, lemon, apples, cherries, grapes, tomatoes, lettuce, green beans, potatoes, rice, wheat, cotton seed, hops and tea. The processing factors calculated from total residues were used for estimation of STMR-P and HR-P values.

Citrus fruits (RAC residues in oranges 0.12, 0.2, 0.19 mg/kg, in lemon 0.26 mg/kg) were processed into marmalade, juice and dried pulp with processing factors of 0.625 (mean of 0.5, 0.75), 0.28 (mean of 0.19, 0.25, 0.26, 0.42) and 7.47, respectively. Based on the STMR value of 0.05 mg/kg for citrus fruits, the STMR-Ps were 0.03 mg/kg for marmalade and 0.014 mg/kg for citrus juice. A maximum residue level of 10 mg/kg and an STMR of 0.374 mg/kg is estimated for citrus dried pulp.

Apples (RAC residues 0.06, 0.11, 0.13, 0.16, 0.23 mg/kg) were processed into juice, sauce, pomace wet, pomace dry, and dried fruit, with processing factors of 0.656 (mean of 0.4, 0.45, 0.77, 0.83, 0.83), 0.75 (mean of 0.6, 0.73, 0.83, 0.83), 1.6, 5.2 (mean of 3.7, 5.7, 6.3) and 0.865 (mean of 0.83, 0.9), respectively. Based on the STMR value of 0.07 mg/kg for apples, the STMR-P for apple

juice was 0.046 mg/kg, 0.053 mg/kg for sauce, 0.11 mg/kg for wet apple pomace, and 0.061 mg/kg for dried apple fruit. A maximum residue level of 5 mg/kg and an STMR of 0.364 mg/kg is estimated for apple pomace, dry.

Cherries, sweet (RAC residues 0.08, 0.08, 0.09, 0.09 mg/kg) were processed into preserve (canned fruits) with a processing factor of <0.6 (mean of <0.56, <0.56, <0.63, <0.63). Based on the STMR value of 0.14 mg/kg for sweet cherries, the STMR-P was 0.084 mg/kg for canned sweet cherries.

Peaches (RAC residue 0.13 mg/kg) were processed into preserve (canned fruits) and jam with processing factors of <0.38 each. Based on the STMR value of 0.12 mg/kg for peaches, nectarines and apricots, the STMR-P was 0.046 mg/kg for canned fruits and jam of peaches, nectarines and apricots.

Grapes (RAC residues 0.05, 0.06, 0.06, 0.07, 0.1, 0.1, 0.2 mg/kg) were processed into wine, juice and raisins with processing factors of 1.17 (mean of 0.86, 1.2, 1.3, 1.33), 0.73 (mean of <0.5, <0.5, 1.2) and 1.05 (mean of 1.0, 1.1), respectively. Based on the STMR value of 0.11 mg/kg for grapes, the STMR-P for wine was 0.13 mg/kg, 0.08 for juice and 0.12 mg/kg for raisins (dried grapes).

Tomatoes (RAC residues 0.05, 0.11, 0.16, 0.44 mg/kg) were processed into paste, puree, ketchup, preserve (canned fruits) and juice with processing factors of 5.73 (mean of 3.4, 5.1, 8.7), 2.3 (mean of 1.89, 2.7), 2.0, 0.91 and 1.37 (mean of 1, 1.3, 1.8) respectively. Based on the STMR value of 0.08 mg/kg for tomato, the STMR-Ps were 0.458 mg/kg for tomato paste, 0.184 mg/kg for puree, 0.16 mg/kg for ketchup, 0.073 mg/kg for canned fruits and 0.11 mg/kg for juice.

Beans, green with pods, (RAC residues 0.29, 0.32 mg/kg) were processed into cooked beans with pods and preserves (canned fruits) with processing factors of 0.975 (mean of 0.81, 1.14) and 0.43 (mean of 0.375, 0.48). Based on the STMR value of 0.4 mg/kg for beans, except broad bean and soya bean, the STMR-Ps were 0.39 and 0.17 mg/kg for cooked beans with pods and their canned fruits.

Potatoes (RAC residue 0.26 mg/kg) were processed into wet peel, chips and granules with processing factors of 0.65, 1.35 and 0.92, respectively. Based on the STMR value of 0.05 mg/kg for potatoes, the STMR-Ps were 0.033 mg/kg for potato wet peel, 0.068 mg/kg for potato chips and 0.046 mg/kg for potato granules.

Rice (RAC residues <0.05 mg/kg) were processed into polished rice, bran and glume. No detectable residues were reported in the processed commodities (<0.05 mg/kg) with one exception of glume (0.08 mg/kg). As the concentration of total residues was at the LOQ in the RAC, no STMR-P values could be estimated.

Wheat (RAC residue 0.02 mg/kg) was processed into milled by-products (bran) and flour with processing factors of 3.5 and 0.5. Based on the STMR value of 0.05 mg/kg for wheat grain, the STMR-Ps were 0.175 mg/kg for wheat milled by-products (bran) and 0.025 for wheat flour. The Meeting recommended a maximum residue level of 0.3 for wheat bran and 0.03 for wheat flour.

Cotton seed (RAC residues 0.54, 0.66, 2.7, 2.9 mg/kg) were processed into hulls, meal, crude oil and refined oil. The processing factors were 0.38, 1.45, <0.09 (mean of <0.019, <0.076, <0.093, <0.17) and <0.09 (mean of <0.019, <0.17) for hulls, meal, crude oil and refined oil. STMR-P values could not be recommended because no maximum residue limit or STMR was estimated for cotton seed.

Hops (RAC residues in kiln-dried cones 5.8, 6.4 mg/kg) were processed into beer with a processing factor of 0.0035 (mean of 0.002, 0.005). Based on the STMR value of 0.7 mg/kg for hops, dry, the STMR-P was 0.0025 mg/kg for beer.

Tea leaf samples were twisted and dried in a tea-making machine. The infusion was prepared by extracting the dried tea leaves with hot water. Only the parent compound imidacloprid was analyzed in dried leaves and the infusion. Therefore, no maximum residue limit, STMR or STMR-P values could be estimated.

Residues in animal commodities

Dietary burden in animals

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

Estimated maximum dietary burden of farm animals.

Commodity	Codex Commodity Group	Residue mg/kg	Basis	% dry matter	Residue dry wt (mg/kg)	Choose diets, %			Residue contribution (mg/kg)		
						Beef cattle	Dairy cattle	poultry	beef cattle	dairy cattle	poultry
Apple pomace, wet	AB	0.11	STMR-P	40	0.275						
Barley grain	GC	0.05	MRL	88	0.057						
Barley straw	AS	1	MRL	100	1						
Citrus pulp, dried	AB	0.374	STMR-P	91	0.41	20	20		0.082	0.082	
Maize grain	GC	0.05	MRL	88	0.057			50			0.0285
Maize forage	AF	0.5	MRL	100	0.5						
Maize stover	AS	0.2	MRL	100	0.2						
Oats grain	GC	0.05	MRL	89	0.056						
Oats forage	AF	5	MRL	100	5	25	60		1.25	3	
Oats straw	AS	1	MRL	100	1						
Potato wet peel	AB	0.033	STMR-P	15	0.22						
Rye grain	GC	0.05	MRL	88	0.057						
Rye forage	AF	2.4	highest residue	100	2.4						
Rye straw	AS	1	MRL	100	1						
Sugar beet leaves and tops	AM	5	highest residue	100	5	20	10		1.0	0.5	
Wheat grain	GC	0.05	MRL	89	0.056						
Wheat forage	AF	2.4	highest residue	100	2.4						
Wheat straw	AS	1	MRL	100	1						
Wheat milled by-products	CF	0.175	STMR-P	88	0.199			50	0.07	0.0199	0.0995
TOTAL						100	100	100	2.402	3.6019	0.128

Estimated STMR dietary burden of farm animals.

Commodity	Codex Commodity Group	Residue mg/kg	Basis	% Dry matter	Residue dry wt (mg/kg)	Choose diets, %			Residue contribution (mg/kg)		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Apple pomace, wet	AB	0.11	STMR-P	40	0.275						
Barley grain	GC	0.05	STMR	88	0.057						

Commodity	Codex Commodity Group	Residue mg/kg	Basis	% Dry matter	Residue dry wt (mg/kg)	Choose diets, %			Residue contribution (mg/kg)		
						Beef cattle	Dairy cattle	Poultry	Beef cattle	Dairy cattle	Poultry
Barley straw	AS	0.056	STMR	100	0.056						
Citrus pulp, dried	AB	0.374	STMR-P	91	0.41	20	20		0.082	0.082	
Maize grain	GC	0.05	STMR	88	0.057			50			0.0285
Maize forage	AF	0.125	STMR	100	0.125						
Maize stover	AS	0.06	STMR	100	0.06						
Oats grain	GC	0.05	STMR	89	0.056						
Oats forage	AF	0.32	STMR	100	0.32	25	60		0.08	0.192	
Oats straw	AS	0.056	STMR	100	0.056						
Potato wet peel	AB	0.033	STMR-P	15	0.22						
Rye grain	GC	0.05	STMR	88	0.057						
Rye forage	AF	0.32	STMR	100	0.32						
Rye straw	AS	0.056	STMR	100	0.056						
Sugar beet leaves and tops	AM	1.8	STMR	100	1.8	20	10		0.36	0.18	
Wheat grain	GC	0.05	STMR	89	0.056						
Wheat forage	AF	0.32	STMR	100	0.32						
Wheat straw	AS	0.056	STMR	100	0.056						
Wheat milled by-products	CF	0.175	STMR-P	88	0.199	35	10	50	0.07	0.0199	0.0995
TOTAL						100	100	100	0.592	0.4739	0.128

The dietary burdens of imidacloprid for estimating MRLs, STMR and HR values for animal commodities (residue concentrations in animal feeds expressed as dry weight) are: 2.4 and 0.59 mg/kg for beef cattle, 3.6 and 0.47 mg/kg for dairy cattle and 0.13 mg/kg each for poultry.

Feeding studies

The Meeting received information on the concentrations of residues arising in tissues and milk in dairy cows dosed with imidacloprid in capsules at the equivalent of 5, 15 or 50 ppm in the diet for 28 days. The mean transfer factors (concentration of residue ÷ concentration in feed) for cattle tissues and milk were consistent at the three dietary levels:

liver 0.05/5, 0.13/15, 0.49/50 = 0.01, 0.009, 0.0098 → 0.01
kidney 0.03/5, 0.09/15, 0.29/50 = 0.006, 0.006, 0.0058 → 0.006
muscle <0.02/5, 0.03/15, 0.12/50 = <0.004, 0.002, 0.0024 → 0.002 (doses 15 and 50 ppm)
fat <0.02/5, <0.02/15, 0.06/50 = <0.004, <0.0013, 0.0012 → 0.0012 (dose 50 ppm)
milk <0.02/5, 0.041/15, 0.15/50 = <0.004, 0.0027, 0.003 → 0.0029 (dose 15 and 50 ppm)

No residues higher than the LOQ of 0.02 mg/kg were found in milk, muscle or fat from cows at the 5 ppm dose level. The highest concentrations in the three animals at 5 ppm in the diet were 0.054 mg/kg in liver and 0.032 mg/kg in kidney. The mean concentrations in the three animals at 5 ppm were 0.05 mg/kg in liver and 0.03 mg/kg in kidney.

In the 15 ppm group, the milk residue reached a plateau directly after the first administration but did not accumulate. With this dose level, the average plateau concentration in milk (day 1) was 0.041 mg/kg. The mean concentrations in the three animals with 15 ppm were 0.03 mg/kg in muscle, <0.02 mg/kg in fat, 0.13 mg/kg in liver and 0.09 mg/kg in kidney. The highest individual concentrations with 15 ppm in the diet were 0.054 mg/kg in milk, 0.033 mg/kg in muscle, <0.02 mg/kg in fat, 0.17 mg/kg in liver, 0.1 mg/kg in kidney.

The Meeting received information on the concentrations of residues in tissues and eggs of laying hens dosed with imidacloprid at the equivalent of 2, 6 or 20 ppm in the diet for 30 days. The mean transfer factors for hen tissues and eggs were consistent at the three dietary levels:

liver 0.04/2, 0.14/6, 0.35/20 = 0.02, 0.023, 0.0175 → 0.02

muscle <0.02/2, 0.02/6, 0.048/20 = <0.01, 0.003, 0.0024 → 0.0027 (doses 6 and 20 ppm)
fat <0.02/2, <0.02/6, <0.02/20 = <0.01, <0.003, <0.001 → 0.001 (dose 20 ppm)
eggs <0.02/2, 0.049/6, 0.13/20 = <0.01, 0.008, 0.0065 → 0.007 (dose 15 and 20 ppm)

No residues higher than the LOQ of 0.02 mg/kg were determined in eggs, muscle or fat from hens at 2 ppm. The highest and the mean concentrations in the three birds at 2 ppm in the diet were: 0.042 mg/kg and 0.04 mg/kg in liver.

In the 6 ppm group, the egg residues reached a plateau about 6 days after the first administration. In this group, the average plateau concentration in eggs was 0.042 mg/kg. The mean concentrations in the three animals in the 6 ppm dose group were 0.02 mg/kg in muscle, <0.02 mg/kg in fat, 0.14 mg/kg in liver. The highest individual concentrations at the dose of 6 ppm in the diet were 0.052 mg/kg in eggs, 0.021 mg/kg in muscle, <0.02 mg/kg in fat, 0.16 mg/kg in liver.

Maximum residue levels

The Meeting agreed that in the case of dairy cattle, extrapolation below the lowest feeding level (5 ppm) was appropriate as the transfer factors were reasonably consistent across the three dietary levels.

As the maximum dietary burdens of beef and dairy cattle (2.4 and 3.6 ppm) were lower than the lowest feeding level of 5 ppm, the highest residues in tissues and milk were therefore calculated by applying the transfer factors to the maximum dietary burdens (transfer factor • dietary burden in mg/kg feed).

As the maximum dietary burden of dairy cows exceeds that for beef cattle, the former (3.6 mg/kg) was used to estimate the maximum residue level in muscle, liver and kidney.

As the STMR dietary burdens of beef and dairy cattle (0.59 and 0.47 ppm) were lower than the lowest feeding level of 5 ppm, the resulting STMRs in tissues and milk were calculated by applying the transfer factors to the STMR dietary burdens.

Dietary burden (ppm) Feeding level [ppm]	Imidacloprid total residue, mg/kg								
	Milk	Muscle		Liver		Kidney		Fat	
	mean	highest	mean	highest	mean	highest	mean	highest	mean
MRL dairy/beef cattle (3.6) [5]	(0.01) <0.02	(0.007) <0.02		(0.036) 0.054		(0.022) 0.032		(0.004) <0.02	
STMR beef cattle (0.59) [5]			(0.0012) <0.02		(0.006) 0.05		(0.0035) 0.03		(0.0007) <0.02
STMR dairy cattle (0.47) [5]	(0.0014) <0.02								

The maximum concentrations of residues expected in tissues are 0.007 mg/kg in muscle, 0.036 mg/kg in liver, 0.022 mg/kg in kidney, 0.004 mg/kg in fat and 0.01 mg/kg in milk. The mean extrapolated concentrations are 0.0012 mg/kg in muscle, 0.006 mg/kg in liver, 0.0035 mg/kg in kidney, 0.0007 mg/kg in fat and 0.0014 mg/kg in milk.

The Meeting estimated maximum residue levels of 0.02* mg/kg for meat (mammalian) and milks. For edible offal (mammalian), the estimated maximum residue level is 0.05 mg/kg. The Meeting recommended that the HR values should be 0.007 mg/kg in meat (mammalian), 0.036 mg/kg in edible offal (mammalian) and 0.004 in fat (mammalian). The estimated STMR values are 0.001 for meat (mammalian), 0.006 mg/kg for edible offal (mammalian), 0 for fat (mammalian) and 0.0014 mg/kg for milks.

The Meeting agreed that in the case of laying hens, extrapolation below the lowest concentration (2 ppm) was appropriate as the transfer factors were reasonably consistent across the three dietary levels. As the maximum and STMR dietary burden of 0.13 mg/kg each was lower than the lowest feeding level of 2 ppm, the resulting residues in tissues and eggs were calculated by applying the transfer factors to the maximum dietary burden (transfer factor • dietary burden in mg/kg).

Dietary burden (ppm) Feeding level [ppm]	Imidacloprid total residue, mg/kg							
	Eggs		Muscle		Liver		Fat	
	highest	mean	Highest	Mean	highest	mean	highest	mean
MRL (0.13) [2]	(0.0009) <0.02		(0.00035) <0.02		(0.0026) 0.042		(0.00013) <0.02	
STMR (0.13) [2]		(0.0009) <0.02		(0.00035) <0.02		(0.0026) 0.04		(0.00013) <0.02

The Meeting estimated maximum residue levels of 0.02* mg/kg for eggs, poultry meat and edible offal. The Meeting recommended that the HR values should be 0.001 mg/kg in eggs, 0.0004 mg/kg in poultry meat, 0.0026 mg/kg in edible offal and 0 in fat. The STMR values are 0.0026 mg/kg in edible offal of poultry, but 0 in poultry eggs, meat and fat.

RECOMMENDATIONS

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

Definition of the residue (for compliance with MRLs and for estimation of dietary intake):
sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid.

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg	HR, mg/kg
CCN	Name	New	Previous		
FP 0226	Apple	0.5		0.07	0.23
DF 0226	Apples, dried			0.061	
JF 0226	Apple juice			0.046	
AB 0226	Apple pomace, dry	5		0.364	
	Apple sauce			0.053	
FS 0240	Apricot	0.5		0.12	0.32
	Apricot jam			0.046	
	Apricot, canned			0.046	
FI 0327	Banana	0.05		0.01	0.05
AS 0640	Barley straw and fodder (dry) ¹	1		0.056	
VP 0061	Beans, except broad bean and soya bean	2		0.4	0.88
	Beans, except broad bean and soya bean, cooked			0.39	
	Beans, except broad bean and soya bean, canned			0.17	
	Beer			0.0025	
VB 0400	Broccoli	0.5		0.08	0.32
VB 0402	Brussels sprouts	0.5		0.08	0.32
VB 0041	Cabbages, head	0.5		0.08	0.32
VB 0404	Cauliflower	0.5		0.08	0.32
GC 0080	Cereals grains	0.05		0.05	0.05
FS 0244	Cherry, sweet	0.5		0.14	0.28
	Cherry, sweet, canned			0.084	
FC 0001	Citrus fruits	1		0.05	0.11
JF 0001	Citrus juice			0.07	
AB 0001	Citrus pulp, dry	10		1.94	
	Citrus marmalade (orange)			0.16	

Commodity		MRL, mg/kg		STMR or STMR-P, mg/kg	HR, mg/kg
CCN	Name	New	Previous		
VC 0424	Cucumber	1		0.31	0.39
DF 0269	Dried grapes			0.12	
MO 0105	Edible offal (Mammalian)	0.05		0.009	0.04
VO 0440	Egg plant	0.2		0.05	0.14
PE 0112	Eggs	0.02*		0	0.001
FB 0269	Grapes	1		0.11	0.61
JF 0269	Grape juice			0.08	
DH 1100	Hops, dry	10		0.7	
VA 0384	Leek	0.05*		0.05	0.05
VL 0482	Lettuce, Head	2		0.9	1.2
AS 0645	Maize fodder ¹	0.2		0.06	
AF 0645	Maize forage ¹	0.5		0.125	
FI 0345	Mango	0.2		0.05	0.15
MM 0095	Meat (from mammals other than marine mammals)	0.02*		0.002 (muscle) 0 (fat)	0.008 (muscle) 0.005 (fat)
VC 0046	Melons, except watermelon	0.2		0.05	0.11
ML0106	Milks	0.02*		0.0024	
FS 0245	Nectarine	0.5		0.12	0.32
	Nectarine jam			0.046	
	Nectarine, canned			0.046	
AF 0647	Oat forage (green) ¹	5		0.32	
AS 0647	Oat straw and fodder, dry ¹	1		0.056	
VA 0385	Onion, Bulb	0.1		0.05	0.06
FS 0247	Peach	0.5		0.12	0.32
	Peach jam			0.046	
	Peach, canned			0.046	
FP 0230	Pear	1		0.38	0.71
TN 0672	Pecan	0.05		0.05	0.05
VO 0051	Peppers	1		0.15	0.48
FS 0014	Plums (including prunes)	0.2		0.05	0.12
PM 0110	Poultry meat	0.02*		0 (muscle) 0 (fat)	0.0004 (muscle) 0 (fat)
PO 0111	Poultry, Edible offal of	0.02*		0.0026	0.0026
VR 0589	Potato	0.5		0.05	0.28
	Potato chips			0.068	
	Potato granules			0.046	
SO 0495	Rape seed	0.05*		0.05	0.05
AF 0650	Rye forage (green) ¹	5		0.32	
AS 0650	Rye straw and fodder, dry ¹	1		0.056	
VC 0431	Squash, Summer	1		0.31	0.39
VO 0447	Sweet corn (corn-on-the-cob)	0.02*		0.01	0.02
VR 0596	Sugar beet	0.05*		0.05	
AM 0596	Sugar beet leaves and tops ¹	5		1.8	
VO 0448	Tomato	0.5		0.08	0.29
	Tomato paste			0.458	
	Tomato puree			0.184	
JF 0448	Tomato juice			0.11	
	Tomato ketchup			0.16	
	Tomato, canned			0.073	
	Triticale forage ¹			0.32	2.4
	Triticale straw and fodder, dry ¹	1		0.056	
VC 0432	Watermelon	0.2		0.05	0.06
CM 0654	Wheat bran, unprocessed	0.3		0.175	
CF 1211	Wheat flour	0.03		0.025	
	Wheat forage ¹			0.32	2.4
AS 0654	Wheat straw and fodder, dry ¹	1		0.056	
	Wine			0.13	

¹Expressed on dry weight basis

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes of imidacloprid, based on the STMRs estimated for 47 commodities, for the five GEMS/Food regional diets were in the range of 0 to 2 % of the ADI (Annex 3). The Meeting concluded that the long-term intake of residues of imidacloprid resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The International Estimated Short term Intake (IESTI) of imidacloprid was calculated for 49 food commodities (and their processing fractions) for which MRLs, STMR values and/or HR values were established and for which data on consumption were available. The results are shown in Annex 4.

The IESTI represented 0 – 4 % of the acute RfD for the general population and 0 – 15 % of the acute RfD for children. The Meeting concluded that the short-term intake of residues of imidacloprid, resulting from its uses that have been considered by the JMPR, is unlikely to present a public health concern.

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- RA-2081/98, Report includes Trial Nos.: 1155-98 (811556), 1156-98 (811564). Spiegel, K. and Anderson, C. 1999.
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- RA-2083/00, Report includes Trial Nos.: R 2000 0584/9. Anderson, C. and Eberhardt, R. 2001.
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- RA-2094/98, Report includes Trial Nos.: 1163-98 (811637), 1164-98 (811645). Anderson, C. 1999.
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- RA-2104/97, Report includes Trial Nos.: 0786-97 (707864), 0469-97 (704695), 0468-97 (704687). Placke, F.J. 1998c.
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- RA-2111/93, Report includes Trial Nos.: 0415-93 (304158). Placke, F.J. 1994e.
- RA-2114/95, Report includes Trial Nos.: 0141-95 (501417), 0633-95 (506338). Blass, W. 1997a.
- RA-2114/99, Report includes Trial Nos.: R 1999 0514/9, R 1999 0515/7, R 1999 0516/5, R 1999 0517/3, R 1999 0523/8, R 1999 0524/6, R 1999 0525/4, R 1999 0526/2, R 1999 0527/0, R 1999 0528/9, R 1999 0529/7, R 1999 0530/0. Spiegel, K. and Elke, K. 2001.
- RA-2116/95, Report includes Trial Nos.: 0636-95 (506362), 0635-95 (506354), 0147-95 (501476). Blass, W. 1997d.
- RA-2117/95, Report includes Trial Nos.: 0638-95 (506389), 0637-95 (506370), 0148-95 (501484). Blass, W. 1997e.
- RA-2118/95, Report includes Trial Nos.: 0640-95 (506400), 0152-95 (501522). Blass, W. 1997b.

- RA-2119/95, Report includes Trial Nos.: 0642-95 (506427), 0641-95 (506419), 0140-95 (501409), 0139-95 (501395). Blass, W. 1997f.
- RA-2120/95, Report includes Trial Nos.: 0119-95 (501190), 0120-95 (501204), 0643-95 (506435), 0644-95 (506443), 0645-95 (506451), 0647-95 (506478), 0648-95 (506486), 0649-95 (506494). Blass, W. 1997g.
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- RA-2130/91. Bachlechner, G. 1993a.
- RA-2131/91. Placke, F.J. 1993e.
- RA-2131/95, Report includes Trial Nos.: 0658-95 (506583), 0657-95 (506575), 0656-95 (506567), 0202-95 (502022). Blass, W. and Heinemann, O. 1997.
- RA-2132/95, Report includes Trial Nos.: 0199-95 (501999), 0653-95 (506532), 0654-95 (506540), 0655-95 (506559). Blass, W. 1998e.
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- RA-2146/96, Report includes Trial Nos.: 0203-95 (502030), 0687-95 (506877). Heinemann, O. and Blass, W. 1997a.
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