Imazalil (110)

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EXPLANATION

Imazalil is an imidazole fungicide with a protective, curative and anti-sporulation mode of action.

Imazalil was first evaluated by the JMPR in 1977. The current ADI, established in 1991, is 0–0.03 mg/kg bw. In 2005, the JMPR set an ARfD of 0.05 mg/kg bw.

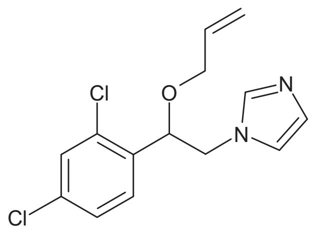
Imazalil was scheduled at the Forty-ninth Session of the CCPR for Periodic Review for residues and toxicology by the 2018 JMPR.

The Meeting received information from the manufacturer on physical and chemical properties, metabolism studies on plants and animals, environmental fate in soil, analytical method and stability in stored analytical samples, use patterns and supervised residue trials, processing studies, and livestock feeding studies.

Imazalil is a racemate, consisting of equal amounts of two enantiomers.

IDENTITY

ISO common Name:	Imazalil
IUPAC Name:	(*RS*)-1-(β-allyloxy-2,4-dichlorophenethyl)imidazole
Chemical Abstracts Name:	1-[2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl]-1*H*-imidazole
CAS No.:	35554-44-0
CIPAC No.:	335 (free base), 335.306 (imazalil sulphate)
Synonyms:	Imazalil (ANSI), chloramisol (Republic of South Africa), enilconazole (BAN, INN)
Molecular Formula:	$C_{14}H_{14}Cl_2N_2O$
Structural Formula:	
Molecular Weight:	297.18 g/mol



Specifications

Specifications for imazalil were developed by the FAO in 2001.

Physical and chemical properties

Pure active (98.1-99.9% purity)

Parameters	Value		Reference
Appearance	Yellow to brown crystalline mass		Wo, 2005
Vapour pressure (25 °C)	1.58×10^{-4} Pa		Rordorf, 1988
Melting point	48.6 °C		Smeykal, 2009
Partition coefficient n-octanol / water	Log K_{OW}: 2.15 (pH 5), 3.53 (pH 9)		Franke, 2008
Solubility in water (20 °C)	0.184 g/L at pH 7.6		Crauwels & Jacobs, 1988
Solubility in organic solvents (20 °C)	Solvent	Solubility (g/L)	
	n-hexane	19	Crauwels & Jacobs, 1988

Parameters	Value		Reference
	methanol, ethanol, propan-2-ol, dichloromethane, acetone, ethyl acetate, diethyl ether, toluene, N,N-dimethyl formamide, tetrahydrofuran, 4-methyl-pentan-2-one, propylene glycol, polyethylene glycol 400, DMSO, acetonitrile, 1,1,1-trichlorethane, butan-2-one	>500	
	n-heptane	54.1	Garnier, 1983
	hexane	52.1	
	petroleum ether	61.6	
	methanol, ethanol, propan-2-ol, butanol, xylene, toluene, benzene, 4-methyl-pentan-2-one, chloroform, dichloromethane, ethyl acetate, isoamyl alcohol	>500	
Density (26°C)	1.348 g/cm^3 at		Crauwels & Jacobs, 1989
Hydrolysis in water	No hydrolysis products were detected at pH 5, 7 and 9, at 25 °C after 61 days. Imazalil is stable to aqueous hydrolysis		Van Leemput, 1982
Photolysis	Imazalil undergoes photolysis in the aquatic environment (DT_{50}- 11-12 days)		Adam, 2008 and Mégel 2009
Dissociation constant	pK_a = 6.53 (>99.9%)		Crauwels & Jacobs, 1988
	pK_a = 6.49 (Imazalil, 98.0%) pK_a = 6.54 (Imazalil sulphate, 99.7%)		Bates, 2002

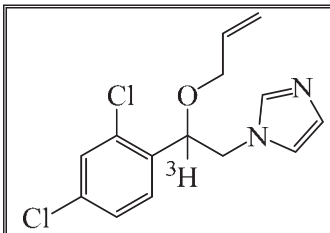
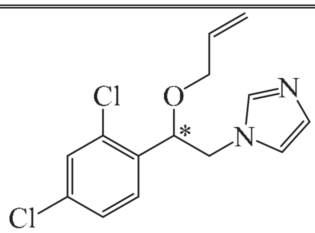
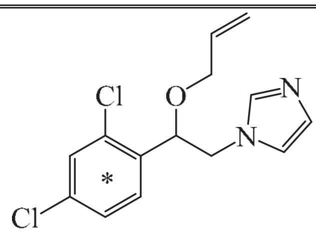
Formulations

Imazalil is available as emulsifiable concentrates, (EC), suspension concentrates (SC), wettable granules (WG), soluble granules (SG), soluble powders (SP) and liquids/solutions (LS/SL).

Formulations	Active ingredient content (g/L)
50 EC, 500EC	500 g/L
75 EC	750
100 EC	100
SC, 400 SC,	200
100 SC	100
75 SP, 750 SP	750
75 SG, 750 SG	750
750 WG	750
75 LS, 7.5 S, 7.5 LS	75
75 WSG	750
100 SL	100
50 ECNA	500
A25	25
Seed treatment	450

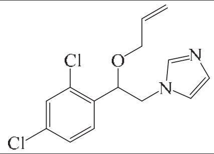
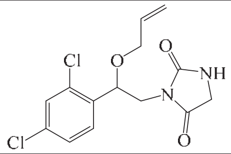
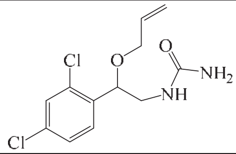
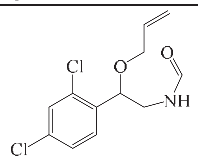
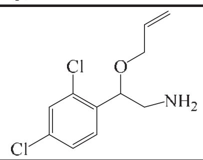
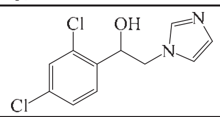
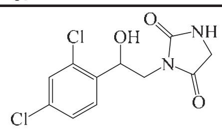
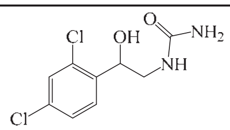
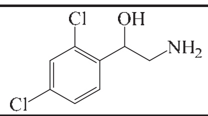
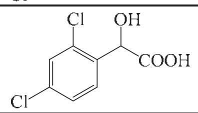
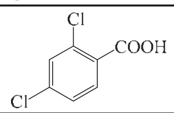
METABOLISM AND ENVIRONMENTAL FATE

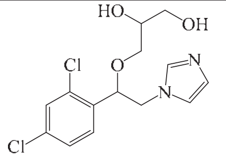
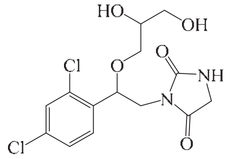
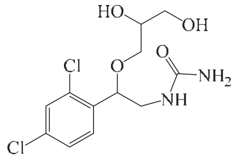
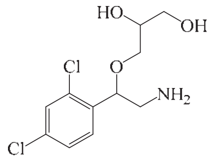
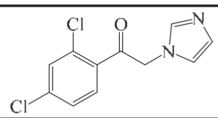
The fate and behaviour of imazalil in animals, plants, and the environment were investigated using [^{14}C] or [^{3}H] labelled test materials as shown below:

		
[^{3}H] Imazalil: tritium label placed on the stereogenic centre	[^{14}C] Imazalil: ^{14}C-label placed on the stereogenic centre	[^{14}C] Imazalil: ^{14}C-label placed in the phenyl ring

Chemical names, structures and code names of metabolites and degradation products of imazalil are summarized in Table 2. Compounds are referred to primarily by the code name.

Table 2 Code names, chemical names and structures of imazalil related substances

Code Name/ Number	Chemical Name	Chemical Structure	Occurrence in
Imazalil	(*RS*)-1-[2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl]-1*H*-imidazole		Ruminant, Poultry, Potato, Tomato, Wheat, Banana, Apple, Orange
R044179	(*RS*)-1-[2-(2,4-dichlorophenyl)-2-(2- propenyloxy)ethyl]-imidazolidine-2,5-dione		Ruminant, Tomato, Cucumber
R092977	(*RS*)-1-[2-(2,4-dichlorophenyl)-2-(2- propenyloxy)ethyl]-urea		Tomato
R055609	*N*-(2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl)formamide		Ruminant, Tomato
R044177	2-(2,4-dichlorophenyl)-2-(2-propenyloxy)-ethan-1-amine		Ruminant, Potato
R014821	(*RS*)-1-(2,4-dichlorophenyl)-2- imidazol-1-yl-ethanol		Rat, Ruminant, Potato, Tomato, Banana, Apple, Orange, Wheat
R043449	(*RS*)-3-[2-(2,4-dichlorophenyl)-2-hydroxyethyl]imidazolidine-2,4-dione		Ruminant Poultry, Tomato
R044085	(*RS*)-*N*-[2-(2,4-dichlorophenyl)-2-hydroxyethyl]urea (±)-*N*-[2-(2,4-dichlorophenyl)-2-hydroxyethyl]urea		Rat, Ruminant, Poultry
R110740	2-amino-1-(2,4-dichlorophenyl)ethan-1-ol		Poultry
R023366	2-(2,4-dichlorophenyl)-2-hydroxyacetic acid		Ruminant
R066996	2,4-dichlorobenzoic acid		Ruminant

Code Name/ Number	Chemical Name	Chemical Structure	Occurrence in
R042639	(*RS*)-1-[[2-(2,4-dichlorophenyl)-2-(2,3-dihydroxypropyl)oxy]ethyl]-1*H*-imidazole (±)-1-[[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-1*H*-imidazole		Rat, Ruminant, Poultry, Tomato, Wheat
R061000	(*RS*)-3-[2-(2,4-dichlorophenyl)-2-(2,3-dihydroxypropoxy)ethyl]imidazolidine- 2,4-dione (±)-1-[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-2,5-imidazolidinedione		Rat, Ruminant, Poultry
R062775	1-(2-(2,4-dichlorophenyl)-2-(2,3-dihydroxypropoxy)ethyl)urea		Ruminant, Poultry
R060998	3-(2-amino-1-(2,4-dichlorophenyl)ethoxy)propane-1,2-diol		Ruminant, Poultry
R045529	1-(2,4-dichlorophenyl)-2-(1*H*-imidazol-1-yl)ethan-1-one		Tomato

Plant metabolism

Imazalil metabolism data were submitted for orange, apple and banana (post-harvest treatment), cucumbers and tomatoes (greenhouse foliar application), seed potatoes, stored potatoes, spring wheat and barley (seed treatment).

Oranges – Post-harvest

Spanish navel oranges (variety *Valencia*) were dipped for 1 minute in an aqueous solution of [^{3}H]-imazalil sulphate, labelled on stereogenic carbon (specific activity: 12.7 µCi/mg), at a rate equivalent to 0.05 kg ai/hL (Meuldermans *et al*., 1979, Report no. R 23979/18 and R 23979/19). After treatment, oranges were dried for 2 hours and randomly separated into five batches of eight oranges per batch and stored on individual plastic holders in a dark room cooled at 12°C and at a relative humidity of about 90%. Samples were taken immediately (2 hours) after dipping, then after 1, 3, 6 and 12 weeks. For all samples, peel and pulp were analysed separately. Results for the whole fruits were calculated using the respective weights of peel and pulp.

The total radioactive residues (TRRs), determined by combustion analysis, remained relatively unchanged over the entire storage duration and were predominantly found in the peel.

Table 3 Distribution of radioactivity in oranges

Storage Duration (weeks)	mg eq/kg (% TRR)		
	Whole Fruit	Peel	Pulp
0 (2 hours)	2.05	5.96 (98.1)	0.11 (1.9)
1	2.59	7.18 (99.4)	0.04 (0.6)
3	1.90	5.82 (99.4)	0.04 (0.6)
6	2.16	6.29 (99.3)	0.04 (0.7)
12	2.08	6.42 (99.2)	0.05 (0.8)

Homogenates of Orange peel and pulp separately adjusted to pH 11 using 1N NaOH and 0.1 M phosphate buffer (pH 7.4) before adjusting to pH 11. The mixture was then extracted with heptane:isoamyl alcohol (95:5 v/v, 3×) followed by successive extractions with chloroform (3×) at pH 9 and 5 and ethyl acetate (3×) at pH 2. Table 4 highlights the distribution of the radioactivity in pulp and peel among the various extracts.

Table 4 Distribution of the peel and pulp radioactivity among the various extracts

	% Radioactivity									
Storage Duration (weeks)	0 (2 hours)		1		3		6		12	
	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp
TRRs (mg eq/kg)	5.96	0.11	7.18	0.04	5.82	0.04	6.29	0.04	6.42	0.05
Organic fraction	93.3	87.8	88.1	ND	86.0	74.2	78.3	75.2	76.4	70.6
pH 11 extract	78.3	84.9	70.5	ND	63.8	67.7	51.8	69.8	45.1	64.9
pH 9 extract	5.1	1.8	7.4	ND	9.5	3.7	12.3	3.5	12.8	3.6
pH 5 extract	9.2	0.2	8.6	ND	10.2	0.4	12.2	0.4	15.6	0.5
pH 2 extract	0.7	0.9	1.6	ND	2.5	2.3	2.1	1.5	2.9	1.5
Aqueous fraction	6.7	12.3	11.9	ND	14.0	25.8	21.7	24.8	23.6	29.4

ND: Not determined

Table 5 Identification/characterization of radioactivity, determined by GLC-assay, in peel and pulp from oranges stored 2 hours to 12 weeks following post-harvest treatment

Analyte	Radioactivity, mg eq/kg (% TRR)									
	0 (2 hours)		1		3		6		12	
	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp	Peel	Pulp
Imazalil	4.6 (78.4)	0.077 (67.7)	5.0 (69.9)	0.022 (45.0)	4.5 (79.0)	0.02 (45.1)	4.1 (66.7)	0.02 (49.2)	3.6 (57.3)	0.025 (44.8)
R014821	0.08 (1.38)	<0.01 (0.01)	0.10 (1.24)	<0.01 (0.01)	0.14 (2.36)	<0.01 (0.01)	0.25 (4.1)	0.011 (11.7)	0.29 (4.6)	0.01 (17.7)

Imazalil accounted for the majority of the radioactivity in the peel, decreasing from 78% TRR, 2 hours after treatment, to 57% TRR, 12 weeks thereafter. Radioactivity in pulp followed a similar trend, decreasing from 68% TRR, 2-hours post-treatment, to 45% TRR following a 12-week storage period. The concentrations of the metabolite R014821 in peel and pulp remained relatively unchanged following the first 3 weeks of storage but reached maximum levels following 12 weeks of storage.

Peel samples were also subjected to extraction with methanol:concentrated ammonia (9:1, v:v) and methanol: glacial acetic acid (9:1, v:v). The methanolic extract, after evaporation, was further extracted with heptane:isoamyl alcohol (95:5, v:v) resulting in heptane fraction and an aqueous fraction. The latter was subjected to the same extraction procedure as that of pulp. The peel post-extraction solids (PES) were dissolved in water and extracted with methanol:concentrated ammonia (9:1, v:v). The resulting residue was successively extracted with methanol (2×, 50ºC) and DMSO (2×, 80ºC). To the methanol extract, 1N NaOH was added prior to extraction with heptane:isoamyl alcohol (95:5, v:v). The distribution of the radioactivity in both the heptane and aqueous fractions is captured in Table 5.

The identity of imazalil and the metabolites was confirmed using either reverse phase radio-HPLC or GLC-assay.

Table 6 Recovery of radioactivity (% TRR) in the fractions obtained following extraction of peel

Storage Duration (weeks)	0 (2 hours)	1	3	6	12
Organic fraction	91.1	90.0	86.6	85.1	76.2
Aqueous fraction	5.2	6.9	8.3	9.2	15.3
Residue	3.7	3.1	9.1	5.7	8.5

Apple – Post-harvest

Apples (*variety* Golden Delicious) were dipped in solutions of unlabelled imazalil-sulfate corresponding to 0.05 kg ai/hL determining the residue level of the parent compound and its metabolites, equivalent to a magnitude of the residue study. (Van Leemput *et al*., 1985, R23979/L9). Fruits were subsequently stored in controlled atmosphere (temperature: 1 - 1.5 °C; relative humidity: 90 - 95%) and sampled immediately (4 hours) after drying and after 1, 2, 4, 6 and 7 months of storage. Another set of apples was treated with a blend of [^{14}C]-imazalil labelled on the 2-ethyl carbon atom (specific activity: 10.13 µCi/mg) and unlabelled imazalil (ratio 1:9) to investigate the degradation of imazalil and to determine the extractability of the radioactivity as a function of storage time. One [^{14}C]-imazalil (specific activity:10.13 µCi/mg) apple was treated with the same radiolabelled imazalil test substance for the volatilization test. The treated apple was incubated in a flow-through system similar to that of soil metabolism studies and was initiated on the same day as the storage samples and continued until the end of the storage period (7 months).

TRRs in apples treated with radiolabelled material ranged from 2.43–3.20 mg eq/kg which were higher than those treated at the same rate with unlabelled imazalil, where residues ranged from 1.5–2.0 mg/kg. Considering the variability in residue levels between the individual fruits from a sample set, a steady decline in residues, as a function of storage time, could not be demonstrated.

Table 7 Radioactive residues in apples dipped in labelled and unlabelled imazalil solutions at 0.05 kg ai/hL

Storage Duration (months)	TRRs [mg eq/kg] / Total Residues [mg/kg]					
	0	1	2	4	6	7
Labelled imazalil [a]	1.93	2.19	2.17	1.92	2.28	2.40
Unlabelled imazalil [b]	1.76	2.00	1.75	2.00	1.67	1.70

[a] Total Radioactive Residues in ^{14}C-imazalil sulphate treated apples, expressed as mg imazalil equivalents/kg. Total Radioactive Residues were determined by combustion analysis.

[b] Total residues of imazalil and R 014821, expressed as mg imazalil equivalents/kg, from apples treated with unlabelled imazalil sulphate. Residues of imazalil and R 014821were determined by GC-ECD.

Treated apples were homogenized in HCl (0.01 N). The homogenates were alkalinized using 1N NaOH and extracted (3×) with heptane/isoamyl alcohol (95:5; v/v). The combined organic phases were extracted with sulphuric acid (0.1 N). Following centrifugation, the organic phase was discarded. The aqueous phase was made alkaline using concentrated ammonia and re-extracted twice with heptane/isoamyl alcohol (95:5; v/v). The radioactivity in the combined extracts was determined by liquid scintillation counting while the radioactivity in the PES was determined by combustion. In the volatilisation study, the radioactivity in the trapping solutions was determined by LSC.

Table 8 Identification/characterization of radioactivity in apples sampled at various intervals of controlled atmosphere storage (dip treatment 0.05 kg ai/hL)

	Radioactivity, mg eq/kg (% TRR)					
Storage Duration (months)	0	1	2	4	6	7
TRRs [mg eq/kg}	1.93	2.19	2.17	1.92	2.28	2.40
Heptane:isoamyl alcohol (95:5; v/v)	1.87 (96.9)	2.04 (93.2)	2.09 (96.6)	1.64 (85.3)	1.92 (84.3)	1.97 (81.8)
Imazalil	1.83 (94.6)	2.01 (91.7)	2.03 (93.7)	1.61 (83.5)	1.84 (80.8)	1.76 (73.1)
R014821	0.04 (2.3)	0.03 (1.5)	0.06 (2.8)	0.03 (1.8)	0.08 (3.5)	0.21 (8.8)
PES	0.06 (3.1)	0.15 (6.8)	0.07 (3.4)	0.28 (14.7)	0.36 (15.7)	0.44 (18.2)

Extracted residues decreased from 97% TRR immediately after treatment to 82% TRR following 7 months of storage. The post-extraction solids (PES) accounting for 18% TRR after 7 months of storage, were not further subjected to hydrolysis. The nature of extracted radioactivity was determined using a reverse-phase radio-HPLC while quantitation of the analytes was determined by GLC analysis. Imazalil and R014821 accounted for most of the radioactivity, where concentrations of imazalil decreased with a corresponding increase in concentrations of R014821, as a function of storage duration. No other transformation product was detected. The radioactivity levels in the trapping solutions did not exceed those in the control (blank) solutions, therefore, volatilization of imazalil and imazalil-derived metabolites does not appear to be a route of metabolism.

Banana – Post-harvest

Ten growing banana plants (with a minimum of six leaves) maintained in a greenhouse were sprayed with ^{3}H-imazalil labelled on the asymmetric carbon atom (specific activity: 3.25 µCi/mg) at a dose rate corresponding to 50 mg ai/application/plant (Meuldermans *et al*., 1980, Report 23 979/21). Plants were treated several times, according to the treatment regime, summarized in Table 9, from the 6-leaf stage to the 15-leaf stage. Before spraying, plastic screens were placed around each plant in order to avoid contamination. The soil in the containers was also covered with a sheet of plastic.

Table 9 Description of the various treatment regimes

Plant No.	Weeks									
	0	2	4	6	8	10	12	14	16	18
1	Control									
2	TS									
3	TS									
4	T	S								
5	T	T	T	T	S					
6	T	T	T	T	TS					
7	T	T	T	T	TS					
8	T	T	T	T	T	S				
9	T	T	T	T	T	T	T	T	TS	
10	T	T	T	T	T	T	T	T	TS	
11	T	T	T	T	T	T	T	T	T	S

T: Treatment

S: Sampling

Five different parts of the plant were investigated: leaves, new leaves emerged, after the last treatment, pseudostem (including petioles), rhizome and roots. Radioactivity in these samples was determined by combustion analysis.

Table 10 Distribution of the radioactivity throughout the banana plants, according to the treatment regimes 2 to 6

Plant Part	Plant No.									
	2		3		4		5		6	
	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]
Roots	0.04	0.26	0.02	0.10	0.05	0.27	0.02	0.14	0.00	0.00
Rhizome	0.12	0.78	0.00	0.00	0.47	2.54	0.00	0.00	0.00	0.00
Pseudostem	0.09	0.57	0.68	4.40	0.36	1.96	0.02	0.16	0.00	0.00
Cigar leaf	-	-	<0.01	0.03	<0.01	0.03	0.00	0.00	-	
New leaf	-	-	-	-	0.14	0.73	0.00	0.00	-	
Leaves	14.93	98.39	14.69	95.50	17.44	94.48	9.78	73.09	18.99[c]	99.43
Withered/ yellow leaves	-	-	-	-	-	-	3.56	26.1	0.11	0.57
Total	15.18	100	15.39	100	18.46	100	13.38	99.39	19.10	100.0

[a] Represents % of radioactivity sprayed onto banana plants

[b] Represents % of radioactivity recovered in whole plant

[c] Includes 1 cigar leaf

Table 11 Distribution of the radioactivity throughout the banana plants, according to the treatment regimes 7 to 11

Plant Part	Plant No.									
	7		8		9		10		11	
	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]
Roots	0.00	0.00	0.00	0.00	0.03	0.22	0.06	0.32	0.02	0.13
Rhizome	0.00	0.00	0.00	0.00	0.12	0.75	0.18	1.04	0.05	0.28

Plant Part	Plant No.									
	7		8		9		10		11	
	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]	% sprayed[a]	% recovered[b]
Pseudostem	0.00	0.00	0.13	0.94	0.18	1.12	0.23	1.28	0.17	1.01
Cigar leaf	0.00	0.00	0.00	0.00	-	-	<0.01	0.02	<0.01	0.03
New leaf	-	-	0.05	0.39	-	-	-	-	0.07	0.39
Leaves	11.10	85.90	12.39	87.40	13.38[c]	84.82	14.03	17.52	14.75	83.89
Withered/ yellow leaves	1.82	14.10	1.60	11.27	2.07	13.10	3.08	79.83	2.51	14.27
Total	12.92	100.0	14.17	100	15.78	100.0	17.59	100.0	17.58	100.0

[a] Represents % of radioactivity sprayed onto banana plants

[b] Represents % of radioactivity recovered in whole plant

[c] Includes 1 cigar leaf

Irrespective of the number of applications and interval between applications and sampling, no more than 20% of radioactivity sprayed was recovered in the plant parts. The remaining 80% of the radioactivity sprayed had apparently landed on the plastic screens surrounding the banana plants. The majority of the radioactivity present on the plants was located in most part on the leaves with limited radioactivity in the remaining plant parts, demonstrating minimal translocation. Leaf homogenates of the various treated plants were repeatedly extracted in methanol:ammonia and methanol. The methanol extract was evaporated, made alkaline and partitioned with heptane:isoamyl alcohol (95:5; v:v) resulting in an alkaline organo-soluble extract. The radioactivity remaining in the aqueous phase after the alkaline extraction was further partitioned at pH 11, 9, 5 with chloroform and pH 2 with ethyl acetate, releasing each time further organo-soluble material and a final aqueous fraction. The remaining residue of the initial solvent extraction was subject to Soxhlet extraction (methanol/NH_4OH), resulting in a final residue and in a Soxhlet extract. The distribution of the radioactivity following the different steps of the extraction procedure is presented in Table 12.

Table 12 Distribution of the radioactivity between the different fractions resulting from the extraction procedure applied to leaf homogenates

	% TRR in leaves
Methanol extract	
Organo-soluble fraction - organo-soluble (after hydrolysis) - in heptane/isoamyl alcohol pH 11 - in $CHCl_3$ pH 9 - in $CHCl_3$ pH 5 - in ethyl acetate pH 2 Aqueous fraction	76.6 - 88.6 50.0 - 75.8 1.8 - 7.0 0.9 - 4.5 1.9 - 4.7 3.1 - 9.5 1.3 - 11.7
PES	
Soxhlet extract	4.2 - 6.7
Unextracted	7.2 - 17.0

Radio-HPLC was used to investigate the nature of the residue in the extracts of leaves collected from 4 selected plants. Imazalil and the metabolite R014821 were identified in the organo-soluble fraction (heptane/isoamyl alcohol) of the methanol extract. Various peaks were also eluted, potentially comprising several unidentified minor metabolites.

Table 13 Levels of free imazalil and the metabolite R014821 in leaves of banana plants

Plant no. (days after last treatment)	% TRR in organo-soluble fraction (% TRR in leaves)	
	Imazalil	R014821
8 (14)	34 (17)	23 (12)
9 (0)	47 (27)	22 (13)
10 (4)	37 (21)	28 (15)
11 (14)	22 (12)	38 (21)

Radiochromatograms of the organo-soluble material after hydrolysis at pH 11, pH 9, pH 5, pH 2 and of the final aqueous fraction of plant no. 11 contained small peaks indicating many minor metabolites. Although one major peak in the pH 2 extract accounting for about half of the radioactivity in this extract and a peak representing 30% of the radioactivity in the aqueous layer was observed, neither of these could be identified. The Soxhlet extract of leaves of plant no. 11 contained R014821 and imazalil as major radioactive compounds.

An additional study was carried out on a banana plant bearing bunches of bananas (Meuldermans *et al*., 1981, R 23 979/25). ^{3}H-imazalil (specific activity; 44.5µCi/mg) was sprayed 5 times at intervals of 2 weeks at 200 mg ai/application. Prior to spraying, the bananas were bagged. TRRs in lower leaves, upper leaves and bananas sampled 24 hours after the last spray application were 27 mg eq/kg, 70 mg eq/kg and <0.03 mg eq/kg, respectively, indicating minimal translocation from the leaves to the bagged bananas. The metabolic pattern was similar to that observed in the first study.

Cucumber

For the translocation study, roots of cucumber seedlings were soaked in a nutrient solution containing 2.4 ppm of [^{3}H]-imazalil (specific activity; 4.5 µCi/mg) for 6 hours and transferred to a blank nutrient solution. For the metabolism study, young cucumber plants were placed in a solution containing 10 ppm of [^{3}H]-imazalil (specific activity; 27.5 µCi/mg). Following a few hours of treatment, plants were washed and placed in a blank nutrient solution. For the leaf experiment of the same study, young cucumber plants were treated with an aqueous solution of [^{3}H]-imazalil (specific activity; 0.4 µCi/mg) applied using a syringe at a rate of 150 ppm. Plants were harvested 12 and 23 days after treatment. All cucumber plants were maintained in a greenhouse (Vonk *et al*., 1979, 44/2).

Cucumber plant parts were extracted with methanol and the PES were extracted with methanol and subsequently with methanol:concentrated HCl (60:1, v:v). After centrifugation, the extracts were concentrated and redissolved in methanol. The remaining PES were extracted with DMSO. Radioactivity in the extracts was measured using LSC, while whole plants and PES were subjected to combustion analysis. Characterization/identification was determined using TLC.

Ten days following the root treatment, radioactivity was detected in all plant parts from 0.4% of the applied radioactivity (AR) in the uppermost new leaves to 3.5-31.1% AR in the mature leaves and 31.1% AR in each of the stem and roots. Greater than 7% AR was detected in the blank nutrient solution, apparently exuded by the roots.

In the metabolism study, plants were collected from the nutrient solution 10, 17 and 24 days after treatment. In the first 10 days of treatment, 35% of the radioactivity taken up by the plants was exuded from the roots into the solution while only 2.5% was exuded in the last 14 days. After collection, the plants were separated into green parts and roots. Both parts were extracted successively with methanol and methanol:HCl (60:1, v:v). As the radioactivity decreased in roots from 10 to 24 days post-treatment, it correspondingly increased in the green parts. Greater than 44% of the recovered radioactivity was extracted while 46-56% of the radioactivity remained bound. The root extract collected 14 days after treatment contained imazalil and polar metabolites while the green parts contained only minor amounts of imazalil with most of the radioactivity present as polar compounds.

Foliar treated cucumber plants were separated into treated leaves and the rest of the plant, excluding roots. The plant parts were extracted in a similar manner to that of the metabolism study above. Several peaks were observed on the TLC plate but the presence of imazalil and any of its metabolites could not be confirmed.

While limited in scope, the study did demonstrate that the radioactivity translocated to a limited extent from the roots to the upper parts of the plant.

Tomato - Foliar

Greenhouse tomatoes were treated with three foliar applications of [U-^{14}C]-imazalil (specific activity; 134.6 µCi/mg) formulated as an emulsifiable concentrate at the rate of 300 g ai/ha/application for a total application rate of 900 g ai/ha (low dose) during the growing period (Rosenwald, 2008, B62706). Additional plants were treated three times at 1500 g ai/ha/application for a seasonal application rate of 4500 g ai/ha (high dose). Treatments were made at 10-day intervals and tomatoes were harvested 1 day after the last application (DALA).

The TRR in the tomatoes represented 0.441 mg eq/kg (low dose) and 2.851 mg eq/kg (high dose).

Table 14 Distribution of radioactivity from tomato juice/pomace after extraction (1 day after the last application)

Tomato fraction	Low Dose (300 g ai/ha)		High Dose (1500 g ai/ha)	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Surface wash	50.1	0.221	54.4	1.551
Juice*	9.8	0.043	8.3	0.238
Pomace	40.1	0.177	37.3	1.062
Total	100.0	0.441	100.0	2.851

* directly analysed by concentrating and HPLC analysis

Tomatoes were washed with acetonitrile, homogenised and centrifuged to separate clear tomato juice (*ca.* 80% w/w) from solid pomace.

Pomace was dried and the radioactivity determined by combustion. Aliquots of the dried pomace were extracted in sequence with acetonitrile, methanol, and acetonitrile:water (1:1, v:v). The PES was subjected to Soxhlet extraction with acetonitrile:HCl at pH 2 for 5 hours and thereafter hydrolysed under reflux with acetonitrile:HCl_{aq} at pH 2 for 4 hours. A second parallel pomace sample was hydrolysed using basic reflux conditions for 4 hours (acetonitrile:$NaOH_{aq}$; pH 14). Aliquots were analysed by LSC followed by HPLC and TLC.

The juice was concentrated, and aliquots were analysed by LSC followed by HPLC and TLC.

Table 15 Identification/Characterisation of radioactive residues in tomato surface wash, juice and pomace

Tomato fraction	Low Dose (300 g ai/ha)		High Dose (1500 g ai/ha)	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Surface wash	50.1	0.22	54.4	1.551
Imazalil	43.9	0.19	45.4	1.29
R055609 /R092977	6.2	0.03	5.1	0.144
R044179	ND	ND	4.0	0.113
Juice	9.8	0.043	8.3	0.238
Imazalil	2.6	0.012	2.4	0.067
R043449	0.4	0.002	0.2	0.006
R042639 /R045529	0.6	0.003	0.6	0.016
R014821	0.3	0.001	0.2	0.004
R055609 /R092977	0.6	0.002	0.4	0.013
R044179	0.3	0.002	0.3	0.008
Unknowns	5.0 (7)	0.022	4.2 (12)	0.123
Pomace	40.1	0.177	37.3	1.062
ACN / MeOH / ACN-water extract	14.7	0.065	12.7	0.362
Imazalil	10.0	0.044	8.4	0.240
R042639 /R045529	0.4	0.002	0.4	0.010
R055609 /R092977	0.7	0.003	ND	ND
R044179	1.6	0.007	1.6	0.046
Unknowns	1.9(2)	0.008	2.3 (2)	0.065
PES	25.4	0.112	24.6	0.700
ACN/HCl pH 2 Soxhlet extract	6.5	0.029	10.3	0.294
Remaining solid	18.9	0.083	14.3	0.406
ACN/HCl pH 2 reflux extract	6.2	0.027	3.1	0.089
Remaining solid	12.7	0.056	11.1	0.317
ACN/NaOH pH 14 reflux extract	21.5	0.095	24.1	0.686
Imazalil	20.5	0.090	23.2	0.663
R042639 /R045529	ND	ND	0.1	0.003
R014821	ND	ND	0.1	0.003
R055609 /R092977	0.4	0.002	0.4	0.012
Unknowns	ND	ND	0.4 (3)	0.014
Remaining solids	3.9	0.017	0.5	0.014
Total Extracted	96.1	0.423	99.5	
Total Identified	88.5	0.392	92.8	
Imazalil	77.0	0.340	79.4	2.26
R043449	0.4	0.002	0.2	0.006
R042639 /R045529	1.0	0.005	1.1	0.029
R014821	0.3	0.001	0.3	0.008
R055609 /R092977	7.9	0.035	5.9	0.168
R044179	1.9	0.009	5.9	0.168
Total Characterized	6.9	0.031	6.9	0.202
Total Unextracted	3.9	0.017	0.5	0.014
Accountability	100		100	

ND: Not Detected

( ): Number of unknowns detected

Highest TRRs were found in the surface wash (≥ 50% TRR) and in pomace (~40% TRR), whereas < 10% of TRR was found in the tomato juice.

Limited extraction of the TRRs in pomace was achieved using acetonitrile, methanol and acetonitrile-water. However, following hydrolysis using basic harsh conditions, the unextracted residues remaining in the pomace only accounted for 3.9% and 0.5% TRR for the normal and high dose levels, respectively.

The parent compound represented 77.0% TRR (0.340 mg eq/kg) for the low dose and 79.4% TRR (2.26 mg eq/kg) for the high dose level. In an amendment to the study (Gassen, 2010, B62706), the ratio of enantiomers of imazalil (i.e., [(±)-1-[2-(2,4-dichlorophenyl)2-(2-propenyloxy) ethyl]-1H-imidazole]) in the juice, pomace extracts and surface wash was investigated using a chiral HPLC column. The total radioactivity of all samples corresponded well with the values reported in Table 15. The results showed the enantiomer ratio was approximately 1 in all samples, indicating no change in the enantiomer ratio and that stereospecific metabolism of imazalil is highly unlikely.

In addition to the parent compound up to five known (R043449, R042639/R045529, R014821, R055609/R092977 and R044179) and fifteen unknown metabolites were detected for both dose levels. None of the identified metabolites exceeded 10% TRR and none of the unknowns exceeded 2% TRR.

Potato

Study 1

Organic potatoes were bought from a supermarket and treated with [^{14}C-U-ring] imazalil (specific activity: 134.6 µCi/mg) formulated as a solution containing 100 g ai/L (Rosenwald, 2009, B69412). Potatoes were treated by drip application onto the peel surface at the recommended rate of 15 g ai/ton corresponding to 15 mg ai/kg potato tuber and at an exaggerated rate of 75 mg ai/kg potato. Eight potato tubers were treated and three tubers were left untreated as control samples. Seven of the treated potatoes were stored for three months under cooled conditions in the dark and planted outdoors in pots. One potato was directly processed after application to assess the distribution of radioactivity between peel and pulp. An additional untreated potato was treated three months later on its day of planting to investigate the difference in timing of application on the TRR under realistic outdoor conditions.

Potatoes were harvested at maturity, separated into aerial parts, roots, tubers and soil samples. Mature potatoes were washed, to remove soil and release any surface radioactivity, and peeled where the peel and peeled tubers were extracted and/or combusted. The dried peel fraction was extracted in sequence with acetone (3x), methanol (2x) and methanol:water (1:1; v:v, 1x). The remaining residue was combusted to determine the radioactivity in the PES. The peeled tuber fraction was lyophilized, homogenized and combusted to determine the TRR. Radioactivity was measured by LSC. Analysis of the aqueous/organic peel extracts was performed by HPLC.

The TRRs in mature potatoes grown from treated seed potatoes were generally below 0.01 mg eq/kg in the whole potato with one exception (>0.01 mg/kg at the high dose level). Thus, no further analytical work was conducted for the characterization of the TRRs.

Table 16 Total radioactive residues in mature potatoes

	Radioactivity, mg eq/kg (% TRR)				
	15 mg ai/kg		75 mg ai/kg		15 mg ai/kg
Commodity	Potato tuber treated before storage	Potato tuber treated before storage	Potato tuber treated before storage	Potato tuber treated before storage	Potato tuber treated directly before planting
Peel	0.004 (71%)	0.005 (69%)	0.007 (76%)	0.037 (81%)	0.0004
Peeled tuber	0.002 (29%)	0.002 (31%)	0.002 (24%)	0.009 (19%)	0.0007
Total	<0.01 mg eq/kg	<0.01 mg eq/kg	<0.01 mg eq/kg	>0.01 mg eq/kg	<0.01 mg eq/kg

The residue level in potatoes grown from freshly treated tubers was up to ten times lower than those measured in potatoes derived from tubers which were treated prior to storage (3 months). In both cases the residue levels in the whole tubers of the low dose were <0.01 mg eq/kg. From 19-31% TRR were located in the peeled tuber while the majority of the residues (69-81% TRR) were found in the peel.

The extractability of ^{14}C-imazalil residues was representatively investigated using a potato treated immediately before planting (Table 17). The extracted radioactivity of the peel fraction represented 95.3% TRR. The corresponding unextracted radioactivity amounted to 4.6% TRR and the rest of the residues, determined in the peeled tuber, represented 0.1% TRR. The total residues summed up from each fraction amounted to 7.55 mg eq/kg.

Table 17 Distribution of the radioactivity in potato treated a rate of 15 mg ai/kg immediately prior to planting

Fraction	Radioactivity	
	% TRR	mg eq/kg fresh weight
Peel		
Acetone extract	61.9	4.67
Methanol extract	33.4	2.52
Unextracted residue	4.6	0.35
Peeled tuber	0.1	0.01
Total tuber	100	7.55

Study 2

[^{14}C]-Imazalil (specific activity: 151.4 µCi/mg) labelled at the benzoyl-ring and formulated as a solution containing100 g ai/L, was applied by simulated drench application to potato tubers (variety *Laura*) at a target rate of 15 mg ai/kg potato tubers (Walther, 2012, C94695). Ten potatoes were treated and stored on a wire grid in storage boxes at approximately 5 °C.

Duplicate potato tuber samples were taken immediately after treatment (day 0) and after 14, 29, 91 and 188 days of storage.

At each sampling interval, potato tubers were weighed and washed by dipping each potato for approximately 10 seconds into acetonitrile:water (1:1, v:v) in a glass jar. The radioactivity in the rinsate was measured by LSC and subjected to chromatographic analysis. Washed potatoes were peeled using a potato peeler. The potato peel was homogenized and extracted at ambient temperature using acetone and methanol:water (1:1, v:v). From day 14 onwards, the solid phase after ambient extractions of potato peel was extracted under reflux conditions with acetonitrile:0.1 N HCl (1:1, v:v) for 4 hours. Peeled potato tubers were washed by dipping each tuber for approximately 10 seconds into acetonitrile:water (1:1, v:v) in a glass jar to minimize the carry-over of radioactive residue from the peel to the tuber. The radioactivity in the rinsate was measured by LSC. The tubers were lyophilized. For samples collected on day 188, an aliquot of the lyophilized tuber material was extracted using acetone and methanol:water (1:1, v:v).

The residue in the surface wash decreased from 64% TRR (11 mg eq/kg) on day 0 to 35% TRR (5.4 mg eq/kg) by day 188. The extracted residue in the peel increased from 33% TRR (5.7 mg eq/kg) on day 0 to 54% TRR (8.7 mg eq/kg) on day 91 and subsequently decreased to 46% TRR on day 188 (7.2 mg eq/kg). The unextracted residue of the peel increased from 3% TRR (0.5 mg eq/kg) on day 0 to a maximum of 23% TRR (3.6 mg eq/kg) observed on day 91. Acidic reflux treatment released up to 6% TRR (0.92 mg eq/kg) by day 188. Residues in potato tubers were approximately 0.2% TRR (0.03 mg eq/kg) except for day 14 where tuber accounted for 0.88% TRR (0.15 mg eq/kg). Radioactivity on potato tubers was partly removed by surface washing. TRRs in washed tubers, determined by combustion analysis, ranged from 0.06–0.12% TRR (0.01–0.02 mg eq/kg), demonstrating negligible penetration of residues from peel into tuber. The radioactivity in the potato extracts was determined by liquid scintillation analysis, and that in solid samples was determined by combustion analysis.

Aliquots of potato surface wash and peel extract from each sampling interval and of tuber extracts from the last sampling interval were analysed for imazalil and its potential metabolites. HPLC-UV was used as a primary method for chromatographic analysis. Imazalil and its metabolites were separated using a C18 column eluted with methanol and water buffered with ammonium acetate. The enantiomers of imazalil were separated by HPLC-UV using a chiral sensitive column and water:acetonitrile (1:1, v:v) eluent. One- and two-dimensional TLC chromatography was used as a confirmatory method.

Imazalil was the predominant residue in the surface wash and peel extracts decreasing from 94% TRR (16.1 mg eq/kg) on day 0 to 73% TRR (10.9 mg eq/kg) on day 188. Since imazalil accounted for <0.02% TRR (<0.003 mg/kg) in the tuber extract, it was not included in the overall concentration of parent compound in surface wash and peel. The metabolites R014821 and R044177 each reached a maximum of 9% TRR (1.4 mg eq/kg) and 3.5% TRR (0.54 mg eq/kg), respectively, in surface wash and peel extracts by day 188. Additional metabolites accounted for less than 2% TRR. These metabolites were characterised in terms of solubility, HPLC retention times and conjugation to plant constituents. Following acidic reflux extraction of the peel solids, the analytes identified included the parent (1.3-4.0 % TRR), metabolites R014821 (0.1-0.2% TRR) and R044177 (0.04-0.1% TRR) as well as other unknown fractions that were likely aglycones of conjugates hydrolysed under acidic reflux conditions.

Enantiomeric analysis of the surface wash and peel extract sampled on day 0, 91 and 188 showed that the ratios of imazalil enantiomers remained unchanged during storage of treated potato and that stereospecific metabolism of imazalil is highly unlikely.

Table 18 Distribution and identification/characterization of the radioactive residues in potatoes stored for 0-188 days

Fraction	Storage intervals									
	0 day [17.16 mg eq/kg)		14 days [17.04 mg eq/kg]		29 days [17.28 mg eq/kg]		91 days [15.97 mg eq/kg]		188 days [15.57 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Surface wash	63.98	10.98	34.98	5.96	35.30	6.10	22.86	3.67	34.82	5.40
Imazalil	61.45	10.54	32.80	5.59	32.60	5.63	21.00	3.35	30.10	4.69
R014821	n.d.	n.d.	0.60	0.10	1.05	0.18	1.03	0.16	2.51	0.39
R044177	n.d.	n.d.	0.27	0.05	0.66	0.11	0.21	0.03	0.92	0.14
Unknowns	2.53	0.44	1.32	0.22	1.0	0.17	0.64	0.10	1.26	0.20
Peel	35.86	6.15	64.13	10.93	64.54	11.18	76.97	12.28	64.99	10.14
Extracted	33.10	5.67	51.80	8.83	51.44	8.92	54.42	8.68	45.74	7.12
Imazalil	32.65	5.60	44.80.	7.63	47.15	8.14	49.60	7.9	36.0	5.60
R014821	n.d.	n.d.	2.81	4.79	2.50	0.43	2.94	0.47	5.86	0.91
R044177	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.43	0.07	2.38	0.37
Unknowns	0.49	0.07	4.21	0.72	1.82	0.31	1.45	0.23	1.45	0.22
PES	2.76	0.47	12.34	2.10	13.09	2.26	22.56	3.59	19.26	3.00
Reflux	n.n	n.n	3.12	0.53	1.54	0.26	5.77	0.92	5.38	0.83
Imazalil	n.a.	n.a.	2.4	0.46	1.30	0.22	4.02	0.64	4.00	0.62
R014821	n.a.	n.a.	0.09	0.02	0.15	0.02	0.19	0.03	0.67	0.10
R044177	n.a.	n.a.	0.10	0.02	0.04	0.01	0.14	0.02	0.12	0.02
Unknowns	n.a.	n.a.	0.54	0.09	0.07	0.01	1.42	0.23	0.56	0.09
Peeled tuber	0.16	0.03	0.88	0.15	0.16	0.03	0.17	0.027	0.18	0.028
Wash	0.05	0.01	0.80	0.13	0.10	0.02	0.08	0.013	0.06	0.010
Unextracted	n.p.	n.p.	n.p.	n.p.	n.p.	n.p.	n.p.	n.p.	0.12	0.010
Combustion	0.10	0.02	0.08	0.01	0.06	0.01	0.09	0.014	0.07	0.018
Total Extracted	97.13	16.66	90.70	15.45	88.38	15.30	83.12	13.29	86.0	13.36
Total Identified	94.10	16.14	83.85	14.29	85.45	15.63	79.60	12.71	82.65	12.85
Imazalil	94.10	16.14	80.0	13.63	81.05	14.01	74.65	11.92	70.1	10.91
R014821	n.d.	n.d.	3.5	0.60	8.7	1.50	4.15	0.66	9.0	1.40
R044177	n.d.	n.d.	0.35	0.06	0.7	0.12	0.8	0.13	3.45	0.54
Total Characterized	3.02	0.51	6.07	1.03	2.89	0.49	3.51	0.56	3.35	0.51
Total Unextracted	2.86	0.49	9.30	1.58	11.62	2.01	16.88	2.68	14.0	2.19
Accountability	100		100		100		100		100	

n.n. Reflux extraction was not needed since the cumulated residue in the surface wash and peel extract was near 100% TRR

n.a.: Not analysed

n.p. Extraction was not performed due to the small amounts of radioactivity involved

n.d.: Not detected

Spring wheat – seed treatment

^{14}C-imazalil, radio-labelled on the stereogenic centre (specific activity: 183.7 µCi/mg) was applied to spring wheat seeds (variety Axona) at a rate of 49.3 g ai/100 kg seed (O'Connor, 1993, 92/JST003/1046). Treated seeds were sown in pots left outdoors under normal environmental conditions. Forage was sampled 42 days after sowing while straw and grain were harvested approximately 5 months after sowing.

TRRs in forage, straw and grain were 1.36, 0.15, 0.003 mg eq/kg, respectively. Due to the very low level of TRRs in grain, no further investigation was carried out.

Several extraction procedures were assayed on forage and straw, leading to variable results. The most appropriate system to obtain an efficient extraction was two successive ambient extractions with a mixture methanol:chloroform:HCl (0.1 N) (2:1:0.8; v:v:v) followed by a Soxhlet extraction with methanol:HCl (0.1 N) (95:5; v:v). Ambient extraction of the remaining residue with HCl (1 N) and subsequently with ammonia hydroxide solution (1 N) extracted further conjugated materials. Finally, reflux extraction with HCl (6 N) then with NaOH (10 N) released additional radioactivity corresponding to incorporated radioactive residues. Identification/characterization was accomplished using 1D- or 2D-TLC and/or reverse-phase HPLC.

Table 19 Distribution and identification of TRRs in forage and mature straw

Fraction	Forage [1.36 mg eq/kg]		Straw [0.15 mg eq/kg]	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Extracted	72.6	0.99	44.0	0.06
Imazalil	24.36	0.33	16.86	0.03
R014821	8.30	0.11	3.79	<0.01
R042639	4.58	0.06	3.74	<0.01
Unknowns (no more than 4)	25.88	0.35	19.23	<0.04
PES	12.6	0.16	16.4	0.09
1N HCl	0.3	<0.01	ND	ND
1N NaOH	4.0	0.05	4.7	<0.01
6 N HCl	5.9	0.08	8.1	0.01
10 N NaOH	2.4	0.03	3.6	<0.01
Remaining solids	7.7	0.11	58.5	0.09
Accountability	93		119	

ND: Not detected

Greater than 72% TRR (0.99 mg eq/kg) in the forage was extracted. Successive mild base and acid hydrolysis of the PES released 4.3% TRR (0.05 mg eq/kg). Subsequent reflux with harsh acid and base released a further 8.3% TRR (0.11 mg eq/kg). The radioactivity remaining in the final PES accounted for 7.7% TRR (0.11 mg eq/kg). Extraction of mature straw released significantly less radioactivity compared to forage (44% TRR; 0.06 mg eq/kg). Mild and harsh acid and base hydrolysis released a further 16.4% TRR (<0.03 mg eq/kg) with 58.5% TRR (0.09 mg eq/kg) remaining in the final PES.

The parent compound was observed in both matrices, decreasing from 0.33 mg eq/kg in forage to 0.03 mg eq/kg in straw. The levels of the metabolites R014821 and R042639 in forage also decreased from 0.11 mg eq/kg and 0.06 mg eq/kg, respectively, to <0.01 mg eq/kg in straw. Several similar unknown compounds, based on retention time, were observed in both forage and straw, none of which accounted for >4.3% TRR. It was noted that the deviations in the accountability may be a result of the extensive work-up of the individual samples as well a lack of a clean-up step prior to analysis, causing potential co-extraction with plant material.

Barley – seed treatment

Barley seeds were treated at 1.8 g/100 kg seed with a 40% ethanolic solution of [^{3}H]-imazalil (specific activity; 0.68 µCi/mL) prior to planting in pots and maintained in a greenhouse at 25ºC (Vonk *et al*., 1979, 44/2). Three weeks after sowing, plants were harvested and the soil, roots, seedcoats and leaves were analysed

Most of the label appeared to have dissipated into the soil with smaller amounts transported from the seed into the plant. Consistent with the cucumber translocation study, less radioactivity was found in the upper leaves compared to the lower leaves.

Table 20 Distribution of radioactivity in soil and plant parts of barley

Part	%Radioactivity applied to seeds	mg eq/kg (dry weight)
Soil	66	
Seed coat	5.5	11
Roots	7	0.25
Leaves and stem	6	2.08
Not recovered	15.5	

When barley roots were extracted with methanol, only 20% of the radioactivity was released. However, when barley roots and leaves were each extracted using toluene:1N NaOH: Na_2SO_4 (4:2:1, v:v:v), 50% of the radioactivity was released. When the methanolic root extract was subjected to TLC analysis, both imazalil and a polar unknown compound, present at significantly lower levels, were observed. No further attempt was made to identify the polar metabolite based on its low concentration.

Figure 1 Proposed Metabolic Pathway of Imazalil in Plants

Animal Metabolism

Lactating Goat

The sulfate salt of ^{14}C-imazalil, radiolabelled on the stereogenic centre (specific activity; 39.7 µCi/mg), was orally administered to a lactating goat by intubation, twice daily for 3 consecutive days (Van Dijk, 1992, 296662; Mannens, 1993, R23979/FK1346). The actual daily dose level was 10.4 mg/kg bw/day corresponding to 180 ppm in the feed (dry matter). The average daily milk production during dosing was 293 mL. Milk was collected twice daily before administration of the test compound while urine and faeces were collected once daily. The goat was sacrificed 16 hours after administration of the last dose.

Table 21 Concentration of radioactivity in milk following each oral dose

Time period after first administration	TRRs (mg eq/kg)
0-8	0.406
8-24	0.619
24-32	0.826
32-48	0.808
48-56	0.968
56-72	0.991

Radioactivity levels in milk increased from the first administered dose to the third but appeared to plateau after the fourth administration (32 hours).

Greater than half of the administered dose (AD) was excreta-related (57.3% AD). Limited radioactivity was eliminated in the milk (0.1% AD) and the tissue burden was low (1.4% AD). The overall recovered radioactivity accounted for 59% AD. Although the radioactivity was not measured in the carcass and GI tract, it was assumed that these matrices accounted for the remaining radioactivity.

At sacrifice (16 hours after last administration), TRRs were 19.8, 9.6, 0.36 and 0.09 mg eq/kg in liver, kidney, muscle (round, loin and flank) and fat (perirenal, omental, subcutaneous), respectively.

Table 22 Balance of radioactivity in goat following oral administration of [^{14}C]-imazalil for 3 consecutive days

Sample	%AD	mg eq/kg
Urine	48.1	-
Feces	4.3	-
Cage wash	4.9	-
Milk (0-72 hours)	0.1	-
Blood	0.3	1.13
Liver	0.9	19.80
Kidney	0.1	9.62
Muscle	0.4	0.36
Fat	<0.05	0.09
Total	59.1	

The distribution of radioactivity in milk, (56-72 hours after the last administration, containing the highest amount of radioactivity) after fat separation using centrifugation, and protein precipitation using acetone, indicated that the majority of the radioactive residue (92.3% TRR) was contained in whey. Levels in fat and protein accounted for 6.9% and 4.0% TRR, respectively. These fractions were not further analysed.

The whey from milk was extracted using acetonitrile and successively partitioned using hexane, dichloromethane and ethyl acetate under neutral and acidic conditions, resulting in a total recovery of 54.2% TRR. The remaining radioactivity in whey was further hydrolysed with 1M HCl (16 hours at 70ºC) and then partitioned with ethyl acetate under neutral, basic and acidic conditions.

The liver was incubated with phosphate buffer prior to successive extractions using methanol, methanol:water (8:2, v:v), acetonitrile, methanol:ammonia (0.5 M) followed by hydrolysis using 0.01M HCl (16 hours at 70ºC). Several other extraction procedures were investigated to improve the extraction efficiency; however, none showed an improvement in the extracted radioactivity. Radioactivity in the unextracted fraction was determined by combustion analysis.

Kidney and muscle were successively extracted with methanol and methanol:water (8:2, v:v). The remaining radioactivity in the tissues was further extracted in a Soxhlet apparatus for 16 hours with methanol. The radioactivity extracted by methanol and methanol:water was combined and partitioned with hexane, dichloromethane and ethyl acetate under neutral conditions. The aqueous phase was acidified with concentrated HCl to pH1.0 and partitioned with ethyl acetate. The aqueous phase was also partitioned under basic conditions (with ammonia at pH10). The remaining aqueous phase of kidney and muscle were acidified with 4N HCl, hydrolysed for 6 hours at 70ºC by refluxing and partitioned with ethyl acetate under acid and neutral condition.

Fat was successively extracted three times with dichloromethane, acetonitrile, acetonitrile:chloroform (1:1, v:v), methanol and finally with methanol in a Soxhlet apparatus. The unextracted residue was dissolved in hexane and partitioned with acetonitrile.

Identification/characterization of the metabolites was accomplished using co-chromatography (TLC) and co-elution (HPLC) with reference standards.

Table 23 Distribution and characterisation/identification of radioactivity in milk whey

Component	% TRR	mg eq/kg
Milk fat	6.9	0.07
Milk Protein (PES)	4.0	0.04
Whey	92.3	0.89
Hexane phase	0.4	0.004
Aqueous phase	91.9	0.88
Combined dichloromethane/ethyl acetate fraction	53.8	0.52
R043449	4.7	0.05
R042639	1.5	0.01
R044085	2.3	0.02
R061000	4.9	0.05
R062775	7.2	0.07
R066996	1.4	0.01
R023366	3.0	0.03
Unknowns (no more than 12)	28.8	0.28
Aqueous fraction	38.1	0.366
Combined ethyl acetate fraction	14.7	0.141
R043449	1.7	0.02
R042639	2.0	0.02
R061000	0.6	0.01
R014821	3.4	0.03
R044177	1.0	0.01
R060998	1.0	0.01
Unknowns (no more than 3)	5.0	0.05
Aqueous fraction	23.4	0.225
Total Extracted	92.3	0.89
Total Identified	34.7	0.33
R043449	6.4	0.06
R042639	3.5	0.03
R044085	2.3	0.02
R061000	5.5	0.06
R062775	7.2	0.07
R066996	1.4	0.01
R023366	3.0	0.03
R014821	3.4	0.03
R044177	1.0	0.01
R060998	1.0	0.01
Total Characterized	33.8	0.33
Total Unextracted	-	-
Accountability	103.2	0.991

Table 24 Distribution and characterisation/identification of radioactivity in liver

Component	% TRR	mg eq/kg
Extracted	80.0	17.12
Organosoluble	41.3	8.84
Hexane phase	6.0	1.28
Imazalil	2.8	0.60
R055609	0.4	0.09
Unknowns (no more than 3)	2.8	0.60
Combined ethyl acetate pH 10 and pH 2 extracts	3.4	0.73
Combined dichloromethane/ethyl acetate fraction	31.9	6.83
Imazalil	3.1	0.66
R055609	4.2	0.90
R043449	2.1	0.45
R044177	0.6	0.13
R014821	0.9	0.19
R042639	2.7	0.58
R044085	4.2	0.90
R061000	4.1	0.88
R060998	0.6	0.13

Component	% TRR	mg eq/kg
R062775 Unknowns (no more than 3)	2.0 7.4	0.43 1.58
Aqueous soluble	23.9	5.12
Combined ethyl acetate fraction	3.3	0.71
Aqueous fraction	20.6	4.41
Unextracted	12.5	2.68
Total Extracted	80.0	17.12
Total Identified	27.7	6.04
Imazalil	5.9	1.26
R055609	4.6	0.99
R043449	2.1	0.45
R044177	0.6	0.13
R014821	0.9	0.19
R042639	2.7	0.58
R044085	4.2	0.90
R061000	4.1	0.88
R060998	0.6	0.13
R062775	2.0	0.43
Total Characterized	10.2	2.18
Total Unextracted	12.5	2.68
Accountability	92.5	19.80

Table 25 Distribution and characterisation/identification of radioactivity in kidney and muscle

Component	Kidney		Muscle	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Extracted	96.6	9.33	97.8	0.34
Organosoluble	48.1	4.65	71.2	0.24
Hexane phase	5.0	0.49	5.9	0.02
Imazalil R043449 Unknown	2.3 ND 2.7	0.22 ND 0.26	1.9 2.4 1.6	0.006 0.008 0.006
Combined dichloromethane/ethyl acetate extracts	43.1	4.16	65.3	0.22
Imazalil R043449 R044085 R061000 R042639 R062775 R023366 R055609 Unknowns (no more than 13)	1.3 ND 2.7 15.0 1.5 0.5 2.9 ND 19.2	0.13 ND 0.26 1.45 0.14 0.05 0.28 ND 1.86	1.0 12.8 8.4 21.2 ND ND ND 7.1 14.8	0.003 0.04 0.03 0.07 ND ND ND 0.02 0.05
Aqueous soluble	42.0	4.06	21.9	0.08
Combined ethyl acetate fraction	9.2	0.89	11.3	0.04
Aqueous fraction	32.8	3.17	10.6	0.04
Unextracted	2.8	0.27	7.1	0.02
Total Extracted	96.6	9.33	97.8	0.34
Total Identified	22.6	2.18	54.8	0.18
Imazalil	3.6	0.35	2.9	0.009
R043449	ND	ND	15.2	0.05
R044085	2.7	0.26	8.4	0.03
R061000	15.0	1.45	21.2	0.07
R042639	1.5	0.14	ND	ND
R062775	0.5	0.05	ND	ND
R023366	2.9	0.28	ND	ND
R055609	ND	ND	7.1	0.02
Total Characterized	21.9	2.12	16.4	0.06
Total Unextracted	2.8	0.27	7.1	0.02
Accountability	99.4	9.60	104.9	0.36

Table 26 Distribution and characterisation/identification of radioactivity in fat

Component	% TRR	mg eq/kg
Extracted	80.9	0.086
Organosoluble	76.9	0.082
Hexane phase	15.3	0.016
Acetonitrile phase	61.6	0.066
Imazalil	5.7	0.006
R044179	24.9	0.027
R043449	9.0	0.010
R044177	6.0	0.006
Unknowns (no more than 3)	16.0	0.017
Methanol + Methanol soxhlet	4.0	0.004
Unextracted	4.8	0.005
Accountability	85.7	0.091

Imazalil was not observed in milk whey, however, a total of 10 minor metabolites were identified ranging in concentrations from 1.0-7.2% TRR (0.01-0.07 mg eq/kg). Up to 15 unknown metabolites, accounting for a total of 33.8% TRR (0.33 mg eq/kg), were characterized. Only one of these exceeded 10% TRR (0.10 mg eq/kg). Radio-HPLC analysis of this metabolite fraction revealed that it comprised of at least 7 metabolites, the most prominent accounting for 8% TRR (0.08 mg eq/kg).

Successive extractions of the liver sample with various organic solvents released 80% TRR. In the hexane and dichloromethane/ethyl acetate extracts, imazalil accounted for a total of 5.9% TRR (1.26 mg /kg). The only other analyte observed in both phases was R055609 and accounted for a total of 4.6% TRR (0.99 mg eq/kg). Eight additional minor metabolites were identified in the dichloromethane/ethyl acetate phase, none of which accounted for greater than 4.2% TRR (0.90 mg eq/kg). A total of six unknown metabolites were observed in both phases, accounting for 10.2% TRR (2.18 mg eq/kg).

An average of 97% TRR was extracted from kidney and muscle, the majority of which partitioned into the organosoluble phase. Imazalil was present in both the hexane and the dichloromethane/ethyl acetate phases accounting for a total of 3.6% TRR (0.35 mg /kg) in kidney and 2.9% TRR (0.009 mg /kg) in muscle. R043449 was not observed in kidney but in muscle, it was the only metabolite observed in both phases, accounting for a total of 15.2% TRR (0.05 mg eq/kg). In both tissues, the major metabolite observed was R06100, accounting for 15% TRR (1.45 mg eq/kg) in kidney and 21.2% TRR (0.07 mg eq/kg) in muscle. Four additional minor metabolites were observed in kidney, representing 0.5-2.9% TRR (0.05-0.28 mg eq/kg). In contrast, only 2 additional minor metabolites were observed in muscle, R044085 (8.4% TRR; 0.03 mg eq/kg) and R055609 (7.1% TRR, 0.02 mg eq/kg). A total of 14 unknown metabolites were observed in kidney ranging in concentration from 0.2-4.9% TRR (0.02-0.47 mg eq/kg). Conversely, only 6 unknown metabolites were observed in muscle, none of which accounted for more than 5.4% TRR (0.02 mg eq/kg).

Up to 81% of the radioactivity in fat was extracted. Following partitioning, only the acetonitrile phase was analysed as the radioactivity in the hexane phase was low (15.3% TRR; 0.016 mg eq/kg). Imazalil was a minor residue in the acetonitrile phase (5.7% TRR; 0.006 mg /kg) as were the metabolites R043449 and R044177 accounting for 9.0% TRR (0.01 mg eq/kg) and 6.0 % TRR (0.006 mg eq/kg), respectively. R044179 was the only predominant metabolite observed, accounting for 25% TRR (0.027 mg eq/kg). A total of three unknown metabolites were detected, however, none represented greater than 0.01 mg eq/kg.

Attempts were made to identify/characterize the radioactivity in the aqueous phases remaining after extraction and/or partitioning of milk, liver and kidney. These fractions were subjected to acid hydrolysis using 4N HCl for 16 hours at 70ºC. The metabolites, analysed by radio-HPLC, demonstrated minimal changes in the metabolic profile following the work up.

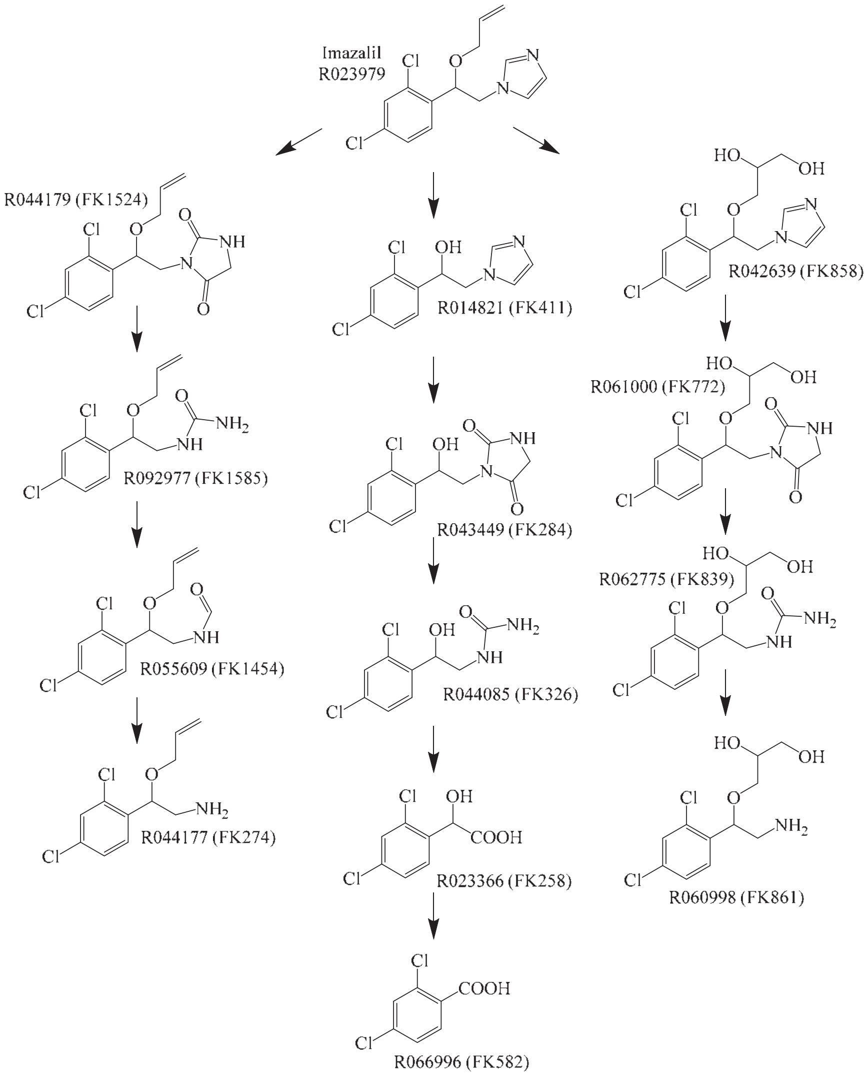


Figure 2 Proposed Metabolic Pathway of Imazalil in Lactating Goats

Laying hen

Ten laying hens were dosed orally for 10 consecutive days with [U-C^{14}]-imazalil sulphate (specific activity; 75 µCi/mg) at 4.6 mg/kg bw/day, equivalent to an average of 66 mg/kg in the feed (dry matter) (Hallifax *et al*., 1993, 93/JST006/0615). Eggs were collected daily immediately prior to dosing while urine and feces were collected once daily. The hens were sacrificed 20 hours after the final dose.

TRRs in eggs (combined yolk and white) reached a plateau (0.8 mg eq/kg) by day 6.

Table 27 Concentration of radioactivity in eggs following each oral dose

Day	TRRs (mg eq/kg)
1	0.25
2	0.37
3	0.66
4	0.68
5	0.74
6	0.81
7	0.73

Day	TRRs (mg eq/kg)
8	0.84
9	0.78
10	0.81

At sacrifice, almost all the administered radioactivity was recovered in excreta (94.7% AD), cage wash and cage debris (4.5% AD). Limited radioactivity was eliminated in the eggs (0.3% AD) and the tissue burden was low (1.8% AD). The content of the GI tract was collected however, the carcass was not retained for analysis. The overall recovered radioactivity accounted for 101%.

TRRs were highest in the GI tract and contents followed by liver, skin, muscle and fat.

Table 28 Distribution of radioactivity in laying hen tissues

Tissue	%AD	TRRs (mg eq/kg)
Fat (abdominal)	0.0	0.119
Fat (skin)	0.0	0.126
Muscle (breast)	0.0	0.130
Muscle (thigh)	0.0	0.165
Skin	0.0	0.435
Liver	0.6	10.2
GI tract and contents	1.2	16.5
Total	1.8	

Samples of liver, muscle, fat and eggs were extracted using methanol/ammonia. The methanol extracts after evaporation of methanol were then partitioned twice with ethyl acetate. The aqueous phases remaining after partition were combined and hydrolysed under acidic (1 M HCl) or basic (1 M NaOH) conditions for 2 hours. After adjusting to pH 2, the hydrolysates were partitioned with ethyl acetate.

For liver and muscle, further extraction was performed by continuous reflux of the solid residue with methanol in a Soxhlet apparatus. Post-extraction solids (PES) remaining after Soxhlet extraction with methanol were subject to mild acid or mild base hydrolysis or digestion by protease at 37 °C in a triethylamine acetate buffer at pH 7.5 in order to release further radioactivity. The acid, base and protease hydrolysates were further characterized by partitioning into ethyl acetate. The aqueous remainder from the protease digestion, containing most of the radioactivity, was further characterized by acid and base reflux, followed by partitioning into ethyl acetate.

Qualitative determination of the metabolites was accomplished using normal phase TLC and quantitation of the metabolites was based on reverse phase radio-HPLC analysis.

Table 29 Distribution and characterisation/identification of radioactivity in liver and muscle

Component	Liver		Muscle	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Extracted	64.0	6.63	63.5	0.09
Organic phase	20.5	2.08	50.5	0.07
R060998	0.7	0.07	ND	ND
R061000	1.5	0.15	ND	ND
R044085	1.0	0.10	ND	ND
R044085/R110740	ND	ND	16.3	0.02
R042639	4.0	0.40	14.7	0.02
Unknowns (no more than 12)	13.3	1.35	19.4	0.03
Aqueous phase	46.0	4.68	13.0	0.02
Hydrolysed	29.4	2.99	5.5	0.01
R062775	2.1	0.21	ND	ND
R061000	7.3	0.75	ND	ND
R042639	3.2	0.33	ND	ND
Unknowns (no more than 9)	16.7	1.68	ND	ND
Non-hydrolysed	16.6	1.69	7.4	0.01
R062775	2.3	0.23	ND	ND
Unknowns (no more than 6)	14.3	1.46	ND	ND
PES	34.0	3.47	40.6	0.06
Soxhlet extraction	12.9	1.31	8.0	0.01
R062775	0.8	0.08	ND	ND

Component	Liver		Muscle	
	% TRR	mg eq/kg	% TRR	mg eq/kg
R061000 R042639 R110740 Unknowns (no more than 6)	0.61 1.2 2.7 7.6	0.06 0.13 0.27 0.75	ND ND ND ND	ND ND ND ND
Residue	21.1	2.15	32.6	0.05
Organic	1.1	0.11	0.0	0.0
Aqueous	13.5	1.39	29.6	0.04
Hydrolysed	3.4	0.35	7.2	0.01
Non-hydrolysed	10.1	1.03	22.4	0.03
Remaining bound	6.3	0.64	3.0	0.004
Total Extracted	92.4	9.46	101.1	0.10
Total Identified	27.3	2.78	31.0	0.004
R062775	5.2	0.53	ND	ND
R061000	9.3	0.95	ND	ND
R042639	8.4	0.86	14.7	0.02
R060998	0.7	0.07	ND	ND
R110740	2.7	0.27	16.3	0.02
R044085	1.0	0.10		
Total Characterized	51.9	5.24	19.4	0.03
Total Unextracted	6.3	0.64	3.0	0.01
Accountability	98.7	10.2	104.1	0.14

ND: Not subjected to chromatographic analysis

Table 30 Distribution and characterisation/identification of radioactivity in fat and eggs

Component	Fat		Eggs	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Extracted	54.1	0.07	84.8	0.66
Organic phase	39.1	0.05	51.5	0.40
Imazalil R043449 R062775 R044085 R061000 R042639 R110740 Unknowns (no more than 8)	10.7 9.8 ND ND ND ND ND 18.6	0.01 0.01 ND ND ND ND ND 0.02	7.7 1.8 3.2 7.8 5.2 11.1 7.3 7.4	0.06 0.01 0.02 0.06 0.04 0.09 0.06 0.06
Aqueous phase	15.0	0.02	33.3	0.26
Hydrolysed	5.8	0.01	18.1	0.14
R062775 R061000 R060998 Unknowns (no more than 4)	ND ND ND ND	ND ND ND ND	3.4 0.9 0.6 11.6	0.03 0.006 0.001 0.09
Non-hydrolysed	9.2	0.01	15.2	0.12
PES	62.2	0.08	14.7	0.12
Organic	0.0	0.0	4.4	0.03
Aqueous	52.3	0.06	7.2	0.06
Hydrolysed	15.2	0.02	2.5	0.02
Non-hydrolysed	37.1	0.04	4.8	0.04
Remaining bound	9.9	0.01	3.1	0.02
Total Extracted	106.4	0.14	96.4	0.76
Total Identified	20.5	0.02	49.0	0.38
Imazalil	10.7	0.01	7.7	0.06
R062775	ND	ND	6.6	0.05
R061000	ND	ND	6.1	0.05
R042639	ND	ND	11.1	0.09
R060998	ND	ND	0.6	0.001
R110740	ND	ND	7.3	0.06
R043449	9.8	0.01	1.8	0.01
R044085	ND	ND	7.8	0.06

Component	Fat		Eggs	
	% TRR	mg eq/kg	% TRR	mg eq/kg
Total Characterized	18.6	0.02	19.0	1.5
Total Unextracted	9.9	0.01	3.1	0.02
Accountability	116.3	0.15	99.5	0.78

ND: Not subjected to chromatographic analysis

Sixty four percent of the radioactivity in liver was extracted using organic solvents and an additional 13% TRR was released using continuous Soxhlet extraction, leaving 6% TRR bound. Imazalil was not observed in liver. While none of the identified metabolites accounted for greater than 10% TRR, R061000 and R042639 had the highest concentrations, 9% TRR (1.0 mg eq/kg) and 8% TRR (0.90 mg eq/kg), respectively. Neither of the remaining minor metabolites R062775 and R060998 and R110740 and R044085 represented more than 5% TRR (0.5 mg eq/kg). The overall identification of metabolites in liver was low (27% TRR), demonstrating that imazalil was rapidly and extensively metabolized to a large number of metabolites, each accounting for minimal radioactivity.

Similarly, in muscle, 72% TRR was extracted using organic solvents followed by Soxhlet extraction of the PES. Imazalil was also not detected in muscle. However, in contrast with liver, only 2 major metabolites were identified, R042639 (15% TRR) and R110740/R044085, together accounting for 16.3% TRR (0.02 mg eq/kg). A substantial portion of the radioactivity was bound (33% TRR) and while the majority was hydrolysed, no distinct metabolite could be identified in either the organosoluble or aqueous fractions of the hydrolysates.

Only 54% TRR in fat was extracted using organic solvents, with the remaining radioactivity found in the PES. Although these bound residues also underwent a similar hydrolysis to that of liver, all released radioactivity partitioned into the aqueous phase, but no metabolites were identified. Only parent (11% TRR; 0.01 mg eq/kg) and metabolite R043449 were identified (9.8% TRR, 0.01 mg eq/kg).

Higher levels (85% TRR) of the radioactivity in eggs were extracted using organic solvents. Imazalil was detected in eggs (8% TRR; 0.06 mg/kg) as were 7 metabolites R062775, R061000, R042639, R060998 , R110740, R043449 and R044085.Of all the identified metabolites, only R042639 accounted for greater than 10% TRR.

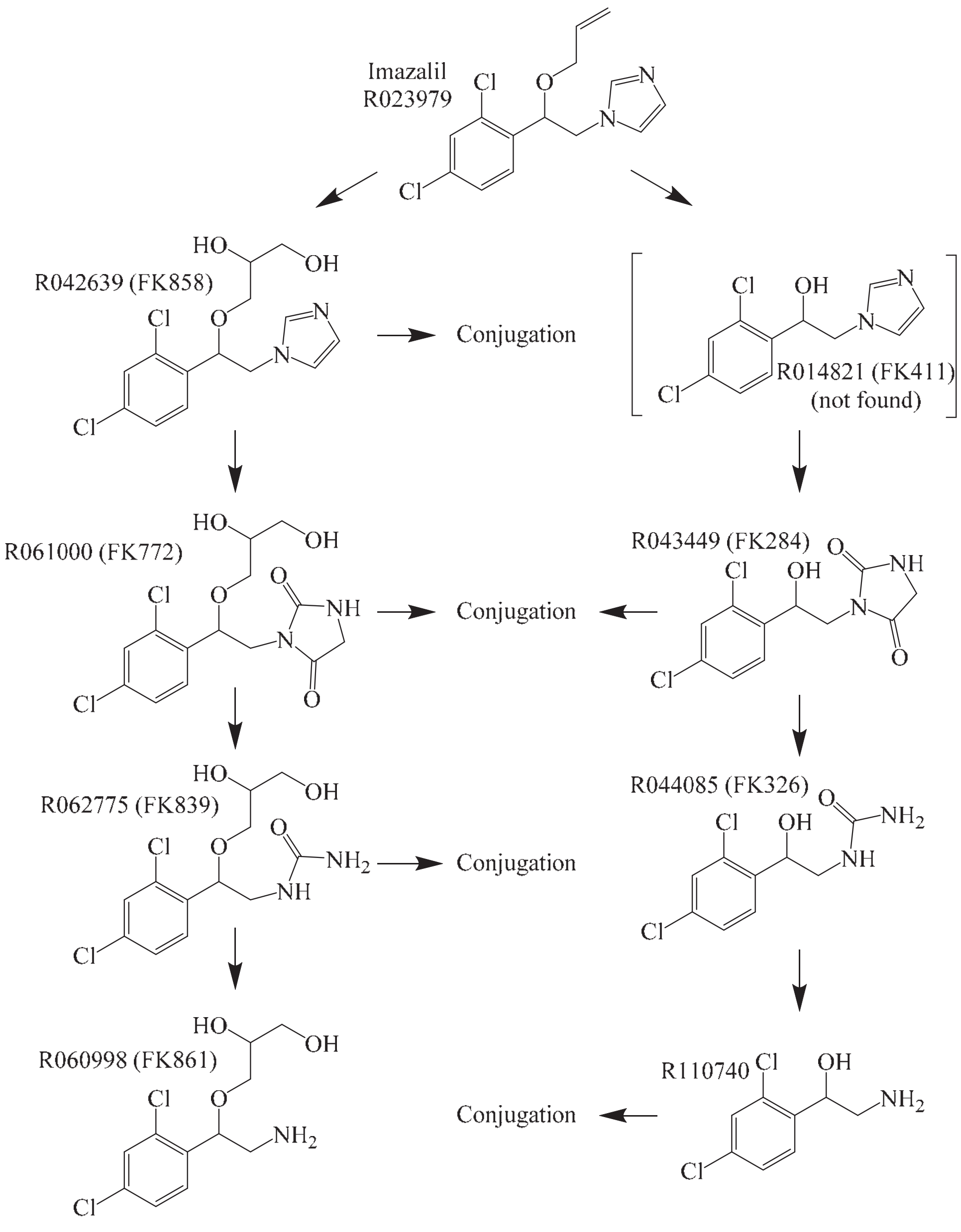


Figure 3 Proposed Metabolic Pathway of Imazalil in Laying Hen

ENVIRONMENTAL FATE IN SOIL

While the Meeting received information on soil aerobic degradation, hydrolysis and photolysis properties of imazalil, studies on the behaviour of [^{14}C]-imazalil in confined rotational crops were not received.

Route of degradation in soil

Aerobic soil degradation

Study 1

A loam soil (from Watervliet, Belgium; $pH_{(KCl)}$ = 7.0, organic matter content = 2.71%) was treated with imazalil sulphate radiolabelled at the 2-ethyl carbon (specific activity: 17.1 µCi/mg) at a rate equivalent to 10 kg ai/ha and incubated at 25 °C for 366 days (Van Leemput, 1984, R 23 979/L8). Analysis was performed using LSC, radio-HPLC and mass spectrometry.

Imazalil was transformed to more polar compounds (extracted metabolites), the majority of which (about 70%) was identified as the metabolite R014821. No other metabolites were identified.

The extracted radioactivity, excluding imazalil, reached a maximum of 5.7% AR after 115 days and declined to 2.5% AR at the end of the incubation period at 366 days, demonstrating no accumulation of radioactivity over time. R014821 did not exceed 10% AR. The concentration of R014821 declined at a rate similar to that of the parent compound.

A large fraction of the AR was mineralized to $^{14}CO_2$ (22% AR), with 32% of the AR transformed to soil bound residues.

Table 31 Distribution of radioactivity, reported in %AR, following 366 days of incubation at 25 °C

Time (days)	Imazalil (%)	Total Extracted (%)	$^{14}CO_2$ (%)	Unextracted (%)	Total (%)
14	86.3	1.1	0.4	7.9	95.7
28	79.1	3.2	1.3	12.7	96.3
62	57.4	5.2	3.9	11.8	78.3
115	45.6	5.7	9.1	16.6	76.8
171	35.6	3.8	13.2	23.7	76.1
222	37.7	—	16.0	25.4	79.2
252	29.8	3.3	17.1	34.5	83.0
366	22.1	2.5	21.6	32.5	78.7

The radioactivity recovered during the experiment was low, typically lower than 80% starting on day 62 until the end of the study.

Imazalil was transformed to three major fractions: the metabolite R014821, CO_2 and unextracted residues. There was no evidence of accumulation of extracted radioactivity, as the concentration was always less than 10% of AR.

Study 2

^{14}C-Imazalil (specific activity; 190 µCi/mg) was applied at a nominal rate of 200 g ai/ha to four soils incubated at 20 °C and one soil at a lower incubation temperature of 10 °C for a period of up to 120 days under aerobic laboratory conditions in the dark (Völkel, 2008, B62717 and Dobson, 2009, B62717a). Soil characteristics are provided in Table 31.

Table 32 Physical, chemical properties of the test soils

Soil Property	Soil I Gartenacker	Soil II Pappelacker	Soil III Fislis	Soil IV 18-Acres
Soil type (USDA)	Silt loam	Sandy loam	Silt loam	Sandy clay loam
Particle size distribution [%][a]				
Sand	35.43	71.77	11.74	48.88
Silt	53.06	21.56	64.04	26.42
Clay	11.51	6.67	24.22	24.70
pH values ($CaCl_2$)	7.18	7.24	7.00	5.90
Organic carbon [%]	2.23	1.23	1.37	3.08
Cation exchange capacity [mval/100 g]	15.07	8.93	18.07	21.66
Maximum water holding capacity [g water/100 g soil]	43.21 [c]	25.01 [c]	26.10 [d]	32.41 [c]
Biomass at start of incubation [mg C/100 g dry soil]	45.8	24.9	27.5	40.4
Biomass at end of incubation [mg C/100g dry soil]	48.1	24.5	35.2 [b]	50.1

[a] soil classification according to USDA soil classification system

[b] value at end of incubation for soil III incubated at 10 °C was '44.1'

[c] water holding capacity at pF 2.0

[d] water holding capacity at pF 2.5

Following application, soils were sampled on days 0, 7, 14, 29, 59, 91 and 120. These were sequentially extracted at room temperature using ethyl acetate (4×), acetonitrile (2×) followed by acetonitrile:water (4:1, v:v) (2×). Extracts were quantified by LSC and TLC analysis. The residual radioactivity remaining in soil after the extraction procedure was quantified by LSC.

Day 120 samples also underwent a harsh extraction procedure under reflux for four hours using acetonitrile:0.1 N HCl (1:1, v:v) to attempt to identify the unextracted residues. The reflux extracts were measured by LSC and analysed by TLC. The residues from the reflux extractions were submitted to organic matter fractionation in order to measure the radioactivity bound to the humic and fulvic acids as well as to the humin fraction of the soil.

TLC was used to quantify the metabolic fractions, while HPLC analysis was used for confirmatory purposes.

Individual recoveries from all soils treated with the test material ranged from 97% to 106% of applied radioactivity (AR). For the four soils incubated at 20 °C, extracted radioactivity decreased during the incubation period, with the fastest rate observed in Soil I. Conversely, unextracted radioactivity increased during the study reaching maximum levels of 39 to 45% of AR. $^{14}CO_2$ also increased steadily during incubation reaching maximums at the end of incubation of 38, 23, 19 and 3% AR for soils I, II, III, and IV, respectively.

In Soil III incubated at 10 °C, a similar distribution of radioactivity was observed where extracted radioactivity decreased to 66% at day 120 with a resulting increase in non-extracted radioactivity and $^{14}CO_2$, reaching maximums of 33 and 3% AR, respectively, at day 120.

Imazalil was found to steadily degrade in soil with half-lives ranging from 46 to 57 days at 20 °C and 113 days at 10ºC, estimated using first order kinetics.

The only radioactive degradation product exceeding 5% of AR was identified as R014821, accounting for up to 10% of AR in the soils.

Harsh extraction of the bound residues showed strong adsorption to the humins and humic acids fractions.

Table 33 Pattern of imazalil degradation and R014821 formation (% AR) following 120 days incubation in the dark at 20 or 10 °C under aerobic conditions

Time (days)	Soil I 20 °C		Soil II 20 °C		Soil III 20 °C		Soil IV 20 °C		Soil III 10 °C	
	Imazalil	R014821	Imazalil	R014821	Imazalil	R014821	Imazalil	R014821	Imazalil	R014821
0	73.1	2.2	93.0	n.d.	89.1	1.0	85.8	1.1	89.1	1.0
7	59.4	3.3	74.1	3.1	74.4	2.8	70.2	2.5	85.5	2.1
14	56.2	3.6	69.7	3.3	67.3	4.1	74.5	3.4	77.4	2.6
29	46.0	4.0	53.5	4.6	54.6	6.1	59.1	5.1	75.7	5.1
59	31.9	3.0	39.2	4.0	37.3	5.5	44.4	8.2	58.7	6.9
91	20.3	2.4	29.9	4.1	27.8	4.9	42.2	8.8	53.8	6.9
120	14.1	2.0	26.1	3.6	21.1	4.8	39.3	9.6	45.4	7.5

Study 3

An aerobic laboratory soil metabolism/degradation study was conducted where imazalil [^{14}C-phenyl-ring] (specific activity; 178 µCi/mg) and imazalil [^{14}C-imidazole-ring] (specific activity: 185 µCi/mg) was applied to a sandy loam soil from Sevelen (Switzerland) (Möndel, 2012, AS223) at an application rate of about 20 µg/100 g dry soil, corresponding to a field application rate of 200 g ai/ha.

Table 34 Characterization of Sevelen test soil

Soil Property	Sevelen [a]
Soil type (USDA)	Sandy loam
Particle size distribution [%]	
Sand (2000-50 µm)	54.63
Silt (<50-2 µm)	36.55
Clay (<2 µm)	8.82
pH values ($CaCl_2$)	7.92
Organic carbon [%]	1.64
Cation exchange capacity [mval/100 g]	8.96

Soil Property	Sevelen [a]
Maximum Water Holding Capacity (MWHC): at pF 1.0 at pF 2.0 at pF 2.5 (projected [b])	 57.29% 36.44% 21.45%
Microbial carbon [mg Cmic/100 g DM] [c] Day 0 Day 120 (with test item and solvent) Day 120 (without test item and solvent) Day 120 (with solvent only)	(two measurements) 22.15 / 55.29 62.06 / 58.35 45.59 / 41.05 60.63 / 59.01

[a] Data according Untersuchungsbericht of AgroLab Swiss GmbH, Labor für Landwirtschaft und Umwelt, Oberfeld 3, 6037 Root, Switzerland, M. Jozic, dated on Nov. 30, 2011

[b] This result was allocated by the monitor and based from a former soil characterization study and was used for moisture adjustment of the soil

[c] Data were determined by the test facility

Soil samples were collected immediately after the application (day 0) and after 2, 7, 15, 30, 58, 90 and 120 days of incubation in the dark at a temperature of 20 °C and at a soil moisture content of about pF 2.5 of the respective soil MWHC.

The soil was sequentially extracted with ethyl acetate (3x), acetonitrile (1x) and acetonitrile:water (4:1, v:v) (1x). Beginning with sampling day 15, a hot reflux extraction with acetonitrile:water (4:1, v:v) was also performed. The combined extracts were submitted to direct LSC and radio-TLC analyses. The unextracted radioactivity was determined by combustion.

The amount of extracted radioactivity decreased continuously over the course of the study in the samples incubated under aerobic conditions and was comparable for both labels. Greater than 100% of AR was extracted on day 0 for both radiolabels. On the last sampling interval (day 120) the extracted radioactivity ranged from 21% AR (phenyl) to 24% AR (imidazole).

The portion of unextracted radioactivity directly after application (day 0) was less than 0.5% AR for both labels, gradually increasing over the study duration, reaching values of 38% AR (phenyl-label) and 30% AR (imidazole-label) by day 120.

High mineralisation was observed, where the amount of $^{14}CO_2$ determined on day 120 ranged from 33% AR (phenyl) to 42% AR (imidazole). The confirmation of $^{14}CO_2$ was done by precipitation. No other volatile compounds were detected.

Table 35 Distribution of radioactivity in Sevelen soil during the incubation period under aerobic conditions

Time [days]	Total Extracted [%]	$^{14}CO_2$ [%]	Unextracted [%]	Total [%]
Phenyl-label				
0	100.89	n.m.	0.49	101.38
2	88.32	0.14	10.11	98.57
7	75.04	0.96	22.11	98.12
15	64.83	5.85	25.62	96.30
30	45.24	16.63	35.18	97.04
58	32.23	24.56	39.86	96.65
90	22.73	31.90	40.93	95.55
120	20.78	33.26	38.36	92.41
Imidazole-label				
0	100.14	n.m.	0.48	100.62
2	88.71	0.52	9.52	98.75
7	77.13	1.90	19.93	98.95
15	70.53	7.40	19.62	97.55
30	54.12	15.87	26.29	96.28
58	36.94	26.04	34.13	97.12
90	28.63	36.19	31.48	96.29
120	23.92	41.93	30.14	95.99

The amount of imazalil steadily decreased with time. Immediately after treatment (day 0), the parent compound represented approximately 100% AR for both radiolabels. On day 120, it accounted for 16% (phenyl) and 20% AR (imidazole).

No radioactive fraction exceeding 5% AR in more than one interval was observed. An unknown fraction exceeded 5% only in one interval reaching an average of 5.8% AR on day 7 (5.6% phenyl and 6.0% imidazole) and subsequent samplings showed a steady decline of the unknown fraction reaching non-detectable levels by day 120, confirming its transient formation. No other unknown fractions exceeded 3% AR.

Metabolite R014821 was detected in only one interval (day 58) of the phenyl-label treated samples and did not exceed 1.2% AR.

Table 36 Pattern of degradation and metabolism of imazalil in the room temperature extracts and harsh reflux extracts of Sevelen soil under aerobic conditions.

		% AR							
Day		0	2	7	15	30	58	90	120
Imazalil	Phenyl	100.3	88.3	68.8	59.5	39.3	26.7	16.2	16.5
	Imidazole	99.6	84.7	70.6	64.9	48.6	33.3	24.9	20.1
R014821	Phenyl	n.d.	n.d.	n.d.	n.d.	n.d.	1.18	n.d.	n.d.
	Imidazole	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
unk-1	Phenyl	0.6	n.d.	0.6	0.5	3	2.1	4.1	1
	Imidazole	0.6	2.2	0.5	0.6	2.2	2.9	2	0.5
unk-2	Phenyl	n.d.	n.d.	5.6	4.8	1.8	n.d.	0.7	n.d.
	Imidazole	n.d.	1.8	6.0	5.0	3.4	0.8	1.7	n.d.
unk-3	Phenyl	n.d.	n.d.	n.d.	n.d.	1.1	1.3	1.2	n.d.
	Imidazole	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
unk-4	Phenyl	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	0.6.	n.d.
	Imidazole	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
unk-5	Phenyl	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d	3.3
	Imidazole	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	3.3

n.d.: Not detected

Using single first order kinetics, the DT_{50} and DT_{90} values for imazalil were 28.1 days and 93.4 days, respectively.

Samples, where unk-2 fraction was found to exceed 5% (from day 7–5.6% in phenyl-label and 6.0% in imidazole-label), were reanalysed in a separate study (Hein, 2013, AS281) and showed levels below 5% of AR. In addition, the unk-2 fraction was analysed separately by TLC and the major fraction corresponded to the metabolite R014821. In re-analysed samples, isolation of the major detected fraction showed that it co-chromatographed either with the parent compound or its metabolite R014821. Therefore, it was concluded that unk-2 corresponds to imazalil or its metabolite R014821 which is probably still strongly bound to the extractable organic matter.

Study 4

The degradation rate of the soil metabolite, R014821, was investigated in three soils (Gartenacker silt loam, Pappelacker sandy loam and Fislis silt loam) under aerobic laboratory conditions at 20 ± 2 °C in the dark (Adam, 2008, AGR3852, B72347).

Table 37 Physical, chemical properties of the test soils

Soil Property	Soil I Gartenacker	Soil II Pappelacker	Soil III Fislis
Soil type (USDA)	Silt loam	Sandy loam	Silt loam
Particle size distribution [%]			
Sand	31.10	71.35	20.60
Silt	58.25	22.34	59.10
Clay	10.65	6.31	20.30
pH values ($CaCl_2$)	7.43	7.58	6.67
Organic carbon [%]	1.72	0.89	1.59
Cation exchange capacity [mval/100 g]	15.14	7.77	20.61
Biomass at start of the test [mg C/100 g dry soil]	36.3	17.9	50.9
Biomass at end of the test [mg C/100g dry soil]	34.1	16.0	43.5

Soils received applications of 0.1976 mg R014821/kg dry soil. Treated soil samples were taken after 0, 1, 3, 7, 14, 33 and 43 days of incubation for soils I and II. Samples at days 60 and 120 were also taken for soil III.

The soil samples were extracted in a shaker with acetonitrile/water (4:1; v/v) four times and subsequently analysed by LC/MS.

R014821 degraded steadily to 7.8, 3.0 and 27.3% in soils I, II and III, respectively after 43 days of incubation. In soil III, the test item was further degraded to 14.0% after 120 days.

Table 38 Amount of R014821 recovered following incubation in the dark at 20 ± 2 °C

Soil	% AR at incubation time [days]								
	0	1	3	7	14	33	43	60	120
Soil I	93.1	84.1	65.3	44.7	21.5	9.8	7.8	NA	NA
Soil II	94.0	92.5	75.4	44.4	22.0	3.9	3.0	NA	NA
Soil III	100.1	94.8	88.1	73.7	57.2	28.3	27.3	25.8	14.0

NA Not applicable

Following first order kinetics, the resulting DT_{50} values at 20 °C for R014821 were 6.9, 6.6 and 22.8 days for soils I, II and III, respectively and corresponding DT_{90} values were 23.0, 22.0 and 75.9 days.

Hydrolysis

Aqueous imazalil solutions of 20 mg/L were incubated at pH 5, 7 and 9 at 25 °C in the dark for periods 0, 20, 41 and 61 days (Van Leemput, 1982, R 23 979/L1).

All of the imazalil was recovered at the end of the incubation period, indicating that hydrolysis is not a route of degradation.

Table 39 Concentration of imazalil recovered from aqueous solutions buffered at various pH values after incubation at 25 °C at different time points

Time (days)	Sample	pH	Imazalil (µg/mL)	mean	pH	Imazalil (µg/mL)	Mean	pH	Imazalil (µg/mL)	mean
0	I	4.88	20.6	19.6	6.87	18.6	18.5	8.96	18.0	17.7
	II	4.89	18.6		6.87	18.4		8.97	17.4	
20	I	4.89	18.0	19.1	6.89	17.7	18.4	8.99	19.9	18.2
	II	4.89	20.2		6.89	19.0		9.00	16.4	
41	I	4.89	18.7	20.3	6.91	17.8	18.8	9.01	19.1	17.4
	II	4.89	21.9		6.90	19.8		9.00	15.6	
61	I	4.89	21.9	20.5	6.91	19.3	19.8	9.00	17.4	17.9
	II	4.89	19.0		6.92	20.2		9.01	18.4	

Photolysis - Aqueous

Duplicate samples of sterile buffer solution at pH 7 treated with imazalil [^{14}C-phenyl-ring] (specific activity; 151 µCi/mg) at a mean initial concentration of about 0.15 mg/L were irradiated for a continuous period of 19 days (Mégel, 2009, AGR 3856). The 19-day continuous irradiation time corresponded to 35 days of midsummer sunlight at latitudes 30-40°N and 36 days of midsummer sunlight at latitude 50°N. Concurrently, a sample of the treated test solution was incubated under the same conditions but in the dark. The irradiated and dark control samples were maintained at a mean temperature of 25 °C throughout the study. Volatile traps consisting of ethylene glycol and sodium hydroxide solutions were set-up to determine the formation of organic volatile compounds and radioactive carbon dioxide, respectively.

Aliquots were taken from the test solutions at 0, 3, 5, 7, 11 and 19 days during the irradiation/incubation period and directly submitted to radiochemical quantification by LSC and chromatographic analysis by HPLC and confirmed by TLC on selected samples. In addition, LC/MS was performed on regenerated sample to identify the photodegradates.

Total mean recoveries were 99% of the applied radioactivity (AR) for the irradiated samples and 101% AR for the dark control.

^{14}C-Imazalil underwent continuous photolysis, decreasing to 44% AR within 7 days of irradiation. By the end of the study, it had declined to 5% AR. In the dark control samples, virtually no photolysis of the test item was observed, on day 19 it still accounted for 98 % AR.

Four photodegradates accounted for more than 10 % AR: R044177, R044179, R055609 and R018238. R044179 and R055609 were the most significant radioactive residues, reaching up to 20% AR and 18% AR on day 19, respectively. R044177 reached its maximum on day 5 (12 % AR) then decreased to 7% AR on day 19.

The photodegradate R018238 was formed much later (from day 7 onwards) and accounted for 10% AR on day 19. All other unidentified radioactive fractions did not individually exceed 10 % AR at any time point.

The formation of radioactive carbon dioxide was insignificant for both the irradiated and dark control samples, not accounting for more than 2% and 0.1% AR, respectively, throughout the study. Not more than 0.1% AR was detected in the ethylene glycol traps of either the irradiated or dark control samples.

The rate of photodegradation of ^{14}C-Imazalil was described using simple first order kinetics and are given in the table below:

Calculated Half-life (DT_{50}) and DT_{90} in Days					
Continuous irradiation		Sunlight 30-40ºN		Sunlight 50ºN	
DT_{50}	DT_{90}	DT_{50}	DT_{90}	DT_{50}	DT_{90}
6.1	20.2	11.1	37.0	11.6	38.6

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received descriptions and validation data for several analytical methods for residues of imazalil and its metabolite R014821 in diverse plant matrices, which were determined to be suitable.

Briefly, most methods involved extraction of the homogenized plant samples with a mixture of organic solvents, to which was added, in many cases, an internal standard. Sodium chloride was also added to many of the homogenates, prior to extraction. Typically, clean-up was performed using liquid-liquid partition or solid phase extraction. Quantification of the residues of imazalil and the alcohol metabolite R014821 was achieved using various detectors.

Recovery and repeatability data for determination of imazalil and R014821residues in crops are presented in Table 40. The average recoveries for each of the analytes was between 70-120% with %RSDs at different fortification levels in different matrices below 20%. The Meeting did not receive any information on radiovalidation.

Table 40 Summary of Plant Analytical Methods

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
Gent 53/3b (Woestenberghs. 1988)	Imazalil	GC/ECD (with internal standard)	heptane:isoamyl alcohol (95:5, v:v)	Citrus whole fruit	0.05	0.05 (2) 0.27 (2) 1.0 (12) 4 (2)	101% 112% 75%, RSD:20% 102%
LOA/SOP/6006.1 (Vanderlinden, 2001, updated 2002, updated 2003)	Imazalil	GC/MS	ethyl acetate/n-hexane (50:50, v:v) and sodium chloride	Lemon whole fruit	0.05	0.05 (6) 0.5 (6) 5 (6)	96%, RSD: 12% 79%, RSD: 9% 88%, RSD: 6%
JAN-0804V (ILV of LOA/SOP/ 6006.1) (Schernikau, 2008)	Imazalil	GC/MS (with internal standard) m/z 215 (quantification) m/z 173 (confirmation) m/z 217 (confirmation)	ethyl acetate/n-hexane (50:50, v:v) and sodium chloride	Orange whole fruit	0.05	Quantitation	
						0.05 (5)	96%, RSD:5%
						0.50 (5)	86%, RSD:13%
						Confirmation	
						0.05 (5)	94%, RSD:5%
						0.50 (5)	88%, RSD:15%
						Confirmation	
						0.05 (5)	94%, RSD:6%
						0.50 (5)	86%, RSD:15%
				Apple whole fruit	0.05	Quantitation	
						0.05 (5)	99%, RSD:2%
						0.50 (5)	104%, RSD:1%
						Confirmation	
						0.05 (5)	101%, RSD:5%
						0.50 (5)	101%, RSD:1%
						Confirmation	

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
						0.05 (5)	99%, RSD:5%
						0.50 (5)	105%, RSD:2%
R23979/AGR 18 (Woestenborghs, 1992)	Imazalil,	GC-ECD (with internal standard)	heptane/isoamyl alcohol mixture (95:5; v/v)	Citrus whole fruit	0.05	0.05 (1) 0.10 (1) 0.20 (2) 0.50 (2) 1.0 (2) 2.0 (2) 5.0 (2) 10.0 (1) 20.0 (1)	102% 101% 101% 96% 100% 103% 99% 108% 95%
				Citrus chopped peel	0.50	0.50 (1) 1.0 (1) 2.5 (1) 5.0 (1) 10.0 (1) 25.0 (1) 50.0 (1)	102% 104% 94% 94% 101% 109% 97%
				Citrus pulp	0.05	0.05 (1) 0.10 (1) 0.20 (1) 0.40 (1) 1.0 (1) 2.0 (1) 5.0 (1)	104% 99% 98% 94% 103% 107% 97%
				Juice	0.05	0.05 (1) 0.10 (1) 0.20 (1) 0.40 (1) 1.0 (1) 2.0 (1) 5.0 (1)	107% 98% 99% 92% 96% 107% 102%
				Molasses	0.05	0.05 (1) 0.10 (1) 0.20 (2) 0.40 (2) 1.0 (2) 2.0 (2) 5.0 (2) 10.0 (1) 20.0 (1)	106% 99% 101% 96% 97% 103% 101% 112% 92%
				Pressed oil	0.05	0.05 (1) 0.10 (1) 0.20 (1) 0.40 (1) 1.0 (1) 2.0 (2) 4.0 (1) 5.0 (1) 10.0 (1) 20.0 (1) 50.0 (1) 100.0 (1) 200.0 (1)	95% 110% 92% 103% 100% 107% 97% 94% 96% 99% 96% 104% 102%
	R014821			Citrus whole fruit	0.02	0.02 (1) 0.05 (2) 0.10 (2) 0.20 (2) 0.50 (2) 1.0 (1) 2.0 (1)	102% 101% 100% 96% 100% 99% 104%

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
				Citrus chopped peel	0.02	0.25 (1) 0.50 (1) 1.0 (1) 2.50 (1) 3.0 (1)	90% 102% 104% 99% 99%
				Citrus pulp	0.02	0.02 (1) 0.04 (1) 0.10 (1) 0.20 (1) 0.50 (1)	102% 96% 102% 98% 101%
				Juice	0.02	0.02 (1) 0.04 (1) 0.10 (1) 0.20 (1) 0.50 (1)	100% 98% 103% 100% 99%
				Molasses	0.02	0.02 (1) 0.04 (2) 0.10 (2) 0.20 (2) 0.50 (2) 1.0 (1) 2.0 (1)	105% 102% 94% 98% 101% 106% 100%
				Pressed oil	0.02	0.02 (1) 0.04(1) 0.10 (2) 0.20 (2) 0.50 (2) 1.0 (1) 2.0 (1) 5.0 (1)	103% 98% 101% 99% 100% 99% 100% 102%
MAK 513/984694 (Todd, 1999)	Imazalil	HPLC-UV (204 nm)	Pulp, peel, dry pomace, marmalade: hexane:acetone (90:10;v:v), sodium sulphate, sodium chloride, celite and sodium hydroxide (0.5M) Juice: sodium chloride and hydrochloric acid (0.5 M)	Orange pulp	0.02	0.02 (5) 0.10 (5) 0.50 (5)	89%, RSD: 8% 86%, RSD: 6% 82%, RSD:6%
				Orange peel	0.05	0.05 (5) 0.25 (5) 1.25 (5)	89%, RSD: 8% 80%, RSD: 8% 95%, RSD: 9%
				Orange dry pomace	0.02	0.02 (5) 0.10 (5) 0.50 (5)	79%, RSD: 10% 79%, RSD: 5% 83%, RSD: 6%
				Orange juice	0.02	0.02 (5) 0.10 (5) 0.50 (5)	83%, RSD: 6% 99%, RSD: 3% 102%, RSD: 1%
				Orange marmalade	0.02	0.02 (5) 0.10 (5) 0.50 (5)	96%, RSD: 4% 96%, RSD: 3% 92%, RSD: 3%
OA01287 (Hubbard, 2006)	Imazalil	GC-MSD	heptane:isoamyl alcohol (95:5; v/v) and sodium chloride	Potato tubers	0.017	0.017 (5) 0.17 (5)	82%; RSD:6% 86%, RSD: 3%
20054080/E1-FPWW (Fillion *et al*. 2000 (modified)) (Pollman, 2006)	Imazalil	HPLC-MS/MS m/z 299→161	acetonitrile:water	Wheat grain	0.01	0.01 (3) 0.1 (3) 1 (2) 50 (2)	95%, RSD: 3% 93%, RSD: 5% 88% 89%
				Wheat straw	0.02	0.02 (3) 0.2 (3)	98%, RSD: 6% 92%, RSD: 8%
				Wheat immature plant	0.02	0.02 (3) 0.2 (3)	85%, RSD: 14% 90%, RSD: 13%
L-00.00-34 - formerly DFG S19 (GAC-0901V) (Lindner, 2009)	Imazalil	HPLC-MS/MS m/z 297→159 (quantification) m/z 297→201	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Lemon	0.01	Quantitation	
						0.01 (5)	110%, RSD:8%
						0.10 (5)	96%, RSD:3%
						Confirmation	

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
		(confirmation)				0.01 (5)	109%, RSD:7%
						0.10 (5)	96%, RSD:3%
L-00.00-115 – QuECHERS SX/10/003 R-27563 (Andrews, 2010)	Imazalil	HPLC-MS/MS (with internal standard) m/z 297→41 (quantification) m/z 297→159 (confirmation)	acetonitrile/water (2:1; v/v)	Citrus fruit	0.01	0.01 (5)	109%, RSD: 3%
						0.10 (5)	106%, RSD:5%
				Citrus peel	0.01	0.01 (5)	106%, RSD: 8%
						0.10 (5)	94%, RSD:6%
				Citrus pulp	0.01	0.01 (5)	97%, RSD: 4%
						0.10 (5)	98%, RSD:7%
						Note: Only recoveries from 1st ion transition reported.	
ILV of SX/10/003 S11-03249 R-29367 (Mende, 2011)	Imazalil	HPLC-MS/MS (with internal standard) m/z 297→41 (quantification) m/z 297→159 (confirmation)	acetonitrile/water (2:1; v/v)	Orange pulp	0.01	Quantitation	
						0.01 (5)	103%, RSD: 8%
						0.10 (5)	100%, RSD:7%
						Confirmation	
						0.01 (5)	104%, RSD: 3%
						0.10 (5)	100%, RSD:2%
				Orange peel	0.01	Quantitation	
						0.01 (5)	95%, RSD: 9%
						0.10 (5)	99%, RSD: 6%
						Confirmation	
						0.01 (5)	98%, RSD: 4%
						0.10 (5)	98%, RSD: 2%
L-00.00-115 – QuECHERS SX/10/026 R-27564 (Andrews, 2011)	R014821	HPLC-MS/MS (with internal standard) m/z 257→69 (quantification) m/z 257→125 (confirmation)	acetonitrile/water (2:1; v/v)	Orange whole fruit	0.01	Quantitation	
						0.01 (6)	82%, RSD: 6%
						0.10 (5)	75%, RSD: 4%
						Confirmation	
						0.01 (6)	88%, RSD: 6%
						0.10 (5)	73%, RSD: 15%
				Orange peel	0.01	Quantitation	
						0.01 (5)	63%, RSD: 8%
						0.10 (5)	64%, RSD: 9%
						Confirmation	
						0.01 (5)	78%, RSD: 6%
						0.10 (5)	72%, RSD: 9%
				Orange pulp	0.01	Quantitation	
						0.01 (5)	82%, RSD: 10%
						0.10 (5)	83%, RSD: 6%
						Confirmation	
						0.01 (5)	85%, RSD: 12%
						0.10 (5)	83%, RSD: 9%
L-00.00-115 – QuECHERS SX/11/016, R-29511 (Austin, 2013)	Imazalil	HPLC-MS/MS m/z 297→41 OR m/z 297→255 (quantitation) m/z 297→159 (confirmation)	acetonitrile:water (2:1; v:v)	Orange marmalade	0.01	Quantitation	
						0.01 (5)	104%, RSD: 2%
						0.1 (5)	105%, RSD: 2%
						Confirmation	
						0.01 (5)	103%, RSD: 1%
						0.1 (5)	99%, RSD: 1%
				Orange essential oil	0.01	Quantitation	
						0.01 (5)	99%, RSD: 2%
						0.1 (5)	102%, RSD: 2%
						Confirmation	
						0.01 (5)	105%, RSD: 7%
						0.1 (5)	101%, RSD: 4%
				Orange dried slices	0.01	Quantitation	
						0.01 (5)	104%, RSD: 5%
						0.1 (5)	97%, RSD: 12%
						Confirmation	

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
						0.01 (5)	99%, RSD: 4%
						0.1 (5)	91%, RSD: 8%
				Orange wet pomace	0.01	Quantitation	
						0.01 (3)	100%, RSD: 1%
						0.1 (3)	101%, RSD: 1%
						Confirmation	
						0.01 (3)	86%, RSD: 2%
						0.1 (3)	87%, RSD: 1%
				Orange dry pomace	0.01	Quantitation	
						0.01 (3)	102%, RSD: 2%
						0.1 (3)	95%, RSD: 1%
						Confirmation	
						0.01 (3)	92%, RSD: 8%
						0.1 (3)	90%, RSD: 3%
				Orange juice	0.01	Quantitation	
						0.01 (3)	103%, RSD: 1%
						0.1 (3)	106%, RSD: 1%
						Confirmation	
						0.01 (3)	104%, RSD: 2%
						0.1 (3)	101%, RSD: 1%
				Orange (canned)	0.01	Quantitation	
						0.01 (3)	99%, RSD: 2%
						0.1 (3)	102%, RSD: 1%
						Confirmation	
						0.01 (3)	100%, RSD: 3%
						0.1 (3)	100%, RSD: 1%
				Orange jam	0.01	Quantitation	
						0.01 (3)	101%, RSD: 1%
						0.1 (3)	101%, RSD: 2%
						Confirmation	
						0.01 (3)	101%, RSD: 2%
						0.1 (3)	101%, RSD: 0%
				Orange jelly	0.01	Quantitation	
						0.01 (3)	103%, RSD: 1%
						0.1 (3)	101%, RSD: 2%
						Confirmation	
						0.01 (3)	103%, RSD: 2%
						0.1 (3)	100%, RSD: 2%
				Orange peel	0.01	Quantitation	
						0.01 (3)	107%, RSD: 1%
						0.1 (3)	103%, RSD: 6%
						Confirmation	
						0.01 (3)	106%, RSD: 3%
						0.1 (3)	103%, RSD: 4%
L-00.00-115 – QuECHERS SX/11/017, R-29512	R014821	HPLC-MS/MS m/z 257→69 (quantitation) m/z 257→125 (confirmation)	acetonitrile:water (2:1; v:v)	Orange marmalade	0.01	Quantitation	
						0.01 (5)	100%, RSD: 5%
						0.1 (5)	88%, RSD: 4%
						Confirmation	
						0.01 (5)	101%, RSD: 4%
						0.1 (5)	94%, RSD: 8%
				Orange essential oil	0.01	Quantitation	
						0.01 (5)	90%, RSD: 13%
						0.1 (5)	92%, RSD: 8%
						Confirmation	
						0.01 (5)	92%, RSD: 8%
						0.1 (5)	90%, RSD: 10%
				Orange dried slices	0.01	Quantitation	
						0.01 (5)	88%, RSD: 11%
						0.1 (5)	86%, RSD: 7%

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
						Confirmation	
						0.01 (5)	86%, RSD: 10%
						0.1 (5)	84%, RSD: 12%
				Orange wet pomace	0.01	Quantitation	
						0.01 (3)	107%, RSD: 1%
						0.1 (3)	99%, RSD: 7%
						Confirmation	
						0.01 (3)	107%, RSD: 1%
						0.1 (3)	103%, RSD: 3%
				Orange dry pomace	0.01	Quantitation	
						0.01 (3)	107%, RSD: 3%
						0.1 (3)	95%, RSD: 7%
						Confirmation	
						0.01 (3)	100%, RSD: 4%
						0.1 (3)	89%, RSD: 5%
				Orange juice	0.01	Quantitation	
						0.01 (3)	93%, RSD: 8%
						0.1 (3)	93%, RSD: 4%
						Confirmation	
						0.01 (3)	87%, RSD: 8%
						0.1 (3)	90%, RSD: 6%
				Orange (canned)	0.01	Quantitation	
						0.01 (3)	85%, RSD: 4%
						0.1 (3)	95%, RSD: 6%
						Confirmation	
						0.01 (3)	89%, RSD: 12%
						0.1 (3)	93%, RSD: 5%
				Orange jam	0.01	Quantitation	
						0.01 (3)	77%, RSD: 7%
						0.1 (3)	72%, RSD: 2%
						Confirmation	
						0.01 (3)	73%, RSD: 2%
						0.1 (3)	72%, RSD: 1%
				Orange jelly	0.01	Quantitation	
						0.01 (3)	84%, RSD: 6%
						0.1 (3)	83%, RSD: 10%
						Confirmation	
						0.01 (3)	80%, RSD: 9%
						0.1 (3)	81%, RSD: 7%
				Orange peel	0.01	Quantitation	
						0.01 (3)	88%, RSD: 2%
						0.1 (3)	92%, RSD: 4%
						Confirmation	
						0.01 (3)	89%, RSD: 1%
						0.1 (3)	87%, RSD: 2%
L-00.00-115 – QuECHERS S16-06757, R-38419, AGR 5515 (Grote, 2017)	Imazalil and R014821	HPLC-MS/MS Imazalil m/z 297→159 (quantitation) m/z 297→201 (confirmation) R014821 m/z 257→125 (quantitation) m/z 257→69 (confirmation)	acetonitrile	Orange whole fruit	0.01/ analyte	Imazalil	
						Quantitation	
						0.01 (5)	105%, RSD: 8%
						0.1 (4)	104%, RSD: 4%
						Confirmation	
						0.01 (5)	103%,RSD:11%
						0.1 (4)	109%, RSD: 7%
						R014821	
						Quantitation	
						0.01 (5)	89%, RSD: 6%
						0.1 (4)	82%, RSD: 4%
						Confirmation	
						0.01 (5)	98%, RSD: 6%
						0.1 (4)	93%, RSD: 6%

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
				Orange pulp	0.01/ analyte	Imazalil	
						Quantitation	
						0.01 (5)	103%, RSD: 5%
						0.1 (4)	98%, RSD: 2%
						Confirmation	
						0.01 (5)	108%, RSD:7%
						0.1 (4)	96%, RSD: 4%
						R014821	
						Quantitation	
						0.01 (5)	93%, RSD: 9%
						0.1 (4)	84%, RSD: 2%
						Confirmation	
						0.01 (5)	97%, RSD: 3%
						0.1 (4)	91%, RSD: 3%
L-00.00-115 – QuECHERS S16-06758,	Imazalil and R014821	HPLC-MS/MS Imazalil m/z 297→159 (quantitation) m/z 297→201 (confirmation) R014821 m/z 257→125 (quantitation) m/z 257→69 (confirmation)	acetonitrile	Lemon whole fruit	0.01/ analyte	Imazalil	
						Quantitation	
						0.01 (3)	106%, RSD: 4%
						0.1 (3)	95%, RSD: 1%
						Confirmation	
						0.01 (3)	102%, RSD: 7%
						0.1 (3)	94%, RSD: 1%
						R014821	
						Quantitation	
						0.01 (3)	94%, RSD: 4%
						0.1 (3)	88%, RSD: 9%
						Confirmation	
						0.01 (3)	86%, RSD:4%
						0.1 (3)	82%, RSD: 5%
				Lemon pulp	0.01 / analyte	Imazalil	
						Quantitation	
						0.01 (3)	98%, RSD: 4%
						0.1 (3)	103%, RSD: 2%
						Confirmation	
						0.01 (3)	98%, RSD: 5%
						0.1 (3)	107%, RSD: 5%
						R014821	
						Quantitation	
						0.01 (3)	89%, RSD: 6%
						0.1 (3)	99%, RSD: 2%
						Confirmation	
						0.01 (3)	92%, RSD:8%
						0.1 (3)	96%, RSD: 2%
L-00.00-34 TRC13-058 R-33728 AGR 4977 (Hamberger, 2013)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation 1) m/z 299→161 (confirmation 2)	acetone and ethyl acetate:cyclohexane (1:1, v:v)	Banana whole fruit	0.01	Quantitation	
						0.01 (3)	86%, RSD:10%
						5.0 (3)	92%, RSD: 8%
						Confirmation	
						0.01 (3)	82%, RSD: 12%
						5.0 (3)	93%, RSD: 9%
						Confirmation	
						0.01 (3)	86%, RSD: 27%
						5.0 (3)	94%, RSD: 7%
				Banana peel	0.01	Quantitation	
						0.01 (5)	100%,RSD:10%
						10.0 (5)	86%, RSD: 10%
						Confirmation	
						0.01 (5)	92%, RSD: 8%
						10.0 (5)	88%, RSD: 8%
						Confirmation	
						0.01 (5)	95%, RSD: 10%

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
						10.0 (5)	86%, RSD: 9%
				Banana pulp	0.01	Quantitation	
						0.01 (6)	103%, RSD: 3%
						0.10 (3)	106%, RSD: 2%
						2.0 (6)	99%, RSD:3%
						Confirmation	
						0.01 (6)	93%, RSD: 14%
						0.10 (3)	107%, RSD: 4%
						2.0 (6)	98%, RSD: 9%
						Confirmation	
						0.01 (6)	95%, RSD: 13%
						0.10 (3)	101%, RSD: 3%
						2.0 (6)	96%, RSD: 10%
L-00.00-34 GAB-08103V (Lindner, 2009)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Cucumber	0.01	Quantitation	
						0.01 (5)	86%, RSD: 3%
						0.10 (5)	84%, RSD: 6%
						Confirmation	
						0.01 (5)	83%, RSD: 5%
						0.10 (5)	83%, RSD: 5%
L-00.00-34 S12-00922 (Fischer, 2013)	Imazalil and R014821	HPLC-MS/MS Imazalil m/z 297→159 (quantitation) m/z 297→201 (confirmation) R014821 m/z 257→69 (quantitation) m/z 257→125 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Cucumber	0.01/ analyte	Imazalil	
						Quantitation	
						0.01 (5)	72%, RSD: 7%
						0.10 (5)	75%, RSD: 2%
						Confirmation	
						0.01 (5)	76%, RSD: 6%
						0.10 (5)	79%, RSD: 4%
						R014821	
						Quantitation	
						0.01 (5)	74%, RSD: 7%
						0.10 (5)	72%, RSD: 2%
						Confirmation	
						0.01 (5)	74%, RSD: 6%
						0.10 (5)	75%, RSD: 4%
L-00.00-34 CET-0703V (Rotzoll, 2007)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Tomato	0.01	Quantitation	
						0.01 (5)	95%, RSD: 6%
						0.50 (5)	87%, RSD: 5%
						Confirmation	
						0.01 (5)	93%, RSD: 7%
						0.50 (5)	84%, RSD: 7%
08C01019-01-VMTO (ILV of CET-0703V) (Hamberger. 2008)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Tomato	0.01	Quantitation	
						0.01 (5)	84%, RSD: 4%
						0.50 (5)	78%, RSD: 8%
						Confirmation	
						0.01 (5)	81%, RSD: 5%
						0.50 (5)	76%, RSD: 7%
L-00.00-34 CET-0702V (Rotzoll, 2007)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Potato	0.01	Quantitation	
						0.01 (5)	101%, RSD: 4%
						0.20 (5)	97%, RSD: 4%
						5.0 (5)	98%, RSD: 7%
						Confirmation	
						0.01 (5)	101%, RSD: 4%
						0.20 (5)	99%, RSD: 4%
						5.0 (5)	96%, RSD: 6%
08C01019-01-VMPO (ILV of CET-0702V) (Hamberger. 2008)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Potato	0.01	Quantitation	
						0.01 (5)	91%, RSD: 2%
						0.20 (5)	79%, RSD: 3%
						5.0 (5)	79%, RSD: 5%
						Confirmation	
						0.01 (5)	93%, RSD: 2%

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
						0.20 (5)	78%, RSD: 2%
						5.0 (5)	79%, RSD: 4%
L-00.00-34 11G04031-01-VMPO (Hamberger, 2011)	R014821	HPLC-MS/MS m/z 257→69 (quantitation) m/z 257→125 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Potato	0.01	Quantitation	
						0.01 (5)	81%, RSD: 8%
						0.10 (5)	76%, RSD: 4%
						3.0 (5)	73%, RSD: 9%
						Confirmation	
						0.01 (5)	83%, RSD: 5%
						0.10 (5)	76%, RSD: 3%
						3.0 (5)	74%, RSD: 9%
S11-03790 (ILV of 11G04031-01-VMPO) (Mende, 2012)	R014821	HPLC-MS/MS m/z 257→69 (quantitation) m/z 257→125 (confirmation)	acetone, ethyl acetate:cyclohexane (1:1, v:v) and sodium chloride	Potato	0.01	Quantitation	
						0.01 (5)	86%, RSD: 6%
						0.10 (5)	80%, RSD: 6%
						3.0 (5)	74%, RSD: 7%
						Confirmation	
						0.01 (5)	85%, RSD: 6%
						0.10 (5)	80%, RSD: 6%
						3.0 (5)	75%, RSD: 5%
L-00.00-115 - QuECHERS D55142 (Zeisler, 2013)	Imazalil	HPLC-MS/MS m/z 297→255 (quantitation) m/z 297→159 (confirmation)	acetonitrile	Potato tuber	0.01	Quantitation	
						0.01 (5)	86%, RSD: 5%
						0.10 (5)	90%, RSD: 3%
						Confirmation	
						0.01 (5)	91%, RSD: 3%
						0.10 (5)	91%, RSD: 2%
				Potato oil	0.01	Quantitation	
						0.01 (5)	101%, RSD: 2%
						0.10 (5)	95%, RSD: 2%
						Confirmation	
						0.01 (5)	99%, RSD: 2%
						0.10 (5)	96%, RSD: 2%
				Potato flakes	0.01	Quantitation	
						0.01 (5)	93%, RSD: 7%
						0.10 (5)	86%, RSD: 1%
						Confirmation	
						0.01 (5)	91%, RSD: 3%
						0.10 (5)	87%, RSD: 2%
				Potato fries	0.01	Quantitation	
						0.01 (5)	108%, RSD: 2%
						0.10 (5)	106%, RSD: 3%
						Confirmation	
						0.01 (5)	106%, RSD: 3%
						0.10 (5)	105%, RSD: 6%
				Potato crisps	0.01	Quantitation	
						0.01 (5)	107%, RSD: 5%
						0.10 (5)	107%, RSD: 5%
						Confirmation	
						0.01 (5)	109%, RSD: 3%
						0.10 (5)	108%, RSD: 4%
L-00.00-115 - QuECHERS D55164 (Zeisler, 2013)	R014821	HPLC-MS/MS m/z 257→69 (quantitation) m/z 257→125 (confirmation)	acetonitrile	Potato tuber	0.01	Quantitation	
						0.01 (5)	77%, RSD: 5%
						0.10 (5)	86%, RSD: 3%
						Confirmation	
						0.01 (5)	80%, RSD: 5%
						0.10 (5)	84%, RSD: 6%
				Potato oil	0.01	Quantitation	
						0.01 (5)	90%, RSD: 6%
						0.10 (5)	94%, RSD: 2%
						Confirmation	
						0.01 (5)	90%, RSD: 10%

Method Identification (Reference)	Analyte	Method Type	Extraction solvents	Commodity	LOQ (mg/kg)	Fortification level in mg/kg (no. of samples)	Mean recovery (%); Relative standard deviation (RSD)
						0.10 (5)	96%, RSD: 3%
				Potato flakes	0.01	Quantitation	
						0.01 (5)	91%, RSD: 8%
						0.10 (5)	90%, RSD: 2%
						Confirmation	
						0.01 (5)	94%, RSD: 3%
						0.10 (5)	90%, RSD: 3%
				Potato fries	0.01	Quantitation	
						0.01 (5)	77%, RSD: 5%
						0.10 (5)	72%, RSD: 5%
						Confirmation	
						0.01 (5)	78%, RSD: 6%
						0.10 (5)	73%, RSD: 4%
				Potato crisps	0.01	Quantitation	
						0.01 (5)	79%, RSD: 6%
						0.10 (5)	77%, RSD: 3%
						Confirmation	
						0.01 (5)	84%, RSD: 8%
						0.10 (5)	76%, RSD: 4%
L-00.00-34 CET-0701V (Rotzoll, 2007)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation)	ethyl acetate:cyclohexane (1:1, v:v)	Cereal grain	0.01	Quantitation	
						0.01 (5)	85%, RSD: 6%
						0.1 (5)	79%, RSD: 8%
						Confirmation	
						0.01 (5)	88%, RSD: 4%
						0.1 (5)	80%, RSD: 7%
				Cereal straw	0.01	Quantitation	
						0.01 (5)	88%, RSD: 6%
						0.1 (5)	84%, RSD: 7%
						Confirmation	
						0.01 (5)	92%, RSD: 4%
						0.1 (5)	82%, RSD: 7%
IF-09/01310918 (ILV of CET-0701V) (Knoch, 2009)	Imazalil	HPLC-MS/MS m/z 297→159 (quantitation) m/z 297→201 (confirmation)	ethyl acetate:cyclohexane (1:1, v:v)	Wheat grain	0.01	Quantitation	
						0.01 (5)	84%, RSD: 4%
						0.1 (5)	79%, RSD: 6%
						Confirmation	
						0.01 (5)	88%, RSD: 3%
						0.1 (5)	80%, RSD: 5%
				Barley straw	0.01	Quantitation	
						0.01 (5)	92%, RSD: 16%
						0.1 (5)	78%, RSD: 18%
						Confirmation	
						0.01 (5)	108%,RSD:15%
						0.1 (5)	102%,RSD:20%

The Meeting received descriptions and validation data for several analytical methods for residues of

imazalil and its metabolites R043449 and R061000 in bovine matrices and imazalil, R042639, R044085 and R110740 in poultry matrices, which were determined to be suitable.

Briefly, most methods involved extraction of the homogenized tissue samples, milk or eggs with either methanol, acetonitrile and/or acetone Typically, clean-up was performed using either, liquid-liquid partitioning with hexane or solid phase extraction on an RP8 cartridge. Quantification of the residues of imazalil and the metabolites was achieved using either GC-ECD (which included derivatization prior to analysis) or LC-MS/MS.

Recovery and repeatability data for determination of imazalil and the metabolite residues in tissues, milk and eggs are presented in Table 40. With the exception of a few recoveries and %RSD, most average recoveries for each of the analytes were between 70-120% with %RSDs <20% at different fortification levels in different matrices.

Table 41 Summary of Animal Analytical Methods

Method Identification (Reference)	Method Type	Extraction Solvents	Commodity	Analyte	Fortification Levels (mg /kg)	n	Mean Recovery %	Relative Standard Deviation (RSD) %
651554 (Van Dijk, 1997)	GC-ECD	Muscle, liver – methanol Kidney – methanol and acetonitrile Fat – methanol, acetone, acetonitrile Milk - acetonitrile and methanol	Bovine (muscle)	Imazalil	0.02	6	84	18
					0.04	7	94	24
					0.1	7	105	17
				R061000	0.02	6	88	11
					0.04	8	103	12
					0.1	6	98	16
				R043449	0.02	6	70	7
					0.04	8	87	21
					0.1	6	95	16
			Bovine (kidney)	Imazalil	0.02	7	89	24
					0.04	6	91	23
					0.1	6	86	9
				R061000	0.02	5	87	7
					0.04	5	83	9
					0.1	6	80	13
				R043449	0.02	6	97	17
					0.04	7	88	19
					0.1	6	83	13
			Bovine (liver)	Imazalil	0.05	4	95	16
					0.1	6	85	21
					0.25	6	107	10
				R061000	0.05	6	95	14
					0.1	6	99	17
					0.25	6	104	12
				R043449	0.05	6	75	21
					0.1	6	89	15
					0.25	6	86	5
			Bovine (fat)	Imazalil	0.02	4	86	21
					0.04	8	88	13
					0.1	8	96	15
					0.25	4	119	4
				R061000	0.04	3	76	15
					0.1	6	101	13
					0.25	3	87	30
				R043449	0.04	5	89	12
					0.1	8	97	14
					0.25	4	87	11
			Bovine (milk)	Imazalil	0.02	6	89	19
					0.05	6	95	15
					0.1	2	88	—
				R061000	0.02	8	89	18
					0.05	6	103	8
					0.1	4	107	9
				R043449	0.02	6	81	10
					0.05	8	93	14
					0.1	4	98	5
687971 (Van Dijk, 1999)	LC-MS	Tissues – methanol Eggs – acetonitrile and methanol	Poultry (muscle)	Imazalil	0.02	6	86	17
					0.04	7	79	14
					0.1	6	77	9
				R042639	0.02	6	91	9
					0.04	8	85	8
					0.1	7	86	10
				R044085	0.02	6	82	8
					0.04	7	87	8
					0.1	7	87	19
			Poultry	Imazalil	0.02	4	113	16

Method Identification (Reference)	Method Type	Extraction Solvents	Commodity	Analyte	Fortification Levels (mg /kg)	n	Mean Recovery %	Relative Standard Deviation (RSD) %
			(liver)		0.04	6	89	6
					0.1	6	71	8
				R042639	0.02	6	115	10
					0.04	6	94	14
					0.1	6	85	13
				R044085	0.02	4	101	28
					0.04	6	98	22
					0.1	5	91	30
				R110740	0.02	2	69	—
					0.04	4	56	8
					0.1	4	51	8
			Poultry (fat)	Imazalil	0.02	5	66	7
					0.04	5	65	8
					0.1	8	87	27
				R042639	0.02	10	84	21
					0.04	8	76	10
					0.1	4	78	12
				R044085	0.02	7	86	15
					0.04	7	77	22
					0.1	6	97	23
			Poultry (egg)	Imazalil	0.01	8	86	16
					0.02	9	92	11
					0.05	10	88	17
				R042639	0.01	8	101	11
					0.02	9	94	15
					0.05	8	87	21
				R044085	0.01	8	90	18
					0.02	10	90	16
					0.05	10	88	13
				R110740	0.01	5	87	25
					0.02	5	80	11
					0.05	6	74	4
803327, AGR361 (Wolf, 2001)	HPLC-MS/MS Imazalil: m/z = 297→159 R043449: m/z = 287→141 R061000: m/z = 361→99	acetonitrile	Bovine (muscle)	Imazalil	0.01	5	105	10
					0.1	5	109	4
				R043449	0.01	5	87	13
					0.1	5	92	10
				R061000	0.01	5	99	7
					0.1	5	90	8
			Bovine (liver)	Imazalil	0.01	5	100	8
					0.1	5	86	12
				R043449	0.01	5	89	13
					0.1	5	99	11
				R061000	0.01	5	95	11
					0.1	5	100	6
			Bovine (kidney)	Imazalil	0.01	5	88	2
					0.1	5	87	5
				R043449	0.01	5	96	6
					0.1	5	92	4
				R061000	0.01	5	105	2
					0.1	5	104	5
			Bovine (fat)	Imazalil	0.01	5	93	5
					0.1	5	91	6
				R043449	0.01	5	85	13
					0.1	5	84	6
				R061000	0.01	5	94	15
					0.1	5	88	7
			Bovine	Imazalil	0.01	5	109	4

Method Identification (Reference)	Method Type	Extraction Solvents	Commodity	Analyte	Fortification Levels (mg /kg)	n	Mean Recovery %	Relative Standard Deviation (RSD) %
			(milk)		0.1	5	95	6
				R043449	0.01	5	86	15
					0.1	5	86	18
				R061000	0.01	5	91	14
					0.1	5	84	11

Method Identification (Reference)	Method Type	Extraction Solvents	Commodity	Analyte	Fortification Levels (mg /kg)	n	Mean Recovery %	Relative Standard Deviation (RSD) %
12G05162-01-VMAT (ILV of 803327 (modified)) (Lang, 2013)	HPLC-MS/MS m/z = 361→99 (quantitation) m/z = 363→99 (confirmation)	acetonitrile	Bovine (muscle)	R061000 361→99	0.01	5	102	13
					0.1	5	91	4
				R061000 363→99	0.01	5	104	15
					0.1	5	90	5
			Poultry (liver)	R061000 361→99	0.01	5	101	9
					0.1	5	100	5
				R061000 363→99	0.01	5	108	12
					0.1	5	101	6
08J01019-01-VMAT (ILV of 803327) (Hamberger, 2008)	HPLC-MS/MS m/z = 297→159 (quantitation) m/z = 297→201 (confirmation)	acetonitrile	Bovine (milk)	Imazalil 297→159	0.01	5	90	4
					0.1	5	88	4
				Imazalil 297→201	0.01	5	91	5
					0.1	5	85	2
			Bovine (muscle)	Imazalil 297→159	0.01	5	87	7
					0.1	5	85	12
				Imazalil 297→201	0.01	5	89	1
					0.1	5	84	12
			Bovine (liver)	Imazalil 297→159	0.01	5	74	2
					0.1	5	70	2
				Imazalil 297→201	0.01	5	77	2
					0.1	5	71	2
			Bovine (kidney)	Imazalil 297→159	0.01	5	76	2
					0.1	5	74	6
				Imazalil 297→201	0.01	5	77	3
					0.1	5	75	5
			Bovine (fat)	Imazalil 297→159	0.01	5	86	4
					0.1	5	89	4
				Imazalil 297→201	0.01	5	88	3
					0.1	5	89	4
JAN-0803V AGR 3956 (Schernikau, 2008)	HPLC-MS/MS m/z = 297→159 (quantitation) m/z = 297→201. (confirmation)	acetonitrile	Poultry (egg)	Imazalil 297→159	0.01	5	100	4
					0.1	5	106	3
				Imazalil 297→201	0.01	5	108	11
					0.1	5	104	4
08J01019-01-VMEG, AGR 4035 (ILV of JAN-0803V) (Hamberger, 2008)	HPLC-MS/MS m/z = 297→159 (quantitation) m/z = 297→201 (confirmation)	acetonitrile	Poultry (egg)	Imazalil 297→159	0.01	5	84	1
					0.1	5	86	2
				Imazalil 297→201	0.01	5	87	3
					0.1	5	87	2
11G04032-01-VMAT (ILV of 803327 (modified))	HPLC-MS/MS m/z = 287→141 (quantitation) m/z = 289→141 (confirmation)	acetonitrile	Bovine (muscle)	R043449 287→141	0.01	5	97	10
					0.1	5	91	7
				R043449 289→141	0.01	5	94	7
					0.1	5	92	5
			Bovine (liver)	R043449 287→141	0.01	5	89	10
					0.1	5	93	7
				R043449	0.01	5	90	9

Method Identification (Reference)	Method Type	Extraction Solvents	Commodity	Analyte	Fortification Levels (mg /kg)	n	Mean Recovery %	Relative Standard Deviation (RSD) %
				289→141	0.1	5	92	7
			Bovine (kidney)	R043449 287→141	0.01	5	93	9
					0.1	5	93	2
				R043449 289→141	0.01	5	94	11
					0.1	5	92	2
			Porc (fat)	R043449 287→141	0.01	5	95	5
					0.1	5	93	2
				R043449 289→141	0.01	5	92	9
					0.1	5	95	4
			Bovine (milk)	R043449 287→141	0.01	5	90	7
					0.1	5	92	4
				R043449 289→141	0.01	5	88	11
					0.1	5	92	4
			Poultry (egg)	R043449 287→141	0.01	5	84	12
					0.1	5	96	6
				R043449 289→141	0.01	5	82	9
					0.1	5	91	7

Applicability of multi-residue methods

Imazalil may be detected in grapes and orange juice using QuEChERS multi-residue methods, where acceptable recoveries were obtained at the LOQs of 0.01 mg/kg and 0.0085 mg/L, respectively.

STABILITY OF RESIDUES IN STORED ANALYTICAL SAMPLES

The freezer storage stability of imazalil in homogenised plant samples fortified with imazalil and its plant-specific metabolite (R014821) was studied.

Studies on storage stability of imazalil and its animal-specific metabolites (R043449, R061000 and R110740) were not provided to the Meeting as all animal samples in the livestock feeding studies were extracted and analysed within 30 days from sampling.

Stability of residues in plant products

The stability of imazalil and imazalil alcohol (R014821) was investigated in orange and its processed commodities, apple and its processed commodities, bananas, melons, tomatoes, potatoes and cereal grain and straw. Samples were fortified with each analyte at various concentrations and stored frozen. Samples were taken for analysis at intervals up to 12 months. While the methods used were concurrently validated at the time of analysis of the stored samples, concurrent method validation was not performed at the time the stored samples of oranges and processed commodities were analysed.

No dissipation of residues of imazalil and R014821 was observed in any of the raw agricultural commodities or the processed commodities. The recoveries of imazalil and R014821 after frozen storage are summarized in Table 42.

Table 42 Stability of imazalil and R014821 residues in various commodities during frozen storage

Commodity	Storage Period (days)	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Reference
	Imazalil				R014821			
Bananas	0	0.05	0.053, 0.049, 0.056, 0.055	100	0.05	0.03, 0.032, 0.043 0.038	100	Garnier, 1992, AGR 4
	84	0.05	0.049, 0.044, 0.037, 0.041	79	0.05	0.059, 0.060, 0.060, 0.060	167	
	168	0.05	0.048, 0.047, 0.046, 0.045	89	0.05	0.055, 0.051, 0.051, 0.052	144	
	266	0.05	0.069, 0.065, 0.062, 0.063	123	0.05	0086, 0.085, 0.083, 0.084	233	
	0	0.2	0.175, 0.189, 0.162 0.172	100	0.2	0.151, 0.171, 0.155, 0.148	100	
	84	0.2	0.158 0.154.	89	0.2	0.187, 0.179,	116	

Commodity	Storage Period (days)	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Reference
	Imazalil				R014821			
			0.150, 0.157			0.175, 0.182		
	168	0.2	0.172, 0.177, 0.160, 0.163	96	0.2	0.173, 0.182, 0.157, 0.162	108	
	266	0.2	0.174, 0.193, 0.185, 0.187	106	0.2	0.209, 0.247, 0.243, 0.257	153	
	0	3.0	3.71, 3.582, 3.663, 3.257	100	Not analysed			
	84	3.0	2.975, 2.887, 2.735, 3.012	82				
	168	3.0	2.188, 2.365, 2.689, 2.227	67				
	266	3.0	2.242, 2.506, 2.603, 2.578	71				
Citrus fruit	0	0.25	0.248, 0.230, 0.250	100	0.25	0.247, 0.243, 0.250	100	Woestenborghs, 1998, AGR 7
	84	0.25	0.246, 0.239, 0.243	100	0.25	0.250, 0.243, 0.247	100	
	168	0.25	0.254, 0.246, 0.245	102	0.25	0.265. 0.256. 0.256	105	
	245	0.25	0.238, 0.217, 0.230	94	0.25	0.239. 0.247. 0.241	98	
	0	10.0	11.2, 10.9, 10.8	100	1.0	1.10. 1.04. 0.999	100	
	84	10.0	10.9, 10.8, 10.8	99	1.0	1.09. 1.05. 1.14	103	
	168	10.0	10.5, 10.5, 10.2	95	1.0	1.00, 0.971, 0.993	94	
	245	10.0	9.42, 9.85, 9.39	87	1.0	0.954, 0.973, 0.913	90	
Dried peel	0	2.50	2.17, 2.08, 2.27	100	0.25	0.231, 0.238, 0.250	100	
	84	2.50	2.23, 2.35 ,2.26	105	0.25	0.252, 0.25, 0.229	102	
	168	2.50	2.31, 2.26, 2.34	106	0.25	0.238, 0.231, 0.247	100	
	245	2.50	2.22, 2.07, 2.04	97	0.25	0.247, 0.231, 0.243	100	
	0	25.0	25.8, 25.2, 26.1	100	2.50	2.34, 2.33, 2.34	100	
	84	25.0	24.3, 24.6, 23.5	94	2.50	2.36, 2.41, 2.39	102	
	168	25.0	28.9, 25.9, 25.2	104	2.50	2.79, 2.51, 2.47	111	
	245	25.0	24.4, 23.6, 22.7	92	2.50	2.30, 2.30, 2.23	98	
Chopped peel	0	2.50	2.23, 2.46, 2.21	100	0.25	0.262, 0.21, 0.242	100	
	84	2.50	2.66, 2.52, 2.48	111	0.25	0.258, 0.238, 0.254	97	
	168	2.50	2.51, 2.34, 2.43	105	0.25	0.248, 0.238, 0.223	92	
	245	2.50	2.23, 2.32, 2.30	99	0.25	0.216, 0.219, 0.233	86	
	0	25.0	25.3, 26.6, 26.4	100	2.50	2.29, 2.45, 2.45	100	
	84	25.0	25.8, 26.3, 25.2	98	2.50	2.53, 2.58, 2.43	105	
	168	25.0	27.9, 26.3, 26.0	102	2.50	2.60, 2.54, 2.42	105	
	245	25.0	23.4, 23.5,	89	2.50	2.38, 2.38,	98	

Commodity	Storage Period (days)	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Reference
	Imazalil				R014821			
			22.9			2.26		
Oil	0	0.25	0.280, 0.255, 0.227	100	0.25	0.255, 0.252, 0.245	100	
	84	0.25	0.264, 0.268, 0.291	108	0.25	0.249, 0.227, 0.245	96	
	168	0.25	0.230, 0.222, 0.271	95	0.25	0.235, 0.211, 0.248	92	
	245	0.25	0.253, 0.240, 0.235	96	0.25	0.237, 0.219, 0.208	88	
	0	25.0	25.1, 26.6, 24.0	100	2.50	2.65 ,2.75, 2.55	100	
	84	25.0	23.8, 24.9, 24.8	97	2.50	2.37, 2.49, 2.51	93	
	168	25.0	25.2, 25.5, 23.2	98	2.50	2.45, 2.32, 2.07	86	
	245	25.0	22.2, 22.9, 21.8	88	2.50	1.82, 1.88, 2.11	73	
Molasses	0	1.0	1.03, 1.03, 1.02	100	0.25	0.249, 0.231, 0.262	100	
	84	1.0	1.13, 1.19, 0.996	105	0.25	0.257, 0.297, 0.258	109	
	168	1.0	0.990, 0.940, 0.969	94	0.25	0.257, 0.258, 0.253	104	
	245	1.0	0.942 ,0.957, 0.973	94	0.25	0.250, 0.245, 0.249	100	
	0	10.0	10.9, 10.9, 11.0	100	1.0	1.05, 1.00, 1.05	100	
	84	10.0	10.3, 10.5, 10.4	96	1.0	0.996, 0.979, 1.00	96	
	168	10.0	10.2, 10.3, 10.9	96	1.0	0.53, 0.966, 0.919	92	
	245	10.0	10.4, 10.6 10.4	96	1.0	1.00, 0.968, 0.992	96	
Juice	0	0.25	0.225, 0.219, 0.213	100	0.025	0.029, 0.028, 0.027	100	
	84	0.25	0.256, 0.234, 0.266	116	0.025	0.027, 0.025, 0.029	96	
	168	0.25	0.259, 0.270, 0.262	120	0.025	0.025, 0.023, 0.023	84	
	245	0.25	0.236, 0.241, 0.241	10	0.025	0.025 0.026, 0.025	90	
	0	1.0	1.03, 0.983, 1.01	100	0.25	0.260 0.257, 0.252	100	
	84	1.0	1.05, 1.06, 0.979	102	0.25	0.256, 0.254, 0.253	99	
	168	1.0	1.04, 1.07, 1.07	105	0.25	0.253, 0.251, 0.243	97	
	245	1.0	1.02, 0.990, 0.929	97	0.25	0.251, 0.251 0.258	99	
Wheat grain	0	0.10	0.081, 0.077, 0.083	100	Not analysed			Schernikau, 2008, CET-0801
	86	0.10	0.087, 0.083	91				
	184	0.10	0.079, 0.076	94				
Barley straw	0	0.10	0.076, 0.076, 0.077	100				
	86	0.10	0.069, 0.070	100				
	184	0.10	0.078, 0.079	94				
Wheat grain	0	0.20	0.197, 0.199	100	Not analysed			Hamberger, 2006, R-20000
	90	0.20	0.200, 0.207	103				
	180	0.20	0.205, 0.199	102				

Commodity	Storage Period (days)	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Reference
	Imazalil				R014821			
Wheat plant	0	0.20	0.193, 0.201	100				
	90	0.20	0.195, 0.183	96				
	180	0.20	0.144, 0.164	78				
Wheat straw	0	0.20	0.193, 0.179	100				
	90	0.20	0.185, 0.193	101				
	180	0.20	0.172, 0.177	93				
Tomato	0	0.10	0.083, 0.089, 0.083	100	Not analysed			Schernikau, 2008, GAB-0729
	90	0.10	0.072, 0.071	89				
	183	0.10	0.077, 0.080	82				
Melon	0	1.0	0.841	100	Not analysed			Byast, 1997, R-8918A
	90	1.0	1.043	124				
	180	1.0	0.904	107				
Apple juice	0	0.10	0.095, 0.092, 0.097	100	0.10	0.096, 0.099, 0.098	100	Zeisler, 2013, R-29503/ R-29504
	120	0.10	0.101, 0.109, 0.106	102	0.10	0.101, 0.101, 0.099	99	
	180	0.10	0.097, 0.098, 0.098	95	0.10	0.107, 0.105, 0.105	104	
	365	0.10	0.105, 0.102, 0.100	100	0.10	0.110, 0.110, 0.108	108	
Apple sauce	0	0.10	0.089, 0.093, 0.090	100	0.10	0.089, 0.001, 0.092	100	
	120	0.10	0.097, 0.100, 0.096	95	0.10	0.105, 0.106, 0.094	101	
	180	0.10	0.093, 0.099, 0.098	94	0.10	0.102, 0.100, 0.099	100	
	365	0.10	0.093, 0.091, 0.094	90	0.10	0.106, 0.109, 0.107	106	
Apple wet pomace	0	0.10	0.087, 0.085, 0.091	100	0.10	0.086, 0.089, 0.090	100	
	120	0.10	0.097, 0.097, 0.092	94	0.10	0.098, 0.092, 0.098	96	
	180	0.10	0.078, 0.073, 0.084	80	0.10	0.100, 0.093, 0.092	96	
	365	0.10	0.081, 0.084, 0.079	80	0.10	0.094, 0.100, 0.095	97	
Apple dry pomace	0	0.10	0.082, 0078, 0.079	100	0.10	0.082, 0.084, 0.086	100	
	120	0.10	0.098, 0.093, 0.092	91	0.10	0.106, 0.097, 0.106	100	
	180	0.10	0.089, 0.082, 0.074	79	0.10	0.095, 0.089, 0.082	85	
	365	0.10	0.092, 0.100, 0.093	91	0.10	0.101, 0.098, 0.104	97	
Apple peel	0	0.10	0.074, 0.078, 0.078	100	0.10	0.077, 0.079, 0.076	100	
	120	0.10	0.090, 0.088, 0.099	96	0.10	0.103, 0.100, 0.105	106	
	180	0.10	0.076, 0.078, 0.074	79	0.10	0.098, 0.092, 0.098	99	
	365	0.10	0.071, 0.070, 0.070	73	0.10	0.099, 0.098, 0.101	103	
Orange marmalade	0	0.51	0.505, 0.487	100	0.50	0.415, 0.384	100	Austin, 2014, R-29509/ R-29510
	120	0.51	0.548, 0.631	117	0.50	0.505. 0.577	102	
	180	0.51	0.542, 0.512	104	0.50	0.487. 0.464	90	
	365	0.51	0.480, 0.478	95	0.50	0.482. 0.499	93	
Orange essential oil	0	0.51	0.468, 0.456	100	0.50	0.412, 0.412	100	
	120	0.51	0.538, 0.542	105	0.50	0.464. 0.459	94	

Commodity	Storage Period (days)	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Spiking Level (mg/kg)	Stored Sample Residues (mg/kg)	Mean % Remaining	Reference
	Imazalil				R014821			
	180	0.51	0.560, 0.565	110	0.50	0.508. 0.499	103	
	365	0.51	0.464, 0.485	92	0.50	0.473. 0.439	93	
Potato tubers	0	0.10	0.091, 0.088, 0.093	100	0.10	0.085, 0.092, 0.089	100	Daneva, 2011, S10-01082; R01824: Lindner, 2012, S11-02829
	31	0.10	0.090. 0.098	120	0.10	0.078, 0.076	87	
	90	0.10	0.073, 0.078	97	0.10	0.075, 0.087	91	
	181	0.10	0.075, 0.075	94	0.10	0.071, 0.072	81	
	273	0.10	0.071, 0.072	87	0.10			

Stability of residues in animal products

Studies on storage stability of imazalil in animal tissues were not provided to the Meeting as all animal samples in the livestock feeding studies were extracted and analysed within 30 days from sampling.

USE PATTERN

Imazalil is a fungicide registered in many countries all over the world. It is either intended for post-harvest use in various fruits such as citrus and banana against fungal storage diseases or as a foliar spray application for the control of powdery mildew in tomato and cucumber grown under glasshouse conditions and on artificial substrate or as a seed treatment on cereals or potato seed pieces.

Table 43 Selected registered uses of imazalil

Crop	Country	Formulation	Application				Days After Application (DAA)
			Method	Rate, kg ai/tonne	Spray conc. kg ai/hL	No. (min RTI)	
Citrus fruit	Argentina	50 g/L, EC	Dip, spray, drench	-	0.0025-0.01	1	n/a
			Wax	-	0.02-0.05	1	n/a
		750 g/kg, SP	Dip	-	0.05-0.1	1	n/a
			Spray	-	0.1-0.2	1	n/a
		400 g/L, SC	Drench	-	0.2	1	n/a
			Spray, wax	-	0.4	1	n/a
	Australia	500 g/L, EC 750 g/kg, SG 750 g/kg, WG	Dip, flood, spray	-	0.05	1	n/a
		500 g/L, EC	Wax	-	0.25-0.4	1	n/a
		750 g/kg, WG 500 g/L, EC	Dip, drench, spray	-	0.05	1	0
		500 g/L, EC	Wax	-	0.4	1	0
		200 g/L, SC	Dip	-	0.04	1	0
			Spray	-	0.1-0.2	1	0
			Wax	-	0.2-0.3	1	0
	Cameroon	750 g/kg, WSG	Dip	-	0.05	1	0
			Spray	-	0.1	1	0
	Central America	750 g/kg, SG	Drench	-	0.3-0.525	1	0
			Dip	-	0.075	1	0
	Chile	750 g/kg, SG	Drench, spray	-	0.0975	1	0
			Wax	-	0.15	1	0
		500 g/L, EC	Drench	-	0.05	1	0
			Spray	-	0.1-0.15	1	0
			Wax	-	0.2-0.4	1	0
	China	500 g/L, EC	Dip		0.025-0.05	1	60
	European countries	75 g/L, SL	Dip, drench	-	0.0375-0.045	1	0
			Spray		0.15-0.20	1	0
		500 g/L, EC	Dip, drench	-	0.05	1	0
			Spray	-	0.1-0.15	1	0
			Wax	-	0.2-0.4	1	0
		75 g/L, SC	Dip, drench	-	0.045	1	0

Crop	Country	Formulation	Application				Days After Application (DAA)
			Method	Rate, kg ai/tonne	Spray conc. kg ai/hL	No. (min RTI)	
			Spray	-	0.15	1	0
		75g/L, SL	Drench	-	0.0375-0.045	1	0
			Spray	-	0.15-0.1875	1	0
		500 g/L, EC	Dip, drench	-	0.5	1	0
			Spray	-	0.15	1	0
			Wax	-	0.2	1	0
		200 g/L, SC	Drench	-	0.04-0.05	1	0
			Spray	-	0.1	1	0
		25%, EC	Fogging	0.006	-	1	0
		7.5 g/L, SL	Dip	-	0.045	1	0
		500 g/L, EC	Dip	-	0.05-0.1	1	0 3 (Portugal)
			Spray	-	0.5	1	0
			Wax	-	0.2	1	0
	Israel	500 g/L, EC	Dip, drench	-	0.1	1	0
			Wax	-	0.2	1	0
	Mexico	500 g/L, EC	Dip	-	0.05-0.2	1	0
			Wax	-	0.2	1	0
	Morocco	500 g/L, EC	Dip, drench	-	0.025	1	0
			Wax	-	0.1	1	0
		200 g/L, SC	Drench	-	0.05	1	0
			Spray	-	0.1	1	0
			Wax	-	0.3	1	0
		500 g/L, EC	Wax	0.0024	-	1	0
		25%, EC	Fogging	0.006	-	1	0
	Peru (mandarins)	500g/L, EC	Wax	0.2-0.004	-	1	0
	South Africa	200 g/L, SC	Dip	-	0.05	1	0
		500 g/L, EC	Dip	-	0.05	1 in addition to wax	0
			Spray	-	0.1	1 in addition to wax	0
			Wax	-	0.3	1 in addition to dip or spray	0
		750 g/kg, WSG	Dip, drench	-	0.05	1	0
		750 g/L, WSP, 750 g/kg, SG	Spray	-	0.1	1	0
	Turkey	75 g/L, SL	Dip, drench	-	0.035	1	0
		500 g/L, EC	Wax	-	0.2	1	0
	Uruguay	750 g/L, SP	Dip	-	0.05	1	0
			Drench	-	0.1	1	0
			Spray	-	0.2	1	0
		500 g/L, EC	Dip or spray	-	0.2	1	0
			Wax	-	0.1-0.2	1	0
		200 g/L, SC	Dip, drench	-	0.05-0.1	1	0
			Spray, wax	-	0.2	1	0
	USA*	75%, WSG	Dip, drench	-	0.05-0.075	1	0
		500 g/L, EC	Spray	-	0.1	1	0
		200 g/L, SC	Dip, drench	-	0.025-0.075	1	0
			Spray	-	0.05-0.1	1	0
			Wax (dilution)	-	0.1-0.2	1	0
		500 g/L, EC	Dip, drench	-	0.05-0.075	1	0
			Spray	-	0.1	1	0
			Wax (dilution)	-	0.2	1	0
			RTU Wax	-	0.4	1	
	Zimbabwe	800 g/L, EC	Spray	-	0.124	1	0
			Wax	-	0.32	1	0
Bananas	Cameroon	750 g/kg, WSG	Drench	-	0.05	1	0
			Spray	-	0.1	1	0
	China	75%, SG	Dip	-	0.10-0.15	1	20

Crop	Country	Formulation	Application				Days After Application (DAA)
			Method	Rate, kg ai/tonne	Spray conc. kg ai/hL	No. (min RTI)	
	Columbia	100g/L, SL	Dip, drench, spray	-	0.04-0.06	1	0
		750 g/kg, SP	Dip, drench	-	0.03-0.06	1	0
	Dominican Republic	75%, WSP	Dip, drench, spray	-	0.04-0.06	1	0
	Ecuador	750 g/kg, SP	Spray	-	0.045	1	1
		750 g/kg, SG	Spray	-	0.06	1	2
	Spain, France	75 g/L, SL	Drench	-	0.0375-0.045	1	0
		750 g/L, SP	Dip, drench	-	0.0375	1	0
	Portugal	500 g/L, EC	Dip, drench	-	0.05	1	3
	Guatemala/ Honduras/ Nicaragua	750 g/kg, SG 750 g/kg, SP	Dip, drench, spray	-	0.04-0.06	1	0
	Israel	500 g/L, EC	Dip, drench, spray	-	0.0375-0.05	1	0
	Philippines	750 g/kg, SP	Dip, spray	-	0.03-0.052	1	0
	Uruguay	75%, WSP	Dip	-	0.05	1	0
			Drench	-	0.10		
			Spray	-	0.15-0.20		
Wheat, barley	USA	100 g/L, EC 450 g/L, EC	Seed treatment	0.05-0.1	n/a	1	n/a`
Fruiting vegetables, cucurbits	Belgium	100 g/L	Foliar spray	-	0.005	3 (7-day interval)	1 (all except melon) 3 (melon)
	Israel	500 g/L, EC	Dip spray	-	0.2	1	0
Tomato	Belgium Netherlands	100 g/L, SL 100 g/L, SC	Foliar spray	-	0.02	3 (7-day interval)	1
	France Belgium Germany Italy Netherlands	2% aerosol 20 g/kg, aerosol	Spot treatment	-	2 g/plant	2 (14-day interval)	1
		60%, aerosol	Spot treatment	-	1.8 g/plant	2 (14-day interval)	1
	New Zealand	20 g/kg, aerosol	Spot treatment	-	Not specified	4 (7-day interval)	3
	Turkey	500 g/L, EC	Foliar spray	-	0.015	Not specified (10-15 day interval)	3
Potato (seed and ware)	Australia	500 g/L, EC 750 g/kg, SG 750 g/kg, WG	Spray	0.015	-	1	n/a
	France, Belgium,	100 g/L, SL	Spray	0.0075-0.015	-	1	n/a
		100 g/L, SC	Spray, Fogging	0.015	-	1	n/a
		100 g/L, SL	Spraying, Fogging	0.015	-	1	n/a
	UK	100 g/L, SC	Spraying	0.0075-0.015	-	1	n/a

* dual use (use of more than one product) permitted in the USA

RESULTS OF SUPERVISED RESIDUE TRIALS ON CROPS

The Meeting received information on supervised field trials for imazalil on the following crops or crop groups:

Crop	Table No.
Citrus fruits	44-51
Banana	52, 53
Cucumber	54
Tomato	55, 56
Potato	
Seed piece treatment	57
Post-harvest	58
Cereal grain	59
Cereal straw	60

Trials were generally well documented with laboratory and field reports. Laboratory reports included method validation with procedural recoveries from spiking at levels similar to those occurring in samples from the supervised trials. Dates of analyses or storage duration were also provided. Although trials included control plots, no control data are recorded in the tables. Unless stated otherwise, residue data are recorded unadjusted for recovery. Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined. Conditions of the supervised residue trials were generally well reported in detailed field reports. Reports provided data on the method of treatment, sample size and sampling date. Where duplicate samples from an un-replicated trial were taken at each sampling time and were analysed separately, the mean of the two analytical results was taken as the best estimate of the residues.

Citrus fruits

Lemons

United States

Seven post-harvest trials in the US were conducted during 1987-1988, where emulsifiable concentrate formulations, containing 500 g/L or 289 g/L of imazalil were applied to lemons either via pack wax, water spray, or a combination of both at total rates of 0.2-0.4 kg ai/hL. All samples were collected immediately after treatment (0-DAA).

A GC/MS method was used to determine residues of imazalil and the metabolite R14821 which reported an LOQ of 0.05 mg/kg/analyte.

The maximum duration in frozen storage was 73 days.

Table 44 Magnitude of the residues of imazalil and R014821 in/on lemons following post-harvest treatment from trials conducted in the USA

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
USA GAP		Dip, drench	0.075	1 combined with wax application			
		Spray	0.1	1 combined with wax application			
		Wax (diluted)	0.2	1 combined with dip, drench or spray application			
		Wax (Ready to use)	0.4	1			

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
F004-P/F005-P San Joaquin, CA, USA 1988	Lemon	500 EC, storage wax 500 EC, pack wax	0.2 0.1	2	peel pulp whole fruit[a]	3.79 0.21 1.13	0.13 <0.05 0.02
F122-P/F123-P Ventura Co, CA, USA 1988	Lemon	500 EC, storage wax 500 EC, pack wax	0.2 0.2	2	peel pulp whole fruit[a]	4.46 0.17 0.84	0.36 <0.05 0.07
F126-P/F127-P Lindsay, CA, USA 1988	Lemon	289 EC, storage wax 289 EC, pack wax	0.2 0.2	2	peel pulp whole fruit[a]	3.70 0.14 1.16	<0.05 <0.05 0.02
F276-P/F277-P Upland, CA, USA 1988	Lemon	500 EC, pack wax	0.2	1	peel pulp whole fruit[a]	2.65 0.20 0.11	0.27 <0.05 0.10
F278-P/F279-P Upland, CA, USA 1988	Lemon	500 EC, pack wax	0.4	1	peel pulp whole fruit[a]	3.35 0.21 1.60	0.38 <0.05 0.17
F280-P/F281-P Upland, CA, USA 1988	Lemon	500 EC, pack wax	0.4	1	peel pulp whole fruit[a]	3.01 0.20 1.45	0.29 <0.05 0.13
F858-0/F862-0 Upland, CA, USA 1987	Lemon	500 EC, pack wax	0.2	1	peel pulp whole fruit[a]	3.54 0.24 1.61	1.32 0.11 0.61

[a] Whole fruit residue calculated based on peel and pulp individual weights and residues

Europe

In the 1997 trials conducted in Spain (De Winter, 1997, AGR247), lemons received four different treatments: 1) drench with different solutions, each containing 75 g/L imazalil (L/D1 and L/D2); 2) drench with different solutions, each containing 75 g/L imazalil + wash + dry (L/DWD1 and L/DWD2); 3) drench with different solutions, each containing 75 g/L imazalil, + wash + wax formulated with different emulsifiable concentrate formulations containing 500 g/L of imazalil + dry (L/DWWD1 and L/DWWD2); or 4) wash + wax formulated with different emulsifiable concentrate formulations containing 500 g/L of imazalil + dry (L/WWD1 and L/WWD2). All samples were collected immediately after treatment (0-DAA). Samples comprised of at least 12 fruit and weighing at least 1 kg. The GC-ECD method Gent 53/3b, for which the LOQ of 0.5 mg/kg was reported, was used to analyse all samples. The maximum duration in frozen storage was 49 days.

One post-harvest trial was conducted in Spain on lemons during 2003 (Gimeno Martos, 2002, TRC03-6). The fruits received either one application of:1) a wax containing 2 g/L of imazalil applied at 0.2 kg ai/hL; 2) an SC formulation containing 200 g/L of imazalil dissolved in a wax and applied at 0.2-0.3 kg ai/hL; and 3) a different SC formulation containing 200 g/L of imazalil dissolved in wax and applied at 0.2 kg ai/hL. Fruits were collected immediately after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg. Residues of imazalil were quantified using a validated GC-MS multi-residue method which reported an LOQ of 0.05 mg/kg. The storage intervals from collection to extraction did not exceed 56 days.

Nine post-harvest trials were conducted in Greece, Italy and Spain, during 2016, where lemons were treated with one post-harvest drench application followed by one storage wax line spray application of an emulsifiable concentrate formulation containing 500 g/L of imazalil (Grote, 2017, S16/06758; Grote, 2017, S17-07772). The post-harvest drench application was applied at a concentration of 0.075 kg ai/hL. After fruits were dried, one post-harvest storage wax line spray application followed at a concentration of 0.20 kg ai/hL. Treated whole lemons were collected immediately after the wax application (0-DAA). and comprised of at least 12 fruit and weighing a minimum of 1 kg. Residues of imazalil and the metabolite R014821 were quantified using a validated QuECHERS, HPLC/MS-MS method. The LOQ of the method was 0.01 mg/kg/analyte. The storage intervals from collection to extraction did not exceed 106 days

Table 45 Magnitude of the residues of imazalil and R014821 in/on lemons following post-harvest treatment from trials conducted in Europe

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
Europe GAP		Dip, drench	0.075	1 combined with wax application			
		Spray	0.1	1 combined with			

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
				wax application			
		Wax (diluted)	0.2	1 combined with dip, drench or spray application			
		Wax (Ready to use)	0.4	1			
S16-06758-01 47100, Arta, Arta, Greece 2017	Lemon / Femminello	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	9.7 (9.5, 9.9) 0.12	0.01 <0.01
S16-06758-02 59032, Plati, Imathia, Greece 2017	Lemon / Maglino	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	7.8 (7.9, 7.7) 0.20 (0.19, 0.20)	0.01 <0.01
S16-06758-03 95032, Belpasso, Sicily, Italy 2017	Lemon / Femminello	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	3.3 0.05	0.01 <0.01
S16-06758-04 95047, Paterno, Sicily, Italy 2017	Lemon / Monachello	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	3.1 0.05	0.02 <0.01
S16-06758-05 46469, Beniparrell, Valencia, Spain 2016	Lemon / Eureka	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	3.0 0.11	0.03 <0.01
S16-06758-06 25003, Lleida, Catalunya, Spain 2017	Lemon / Eureka	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	5.0 0.23 (0.22, 0.23)	0.02 <0.01
S17-07772-01 59032, Plati, Imathia, Greece, 2017	Lemon / Maglino	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	6.4 0.27	0.05 <0.01
S17-07772-02 46469, Beniparrell, Valencia, Spain, 2017	Lemon / Fino	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	4.1 0.36	0.01 <0.01
S17-07772-03 46469, Beniparrell, Valencia, Spain, 2018	Lemon / Fino	500 EC, drench 500 EC, wax	0.075 0.20	2	whole fruit pulp	5.7 (5.8, 5.5) 0.18 (0.18, 0.17)	0.02 <0.01
AGR247 El Puig, Valencia, Spain 1997	Lemon	S-7.5, drench L/D1	0.045	1	fruit	1.42 (1.53, 1.30)	Not analysed
		S-7.5, drench L/D2	0.045	1	fruit	1.45 (1.61, 1.29)	
	Lemon	S-7.5, drench L/DWD1	0.045	1	fruit	0.87 (0.98, 0.76)	
		S-7.5, drench L/DWD2	0.045	1	fruit	1.10 (1.23, 0.97)	
	Lemon	S-7.5, drench 50 EC, wax L/DWWD1	0.045 0.4	2	fruit	4.24 (4.32, 4.15)	
		S-7.5, drench 50 EC, wax L/DWWD2	0.045 0.4	2	fruit	4.54 (4.49, 4.59)	
	Lemon	50 EC, wax L/WWD1	0.4	1	fruit	2.24 (2.26, 2.21)	

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
		50 EC, wax L/WWD2	0.4	1	fruit	2.38 (2.30, 2.47)	
TRC03-6 Valencia, Spain 2003	Lemon	2 SC, wax	0.2	1	fruit	1.84	
		200 SC, wax	0.2	1	fruit	1.97	
			0.3	1	fruit	2.92	
		200 SC, wax	0.2	1	fruit	2.08	

Australia

Six post-harvest supervised trials were conducted in Australia on lemons during 2005 (Ridley, 2005, DEGROOT/GLP/04/03-1a). The test substance was a suspension concentration formulation containing 200 g/L of imazalil. Three treatment methods were assessed: dipping (0.04-0.05 kg ai/hL), non-recovery spray (0.1-0.2 kg ai/hL) and waxing (0.2-0.3 kg ai/hL). Lemons were collected immediately after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg.

Residues of imazalil were quantified using a published multi-residue LC/MS method employing acetonitrile extraction/partitioning and dispersive solid phase extraction (Journal of AOAC Intern. Vol. 86, no. 2, 2003). The LOQ of the method was 0.2 mg/kg. The storage intervals from collection to extraction did not exceed 46 days.

Table 46 Magnitude of the residues of imazalil and R014821 in/on lemons following post-harvest treatment from trials conducted in Australia

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Imazalil Residues (mg/kg)
Australia GAP		Dip, drench	0.075	1 combined with wax application		
		Spray	0.1	1 combined with wax application		
		Wax (diluted)	0.2	1 combined with dip, drench or spray application		
		Wax (Ready to use)	0.4	1		
4665 West Gosford NSW 2250, Australia 2005	Lemon / NS	200 SC, dip	0.04	1	whole fruit	5.0
			0.05	1	peel pulp whole fruit	14.0 <0.2 6.2
4667 Kulnura NSW 2250, Australia 2005	Lemon / NS	200 SC, spray	0.1	1	whole fruit	1.6
			0.2	1	whole fruit	3.5
4669 Kulnura NSW 2250, Australia 2005	Lemon / NS	200 SC, wax	0.2	1	whole fruit	1.8
			0.3	1	peel pulp whole fruit	8.3 0.3 2.6

Oranges

Many of the trials were conducted with formulations containing imazalil and pyrimethanil or thiabendazole, however, only residues of imazalil are reported herein.

United States of America

Post-harvest trials in the USA were conducted during 1987, where emulsifiable concentrate formulations, containing 500 g/L or 289 g/L of imazalil were applied to oranges either via pack wax, water spray, dip or foam, or a combination of these, at total rates of 0.1-0.68 kg ai/hL. All samples were collected immediately after treatment (0-DAA). Samples comprised of at least 12 fruits and weighing at least 1 kg.

A GC/MS method was used to determine residues of imazalil and the metabolite R014821 which reported an LOQ of 0.05 mg/kg/analyte.

The maximum duration in frozen storage was 71 days.

Table 47 Magnitude of the residues of imazalil and R014821 in/on oranges following post-harvest treatment from trials conducted in the USA

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
USA GAP		Dip, drench	0.075	1 combined with wax application			
		Spray	0.1	1 combined with wax application			
		Wax (diluted)	0.2	1 combined with dip, drench or spray application			
		Wax (Ready to use)	0.4	1			
F846-0/F849-0 Orange, CA, USA 1987	Orange / Valencia	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	2.70 0.12 0.89	0.34 <0.05 0.10
F852-0/F853-0 Orange, CA, USA 1987	Orange / Valencia	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	1.93 0.10 0.69	0.14 <0.05 0.05
F854-0/F855-0 Irvine, CA, USA 1987	Orange / Valencia	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	2.22 0.08 0.57	0.19 <0.05 0.05
F856-0/F857-0 Irvine, CA, USA 1987	Orange / Valencia	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	2.08 0.10 0.65	0.13 <0.05 0.04
F997-0/F998-0 Riverside, CA, USA 1987	Orange / Valencia	500 EC, pack wax	0.1	1	peel pulp whole fruit[1)]	4.73 0.27 1.73	0.44 <0.05 0.15
F999-0/F1000-0 Riverside, CA, USA 1987	Orange / Valencia	500 EC, water spray 500 EC, pack wax	0.1 0.1	2	peel pulp whole fruit[1)]	2.32 0.16 0.72	0.30 <0.05 0.08
F1001-0/F1002-0 Riverside, CA, USA 1987	Orange / Valencia	500 EC, water spray	0.1	1	peel pulp whole fruit[1)]	4.11 0.18 1.17	0.56 <0.05 0.15
F1007-0/F1008-0 Arlington Heights, CA, USA 1987	Orange / Valencia	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	1.00 0.07 0.36	0.08 <0.05 0.02
F1009-0/F1010-0 Arlington Heights, CA, USA 1987	Orange / Valencia	500 EC, dip	0.2	1	peel pulp whole fruit[1)]	40.1 0.19 12.38	0.87 <0.05 0.27
F1011-0/F1012-0 Arlington Heights, CA, USA 1987	Orange / Valencia	500 EC, water spray	0.2	1	peel pulp whole fruit[1)]	1.66 0.10 0.55	0.11 <0.05 0.03
F1013-0/F1014-0 Arlington Heights, CA, USA 1987	Orange / Valencia	500 EC, foam	0.2	1	peel pulp whole fruit[1)]	0.79 0.20 0.39	0.06 <0.05 0.02

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
F1015-0/F1016-0 Arlington Heights, CA, USA 1987	Orange / Valencia	500 EC, foam	0.2	1	peel pulp whole fruit[1)]	0.98 0.07 0.36	0.07 <0.05 0.02
F1017-0/F1018-0 Arlington Heights, CA, USA 1987	Orange / Valencia	500 EC, foam	0.2	1	peel pulp whole fruit[1)]	0.80 0.08 0.33	0.07 <0.05 002
F105-P/F106-P Lake Alfred, Florida, USA 1988	Orange / Hamlin	500 EC, pack wax	0.4	1	peel pulp whole fruit[1)]	22.12 0.39 6.43	0.66 <0.05 0.21
F663-0/F664-0 Vero Beach, Florida, USA 1987	Orange / Navel	500 EC, water spray	0.1	1	peel pulp whole fruit[1)]	1.33 <0.05 0.26	0.12 <0.05 0.02
F665-0/F666-0 Vero Beach, Florida, USA 1987	Orange / Navel	500 EC, water spray	0.2	1	peel pulp whole fruit[1)]	1.01 <0.05 0.21	0.07 <0.05 0.01
F667-0/F668-0 Vero Beach, Florida, USA 1987	Orange / Navel	500 EC, pack wax	0.1	1	peel pulp whole fruit[1)]	2.25 0.17 0.48	0.14 <0.05 0.03
F669-0/F670-0 Vero Beach, Florida, USA 1987	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.42 0.14 0.64	0.18 <0.05 0.03
F671-0/F672-0 Vero Beach, Florida, USA 1987	Orange / Navel	500 EC, water spray 500 EC, pack wax	0.1 0.1	2	peel pulp whole fruit[1)]	0.96 <0.05 0.19	0.10 <0.05 0.02
F006-P/F007-P San Joaquin, Ca, USA 1987	Orange / Navel	289 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	4.65 0.16 1.52	0.11 <0.05 0.04
F008-P/F009-P San Joaquin, Ca, USA 1987	Orange / Navel	289 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	5.32 0.15 2.15	0.12 <0.05 0.05
F132-P/F133-P Exeter, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.89 <0.05 1.34	0.09 <0.05 0.03
F134-P/F135-P Exeter, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.24 <0.05 0.96	0.07 <0.05 0.02
F136-P/F137-P Riverside, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.14	1	peel pulp whole fruit[1)]	3.36 0.08 1.06	0.06 <0.05 0.03
F138-P/F139-P Riverside, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.3	1	peel pulp whole fruit[1)]	9.40 0.26 3.11	0.13 <0.05 0.04
F140-P/F141-P Riverside, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.3	1	peel pulp whole fruit[1)]	9.02 0.31 2.93	0.10 <0.05 <0.05
F120-P/F121-P Exeter, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.15 0.13 1.01	0.28 <0.05 0.14
F124-P/F125-P Strathmore, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.06 0.10 0.84	0.36 <0.05 0.07

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
F128-P/F129-P San Joaquin, CA, USA 1988	Tangerine	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.51 0.12 1.13	0.06 <0.05 0.02
F130-P/F131-P Ventura Co., CA USA 1988	Orange / Navel	289 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	240 0.09 0.89	0.11 <0.05 0.05
F258-P/F259-P Strathmore, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	4.09 0.05 1.33	0.56 <0.05 0.19
F260-P/F261-P Exeter, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	5.44 0.13 1.85	0.24 <0.05 0.08
F262-P/F263-P Lindsay, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	2.43 0.10 0.92	0.10 <0.05 <0.05
F264-P/F265-P Ventura Co., CA, USA 1988	Orange / Navel	289 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.38 0.23 1.28	0.12 <0.05 0.05
F266-P/F267-P Ventura Co., CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	4.41 0.12 1.47	<0.05 <0.05 0.02
F268-P/F269-P Orange Cove, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	5.79 0.09 2.03	0.14 <0.05 0.05
F270-P/F271-P Terra Bella, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	3.24 0.08 1.24	<0.05 <0.05 <0.05
F286-P/F287-P Ventura Co., CA, USA 1988	Tangerine	289 EC, pack wax	0.2	1	peel pulp whole fruit[1)]	2.34 0.12 0.81	0.09 <0.05 0.03
F361-P/F362-P Dinuba, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.332	1	peel pulp whole fruit[1)]	3.95 0.10 1.38	0.24 0.11 0.15
F363-P/F364-P Dinuba, CA, USA 1988	Orange / Navel	500 EC, pack wax	0.68	1	peel pulp whole fruit[1)]	6.22 0.14 2.15	0.47 0.05 0.19

[a] Whole fruit residue calculated based on peel and pulp individual weights and residues

Europe

In the 1997 trials conducted in Spain (De Winter, 1997, AGR244/AGR245), Satsuma mandarins (C) or Navelina oranges (N) received four different treatments: 1) drench with different solutions, each containing 75 g/L imazalil (C/D1 and C/D2 or N/D1 and N/D2); 2) drench with different solutions, each containing 75 g/L imazalil, + wash + dry (C/DWD1 and C/DWD2 or N/DWD1 and N/DWD2); 3) drench with different solutions, each containing 75 g/L imazalil + wash + wax formulated with different soluble concentrates each containing 500 g/L of imazalil + dry (C/DWWD1 and C/DWWD2 or N/DWWD1 and N/DWWD2); or 4) wash + wax formulated with different soluble concentrates each containing 500 g/L of imazalil + dry (C/WWD1 and C/WWD2 or N/WWD1 and N/WWD2). All samples were collected immediately after treatment, with the exception of the C/DWD1, C/DWD2, N/DWD1 and N/DWD2 samples which were collected 3 days after application. Samples comprised of at least 12 fruit and weighing at least 1 kg. The GC-ECD method Gent 53/3b, for which the LOQ of 0.5 mg/kg was reported, was used to analyse all samples. The maximum duration in frozen storage was 27 days.

Five post-harvest trials were conducted in Spain during 2001 where oranges received single applications of a solution (containing 75 g/L of imazalil) or an emulsifiable concentrate formulation (containing 500 g/L of imazalil) (Serrano Delgado, 2002, TRC02-3). In the first trial, the EC formulation was applied as a spray with wax at a rate of 0.4 kg ai/hL while in the second and third trials, both formulations were each applied individually as spray applications, without wax, at 0.135-0.15 g ai/hL. The last two trials involved individual drench applications with each formulation at 0.05 kg ai/hL. All treated fruits were collected immediately after treatment (0-DAA). Residues of imazalil were determined using a GC-MS method where the LOQ was reported as 0.05 mg/kg. The maximum duration in frozen storage was 27 days.

Post-harvest trials were conducted in Spain on mandarins and oranges during 2001 (Serrano Delgado, 2002, TRC01-5). The fruits received one fogging application of a flowable dispersion formulation, containing 25% imazalil, at a rate of 0.06 kg ai/ton of fruit. Fruits were collected 0, 5, 15 and 30 days after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg.

Residues of imazalil were quantified using the validated GC-ECD DFG S19 multi-residue method. The LOQs of the method were 0.02 mg/kg (pulp), 0.05 mg/kg (peel, fruit). The storage intervals from collection to extraction did not exceed 148 days.

One post-harvest trial was conducted in Spain on oranges during 2003 (Gimeno Martos, 2002, TRC03-6). The fruits received either one application of:1) a wax containing 2 g/L of imazalil applied at 0.2 kg ai/hL; 2) an SC formulation containing 200 g/L of imazalil dissolve in a wax and applied at 0.2-0.3 kg ai/hL; and 3) a different SC formulation containing 200 g/L of imazalil dissolved in wax and applied at 0.2 kg ai/hL. Fruits were collected immediately after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg.

Residues of imazalil were quantified using a validated GC-MS multi-residue method which reported an LOQ of 0.05 mg/kg. The storage intervals from collection to extraction did not exceed 56 days.

Four post-harvest trials were conducted in Spain on oranges during 2004 (Gimeno Martos, 2004, TRC04-9/10/11/12). For two of the four trials, fruits received either one spray wax application of an emulsifiable concentrate formulation, containing 500 g/L of imazalil, at a rate of 0.2-0.5 kg ai/hL, one spray wax application of a soluble concentrate formulation, containing 150 g/L or 200 g/L of imazalil, at a rate of 0.2-0.5 kg ai/hL or a dual treatment consisting of a drench application at 0.05 kg ai/hL followed by a spray wax application at 0.3kg ai/hL. In the two remaining trials, all treatments were drench applications using solutions, containing 75 g/L of imazalil or 100 g/L of imazalil, at rates of 0.04-0.05 kg ai/hL. Oranges were collected immediately after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg.

Residues of imazalil were quantified using a validated GC-MS method. The LOQ of the method was 0.05 mg/kg. The storage intervals from collection to extraction did not exceed 122 days.

Six post-harvest supervised trials were conducted in Spain on mandarins during 2004 (Fernandez, 2004, 20047001-20047003/S1-FPMD). In two of the six trials, the fruits received one spray wax application of a suspension concentrate formulation, containing 400 g/L of imazalil, or an emulsifiable concentrate, containing 500 g/L of imzalil, at a rate of 0.2-0.3 kg ai/hL. For one of the treatment regimes, mandarins received a drench application of either formulation at a rate of 0.05 kg ai/hL prior to the spray wax application described above. In two separate trials, the fruits received one drench application of a suspension concentrate formulation, containing 200 g/L of imazalil, or an emulsifiable concentrate, containing 75 g/L of imzalil, at a rate of 0.04-0.05 kg ai/hL. In the remaining two trials, the fruits received one drench application of a solution containing 100 g/L of imazalil, at a rate of 0.04-0.05 kg ai/hL. Mandarins were collected immediately after treatment (0-DAA) and comprised of at least 24 fruit and weighing at least 3 kg.

Residues of imazalil were quantified using a validated liquid chromatography with diode array detector (LC/DAD). The LOQ of the method was 0.05 mg/kg. The storage intervals from collection to extraction did not exceed 58 days.

Post-harvest trials were conducted in Spain, Italy and Greece on mandarins and oranges during 2011-2012 (Grote, 2013, S11-03184/AGR 4726 to S11-03190/AGR 4732). The fruits received one dip, drench or spray application of a suspension/emulsion concentrates or solution formulations, containing concentrations of imazalil ranging from 75 g/L to 500 g/L, at rates ranging from 0.045-0.15 kg ai/hL. Fruits were collected immediately after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg.

Residues of imazalil and the metabolite R14821 were quantified using a validated QuECHERS, HPLC/MS-MS method. The LOQ of the method was 0.01 mg/kg/analyte. The storage intervals from collection to extraction did not exceed 236 days.

Nine post-harvest trials were conducted in Greece, Italy and Spain, during 2016 to 2018, where oranges were treated with one post-harvest drench application followed by one storage wax line spray application using an emulsifiable concentrate formulation containing 500 g/L of imazalil (Grote, 2017, S16/06757; Grote, 2018, S17-0771). The post-harvest drench application was applied at concentrations of 0.057-0.075 kg ai/hL. After fruits were dried, one post-harvest storage wax line spray application followed at concentrations of 0.20-0.46 kg ai/hL. Treated whole oranges were collected immediately after the wax application (0-DAA). and comprised of at least 12 fruits and weighing a minimum of 1 kg. Residues of imazalil and the metabolite R014821 were quantified using a validated QuECHERS, HPLC/MS-MS method. The LOQ of the method was 0.01 mg/kg/analyte. The storage intervals from collection to extraction did not exceed 100 days.

Table 48 Magnitude of the residues of imazalil and R014821 in/on oranges and mandarins following post-harvest treatment from trials conducted in Europe

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
USA GAP	Citrus fruit	Dip, drench	0.075	1 combined with wax application	0			
		Spray	0.1	1 combined with wax application	0			
		Wax (diluted)	0.2	1 combined with dip, drench or spray application	0			
		Wax (Ready to use)	0.4	1	0			
AGR244 Valencia, Spain 1997	Mandarin / Satsuma	S-7.5, drench C/D1	0.04	1	0	whole fruit	2.36 (2.33, 2.38)	Not analysed
		S-7.5, drench C/D2	0.04	1	0	whole fruit	2.05 (2.08, 2.03}	
	Mandarin / Satsuma	S-7.5, drench C/DWD1	0.04	1	3	whole fruit	2.60 (2.58, 2.61)	
		S-7.5, drench C/DWD2	0.04	1	3	whole fruit	1.63 (1.61, 1.65)	
	Mandarin / Satsuma	S-7.5, drench 50 EC, spray wax C/DWWD1	0.04 0.4	2	0	whole fruit	3.59 (3.59, 3.59)	
		S-7.5, drench 50 EC, spray wax C/DWWD2	0.04 0.4	2	0	whole fruit	2.73 (2.73, 2.73)	
	Mandarin / Satsuma	50 EC, spray wax C/WWD1	0.4	1	0	whole fruit	1.73 (1.68, 1.77)	
		50 EC, spray wax C/WWD2	0.4	1	0	whole fruit	2.01 (2.00, 2.01)	
AGR245 Valencia, Spain 1997	Orange / Navelina	S-7.5, drench N/D1	0.045	1	0	whole fruit	1.64 (1.61, 1.68)	Not analysed
		S-7.5, drench N/D2	0.045	1	0	whole fruit	1.38 (1.38, 1.39)	
	Orange / Navelina	S-7.5, drench N/DWD1	0.045	1	3	whole fruit	2.04 (2.05, 2.02)	
		S-7.5, drench N/DWD2	0.045	1	3	whole fruit	0.92 (0.88, 0.95)	
	Orange / Navelina	S-7.5, drench 50 EC, spray wax N/DWWD1	0.045 0.4	2	0	whole fruit	2.53 (2.55, 2.51)	
		S-7.5, drench 50 EC, spray wax N/DWWD2	0.045 0.4	2	0	whole fruit	1.88 (1.88, 1.88)	
	Orange / Navelina	50 EC, spray wax N/WWD1	0.4	1	0	whole fruit	1.55 (1.56. 1.54)	
		50 EC, spray wax N/WWD2	0.4	1	0	whole fruit	1.63 (1.61, 1.66)	
TRC02-3 Valencia, Spain 2002	Orange / Valencia Late	50EC, spray wax	0.4	1	0	whole fruit	1.46 (1.51, 1.05, 1.68, 1.59)	Not analysed

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
		50EC, spray	0.15	1	0	whole fruit	0.360 (0.281, 0.391, 0.358, 0.408)	
		S-7.5, spray	0.135	1	0	whole fruit	0.331 (0.310, 0.320, 0.368, 0.327)	
		50EC, drench	0.05	1	0	whole fruit	1.721 (1.23, 2.00, 1.95, 1.70)	
		S-7.5, drench	0.045	1	0	whole fruit	2.33 (2.11, 2.55, 2.46, 2.20)	
TRC01-5 R1 Valencia, Spain 2001	Orange / Navelina	25% FD, fogging	0.006 kg ai/ton	1	1 5 5 15 30	whole fruit peel pulp whole fruit whole fruit	0.451 0.297 <0.02 0.229 0.277	Not analysed
TRC01-5 R2 Valencia, Spain 2001	Orange / Navel	25% FD, fogging	0.006 kg ai/ton	1	1 5 5	whole fruit peel pulp	0.337 0.446 <0.02	Not analysed
TRC01-5 R3 Picaña, Spain 2001	Orange / Navel	25% FD, fogging	0.006 kg ai/ton	1	1 5 5 15 30	whole fruit peel pulp whole fruit whole fruit	0.829 2.007 0.029 0.640 0.301	
TRC01-5 R4 Picaña, Spain 2001	Orange / Navelina	25% FD, fogging	0.006 kg ai/ton	1	1 5 5	whole fruit peel pulp	0.563 2.267 0.036	
TRC01-5 R5 Valencia, Spain 2001	Mandarin / Satsuma	25% FD, fogging	0.006 kg ai/ton	1	1 5 5 15 30	whole fruit peel pulp whole fruit whole fruit	0.390 0.687 0.043 0.253 <0.05	
TRC01-5 R6 Valencia, Spain 2001	Mandarin / Clemenules	25% FD, fogging	0.006 kg ai/ton	1	1 5 5	whole fruit peel pulp	0.593 0.852 <0.02	
TRC01-5 R7 Picaña, Spain 2001	Mandarin / Clemenules	25% FD, fogging	0.006 kg ai/ton	1	1 5 5 15 30	whole fruit peel pulp whole fruit whole fruit	1.533 2.471 <0.02 0.990 0.670	
TRC01-5 R8 Picaña, Spain 2001	Mandarin / Satsuma	25% FD, fogging	0.006 kg ai/ton	1	1 5 5	whole fruit peel pulp	0.950 1.773 <0.02	
TRC03-6 Valencia, Spain 2003	Orange / Valencia Late	2 g/L, wax	0.2	1	0	whole fruit	0.525	
		200 SC, spray wax	0.2	1	0	whole fruit	0.466	
		200 SC, spray wax	0.3	1	0	whole fruit	0.848	
		200 SC, spray wax	0.2	1	0	whole fruit	1.09	
S04S005R La Font d'en Carrós, Valencia, Spain 2004	Mandarin / Fortunas	200 SL, drench	0.04	1	0	Whole fruit	1.43	Not analysed
		200 SL, drench	0.05	1	0	whole fruit	1.42	
S04S006R La Font d'en Carrós, Valencia, Spain 2004	Mandarin / Ortanique	200 SL, drench	0.04	1	0	whole fruit	0.769	Not analysed
		200 SL, drench	0.05	1	0	whole fruit	1.46	
S04S001R	Mandarin /	400 SC, spray wax	0.2	1	0	whole fruit	0.617	Not analysed

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
La Font d'en Carrós, Valencia, Spain 2004	Fortunas	400 SC, spray wax	0.3	1	0	whole fruit	0.842	
		500 EC, spray wax	0.2	1	0	whole fruit	0.763	
		500 EC, spray wax	0.3	1	0	whole fruit	1.29	
		400 SC, drench 400 SC, spray wax	0.05 0.3	2	0	whole fruit	1.96	
S04S002R La Font d'en Carrós, Valencia, Spain 2004	Mandarin / Ortanique	400 SC, spray wax	0.2	1	0	whole fruit	0.608	Not analysed
		400 SC, spray wax	0.3	1	0	whole fruit	1.22	
		S-7.5 spray wax	0.2	1	0	whole fruit	0.617	
		S-7.5 spray wax	0.3	1	0	whole fruit	0.920	
		400 SC, drench 400 SC, spray wax	0.05 0.3	2	0	whole fruit	1.78	
S04S003R La Font d'en Carrós, Valencia, Spain 2004	Mandarin / Fortunas	400 SC, drench	0.04	1	0	whole fruit	0.969	Not analysed
		400 SC, drench	0.05	1	0	whole fruit	1.30	
		S-7.5, drench	0.04	1	0	whole fruit	2.40	
		S-7.5 drench	0.05	1	0	whole fruit	4.84	
S04S004R La Font d'en Carrós, Valencia, Spain 2004	Mandarin / Ortanique	400 SC, drench	0.04	1	0	whole fruit	0.677	Not analysed
		400 SC, drench	0.05	1	0	whole fruit	1.51	
		S-7.5, drench	0.04	1	0	whole fruit	1.08	
		S-7.5 drench	0.05	1	0	whole fruit	2.19	
TRC04-9 La Font d'en Carrós, Valencia, Spain 2004	Orange / Valencia	500 EC, spray wax	0.2	1	0	whole fruit	0.555	Not analysed
		500 EC, spray wax	0.3	1	0	whole fruit	0.734	
		525 SC, spray wax	0.2	1	0	whole fruit	0.965	
	Orange / Lane late	500 EC, spray wax	0.2	1	0	whole fruit	0.935	
		500 EC, spray wax	0.3	1	0	whole fruit	1.100	
		525 SC, spray wax	0.2	1	0	whole fruit	0.772	
TRC04-10 La Font d'en Carrós, Valencia, Spain 2004	Orange / Valencia	500 EC, spray wax	0.2	1	0	whole fruit	0.555	Not analysed
		500 EC, spray wax	0.3	1	0	whole fruit	0.734	
		400SC, spray wax	0.2	1	0	whole fruit	0.910	
		400SC, spray wax	0.3	1	0	whole fruit	1.46	
		400 SC, drench 400SC, spray wax	0.05 0.3	2	0	whole fruit	2.60	
	Orange / Lane late	500 EC, spray wax	0.4	1	0	whole fruit	0.935	
		500 EC, spray wax	0.5	1	0	whole fruit	1.10	
		400SC, spray wax	0.4	1	0	whole fruit	1.14	
		400SC, spray wax	0.5	1	0	whole fruit	1.52	
		400 SC, drench 400SC, spray wax	0.05 0.3	2	0	whole fruit	2.15	
TRC04-11 La Font d'en Carrós, Valencia, Spain 2004	Orange / Valencia	S-7.5, drench	0.04	1	0	whole fruit	2.27	Not analysed
		S-7.5, drench	0.05	1	0	whole fruit	2.59	
		200 SL, drench	0.04	1	0	whole fruit	2.08	
		200 SL, drench	0.05	1	0	whole fruit	2.89	
	Orange / Lane late	S-7.5, drench	0.04	1	0	whole fruit	2.53	
		S-7.5, drench	0.05	1	0	whole fruit	4.95	
		200 SL, drench	0.04	1	0	whole fruit	2.00	
		200 SL, drench	0.05	1	0	whole fruit	3.12	
TRC04-12 La Font d'en	Orange / Valencia	S-7.5, drench	0.04	1	0	whole fruit	2.27	Not analysed
		S-7.5, drench	0.05	1	0	whole fruit	2.59	

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
Carrós, Valencia, Spain 2004		200 SL, drench	0.04	1	0	whole fruit	1.09	
		200 SL, drench	0.05	1	0	whole fruit	1.36	
	Orange / Lane late	S-7.5, drench	0.04	1	0	whole fruit	2.53	
		S-7.5, drench	0.05	1	0	whole fruit	4.95	
		200 SL, drench	0.04	1	0	whole fruit	1.40	
		200 SL, drench	0.05	1	0	whole fruit	2.06	
S11-03184-01 41927, Mairena, Andalusia, Spain 2011	mandarins / Oronules	400 EC, dip	0.05	1	0	whole fruit pulp	2.66 0.177	0.018 <0.01
					28	whole fruit pulp	1.53 0.070	0.059 <0.01
					57	whole fruit pulp	1.68 0.082	0.096 <0.01
					86	whole fruit pulp	1.34 0.122	0.127 0.014
S11-03184-02 46650, Canals, Valencia, Spain 2011	mandarins / Clemenules	400 EC, drench	0.05	1	0	whole fruit pulp	2.26 0.020	0.012 <0.01
					30	whole fruit pulp	1.69 0.067	0.065 <0.01
					61	whole fruit pulp	1.03 0.135	0.077 0.017
S11-03184-03 57200, Kolchiko, Thessaloniki, Geece 2012	mandains / Clementins	400 EC, dip	0.05	1	0	whole fruit pulp	1.77 0.368	<0.01 <0.01
S11-03184-04 40016, San Giorgio de Piano, Bologna, Italy 2011	mandarins / Simeto	400 EC, drench	0.05	1	0	whole fruit pulp	3.34 0.477	0.020 <0.01
S11-03184-05 41927, Mairena, Andalusia, Spain 2011	orange / New Hall	400 EC, dip	0.05	1	0	whole fruit pulp	2.90 0.120	<0.01 <0.01
					28	whole fruit pulp	1.71 0.022	0.040 <0.01
					57	whole fruit pulp	1.08 <0.01	0.061 <0.01
					86	whole fruit pulp	0.908 <0.01	<0.01 <0.01
S11-03184-06 46650, Canals, Valencia, Spain 2011	orange / Navelina	400 EC, drench	0.05	1	0	whole fruit pulp	2.81 0.087	<0.01 <0.01
					30	whole fruit pulp	1.47 0.017	0.043 <0.01
					61	whole fruit pulp	0.524 0.035	0.060 0.016
					85	whole fruit pulp	0.564 0.147	0.084 0.018
S11-03184-07 57200, Kolchiko, Thessaloniki, Geece 2012	orange / Kyno	400 EC, dip	0.05	1	0	whole fruit pulp	1.56 0.066	0.813 0.112
S11-03184-08 40016, San Giorgio de Piano, Bologna, Italy 2011	orange / Navel	400 EC, drench	0.05	1	0	whole fruit pulp	2.46 0.689	2.03 0.907

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
S11-03185-01 25198, Lleida, Barcelona, Spain 2011	mandarin / Satsuma	400 EC, spray	0.15	1	0	whole fruit pulp	0.373 0.012	0.012 <0.01
S11-03185-02 25198, Lleida, Barcelona, Spain 2011	mandarin / Orogrande	400 EC, spray	0.15	1	0	whole fruit pulp	0.530 0.010	0.021 <0.01
S11-03185-03 95040, Belpasso, Sicily, Italy 2012	mandarin / Avana	400 EC, spray	0.15	1	0	whole fruit pulp	0.709 <0.01	0.012 <0.01
S11-03185-04 40003, Agia, Larissa, Greece 2012	mandarin / Ortanik	400 EC, spray	0.15	1	0	whole fruit pulp	1.83 0.166	0.037 <0.01
S11-03185-05 25198, Lleida, Barcelona, Spain 2011	orange / Navelina	400 EC, spray	0.15	1	0	whole fruit pulp	0.866 0.049	<0.01 <0.01
S11-03185-06 25198, Lleida, Barcelona, Spain 2011	orange / New Hall	400 EC, spray	0.15	1	0	whole fruit pulp	0.529 0.048	<0.01 <0.01
S11-03185-07 95040, Belpasso, Sicily, Italy 2012	orange / Tarocco Sciara	400 EC, spray	0.15	1	0	whole fruit pulp	0.198 <0.01	<0.01 <0.01
S11-03185-08 40003, Agia, Larissa, Greece 2012	orange / Merlin	400 EC, spray	0.15	1	0	whole fruit pulp	2.40 0.044	0.018 <0.01
S11-03186-01 41805, Mairena del Aljarafe, Andalusia, Spain 2011	mandarin / Oronules	7.5 SL, dip	0.045	1	0	whole fruit pulp	1.13 0.167	<0.01 <0.01
S11-03186-02 46650, Canals, Valencia, Spain 2011	mandarin / Clemenules	7.5 SL, dench	0.045	1	0	whole fruit pulp	2.12 0.081	0.019 <0.01
S11-03186-03 57200, Kolchiko, Thessaloniki, Greece 2011	mandarin / Clementines	7.5 SL, dip	0.045	1	0	whole fruit pulp	2.72 0.430	0.027 <0.01
S11-03186-04 40016, San Giorgio di Piano, Bologna, Italy 2011	mandarin / Simeto	7.5 SL, dench	0.045	1	0	whole fruit pulp	2.76 0.698	0.014 <0.01
S11-03186-05: 41805, Mairena del Aljarafe, Andalusia, Spain 2011	orange / New Hall	7.5 SL, dip	0.045	1	0	whole fruits pulp	1.39 0.160	<0.01 <0.01
S11-03186-06 46650, Canals, Valencia, Spain 2011	orange / Navelina	7.5 SL, dench	0.045	1	0	whole fruit pulp	1.39 0.068	<0.01 <0.01

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
S11-03186-07 57200, Kolchiko, Thessaloniki, Greece 2011	orange / Kyno	7.5 SL, dip	0.045	1	0	whole fruit pulp	2.58 0.210	<0.01 <0.01
S11-03186-08 40016, San Giorgio di Piano, Bologna, Italy 2011	orange / Navel	7.5 SL, dench	0.045	1	0	whole fruit pulp	1.84 0.370	<0.01 <0.01
S11-03186-05: 41805, Mairena del Aljarafe, Andalusia, Spain 2011	orange / New Hall	7.5 SL, dip	0.045	1	0	whole fruit pulp	1.39 0.160	<0.01 <0.01
S11-03186-06 46650, Canals, Valencia, Spain 2011	orange / Navelina	7.5 SL, dench	0.045	1	0	whole fruit pulp	1.39 0.068	<0.01 <0.01
S11-03186-07 57200, Kolchiko, Thessaloniki, Greece 2011	orange / Kyno	7.5 SL, dip	0.045	1	0	whole fruit pulp	2.58 0.210	<0.01 <0.01
S11-03186-08 40016, San Giorgio di Piano, Bologna, Italy 2011	orange / Navel	7.5 SL, dench	0.045	1	0	whole fruit pulp	1.84 0.370	<0.01 <0.01
S11-03187-01: 25198, Lleida, Barcelona, Spain 2011	mandarin / Satsuma	7.5 SL, spray	0.15	1	0	whole fruit pulp	0.547 0.022	0.022 <0.01
S11-03187-02 25198, Lleida, Barcelona, Spain 2011	mandarin / Orogrande	7.5 SL, spray	0.15	1	0	whole fruit pulp	1.16 0.019	0.032 <0.01
S11-03187-03 95040, Belpasso, Sicily, Italy 2012	mandarin / Avana	7.5 SL, spray	0.15	1	0	whole fruit pulp	1.05 0.016	0.019 <0.01
S11-03187-04 40003, Agia, Larissa, Greece 2012	mandarin / Ortanik	7.5 SL, spray	0.15	1	0	whole fruit pulp	1.84 0.974	0.036 0.019
S11-03187-05 25198, Lleida, Barcelona, Spain 2011	orange / Navelina	7.5 SL, spray	0.15	1	0	whole fruit pulp	0.228 0.027	<0.01 <0.01
S11-03187-06 46181, 25198, Lleida, Barcelona, Spain 2011	orange / New Hall	7.5 SL, spray	0.15	1	0	whole fruit pulp	0.328 0.032	<0.01 <0.01
S11-03187-07 95040, Belpasso, Sicily, Italy 2012	orange / Tarocco Sciara	7.5 SL, spray	0.15	1	0	whole fruit pulp	1.08 <0.01	<0.01 <0.01

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
S11-03187-08 40003, Agia, Larissa, Greece 2012	orange / Merlin	7.5 SL, spray	0.15	1	0	whole fruit pulp	1.34 0.046	0.022 <0.01
S11-03188-01: 41805, Mairena del Aljarafe, Andalusia, Spain 2011	mandarin / Oronules	500 EC, dip	0.05	1	0	whole fruit pulp	2.37 0.183	0.020 <0.01
S11-03188-02 46650, Canals, Valencia, Spain 2011	mandarin / Clemenules	500 EC, drench	0.05	1	0	whole fruit pulp	1.48 0.014	0.010 <0.01
S11-03188-03: 57200, Kolchiko, Thessaloniki, Greece 2011	mandarin / Clementines	500 EC, dip	0.05	1	0	whole fruit pulp	2.44 0.272	0.016 <0.01
S11-03188-04 40016, San Giorgio di Piano, Bologna, Italy 2011	mandarin / Simeto	500 EC, drench	0.05	1	0	whole fruit pulp	2.55 0.395	0.010 <0.01
S11-03188-05: 41805, Mairena del Aljarafe, Andalusia, Spain 2011	orange / New Hall	500 EC, dip	0.05	1	0	whole fruit pulp	1.89 0.189	<0.01 <0.01
S11-03188-06 46650, Canals, Valencia, Spain 2011	orange / Navelina	500 EC, drench	0.05	1	0	whole fruit pulp	2.17 0.078	<0.01 <0.01
S11-03188-07: 57200, Kolchiko, Thessaloniki, Greece 2011	orange / Kyno	500 EC, dip	0.05	1	0	whole fruit pulp	2.14 0.142	0.013 <0.01
S11-03188-08 40016, San Giorgio di Piano, Bologna, Italy 2011	orange / Navel	500 EC, drench	0.05	1	0	whole fruit pulp	2.69 0.122	<0.01 <0.01
S11-03189-01 25198, Lleida, Barcelona, Spain 2011	mandarin / Satsuma	500EC, spray	0.15	1	0	whole fruit pulp	0.212 0.011	<0.01 <0.01
S11-03189-02 25198, Lleida, Barcelona, Spain 2011	mandarin / Orogrande	500EC, spray	0.15	1	0	whole fruit pulp	0.398 <0.01	0.017 <0.01
S11-03189-03 95040, Belpasso, Sicily, Italy 2012	mandarin / Avana	500EC, spray	0.15	1	0	whole fruit pulp	0.996 0.013	0.016 <0.01
S11-03189-04 40003, Agia, Larissa, Greece 2012	mandarin / Ortanik	500EC, spray	0.15	1	0	whole fruit pulp	2.067 0.711	0.032 <0.01

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
S11-03189-05 25198, Lleida, Barcelona, Spain 2011	orange / Navelina	500EC, spray	0.15	1	0	whole fruit pulp	0.514 0.055	<0.01 <0.01
S11-03189-06 46181, 25198, Lleida, Barcelona, Spain 2011	orange / New Hall	500EC, spray	0.15	1	0	whole fruit pulp	0.644 0.044	<0.01 <0.01
S11-03189-07 95040, Belpasso, Sicily, Italy 2012	orange / Tarocco Sciara	500EC, spray	0.15	1	0	whole fruit pulp	0.962 <0.01	<0.01 <0.01
S11-03189-08 40003, Agia, Larissa, Greece 2012	orange / Merlin	500EC, spray	0.15	1	0	whole fruit pulp	2.106 0.032	0.018 <0.01
S11-03190-02 95040, Belpasso, Sicily, Italy 2012	mandarin / Avana	500EC, wax spray	0.2	1	0	whole fruit pulp	0.404 <0.01	<0.01 <0.01
S11-03190-03 40003, Agia, Larissa, Greece 2012	mandarin / Ortanik	500EC, wax spray	0.2	1	0	whole fruit pulp	0.299 0.039	<0.01 <0.01
S11-03190-05 95040, Belpasso, Sicily, Italy 2012	orange / Tarocco Sciara	500EC, wax spray	0.2	1	0	whole fruit pulp	0.518 <0.01	<0.01 <0.01
S11-03190-06 40003, Agia, Larissa, Greece 2012	orange / Merlin	500EC, wax spray	0.2	1	0	whole fruit pulp	0.492 <0.01	<0.01 <0.01
S11-03190-07 25198, Lleida, Barcelona, Spain 2012	mandarin / Fortuna	500EC, wax spray	0.2	1	0	whole fruit pulp	0.197 0.013	<0.01 <0.01
S11-03190-08 25198, Lleida, Barcelona, Spain 2012	orange / Navelina	500EC, wax spray	0.2	1	0	whole fruit pulp	0.388 <0.01	<0.01 <0.01
S16-06757-01 25100 Aigio, Achaia, Greece 2017	Orange / Navel	500 EC, drench 500 EC, wax	0.075 0.20	1 1	0	whole fruits pulp	4.2 0.07	0.02 <0.01
S16-06757-02 50932, Plati, Imathia, Greece 2017	Orange / Navelines	500 EC, drench 500 EC, wax	0.075 0.20	1 1	0	whole fruit pulp	2.7 0.06	0.02 <0.01
S16-06757-03 95032, Belpasso, Sicily, Italy 2017	Orange / Tarocco	500 EC, drench 500 EC, wax	0.075 0.20	1 1	0	whole fruit pulp	3.0 0.07	<0.01 <0.01
S16-06757-04 95047, Paterno, Sicily, Italy 2017	Orange / Moro	500 EC, drench 500 EC, wax	0.075 0.20	1 1	0	whole fruit pulp	4.4 0.26 (0.24, 0.28)	0.02 <0.01

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Residues (mg/kg)	
							Imazalil	R014821
S16-06757-05 46469, Beniparrell, Valencia, Spain 2016	Orange / Navelina	500 EC, drench 500 EC, wax	0.075 0.20	1 1	0	whole fruit pulp	3.6 0.06	<0.01 <0.01
S16-06757-06 25003, Lleida, Catalunya, Spain 2017	Orange / Navelina	500 EC, drench 500 EC, wax	0.075 0.20	1 1	0	whole fruit pulp	4.8 0.13 (0.12, 0.13)	0.01 <0.01
S17-07771-01 59032, Plati, Imathia, Greece, 2017	Orange / Merlin	500 EC, drench 500 EC, wax	0.057 0.46	1 1	0	whole fruit pulp	3.0 0.17	<0.01 <0.01
S17-07771-02 46469, Beniparrell, Valencia, Spain, 2017	Orange / Navelina	500 EC, drench 500 EC, wax	0.063 0.20	1 1	0	whole fruit pulp	2.5 0.11	0.01 <0.01
S17-07771-03 46469, Beniparrell, Valencia, Spain, 2018	Orange / Lane Late	500 EC, drench 500 EC, wax	0.065 0.21	1 1	0	whole fruit pulp	3.4 (3.3, 3.4) 0.21 (0.21, 0.20)	0.03 <0.01

Australia

Three post-harvest supervised trials were conducted in Australia on oranges during 2005 (Ridley, 2005, DEGROOT/GLP/04/03-1a). The test substance was a suspension concentration formulation containing 200 g/L of imazalil. Three treatment methods were assessed: dipping (0.04-0.05 kg ai/hL), non-recovery spray (0.1-0.2 kg ai/hL) and spray wax (0.2-0.3 kg ai/hL). Oranges were collected immediately after treatment (0-DAA) and comprised of at least 12 fruit and weighing at least 2 kg.

Residues of imazalil were quantified using a published multi-residue LC/MS method employing acetonitrile extraction/partitioning and dispersive solid phase extraction (Journal of AOAC Intern. Vol. 86, no. 2, 2003). The LOQ of the method was 0.2 mg/kg. The storage intervals from collection to extraction did not exceed 46 days.

Table 49 Magnitude of the residues of imazalil in oranges following post-harvest treatment from trials conducted in Australia

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Imazalil Residues (mg/kg)
Australia GAP	Citrus fruit	Dip, drench	0.075	1 combined with wax application	0		
		Spray	0.1	1 combined with wax application	0		
		Wax (diluted)	0.2	1 combined with dip, drench or spray application	0		
		Wax (Ready to use)	0.4	1	0		
4666 West Gosford NSW 2250, Australia 2005	Orange / Navel	200 SC, dip	0.04	1	0	whole fruit	3.2
			0.05	1	0	peel pulp whole fruit	27.0 0.3 10.0
4668	Orange /	200 SC, spray	0.1	1	0	whole fruit	5.7

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Imazalil Residues (mg/kg)
Kulnura NSW 2250, Australia 2005	Navel		0.2	1	0	whole fruit	5.9
4670 Kulnura NSW 2250, Australia 2005	Orange / Navel	200SC, spray wax	0.2	1	0	whole fruit	2.0
			0.3	1	0	peel pulp whole fruit	6.2 <0.2 0.3

New Zealand

Three post-harvest supervised trials were conducted in New Zealand, during 2002, where satsuma mandarins received post-harvest spray applications of a water soluble powder formulation containing 750 g/kg of imazalil (Steven, 2003, report no. not available). The post-harvest spray application was applied at a concentration of 0.075 kg ai/hL or twice this rate, 0.150 kg ai/hL. Treated mandarins were collected immediately after the wax application (0-DAA) up to 20 DAA. and comprised of at least 12 fruit and weighing a minimum of 1 kg at each interval. Residues of imazalil were quantified using the GC multi-residue method CD.SC.3019. The LOQ of the method was 0.02 mg/kg. The storage intervals from collection to extraction did not exceed 25 days.

Table 50 Magnitude of the residues of imazalil in/on oranges following post-harvest treatment from trials conducted in New Zealand

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Imazalil Residues (mg/kg)
Gisborne, New Zealand 2002	Satsuma mandarin / Miyagava	75 WSP, spray	0.075	1	0 0 0 3 3 3 5 5 5 10 10 10 15 15 15 20 20 20	peel pulp whole fruit[a] peel pulp whole fruit peel pulp whole fruit peel pulp whole fruit peel pulp whole fruit peel pulp whole fruit	0.39 <0.02 0.11 0.47 <0.02 0.12 0.45 <0.02 0.12 0.38 <0.02 0.10 0.47 <0.02 0.14 0.34 <0.02 0.10
			0.150	1	0 0 0 3 3 3 5 5 5 10 10 10 15 15 15 20 20 20	peel pulp whole fruit peel pulp whole fruit peel pulp whole fruit peel pulp whole fruit peel pulp whole fruit peel pulp whole fruit	0.54 <0.02 0.14 0.87 0.02 0.23 0.88 <0.02 0.22 0.86 <0.02 0.23 0.93 <0.02 0.26 0.82 0.025 0.23

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Imazalil Residues (mg/kg)
Tauranga New Zealand 2002	Satsuma mandarin / Silverhill	75 WSP, spray	0.075	1	0	peel	0.089
					0	pulp	<0.02
					0	whole fruit	0.032
					3	peel	0.063
					3	pulp	<0.02
					3	whole fruit	0.023
					5	peel	0.047
					5	pulp	<0.02
					5	whole fruit	0.017
					10	peel	0.046
					10	pulp	<0.02
					10	whole fruit	0.016
					15	peel	0.047
					15	pulp	<0.02
					15	whole fruit	0.017
					20	peel	0.047
					20	pulp	<0.02
					20	whole fruit	0.017
			0.150	1	0	peel	0.16
					0	pulp	<0.02
					0	whole fruit	0.06
					3	peel	0.19
					3	pulp	<0.02
					3	whole fruit	0.07
					5	peel	0.07
					5	pulp	<0.02
					5	whole fruit	0.03
					10	peel	0.07
					10	pulp	<0.02
					10	whole fruit	0.03
					15	peel	0.10
					15	pulp	<0.02
					15	whole fruit	0.04
					20	peel	0.08
					20	pulp	<0.02
					20	whole fruit	0.03
Kerikeri New Zealand 2002	Satsuma mandarin / Miho	75 WSP, spray	0.075	1	0	peel	0.90
					0	pulp	<0.02
					0	whole fruit	0.27
					3	peel	0.78
					3	pulp	<0.02
					3	whole fruit	0.24
					5	peel	1.2
					5	pulp	<0.02
					5	whole fruit	0.36
					10	peel	0.81
					10	pulp	<0.02
					10	whole fruit	0.25
					15	peel	0.89
					15	pulp	<0.02
					15	whole fruit	0.28
					20	peel	0.79
					20	pulp	<0.02
					20	whole fruit	0.25

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	No.	DAA (days)	Portion analysed	Imazalil Residues (mg/kg)
			0.150	1	0	peel	2.7
					0	pulp	0.12
					0	whole fruit	0.85
					3	peel	2.2
					3	pulp	0.08
					3	whole fruit	0.70
					5	peel	2.9
					5	pulp	0.07
					5	whole fruit	0.87
					10	peel	1.65
					10	pulp	0.04
					10	whole fruit	0.49
					15	peel	2.0
					15	pulp	0.04
					15	whole fruit	0.60
					20	peel	1.9
					20	pulp	0.03
					20	whole fruit	0.59

[a] Residues in whole fruit were determined based on the individual residues in peel and pulp as well as the weight of each fraction

Grapefruits

United States of America

Ten post-harvest trials in the USA were conducted during 1987-1988, where emulsifiable concentrate formulations, containing 500 g/L or 68 g/L of imazalil were applied to grapefruits either via pack wax, water spray, or a combination of both at total rates of 0.1-0.4 kg ai/hL. All samples were collected immediately after treatment (0-DAA).

A GC/MS method was used to determine residues of imazalil and the metabolite R014821 which reported an LOQ of 0.05 mg/kg/analyte.

The maximum duration in frozen storage was 60 days.

Table 51 Magnitude of the residues of imazalil and R014821 in/on grapefruits following post-harvest treatment from trials conducted in the USA

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg i/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
USA GAP		Dip, drench	0.075	1 combined with wax application			
		Spray	0.1	1 combined with wax application			
		Wax (diluted)	0.2	1 combined with dip, drench or spray application			
		Wax (Ready to use)	0.4	1			
F114-P/F115-P Lake Alfred, Florida, USA 1988	Grapefruit / Marsh	500 EC, pack wax	0.4	1	peel pulp whole fruit[a]	16.11 0.47 5.07	0.44 <0.05 0.15

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg i/hL)	No.	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
F831-0/F832-0 Riverside, CA, USA 1987	Grapefruit	500 EC, pack wax	0.2	1	peel pulp whole fruit[a]	2.57 0.05 0.84	0.16 <0.05 0.06
F833-0/F834-0 Riverside, CA, USA 1987	Grapefruit	500 EC, water spray 500 EC, pack wax	0.1 0.1	2	peel pulp whole fruit[a]	2.69 0.10 0.85	0.24 <0.05 0.08
F835-0/F836-0 Riverside, CA, USA 1987	Grapefruit	500 EC, water spray 500 EC, pack wax	0.1 0.1	2	peel pulp whole fruit[a]	2.92 0.10 0.95	0.12 <0.05 0.04
F837-0/F841-0 Riverside, CA, USA 1987	Grapefruit	68 EC, pack wax	0.2	1	peel pulp whole fruit[a]	2.05 0.08 0.68	0.38 <0.05 0.13
F844-0/F845-0 Riverside, CA, USA 1987	Grapefruit	500 EC, pack wax	0.2	1	peel pulp whole fruit[a]	3.02 0.14 1.22	0.10 <0.05 0.04
F987-0/F988-0 Arlington Heights, CA, USA 1987	Grapefruit	500 EC, pack wax	0.2	1	peel pulp whole fruit[a]	4.26 0.10 1.49	0.40 <0.05 0.14
F989-0/F991-0 Riverside, CA, USA 1987	Grapefruit	500EC, water spray	0.1	1	peel pulp whole fruit[a]	1.74 0.09 0.61	0.23 <0.05 0.08
F993-0/F994-0 Riverside, CA, USA 1987	Grapefruit	500 EC, water spray 500 EC, pack wax	0.1 0.1	2	peel pulp whole fruit[a]	2.36 0.10 0.84	0.20 <0.05 0.07
F995-0/F996-0 Riverside, CA, USA 1987	Grapefruit	500 EC, pack wax	0.1	1	peel pulp whole fruit[a]	1.43 0.08 0.60	0.13 <0.05 0.05
F654-0/F655-0 Vero Beach, Florida, USA 1987	Grapefruit	500 EC, water spray	0.1	1	peel pulp whole fruit[a]	1.74 <0.05 0.50	0.15 <0.05 0.04

[a] Whole fruit residue calculated based on peel and pulp individual weights and residues

Banana

Europe

Four trials were conducted in Spain in 2013 (Gimeno, 2013, TRC13-058). The test substance was a soluble granule formulation containing 750g/kg of imazalil. In each of the trials imazalil was applied once as a dip application at a rate of 0.06 kg ai/hL. At least 24 individual banana fruits (finger) weighing a minimum of 2 kg were collected on the day of application and at 14, 21, 28 and 34 days after application.

At all sampling intervals, except day 34, whole banana fruits were separated into peel and pulp, analysed individually from which residues in the whole fruit were determined. In addition, whole bananas sampled 0, 14 and 34 DAA were also analysed whole. The residues of imazalil and the metabolite R014821 were quantified using the LC-MS/MS multi residue method L 00.00-34 (formerly DFG method S-19), with an LOQ of 0.01 mg/kg/analyte.

Samples were stored deep-frozen for a maximum of 4 months (115 days).

Table 52 Magnitude of the residues of imazalil and R014821 in/on bananas following post-harvest treatment from trials conducted in Europe

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	DAA (days)	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
Spain		Dip, drench	0.0375	0			
TRC13-058 R1 46192 Monserrat,	Banana / Pequeña	75 SG, Dip	0.06	0	peel	5.86	0.05
				0	pulp	0.05	<0.01

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	DAA (days)	Portion analysed	Residues (mg/kg)	
						Imazalil	R014821
Valencia, Spain 2013[b]	enana			0	whole fruit[a]	2.37	0.02
				0	whole fruit	2.39	0.02
				14	peel	7.45	0.08
				14	pulp	0.14	<0.01
				14	whole fruit[a]	3.10	0.03
				14	whole fruit	2.63	0.04
				21	peel	7.87	0.17
				21	pulp	0.31	<0.01
				21	whole fruit[a]	2.64	0.05
				28	peel	7.59	0.17
				28	pulp	0.42	<0.01
				28	whole fruit[a]	2.56	0.05
				34	whole fruit	2.12	0.04
TRC13-058 R2 46192 Monserrat, Valencia, Spain 2013[b]	Banana / Gran enana	75 SG, Dip	0.06	0	peel	7.57	0.05
				0	pulp	0.57	<0.01
				0	whole fruit[a]	3.34	0.02
				0	whole fruit	3.31	0.03
				14	peel	6.97	0.07
				14	pulp	0.26	<0.01
				14	whole fruit[a]	3.14	0.03
				14	whole fruit	3.27	0.04
				21	peel	8.64	0.14
				21	pulp	0.54	<0.01
				21	whole fruit[a]	2.82	0.04
				28	peel	8.98	0.16
				28	pulp	0.93	<0.01
				28	whole fruit[a]	3.14	0.05
				34	whole fruit	2.61	0.03
TRC13-058 R3 46013 Marcavalencia, Valencia, Spain 2013 [b]	Banana / Brier	75 SG, Dip	0.06	0	peel	6.63	0.05
				0	pulp	0.72	<0.01
				0	whole fruit[a]	2.94	0.02
				0	whole fruit	2.33	0.03
				14	peel	6.43	0.07
				14	pulp	0.08	<0.01
				14	whole fruit	2.57	0.03
				14	whole fruit	2.54	0.05
				21	peel	8.64	0.13
				21	pulp	0.14	<0.01
				21	whole fruit[a]	2.55	0.04
				28	peel	9.15	0.13
				28	pulp	0.23	<0.01
				28	whole fruit[a]	2.68	0.04
				34	whole fruit	2.80	0.03
TRC13-058 R4 46013 Marcavalencia, Valencia, Spain 2013[b]	Banana / Gruesa palmera	75 SG, Dip	0.06	0	peel	5.56	0.05
				0	pulp	0.08	<0.01
				0	whole fruit[a]	2.38	0.02
				0	whole fruit	2.21	0.02
				14	peel	6.56	0.08
				14	pulp	0.05	<0.01
				14	whole fruit[a]	2.90	0.04
				14	whole fruit	2.57	0.05
				21	peel	7.60	0.14
				21	pulp	0.06	<0.01
				21	whole fruit[a]	2.36	0.05
				28	peel	8.72	0.15
				28	pulp	0.15	<0.01
				28	whole fruit[a]	2.73	0.05
				34	whole fruit	2.71	0.04

[a] Whole fruit residue calculated based on peel and pulp individual weights and residues

[b] Trials R1/R2 and R3/R4 were deemed dependent based on timing and location of treatment

Central America

A total of six residue trials were carried out in Central America (Honduras and Panama) in 1991 (Garnier, 1991, AGR-3). The test substance was a soluble powder (SP) formulation containing 750g/kg of imazalil. In each of the trials imazalil was applied once as a dip-static, drench, dip-dynamic or non-recovery spray at application rates of 0.03-0.09 kg ai/hL. Samples of green bananas were taken at 7 or 14 days after application and samples of mature bananas were taken at 22, 25 or 27 days after application.

Whole green and yellow banana fruits and pulp were analysed for imazalil and the metabolite R014821 using a GC-method, not further detailed in the report with an LOQ of 0.05 mg/kg/analyte.

Samples were stored deep-frozen for a maximum of 108 days.

Table 53 Magnitude of the residues of imazalil and R014821 in/on bananas following post-harvest treatment from trials conducted in Central American countries

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	Growth Stage at treatment	Portion analysed	DAA (days)	Residues (mg/kg)	
							Imazalil	R014821
Spain		Dip, drench	0.0375			0		
AGR3/EXP1 Bocas, Panama 1991	Banana	Dip-static for 3 seconds	0.03	green fruit	whole fruit	14	1.014	<0.05
				mature	pulp	22	<0.05	<0.05
					whole fruit	22	0.746	<0.05
		Dip-static for 5 seconds	0.03	green fruit	whole fruit	14	1.08	<0.05
				mature	pulp	22	0.063	<0.05
					whole fruit	22	1.683	<0.05
		Dip-static for 15 seconds	0.03	green fruit	whole fruit	14	1.244	<0.05
				mature	pulp	22	<0.05	<0.05
					whole fruit	22	1.296	<0.05
		Dip-static for 30 seconds	0.03	green fruit	whole fruit	14	0.996	<0.05
				mature	pulp	22	<0.05	<0.05
					whole fruit	22	1.693	<0.05
		Dip-static for 60 seconds	0.03	green fruit	whole fruit	14	1.018	<0.05
				mature	pulp	22	<0.05	<0.05
					whole fruit	22	1.12	<0.05
		Dip-static for 300 seconds	0.03	green fruit	whole fruit	14	1.337	<0.05
				mature	pulp	22	<0.05	<0.05
					whole fruit	22	0.998	<0.05
		Dip-static for 3 seconds	0.06	green fruit	whole fruit	14	1.404	<0.05
				mature	pulp	22	0.068	<0.05
					whole fruit	22	2.335	<0.05
		Dip-static for 5 seconds	0.06	green fruit	whole fruit	14	1.855	<0.05
				mature	pulp	22	0.113	<0.05
					whole fruit	22	2.685	<0.05
		Dip-static for 15 seconds	0.06	green fruit	whole fruit	14	1.192	<0.05
				mature	pulp	22	0.091	<0.05
					whole fruit	22	2.906	<0.05
		Dip-static for 30 seconds	0.06	green fruit	whole fruit	14	1.547	<0.05
				mature	pulp	22	0.066	<0.05
					whole fruit	22	2.031	<0.05

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	Growth Stage at treatment	Portion analysed	DAA (days)	Residues (mg/kg)	
							Imazalil	R014821
		Dip-static for 60 seconds	0.06	green fruit	whole fruit	14	1.789	<0.05
				mature	pulp	22	0.066	<0.05
					whole fruit	22	1.652	<0.05
		Dip-static for 300 sec.	0.06	green fruit	whole fruit	14	2.324	<0.05
				mature	pulp	22	0.079	<0.05
					whole fruit	22	1.832	<0.05
AGR3/EXP2 Armuelles, Panama 1990	Banana	Drench for 3 seconds	0.04	mature	pulp	23	0.08	<0.05
					whole fruit		0.82	<0.05
		Drench for 3 seconds	0.04	mature	pulp	23	0.25	<0.05
					whole fruit		0.50	<0.05
		Drench for 3 seconds	0.04	mature	pulp	23	0.08	<0.05
					whole fruit		0.35	<0.05
		Drench for 30 seconds	0.04	mature	pulp	23	0.21	<0.05
					whole fruit		0.53	<0.05
		Drench for 60 seconds	0.04	mature	pulp	23	0.19	<0.05
					whole fruit		0.64	<0.05
		Drench for 300 sec.	0.04	mature	pulp	23	0.21	<0.05
					whole fruit		0.79	<0.05
		Drench for 600 sec.	0.04	mature	pulp	23	0.18	<0.05
					whole fruit		1.04	0.07
		Drench for 900 sec.	0.04	mature	pulp	23	0.19	<0.05
					whole fruit		0.81	0.06
		Drench for 3 seconds	0.06	mature	pulp	23	0.16	<0.05
					whole fruit		0.78	<0.05
		Drench for 30 seconds	0.06	mature	pulp	23	0.34	<0.05
					whole fruit		0.95	0.06
		Drench for 60 seconds	0.06	mature	pulp	23	0.21	<0.05
					whole fruit		0.93	0.08
		Drench for 300 sec.	0.06	mature	pulp	23	0.39	<0.05
					whole fruit		1.08	0.08
		Drench for 600 sec.	0.06	mature	pulp	23	0.37	<0.05
					whole fruit		1.14	0.22
		Drench for 900 sec.	0.06	mature	pulp	23	0.37	<0.05
					whole fruit		0.82	0.14
AGR3/EXP3 Tela, Honduras 1991	Banana	Dip-dynamic for 1 seconds	0.03	green fruit	whole fruit	14	0.306	<0.05
				mature	pulp	27	<0.05	<0.05
					whole fruit		0.197	<0.05
		Dip-dynamic for 5 seconds	0.03	green fruit	whole fruit	14	0.224	<0.05
				mature	pulp	27	<0.05	<0.05
					whole fruit		0.148	<0.05
		Dip-dynamic for 30 seconds	0.03	green fruit	whole fruit	14	0.386	<0.05
				mature	pulp	27	<0.05	<0.05
					whole fruit		0.237	<0.05
		Dip-dynamic for 60 seconds	0.03	green fruit	whole fruit	14	0.483	<0.05
				mature	pulp	27	0.096	<0.05
					whole fruit		0.585	<0.05
		Dip-dynamic for 300 seconds	0.03	green fruit	whole fruit	14	0.609	<0.05
				mature	pulp	27	0.066	<0.05
					whole fruit		0.393	<0.05
		Dip-dynamic for 1 seconds	0.06	green fruit	whole fruit	14	0.924	<0.05
				mature	pulp	27	<0.05	<0.05

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	Growth Stage at treatment	Portion analysed	DAA (days)	Residues (mg/kg)	
							Imazalil	R014821
					whole fruit		0.765	<0.05
		Dip-dynamic for 5 seconds	0.06	green fruit	whole fruit	14	0.604	<0.05
				mature	pulp	27	0.161	<0.05
					whole fruit		0.742	<0.05
		Dip-dynamic for 15 seconds	0.06	green fruit	whole fruit	14	0.953	<0.05
				mature	pulp	27	0.24	<0.05
					whole fruit		0.693	<0.05
		Dip-dynamic for 30 seconds	0.06	green fruit	whole fruit	14	1.224	<0.05
				mature	pulp	27	0.203	<0.05
					whole fruit		0.833	<0.05
		Dip-dynamic for 60 seconds	0.06	green fruit	whole fruit	14	2.143	<0.05
				mature	pulp	27	0.157	<0.05
					whole fruit		1.298	<0.05
		Dip-dynamic for 1 second	0.03	green fruit	whole fruit	7	0.717 (0.516, 0.915, 0.783, 0.655)	<0.05 (<0.05, <0.05, <0.05, <0.05)
				mature	whole fruit	25	0.426 (0.394, 0.391, 0.437, 0.483)	<0.05 (<0.05, <0.05, <0.05, <0.05)
		Dip-dynamic for 300 sec.	0.03	green fruit	whole fruit	7	1.544 (1.484, 1.498, 1.669, 1.526)	<0.05 (<0.05, <0.05, <0.05, <0.05)
				mature	whole fruit	25	1.271 (1.422, 1.456, 1.271, 0.936)	<0.05 (<0.05, <0.05, <0.05, <0.05)
		Dip-dynamic for 1 second	0.06	green fruit	whole fruit	7	1.445 (1.555, 1.332, 1.058, 1.833)	<0.05 (<0.05, <0.05, <0.05, <0.05)
				mature	whole fruit	25	1.110 (1.146, 1.157, 0.995, 1.141)	<0.05 (<0.05, <0.05, <0.05, <0.05)
		Dip-dynamic for 300 sec.	0.06	green fruit	whole fruit	7	1.347 (1.607, 1.436, 1.247, 1.098)	<0.05 (<0.05, <0.05, <0.05, <0.05)
				mature	whole fruit	25	1.309 (1.265, 1.449, 1.548, 0.972)	<0.05 (<0.05, <0.05, <0.05, <0.05)
AGR3/EXP4 Tela, Honduras 1990/91	Banana	Spray-dynamic for 3 seconds	0.03	green fruit	whole fruit	14	0.808	<0.05
				mature	pulp	20	0.063	<0.05
					whole fruit		0.361	<0.05
		Spray-dynamic for 30 seconds	0.03	green fruit	whole fruit	14	0.525	<0.05
				mature	pulp	20	0.058	<0.05
					whole fruit		0.502	<0.05
		Spray-dynamic for 60 seconds	0.03	green fruit	whole fruit	14	0.314	<0.05
				mature	pulp	20	0.054	<0.05
					whole fruit		0.383	<0.05
		Spray-dynamic for 300 sec.	0.03	green fruit	whole fruit	14	0.283	<0.05
				mature	pulp	20	0.064	<0.05
					whole fruit		0.367	<0.05
		Spray-dynamic for 600 sec.	0.03	green fruit	whole fruit	14	0.487	<0.05

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	Growth Stage at treatment	Portion analysed	DAA (days)	Residues (mg/kg)	
							Imazalil	R014821
				mature	pulp	20	0.105	<0.05
					whole fruit		0.492	<0.05
		Spray-dynamic for 900 sec.	0.03	green fruit	whole fruit	14	0.911	<0.05
				mature	pulp	20	0.093	<0.05
					whole fruit		0.564	<0.05
		Spray-dynamic for 3 seconds	0.06	green fruit	whole fruit	14	1.465	<0.05
				mature	pulp	20	0.057	<0.05
					whole fruit		0.917	<0.05
		Spray-dynamic for 30 seconds	0.06	green fruit	whole fruit	14	0.89	<0.05
				mature	pulp	20	0.088	<0.05
					whole fruit		0.777	<0.05
		Spray-dynamic for 60 seconds	0.06	green fruit	whole fruit	14	1.106	<0.05
				mature	pulp	20	0.100	<0.05
					whole fruit		0.925	<0.05
		Spray-dynamic for 300 sec.	0.06	green fruit	whole fruit	14	1.016	<0.05
				mature	pulp	20	0.126	<0.05
					whole fruit		1.16	<0.05
		Spray-dynamic for 600 sec.	0.06	green fruit	whole fruit	14	0.738	<0.05
				mature	pulp	20	0.122	<0.05
					whole fruit		1.154	<0.05
		Spray-dynamic for 900 sec.	0.06	green fruit	whole fruit	14	1.112	<0.05
				mature	pulp	20	0.151	<0.05
					whole fruit		1.215	<0.05
AGR3/EXP5 Armuelles, Panama 1990	Banana	Non-recovered spray for 3 seconds	0.04	mature	pulp	22	<0.05	<0.05
					whole fruit		<0.05	<0.05
		Non-recovered spray for 30 seconds	0.04	mature	pulp	22	0.095 (0.110, 0.080)	<0.05, (<0.05, <0.05)
					whole fruit		0.240 (0.260, 0.220)	<0.05, (<0.05, <0.05)
		Non-recovered spray for 60 seconds	0.04	mature	pulp	22	0.090 (0.080, 0.100)	<0.05, (<0.05, <0.05)
					whole fruit		0.535 (0.650, 0.420)	0.051 (0.060, <0.05)
		Non-recovered spray for 300 sec.	0.04	mature	pulp	22	0.140 (0.120, 0.160)	<0.05, (<0.05, <0.05)
					whole fruit		0.650 (0.730, 0.570)	0.051 (<0.05, 0.060)
		Non-recovered spray for 600 sec.	0.04	mature	pulp	22	0.130 (0.140, 0.120)	<0.05, (<0.05, <0.05)
					whole fruit		0.895 (0.710, 1.080)	<0.05, (<0.05, <0.05)
		Non-recovered spray for 900 sec.	0.04	mature	pulp	22	0.150 (0.100, 0.200)	<0.05, (<0.05, <0.05)
					whole fruit		1.165 (1.750, 0.580)	0.081 (0.120, <0.05)
		Non-recovered spray for 3 seconds	0.06	mature	pulp	22	0.06	<0.05
					whole fruit		0.42	<0.05
		Non-recovered spray for 30 seconds	0.06	mature	pulp	22	0.140 (0.140, 0.140)	<0.05, (<0.05, <0.05)
					whole fruit		0.705 (0.820, 0.590)	<0.05, (<0.05, <0.05)

Reference No., Location, Year	Crop / Variety	Formulation, Method of Treatment	Application rate per treatment (kg ai/hL)	Growth Stage at treatment	Portion analysed	DAA (days)	Residues (mg/kg)	
							Imazalil	R014821
		Non-recovered spray for 60 seconds	0.06	mature	pulp	22	0.155 (0.190, 0.120)	<0.05, (<0.05, <0.05)
					whole fruit		0.665 (0.640, 0.690)	0.056 (0.070, <0.05)
		Non-recovered spray for 300 sec.	0.06	mature	pulp	22	0.165 (0.170, 0.160)	<0.05, (<0.05, <0.05)
					whole fruit		0.695 (0.680, 0.710)	<0.05, (<0.05, <0.05)
		Non-recovered spray for 600 sec.	0.06	mature	pulp	22	0.205 (0.170, 0.240)	<0.05, (<0.05, <0.05)
					whole fruit		1.025 (1.130, 0.920)	0.085 (0.110, 0.060)
		Non-recovered spray for 900 sec.	0.06	mature	pulp	22	0.330 (0.430, 0.230)	<0.05, (<0.05, <0.05)
					whole fruit		1.175 (1.140, 1.210)	0.095 (0.110, 0.080)

Cucumber

Europe

Twelve supervised trials were carried out in Europe on greenhouse (protected) cucumbers in 2008, 2012 and 2013 (Fischer, 2009, S08-01482; 2012, S12-00922; 2013, S13-0458). In ten of the trials three foliar spray applications were made, while in the remaining two trials, four spray applications were made of an emulsifiable concentrate formulation containing 100 g/L of imazalil. Application rates ranged from 0.007 to 0.008 kg ai/hL, and in the case of exaggerated rates, ranged from 0.023 to 0.03 kg ai/hL.
Samples from both Northern and Southern European trials were taken for analysis at 0-7 days after the last application. Fruit was analysed for imazalil by the LC-MS/MS method GAB-08103V based on DFG method S-19 (multi residue method L 00.00-34). In the 2012/2013 trials, residues of the metabolite R014821 were analysed by the LC-MS/MS method 11G04031-013VMPO that is also based on DFG method S-19, with an LOQ of 0.01 mg/kg/analyte for both methods.
Cucumber samples were stored deep-frozen for a maximum of 6.6 months (200 days).

Table 54 Magnitude of the residues of imazalil and R014821 in/on cucumbers following foliar application from trials conducted in Europe

Reference No., Location, Year	Crop / Variety	Method of Treatment	Application rate per treatment			No.	RTI (days)	Portion analysed	DAA (days)	Residues (mg/kg)	
			kg ai/ha	Water (L/ha)	kg ai/hL					Imazalil	R014821
Belgium GAP		Foliar spray			0.005	3	7		1		
S08-01482-01 11140 Conil de la Frontera, Cádiz, Spain 2008	Cucumber / Alanis	Foliar spray	0.304 0.302 0.316	1215 1206 1264	0.025 0.025 0.025	3	- 7 7	whole fruit	0	0.06	Not analysed
									1	0.03	
									3	0.02	
S08-01482-02 11540 Sanlúcar de Barrameda Cádiz, Spain 2008	Cucumber / Alanis	Foliar spray	0.293 0.310 0.313	1186 1333 1342	0.025 0.023 0.023	3	- 7 7	whole fruit	0	0.09	Not analysed
									1	0.07	
S08-01482-03 40057 Granarolo dell'Emilia, Bologna, Italy 2008	Cucumber / Akito	Foliar spray	0.313 0.300 0.316	1044 1000 1052	0.03 0.03 0.03	3	- 7 7	whole fruit	0	0.11	Not analysed
									1	0.03	
									3	0.03	
S08-01482-04 57200 Profitis	Cucumber / Palmera	Foliar spray	0.301 0.304	1003 1013	0.03 0.03	3	- 7	whole fruit	0	0.05	Not analysed

Reference No., Location, Year	Crop / Variety	Method of Treatment	Application rate per treatment			No.	RTI (days)	Portion analysed	DAA (days)	Residues (mg/kg)	
			kg ai/ha	Water (L/ha)	kg ai/hL					Imazalil	R014821
Thessaloniki, Greece 2008			0.303	1010	0.03		7		1	0.07	
S08-01482-05 56250 Sulniac Bretagne, France 2008	Cucumber / Dominica	Foliar spray	0.329 0.298 0.329	1096 992 1096	0.03 0.03 0.03	3	- 7 7	whole fruit	0	0.06	Not analysed
									1	0.05	
									3	0.02	
S08-01482-06 3015 Csány Heves, Hungary 2008	Cucumber / Majestosa	Foliar spray	0.300 0.303 0.298	1000 1010 994	0.03 0.03 0.03	3	- 7 7	whole fruit	0	0.25	Not analysed
									1	0.18	
S08-01482-07 Roydon Hamlet Essex, Great Britain 2008	Cucumber / Aviance	Foliar spray	0.306 0.300 0.303	1020 1000 1010	0.03 0.03 0.03	3	- 7 4	whole fruit	0	0.23	Not analysed
									1	0.20	
									3	0.11	
S08-01482-08 76698 Upstadt-Weiher, Baden-Württemberg, Germany 2008	Cucumber / Tornac	Foliar spray	0.306 0.309 0.341	1117 1130 1247	0.027 0.027 0.027	3	- 7 7	whole fruit	0	0.15	Not analysed
									1	0.04	
S12-00922-01 27478 Altenbruch, Lower Saxony, Germany 2012	Cucumber / Cordoba	Foliar spray	0.075 0.073 0.075	1009 978 1001	0.0075 0.0075 0.0075	3	- 5 5	whole fruit	0	0.03	<0.01
									1	0.02	<0.01
									3	0.01	<0.01
									7	<0.01	<0.01
S12-00922-02 71277 Perouse-Rutesheim, Germany 2012	Cucumber / Indira	Foliar spray	0.081 0.0741 0.072	1080 990 957	0.0075 0.0075 0.0075	3	- 5 5	whole fruit	0	0.02	<0.01
									1	0.01	<0.01
									3	<0.01	<0.01
									7	<0.01	<0.01
S13-04508-01 76698 Ubstadt-Weiher, Germany 2013	Cucumber / Khassib	Foliar spray	0.081 0.082 0.077 0.075	1079 1099 1020 1004	0.008 0.007 0.008 0.007	4	- 5 5 5	whole fruit	0	0.02	<0.01
									1	0.02	<0.01
									3	0.01	<0.01
									7	<0.01	<0.01
S13-04508-02 6662 PK Elst, Gelderland, Netherlands 2013	Cucumber / Cratos	Foliar spray	0.077 0.076 0.077 0.074	1020 1009 1028 980	0.008 0.008 0.007 0.008	4	- 5 5 5	whole fruit	0	0.04	<0.01
									1	0.05	<0.01
									3	0.03	<0.01
									7	0.02	<0.01

Tomato

Europe – Foliar Spray Application

Eight supervised trials were carried out in Europe on greenhouse (protected) tomatoes in 2007 (Fischer, 2008, 20074117/E1-FGTO). Three spray applications of a 100 g/L EC formulation of imazalil were made at rates of 0.02 to 0.025 kg ai/hL. In two trials the spray concentration was 0.03 kg ai/hL. Mature tomatoes were sampled 0, 1 and 3 days after the last application.
Fruits were analysed for imazalil by the LC-MS/MS method CET-0703V based on DFG method S-19 (multi residue method L 00.00-34), with an LOQ of 0.01 mg/kg.
Tomato samples were stored deep-frozen for a maximum of 4.4 months (134 days).

Table 54 Magnitude of the residues of imazalil in/on greenhouse tomato following foliar application from trials conducted in Europe

Reference No., Location, Year	Crop / Variety	Method of Treatment	Application rate per treatment			No.	RTI (days)	Portion analysed	DAA (days)	Residues (mg/kg)
			kg ai/ha	Water (L/ha)	kg ai/hL					Imazalil
Belgium GAP		Foliar spray			0.02	3	7		1	
F07W313R 56450 Theix, Bretagne, France 2007	Tomato / Palmiro	Foliar Spray	0.282 0.279 0.265	1129 1118 1059	0.025 0.025 0.025	3	- 7 8	Fruit	0 1 3	0.08 0.08 0.06
F07W314R 56400 Pluneret, Bretagne, France 2007	Tomato / Brazil	Foliar Spray	0.287 0.290 0.267	1147 1160 1167	0.025 0.025 0.025	3	- 9 10	Fruit	0 1 3	0.04 0.03 0.04
F07W315R 56250 Sulniac, Bretagne, France 2007	Tomato / Mugello	Foliar Spray	0.297 0.330 0.273	1187 1320 1093	0.025 0.025 0.025	3	- 10 8	Fruit	0 1 3	0.08 0.16 0.08
F07W316R 56680 Plouhinec, Bretagne, France 2007	Tomato / Felicia	Foliar Spray	0.271 0.274 0.286	1086 1095 1143	0.025 0.025 0.025	3	- 9 10	Fruit	0 1 3	0.05 0.03 0.04
S07W204R 03320 Torrellano, Alicante, Spain 2007[a]	Tomato / Denis	Foliar Spray	0.290 0.290 0.292	966 968 974	0.030 0.030 0.030	3	- 8 7	Fruit	0 1 3	0.06 0.15 0.04
S07W205R 03320 Torrellano, Alicante, Spain 2007[a]	Tomato / Boludo	Foliar Spray	0.300 0.295 0.292	1000 985 973	0.030 0.030 0.030	3	- 8 6	Fruit	0 1 3	0.26 0.14 0.15
GR07W213R 57008 Magnisia-Ionia, Thessaloniki, Greece 2007[b]	Tomato / Neely	Foliar Spray	0.288 0.316 0.302	1441 1580 1510	0.020 0.020 0.020	3	- 7 7	Fruit	0 1 3	0.40 0.08 0.05
GR07W214R 57008 Magnisia-Ionia, Thessaloniki, Greece 2007[b]	Tomato / Elpida	Foliar Spray	0.301 0.305 0.293	1502 1526 1466	0.020 0.020 0.020	3	- 7 7	Fruit	0 1 3	0.10 0.09 0.05

[a] Trials considered dependent based on location and timing of applications.

[b] Trials considered dependent based on location and timing of applications.

Europe – Spot treatment application

Eight supervised trials were carried out in Europe on greenhouse (protected) tomatoes in 1996/97 (De Winter, 1998, AGR-178 to AGR 185; Van den Heuvel, 1997, 97.616). One local spot spray application of a ready to use 20 g/L spray solution of imazalil was made by spraying for one second to two different locations at the tomato stem.

Mature tomatoes were sampled 0, 1 and 3 days after the last application. Fruits were analysed for imazalil by a validated gas chromatographic method (Report 56 - AGR 16, 1992) for the determination of imazalil residues with a method LOQ of 0.05 mg/kg.

Tomato samples were stored deep-frozen for a maximum of 4 months (121 days).

Table 55 Magnitude of the residues of imazalil in/on greenhouse following spot treatment from trials conducted in Europe

Reference No., Location, Year	Crop / Variety	Method of Treatment	Application rate per treatment (kg ai/hL)	DAA (days)	Portion analysed	Residues (mg/kg)
						Imazalil
AGR 178 4813 Breda, Netherlands 1996	Tomato / Rivido	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05
AGR 179 4741 Hoeven, Netherlands 1996	Tomato / Chaser	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05
AGR 180 4813 Breda, Netherlands 1996	Tomato / Chaser	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05
AGR 181 4873 Etten-Leur, Netherlands 1996	Tomato / Jamaica	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05
AGR 182 4873 Etten-Leur, Netherlands 1997	Tomato / Chaser	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05
AGR 183 4813 Breda, Netherlands 1997	Tomato / Jamaica	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05
AGR 184 4741 Hoeven, Netherlands 1997	Tomato / Chaser	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05
AGR 185 4855 Galder, Netherlands 1997	Tomato / Jamaica	Local spray of 1 second to two different locations at the tomato stems	2.0	0	fruit	<0.05
				1		<0.05
				3		<0.05

Potato

Europe – Seed-piece treatment

Seventeen supervised seed treatment trials on potato were carried out in Europe in 2004 and 2008 (Hubbard, 2006, OA01287; Bamber, 2006, 577-04-MAK-ROL; Moller, 2005, 460501; Fischer, 2009, S08-01236/S08-01237). Potato seed pieces were treated with a 100 g/L SL formulation of imazalil at a nominal application rate of 0.015 kg ai/tonne. The spray concentration was 0.75 kg ai/hL in the 2004 trials and 1.25 kg ai/hL in the 2008 trials.

Treated seed pieces were stored for approximately 180 days (2004 trials) or 11–44 days (2008 trials) prior to planting. Mature potato tubers were harvested after a growing period of 68–71 days (2004) to 103–150 days (2008).

Potato tubers were analysed for imazalil using a GC-MS method with an LOQ of 0.017 mg/kg (OA01287) and the DFG method S-19 (multi residue method L-00.00-34) with an LOQ of 0.01 mg/kg (S08-01236 and S08-01237).

Samples of potato tubers were stored deep-frozen for a maximum of 7.5 months (229 days).

Table 56 Magnitude of the residues of imazalil in/on potato following seed treatment from trials conducted in Europe

Trial No., Location, Year	Crop / Variety	Application rate per treatment		Portion analysed	DAA / DAP (days)	Residues (mg/kg)
		kg ai/tonne	kg ai/tonne			
Belgium GAP		0.015				
Trial 577/POT/1-A Bicester, Oxon, OX27	Potato / Premiere	0.015	0.75	mature tubers	248 / 71	<0.017

Trial No., Location, Year	Crop / Variety	Application rate per treatment		Portion analysed	DAA / DAP (days)	Residues (mg/kg)
		kg ai/tonne	kg ai/tonne			
9AS, United Kingdom, United Kingdom 2004						
Trial 577/POT/1-B 5500 Middelfart, Denmark 2004	Potato / Inova	0.015	0.75	mature tubers	248 / 68	<0.017
S08-01236-01 69168 Wiesloch, Baden-Württemberg, Germany 2008	Potato / Agria	0.016	1.25	mature tubers	106 / 93	<0.01
S08-01236-02 82269 Walleshausen, Bavaria, Germany 2008	Potato / Agria	0.016	1.25	mature tubers	156 / 112	<0.01
S08-01236-03 45300 Ezanville, Sermaises, France 2008	Potato / Agria	0.016	1.25	mature tubers	129 / 106	<0.01
S08-01236-04 45300 Stotzheim, Alsace, France 2008	Potato / Agria	0.016	1.25	mature tubers	134 / 112	<0.01
S08-01236-05 2454 Ivàncsa, Fejér, Hungary 2008	Potato / Agria	0.016	1.25	mature tubers	157 / 146	<0.01
S08-01236-06 5310 Kisùjszàllàs, Jàzs-Nagykun-Szolnok, Hungary 2008	Potato / Agria	0.016	1.25	mature tubers	154 / 142	<0.01
S08-01236-07 NG20 9HU, Whaley, Mansfield, UK 2008	Potato / Agria	0.016	1.25	mature tubers	142 / 117	<0.01
S08-01236-08 B78 2LQ Drayton Bussett, Tamworth, UK 2008	Potato / Agria	0.016	1.25	mature tubers	182 / 150	<0.01
S08-01237-01 84210 Pernes-les-Fontaines, Provence-Alpes-Côte d'Azur, France 2008	Potato / Agria	0.016	1.25	mature tubers	130 / 106	<0.01
S08-01237-02 66033 Kato Vrontou, K. Nevrokopi, Greece 2008	Potato / Agria	0.016	1.25	mature tubers	142 / 121	<0.01
S08-01237-03 40013 Trebbo Di Reno, Bologna, Italy 2008	Potato / Agria	0.016	1.25	mature tubers	127 / 113	<0.01

Trial No., Location, Year	Crop / Variety	Application rate per treatment		Portion analysed	DAA / DAP (days)	Residues (mg/kg)
		kg ai/tonne	kg ai/tonne			
S08-01237-04 40023 Castel Guelfo, Bologna Italy 2008	Potato / Agria	0.016	1.25	mature tubers	128 / 112	<0.01
S08-01237-05 57008 Ionia, Thessaloniki, Greece 2008	Potato / Agria	0.016	1.25	mature tubers	143 / 112	<0.01
S08-01237-06 58002 Arnissa, Pella, Greece 2008	Potato / Agria	0.016	1.25	mature tubers	147 / 103	<0.01
S08-01237-07 66620 Brouilla, Languedoc-Roussillon, France 2008	Potato / Agria	0.016	1.25	mature tubers	117 / 107	<0.01
S08-01237-08 50490, Villarreal de Huerva, Zaragoza, Spain 2008	Potato / Agria	0.016	1.25	mature tubers	147 / 132	<0.01

Europe – Post-harvest application

Fifteen supervised trials on potatoes were carried out in Europe where potatoes received a single post-harvest application, immediately prior to storage, of a liquid formulation containing 100 g/L of imazalil (Bamber, 2000, 651-55-MAK-POT; Byast, 2001, OA00549; Fischer, 2007, 20074115/G1-FDPO; Samrau, 2013, S11-02135/ S12-03969; Kreke, 2013, D47918; Zeisler, 2013, D55377).

Eight trials were conducted in 1999-2000 in the UK and one trial was conducted in Germany in 2007 where tubers were treated at a dose rate of 0.009-0.014 kg ai/tonne. Tubers were sampled at various intervals ranging from 0 - 190 days after treatment.

In 2011 and 2012, four additional trials were conducted in Germany where potatoes received a single post-harvest application at 0.015-0.016 kg ai/tonne with no waiting period (PHI = 0).

Two trials were carried out in Switzerland in 2012, also supporting one post-harvest application to potatoes at a dose rate of 0.015 kg ai/tonne. Residues were determined at a PHI of 29 days after the application.

In the 1999/2000 trials, potato tubers were analysed for imazalil using a validated HPLC-UV method with an LOQ of 0.06 mg/kg. In the 2007 trial, potato tubers were analysed for imazalil using the HPLC-MS/MS method CET-0702V with an LOQ of 0.01 mg/kg. In the trials performed in Germany in 2011/12, residues of imazalil and the metabolite R014821 were analysed using the HPLC-MS/MS methods CET-0702V and 11G04031-013VMPO with an LOQ of 0.01 mg/kg/analyte. For the trial performed in Switzerland, potato tubers were analysed for imazalil and R014821 using the QuEChERS method with an LOQ of 0.01 mg/kg/analyte.

Samples of potato tubers were stored deep-frozen for a maximum of 8.7 months (264 days).

Table 57 Magnitude of the residues of imazalil in/on potato following post-harvest treatment from trials conducted in the United Kingdom

Trial No., Location, Year	Crop / Variety	Application rate per treatment		DAA (days)	Portion Analysed	Residues (mg/kg)	
		kg ai/tonne	(kg ai/hL)			Imazalil	R014821
Belgium GAP		0.015					
651/POT/1, Redbourne, United Kingdom, 1999	Potato / Cara	0.009	0.47	0	tubers	0.36	Not analysed
				6		1.03 (1.10, 1.14, 0.85)	

Trial No., Location, Year	Crop / Variety	Application rate per treatment		DAA (days)	Portion Analysed	Residues (mg/kg)	
		kg ai/tonne	(kg ai/hL)			Imazalil	R014821
				20		0.93 (1.30, 0.53, 0.97)	
				42		1.04 (1.57, 0.83, 0.71)	
				92		0.78 (0.92, 0.81, 0.62)	
		0.014	0.70	0	tubers	0.93	Not analysed
				6		1.51 (1.58, 1.72, 1.23)	
				20		1.28 (1.84, 1.18, 0.81)	
				42		1.39 (1.25, 2.37, 0.56)	
				92		1.22 (1.37, 1.90, 0.41)	
651/POT/2, Catthorpe, United Kingdom, 1999	Potato / Nadine	0.009	0.47	0	tubers	0.23	Not analysed
				7		0.60 (0.67, 0.53, 0.60)	
				21		0.39 (0.51, 0.46, 0.19)	
				42		0.35 (0.35, 0.37, 0.32)	
				91		0.46 (0.37, 0.36, 0.65)	
		0.014	0.70	0	tubers	0.48	Not analysed
				7		0.88 (0.50, 1.09, 1.06)	
				21		0.66 (0.65, 0.65, 0.67)	
				42		0.60 (0.58, 0.60, 0.63)	
				91		0.38 (0.36, 0.53, 0.25)	
651/POT/3, Owmby, United Kingdom, 1999	Potato / Marfona	0.009	0.47	41	tubers	0.16 (0.14, 0.18, 0.16)	Not analysed
				91		0.13 (0.13, 0.09, 0.18)	
		0.014	0.70	41	tubers	0.22 (0.22, 0.24, 0.20)	Not analysed
				91		0.17 (0.15, 0.17, 0.18)	
651/POT/4, Kinross, United Kingdom, 1999	Potato / Maris Piper	0.009	0.47	42	tubers	0.25 (0.38, 0.08, 0.29)	Not analysed
				85		0.24 (0.56, 0.11, 0.06)	
		0.014	0.70	42	tubers	0.54 (0.42, 0.37, 0.83)	Not analysed
				85		0.47 (0.40, 0.65, 0.37)	
651/POT/5, Harborough Magna, United Kingdom, 1999	Potato / Pentland Dell	0.009	0.47	0	tubers	0.60	Not analysed
				8		0.48	
				21		0.62	
				42		0.60 (0.67, 0.82, 0.33, 0.56)	
				91		0.43 (0.33, 0.40, 0.39, 0.58)	
		0.014	0.70	0	tubers	1.60	Not analysed
				8		1.69	
				21		1.83	

Trial No., Location, Year	Crop / Variety	Application rate per treatment kg ai/tonne	(kg ai/hL)	DAA (days)	Portion Analysed	Residues (mg/kg) Imazalil	R014821
				42		1.71 (1.72, 2.07, 1.43, 1.62)	
				91		0.80 (0.85, 0.11, 1.25, 0.98)	
651/POT/6, Ansley, United Kingdom, 1999	Potato / Romano	0.009	0.47	0	tubers	0.43	Not analysed
				7		0.33	
				21		0.20	
				42		0.37	
				91		0.08	
				190		0.11 (0.11, 0.09, 0.13)	
		0.014	0.70	0	tubers	0.15	Not analysed
				7		0.46	
				21		0.33	
				42		0.37	
				91		0.20	
				190		0.27 (0.28, 0.23, 0.30)	
651/POT/7, Trerulefoot, United Kingdom, 1999	Potato / Cara	0.009	0.47	55	tubers	0.35	Not analysed
				102		0.27	
				181		0.35 (0.23, 0.39, 0.43)	
		0.014	0.70	55	tubers	0.78	Not analysed
				102		0.46	
				181		0.42 (0.29, 0.46, 0.50)	
651/POT/8, Bardney, United Kingdom, 1999	Potato / Estima	0.009	0.47	42	tubers	1.18	Not analysed
				67		0.60 (0.61, 0.70, 0.49)	
				97		0.52 (1.05, 0.65, 0.27, 0.12)	
		0.014	0.70	42	tubers	0.83	Not analysed
				67		0.84 (0.69, 0.95, 0.89)	
				97		0.70 (0.65, 0.50, 0.67, 0.98)	
20074115/G1-FDPO, G07W557R-A-002, Germany, 2007	Potato / Cilena	0.014	1.39	1	tubers	2.65	Not analysed
S11-02135-01, Dollern, Germany, 2011	Potato / Belana	0.015	10	0	tubers	4.1 (4.5, 3.6)	0.03 (0.04, 0.02)
				29		2.7	0.08
				62		2.1	0.12
				89		1.6	0.13
				117		1.3	0.17
				152		1.2	0.24
				180		0.9	0.23
S11-02135-02, Brest, Germany, 2011	Potato / Cilena	0.015	10	0	tubers	4.6 (4.9, 4.3)	0.04 (0.05, 0.03)
				28		2.1	0.15
				61		1.1	0.14
				89		1.2	0.22
				117		1.0	0.22
				147		1.4	0.26
				180		1.0	0.22

Trial No., Location, Year	Crop / Variety	Application rate per treatment		DAA (days)	Portion Analysed	Residues (mg/kg)	
		kg ai/tonne	(kg ai/hL)			Imazalil	R014821
S12-03969-01, Stade, Germany, 2012[a]	Potato / Belana	0.015	0.728	0	tubers	3.4	0.02
S12-03969-02, Stade, Germany, 2012[a]	Potato / Cilena	0.016	0.728	0	tubers	4.5	0.03
A/CH/F/12/1, Baselland, Switzerland, 2012[b]	Potato / Desiree	0.015	0.735	29	tubers	6.25	0.24
A/CH/F/12/2, Baselland, Switzerland, 2012[b]	Potato / Agria	0.015	0.735	29	tubers	4.5	0.26

[a] Trials considered dependent based on location and timing of application.

[b] Trials considered dependent based on location and timing of application.

Cereal grains

Sixteen supervised seed treatment trials on wheat (4) and barley (12) were carried out in Europe in 2005/2006 (Pollman, 2006, R-18884/R18885) and 2008 (Fischer, 2008, 20074129/E1-FDCE/20074129/E2-FDCE). Seed treatment rates ranged from 0.048-0.078 kg ai/tonne, either with a 30 g/L FS formulation of imazalil (2005/2006) or with a 50 g/L LS formulation (2008). The products were used undiluted.

Treated cereal seeds were stored for 7 - 46 days before sowing in the field. Samples of cereal grain were taken 96 - 138 days after planting (DAP) for spring cereals and 287 - 302 DAP for winter cereals

Samples were analysed for imazalil using a method by Fillion *et al*. 2000 (modified) with an LOQ of 0.01 mg/kg for grain (R-18884 and R-18885) and the DFG method S-19 (multi residue method L-00.00-34) with an LOQ of 0.01 mg/kg (20074129/E1-FDCE and 20074129/E2-FDCE).

Samples of cereal grain were stored deep-frozen for a maximum of 7 months (212 days).

Table 58 Magnitude of the residues of imazalil in/on wheat and barley grain following seed treatment from trials conducted in Europe

Reference No., Trial, Country	Crop / Variety	Application rate per treatment		DAP (days)	Portion analysed	Residues of Imazalil (mg/kg)
		kg ai/tonne	kg ai/hL			
USA GAP	Wheat and barley	0.1		n/a		
G06W002R, Niedersachsen, Germany, 2006[a]	Spring wheat / Monsun	0.048	3	115	grain	<0.01
G06W003R, Niedersachsen, Germany, 2006[a]	Spring wheat /Monsun	0.048	3	132	grain	<0.01
G06W004R, Niedersachsen, Germany, 2006[1]	Spring wheat / Monsun	0.048	3	96	grain	<0.01
PL05W013R, Wielkopolska, Poland, 2005/06	Winter wheat / Pegassos	0.048	3	297	grain	<0.01
G05W172R, Niedersachsen, Germany, 2005/06	Winter barley / Candesse	0.048	3	305	grain	<0.01
G05W173R, Baden-Wurttemberg, Germany, 2005/06	Winter barley / Jessica	0.048	3	314	grain	<0.01
PL05W014R, Wielkopolska, Poland, 2005/06	Winter barley / Merlot	0.048	3	302	grain	<0.01
H05W010R, Fejér County, Hungary, 2006	Spring barley /Tocada	0.048	3	118	grain	<0.01
G07W621R, Niedersachsen, Germany, 2008	Spring barley / Power	0.078	—	120	grain	<0.01
G07W622R, Niedersachsen, Germany, 2008	Spring barley / Power	0.078	—	120	grain	<0.01
G07W623R, Baden-Wurttemberg, Germany, 2008	Spring barley / Power	0.078	—	127	grain	<0.01

Reference No., Trial, Country	Crop / Variety	Application rate per treatment		DAP (days)	Portion analysed	Residues of Imazalil (mg/kg)
		kg ai/tonne	kg ai/hL			
F07W320R, Alsace, France, 2008	Spring barley / Power	0.078	—	138	grain	<0.01
F07W321R, Languedoc-Roussilon, France, 2008	Spring barley / Tipple	0.075	—	127	grain	<0.01
F07W322R, Languedoc-Roussilon, France, 2008	Spring barley / Power	0.075	—	127	grain	<0.01
I07W127R, Emilia Romagna, Italy, 2008	Spring barley / Tipple	0.078	—	131	grain	<0.01
I07W128R, Emilia Romagna, Italy, 2008	Spring barley / Power	0.074	—	124	grain	<0.01

[a] All trials were considered dependent based on location and timing of application

Primary Feed Commodities of Plant Origin

Straw, fodder and forage of cereal grains and grasses

Sixteen supervised seed treatment trials on wheat (4) and barley (12) were carried out in Europe in 2005/2006 (Pollman, 2006, R-18884/R18885) and 2008 (Fischer, 2008, 20074129/E1-FDCE/20074129/E2-FDCE). Cereal grains were seed treated at rates of 0.048-0.078 kg ai/tonne, either with a 30 g/L FS formulation of imazalil (2005/2006) or with a 50 g/L LS formulation (2008). The products were used undiluted.

Treated cereal seeds were stored for 7 - 46 days before sowing in the field. Samples of cereal straw were taken 96 - 138 days after planting (DAP) for spring cereals and 287 - 302 DAP for winter cereals. For all trials, whole plant without root samples were taken at an immature stage, in the range of 48 - 99 DAP.

Samples were analysed for imazalil using a method by Fillion *et al*. 2000 (modified) with an LOQ of 0.02 mg/kg for plant and straw (R-18884 and R-18885) and the DFG method S-19 (multi residue method L-00.00-34) with an LOQ of 0.02 mg/kg (20074129/E1-FDCE and 20074129/E2-FDCE).

Samples of cereal plant and straw were stored deep-frozen for a maximum of 7 months (212 days).

Table 59 Magnitude of the residues of imazalil in/on wheat and barley plants and straw following seed treatment from trials conducted in Europe

Reference No., Trial, Country	Crop / Variety	Application rate per treatment		DAP (days)	Portions analysed	Residues of Imazalil (mg/kg)
		kg ai/tonne	kg ai/hL			
USA	Wheat and barley	0.1		n/a		
G06W002R, Niedersachsen, Germany, 2006[a]	Spring wheat / Monsun	0.048	3	50	plant w/o roots	<0.02
				65	plant w/o roots	<0.02
				115	straw	<0.02
G06W003R, Niedersachsen, Germany, 2006[a]	Spring wheat /Monsun	0.048	3	59	plant w/o roots	<0.02
				81	plant w/o roots	<0.02
				132	straw	<0.02
G06W004R, Niedersachsen, Germany, 2006[a]	Spring wheat / Monsun	0.048	3	68	plant w/o roots	<0.02
				96	straw	<0.02
PL05W013R, Wielkopolska, Poland, 2005/06	Winter wheat / Pegassos	0.048	3	297	straw	<0.02
G05W172R, Niedersachsen, Germany, 2005/06	Winter barley / Candesse	0.048	3	271	plant w/o roots	<0.02
				305	straw	<0.02
G05W173R, Baden-Wurttemberg, Germany,	Winter barley / Jessica	0.048	3	258	plant w/o roots	<0.02

Reference No., Trial, Country	Crop / Variety	Application rate per treatment		DAP (days)	Portions analysed	Residues of Imazalil (mg/kg)
		kg ai/tonne	kg ai/hL			
2005/06				314	straw	<0.02
PL05W014R, Wielkopolska, Poland, 2005/06	Winter barley / Merlot	0.048	3	302	straw	<0.02
H05W010R, Fejér County, Hungary, 2006	Spring barley /Tocada	0.048	3	96	plant w/o roots	<0.01
				118	straw	<0.01
G07W621R, Niedersachsen, Germany, 2008	Spring barley / Power	0.078	—	48	plant w/o roots	<0.01
				120	straw	<0.01
G07W622R, Niedersachsen, Germany, 2008	Spring barley / Power	0.078	—	48	plant w/o roots	<0.01
				120	straw	<0.01
G07W623R, Baden-Wurttemberg, Germany, 2008	Spring barley / Power	0.078	—	69	plant w/o roots	<0.01
				127	straw	<0.01
F07W320R, Alsace, France, 2008	Spring barley / Power	0.078	—	73	plant w/o roots	<0.01
				138	straw	<0.01
F07W321R, Languedoc-Roussilon, France, 2008	Spring barley / Tipple	0.075	—	84	ears	<0.01
				84	rest of plant	<0.01
				127	straw	<0.01
F07W322R, Languedoc-Roussilon, France, 2008	Spring barley / Power	0.075	—	84	ears	<0.01
				84	rest of plant	<0.01
				127	straw	<0.01
I07W127R, Emilia Romagna, Italy, 2008	Spring barley / Tipple	0.078	—	97	ears	<0.01
				97	rest of plant	<0.01
				131	straw	<0.01
I07W128R, Emilia Romagna, Italy, 2008	Spring barley / Power	0.074	—	99	ears	<0.01
				99	rest of plant	<0.01
				124	straw	<0.01

[a] All trials were considered dependent based on location and timing of application.

Fate of residues during processing

High temperature hydrolysis

The hydrolytic stability of imazalil was investigated in aqueous buffer solutions at three pH values and temperatures to simulate processing practice (Flörchinger, 2010, S10-02412). The study was performed at pH 4, 5 and 6 and temperatures of 90 °C, 100 °C and 120 °C, for 20, 60 and 20 minutes, respectively. The range of hydrolytic conditions used represents the processes of pasteurisation, baking/brewing/boiling and sterilisation. Buffer solutions containing imazalil were incubated in glass vessels. The test item was tested at concentrations of approximately 11.4 mg/L buffer solution. At time 0 and after 20 or 60 minutes incubation, an aliquot of the test solution was diluted with acetonitrile:water (1:1, v:v) and analysed using an HPLC-MS/MS method. The temperatures were maintained constant throughout incubation and no significant variation of the pH values was observed in the buffered solutions.

Table 60

Test vessel	Imazalil content (mg/L)		
	pH 4 (90ºC)	pH5 (100ºC)	pH6 (120ºC)

	0 min	20min	0min	60min	0min	20min
1	12.1	11.3	12.1	11.5	11.8	11.2
2	11.9	11.3	11.1	11.4	11.4	11.8

Imazalil was shown to be hydrolytically stable for all hydrolytic conditions tested in this study: at pH 4 and 90 °C simulating pasteurisation, at pH 5 and 100 °C simulating baking/brewing/boiling and at pH 6 and 120 °C simulating the process of sterilisation. There was no evidence of any hydrolysis or reaction products formed during incubation.

Citrus fruits

Shortly after being harvested, lemons received a pre-storage treatment with a wax formulated with a solution containing imazalil at 0.2 kg ai/hL. After a storage period of 8 weeks at 13-15 °C, lemons were treated again with wax containing imazalil at 0.4 or 0.8 kg ai/hL. Oranges and grapefruits received only one single post-harvest treatment with a wax containing imazalil at 0.4 kg/hL. The interval from treatment to processing was 4 days (Goodwine, 1992, AGR8).

The effects of washing were examined in lemons stored 8 weeks after the first post-harvest treatment and again immediately after the post-storage treatment. In oranges and grapefruit, the effect of washing was investigated immediately after treatment

Table 61 Levels of imazalil and R014821 in citrus whole fruit - effects of washing

Sample	Application rate (kg ai/hL)	Average Residues (mg/kg)			Processing Factors	
		Imazalil	R014821	Total[a]	Imazalil	Total
Lemons						
Prior to storage	0.2	1.04	0.05	1.10	-	-
After storage (unwashed)		0.80	0.34	1.19	-	-
After storage (washed)		0.68	0.31	1.04	0.65	0.87
After storage (unwashed)	0.2 + 0.4	6.69	0.36	7.11	-	-
After storage (washed)		2.73	0.34	3.12	0.40	0.44
After storage (unwashed)	0.2 + 0.8	11.4	0.41	11.9	-	-
After storage (washed)		4.58	0.33	4.96	0.40	0.42
Oranges						
Unwashed	0.4	3.31	<0.05	3.37	-	-
Washed		0.88	<0.05	0.94	0.26	0.28
Unwashed	0.8	7.43	<0.05	7.49	-	-
Washed		2.36	<0.05	2.42	0.32	0.32
Grapefruits						
Unwashed	0.4	3.99	<0.05	4.05	-	-
Washed		1.09	<0.05	1.15	0.27	0.28
Unwashed	0.8	8.08	<0.05	8.14	-	-
Washed		3.57	<0.05	3.62	0.44	0.45

[a] Expressed as imazalil equivalents.

Washing appears to greatly reduce total residues of imazalil with processing factors (PF) in the range of 0.28 - 0.44. This effect is lower after eight weeks of storage with a processing factor of 0.87.

Washed fruits were subsequently processed, according to commercial practices, into juice, finisher pulp, fresh and dried peel, oil, oil emulsion and molasses.

The RAC and processed commodity samples were analysed using the validated LC-MS/MS method AGR18.The Limit of Quantitation (LOQ) for imazalil was 0.05 mg/kg. The citrus RAC and processed commodities were stored frozen for up to 268 days.

Table 62 Concentration of imazalil and R014821 residues in orange processed fractions and processing factors

Process	Application Rate (kg ai/hL)	Average Residues (mg/kg)			Processing Factors	
		Imazalil	R014821	Totala	Imazalil	Total
Lemons						
Whole fruit (unwashed)	0.2+0.4	6.69	0.36	7.11	—	—
Whole fruit (washed)		2.73	0.34	3.12	0.41	0.44
Juice		0.31	<0.05	0.37	0.05	0.05
Finisher pulp		0.55	<0.25	0.8	0.08	0.11
Chopped fresh peel		2.58	0.61	3.29	0.38	0.46
Cold pressed oil		29	<0.05	29.1	4.3	4.09
Oil emulsion water (waste)		3.29	<0.05	3.34	0.49	0.47
Dried peel		6.71	1.88	8.88	1	1.25
Molasses		3.74	1.29	5.23	0.56	0.74
Whole fruit (unwashed)	0.2+0.8	11.4	0.41	11.9	—	—
Whole fruit (washed)		4.58	0.33	4.96	0.4	0.42
Juice		0.44	<0.05	0.49	0.04	0.04
Finisher pulp		0.61	<0.05	0.66	0.05	0.06
Chopped fresh peel		3.92	0.54	4.54	0.34	0.38
Cold pressed oil		29.6	0.15	29.8	2.6	2.5
Oil emulsion water (waste)		4.71	<0.05	4.76	0.41	0.4
Dried peel		11.9	2.02	14.2	1.04	1.19
Molasses		5.97	1.06	7.2	0.52	0.6
Orange						
Whole fruit (unwashed)	0.4	3.31	<0.05	3.37	—	—
Whole fruit (washed)		0.88	<0.05	0.94	0.27	0.28
Juice		<0.05	<0.05	<0.11	<0.02	<0.03
Finisher pulp		<0.50	<0.25	<0.75	<0.2	<0.23
Chopped fresh peel		1.58	<0.25	1.87	0.48	0.55
Cold pressed oil		78.9	0.16	79.1	23.8	23.5
Oil emulsion water (waste)		1	<0.05	1.06	0.3	0.31
Dried peel		7.66	0.29	8	2.31	2.37
Molasses		0.76	0.13	0.91	0.23	0.27
Whole fruit (unwashed)	0.8	7.43	<0.05	7.49	—	—
Whole fruit (washed)		2.36	<0.05	2.42	0.32	0.32
Juice		0.1	<0.05	0.16	0.01	0.02
Finisher pulp		<0.50	<0.25	<0.75	<0.10	<0.10
Chopped fresh peel		2.19	<0.05	2.24	0.29	0.3
Cold pressed oil		248	0.46	249	33.4	33.2
Oil emulsion water (waste)		2.62	<0.05	2.67	0.5	0.36
Dried peel		18.1	0.38	18.6	2.44	2.48
Molasses		2.24	0.23	2.5	0.3	0.33
Grapefruit						
Whole fruit (unwashed)	0.4	3.99	<0.05	4.05	—	—
Whole fruit (washed)		1.09	<0.05	1.15	0.27	0.28
Juice		<0.05	<0.05	<0.11	<0.02	<0.03
Finisher pulp		<0.50	<0.25	<0.75	<0.20	<0.20
Chopped fresh peel		1.34	<0.05	1.63	0.34	0.4
Cold pressed oil		46.7	0.04	46.7	11.7	11.5
Oil emulsion water (waste)		2.61	<0.05	2.66	0.65	0.66
Dried peel		4.39	<0.05	4.44	1.1	1.1
Molasses		1.51	<0.05	1.56	0.38	0.39
Whole fruit (unwashed)	0.8	8.08	<0.05	8.14	—	—
Whole fruit (washed)		3.57	<0.05	3.62	0.44	0.45
Juice		0.073	<0.05	0.13	0.01	0.02
Finisher pulp		<0.50	<0.25	<0.75	<0.10	<0.10
Chopped fresh peel		3.4	<0.05	3.45	0.42	0.42
Cold pressed oil		146	0.1	146	18.1	18

Process	Application Rate (kg ai/hL)	Average Residues (mg/kg)			Processing Factors	
		Imazalil	R014821	Total[a]	Imazalil	Total
Oil emulsion water (waste)		5.89	<0.05	5.94	0.72	0.73
Dried peel		12.7	<0.05	12.8	1.57	1.57
Molasses		4.45	0.06	4.53	0.55	0.56
Washing (median PF)					0.36	0.35
Juice (median PF)					<0.02	0.03
Finisher pulp (median PF)					<0.20	0.11
Chopped fresh peel (median PF)					0.36	0.41
Cold pressed oil (median PF)					11.9	14.8
Oil emulsion water (waste) (median PF)					0.5	0.44
Dried peel (median PF)					1.34	1.41
Molasses (median PF)					0.45	0.38

[a] Expressed as imazalil equivalents

Orange – Study 1

One processing study conducted in Spain in 1998 was carried out on oranges (Wilson, 1998, R-10465). Following 10 days of cold storage, the oranges received either a drench application of an emulsifiable concentrate containing 500 g/L of imazalil at 0.05 kg ai/hL or a drench application at 0.05 kg ai/hL and after 10, 25, 40 or 56 days of cold storage an on-line spray application at 0.4 kg ai/hL. Treated oranges were collected immediately after treatment and transported to the processing facility under refrigerated conditions. At the processing facility, samples were processed within one day of receipt to juice and marmalade according to commercial practices. For juice, the whole oranges were cut in two parts and put piece by piece on the head of a juice extractor. The orange juice obtained was collected and a fraction of the wet pomace was stored while the remaining pomace was dried in an oven for 1-3 days.

For marmalade, the oranges were peeled and divided into quarters. Half of the peel (without pith) was incorporated with the orange quarters. The mixture was left to steep with the same weight of water for about 24 hours. After straining, half of the solid phase and half of the liquid phase were discarded. An equal weight of sugar was added to the remaining solid phase and combined with the liquid phase. The mixture was left to concentrate until the Brix degree reached approximately 62%. After cooling, the pH of the marmalade was adjusted to 3.5 with citric acid.

The HPLC-UV method MAK 513/984694 was used to determine residues of imazalil in the oranges, juice, pomace (wet and dry) and marmalade. The limit of quantification (LOQ) was 0.02 mg/kg for pulp, pomace, juice and marmalade and 0.05 mg/kg for peel. The maximum duration between sampling of the RAC and processed commodities and analysis was 16 weeks.

The processing study indicated that imazalil residues concentrated in wet and dry pomace yet they were reduced in juice and marmalade.

Table 63 Concentration of imazalil residues in orange processed fractions and processing factors

Application regime	Storage interval between treatments (days)	Commodity	Imazalil Residues (mg/kg)	PF
Drench at 0.05 kg ai/hL	NA	RAC fruit	2.54	—
		Wet pomace	4.41	1.74
		Dry pomace	11.38	4.48
		Juice	0.84	0.33
		Marmalade	0.71	0.28
Drench at 0.05 kg ai/hL + Spray at 0.4 kg ai/hL	10	RAC fruit	1.84	—
		Wet pomace	4.22	2.29
		Dry pomace	8.95	4.86
		Juice	0.26	0.14
		Marmalade	0.50	0.27
Drench at 0.05 kg ai/hL + Spray at 0.4 kg ai/hL	25	RAC fruit	2.52	—
		Wet pomace	5.14	2.04
		Dry pomace	11.06	4.39
		Juice	0.26	0.10
		Marmalade	0.64	0.25
Drench at 0.05 kg ai/hL	40	RAC fruit	2.19	—
		Wet pomace	4.45	2.03

Application regime	Storage interval between treatments (days)	Commodity	Imazalil Residues (mg/kg)	PF
+ Spray at 0.4 kg ai/hL		Dry pomace	8.86	4.05
		Juice	0.23	0.11
		Marmalade	0.54	0.25
Drench at 0.05 kg ai/hL + Spray at 0.4 kg ai/hL	56	RAC fruit	2.86	—
		Wet pomace	5.66	1.98
		Dry pomace	11.52	4.03
		Juice	0.25	0.35
		Marmalade	0.43	0.15
Wet pomace (median PF)				2.0
Dry pomace (median PF)				4.4
Juice (median PF)				0.14
Marmalade (median PF)				0.25

Orange – Study 2

Two processing trials were conducted in Spain in 2012 where oranges (varieties Navelina and Lane Late) were drenched using an emulsifiable concentrate containing 500 g/L of imazalil at a nominal application rate of 0.05 kg ai/hL (Auston, 2013, R-29507/R-29508). After storage for 31 days at approximately 5 ºC, the treated oranges were transported to the processing facility where they were washed and processed into juice, marmalade, jam, jelly, essential oil, canned oranges and dried oranges, according to commercial practices.

The determination of imazalil and the metabolite R014821 in citrus processed commodities was performed using a QuEChERS extraction and dispersive SPE method followed by LC-MS/MS analysis. The limit of quantification (LOQ) was 0.01 mg/kg/analyte.

The longest period between sampling and extraction of the whole orange fractions was 129 days which is within the demonstrated storage stability period of citrus. The longest period between sampling and extraction of processed fractions was 352 days which is within the demonstrated storage stability period of citrus processing fractions marmalade and essential oil.

The imazalil residue levels were higher in the peel than in the peeled orange and stayed in the peel during processing. Residues increased in the essential oil processing, as it uses only the peel, and in the dried sliced oranges, likely due to dehydration. Imazalil residues decreased in marmalade as processing to marmalade uses only some of the peel, and in juice, jam, jelly and canned orange processing as they do not use the peel. Washing had an insignificant effect on the imazalil residues.

Table 64 Concentration of imazalil and R014821 residues in orange processed fractions and processing factors

Process	DAA (days)	Residues (mg/kg)			Processing Factors	
		Imazalil	R014821	Total[a]	Imazalil	Total
Trial SX11014/1						
Fruit (RAC)	31	0.972	0.024	1.00	—	—
Fruit (washed)		0.908	0.026	0.938	0.93	0.94
Juice		0.027	<0.01	0.037	0.03	0.04
Marmalade		0.544	<0.01	0.554	0.56	0.56
Jam		0.031	<0.01	0.041	0.03	0.04
Jelly		0.023	<0.01	0.033	0.02	0.03
Essential oil		90.4	0.175	90.6	93	91
Canned orange		<0.01	<0.01	<0.02	<0.02	<0.02
Dried orange		1.58	0.061	1.65	1.6	1.7
Wet pomace		2.18	0.049	2.24	2.2	2.2
Dry pomace		6.52	0.179	6.73	6.7	6.7
Trial SX11014/2						
Fruit (RAC)	31	0.636	0.024	0.664	—	—
Fruit (washed)		0.680	0.028	0.712	1.1	1.1
Juice		0.034	<0.01	0.046	0.05	0.07
Marmalade		0.430	0.014	0.446	0.68	0.67
Jam		0.023	<0.01	0.035	0.04	0.05
Jelly		0.017	<0.01	0.029	0.03	0.04
Essential oil		80.8	0.190	81.0	127	122
Canned orange		0.025	<0.01	0.037	0.04	0.06
Dried orange		2.03	0.079	2.12	3.2	3.2

Process	DAA (days)	Residues (mg/kg)			Processing Factors	
		Imazalil	R014821	Total[a]	Imazalil	Total
Wet pomace		1.73	0.081	1.82	2.7	2.7
Dry pomace		6.08	0.283	6.41	9.6	9.7
Washing (mean PF):					0.97	0.98
Juice (mean PF):					0.04	0.05
Marmalade (mean PF):					0.62	0.61
Jam (mean PF):					0.04	0.05
Jelly (mean PF):					0.03	0.04
Essential oil (mean PF):					110	106
Canned orange (mean PF):					0.03	0.04
Dried orange (mean PF):					2.4	2.4
Wet pomace (mean PF):					2.5	2.5
Dried pomace (mean PF):					8.3	8.2

[a] Expressed as imazalil equivalents

Potato

Two processing trials were carried out in Switzerland in 2012 on potatoes (varieties Désirée and Agria) following a post-harvest treatment with a soluble liquid formulation containing 100 g/L of imazalil (Kreke, 2013, R-29521/ Zeisler, 2013, R-29522). The post-harvest application was carried out by placing the potatoes in a spraying chamber and treating at an application rate of 0.735 kg ai/hL corresponding to 15 g ai/ton potatoes. Treated potatoes were initially washed with tap water and placed in a cold storage for one month. The washed potatoes were either divided into two parts, peeled potato and peel or processed into baked, boiled, microwaved, canned and steamed potatoes, crisps, fries and flakes (dehydrated), according to typical food preparation practices.

The method used for analysis of imazalil and R014821 was based on the QuEChERS multi-residue method (L00.00-115/D55142). Concentrations of imazalil and of R014821 were determined by LC-MS/MS detection. The limit of quantification (LOQ) was 0.01 mg/kg/analyte.

The maximum storage interval from first sampling until analysis of treated tubers (RAC) was 6 months. The maximum storage duration of processed commodities was 11 months, while the demonstrated storage stability of imazalil in potato is 9 months. Nevertheless, no dissipation of imazalil and R014821 residues is anticipated in the additional 2 months of storage.

Total residues of imazalil and R014821 concentrated in microwaved potatoes while residues reduced in baked, boiled, peeled, canned and steamed potatoes, crisps, fries and flakes.

Table 65 Concentration of imazalil and R014821 residues in potato processed fractions and processing factors

Process	DAA (days)	Residues (mg/kg)			Processing Factors	
		Imazalil	R014821	Total[a]	Imazalil	Total
Trial A/CH/F/12/1						
Tubers (RAC)	29	6.25	0.244	6.53	–	–
Tubers (washed)		3.81*	0.117	3.95	0.61	0.60
Baked (without foil)		2.49	0.062	2.56	0.40	0.39
Baked with aluminium foil		2.31*	0.039	2.36	0.37	0.36
Boiled (washed with peel)		3.13*	0.044	3.18	0.50	0.49
Microwaved		6.80	0.119	6.94	1.1	1.1
Peel		19.6	0.446	20.1	3.2	3.1
Peeled		0.017	< 0.01	0.027	0.003	0.004
Steamed		0.335**	<0.01	0.345	0.05	0.05
Fries		0.099*	< 0.01	0.109	0.02	0.02
Crisps		0.121	< 0.01	0.131	0.02	0.02
Canned (after sterilization)		0.042*	< 0.01	0.052	0.01	0.01
Flakes		< 0.01	< 0.01	<0.02	<0.002	<0.004
Trial A/CH/F/12/2						
Tubers (RAC)	29	4.91	0.262	5.21	–	–
Tubers (washed)		4.24*	0.147	4.41	0.86	0.85

Process	DAA (days)	Residues (mg/kg)			Processing Factors	
		Imazalil	R014821	Total[a]	Imazalil	Total
Baked (without foil)		3.95	0.100	4.07	0.80	0.78
Baked with aluminium foil		2.96*	0.046	3.01	0.60	0.58
Boiled (washed with peel)		1.36*	0.022	1.39	0.28	0.27
Microwaved		7.76	0.159	7.94	1.6	1.5
Peel		13.3	0.360	13.7	2.7	2.6
Peeled		0.020	<0.01	0.030	0.01	0.01
Steamed		0.053*	<0.01	0.063	0.01	0.01
Fries		0.039*	<0.01	0.049	0.01	0.01
Crisps		0.100	<0.01	0.110	0.02	0.02
Canned (after sterilization)		0.012*	<0.01	0.022	0.002	0.004
Flakes		< 0.01***	<0.01	<0.02	<0.002	<0.004
Washed (mean PF):					0.74	0.72
Baked without foil (mean PF):					0.60	0.59
Baked with aluminium foil (mean PF):					0.49	0.47
Boiled (mean PF):					0.39	0.38
Microwaved (mean PF):					1.4	1.3
Peeled (mean PF):					0.01	0.01
Peel (mean PF):					3.0	2.9
Steamed (mean PF):					0.03	0.03
Fries (mean PF):					0.02	0.01
Crisps (mean PF):					0.02	0.02
Canned (mean PF):					0.01	0.01
Flakes (mean PF):					<0.002	<0.004

[a] Expressed as imazalil equivalents

*mean value of triple analysis

**mean value of quadruple analysis

***mean value of double analysis

Residues in animal commodities

Dairy Cattle

Twelve dairy cows (Holstein, 2–8 years old, 520-740 kg bw) were divided into four test groups (van Dijk, 1998, 663186), where the first group of three cows served as a control group while the remaining three groups consisted of three cows each. Animals were dosed with imazalil twice daily for 28 consecutive days by means of gelatine capsules administered with a balling gun. The doses administered to the low, mid and high dose treatment groups were 46.4, 148.6 and 440.5 mg of imazalil/kg feed/day, respectively. During the study, daily feed consumption and milk production were constant during the administration period of 4 weeks and were on average 22 kg/day and 20 kg/day, respectively. Cows were sacrificed 24 hours after administration of the last dose. Milk collection included the morning and evening milkings on the first day of administration of imazalil and every subsequent evening and morning thereafter. All milk samples were frozen at –20 °C and analysed within 30 days of collection. Tissue samples collected at sacrifice included liver, kidney, mesenteral and perirenal fat, round and loin muscle. All tissue samples were stored frozen and analysed within 30 days of collection. As milk and tissue samples were stored for up to 30 days, no concurrent storage stability study was conducted.

Samples were analysed for residues of imazalil, R06100 and R043449 using the validated GC-ECD method 651554.

Residues of imazalil, R06100 and R043449 were <LOQ (0.02 mg/kg) in milk samples collected from the low and mid dose groups.

Table 66 Residues in whole milk following administration of imazalil to dairy cows at the highest feeding level (440.5 mg/kg feed/day)

Day	High dose (440.5 mg/kg feed/day)		
	Imazalil	R06100	R043449
0	<0.02 (<0.02, <0.02, <0.02)	<0.02 (<0.02, <0.02, <0.02)	<0.02 (<0.02, <0.02, <0.02)
1	0.023 (0.029, <0.02, <0.02)	0.02 (0.02, <0.02, <0.02)	0.058 (0.063, 0.055, 0.055)
2	0.046 (0.072, 0.036, 0.043)	0.025 (0.028, 0.026, <0.02)	0.109 (0.130, 0.116, 0.080)
3	0.120 (0.110, 0.092, 0.158)	0.026 (0.031, 0.025, 0.021)	0.193 (0.209, 0.204, 0.166)
4	0.126 (0.043, 0.206, 0.129)	0.029 (0.027, 0.027, 0.032)	0.159 (0.094, 0.250, 0.133)
5	0.060 (0.035, 0.064, 0.081)	0.036 (0.029, 0.041, 0.039)	0.170 (0.113, 0.230, 0.168)
6	0.066 (0.046, 0.075, 0.077)	0.057 (0.050, 0.069, 0.052)	0.172 (0.137, 0.228, 0.152)
7	0.072 (0.067, 0.072, 0.078)	0.047 (0.055, 0.046, 0.041)	0.171 (0.180, 0.200, 0.134)
8	0.058 (0.051, 0.064, 0.058)	0.030 (0.031, 0.033, 0.027)	0.141 (0.124, 0.187, 0.113)
12	0.036 (0.030, 0.039, 0.039)	0.028 (0.031, 0.030, 0.024)	0.133 (0.126, 0.160, 0.113)
15	0.049 (0.047, 0.057, 0.043)	0.043 (0.047, 0.049, 0.033)	0.084 (0.112, 0.049, 0.092)
19	0.032 (0.026, 0.037, 0.033)	0.073 (0.062, 0.108, 0.049)	0.098 (0.097, 0.108, 0.089)
22	0.026(0.025, 0.028, 0.026)	0.026 (0.030, 0.026, 0.023)	0.107 (0.107, 0.127, 0.086)
28	<0.02 (<0.02, <0.02, <0.02)	0.020 (0.020, 0.021, <0.02)	0.067 (0.063, 0.084, 0.053)

In muscle, residues of imazalil, R06100 and R043449 were <LOQ at the low dose feeding level. At the mid dose feeding level, residues of imazalil and R06100 were <LOQ and at the highest feeding level, only residues of imazalil remained <LOQ.

In fat, residues of imazalil, R06100 and R043449 were <LOQ at the low and mid dose feeling levels.

Table 67 Residues in muscle of R06100 in the high dose group and residues of R043449 in the mid and high dose groups.

Tissue	Mid dose (148.6 mg/kg feed/day)	High dose (440.5 mg/kg feed/day)		
	R043449	Imazalil	R06100	R043449
Muscle	0.031 (0.038, 0.037, 0.017)	<0.02 (<0.02, <0.02, <0.02)	0.034 (0.063, 0.021, <0.02)	0.094 (0.097, 0.103, 0.082)
Fat	<0.02 (<0.02, <0.02, <0.02)	0.136 (0.128, 0.070, 0.210)	0.033 (0.033, 0.023, 0.043)	0.051 (0.057, 0.017, 0.078)

In liver and kidney, quantifiable residues of imazalil, R06100 and R043449 were observed at all dose levels.

Table 68 Residues of imazalil, R06100 and R043449 in liver and kidney at all dose levels.

Tissue	Low dose (46.4 mg/kg feed/day)			Mid dose (148.6 mg/kg feed/day)			High dose (440.5 mg/kg feed/day)		
	Imazalil	R06100	R043449	Imazalil	R06100	R043449	Imazalil	R06100	R043449
Liver	0.326 (0.324, 0.362, 0.259)	0.153 (0.075, 0.224, 0.159)	0.153 (0.113, 0.213, 0.133)	2.156 (2.342, 2.640, 1.488)	0.329 (0.313, 0.501, 0.164)	0.336 (0.408, 0.433, 0.166)	9.206 (7.873, 11.562, 8.184)	0.773 (0.673, 0.778, 0.868)	1.081 (0.837, 1.508, 0.897)
Kidney	0.020 (<0.02, 0.033, 0.022)	0.069 (0.059, 0.070, 0.078)	0.022 (<0.02, <0.02, 0.026)	0.197 (0.256, 0.178, 0.157)	0.236 (0.251, 0.264, 0.192)	0.054 (0.064, 0.040, 0.060)	0.910 (0.624, 1.214, 0.892)	1.005 (0.690, 1.284, 1.040)	0.676 (0.386, 1.064, 0.578)

Laying Hen

Fourty hens (White leghorn, 7 months old, 1.32-1.77 kg bw) were divided into four test groups (van Dijk, 1999, 687982), where each group consisted of 10 hens, one of which served as a control group, while each of the remaining three groups was divided into 3 subgroups of 3, 3, and 4 hens. Animals were dosed with imazalil once daily for 28 consecutive days by means of gelatine capsules. The doses administered to the low, mid and high dose treatment groups were 0.225, 0.728 and 2.56 mg of imazalil/kg feed/day, respectively. During the study, daily feed consumption and egg production were constant during the administration period of 4 weeks and were 104-109 g/hen and 93-100%, respectively. Hens were sacrificed 24 hours after administration of the last dose. Eggs were collected daily after dose administration. All egg samples were frozen at -20 °C and analysed within 30 days of collection.

Tissue samples collected at sacrifice included liver, fat and muscle. All tissue samples were stored frozen and analysed within 14 days of collection. As egg and tissue samples were stored for up to 30 days, no concurrent storage stability study was conducted.

Tissue and egg samples were analysed for residues of imazalil, R042639 and R044085, while eggs were also analysed for residues of the metabolite R110740. The validated LC-MS method 687971 was used to conduct the analysis.

Residues of imazalil, R042639 and R044085 were each <LOQ (0.02 mg/kg/analyte) in tissue samples collected at all dose levels. Similarly, residues of imazalil, R042639, R044085 and R110470 in eggs were each <LOQ (0.01 mg/kg/analyte) at all dosing levels. Hence, there was no evidence of transfer of imazalil from the feed into poultry tissues and eggs, and no metabolic degradation of imazalil into the metabolites in any of the poultry matrices.

APPRAISAL

Imazalil is an imidazole fungicide with a protective, curative and anti-sporulation mode of action.

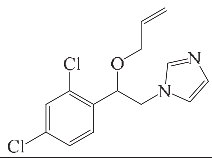
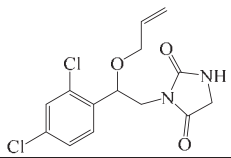
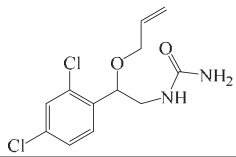
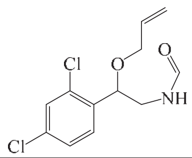
Imazalil was first evaluated by the JMPR in 1977. The current ADI, established in 1991, is 0–0.03 mg/kg bw. In 2005, the JMPR set an ARfD of 0.05 mg/kg bw.

Imazalil was scheduled at the Forty-ninth Session of the CCPR for Periodic Review for residues and toxicology by the 2018 JMPR.

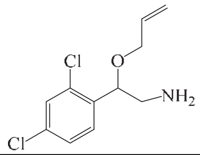
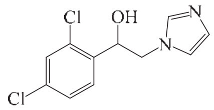
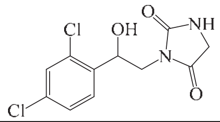
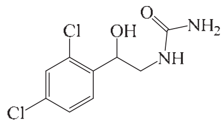
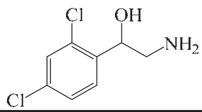
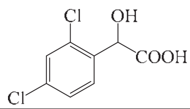
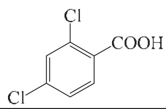
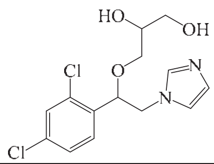
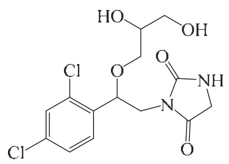
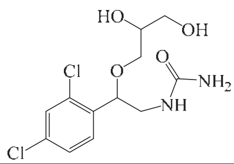
The Meeting received information from the manufacturer on physical and chemical properties, metabolism studies on plants and animals, environmental fate in soil, analytical methods and stability in stored analytical samples, use patterns and supervised residue trials, processing studies, and livestock feeding studies.

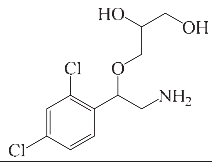
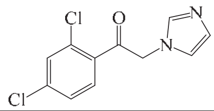
Imazalil is a racemate, consisting of equal amounts of two enantiomers.

Metabolites referred to in the appraisal are addressed by their company code numbers:

List of metabolites and degradates of imazalil			
Code Name/ Number	Chemical Name	Chemical Structure	Occurrence in
Imazalil	(*RS*)-1-[2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl]-1*H*-imidazole		Ruminant, Poultry, Potato, Tomato, Wheat, Banana, Apple, Orange
R044179	(*RS*)-1-[2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl]-imidazolidine-2,5-dione		Ruminant, Tomato, Cucumber
R092977	(*RS*)-1-[2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl]-urea		Tomato
R055609	*N*-(2-(2,4-dichlorophenyl)-2-(2-propenyloxy)ethyl)formamide		Ruminant, Tomato

List of metabolites and degradates of imazalil

Code Name/ Number	Chemical Name	Chemical Structure	Occurrence in
R044177	2-(2,4-dichlorophenyl)-2-(2-propenyloxy)-ethan-1-amine		Ruminant, Potato
R014821	(*RS*)-1-(2,4-dichlorophenyl)-2- imidazol-1-yl-ethanol		Rat, Ruminant, Potato, Tomato, Banana, Apple, Orange, Wheat
R043449	(*RS*)-3-[2-(2,4-dichlorophenyl)-2-hydroxyethyl]imidazolidine-2,4-dione		Ruminant Poultry, Tomato
R044085	(*RS*)-*N*-[2-(2,4-dichlorophenyl)-2-hydroxyethyl]urea (±)-*N*-[2-(2,4-dichlorophenyl)-2-hydroxyethyl]urea		Rat, Ruminant, Poultry
R110740	2-amino-1-(2,4-dichlorophenyl)ethan-1-ol		Poultry
R023366	2-(2,4-dichlorophenyl)-2-hydroxyacetic acid		Ruminant
R066996	2,4-dichlorobenzoic acid		Ruminant
R042639	(*RS*)-1-[[2-(2,4-dichlorophenyl)-2-(2,3-dihydroxypropyl)oxy]ethyl]-1*H*-imidazole (±)-1-[[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-1*H*-imidazole		Rat, Ruminant, Poultry, Tomato, Wheat
R061000	(*RS*)-3-[2-(2,4-dichlorophenyl)-2-(2,3-dihydroxypropoxy)ethyl]imidazolidine- 2,4-dione (±)-1-[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-2,5-imidazolidinedione		Rat, Ruminant, Poultry
R062775	1-(2-(2,4-dichlorophenyl)-2-(2,3-dihydroxypropoxy)ethyl)urea		Ruminant, Poultry

List of metabolites and degradates of imazalil			
Code Name/ Number	Chemical Name	Chemical Structure	Occurrence in
R060998	3-(2-amino-1-(2,4-dichlorophenyl)ethoxy)propane-1,2-diol		Ruminant, Poultry
R045529	1-(2,4-dichlorophenyl)-2-(1*H*-imidazol-1-yl)ethan-1-one		Tomato

Based on the physical chemical properties, imazalil is only slightly volatile and is soluble in water and polar solvents. Imazalil is stable to aqueous hydrolysis yet undergoes continuous photolysis in aquatic environments (DT_{50} = 11–12 days). Based on the Log Kow, imazalil is not likely to sequester to fatty matrices.

Plant metabolism

The Meeting received plant metabolism studies with imazalil following post-harvest treatment to oranges, apples and potatoes, foliar applications to bananas, greenhouse cucumbers and tomatoes and seed treatment of potatoes and cereal grains.

Oranges – Post-harvest treatment

Spanish navel oranges (variety *Valencia*) were dipped in an aqueous solution of [^{3}H]-imazalil sulphate, labelled on the stereogenic carbon, at a rate equivalent to 0.05 kg ai/hL (0.2-fold critical GAP). After treatment, the oranges were stored in a dark room. Samples were taken immediately (2 hours) after dipping, then after 1, 3, 6 and 12 weeks. For all samples, peel and pulp were analysed separately. Results for the whole fruits were calculated using the respective weights of peel and pulp.

The total radioactive residues (TRRs) remained relatively unchanged over the entire storage duration in whole fruit, peel and pulp, ranging from 1.9–2.6 mg eq/kg, 5.8–7.2 mg eq/kg and 0.04–0.1 mg eq/kg, respectively, with the majority of the radioactivity predominantly found in the peel.

Sequential extraction of peel and pulp samples with methanol:concentrated ammonia, methanol: glacial acetic acid and heptane:isoamyl alcohol released 76–93% TRR and 71–88% TRR, respectively, The radioactivity in the post-extraction solids ranged from 3–9% TRR.

Imazalil accounted for the majority of the radioactivity in the peel, decreasing from 78% TRR, 2 hours after treatment, to 57% TRR, 12 weeks thereafter. Radioactivity in pulp followed a similar trend, decreasing from 68% TRR, 2-hours post-treatment, to 45% TRR following a 12-week storage period. The concentrations of the alcohol metabolite, R014821, in peel and pulp reached maximum levels following 12 weeks of storage (4.6% TRR [0.29 mg eq/kg]; 17.7% TRR [0.01 mg eq/kg], respectively). A limitation of the study included the lack of accountability of the applied radioactivity including identification of all potential metabolites.

Apple – Post-harvest treatment

Apples (*variety* Golden Delicious) were dipped in solutions of unlabelled imazalil-sulfate at a rate of 0.05 kg ai/hL to determine the residue level of the parent compound and its metabolites, equivalent to a magnitude of the residue study. Another set of apples was treated with a blend of [^{14}C]-imazalil labelled on the stereogenic carbon and unlabelled imazalil (ratio 1:9) to investigate the degradation of imazalil and to determine the extractability of the radioactivity as a function of storage time. Fruits were subsequently stored in a controlled atmosphere and sampled immediately (4 hours) after dipping and after 1, 2, 4, 6 and 7 months of storage. One [^{14}C]-imazalil apple was treated with the same radiolabelled imazalil test substance for the volatilization test. The treated apple was incubated in a flow-through system similar to that of soil metabolism studies and was initiated on the same day as the storage samples and continued until the end of the storage period (7 months).

Residues in apples treated with radiolabelled material ranged from 2.4–3.2 mg eq/kg which were higher than those treated at the same rate with unlabelled imazalil, where residues ranged from 1.5–2.0 mg/kg.

Residues extracted using heptane:isoamyl alcohol (95:5, v:v) accounted for 97% TRR immediately after treatment and decreased to 82% TRR following 7 months of storage. Imazalil and R014821 accounted for most of the extracted radioactivity, where levels of imazalil decreased (95% TRR to 73% TRR) with a corresponding increase in R014821 (2% TRR to 9% TRR), as a function of storage duration. No other transformation product was identified. The radioactivity levels in the trapping solutions did not exceed those in the control (blank) solutions. Therefore, volatilization of imazalil and imazalil-derived metabolites does not appear to be a route of dissipation.

Tomato – Foliar application

Greenhouse tomatoes were treated with three foliar applications of [^{14}C- U-ring]-imazalil formulated as an emulsifiable concentrate at a rate of 300 g ai/ha per application for a total application rate of 900 g ai/ha (low dose; 4.5-fold cGAP)) during the growing period. Additional plants were treated three times at 1500 g ai/ha per application for a seasonal application rate of 4500 g ai/ha (high dose; 22.5-fold cGAP). Treatments were made at 10-day intervals and tomatoes were harvested 1 day after the last application (DALA).

The TRR in the tomatoes represented 0.44 mg eq/kg (low dose) and 2.85 mg eq/kg (high dose).

Highest TRRs were in the acetonitrile surface wash (≥ 50% TRR) followed by pomace (~40% TRR), with < 10% TRR found in the tomato juice.

Extraction of the TRRs in pomace using acetonitrile, methanol and acetonitrile-water was limited, ranging from 13–15% TRR. However, following hydrolysis of the PES using basic harsh conditions, the unextracted residues remaining in the pomace accounted for 4.0% and 0.5% TRR for the low and high dose rates, respectively.

In whole tomato fruit, the free parent compound represented 54% TRR (0.23–1.5 mg eq/kg) for both dose levels, while the conjugated parent, released following hydrolysis of the PES, accounted for 20–23% TRR (0.09–0.66 mg eq/kg). In addition to the parent compound, up to five known metabolites (R043449, R042639/R045529, R014821, R055609/R092977 and R044179) were detected at both dose levels, none of which exceeded 10% TRR (≤ 0.17 mg eq/kg).

Enantiomeric analysis of the juice, pomace extracts and surface wash revealed that no change in the enantiomer ratio is expected and that stereospecific metabolism of imazalil is highly unlikely.

Potato – Post-harvest treatment

[^{14}C]-Imazalil uniformly labelled in the phenyl ring and formulated as a solution containing100 g ai/L, was applied by simulated drench application to ten potato tubers (variety *Laura*) at a target rate of 15 mg ai/kg potato tubers (1-fold cGAP), after which they were stored on a wire grid in storage boxes at approximately 5 °C.

TRRs in potato tuber samples, taken immediately after treatment (day 0) and after 14, 29, 91 and 188 days of storage, decreased slightly from 17.2 mg eq/kg to 15.6 mg eq/kg.

At each sampling interval, potato tubers were washed with acetonitrile:water. The residue in the surface wash decreased from 64% TRR (11 mg eq/kg) on day 0 to 35% TRR (5.4 mg eq/kg) by day 188. Washed potatoes were subsequently peeled. The extracted residue in the peel increased from 33% TRR (5.7 mg eq/kg) on day 0 to 46% TRR on day 188 (7.2 mg eq/kg). The unextracted peel residue increased from 3% TRR (0.5 mg eq/kg) on day 0 to a maximum of 23% TRR (3.6 mg eq/kg) on day 91. Residues released following acidic reflux were significantly lower than those in the surface wash and the pomace extracts. Residues in peeled tubers were approximately 0.2% TRR (0.03 mg eq/kg) except for day 14 where the radioactivity in the tuber accounted for 0.9% TRR (0.15 mg eq/kg). Based on the distribution of the radioactivity in the treated potato tubers, limited penetration was observed from the peel into the pulp.

HPLC-UV analysis of the surface wash and peel extracts confirmed the presence of imazalil as the predominant residue , decreasing from 94% TRR (16.1 mg eq/kg) on day 0 to 70% TRR (10.9 mg eq/kg) on day 188. The metabolites R014821 and R044177 reached a maximum of 9.0% TRR (1.4 mg eq/kg) and 3.4% TRR (0.54 mg eq/kg), respectively, in surface wash and peel extracts by day 188. Imazalil accounted for < 0.02% TRR (< 0.003 mg/kg) in the tuber extract. Unknown metabolites ranged from 3–6% TRR (0.5–1.0 mg eq/kg). Following acidic reflux extraction of the peel solids, the analytes identified included the parent (1.3–4.0 % TRR), metabolites R014821 (0.1–0.2% TRR) and R044177 (0.04–0.1% TRR) as well as other unknown fractions that were likely aglycones of conjugates hydrolysed under acidic reflux conditions.

Enantiomeric analysis of the surface wash and peel extract sampled on day 0, 91 and 188 showed that the ratios of imazalil enantiomers remained unchanged during storage of treated potato and that stereospecific metabolism of imazalil is highly unlikely.

Spring wheat – Seed treatment

^{14}C-imazalil, radiolabelled on the stereogenic carbon, was applied to spring wheat seeds (variety *Axona*) at a rate of 49.3 g ai/100 kg seed (5-fold cGAP). Treated seeds were sown in pots left outdoors. Forage was sampled 42 days after sowing while straw and grain were harvested approximately 5 months after sowing.

TRRs in forage, straw and grain were 1.36, 0.15 and 0.003 mg eq/kg, respectively. Due to the very low level of TRRs in grain, no further investigation was carried out.

Greater than 72% TRR (0.99 mg eq/kg) in the forage was extracted using methanol:chloroform:HCl (0.1 N) (2:1:0.8; v:v:v) followed by Soxhlet extraction with methanol:HCl (0.1 N). Successive mild and harsh acid and base hydrolysis of the PES released 12.6% TRR (0.16 mg eq/kg). The radioactivity in the remaining bound residues accounted for 7.7% TRR (0.11 mg eq/kg). Similar extraction of mature straw released significantly less radioactivity compared to forage (44% TRR; 0.06 mg eq/kg). Mild and harsh acid and base hydrolysis released a further 16.4% TRR (< 0.03 mg eq/kg) with 58.5% TRR (0.09 mg eq/kg) remaining in the bound residues.

The parent compound was observed in both matrices, 24% TRR (0.33 mg /kg) in forage and 17% TRR (0.03 mg /kg) in straw. The levels of the metabolites R014821 and R042639 in forage were 8% TRR (0.11 mg eq/kg) and 5% TRR (0.06 mg eq/kg), respectively, while both were <4% TRR (< 0.01 mg eq/kg) in straw. Several unknown compounds with similar retention times were observed in both forage and straw, none of which accounted for > 4.3% TRR.

Banana, Cucumber – Foliar application / Barley, Potato – Seed treatment

Various experiments were conducted on foliar-treated greenhouse-grown bananas and cucumber seedlings and seed-treated barley and potatoes.

While limited in scope, these studies demonstrated that the majority of the radioactivity present in the banana, cucumber, barley and potato plants was located predominantly on the site of application with limited radioactivity in the untreated plant parts including edible commodities (bananas, cucumbers, barley grain and potato tubers), demonstrating minimal translocation.

In summary, the metabolism of imazalil is adequately understood in fruits and vegetables following post-harvest, foliar-treated greenhouse vegetables and seed treated cereal grains. The majority of the radioactive residues in oranges, apples and potatoes, following post-harvest treatment, were located on the surface of the crops with limited penetration from the peel to the pulp. The predominant analyte was the parent, imazalil with the metabolite R014821 which accounted for significantly lower levels than those of the parent.

Following foliar application of imazalil to greenhouse grown tomatoes, imazalil accounted for the majority of the identified radioactivity, however, six metabolites were also identified at lower levels than those of the parent, demonstrating a more extensive metabolic profile than that following post-harvest treatment.

In the spring wheat seed-treatment metabolism study, the metabolism of imazalil was similar to that following post-harvest treatment, where limited metabolism of imazalil was observed with R014821 and R042639 being the only two metabolites identified,

While limited in scope, the banana and cucumber (foliar application) and barley and potato (seed treatment) studies demonstrated that the majority of the radioactivity present on the plants was located in most part on the site of application with limited radioactivity in the untreated plant parts including edible commodities (bananas, cucumbers, barley grain and potato tubers), demonstrating minimal translocation.

The degradation of ^{14}C-imazalil proceeds predominantly via O-dealkylation to form metabolite R014821, While less predominant, imazalil also undergoes dihydroxylation followed by cleavage of the imidazole ring and hydroxylation of the alkyl chain. The major plant metabolite, R014821, was identified as a major metabolite in rats.

Animal metabolism

The Meeting received animal metabolism studies with imazalil in goats, hens and rats. Evaluation of the rat metabolism study was carried out by the WHO Core Assessment Group.

Lactating goat

The metabolism of ^{14}C-imazalil, radiolabelled on the stereogenic carbon, was investigated in a lactating goat, dosed orally by intubation, twice daily at a dose level of 188 ppm for 3 consecutive days. The goat was sacrificed 6 hours after administration of the last dose.

Greater than half of the administered dose (AD) was excreta-related (57% AD). Limited radioactivity was eliminated in the milk (0.1% AD) and the tissue burden was low (1.4% AD). The overall recovered radioactivity accounted for 59% AD.

At sacrifice, TRRs were highest in liver (19.8 mg eq/kg), followed by kidney (9.6 mg eq/kg), muscle (round, loin and flank; 0.36 mg eq/kg) and fat (perirenal, omental, subcutaneous; 0.09 mg eq/kg).

Successive extractions of milk and tissues using various organic solvents released up to 97% of the radioactivity .The unextracted residues ranged from 5–12% TRR.

The distribution of radioactivity in milk (56–72 hours after the last administration, containing the highest amount of radioactivity) indicated that the majority of the radioactive residue (92% TRR) was contained in whey, while those in fat and protein accounted for 7% and 4% TRR, respectively. These fractions were not further analysed. Imazalil was not observed in milk whey, however, a total of 10 minor metabolites were identified, ranging from 1.0–7.2% TRR (0.01–0.07 mg eq/kg).

In liver, imazalil accounted for a total of 6% TRR (1.3 mg/kg). Nine additional minor metabolites were identified, none of which accounted for greater than 5% TRR (0.99 mg eq/kg).

Imazalil accounted for a total of 4% TRR (0.4 mg /kg) in kidney and 3% TRR (0.009 mg/kg) in muscle. The metabolite R061000 was a predominant metabolite in kidney representing 15% TRR (1.45 mg eq/kg), In muscle, R043449 and R061000, were both major metabolites accounting for 15% TRR (0.05 mg eq/kg) and 21% TRR (0.07 mg eq/kg), respectively. Four additional minor metabolites were observed in kidney, representing 0.5–3% TRR (0.05–0.3 mg eq/kg). In contrast, only 2 additional minor metabolites were observed in muscle, accounting for 7–8% TRR (0.02–0.03 mg eq/kg).

Imazalil was a minor residue (6% TRR; 0.006 mg /kg) in fat, as was the metabolite R043449, accounting for 9% TRR (0.02 mg eq/kg). R044179 was the only predominant metabolite observed, accounting for 25% TRR (0.03 mg eq/kg).

Up to 15 unknown metabolites were detected in milk whey and tissues, ranging from 0.2–34% TRR (0.01–2.2 mg/kg). In some cases, radio-HPLC analysis of metabolite fractions revealed that they comprised of several individual metabolites, none of which accounted for greater than 10% TRR. There was no evidence of conjugates in any of the ruminant matrices.

Laying hen

Ten laying hens were dosed orally for 10 consecutive days with [U-C^{14}]-imazalil at 66 ppm in the feed (dry matter). The hens were sacrificed 20 hours after the final dose.

Almost all the administered radioactivity was recovered in excreta (95% AD), cage wash and cage debris (4% AD). Limited radioactivity was eliminated in the eggs (0.3% AD) and the tissue burden was low (2% AD). The overall recovered radioactivity accounted for 101%.

TRRs were highest in liver (10 mg eq/kg) followed by skin (0.44 mg eq/kg), muscle (0.13–0.16 mg eq/kg) and fat (0.12–0.13 mg eq/kg).

Extraction of the eggs and tissues using methanol:ammonia followed by Soxhlet extraction of the PES released 54–85% of the TRRs.

Imazalil was not observed in liver as it underwent rapid and extensive metabolism to a large number of metabolites. Six metabolites were identified, including R016000, however, none accounted for greater than 10% TRR (≤ 0.95 mg eq/kg).

Imazalil was also not detected in muscle. However, in contrast with liver, only 2 major metabolites were identified, R042639 (15% TRR) and R110740/R044085, together accounting for 16% TRR (0.02 mg eq/kg). Following Soxhlet extraction of the PES, 33% TRR (0.05 mg eq/kg) remained unextracted. While, 30% TRR was further released following mild acid or base hydrolysis, no attempt to characterise the released radioactivity was undertaken.

In fat, only parent (11% TRR; 0.01 mg eq/kg) and the metabolite R043449 were identified (10% TRR, 0.01 mg eq/kg).

Imazalil was detected in eggs (8% TRR; 0.06 mg/kg) as were several metabolites accounting for a total of ≤ 11% TRR (≤ 0.09 mg eq/kg).

The Meeting concluded that, in all species investigated (goats, hens and rats), the total administered radioactivity was predominantly eliminated in excreta. The metabolic profiles differed quantitatively between the species, yet qualitatively there are no major differences with the exception that the metabolism in goats was more extensive than hens and rats. The routes and products of metabolism were similar across all animals, resulting from 1) dihydroxylation followed by cleavage of the imidazole ring; 2) hydroxylation of the alkyl chain followed by oxidation and cleavage of the imidazole ring; 3) dihydroxylation of the alkyl chain followed by oxidation and cleavage of the imidazole ring.

Environmental fate

While the Meeting received information on soil aerobic degradation, hydrolysis and photolysis properties of imazalil; studies on the behaviour of [^{14}C]-imazalil in confined rotational crops were not received.

Aerobic degradation in soil

The degradation of ^{14}C-imazalil radiolabelled at the stereogenic carbon was investigated in various soil types (including loam, sandy loam, silt loam and sandy clay loam) under aerobic laboratory conditions (10–25 °C for 120–366 days).

Following first order kinetics, the resulting DT_{50} values for imazalil ranged from 28–113 days.

The only radioactive degradation product exceeding 5% of the applied radioactivity (AR) was identified as R014821, accounting for up to 10% of AR. No other metabolites were identified.

The degradation rate of the soil metabolite, R014821, was also investigated in three soils (silt loam, sandy loam and silt loam) under aerobic laboratory conditions at 20 °C.

Following first order kinetics, the resulting DT_{50} values for R014821 ranged from 7–23 days.

Photolysis - Aqueous

Sterile buffer solutions maintained at pH 7 were treated with imazalil [^{14}C-phenyl-ring] at a mean initial concentration of about 0.15 mg/L and irradiated for a continuous period of 19 days.

^{14}C-Imazalil underwent continuous photolysis. By the end of the study, it had declined to 5% AR. In the dark control samples, virtually no hydrolysis of the test item was observed. Four photodegradates, accounting for greater than 10% of the applied radioactivity were formed: R044177, R044179, R055609 and R018238.

Overall, the Meeting concluded that the degradation of imazalil in soil proceeds via hydroxylation of the alkyl chain to form the alcohol metabolite R014821. Imazalil is moderately persistent in soil under field conditions (DT_{50} values ranging from 28–113 days), stable to hydrolysis yet undergoes photolysis in the aquatic environment (DT_{50} values ranging from 11–12 days).

Methods of analysis

QuEChERS based multi-residue methods have been reported in the scientific literature for the analysis of imazalil in orange juice and grapes. Adequate recoveries were obtained at LOQ's of 8.5 µg/L and 0.05 mg/kg, respectively.

The Meeting received the description and validation data for several analytical methods capable of quantifying imazalil and the alcohol metabolite, R014821, in plant commodities. Extraction of the residues was accomplished using various solvents such as heptane:isoamyl alcohol, hexane:acetone , ethyl acetate:hexane, acetonitrile:water and acetonitrile. Liquid partitioning was typically used for the clean-up step. The residue analytical methods relied on GC-ECD, GC-MS or LC-MS/MS detectors. The typical LOQ achieved for the plant commodities using the LC-MS/MS methods is 0.01 mg/kg. Methods were successfully validated by independent laboratories, demonstrating good reproducibility. The Meeting did not receive any information on radiovalidation.

The Meeting received descriptions and validation data for several analytical methods for residues of imazalil and its metabolites R043449 and R061000 in ruminant matrices and imazalil, R042639, R044085 and R110740 in poultry matrices. Extraction solvents used for each of the matrices were made up of different combinations of methanol and/or acetonitrile and/or acetone. Clean-up of the extracts was performed using liquid partitioning or an RP8 column eluted with various solvents. The residue analytical methods relied predominantly on GC-ECD and LC-MS/MS detectors. The LOQs achieved for all animal commodities were 0.02, 0.04 or 0.05 mg/kg (GC-ECD) and 0.01 mg/kg (LC-MS/MS). The LC-MS/MS methods were successfully validated by independent laboratories, demonstrating good reproducibility. The Meeting did not receive any information on radiovalidation.

Stability of pesticide residues in stored analytical samples

The Meeting received storage stability studies on imazalil and imazalil alcohol (R014821) in orange and its processed commodities, apple and its processed commodities, bananas, melons, tomatoes, potatoes and cereal grain and straw. Samples were fortified with each analyte at various concentrations and stored frozen. Samples were taken for analysis at intervals up to 12 months.

No dissipation of residues of imazalil and R014821 was observed in any of the raw agricultural commodities or the processed commodities.

Studies on storage stability of imazalil in animal tissues were not provided to the Meeting as all animal samples in the livestock feeding studies were extracted and analysed within 30 days from sampling.

Definition of the residue

The nature of the imazalil residues was investigated in oranges, apples and potatoes following post-harvest treatment, bananas, greenhouse cucumbers and tomatoes following foliar treatment, and potatoes, spring wheat and barley following seed treatment.

The majority of the radioactive residues in oranges, apples and potatoes, following post-harvest treatment were located on the surface of the crops with limited penetration from the peel to the pulp. The predominant analyte identified immediately after treatment (0-DAT) was the parent, imazalil (78–95% TRR). In all three studies, residues of imazalil decreased steadily as a function of storage duration with a corresponding increase in the residues of the metabolites R014821, and to a lesser extent R044177 (potato).

Following foliar application to greenhouse grown tomatoes at exaggerated rates (4.5–22.5–fold critical GAP), free imazalil accounted for the majority of the identified radioactivity (54% TRR). While five minor metabolites were also identified, none exceeded 10% TRR (0.002–0.17mg eq/kg).

In the spring wheat seed-treatment metabolism study conducted at exaggerated rates (10-fold cGAP), imazalil accounted for the majority of the extracted radioactivity (17–24% TRR) in forage and straw. The only metabolites identified in these feed commodities were R014821 and R042639, each representing < 10% TRR (≤ 0.11 mg eq/kg).

Imazalil was the only analyte present as a major compound in all tested plant matrices. Suitable analytical methods are available to analyse the parent compound. The Meeting considered that imazalil was a suitable marker for enforcement of MRLs for fruits, vegetables and cereal crops.

In deciding which compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence and the toxicological properties of the metabolite R014821. The Meeting concluded that the toxicity of R014821, observed in plants, would be covered by the parent compound, given its toxicity profile as well as its detection in rats at significant levels.

Following foliar spray applications to greenhouse-grown cucumbers, residues of R014821 were consistently low ≤ 0.01 mg/kg. While residues of R014821 were not analysed in the greenhouse tomato supervised residue trials, it did not account for more than 0.3% of the parent residues in the tomato metabolism study. However, the tomato metabolism study showed that the conjugated form of the parent compound accounted for up to 50% of the levels measured of the parent. Therefore, conjugated imazalil is likely to contribute to the dietary exposure of foliar-treated crops, yet it would not be relevant to post-harvest and seed treatment uses.

In the spring wheat seed treatment trials, all residues of imazalil were < 0.01 mg/kg in wheat grain. Although residues of R014821 were not measured in these trials, TRRs in grain from the metabolism study were 0.003 mg eq/kg. Therefore, it is reasonable to expect that levels of this metabolite will be significantly below < 0.01 mg/kg.

In the post-harvest treatment metabolism studies and the post-harvest treatment trials, the alcohol metabolite R014821 in citrus (whole fruit) and potatoes was not detected at the critical GAP PHI of 0-days. As residues of imazalil decreased with an increase in storage duration, residues of the metabolite were consistently below10% those of the parent compound and/or < 0.01 mg eq/kg. Therefore, R014821 is not likely to contribute to the total exposure of imazalil in the diet.

Noting the above, the Meeting decided the residue definition for dietary risk assessment for plant commodities should be free and conjugated imazalil.

The nature of the imazalil residues was investigated in lactating goat and laying hen following oral administration of the test substance. In the lactating goat metabolism study (180 ppm feed), imazalil was extensively metabolised. No imazalil was observed in milk whey and none of the identified metabolites accounted for greater than 10% TRR. In tissues, the parent represented less than 6% TRR. Several metabolites were observed in liver, however, none accounted for greater than 5% TRR. Two major metabolites, R061000 (kidney and muscle) and R043449 (muscle) were detected at higher concentrations (15–21% TRR and 15% TRR, respectively) than that of the parent (4% TRR). In fat, R061000 was not observed yet R043449 and R044179 accounted for 9% TRR (0.01 mg eq/kg) and 25% TRR (0.03 mg eq/kg), respectively.

In the dairy cattle feeding study at the lowest feeding level (equivalent to 1.4-fold the maximum estimated dietary burden), residues of imazalil were less than 0.02 mg/kg in milk, muscle and fat, while residues of imazalil were < 0.02–0.36 mg/kg in liver and kidney.

In the laying hen metabolism study (66 ppm feed), imazalil was also extensively metabolised and only detected in eggs and fat, representing 8% TRR (0.06 mg/kg) and 11% TRR (0.01 mg/kg), respectively. The metabolite R061000 was only observed in liver and eggs, accounting for 6–9% TRR (0.05–0.95 mg eq/kg) and the metabolite R043449 was only observed in fat and eggs at levels of 2–10% TRR (0.01 mg eq/kg).

In the poultry feeding study, residues of imazalil were below 0.02 mg/kg (LOQ) in tissues and below 0.01 mg/kg (LOQ) in eggs at all dose levels tested.

At the maximum estimated dietary burden, imazalil residues are only expected in ruminant matrices (liver and kidney). Therefore, the Meeting decided that the parent was a suitable marker for all animal matrices.

Suitable methods are available for imazalil in animal commodities.

The Meeting concluded that for enforcement of MRLs, imazalil is a suitable marker for animal matrices.

In deciding which compounds should be included in the residue definition for risk assessment, the Meeting considered the likely occurrence and the toxicological properties of the metabolites R061000, R043449 and R044179.

In the lactating goat metabolism study, R061000 and R043449 may contribute to the consumer exposure as they collectively account for a significant proportion of the TRRs in milk whey (12% TRR; 0.15 mg eq/kg), liver (6.2% TRR; 1.33 mg eq/kg), kidney (15% TRR, 1.45 mg eq/kg), muscle (36.4% TRR, 0.12 mg eq/kg) and fat (9% TRR, 0.01 mg eq/kg). Moreover, the levels of these metabolites in liver were similar to that of the parent. In kidney and muscle, the levels of metabolites were 5–13-fold higher than those of imazalil while in fat, the metabolite R043449 was approximately 2-fold higher than parent. The major metabolite R044179 detected in fat (25% TRR, 0.03 mg eq/kg) is also expected to contribute to the consumer exposure considering it represents up to5-fold the levels of the parent (6% TRR; 0.006 mg eq/kg).

In the dairy cattle feeding study, residues of imazalil, R061000 and R043449 were not quantifiable (< 0.02 mg/kg) in milk, muscle and fat at the lowest dose level tested (1.4-fold the maximum estimated dietary burden) while the residues of these metabolites were collectively similar to that of the parent in liver and 5-fold greater than that of the parent in kidney. Although residues of R044179 were not measured in the feeding study, considering the rate of exaggeration of the metabolism study, these are not anticipated to be present at measureable levels in fat, when cattle are fed a diet equivalent to the maximum estimated dietary burden.

In the poultry metabolism study, imazalil and R043449, present only in fat and eggs (0.01–0.06 mg eq/kg), are not expected to contribute significantly to the consumer exposure. Conversely, R061000 was the only major metabolite present at 9% TRR (0.95 mg eq/kg) in liver, which may contribute to the overall consumer exposure.

The Meeting concluded that the toxicity of R061000, observed in ruminants and poultry, would be covered by the parent compound, given its toxicity profile as well as its detection in rats at significant levels. For the metabolite R043449, as no specific data were available on the toxicity of the metabolite the TTC approach was applied[3]. The exposure of R043449 is below the TTC for a Cramer Class III compound (1.5 µg/kg bw) and is therefore unlikely to present a public health concern based on the uses considered by the Meeting.

Noting the above, the Meeting decided the residue definition for dietary risk assessment for animal commodities should be the sum of imazalil and R061000, expressed as imazalil equivalents

Definition of the residue for compliance with the MRL for plant commodities: *imazalil*

Definition of the residue for dietary risk assessment for plant commodities: *free and conjugated imazalil.*

Definition of the residue for compliance with the MRL for animal commodities: *imazalil*

Definition of the residue for dietary risk assessment for animal commodities: sum of imazalil and the metabolite R061000 ((RS)-3-[2-(2,4-dichlorophenyl)-2-(2,3- dihydroxypropoxy)ethyl]imidazolidine- 2,4-dione (+)-1-[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-2,5-imidazolidinedione), expressed as imazalil equivalents.

The Log K_{ow} of imazalil is 2.15, indicating a potential to sequester into fatty matrices. In lactating goats, the ratio of residues (sum of imazalil and R061000) in fat to muscle was 1-fold. The Meeting considered the residue not fat-soluble.

Results of supervised residue trials on crops

The Meeting noted that supervised residue trials were not provided for melons (except watermelons), Japanese persimmons, raspberries (red, black) and strawberries and that the use on pome fruits is no longer supported by the manufacturer. Therefore, the Meeting withdraws its previous recommendations of 2 mg/kg for melons (except watermelons), 2 mg/kg for Japanese persimmons, 5 mg/kg for pome fruits and 2 mg/kg each for raspberries (red, black) and strawberries.

Citrus fruits

The critical GAP for citrus fruits is in the USA, a combination of 2 post-harvest applications, dip or drench at 0.075 kg ai/hL followed by a wax application of 0.2 kg ai/hL, for a total application rate of 0.275 kg ai/hL. The minimum post-treatment interval is 0 days.

Lemons

A total of nine independent supervised residue trials were conducted in Greece, Italy and Spain approximating critical GAP in the USA, where residues of imazalil in whole lemons were, in ranked order (n = 9): 3.0, 3.1, 3.3, 4.1, 5.0, 5.7, 6.4, 7.8 and 9.7 mg/kg.

Mandarins

Only two trials were conducted on mandarins in accordance to the USA critical GAP, therefore, there were an insufficient number of trials to allow the Meeting to estimate a maximum residue level.

Oranges

A total of 12 independent supervised residue trials were conducted in Greece, Italy and Spain, approximating the critical GAP in the USA, where residues of imazalil found in whole oranges were, in ranked order: 1.8, 2.0, 2.2, 2.5, 2.6, 2.7, 3.0, 3.4, 3.6, 4.2, 4.4 and 4.8 mg/kg.

[3] See Toxicology section of the 2018 JMPR Report for further details

Grapefruits

No trials conducted on grapefruit reflected the USA critical GAP; therefore, the Meeting could not estimate a maximum residue level.

Summary – Citrus fruits

While the USA label is approved for use on the entire citrus fruit crop group, the Meeting only received adequate trials for oranges and lemons, Therefore, the Meeting recommended maximum residue levels 15 mg/kg for the subgroup of Lemons and limes and 8 mg/kg for the Subgroup of Oranges, Sweet, Sour, both of which were based on the mean + 4SD. The Meeting withdraws its previous recommendation of 5 mg/kg for citrus fruits.

For dietary risk assessment, the residues of imazalil in lemon pulp were, in ranked order: 0.05 (2), 0.11, 0.12, 0.18, 0.20, 0.23, 0.27 and 0.36 mg/kg. The Meeting estimated a HR and STMR of 0.36 mg/kg and 0.18 mg/kg, respectively.

For dietary risk assessment, the residues of imazalil in orange pulp, analysed in 16 of the 18 trials, were in ranked order: 0.05 (2), 0.06 (2), 0.07 (2), 0.11 (2), 0.12, 0.13, 0.18, 0.20, 0.21, 0.23, 0.26 and 0.27 mg/kg. The Meeting estimated a HR and STMR of 0.27 mg/kg and 0.12 mg/kg, respectively.

Bananas

The critical GAP for post-harvest treatment of bananas is in Honduras, with a dip application at 0.11 kg ai/hL and 0-day post-treatment interval. However, the Meeting could not recommend a maximum residue level as none of the trials reflected this critical GAP.

In Columbia, the critical GAP for post-harvest treatment of bananas is a dip application at 0.06 kg ai/hL and a 0-day post-treatment interval. A total of 2 independent trials were conducted in Spain in accordance with the critical GAP, where residues of imazalil in whole fruit were, in ranked order: 2.94 and 3.34 mg/kg.

As there were an insufficient number of trials to estimate a maximum residue level on bananas, the Meeting considered the residue decline trials conducted in European countries, where little dissipation of imazalil residues, as a function of time post-harvest, was observed. Therefore, the Meeting decided to combine the residue data from the thirteen independent trials conducted in Honduras and Panama with those conducted in Spain, at rates approximating the critical GAP of Columbia, and at longer post-treatment intervals. In ranked order, the combined residues of imazalil in whole banana fruit were: 0.74, 0.92, 0.95, 1.22, 1.35, 1.44, 1.79, 2.03, 2.14 2.32, 2.34, 2.68, 2.91, 2.94 and 3.34 mg/kg.

The Meeting estimated a maximum residue level of 6 mg/kg (based on mean + 4SD).

For dietary risk assessment, the residues of imazalil in banana pulp were, in ranked order: < 0.05, 0.07 (3), 0.08, 0.09, 0.11, 0.16 (2), 0.20, 0.24, 0.57 and 0.72 mg/kg. The Meeting estimated a HR and STMR of 0.72 mg/kg and 0.11 mg/kg, respectively.

The IESTI represented greater than 100% of the ARfD of 0.05 mg/kg bw in the case of bananas (120% children).

An alternative GAP exists in France for the dip treatment of bananas at 0.0375 kg ai/hL and a 0-day post-treatment interval. Thirteen post-harvest dip treatment trials were conducted at 0.03 kg ai/hL in Honduras and Panama. Considering the limited decline in imazalil residues as a function of storage duration, residues from the 14 and 22-day post-treatment intervals were used in calculating the maximum residue level.

The residues in whole fruit, in ranked order, were: 0.22, 0.31, 0.39, 0.58, 0.61, 0.71, 1.01, 1.12, 1.30, 1.34, 1.54, 1.68 and 1.69 mg/kg. The Meeting estimated a maximum residue level of 3 mg/kg (based on CF × 3 mean). The Meeting replaces its previous recommendation of 2 mg/kg for bananas.

For dietary risk assessment, the residues of imazalil in banana pulp (n = 11 as two trials did not analyse residues in pulp) were, in ranked order: < 0.05 (8), 0.06, 0.07 and 0.10 mg/kg. The Meeting estimated a HR and STMR of 0.10 mg/kg and 0.05 mg/kg, respectively.

Cucumber

In Belgium, the critical GAP for imazalil on greenhouse cucumbers is three foliar spray applications at 0.005 kg ai/hL, 7 day re-treatment interval, and a PHI of 1 day.

Two independent trials were conducted in European countries, where three foliar applications were made at 0.0075 kg ai/hL/application (higher than the critical GAP).

The Meeting agreed to utilise the proportionality approach to estimate residues matching critical GAP. Unscaled imazalil residues in cucumbers were 0.01 and 0.02 mg/kg. Using a scaling factor of 1.5, scaled residues were 0.007 and 0.013 mg/kg.

The Meeting concluded there were insufficient trials to estimate a maximum residue level. The meeting recommends withdrawing its previous recommendations of 0.5 mg/kg for cucumbers and gherkins.

Tomato

In Belgium, the critical GAP for imazalil on greenhouse tomatoes is three foliar spray applications at 0.02 kg ai/hL, a 7 day re-treatment interval, and a PHI of 1 day.

Six independent supervised residue trials were conducted in accordance with the critical GAP where imazalil residues, in ranked order, were 0.04 (2), 0.08, 0.09, 0.15 and 0.16 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg. For dietary risk assessment, the total residues of free and conjugated imazalil (using an adjustment factor of 1.5 to account for conjugated imazalil) were, in ranked order: 0.06 (2), 0.12, 0.14, 0.22 and 0.24 mg/kg. The Meeting estimated a HR of 0.24 mg/kg and a STMR of 0.13 mg/kg.

Potato- Post-harvest treatment

In Europe, the critical GAP for imazalil as a post-harvest treatment is 0.015 kg ai/tonne with a minimum post-treatment interval of 0-day.

Eight independent supervised residue trials were conducted in Germany and the UK on stored potatoes receiving a single post-harvest application, in accordance with the critical GAP. The residues of imazalil in potatoes, in ranked order, were 0.46, 0.88, 1.4, 1.8, 2.6, 4.1, 4.5 and 4.6 mg/kg. The Meeting estimated a maximum residue level of 9 mg/kg (using CF × 3 mean), a HR of 4.6 mg/kg and a STMR of 2.2 mg/kg. The Meeting replaces its previous recommendation of 5 mg/kg.

Cereal grains

The critical GAP for the seed treatment of wheat and barley is in the USA at 0.1 kg ai/tonne.

No trials were provided matching the critical GAP for wheat.

Five independent supervised seed treatment trials on barley were carried out in various European countries in 2005–2006 in accordance with the critical GAP. All residues of imazalil in barley grain samples were < 0.01 mg/kg.

The supervised seed treatment trials on wheat, carried out in Germany and Poland in 2005-2006, were not conducted in accordance with the critical GAP. However, the Meeting noted that, in the spring wheat metabolism study conducted at 5-fold the critical GAP, residues of imazalil in grain were < 0.01 mg/kg.

Considering the residue profile in barley and wheat are the same following seed treatment use, the datasets can be combined for mutual support.

The Meeting estimated a maximum residue level of 0.01 (*) mg/kg and a STMR of 0 mg/kg for barley, and triticale and maintains its previous recommendation of 0.01 (*) mg/kg for wheat.

Animal feed items

The critical GAP for the seed treatment of wheat is in the USA at 0.1 kg ai/tonne.

Straw, Fodder and Forage of Cereal Grains

No trials matching the critical GAP for wheat were provided to the Meeting.

When comparing the barley trials with the spring wheat metabolism study, the Meeting considered the residue profile in barley and wheat forage and straw to be the same following seed treatment use.

Five independent supervised seed treatment trials on barley were carried out in various European countries in 2005–2006 in accordance with the USA critical GAP. The residues of imazalil, in all barley forage/whole plant and straw samples were < 0.01 mg/kg.

Noting the above, the Meeting estimated a highest residue of 0.01 mg/kg and median residue of 0.01 mg/kg for each barley forage (whole plant) and wheat forage (whole plant).

For barley straw and fodder (dry) and triticale straw and fodder (dry), the Meeting estimated maximum residue levels of 0.01 mg/kg, highest residues of 0.01 mg/kg and median residues of 0.01 mg/kg for each commodity. The Meeting recommends a maximum residue level, highest residue and median residue of 0.01, 0.01 and 0.01 mg/kg, respectively, for wheat straw and fodder (dry), to replace its previous recommendation of 0.1 mg/kg.

Fate of residues during processing

High temperature hydrolysis

Imazalil was shown to be hydrolytically stable for all hydrolytic conditions tested in this study: at pH 4 and 90 °C simulating pasteurisation, at pH 5 and 100 °C simulating baking/brewing/boiling and at pH 6 and 120 °C simulating the process of sterilisation. There was no evidence of any hydrolysis or reaction products formed during incubation.

Processing

The Meeting received information on the fate of imazalil residues during the processing of citrus fruits and potatoes. Processing factors calculated for imazalil for the processed commodities of the citrus fruits and potatoes are shown in the tables below. Processing factors, best estimates, HR-Ps and STMR-Ps were calculated.

Citrus

As the residue concentrations of imazalil in all orange and lemon processed commodities are not higher than the estimated maximum residue level for the oranges subgroup, separate maximum residue levels will not be estimated for any of the orange processed commodities.

The Meeting could not recommend maximum residue levels for grapefruit in the absence of sufficient trials, therefore, HR-Ps and STMR-Ps for grapefruit processed commodities could not be determined.

Commodity	Calculated Processing Factors	Best Estimate	RAC STMR, mg/kg	STMR-P, mg/kg	RAC HR, mg/kg	HR-P, mg/kg
Oranges						
Juice	0.01, < 0.02, 0.03, 0.05, 0.10, 0.11, 0.14, 0.33, 0.35	0.10 (median)	0.09	0.01	0.26	0.03
Chopped fresh peel	0.29, 0.48	0.39 (mean)		0.04		0.10
Cold pressed oil	23.8, 33.4	28.6 (mean)		2.6		7.4
Marmalade	0.15, 0.25, 0.25, 0.27, 0.28, 0.56, 0.68	0.27 (median)		0.02		0.07
Jam	0.03, 0.04	0.04 (mean)		0.004		0.01
Jelly	0.02, 0.03	0.03 (mean)		0.003		0.008
Canned orange	< 0.02, 0.04	0.03 (mean)		0.003		0.008
Dry pomace (dried pulp)	4.0, 4.0, 4.4, 4.5, 4.9, 6.7, 9.6	4.5 (median)		0.40		1.2
Lemons						
Juice	0.05,0.04	0.05 (mean)	0.18	0.01	0.36	0.02
Chopped fresh peel	0.38, 0.34	0.36 (mean)		0.06		0.13
Cold pressed oil	4.3, 2.6	3.5 (mean)		0.6		1.3
Dried peel	1.0, 1.0	1.0 (mean)		0.2		0.36

Potato

As the residue concentrations of imazalil in all potato processed commodities are not higher than the estimated maximum residue level for potato, separate maximum residue levels will not be estimated for any of the potato processed commodities.

Commodity	Calculated Processing Factors	Best Estimate	RAC STMR, mg/kg	STMR-P, mg/kg	RAC HR, mg/kg	HR-P, mg/kg
Baked, with peel (without foil)	0.40, 0.80	0.60 (mean)	2.2	1.3	4.6	2.8
Baked, with peel (with aluminium foil)	0.37, 0.60	0.49 (mean)		1.1		2.3
Boiled, washed with peel	0.28, 0.50	0.39 (mean)		0.86		1.8
Microwaved, with peel	1.1,1.6	1.4 (mean)		3.1		6.5
Peel	2.7, 3.2	3.0 (mean)		6.6		13.8
Peeled	0.003, 0.01	0.01 (mean)		0.02		0.05
Steamed, washed and	0.01, 0.05	0.03 (mean)		0.07		0.14

Commodity	Calculated Processing Factors	Best Estimate	RAC STMR, mg/kg	STMR-P, mg/kg	RAC HR, mg/kg	HR-P, mg/kg
peeled						
Fries	0.01, 0.02	0.02 (mean)		0.04		0.09
Crisps	0.02, 0.02	0.02 (mean)		0.04		0.09
Canned (after sterilization)	0.002, 0.01	0.01 (mean)		0.02		0.05
Flakes	< 0.002, < 0.002	< 0.002 (mean)		0.004		0.009

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels arising in tissues and milk when three groups of dairy cows were fed with a diet containing 46.4, 148.6 and 440.5 ppm feed/day for 28 consecutive days. As the estimated dietary burdens for beef and dairy cattle are lower than the lowest feeding level tested, residues of imazalil and its metabolites will only be reported for the low dose feeding level.

Residues of imazalil, R06100 and R043449 in milk were each <LOQ (0.02 mg/kg).

In muscle, residues of imazalil, R06100 and R043449 were each <LOQ (0.02 mg/kg).

In fat, residues of imazalil, R06100 and R043449 were each <LOQ (0.02 mg/kg).

In liver, quantifiable residues of imazalil, R06100 and R043449 were observed at the low dose feeding level, where mean (maximum) residues were 0.326 (0.362), 0.153 (0.224) and 0.153 (0.213) mg/kg, respectively.

Similarly, in kidney, lower yet quantifiable residues of imazalil, R06100 and R043449 were observed at the low dose feeding level, where mean (maximum) residues were 0.020 (0.033), 0.069 (0.078) and 0.022 (0.026) mg/kg, respectively.

The Meeting also received information on the residue levels arising in tissues and eggs when groups of laying hens were fed with a diet containing imazalil at rates of 0.225, 0.728 and 2.56 ppm feed/day for 28 consecutive days. Residues of imazalil, R042639 and R044085 were each <LOQ (0.02 mg/kg/analyte) in tissue samples collected at all dose levels. Similarly, residues of imazalil, R042639, R044085 and R110470 in eggs were each <LOQ (0.01 mg/kg/analyte) at all dosing levels.

Estimated dietary burdens of farm animals

Maximum and mean dietary burden calculations for imazalil are based on the feed items evaluated for cattle and poultry as presented in Annex 6. The calculations were made according to the livestock diets from Australia, the EU, Japan and USA-Canada in the OECD feeding tables listed in Appendix IX of the 2016 edition of the FAO Manual.

The post-treatment application of imazalil to oranges and potatoes and seed treatment use on wheat and barley resulted in residues in the following feed items: dried pulp, potato culls, potato peel (waste), wheat and barley straw and grain. Based on the named feed items, the calculated maximum animal dietary burden for dairy or beef cattle was in EU, followed by US/Canada and Australia. For poultry broiler or layer, the calculated maximum dietary burden was in the EU.

	Livestock dietary burden, imazalil							
	US/Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	23.4	19.8	28.9[a]	25.3[b]	5.08	3.88	-	23.4
Dairy Cattle	7.81	6.61	23.4[c]	19.8[d]	2.46	1.26	-	7.81
Poultry, broiler	-	-	2.3	1.1	-	-	-	-
Poultry, layer	-	-	2.3[e]	1.1[f]	-	-	-	-

[a] Suitable for maximum residue level estimate for meat, fat and edible offal of mammals

[b] Suitable for STMR estimate for meat, fat and edible offal of mammals

[c] Suitable for maximum residue level estimate for milk

[d] Suitable for STMR estimate for milk

[e] Suitable for maximum residue level estimate for eggs, meat, fat and edible offal

[f] Suitable for STMR estimate for eggs, meat, fat and edible offal

Animal commodities residue level estimation

Anticipated residues for maximum residue level recommendation resulting from the dietary burdens and based on the feeding studies are summarised below:

	Feed level for milk residues (ppm)	Imazalil Residues in milk (mg/kg)	Feed level for tissue residues (ppm)	Imazalil Residues (mg/kg)			
				Muscle	Liver	Kidney	Fat
maximum residue level Estimation - Beef or Dairy Cattle							
Feeding level	46.4	< 0.02	46.4	< 0.02	0.36	0.03	< 0.02
Dietary burden and anticipated residues	23.4	< 0.02	28.9	< 0.02	0.22	0.02	< 0.02

Anticipated residues for dietary exposure assessment resulting from the dietary burdens and based on the feeding studies are summarised below:

	Feed level for milk residues (ppm)	Total imazalil residues [a] in milk (mg/kg)	Feed level for tissue residues (ppm)	Total imazalil residues[1] (mg/kg)			
				Muscle	Liver	Kidney	Fat
HR Estimation – Beef Cattle							
Feeding level	-	-	46.4	< 0.04	0.80	0.14	< 0.04
Dietary burden and anticipated residues	-	-	28.9	< 0.04	0.50	0.09	< 0.04
STMR Estimation – Beef or Dairy Cattle							
Feeding level	46.4	< 0.04	46.4	< 0.04	0.63	0.11	< 0.04
Dietary burden and anticipated residues	19.8	< 0.04	25.3	< 0.04	0.34	0.06	< 0.04

[a] Total residues of imazalil and R061000, expressed as parent equivalents.

The Meeting estimated a maximum residue level and STMR of 0.02(*) mg/kg and 0 mg/kg, respectively for milks. For meat (from mammals other than marine mammals), the Meeting estimated a maximum residue level, HR and STMR of 0.02(*) mg/kg, 0.04 mg/kg and 0.04 mg/kg and for mammalian fat (except milk fats), the Meeting estimated a maximum residue level, HR and STMR of 0.02 mg/kg, 0.04 mg/kg and 0.04 mg/kg. For edible offal (mammalian), a maximum residue level, HR and STMR of 0.3 mg/kg, 0.50 mg/kg and 0.34 mg/kg, respectively, based on liver residue are recommended. For kidney, the HR and STMR are 0.09 and 0.06 mg/kg, respectively.

In the poultry feeding study, residues of imazalil from all feeding levels were < 0.01 mg/kg in eggs and < 0.02 mg/kg in fat, muscle and liver. Therefore, the meeting estimated a maximum residue level of 0.01(*) mg/kg for eggs and 0.02(*) mg/kg for each poultry meats, fats and edible offal of poultry. Based on an estimated dietary burden for poultry of 1.3 ppm, the Meeting estimated HRs and STMRs of 0.02 and 0.02 mg/kg for eggs and 0.04 mg/kg and 0.04 mg/kg for poultry meats, fats and edible offal of poultry.

RECOMMENDATIONS

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

Definition of the residue for compliance with the MRL for plant commodities: *imazalil.*

Definition of the residue for dietary risk assessment for plant commodities: *free and conjugated imazalil.*

Definition of the residue for compliance with the MRL for animal commodities: *imazalil*.

Definition of the residue for dietary risk assessment for animal commodities: sum of imazalil and the metabolite R061000 ((RS)-3-[2-(2,4-dichlorophenyl)-2-(2,3- dihydroxypropoxy)ethyl]imidazolidine- 2,4-dione (±)-1-[2-(2,4-dichlorophenyl)-2-[(2,3-dihydroxypropyl)oxy]ethyl]-2,5-imidazolidinedione), expressed as imazalil equivalents.

The residue is not fat-soluble.

CCN	Commodity Name	Recommended maximum residue level, mg/kg		STMR, STMR-P or median, mg/kg	HR, HR-P or highest, mg/kg
		New	Previous		
FO0327	Banana	3 Po	2 Po	0.05	0.10
GC 0640	Barley	0.01*	-	0	-
AS 0640	Barley straw and fodder (dry)	0.01	-	0.01	0.01
FC0001	Citrus Fruit	W	5 Po		
VC 0424	Cucumber	W	0.5		
MO 0096	Edible offal (mammalian)	0.3	-	0.34 (liver) 0.06 (kidney)	0.50 (liver) 0.09 (kidney)
PE 0112	Eggs	0.01*	-	0.02	0.02
VC0425	Gherkins	W	0.5		
FC0002	Lemons and limes, Subgroup of (includes all commodities in this subgroup)	15 Po	-	0.18	0.36
MF 0100	Mammalian fats (except milk fats)	0.02	-	0.04	0.04
MM 0095	Meat (from mammals other than marine mammals)	0.02*	-	0.04	0.04
VC 0046	Melons, except Watermelon	W	2 Po		
ML 0106	Milks	0.02*	-	0	-
FC0004	Oranges, sweet, sour, Subgroup of (includes all commodities in this subgroup)	8 Po	-	0.09	0.26
FT 0307	Persimmon, Japanese	W	2 Po		
FP 0009	Pome fruits	W	5 Po		
VR0589	Potato	9 Po	5 Po	2.2	4.6
PO 0111	Poultry, edible offal of	0.02*	-	0.04	0.04
PF 0111	Poultry fats	0.02*	-	0.04	0.04
PM 0110	Poultry meat	0.02*	-	0.04	0.04
FB 0272	Raspberries, red and black	W	2		
FB 0275	Strawberry	W	2		
VO0448	Tomato	0.3	-	0.13	0.24
GC 0653	Triticale	0.01*	-	0	-
AS 0653	Triticale straw and fodder (dry)	0.01	-	0.01	0.01
AS 0654	Wheat straw and fodder (dry)	0.01	0.1	0.01	0.01
JF 0004	Orange juice			0.01	0.03
	Orange peel			0.04	0.10
	Orange oil			2.6	7.4
	Marmalade			0.02	0.07
	Jam			0.004	0.01
	Canned orange			0.003	0.008
	Potato peel			6.6	13.8
	Peeled potato			0.02	0.05
	Baked potato with peel			1.3	2.8
	Boiled potato with peel			0.86	1.8
	Potato fries			0.04	0.09
	Potato crisps			0.04	0.09
	Potato flakes			0.004	0.009
	Barley forage (whole plant)			0.01	0.01
AF 0654	Wheat forage (whole plant)			0.01	0.01

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The ADI for imazalil is 0–0.03 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for imazalil were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the JMPR. The results are shown in Annex 3 of the 2018 JMPR Report. The IEDIs ranged from 2–40% of the maximum ADI.

The Meeting concluded that long-term dietary exposure to residues of imazalil from uses considered by the JMPR is unlikely to present a public health concern.

Acute dietary exposure

The ARfD for imazalil is 0.05 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for imazalil were calculated for the food commodities and their processed commodities for which HRs/HR-Ps or STMRs/STMR-Ps were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2018 JMPR Report. The IESTIs varied from 0–40% of the ARfD for children and 0–90% for the general population.

The Meeting concluded that acute dietary exposure to residues of imazalil from uses considered by the present Meeting is unlikely to present a public health concern.

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Reference Number	Author(s)	Year	Study Title
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