IMIDACLOPRID (206)

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EXPLANATION

Imidacloprid is a systemic insecticide which has been used widely in many crops for years. It was first evaluated by JMPR in 2001 (T) and 2002 (R). An ADI of 0-0.06 mg/kg bw and an ARfD of 0.4 mg/kg bw was established. The compound was evaluated for residues in 2006, 2008 and 2012. In 2002 the Meeting agreed that the residue definition for compliance with MRLs and for estimation of dietary intake for plant and animal commodities should be the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid. It was listed by the 46th Session of CCPR (2014) for the evaluation of 2015 JMPR for additional MRLs.

The residue studies were submitted by the manufacturer and member countries for additional MRLs for stone fruit, olive, curly kale, soya bean, tea, goji (China) and basil (Thailand).

RESIDUE ANALYSIS

Analytical methods

Samples of cherries, plum and peach were fortified with an equimolar solution of imidacloprid, desnitro imidacloprid (WAK4140, M09), olefin imidacloprid (WAK3745, M06), 5-hydroxyl imidacloprid (WAK4103, M01) and 6-chloronicotinic acid (6-CNA, M14), and were analysed for combined residues of those compounds by GC-MS using a modification of the Bayer Method 00200-reformated, Report No 102624-R1 dated 02/23/94 (see JMPR 2002, 2006 and 2012). At the LOQ of 0.05 mg/kg (expressed as imidacloprid), the recoveries were 98±12% for cherries, 92, 104, 115% for plum and 93±18% for peach.

The Meeting received information on the analytical method (Method 00834) for the determination of imidacloprid residues as well as the total residue of imidacloprid (including parent and all metabolites containing the 6-chloropyridinyl moiety) in plant materials (Schöning, 2003: MR-122/03).

Imidacloprid and related metabolites are extracted with a mixture of methanol/water (3/1, v/v) in the presence of diluted sulphuric acid (10%). Oil samples are dissolved in n-hexane and the residues are extracted twice with water. For the determination of the imidacloprid, an aliquot of the extract is partitioned against cyclohexane/ethyl acetate (1/1, v/v) using a Chromabond XTR column (diatomaceous earth). The organic solution is redissolved in acetonitrile/water (2/8, v/v + 2 mL/L formic acid). Quantitation is performed by reversed phase HPLC-MS/MS. For determination of the total residue of imidacloprid, a corresponding aliquot of the extract is evaporated to the aqueous remainder and dissolved in water. Imidacloprid and all metabolites containing the 6-chloropicolyl moiety are oxidised with alkaline $KMnO_4$ to yield 6-CNA. Following acidification and subsequent neutralisation of the excess oxidant, the 6-CNA is extracted from the aqueous solution using *tert*-butylmethylether (MTBE). The ether phase is dried, the solvent is evaporated and the remainder dissolved in acetonitrile/water (2/8, v/v + 2 mL/L formic acid). These solutions are subjected to analysis by HPLC-MS/MS.

The recoveries for imidacloprid ranged from 83 to 112% at fortification levels of 0.01 and 1.0 mg/kg. The mean recoveries for the parent compound were between 89 and 110% with relative standard deviations (RSD) up to 11.6%. The recoveries for the total residue of imidacloprid fortified as parent compound ranged from 75 to 102% at fortification levels of 0.05 to 2.0 mg/kg. The mean recoveries for the parent compound were between 77 and 93% with RSD values up to 6.1%. The recoveries for the total residue of imidacloprid fortified as a mixture of 6-CNA and desnitro-imidacloprid (1:1, w/w) ranged from 64 to 108% at fortification levels of 0.0567 to 1.134 mg/kg parent equivalents. The mean recoveries for the metabolite mixture were between 73 and 97% with relative standard deviations up to 16%. The LOQ is 0.01 mg/kg for imidacloprid and 0.05 mg/kg for total residue of imidacloprid, expressed as imidacloprid.

The method as modified in 00834/M001 (Schöning, 2004: MR-153/03) contains no changes in the analytical procedure compared to the original method 00834 but it incorporates an internal standard procedure to the method. Method 00834/M001 was validated for the determination of residues of imidacloprid parent compound as well as the total residue of imidacloprid (including parent and all metabolites containing the 6-chloropyridinyl moiety) in plant materials. The recoveries for imidacloprid ranged from 80 to 104% at fortification levels of 0.01 and 2.0 mg/kg (mean recoveries: 88 to 99%, RSDs: 1.5 to 7.4%). The recoveries for the total residue of imidacloprid ranged from 66 to 106% at fortification levels of 0.05 (0.0567 mg/kg as mixture of 6-CNA and desnitro metabolite (1:1, w/w) calculated as imidacloprid) to 2.0 mg/kg (mean recoveries: 75 to 101%, RSDs: 1.1 to 9.8%).

The analytical method 00834/M002 (Schöning, 2010: MR-09/169) was developed for the determination of residues of imidacloprid, 5-hydroxyl imidacloprid (WAK4103, M01) and olefin imidacloprid (WAK3745, M06) in plant materials. Imidacloprid and its metabolites are extracted from tomato (fruit), bean (bean with pod), orange (fruit), rape (seed), cereals (grain) and tobacco (green leaf and dried leaf) with methanol/water (3/1, v/v) using a blender. After filtration an aliquot of the extract was evaporated to the aqueous remainder and further the stable isotopically labelled analytes are added for tomato (fruit), bean (bean with pod), orange (fruit), rape (seed), cereals (grain) and tobacco (green leaf). Parts of the solutions are transferred into an HPLC vial and subjected to reversed phase HPLC-MS/MS in the positive ion mode without further clean-up. Recoveries were determined at fortification levels of 0.01 mg/kg (LOQ level, 0.05 mg/kg for tobacco), and 0.10 mg/kg (0.5 mg/kg for tobacco) (each compound expressed as parent equivalent). Mean recoveries for each fortification level ranged from 70 to 107% with RSD up to 12% for all matrices.

The supplemental method 00300/E007 (Schöning, 2010: MR-158/00) has no changes in the analytical procedure compared to the original method 00300. The method was validated for additional matrices of olive fruit, grape pomace and cacao bean. For imidacloprid, recoveries were determined by spiking control samples with imidacloprid at fortification levels of 0.01 and 0.20 mg/kg. The recoveries were in the range from 68 to 110%, the mean recoveries for each matrix ranged from 74 to 90% with a mean RSD ranging from 3.3 to 17.3%. For the total residue of imidacloprid, recoveries were determined by spiking control samples with imidacloprid (fortification levels of 0.05 and 0.5 mg/kg) or with a mixture of 6-CNA and desnitro imidacloprid (0.02 mg/kg each corresponding to 0.0567 mg/kg calculated as imidacloprid). The recoveries were in the range from 64 to 98%, the mean recoveries for each matrix ranged from 70 to 96% with a mean RSD ranging from 2.2 to 12%.

The results for olive are summarized in Tables 1 and 2.

Table 1 Recovery results obtained for the determination of imidacloprid from olive and its processed commodities

Commodity	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference Method
Olive, fruit	0.01 1.0	5 5	83 – 96 106 – 111	92 108	5.6 1.7	MR-122/03 00834 (m/z 258→175)
Olive, oil	0.01 1.0	5 5	97 – 98 96 – 100	98 99	0.5 1.8	
Olive, pomace	0.01 1.0	5 5	94 – 100 109 – 112	97 110	2.5 1.1	
Olive, fruit	0.01 2.0	5 5	80 – 96 96 – 99	88 97	7.4 1.5	MR-153/03 00834/M001
Olive, fruit	0.01 0.20	3 3	78 – 104 87 – 93	87 90	17 3.3	MR-158/00 00300/E007

Table 2 Recovery results obtained for the determination of total residue of imidacloprid from olive and its processed commodities

Commodity	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference Method
Olive, fruit	0.05	5	80 – 86	83	2.9	MR-122/03
	2.0	5	78 – 80	79	1.1	00834
	0.0567*	5	75 – 91	82	7.2	(m/z 158→122
	1.134*	5	70 – 79	74	5.8	
Olive, oil	0.05	5	83 – 85	84	1.2	
	2.0	5	78 – 92	84	6.1	
	0.0567*	5	77 – 83	80	3.4	
	1.134*	5	79 – 84	82	2.5	
Olive, pomace	0.05	5	88 – 95	91	3.3	
	2.0	5	77 – 82	80	2.6	
	0.0567*	5	85 – 95	92	4.6	
	1.134*	5	86 – 94	90	3.4	
Olive, fruit	0.05	5	81 – 89	85	3.9	MR-153/03
	2.0	5	76 – 95	88	9.8	00834/M001
	0.0567*	5	80 – 97	91	7.3	
Olive, fruit	0.05	3	64 – 79	73	11	MR-158/00
	0.50	3	73 – 82	78	5.9	00300/E007
	0.0567*	3	68 – 72	70	2.9	

* Mixture of 6-CNA (0.02 mg/kg) and desnitro metabolite (0.02 mg/kg), (1/1, w/w) calculated as imidacloprid

The Meeting has received information on the analytical method (NY/T 1275-2007) for the detection, quantitative analysis and confirmation of imidacloprid residues in fresh and dried goji berries (Niu, 2014: IG-01).

Imidacloprid is extracted from goji samples by homogenizing with acetonitrile. After adding sodium chloride, the sample is shaked and centrifuged. An aliquot is concentrated, and purified by solid phase extraction using amino cartridges. Imidacloprid residues were analysed by reversed-phase HPLC-UV (275 nm). The method was validated in fresh or dried goji samples. Control samples were spiked with a standard solution of imidacloprid at fortified level of 0.02, 0.05 and 0.1 mg/kg, and recoveries of imidacloprid with this method ranged from 69–87% (mean: 72–84% with RSD of 2.6–3.5%) in fresh goji samples, while recoveries ranged from 76–100% (mean: 79–100% with RSD of 0–11%) in dried goji samples. The LOQ is 0.02 mg/kg for both matrices.

Table 3 Recovery results obtained for the determination of imidacloprid residue from goji berries

Commodity	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference Method
Goji, fresh	0.02	5	70 – 76	72	2.6	IG-01
	0.05	5	69 – 78	73	4.1	Yan Niu, 2014
	0.10	5	78 – 87	84	3.5	
Goji, dried	0.02	5	100	100	0.0	
	0.05	5	70 – 100	85	11	
	0.10	5	76 – 82	79	3.0	

The Meeting has also received information on the analytical method (NT-001-P04-01) used for the determination of residues of imidacloprid in soya bean matrices (seed, forage, hay, meal, hull, refined oil, defatted flour and aspirated grain fractions) (Gould *et al*., 2005: 201591).

This analytical method is based on earlier methods 00200 and 00834 and is designed to make use of the equipment and techniques available at the analytical laboratory. The total residue of imidacloprid is analysed by a common moiety method and quantified by using isotopically-labelled internal standards and HPLC-MS/MS. The method is validated by measuring the concurrent recoveries of each analyte (imidacloprid, desnitro imidacloprid, 5-hydroxy imidacloprid, olefin imidacloprid, and 6-CNA) individually in separate control samples of soya bean seed, forage, and

hay, as well as processed commodities of meal, hull, refined oil, defatted flour and aspirated grain fractions. Additionally, the method is further validated by measuring the concurrent recoveries of an imidacloprid/desnitro mixture (1:1, w/w) in these same matrices at various fortification levels. The validation was performed concurrently during the studies RANTY002 and RANTY003-1 (see Table 4).

Table 4 Recovery results obtained for the determination of imidacloprid from soya bean matrices

Analyte	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference
Seed						RANTY002 Mackie, 2006
Imaidacloprid	0.050	1	72			
Desnitro imidacloprid	0.050	1	59			
5-hydroxy imidacloprid	0.050	1	67			
Olefin imidacloprid	0.050	1	58			
6-CNA	0.050	1	79			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.050 0.10 2.0	5 5 3	75 – 93 67 – 99 69 – 79	85 82 74	9.3 15 6.8	
Forage						
Imaidacloprid	0.025	2	69, 92	81		
Desnitro imidacloprid	0.025	2	72, 75	74		
5-hydroxy imidacloprid	0.025	2	83, 87	85		
Olefin imidacloprid	0.025	2	83, 95	89		
6-CNA	0.025	2	66, 81	74		
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.025 2.0 7.5 10	4 10 3 3	60 – 88 74 – 90 81 – 86 74 – 78	75 81 84 77	16 7.2 3.0 3.0	
Hay						
Imaidacloprid	0.010	2	80, 85	83		
Desnitro imidacloprid	0.010	2	89, 92	91		
5-hydroxy imidacloprid	0.010	2	77, 99	88		
Olefin imidacloprid	0.010	2	74, 85	79		
6-CNA	0.010	2	78, 94	86		
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.010 2.0 30	4 8 3	57 – 85 72 – 95 87 – 90	75 80 88	17 9.5 1.7	
Seed						RANTY003 Krolski, 2006
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	3 3	73 – 87 79 – 88	81 84	8.9 5.4	
Meal						
Imaidacloprid	0.20	1	72			
Desnitro imidacloprid	0.20	1	76			
5-hydroxy imidacloprid	0.20	1	79			
Olefin imidacloprid	0.20	1	79			
6-CNA	0.20	1	84			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	4 6	64 – 74 74 – 84	71 80	6.7 4.6	
Hull						

Analyte	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD	Reference
Imaidacloprid	0.20	1	91			
Desnitro imidacloprid	0.20	1	92			
5-hydroxy imidacloprid	0.20	1	92			
Olefin imidacloprid	0.20	1	94			
6-CNA	0.20	1	78			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	4 6	69 – 78 70 – 84	75 77	5.7 6.5	
Oil						
Imaidacloprid	0.20	1	87			
Desnitro imidacloprid	0.20	1	72			
5-hydroxy imidacloprid	0.20	1	84			
Olefin imidacloprid	0.20	1	82			
6-CNA	0.20	1	92			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.10 2.0	3 2	82 – 93 71, 73	87 72	6.6	
Flour						
Imaidacloprid	0.20	1	86			
Desnitro imidacloprid	0.20	1	88			
5-hydroxy imidacloprid	0.20	1	87			
Olefin imidacloprid	0.20	1	84			
6-CNA	0.20	1	79			
Imidacloprid/desnitro imidacloprid mixture (1:1)*	0.20 2.0	4 6	64 – 76 78 – 84	71 81	8.1 2.7	
AGF						
Imidacloprid/desnitro imidacloprid mixture (1:1)*	30 150	2 2	72, 79 60, 74	75 67		

* The fortification level given is the total mg/kg of both analytes in the mixture.

The analytical method 01389 was developed for the determination of residues of imidacloprid, its 2 metabolites 5-hydroxy imidacloprid and olefin imidacloprid, and of the total residue of imidacloprid determined as 6-CNA in/on plant materials (Richter, 2014: P 3009 G). Imidacloprid and its metabolites are extracted from whole orange fruit, tomato fruit, wheat grain, dry beans, olive fruit, tea (green tea and black tea), hop cones (green and dried), tobacco (green leaves and fermented tobacco), coffee (green beans and roasted coffee), and cocoa (green beans and roasted beans) with methanol/water (3/1, v/v). For the individual analytes, an aliquot of the extract is cleaned-up with liquid/liquid SPE. For the common moiety analysis, an aliquot of the extract is made by alkaline oxidation under reflux and liquid/liquid partition. Final extracts of both branches are subjected to reversed phase HPLC-MS/MS.

The LOQ (expressed as imidacloprid equivalents) for each analyte is 0.01 mg/kg. For dried, fermented and roasted difficult matrices (dried hop cones, fermented tobacco leaves, roasted cocoa beans, roasted coffee beans, black tea) the LOQ increased to 0.05 mg/kg, because validation attempts for dried hop cones and roasted coffee beans at 0.01 mg/kg failed. For the total residue of imidacloprid, the LOQ is 0.05 mg/kg for all matrices.

Table 5 Recovery results obtained for the determination of imidacloprid from tea (green tea and black tea)

Analyte	Fortification level (mg/kg)	N	Recovery range (%)	Mean recovery (%)	% RSD
Green tea					
Imidacloprid	0.01 0.10	5 5	84 – 117 82 – 109	100 100	16 12
5-hydroxy imidacloprid	0.01 0.10	5 5	63 – 99 67 – 86	79 76	20 9.3
Olefin imidacloprid	0.01 0.10	5 5	70 – 112 77 – 101	91 90	19 12
6-chloronicotinic acid	0.05 0.50	5 5	70 – 82 79 – 101	76 87	5.7 10
Black tea					
Imidacloprid	0.05 0.50	5 5	80 – 86 79 – 96	83 86	3.3 7.9
5-hydroxy imidacloprid	0.05 0.50	5 5	90 – 95 89 – 97	93 92	2.5 3.1
Olefin imidacloprid	0.05 0.50	5 5	78 – 95 84 – 93	89 88	9.2 3.7
6-chloronicotinic acid	0.05 0.50	5 5	74 – 95 69 – 84	82 75	10 8.1

Stability of pesticide residues in stored analytical samples

The storage stability of imidacloprid and various important metabolites was tested in various plant and animal materials. Tests on animal samples were carried out to assess the stability of the total residue. For plants, tests were carried out to assess the stability of the total residue and on plants to assess the stability of residues of the active substance and of the total residue. The results indicate that imidacloprid and the tested metabolites are stable for a minimum of approximately 2 years in plants and for at least 1 year in animal commodities (see JMPR 2002, 2006, 2008 and 2012).

The Meeting has received data on the storage stability of imidacloprid, 5-hydroxy imidacloprid and olefin imidacloprid in various plant matrices for a period of 36 months (Schoening and Diehl, 2014: MR-09/182, P642094733). Samples of wheat (grain), orange (fruit), tomato (fruit), bean (seed) and rape seed were fortified with imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid at a level of 0.1 mg/kg. The samples stored at an average temperature of -18°C or below were analysed at the nominal storage interval of 0, 30, 90, 180, 360, 540, 720, 900 and 1080 days.

At each storage interval imidacloprid and its metabolites 5-hydroxy and olefin were determined in the stored control samples and in the stored spiked samples according to the analytical method 00834/M002. Procedual recovery experiments at fortification levels of 0.10 mg/kg (0.01 mg/kg for 0 day storage interval) were also performed for each analyte at each storage interval. For all matrices the LOQ was 0.01 mg/kg for imidacloprid and its metabolite 5-hydroxy and olefin expressed as imidacloprid equivalent.

Table 6 Recovery of imidacloprid from stored fortified samples of plant matrices

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Wheat, grain			
0	91, 94	90, 91, 94, 95, 101	94
38	83, 90	85, 87, 96	89
90	88, 92	88, 92, 93	91

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
180	63, 80	81, 85, 102	89
361	93, 94	96, 98, 100	98
542	90, 95	99, 104, 105	103
719	77, 89	86, 87, 94	89
908	107, 110	91, 104, 129	108
1082	99, 106	94, 110, 110	105
Orange, fruit			
0	87	82, 82, 90, 92, 93	88
35	92, 94	75, 92, 96	88
91	84, 89	95, 100, 101	99
182	106, 113	93, 100, 112	102
366*/360	97, 101	106, 112, 117	112
540	96, 106	105, 110, 114	110
721	83, 88	82, 87, 93	87
912	107, 109	90, 115, 117	107
1080	97	106, 107, 107	107
Tomato, fruit			
0	90, 95	98, 98, 101, 102, 113	102
35	95, 100	88, 100, 100	96
90	93, 101	105, 107, 112	108
181	95, 99	106, 112, 113	110
360	94, 100	98, 105, 109	104
540	102, 105	109, 112, 116	112
720	86, 92	74, 79, 85	79
903	105, 112	108, 112, 120	113
1078	100, 108	110, 116, 124	117
Bean, seed			
0	74, 75	89, 91, 94, 95, 96	93
34	90, 102	85, 87, 88	87
90	85, 93	81, 82, 84	82
180	95, 96	101, 105, 111	106
359	87, 92	94, 97, 97	96
540	102, 109	94, 103, 106	101
720	90, 93	87, 95, 95	92
910	92, 95	89, 100, 106	98
1077	92, 93	97, 98, 104	100
Rape, seed			
0	79, 83	73, 90, 91, 91, 93	88
33	77, 82	73, 74, 80	76
90	84, 85	70, 75, 87	77
180	104, 107	103, 104, 111	106
361	99, 100	86, 87, 89	87
540	82, 85	59, 64, 66	64
719	85, 90	76, 89, 95	87
901	84, 85	78, 90, 95	88
1076	89, 100	82, 85, 92	86

* for procedual recoveries

Table 7 Recovery of 5-hydroxy imidacloprid from stored fortified samples of plant matrices

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Wheat, grain			
0	70, 73	77, 95, 96, 96, 102	93
38	87, 98	96, 100, 102	99
90	90, 91	70, 80, 86	79
180	90, 92	73, 83, 85	80
361	97, 98	100, 105, 105	103
542	89, 96	98, 101, 102	100

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
719	83, 96	94, 97, 103	98
908	103, 104	101, 102, 105	103
1082	100, 103	102, 105, 108	105
Orange, fruit			
0	79	99, 101, 103, 109, 112	105
35	98, 104	103, 105, 109	106
91	89, 98	98, 99, 99	99
182	109, 114	73, 76, 89	79
366*/360	93, 100	95, 101, 101	99
540	95, 106	95, 99, 103	99
721	100, 109	79, 103, 106	96
912	97, 99	96, 103, 111	103
1080	101	76, 92, 103	90
Tomato, fruit			
0	93, 109	98, 98, 99, 102, 104	100
35	105, 106	94, 99, 105	99
90	99, 102	95, 99, 104	99
181	106, 112	105, 106, 109	107
360	97, 105	91, 93, 94	93
540	107, 108	108, 115, 120	114
720	93, 94	95, 98, 99	97
903	91, 95	95, 101, 102	99
1078	84, 91	89, 94, 101	95
Bean, seed			
0	73, 73	72, 75, 80, 82, 86	79
34	99, 100	83, 84, 85	84
90	78, 94	83, 84, 84	84
180	101, 103	83, 97, 99	93
359	79, 84	93, 95, 97	95
540	115, 117	68, 83, 97	83
720	92, 93	91, 94, 95	93
910	89, 91	90, 93, 103	95
1077	107, 111	104, 108, 111	108
Rape, seed			
0	86, 86	86, 88, 89, 91, 96	90
33	89, 94	78, 85, 92	85
90	90, 93	76, 78, 78	77
180	105, 105	96, 99, 103	99
361	94, 98	84, 87, 92	88
540	103, 111	77, 86, 93	85
719	91, 91	88, 101, 103	97
901	91, 98	77, 81, 85	81
1076	89, 95	95, 96, 99	97

* for procedual recoveries

Table 8 Recovery of olefin imidacloprid from stored fortified samples of plant matrices

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Wheat, grain			
0	108, 114	79, 80, 82, 87, 90	84
38	77, 80	84, 85, 90	86
90	87, 91	86, 88, 96	90
180	90, 93	84, 85, 88	86
361	109, 110	92, 93, 95	93
542	90, 98	97, 105, 113	105
719	85, 90	93, 88, 93	88
908	103, 107	96, 99, 105	100
1082	98, 106	105, 109, 110	108

Storage interval (days)	Recovery (%) [0.10 mg/kg fortification]		
	Procedual	% remaining	Mean
Orange, fruit			
0	92	86, 87, 91, 92, 95	90
35	87, 94	87, 89, 97	91
91	80, 87	92, 95, 98	95
182	78, 89	60, 70, 75	68
366*/360	104, 107	88, 92, 92	91
540	93, 107	111, 112, 119	114
721	89, 97	89, 92, 99	93
912	86, 90	100, 102, 104	102
1080	102	67, 70, 105	81
Tomato, fruit			
0	85, 87	90, 101, 101, 102, 103	99
35	95, 97	90, 92, 96	93
90	85, 87	96, 96, 97	96
181	83, 86	85, 91, 95	90
360	105, 117	92, 96, 105	98
540	111, 113	93, 94, 98	95
720	94, 96	91, 92, 93	92
903	99, 104	108, 112, 120	113
1078	79, 84	74, 74, 95	81
Bean, seed			
0	69, 73	67, 70, 82, 83, 87	78
34	88, 96	70, 70, 71	70
90	80, 82	76, 77, 80	78
180	73, 76	71, 78, 81	77
359	81, 88	66, 80, 86	77
540	106, 108	99, 100, 104	101
720	87, 90	81, 87, 93	87
910	88, 89	79, 83, 101	88
1077	98, 100	101, 102, 111	105
Rape, seed			
0	84, 87	67, 71, 72, 72, 76	72
33	80, 86	79, 83, 91	84
90	94, 109	73, 76, 97	82
180	80, 83	65, 67, 72	68
361	97, 101	83, 85, 90	86
540	81, 86	74, 80, 94	83
719	81, 86	81, 84, 86	84
901	97, 103	83, 84, 85	84
1076	70, 81	70, 77, 79	75

* for procedual recoveries

Storage stability results indicated that residues of imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid were stable for at least 36 months under freezer conditions at about -18°C or below in wheat (grain), orange (fruit), tomato (fruit), bean (seed) and rape seed.

The Meeting has also received data on the storage stability of imidacloprid, olefin imidacloprid and 6-CNA in basil for a period of 9 months. Samples were fortified with imidacloprid, olefin imidacloprid and 6-CNA at a level of 0.50 mg/kg. The samples stored at -20°C were analysed at the storage interval of 0, 3, 6 and 9 months.

Imidacloprid, olefin imidacloprid and 6-CNA were determined in the control samples and in the stored fortified samples according to the analytical method 01389.

Table 9 Recovery of imidacloprid and its metabolites from stored fortified samples of basil

Storage interval (months)	Recovery (%) [0.50 mg/kg fortification]		
	Procedural	% remaining	Mean
Imidacloprid			

Storage interval (months)	Recovery (%) [0.50 mg/kg fortification]		
	Procedural	% remaining	Mean
0	78	76, 80	78
3	80	74, 82	78
6	77	70, 91	81
9	89	72, 79	76
Olefin imidacloprid			
0	79	77, 81	79
3	95	93, 96	95
6	73	76, 82	79
9	96	79, 90	85
6-CNA			
0	83	79, 87	83
3	95	93, 95	94
6	88	79, 85	82
9	82	79, 84	81

USE PATTERN

The Meeting received labels from Italy, Japan, Spain and the USA. The authorized uses relevant to the supervised residue trials data submitted to the current Meeting are summarized in Table 10.

Table 10 Registered uses of imidacloprid relevant to the residue evaluation by the current Meeting

Crop	Country	Formulation		Application					PHI, days
		Type	Conc. of imidacloprid	Method	kg ai/ha	kg ai/hL	L/ha	No. max	
Stone fruits									
Stone fruits	USA	SC	550 g/L	Soil	0.28-0.43 (max 0.43/year)			1	21
				Pre-plant, root dip	14.3 mL/38 L root dip solution			1	-
Stone fruits (Apricot, Nectarine, Peach)				Foliar	0.056-0.11 (max 0.34/year)		468 (G) 234 (A)	3-6	0 (7 days interval)
Stone fruits (Cherries, Plums, Plumcot, Prune)					0.056-0.11 (max 0.56/year)		468 (G) 234 (A)	5-10	7 (10 days interval)
Assorted tropical and sub-tropical fruits – edible peel									
Olive	Italy	OD	200 g/L	Foliar		0.01-0.013		1	28
Olive	Spain	SL	200 g/L	Foliar		0.01		2	7 (30 days interval)
				Foliar (a)	0.01-0.02		50-100	4	7 (7-10 days interval)
Brassica vegetables									
Cabbages (including cauliflower, broccoli and other brassica cabbage, cabbage head, leafy brassica, kohlrabi)	Italy	OD	200 g/L	Foliar		0.01		1	14
Cabbage (cabbage head, leafy brassica)	Italy	OD	75 g/L	Foliar	0.075-0.094			1-2	7
Fruiting vegetables, other than Cucurbits – subgroup Tomatoes									

Crop	Country	Formulation		Application					PHI, days
		Type	Conc. of imidacloprid	Method	kg ai/ha	kg ai/hL	L/ha	No. max	
Goji berry	China	EC	50 g/L	Foliar		0.003-0.005		3	3
Pulses									
Soya bean	USA	FS	480 g/L	Seed treatment	63-125 g ai/100 kg seed			1	-
		SC	550 g/L	Foliar	0.053 (max 0.16/year)			3	21 (7 days interval)
Herbs									
Basil	Thailand	WG	700 g/kg	Foliar		0.021-0.042		(b)	7
Teas									
Tea	Japan	WG	500 g/kg	Foliar		0.005-0.01	2000-4000	1	7

(a) spray solution containing a hydrolysed protein mixture at 1-2%, coarse drop application to parts facing south. Use only one of the two authorized methods (spray or bait) during the growing season of one crop.

(b) apply when infested

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

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

Group	Commodity	Table
Stone fruits	Cherries	Table 11
	Plum	Table 12
	Peach	Table 13
Assorted tropical and sub-tropical fruits–edible peel	Olive	Table 14–16
Leafy vegetables	Kale	Table 17
Fruiting vegetables, other than Cucurbits	Goji berry	Table 18
Pulses	Soya bean	Table 19
Herbs	Basil	Table 20
Teas	Tea	Table 21, 22
Legume animal feeds	Soya bean fodder and forage	Table 23

Imidacloprid formulations were applied by foliar treatment. Each of the field trial sites generally consisted of an untreated control plot and treated plots. Residues, application rates and spray concentrations have generally been rounded to two significant figures.

Residue values from the trials, which have been used for the estimation of maximum residue levels, STMRs and HRs are underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses and duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except when residues were found in samples from control plots. Residue data are not corrected for percent recovery.

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

Stone fruits

Cherries

Twelve residue trials for cherries were conducted in the USA (Dorschner, 2002: 111045). The 192 g/L SC formulation was applied five or six times as foliar spray at application rates 0.11-0.13 kg ai/ha. The total residue of imidacloprid was determined according to method 102624-R1 (based on the method 00200). The LOQ was 0.050 mg/kg.

Table 11 Imidacloprid residues on cherries from supervised trials in USA

Cherries country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
GAP, USA	*SC*	*Max 0.56 kg ai/ha /year*				*7*		
USA, 1999 Bridgeton/NJ (Montmorency tart cherry) 99-NJ17	SC	0.11 0.11 0.11 0.11 0.11	1031 1040 1025 1022 1027	100% petal fall Fruiting Green fruit First red fruit Ripening fruit	5	6	2.4, 2.5 Mean 2.5	111045 IR-4 PR. 07202 Dorschner, 2002
USA, 1999 Fennville/MI (Montmorency tart cherry) 99-MI09[a]	SC	0.11 0.11 0.11 0.11 0.11	953 931 944 939 935	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	0 3 7 14	1.2, 1.2 1.0, 1.1 1.0, 1.1 Mean 1.1 0.94, 1.0	Sampling to analysis: 189-250 days
USA, 1999 Fennville/MI (Montmorency tart cherry) 99-MI10[b]	SC	0.11 0.11 0.11 0.11 0.11	934 955 937 957 942	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	7	1.2, 1.5 Mean 1.4	
USA, 1999 Fennville/MI (Montmorency tart cherry) 99-MI11[c]	SC	0.11 0.11 0.11 0.11 0.11	936 950 943 931 941	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	7	0.88, 0.93 Mean 0.90	
USA, 1999 Traverse City/MI (Emperor Francis sweet cherry) 99-MI12[d]	SC	0.11 0.12 0.11 0.11 0.12	555 584 564 563 584	Pea-sized fruit 14-mm fruit 15-mm fruit 16-mm fruit 22-mm fruit	5	7	0.33, 0.34 Mean 0.34	
USA, 1999 Traverse City/MI (Hedelfingen sweet cherry) 99-MI13[e]	SC	0.12 0.11 0.11 0.12 0.12	579 562 564 578 579	- 14-mm fruit 17-mm fruit 22-mm fruit 24-25-mm fruit	5	7	0.39, 0.43 Mean 0.41	
USA, 1999 Grandview/WA (Bing sweet cherry) 99-WA19	SC	0.11 0.11 0.11 0.11 0.11	618 1149 1041 1094 1221	Bloom Fruiting Small fruit Fruiting Fruiting	5	8	0.24, 0.24 Mean 0.24	
USA, 1999 Buhl/ID (Bing sweet cherry) 99-ID07	SC	0.12 0.13 0.13 0.13 0.13 0.13	930 943 938 943 940 946	Late bloom Fruiting Fruiting Fruiting Fruiting Fruiting	6	7	0.55, 0.60 Mean 0.57	
USA, 1999 Caldwell/ID (Lambert sweet cherry) 99-ID08	SC	0.11 0.11 0.11 0.12 0.12 0.12	915 924 928 935 931 933	Bloom Part bloom Fruiting Fruiting Fruiting Maturing	6	7	0.62, 0.63 Mean 0.63	
USA, 1999 Hood River/OR (Bing sweet	SC	0.11 0.11 0.11	1890 1777 1833	Fruiting Fruiting A few turning pink	5	7	0.35, 0.36 Mean 0.36	

Cherries country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
cherry) 99-OR02		0.11 0.11	1813 1828	Fruit ripening Red fruit				
USA, 1999 Stockton/CA (Bing sweet cherry) 99-CA115[f]	SC	0.11 0.11 0.11 0.11 0.11	946 936 944 932 933	99% petal fall Fruiting Fruiting Immature fruit Immature fruit	5	7	0.22, 0.28 Mean 0.25	
USA, 1999 Stockton/CA (Dawson sweet cherry) 99-CA116[g]	SC	0.11 0.11 0.11 0.12 0.11	939 926 931 949 935	99% petal fall Fruiting Fruiting Immature fruit Immature fruit	5	7	0.45, 0.62 Mean 0.53	

Portion analysed: Fruit

[a] Application date: 19 May–29 June 1999, Trial site: Trevor Nochols Research Complex, 124th Ave., Fennville

[b] Application date: 26 May–2 July 1999, Trial site: Trevor Nochols Research Complex, 124th Ave., Fennville

[c] Application date: 25 May – 5 July 1999, Trial site: Trevor Nochols Research Complex, 124th Ave., Fennville

[d] Application date: 21 May–1 July 1999,

Trial site: NW Michigan Horticultural Research Station, 6686 S. Center Highway, Traverse City

[e] Application date: 1 June – 12 July 1999, Trial site: NW Michigan Horticultural Research Station, 6686 S. Center Highway, Traverse City

[f] Application date: 16 April – 20 May 1999, Trial site: 7700 Cherokee Lane, Stockton

[g] Application date: 16 April – 20 May 1999, Trial site: 7700 Cherokee Lane, Stockton

Plum

Eight residue trials were conducted in the USA on plums according to the US GAP (Dorschner, 2002: 111044). The 192 g/L SC formulation was applied 5 times at the rate of 0.11 kg ai/ha with an application interval of 8-12 days. The total residue of imidacloprid were quantified with method 102624-R1 (based on the method 00200) at an LOQ of 0.05 mg/kg.

Table 12 Imidacloprid residues on plums from supervised trials in USA

Plum country, year (variety)	Application					DALA Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
GAP, USA	*SC*	*Max 0.56 /year*				*7*		
USA, 1999 Bridgeton/NJ (Superior plum) 99-NJ16	SC	0.11 0.11 0.11 0.11 0.11	738 727 724 726 736	Fruiting Green sizing fruit Fruiting Fruiting Fruit enlarging	5	7	0.64, 0.70 Mean 0.67	111044 IR-4 PR. 07279 Dorschner, 2002
USA, 1999 Fennville/MI (Ealy Golden plum) 99-MI08	SC	0.11 0.11 0.11 0.11 0.11	922 939 942 917 927	Immature fruit Immature fruit Immature fruit Immature fruit Immature fruit	5	7	0.38, 0.46 Mean 0.42	Sampling to analysis: 75-235 days
USA, 1999 Gervais/OR (Brooks plum) 99-OR21	SC	0.11 0.11 0.11 0.11 0.11	1024 974 987 964 971	Green fruit Green fruit Fruit growth Ripening fruit Fruit maturing	5	6	0.089, 0.10 Mean 0.095	
USA, 1999 Gervais/OR (Brooks plum) 99-OR22	SC	0.12 0.11 0.11 0.11 0.11	770 756 766 747 746	Growing fruit, green Fruit growth Fruit growth Beginning to ripen Fruit ripening	5	7	0.077, 0.086 Mean 0.082	
USA, 1999 Buhl/ID (Simca Rosa	SC	0.11 0.11 0.11	924 936 938	Fruiting Fruiting Fruiting	5	7	0.16, 0.27 Mean 0.22	

Plum country, year (variety)	Application					DALA Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	water, L/ha	Treatment	no.			
plum) 99-ID05		0.11 0.11	933 933	Fruiting Fruiting				
USA, 1999 Caldwell/ID (Empress plum) 99-ID06	SC	0.11 0.11 0.11 0.11 0.11	929 940 929 940 930	Fruiting Fruiting Fruiting Fruiting Fruiting	5	7	0.32, 0.35 Mean <u>0.34</u>	
USA, 1999 Kerman/CA (French prunes) 99-CA79	SC	0.11 0.11 0.11 0.11 0.11	1403 1417 1430 1398 1395	Small green prunes Fruit 0.5-1 inch Fruit 1-1.5 inch Coloring prunes Fruiting	5	0 3 6 13	0.44, 0.52 0.44, 0.46 0.30, 0.41 Mean 0.36 0.39, 0.39 Mean <u>0.39</u>	
USA, 1999 Chowchilla/CA (Fortune plums) 99-CA80	SC	0.11 0.11 0.11 0.11 0.11	1409 1415 1401 1407 1416	Fruit 0.75-1 inch Fruit 1.5-2 inch Fruit 1.5-2.5 inch Coloring fruit Fruiting	5	7	0.12, 0.19 Mean <u>0.15</u>	

[a] Portion analysed: Fruit without pit and stem

Peach

Sixteen side-by-side residue trials were conducted in the USA on peaches according to the US GAP (Harbin & Woodard, 2000: 109238). The 192 g/L SC formulation was applied 3 times at the rate of 0.11 kg ai/ha with application intervals of 7 days. Two different application scenarios (concentrated and dilute spraying) were tested within the same location. The total residue of imidacloprid were quantified with method 102624-R1 (based on the method 00200) at an LOQ of 0.05 mg/kg.

Table 13 Imidacloprid residues on peaches from supervised trials in USA

Peach country, year (variety)	Application							DALA Days	Residues, mg/kg [a]		Ref
	Form	kg ai/ha	kg ai/hL		L/ha		no.				
			dil	conc	dil	conc			dil	conc	
GAP, USA	*SC*	*Max 0.34 /year*						*0*			
USA, 1998 Fresno/CA (Red top) FCA-PO001-98D	SC	0.11 0.11 0.11	0.0030 0.0029 0.0027	0.023 0.023 0.028	3714 3770 4022	485 485 391	3	0 7 14 21	<u>0.10</u> 0.099 0.066 0.059	0.094 0.058 0.074 0.051	109238 Harbin & Woodard, 2000
USA, 1998 Tulare/CA (Carson) BAY-PO002-98H	SC	0.11 0.11 0.11	0.0047 0.0048 0.0047	0.018 0.018 0.019	2338 2271 2324	615 628 575	3	0	<u>0.34</u>	0.25	Sampling to analysis: 378-414 days
USA, 1998 Porterville/CA (Red sun) BAY-PO003-98H	SC	0.11 0.11 0.11	0.0042 0.0052 0.0042	0.016 0.020 0.020	2605 2118 2598	684 561 549	3	0	<u>0.25</u>	0.15	
USA, 1998 Gridley/CA (Lodell 19440 ex erly) BAY-PO004-98H	SC	0.11 0.11 0.11	0.0056 0.0052 0.0052	0.020 0.019 0.020	1962 2104 2106	541 574 542	3	0	0.36	<u>0.37</u>	
USA, 1998 Colony/OK (Glohaven) BAY-PO005-98H	SC	0.11 0.11 0.11	0.0047 0.0045 0.0041	0.020 0.019 0.017	2327 2418 2715	543 572 633	3	0	0.48	<u>0.77</u>	
USA, 1998 Centralia/IL (Crest haven BAY-PO006-98H	SC	0.11 0.11 0.11	0.0032 0.0033 0.0033	0.018 0.018 0.018	3391 3328 3374	618 613 595	3	0	<u>0.38</u>	0.33	

Peach country, year (variety)	Application						no.	DALA Days	Residues, mg/kg [a]		Ref
	Form	kg ai/ha	kg ai/hL		L/ha						
			dil	conc	dil	conc			dil	conc	
USA, 1998 Morven/GA (Gold prince) BAY-PO007-98H	SC	0.11 0.11 0.11	0.0047 0.0045 0.0046	0.020 0.021 0.018	2334 2430 2372	540 525 601	3	0	0.38	0.32	
USA, 1998 Hereford/PA (Glohaven) BAY-PO008-98H	SC	0.11 0.11 0.11	0.0034 0.0034 0.0034	0.018 0.018 0.018	3224 3222 3237	623 603 615	3	0	0.28	0.19	

[a] Portion analysed: Fruit

Assorted tropical and sub-tropical fruits–edible peel & Oilseed

Olives

Eight trials on olives were conducted in Spain, Portugal, Italy and Greece (Schöning & Berkum, 2009: RA-2032/07, Schöning, Reneke & Krusell, 2011: 08-2001). The 200 g/L OD formulation was applied 5 times as a low pressure bait application with 0.020 kg ai/ha, corresponding to a concentration of 0.02 kg ai/hL and a spray volume of 100 L/ha. Only the south side (25% of the whole trees) was treated but samples were taken randomly from the whole trees. The application rate was related to the size of the plot and not just to the area actually treated. At each application the additive Buminal (hydrolyzed protein) was used (1.5%). The application intervals were 9 to 13 days.

All trials were analysed for imidacloprid parent compound and the total residue of imidacloprid according to method 00834/M001. Additionally, the samples taken in 2008 were analysed for imidacloprid parent compound and the metabolites 5-hydroxy imidacloprid and olefin imidacloprid according to method 00834/M002 (not be shown in Table 5).

Table 14 Imidacloprid residues on olives from supervised trials in Southern Europe

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage[a]	no.		Parent	Total	
GAP, Spain	*SL*	*0.01-0.02*		*50-100*		*4*	*7*			
Spain, 2007 Cataluña (Morrut) R2007 0408/9	OD	0.02	0.02	100	81-85	5	-0 0 4 7	0.27 0.45 - 0.40	0.47 0.64 0.56 0.71	RA-2032/07 Schöning & Berkum, 2009
Portugal, 2007 Ribatejo e Oeste (Galega) R2007 0409/7	OD	0.02	0.02	100	81-88	5	-0 0 7	0.33 0.70 0.40	0.81 1.3 1.1	Sampling to analysis: 63-141 days
Italy, 2007 Sicilia (Nocellara Etnea) R2007 0439/9	OD	0.02	0.02	100	78-80	5	-0 0 3 7	0.03 0.04 - 0.03	0.15 0.14 0.17 0.14	
Italy, 2007 Puglia (Corato) R2007 0440/2	OD	0.02	0.02	100	75-80	5	-0 0 7	0.13 0.30 0.30	0.33 0.57 0.63	
Spain, 2008 Cataluña (Vera) 08-2001-01	OD	0.02	0.02	100-114	80-88	5	-0 0 4 8	0.04 0.17 0.07 0.05	0.12 0.24 0.12 0.11	08-2001 Schöning, Reineke & Krusell, 2011
Italy, 2008 Sicilia (Bella di Spagna) 08-2001-02	OD	0.02	0.02	100	80-85	5	-0 0 3 7	< 0.01 0.04 0.02 0.02	< 0.05 0.06 < 0.05 < 0.05	Sampling to analysis: 484-

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage[a]	no.		Parent	Total	
Portugal, 2008 Ribatejo e Oeste (Galega) 08-2001-03	OD	0.02	0.02	100	79-88	5	0 7	0.37 0.42	0.45 0.49	534 days
Greece, 2008 Katerini (Megaron) 08-2001-04	OD	0.02	0.02	100	76-81	5	0 7	0.22 0.11	0.34 0.22	

Portion analysed: Fruit

[a] Code of BBCH scale

[b] -0: the date before last treatment

Eight trials on olives were conducted in Spain, Italy, Portugal and Greece (Anderson & Eberhardt, 2002: RA-2065/00, Schöning, 2002: RA-2034/01). The 200 g/L SL formulation was applied twice as a spray application with 0.10 kg ai/ha, corresponding to a concentration of 0.0125 kg ai/hL and a spray volume of 800 L/ha. The application intervals were 28 to 32 days.

Fruits taken at day 0 at and the PHI of 28 days after the last application were analysed for parent compound whereas fruits of all sampling dates were analysed for the total residue of imidacloprid. Both analytes were either analysed according to method 00300/E007 or method 00834.

Table 15 Imidacloprid residues on olives from supervised trials in Southern Europe

Olive country, year (variety)	Application						DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage*	no.		Parent	Total	
GAP, Spain	*SL*		*0.01*			*2*	*28*			
Spain, 2000 (Vera) R2000 0073/1	SL	0.10 0.10	0.013 0.013	800 800	81 85	2	0 6 11 21 28 35	0.12 0.02 	0.25 0.27 0.28 0.29 0.25 0.22	RA-2065/00 Anderson & Eberhardt, 2002
Italy, 2000 (Nocellara Etnea) R2000 0313/7	SL	0.10 0.093	0.013 0.013	800 743	87 87/88	2	0 7 14 22 28 35	0.10 < 0.01 	0.11 0.11 0.08 < 0.05 0.05 0.05	Sampling to analysis: 243-354 days
Portugal, 2000 (Blanqueta) R2000 0314/5	SL	0.10 0.10	0.013 0.013	800 800	No data 82	2	0 6 14 21 28 35	0.18 0.03 	0.60 0.36 0.19 0.16 0.21 0.07	
Greece, 2000 (Manaki) R2000 0315/3	SL	0.10 0.10	0.013 0.013	800 800	79 85	2	0 7 14 21 28 35	0.32 0.14 	0.59 0.71 0.51 0.67 0.73 0.43	
Spain, 2001 (Vera) R2001 0090/6	SL	0.11 0.10	0.013 0.013	904 800	79 79-81	2	0 6 14 19 27 35	0.14 0.09 0.05 0.02 	0.28 0.26 0.26 0.26 0.22 0.24	RA-2034/01 Schöning, 2002 Sampling to analysis: 134-195 days
Italy, 2001 (Nocellara Etnea) R2001 0091/4	SL	0.10 0.10	0.013 0.013	800 800	78 78-80	2	0 7 14	0.14 0.04 0.02	0.22 0.14 0.12	

Olive country, year (variety)	Application						DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage*	no.		Parent	Total	
							20 28 35	 0.01	0.11 0.15 0.13	
Portugal, 2001 (Picual) R2001 0092/2	SL	0.10 0.10	0.013 0.013	800 800	75/76 79/80	2	0 7 14 21 28 35	0.31 0.11 0.09 0.06	0.39 0.22 0.19 0.10 0.16 0.16	
Greece, 2001 (Manaki) R2001 0093/0	SL	0.10 0.10	0.013 0.013	800 800	79 82	2	0 8 15 22 28 35	0.22 0.01 < 0.01 < 0.01	0.29 0.08 0.06 0.05 0.06 0.06	

Portion analysed: Fruit

* Code of BBCH scale

Eight trials on olives were conducted in Italy, Spain, Portugal and Greece (Schöning & Krusell, 2011: 09-2087, Schöning & Bauer, 2011: 10-2151). The 200 g/L OD formulation was applied once as a spray application with 0.15 kg ai/ha, corresponding to a concentration of 0.0125–0.0188 kg ai/hL and a spray volume of 800–1200 L/ha.

The samples were analysed for imidacloprid parent compound and the metabolites 5-hydroxy imidacloprid and olefin imidacloprid according to method 00834/M002 as well as for the total residue of imidacloprid according to method 00834/M001.

Table 16 Imidacloprid residues on olives from supervised trials in Southern Europe

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage [a]	no.		Parent	Total	
GAP, Italy	*OD*		*0.01-0.013*			*1*	*28*			
Italy, 2009 (Nocellara Etnea) 09-2087-01	OD	0.15	0.015	1000	78	1	-0 0 7 14 28 35	< 0.01 0.33 0.16 0.06 < 0.01 < 0.01	< 0.05 0.31 0.29 0.30 0.23 0.28	09-2087 Schöning & Krusell, 2011 Sampling to analysis: 266-428 days
Spain, 2009 (Arbequina) 09-2087-02	OD	0.15	0.015	1000	85	1	-0 0 7 13 28 35	< 0.01 1.02 0.44 0.27 0.14 0.10	< 0.05 0.93 0.79 0.74 0.75 0.77	
Portugal, 2009 (Cobrançosa) 09-2087-03	OD	0.15	0.019	800	80	1	0 28	0.70 0.51	0.51 0.81	
Italy, 2009 (Nocellara Etnea) 09-2087-04	OD	0.15	0.013	1200	81	1	0 28	0.32 0.01	0.29 0.23	
Italy, 2010 (Bella di Spagna) 10-2151-01	OD	0.15	0.015	1000	78	1	0 7 14 28 35	0.21 0.16 0.07 < 0.01 < 0.01	0.16 0.18 0.13 0.26 0.20	10-2151 Schöning & Bauer, 2011
Spain, 2010 (Arbequina) 10-2151-02	OD	0.15	0.019	800	81	1	0 8 14	0.40 0.20 0.16	0.16 0.35 0.38	Sampling to analysis: 79-129 days

Olive country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	Growth stage [a]	no.		Parent	Total	
							28 35	0.04 0.04	0.41 0.43	
Portugal, 2010 (Cobrançosa) 09-2087-03	OD	0.15	0.019	800	81	1	0 30	0.77 0.43	0.68 0.61	
Greece, 2010 (Amphisses) 10-2151-04	OD	0.15	0.019	800	79	1	0 28	0.34 < 0.01	0.24 0.12	

Portion analysed: Fruit

[a] Code of BBCH scale

[b] -0: The date before last treatment

Leafy vegetables (including Brassica leafy vegetables)

Kale

Four trials were conducted on curly kale in Spain and Italy (Schmeer, Krusell & Bauer, 2010: 08-2029, Ballesteros, 2011: 09-2002). The OD formulation containing 75 g/L imidacloprid and 10 g/L deltamethrin was applied twice as a spray application with 0.094 kg ai/ha, corresponding to a concentration of 0.012–0.016 kg ai/hL and a spray volume of 600–800 L/ha. The application interval was 13–18 days.

The samples were analysed for the parent imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid according to method 00834/M002. The total residue of imidacloprid was determined as 6-CNA common moiety according to method 00834/M001.

Table 17 Imidacloprid residues on curly kale from supervised trials in Spain and Italy

Kale country, year (variety)	Application						DALA Days [b]	Residues, mg/kg		Ref
	Form	kg ai/ha	Kg ai/hL	L/ha	Growth stage [a]	no.		Parent	Total	
GAP, Italy	*OD*	0.075-0.094				*2*	*7*			
Spain, 2008 (Reflex F1) 08-2029-01	OD	0.094 0.094	0.016 0.012	600 800	47 49	2	-0 0 3 6 13	< 0.01 2.9 0.46 0.09 0.02	0.46 3.7 1.8 <u>1.1</u> 0.82	08-2029 Schmeer, Krusell & Bauer, 2010
Italy, 2008 (Nero di Toscana) 08-2029-02	OD	0.094 0.094	0.012 0.012	800 800	42 46	2	-0 0 3 7 14	0.03 2.5 0.64 0.20 0.10	0.34 3.3 2.2 <u>1.5</u> 0.99	Sampling to analysis: 307-596 days
Spain, 2009 (Reflex F1) 09-2002-01	OD	0.094 0.094	0.016 0.016	600 600	42 45	2	-0 0 3 7 15	0.16 2.9 0.99 0.34 0.04	0.93 4.1 2.8 <u>2.0</u> 1.1	09-2002 Ballesteros, 2011
Italy, 2009 (Nero di Toscana) 09-2002-02	OD	0.094 0.094	0.012 0.012	800 800	41 43	2	-0 0 3 6 14	0.02 1.5 0.50 0.31 0.09	0.36 2.3 1.0 <u>1.0</u> 0.64	Sampling to analysis: 85-198 days

Portion analysed: Leaf

[a] Code of BBCH scale

[b] 0: The date before last treatment

Fruiting vegetables, other than Cucurbits–subgroup Tomatoes

Goji berry

Six trials were conducted on goji in China, using the EC formulation containing 50 g/L imidacloprid. The EC formulation was applied with three foliar applications at a concentration of 0.005 kg ai/hL. The application interval was 10 days.

After fresh goji were collected from field trial, 5 g potassium carbonate (0.5% of the weight of fresh goji sample) was added per 1000 g sample, then well mixed and stood for 30 min. The sample was dried in sunshine or under blast drying under 45-50°C. The water content of goji is 70-80%. So the weight of dried goji is about 20-30% of that before drying.

The analytical method NY/T 1275-2007 & GB/T 23201-2008 was used to determine the residue of imidacloprid on goji. The LOQ was 0.02 mg/kg.

Table 18 Imidacloprid residues on goji from supervised trials in China

Goji country, year (variety)	Application			DALA Days	Portion analysed	Residues, mg/kg* Imidacloprid	Ref
	Form	kg ai/hL	no.				
GAP, China	*EC*	*0.003-0.005*	*3*	*3*			
China, 2010 Yinchuan/ Ningxia Hui (Ningqi No. 1) NX-01	EC	0.005	3	1 2 3 5 7 10 14	Fresh fruits	0.078, 0.099, 0.11 (0.096) 0.052, 0.054, 0.082 (0.063) 0.021, 0.032, 0.067 (0.040) < 0.02, < 0.02, < 0.02 (< 0.02) < 0.02, < 0.02, < 0.02 (< 0.02) < 0.02, < 0.02, < 0.02 (< 0.02) < 0.02, < 0.02, < 0.02 (< 0.02)	R-IG-03 Niu, 2014 Sampling to analysis: 131-150 days
				5 7 10 14	Dried fruits	< 0.02, 0.023, 0.038 (0.027) 0.059, 0.062 (0.061) 0.054, 0.055 (0.055) 0.025, 0.027, 0.027 (0.026)	
China, 2010 Zhongning/ Ningxia Hui (Ningqi No. 1) NX-02	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.32, 0.34, 0.69 (0.45) 0.54, 0.64, 0.69 (0.62) 0.36, 0.43, 0.47 (0.42) 0.033, 0.035, 0.036 (0.035) 0.029, 0.030, 0.030 (0.030) 0.051, 0.055, 0.058 (0.055) < 0.02, < 0.02, 0.024 (0.021) < 0.02, < 0.02, < 0.02 (< 0.02)	R-IG-04 Niu, 2014 Sampling to analysis: 129-149 days
				5 7 10 14	Dried fruits	0.063, 0.092, 0.10 (0.085) < 0.02, 0.025, 0.034 (0.026) < 0.02, < 0.02, < 0.02 (< 0.02) < 0.02, < 0.02, 0.024 (0.021)	
China, 2010 Bayannaoer/ Inner Mongolia (Ningqi No. 1) IM-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.77, 0.84, 0.99 (0.87) 0.57, 0.61, 0.67 (0.62) 0.57, 0.59, 0.78 (0.65) 0.36, 0.39, 0.41 (0.39) 0.28, 0.31, 0.35 (0.31) 0.25, 0.26, 0.26 (0.26) 0.072, 0.094, 0.12 (0.095) < 0.02, < 0.02, < 0.02 (< 0.02)	R-IG-05 Zhang, 2014 Sampling to analysis: 130-150 days
				5 7 10 14	Dried fruits	0.42, 0.64 (0.53) 0.49, 0.54 (0.52) 0.29, 0.30, 0.36 (0.32) 0.24, 0.31 (0.28)	

Goji country, year (variety)	Application Form	kg ai/hL	no.	DALA Days	Portion analysed	Residues, mg/kg* Imidacloprid	Ref
China, 2010 Baiyin/ Gansu (Ningqi No. 1) GS-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.38, 0.44, 0.85 (0.56) 0.25, 0.45, 0.47 (0.39) 0.26, 0.32, 0.48 (0.35) 0.17, 0.19, 0.20 (0.19) 0.10, 0.22 (0.16) 0.054, 0.066, 0.13 (0.083) 0.012, 0.030 (0.021) < 0.02, < 0.02, < 0.02 (< 0.02)	R-IG-06 Liu, 2014 Sampling to analysis: 124-144 days
				5 7 10 14	Dried fruits	0.10, 0.11, 0.14 (0.12) 0.049, 0.050, 0.17 (0.090) < 0.02, 0.029, 0.040 (0.030) < 0.02, < 0.02, 0.026 (0.022)	
China, 2010 Xinjiang Uygur (Ningqi No. 1) XJ-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.29, 0.34, 0.41 (0.35) 0.30, 0.31, 0.41 (0.34) 0.30, 0.31, 0.39 (0.33) 0.24, 0.28, 0.31 (0.28) 0.28, 0.30, 0.37 (0.32) 0.22, 0.27, 0.30 (0.26) 0.25, 0.27, 0.28 (0.27) 0.020, 0.14, 0.14 (0.10)	R-IG-07 Gou, 2014 Sampling to analysis: 122-142 days
				5 7 10 14	Dried fruits	0.044, 0.047, 0.086 (0.059) 0.059, 0.14, 0.17 (0.12) 0.054, 0.075, 0.076 (0.068) 0.051, 0.068, 0.070 (0.063)	
China, 2010 Haixi, Qinghai (Ningqi No. 1) QH-01	EC	0.005	3	1 2 3 5 7 10 14 21	Fresh fruits	0.40, 0.47, 0.51 (0.46) 0.17, 0.37, 0.56 (0.37) 0.23, 0.30, 0.44 (0.32) 0.16, 0.21, 0.25 (0.21) 0.17, 0.17, 0.17 (0.17) 0.13, 0.17, 0.20 (0.17) 0.27, 0.44 (0.36) 0.083, 0.18, 0.39 (0.22)	R-IG-08 Gou, 2014 Sampling to analysis:
				5 7 10 14	Dried fruits	0.74, 0.74, 0.78 (0.75) 0.22, 0.46, 0.46 (0.38) 0.62, 0.63 (0.63) 0.28, 0.28, 0.29 (0.28)	

* Average in parentheses

Pulses

Soya bean (dry)

Twenty-one field residue trials were carried out with imidacloprid in soya beans in the USA and Canada using the 480 g/L SC formulation (Mackie, 2006: RANTY002). Soya bean seeds were treated at a rate of 0.125 kg ai/100 kg seed. The growing soya bean plants were subsequently treated with three foliar applications at a target rate of 0.053 kg ai/ha for a total seasonal application of 0.16 kg ai/ha. In each of the 21 residue trials, the treated plot was divided in two sub-plots A and B; from sub-plot A, forage and hay was harvested, and from sub-plot B, soya bean seeds. The application intervals, once foliar treatment was initiated, generally ranged between 5 and 7 days.

The analytical method NT-001-P04-01 (common moiety method) was used to determine the total residue of imidacloprid in soya bean seeds.

Table 19 Imidacloprid residues on soya bean seeds from supervised trials in USA and Canada

Soya bean seed country, year (variety)	Application Form	kg ai/ha	Seeding density water, L/ha	no.	DALA Days	Residues, mg/kg* Total imidacloprid	Ref
GAP, USA	*SC*	*63-125 g ai/100 kg seed*		*1*	*21*		

Soya bean seed country, year (variety)	Application				DALA Days	Residues, mg/kg* Total imidacloprid	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.			
		0.053 *Max 0.16 kg ai/ha /year*		*3*			
USA, 2004	SC	0.070	56.3 kg/ha	1	7	0.035, 0.036 (0.035)	RANTY002 Mackie, 2006
Tifton/GA		0.053	141	3	14	0.022, 0.031 (0.027)	
(DK 5386)		0.053	141		21	0.047, 0.054 (0.050)	
NT001-04D		0.053	140		28	0.037, 0.039 (0.038)	
					34	0.042, 0.043 (0.042)	Sampling to analysis: max 450 days
USA, 2004	SC	0.082	65.9 kg/ha	1	19	0.17, 0.25 (0.21)	
Bumpass/VA		0.052	162	3			
(Pioneer 9492RR)		0.053	163				
NT002-04H		0.053	163				
USA, 2004	SC	0.091	72.5 kg/ha	1	20	0.35, 0.41 (0.38)	
Leland/MS		0.054	151	3			
(Pioneer 9492RR)		0.052	151				
NT003-04H		0.052	157				
USA, 2004	SC	0.098	78.5 kg/ha	1	21	0.64, 0.71 (0.67)	
Proctor/AR		0.054	140	3			
(DK 5386)		0.054	140				
NT004-04H		0.053	139				
USA, 2004	SC	0.087	69.4 kg/ha	1	21	0.32, 0.43 (0.38)	
Newport/AR		0.053	185	3			
(DK 5386)		0.054	189				
NT005-04H		0.054	188				
USA, 2004	SC	0.10	79.9 kg/ha	1	8	0.039, 0.041 (0.040)	
Stilwell/KS		0.055	153	3	14	0.038, 0.042 (0.040)	
(Pioneer 93B68)		0.056	153		20	0.024, 0.030 (0.027)	
NT006-04D		0.053	145		27	0.030, 0.031 (0.031)	
					34	0.033, 0.037 (0.035)	
USA, 2004	SC	0.090	72.2 kg/ha	1	19	0.17, 0.19 (0.18)	
Seymour/IL		0.054	132	3			
(Pioneer 93B68)		0.054	132				
NT007-04H		0.054	132				
USA, 2004	SC	0.093	74.2 kg/ha	1	20	0.066, 0.15 (0.11)	
Springfield/NE		0.054	139	3			
(S2802-4)		0.053	140				
NT008-04H		0.053	133				
USA, 2005	SC	0.096	76.9 kg/ha	1	21	0.18, 0.20 (0.19)	
Sabin/MN		0.053	153	3			
(Northrup King)		0.055	158				
NT009-04HA		0.054	152				
USA, 2004	SC	0.092	74.0 kg/ha	1	20	0.40, 0.46 (0.43)	
Carlock/IL		0.053	121	3			
(S2802-4)		0.052	119				
NT010-04H		0.055	124				
USA, 2004	SC	0.15	122 kg/ha	1	19	0.45, 0.52 (0.48)	
Bagley/IA		0.053	130	3			
(S2802-4)		0.051	132				
NT011-04H		0.051	134				
USA, 2004	SC	0.084	67.3 kg/ha	1	19	0.61, 0.65 (0.63)	
Marysville/OH		0.053	140	3			
(S2802-4)		0.053	141				
NT012-04H		0.053	140				
USA, 2004	SC	0.090	71.6 kg/ha	1	21	0.94, 2.0 (1.5)	
Dumfries/MN		0.054	176	3			
(Pioneer 92B13)		0.055	179				
NT013-04H		0.053	178				
USA, 2004	SC	0.071	57.2 kg/ha	1	20	0.56, 0.65 (0.61)	
Northwood/ND		0.052	185	3			
(S02-G2)		0.052	186				
NT014-04H		0.053	187				
USA, 2004	SC	0.092	73.8 kg/ha	1	21	0.73, 0.73 (0.73)	

Soya bean seed country, year (variety)	Application				DALA Days	Residues, mg/kg* Total imidacloprid	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.			
Gardner/ND (Pioneer 90B51) NT015-04H		0.052 0.053 0.053	182 181 161	3			
USA, 2004 New Holland/OH (Pioneer 93B68) NT016-04H	SC	0.094 0.053 0.054 0.054	74.9 kg/ha 148 153 150	1 3	32	0.025, 0.029 (0.027)	
USA, 2004 Kirksville/MO (Pioneer 93B68) NT017-04H	SC	0.085 0.053 0.052 0.054	68.3 kg/ha 165 170 178	1 3	21	1.4, 1.6 (1.5)	
USA, 2004 Ellendale/MN (Pioneer 92B13) NT018-04H	SC	0.11 0.054 0.055 0.054	83.9 kg/ha 154 156 160	1 3	21	0.57, 0.67 (0.62)	
USA, 2004 Carlyle/IL (Pioneer 93B68) NT019-04H	SC	0.087 0.053 0.053 0.052	69.3 kg/ha 92 137 93	1 3	21	0.039, 0.065 (0.052)	
USA, 2004 Rockwood/ON (S02-G2) NT020-04H	SC	0.094 0.053 0.053 0.053	75.2 kg/ha 94 95 97	1 3	25	0.034, 0.069 (0.052)	
USA, 2004 Bright/ON (Pioneer 90B51) NT021-04H	SC	0.094 0.054 0.053 0.053	75.2 kg/ha 92 101 89	1 3	25	0.093, 0.096 (0.094)	

* Average in parentheses

Herbs

Basil

Four field residue trials were carried out with imidacloprid in basil in Thailand using the 700 g/kg WG formulation. The growing basil plants were treated with two foliar applications at a target concentration of 0.042 kg ai/hL. The application intervals were 7 or 8 days.

The on-line multi residue methods applied for the determination of imidacloprid residues was based on extraction with a mixture of acetone, dichloromethane and sodium chloride water solution. The concentrated extract is cleaned up on silica gel column and detection with HPLC-MS/MS (Steinwandter, 1985). The LOQ for imidacloprid was 0.01 mg/kg. The total residue of imidacloprid (6-CNA common moiety analysis) was determined according to method 01389. The LOQ for total imidacloprid was 0.05 mg/kg.

Table 20 Imidacloprid residues on basil from supervised trials in Thailand [a]

Basil country, year (variety)	Application				DALA Days	Residues, mg/kg*		Ref.
	Form	kg ai/ha	kg ai/hL	no.		Imidacloprid	Total imidacloprid	
GAP, Thailand	*WG*		*0.021-0.042*		*7*			
Thailand, 2014 Nakornpratom (White Holy basil)	WG	0.34	0.042	2	0 1 3 5 7 10 14	16, 24, 26 (22) 8.4, 14, 22 (15) 4.7, 5.0, 5.4 (5.0) 1.5, 2.8, 3.0 (2.4) 0.94, 1.1, 1.4 (1.1) 0.34, 0.41, 0.55 (0.43) 0.04, 0.07, 0.20 (0.10)	16, 28, 38 (27) 16, 25, 40 (27) 4.2, 4.8, 5.8 (4.9) 2.8, 3.5, 5.1 (3.8) 2.5, 4.3, 5.3 (4.0) 3.3, 5.6, 6.5 (5.1) 3.3, 3.8, 3.8 (3.6)	Imida- basil-1 Chaiyanboon, 2014 Sampling to analysis: 268-284 days
Thailand, 2014	WG	0.32	0.042	2	0	31, 46, 49 (42)	40, 49, 50 (46)	Imida-basil-2

Basil country, year (variety)	Application				DALA Days	Residues, mg/kg*		Ref.
	Form	kg ai/ha	kg ai/hL	no.		Imidacloprid	Total imidacloprid	
Saraburi (Red Holy basil)					1 3 5 7 10 14	10, 11, 12 (11) 1.1, 2.8, 3.4 (2.4) 1.2, 1.2, 1.3 (1.2) 0.41, 0.43, 0.49 (0.44) 0.15, 0.21, 0.21 (0.19) < 0.01,< 0.01, 0.03 (0.017)	19, 21, 21 (20) 23, 23, 25 (23) 8.1, 10, 13 (11) 6.1, 6.2, 7.3 (6.5) 4.4, 4.5, 4.8 (4.6) 2.7, 3.4, 3.6 (3.2)	Thongsam, 2014 Sampling to analysis: 256-271 days
Thailand, 2014 Nakhonpathom (Sweet basil)	WG	0.32	0.042	2	0 1 3 5 7 10 14	24, 24, 25 (24) 13, 15, 15 (14) 4.1, 4.3, 4.8 (4.4) 2.1, 2.2, 2.3 (2.2) 0.98, 1.1, 1.2 (1.1) 0.29, 0.32, 0.37 (0.33) 0.17, 0.18, 0.26 (0.20)	23, 25, 29 (26) 18, 19, 20 (19) 8.4, 8.7, 9.7 (8.9) 7.3, 7.3, 8.3 (7.6) 4.2, 4.7, 5.7 (4.9) 2.4, 2.5, 2.9 (2.6) 0.95, 1.2, 1.9 (1.3)	Imida-basil-3 Pongpinyo 2014 Sampling to analysis: 215-232 days
Thailand, 2014 Supanburi (Sweet basil)	WG	0.26	0.042	2	0 1 3 5 7 10 14	18, 19, 21 (19) 3.9, 4.1, 4.1 (4.0) 1.5, 1.7, 1.7 (1.6) 0.64, 0.70, 0.74 (0.69) 0.19, 0.22, 0.23 (0.21) 0.06, 0.07, 0.07 (0.07) 0.02, 0.03, 0.06 (0.04)	21, 24, 30 (25) 10, 11, 14 (12) 6.0, 6.1, 6.4 (6.1) 4.7, 6.0, 6.2 (5.6) 3.9, 4.5, 4.7 (4.3) 1.8, 1.9, 2.3 (2.0) 0.42, 0.54, 0.96 (0.64)	Imida-basil-4 Phaikaew 2014 Sampling to analysis: 264-280 days

[a] Portion analysed: whole commodity

* Average in parentheses

Tea, Green, Black

A total of eight trials (four decline and four harvest trials) were conducted at four different trial locations during the dry season (Manikandan, 2015: RANTN021). Four trials were conducted in spring and the other remaining four trials in autumn. Imidacloprid 700 g/kg WG formulation was applied in a spray application once at 0.40 kg ai/ha. In decline trials tea shoots (two to three leaves and a bud) were harvested 0, 3, 7 and 10 days after the application. In three of them duplicate composite samples were taken and in one decline trial only single samples were taken. In harvest trials, tea shoots were harvested immediately after the application and 7 days thereafter. In all the decline and harvest trials a portion of the tea shoots harvested 7 days after application was used to manufacture green and black tea.

For green tea production, tea shoots comprising of two to three leaves and bud harvested from experimental plots were subjected for steaming for about 10 min. Then the leaves were allowed to cool down to room temperature and subjected to CTC (Crush, Tear and Curl) manufacturing process. The tea leaves were dried for about 20 min in a fluidized bed drier, and cooled to ambient temperature.

For black tea production, the tea leaves were spread to a thickness of about 6.4 cm in a miniature withering trough and allowed to wither for 15-16 hours. The withered leaves were put into a rolling machine and rolled. The rolled leaves were then passed through a CTC machine. Afterwards, the tea was fermented at a humidity of 90%. The fermented tea was dried in a fluid bed drier. The dried tea was allowed to cool at ambient conditions.

All samples were analysed for imidacloprid parent compound and its metabolites and the total residue of imidacloprid according to method 01389. The LOQ was 0.01 mg/kg for imidacloprid in fresh and green tea leaves and 0.05 mg/kg in black tea. The LOQ of the total residue of imidacloprid was 0.05 mg/kg in all sample materials.

Table 21 Imidacloprid residues on tea (fresh leaves) from supervised trials in India

Tea	Application	DALA	Residues, mg/kg*	Ref

country, year (variety)	Form	kg ai/ha	kg ai/hL	L/ha	no.	Days	Parent	Total	
GAP, Japan	*WG*		0.005-0.01	*2000-4000*	*1*	*7*			
India, 2013 Gudalur (Assam Jat/ Seedling tea) S1	WG	0.40	0.089	450	1	0 3 7 10	54, 58 (56) 0.77, 0.99 (0.88) 0.40, 0.52 (0.46) 0.32, 0.37 (0.35)	53, 59 (56) 1.3, 1.4 (1.4) 0.70, 0.82 (0.76) 0.58, 0.65 (0.62)	Sampling to analysis: 207-370 days
India, 2012 Meppadi (Assam Jat/ Seedling tea) S2	WG	0.40	0.089	450	1	0 3 7 10	43, 46 (45) 7.7, 8.6 (8.2) 0.93, 1.1 (1.0) 0.95, 1.0 (0.98)	48, 51 (50) 12, 13 (13) 2.7, 2.8 (2.8) 2.8, 2.8 (2.8)	Sampling to analysis: 189-298 days
India, 2012 Coonoor (Assam Jat/ UPASI-9) S3	WG	0.40	0.089	450	1	0 7	21 c:0.038 1.5	25 2.3	Sampling to analysis: 175-291 days
		1.2	0.27	450	1	0 7	116 4.4, 4.5 (4.5)	151 7.1, 7.2 (7.2)	
India, 2013 Valparai (Assam Jat/ Seedling tea) S4	WG	0.40	0.089	450	1	0 7	39 0.64	48 1.2	Sampling to analysis: 175-281 days
		1.2	0.27	450	1	0 7	124 1.4, 1.4 (1.4)	155 2.6, 3.2 (2.9)	
India, 2012 Valparai (Assam Jat/ Mixed seedling tea) N1	WG	0.40	0.089	450	1	0 3 7 10	46, 51 (49) c:0.040, 0.26 7.2, 7.4 (7.3) 2.8, 4.2 (3.5) c:0.20 2.8, 3.1 (3.0)	51, 56 (54) c:0.29 10, 11 (11) 4.3, 6.1 (5.2) c:0.36 4.0, 4.0 (4.0)	Sampling to analysis: 39-96 days
India, 2011 Coonoor (Assam Jat/ UPASI-9) N2	WG	0.40	0.089	450	1	0 3 7 10	7.5 0.88 0.77 0.57	7.9 0.92 0.87 0.76	Sampling to analysis: 46-100 days
India, 2010 Meppadi (Assam Jat/ Seedling tea) N3	WG	0.40	0.089	450	1	0 7	54 0.48	59 1.0	Sampling to analysis: 56-102 days
India, 2011 Gudalur (Assam Jat/ TRI-2024) N4	WG	0.40	0.089	450	1	0 7	31 1.4	32 2.4	Sampling to analysis: 54-101 days

* Average in parentheses

c: control sample

Table 22 Imidacloprid residues on tea (Green tea and Black tea) from supervised trials in India

Tea country, year (variety)	Application					DALA Days	a	Residues, mg/kg*		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	no.			Parent	Total	
GAP, Japan	*WG*		0.005-0.01	*2000-4000*	*1*	*7*				Sampling to analysis
India, 2013 Gudalur (Assam Jat/ Seedling tea) S1	WG	0.40	0.089	450	1	7	G	1.7, 1.7 (1.7) c:0.011, 0.013	2.7, 3.1 (2.9)	269-310 days
							B	1.3, 1.6 (1.5)	3.1, 3.4 (3.3)	264-313 days
India, 2012 Meppadi	WG	0.40	0.089	450	1	7	G	4.7, 4.8 (4.8) c:0.018, 0.023	11, 12 (12) c:0.40	253-294

Tea country, year (variety)	Application					DALA Days	[a]	Residues, mg/kg*		Ref
	Form	kg ai/ha	kg ai/hL	L/ha	no.			Parent	Total	
(Assam Jat/ Seedling tea) S2										days
							B	5.1, 5.1 (5.1)	12, 12 (12)	248-297 days
India, 2012 Coonoor (Assam Jat/ UPASI-9) S3	WG	0.40	0.089	450	1	7	G	5.4 c:0.13	11 c:0.18	236-283 days
							B	4.0 c:0.13	12 c:0.47	231-286 days
		1.2	0.27	450	1	7	G	16, 16 (16)	34, 34 (34)	
							B	13, 14 (14)	33, 35 (34)	
India, 2013 Valparai (Assam Jat/ Seedling tea) S4	WG	0.40	0.089	450	1	7	G	2.0 c:0.012	5.5 c:0.099	236-280 days
							B	2.2 c:0.54	5.1 c:0.96	231-280 days
		1.2	0.27	450	1	7	G	4.8, 4.8 (4.8)	12, 12 (12)	
							B	4.1, 4.9 (4.5)	13, 13 (13)	
India, 2012 Valparai (Assam Jat/ Mixed seedling tea) N1	WG	0.40	0.089	450	1	7	G	16, 16 (16) c:0.77, 0.90	22, 23 (23) c:0.91, 1.1	56-100 days
							B	15, 15 (15) c:0.33, 0.51	27, 29 (28) c:0.70, 0.86	62-103 days
India, 2011 Coonoor (Assam Jat/ UPASI-9) N2	WG	0.40	0.089	450	1	7	G	2.8 c:0.12	3.0 c:0.21	64-107 days
							B	1.8	2.7	70-110 days
India, 2010 Meppadi (Assam Jat/ Seedling tea) N3	WG	0.40	0.089	450	1	7	G	1.0 c:0.056	2.9 c:0.14	68-111 days
							B	0.90	2.7	72-112 days
India, 2011 Gudalur (Assam Jat/ TRI-2024) N4	WG	0.40	0.089	450	1	7	G	5.2 c:0.031	7.3 c:0.077	65-108 days
							B	2.7 c:0.070	5.1 c:0.19	71-111 days

* Average in parentheses

[a] commodity, G: green tea, B: black tea

c: control sample

Legume animal feeds

Soya bean fodder and forage (green)

Twenty-one field residue trials were carried out with imidacloprid in soya beans in the USA and Canada using the 480 g/L SC formulation (Mackie, 2006: RANTY002). Soya bean seeds were treated at a rate of 0.125 kg ai/100 kg seed. The growing soya bean plants were subsequently treated with three foliar applications at a target rate of 0.053 kg ai/ha for a total seasonal application of 0.16 kg ai/ha. Sample materials were forage and hay. In each of the 21 residue trials, the treated plot was divided in two sub-plots A and B; from sub-plot A, forage and hay was harvested, and from sub-

plot B, soya bean seeds. The application intervals, once foliar treatment was initiated, generally ranged between 5 and 7 days.

The analytical method NT-001-P04-01 (common moiety method) was used to determine the total residue of imidacloprid in soya bean forage and hay.

Table 23 Imidacloprid residues on soya bean forage and hay from supervised trials in USA and Canada

Soya bean forage/hay country, year (variety)	Application				DALA Days	Portion analysed	Residues, mg/kg *	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.				
GAP, USA	*SC*	*63-125 g ai/100 kg seed*		*1*	*0*			
		0.053		*3*				
		Max 0.16 kg ai/ha /year						
USA, 2004 Tifton/GA (DK 5386) NT001-04D	SC	0.067	53.8 kg/ha	1	0	Forage	3.5, 4.2 (3.9)	RANTY002 Mackie, 2006 Sampling to analysis: max 325 days for forage, max 336 days for hay
		0.053	140	3	1		1.7, 1.8 (1.7)	
		0.053	143		3		1.4, 1.5 (1.5)	
		0.053	138		7		0.89, 1.1 (1.0)	
					10		0.86, 0.90 (0.88)	
					0	Hay	9.2, 9.7 (9.4)	
					1		4.3, 4.4 (4.4)	
					3		4.1, 5.1 (4.6)	
					7		3.2, 4.0 (3.6)	
					10		1.9, 2.2 (2.0)	
USA, 2004 Bumpass/VA (Pioneer 9492RR) NT002-04H	SC	0.082	65.9 kg/ha	1	0	Forage	1.5, 1.8 (1.6)	
		0.055	135	3				
		0.054	134			Hay	8.0, 11 (9.6)	
		0.055	135					
USA, 2004 Leland/MS (Pioneer 9492RR) NT003-04H	SC	0.064	51.0 kg/ha	1	0	Forage	4.2, 4.6 (4.4)	
		0.053	151	3				
		0.056	147			Hay	17, 24 (21)	
		0.053	148					
USA, 2004 Proctor/AR (DK 5386) NT004-04H	SC	0.098	78.5 kg/ha	1	0	Forage	2.5, 3.0 (2.7)	
		0.055	42	3				
		0.052	136			Hay	5.4, 6.0 (5.7)	
		0.052	140					
USA, 2004 Newport/AR (DK 5386) NT005-04H	SC	0.087	69.4 kg/ha	1	0	Forage	3.5, 4.2 (3.8)	
		0.054	186	3				
		0.053	185			Hay	21, 21 (21)	
		0.055	190					
USA, 2004 Stilwell/KS (Pioneer 93B68) NT006-04D	SC	0.11	86.1 kg/ha	1	0	Forage	1.1, 1.2 (1.1)	
		0.056	149	3	1		0.79, 1.4 (1.1)	
		0.056	143		3		0.90, 0.94 (0.92)	
		0.051	134		7		0.62, 0.78 (0.70)	
					10		0.61, 0.78 (0.69)	
					0	Hay	3.5, 4.3 (3.9)	
					1		3.0, 3.6 (3.3)	
					3		3.4, 4.7 (4.0)	
					7		1.7, 2.0 (1.8)	
					10		2.8, 3.0 (2.9)	
USA, 2004 Seymour/IL (Pioneer 93B68) NT007-04H	SC	0.089	71.1 kg/ha	1	0	Forage	2.3, 2.8 (2.6)	
		0.055	129	3				
		0.055	128			Hay	9.0, 9.4 (9.2)	
		0.054	128					
USA, 2004 Springfield/NE (S2802-4) NT008-04H	SC	0.093	74.2 kg/ha	1	0	Forage	1.9, 2.3 (2.1)	
		0.053	134	3				
		0.053	135			Hay	6.1, 6.9 (6.5)	
		0.053	131					
USA, 2005 Sabin/MN (Northrup King) NT009-04HA	SC	0.096	76.9 kg/ha	1	0	Forage	1.7, 1.9 (1.8)	
		0.055	163	3				
		0.053	154			Hay	3.8, 5.3 (4.5)	
		0.053	149					

Soya bean forage/hay country, year (variety)	Application				DALA Days	Portion analysed	Residues, mg/kg *	Ref
	Form	kg ai/ha	Seeding density water, L/ha	no.				
USA, 2004 Carlock/IL (S2802-4) NT010-04H	SC	0.092 0.055 0.053 0.054	74.0 kg/ha 121 118 119	1 3	0	Forage Hay	3.2, 3.3 (3.2) 12, 15 (14)	
USA, 2004 Bagley/IA (S2802-4) NT011-04H	SC	0.15 0.051 0.052 0.052	122 kg/ha 106 155 156	1 3	0	Forage Hay	1.9, 2.3 (2.1) 8.3, 10 (9.1)	
USA, 2004 Marysville/OH (S2802-4) NT012-04H	SC	0.084 0.053 0.053 0.053	67.3 kg/ha 160 141 141	1 3	0	Forage Hay	4.1, 8.9 (6.5) 5.8, 16 (11)	
USA, 2004 Dumfries/MN (Pioneer 92B13) NT013-04H	SC	0.090 0.053 0.054 0.053	71.6 kg/ha 174 176 175	1 3	0	Forage Hay	2.3, 2.5 (2.4) 15, 15 (15)	
USA, 2004 Northwood/ND (S02-G2) NT014-04H	SC	0.071 0.052 0.052 0.052	57.2 kg/ha 140 140 139	1 3	0	Forage Hay	3.7, 4.5 (4.1) 8.2, 8.9 (8.5)	
USA, 2004 Gardner/ND (Pioneer 90B51) NT015-04H	SC	0.092 0.054 0.053 0.054	73.8 kg/ha 200 171 172	1 3	0	Forage Hay	4.3, 4.8 (4.6) 21, 24 (22)	
USA, 2004 New Holland/OH (Pioneer 93B68) NT016-04H	SC	0.094 0.054 0.054 0.053	74.9 kg/ha 145 145 146	1 3	0	Forage Hay	3.2, 3.8 (3.5) 5.7, 9.3 (7.5)	
USA, 2004 Kirksville/MO (Pioneer 93B68) NT017-04H	SC	0.085 0.051 0.052 0.053	68.3 kg/ha 149 169 173	1 3	0	Forage Hay	3.0, 4.0 (3.5) 11, 14 (13)	
USA, 2004 Ellendale/MN (Pioneer 92B13) NT018-04H	SC	0.11 0.054 0.054 0.053	83.9 kg/ha 152 147 146	1 3	0	Forage Hay	3.3, 4.3 (3.8) 9.5, 10 (9.9)	
USA, 2004 Carlyle/IL (Pioneer 93B68) NT019-04H	SC	0.087 0.054 0.052 0.054	69.3 kg/ha 145 120 150	1 3	0	Forage Hay	3.5, 5.0 (4.2) 12, 14 (13)	
USA, 2004 Rockwood/ON (S02-G2) NT020-04H	SC	0.094 0.054 0.053 0.053	75.2 kg/ha 101 108 104	1 3	0	Forage Hay	2.8, 3.2 (3.0) 15, 20 (18)	
USA, 2004 Bright/ON (Pioneer 90B51) NT021-04H	SC	0.094 0.053 0.054 0.055	75.2 kg/ha 105 113 107	1 3	0	Forage Hay	2.7, 3.4 (3.1) 14, 16 (15)	

* Average in parentheses

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting has received information on the fate of imidacloprid residues during the processing of plum, olive, soya bean seeds and tea. Processing factors have been calculated for imidacloprid residues in olive, soya bean seeds and tea.

The processing trials for cherry and peach were submitted in 2002.

Plum

The trial was conducted in the USA on plums according to the US GAP (Dorschner, 2002: 111044). The 192 g/L SC formulation was applied 5 times at the rate of 0.11 kg ai/ha with an application interval of 8-12 days. The plums were taken to a commercial drier and dried for 25.5 hours. The total residue of imidacloprid were quantified with method 102624-R1 (based on the method 00200) at an LOQ of 0.05 mg/kg.

Table 24 Imidacloprid residues in processed commodities of plum from supervised trials

country, year (variety)	Application			DALA Days	Commodity	Residues, mg/kg	
	kg ai/ha	water, L/ha	no.			Total	
						mg/kg	PF
USA, 1999 Kerman/CA (French prunes) 99-CA79	0.11	1395-1430	5	6	Fruit (RAC) Dried (prunes)	0.30, 0.41 mean 0.36 1.0, 1.1 mean 1.1	 3.1

Portion analysed: Fruit without pit and stem

Olive

Processing studies for olive were conducted in Germany to determine the concentration of residues of imidacloprid in/on olive fruits and processing products of olive (Schöning and Eberhardt, 2002: RA-3034/01, Schoening, *et al*., 2003: RA-3155/02). The SL formulation containing 200 g/L of imidacloprid was sprayed twice to olive trees with an application rate of 0.10–0.11 kg ai/ha, corresponding to a concentration of 0.013 kg ai/hL and a spray volume of 800–904 L/ha. The olives were harvested after two spray applications of the SL formulation in Spain, Italy, Greece and Portugal (RA-2034/01 and RA-2155/02). Samples in 2001 were taken from the treated and the control plots on day 6 or 7 after the last treatment, while in 2002, at day 28 or 30. The samples from the treated plots were processed. Imidacloprid and the total residue of imidacloprid were analysed according to methods 00300/E007, 00300/E010 and 00834. The LOQ was 0.01 mg/kg for imidacloprid. For the total residue of imidacloprid the LOQ was 0.2 mg/kg for press cake and 0.05 mg/kg for all other sample materials.

Preparation of washed olives, washing water, press cake, separation water and crude oil

The olives were washed in standing water, one part of the olives was weighed and stored deep-frozen. The remaining part of the washed olives was crushed into olive pulp using a cutter. After addition of NaCl (1%, w/w), the olive pulp was pressed to obtain press cake and a water/oil emulsion. A sample of press cake was taken. The water/oil emulsion was separated into crude oil and separation water using a centrifuge; both fractions were taken for analysis.

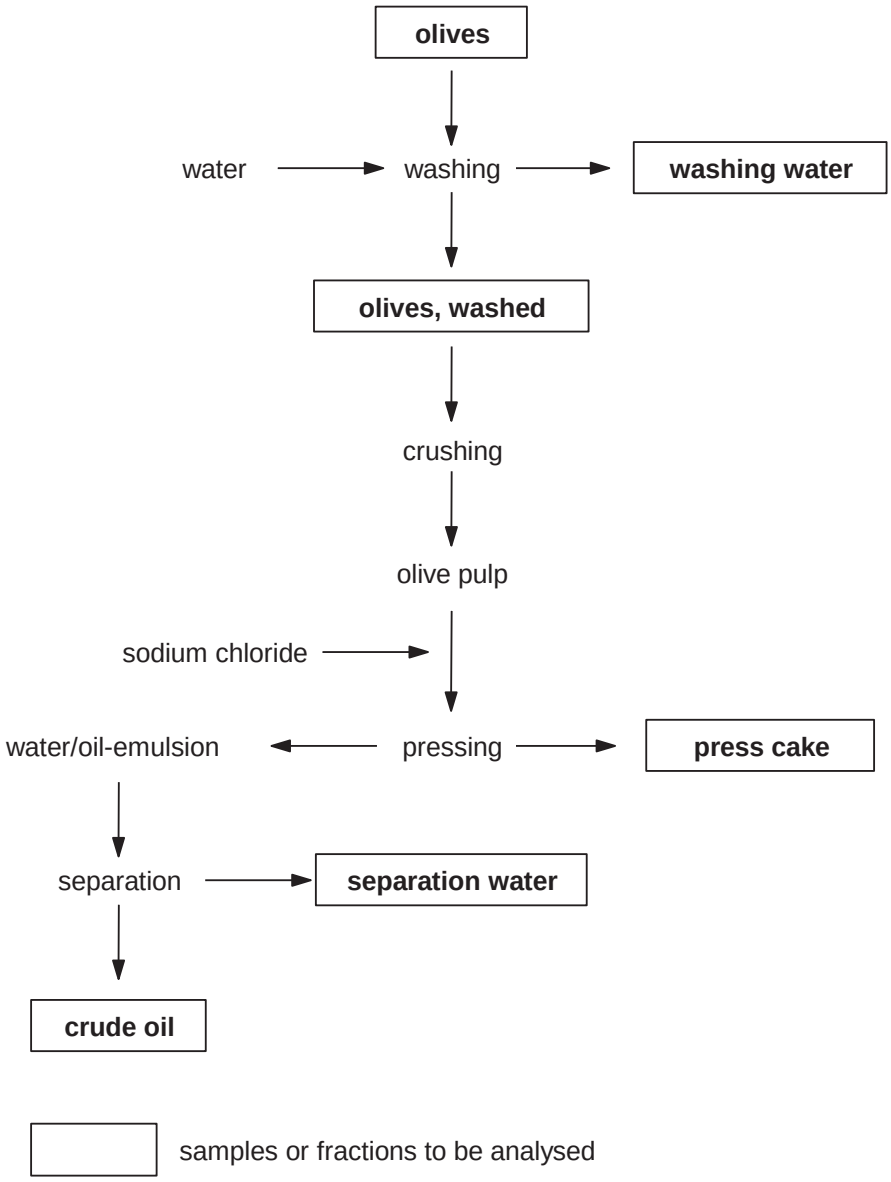

Figure 1 Flow chart of the preparation of washed olive, washing water, press cake, separation water and crude oil

Preparation of refined oil

The crude oil was preclarified by heating, removal of precipitated compounds and one part of the oil was taken for analysis. The remaining part of the preclarified oil was neutralized. After neutralisation, one part of the neutralised crude oil was taken for analysis.

The subsequent processes (bleaching, filtration and steaming) were all carried out in a vacuum. A sample of refined oil was taken for analysis

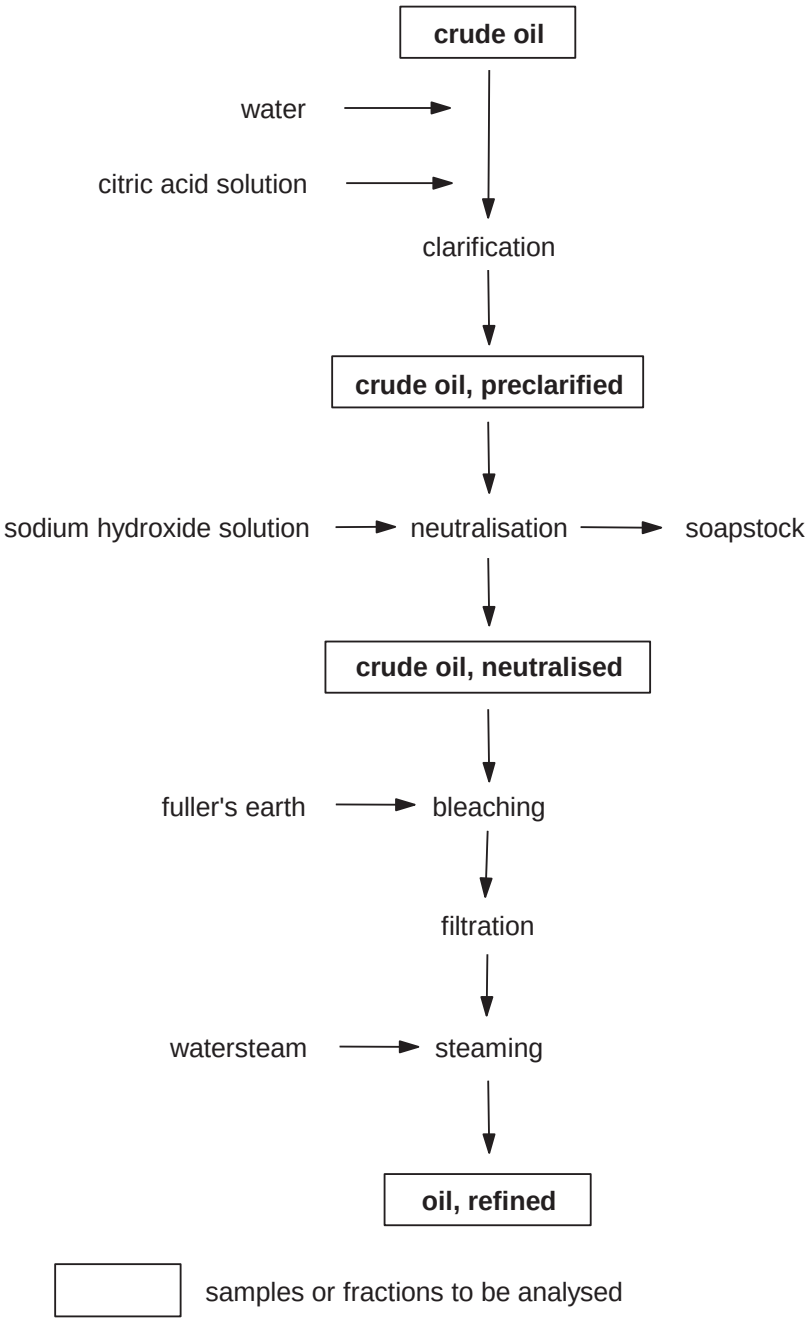

Figure 2 Flow chart of the preparation of refined oil

Table 25 Imidacloprid residues in processed commodities of olive from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg			
	kg ai/ha	kg ai/hL	water, L/ha	no.			Parent		Total	
							mg/kg	PF	mg/kg	PF
Spain, 2001 (Vera) R2001 0090/6	0.11 0.10	0.013 0.013	904 800	2	6	Fruit (RAC) Fruit, washed Press cake Crude oil Washing water Separation water	0.09 0.048 0.13 0.01 < 0.01 0.04	 0.53 1.4 0.1 < 0.1 0.44	0.26 0.18 0.21 < 0.05 < 0.05 0.13	 0.69 0.81 < 0.1 9 < 0.1 9 0.50

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg			
	kg ai/ha	kg ai/hL	water, L/ha	no.			Parent		Total	
							mg/kg	PF	mg/kg	PF
Italy, 2001 (Nocellara Etnea) R2001 0091/4	0.10 0.10	0.013 0.013	800 800	2	7	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.04 0.03 0.04 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.01	 0.75 1.0 < 0.25 < 0.25 < 0.25 < 0.25 < 0.25 0.25	0.14 0.09 < 0.2 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	 0.64 <1.4 < 0.3 6 < 0.3 6 < 0.3 6 < 0.3 6 < 0.3 6
Portugal, 2001 (Picual) R2001 0092/2	0.10 0.10	0.013 0.013	800 800	2	7	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.11 0.10 0.14 0.02 0.01 < 0.01 < 0.01 < 0.01 0.06	 0.91 1.3 0.18 0.091 < 0.09 < 0.09 < 0.09 0.55	0.22 0.19 0.24 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.08	 0.86 1.1 < 0.2 3 < 0.2 3 < 0.2 3 < 0.2 3 0.36
Portugal, 2002 (Cobrancosa) R2002 0697/6	0.10 0.10	0.013 0.013	800 800	2	28	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.02 0.03 0.04 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 0.02	 1.5 2.0 < 0.5 < 0.5 < 0.5 < 0.5 < 0.5 1.0	0.05 < 0.05 0.06 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 0.05	 <1.0 1.2 <1.0 <1.0 <1.0 <1.0 <1.0 1.0
Greece, 2002 (Megaritiki) R2002 0698/4	0.10 0.10	0.013 0.013	800 800	2	30	Fruit (RAC) Fruit, washed Press cake Crude oil Crude oil, preclarified Crude oil, neutralized Refined oil Washing water Separation water	0.18 0.14 0.18 0.03 0.02 < 0.01 < 0.01 < 0.01 0.12	 0.78 1.0 0.17 0.11 < 0.06 < 0.06 < 0.06 0.67	0.76 0.67 0.62 0.09 0.07 < 0.05 < 0.05 < 0.05 0.77	 0.88 0.82 0.12 0.092 < 0.0 7 < 0.0 7 < 0.0 7 1.0

Soya bean seeds

A field trial was conducted to measure the magnitude of imidacloprid residue on soya beans treated with three foliar applications of the SC formulation containing 480 g/L of imidacloprid at a target rate of 0.263 kg ai/ha/application with 7 days between applications. Each application was made at a concentrated spray volume, 160–170 L/ha. The treatment rate is equivalent to a 5× the maximum recommended label use rate on soya beans. Single control and treated samples of soya bean seed were collected at normal commercial harvest (BBCH 89), corresponding to a 20-day PHI. Soya bean seed was processed into the commodities of meal, hulls, refined oil, and defatted flour. Aspirated grain

fractions were also collected. Processing was performed using procedures which simulated commercial processing practices (Krolski, 2006: RANTY003).

The total imidacloprid residue was quantitated as 6-chloronicotinic acid (6-CNA) by HPLC-MS/MS using isotopically labeled internal standards (method NT-001-P04-01). Method validation and concurrent recoveries were performed to demonstrate acceptable method performance. The LOQ for the total residue of imidacloprid was 0.05 mg/kg in soya bean seed, 0.10 mg/kg in soya bean refined oil, 0.20 mg/kg in soya bean meal, hulls, and defatted flour, and 30 mg/kg in soya bean aspirated grain fractions.

Preparation of aspirated grain fractions

After determining the moisture content of soya bean (RAC), the samples were dried to a moisture content of 10–13%. The samples were then placed in a dust generation room and moved in the system. Aspiration was used to remove light impurities. The light impurities were classified by sieving.

Preparation of hull, meal, defatted flour and refined oil

Soya beans were fed to a disc mill to crack the hull and liberate the kernel. After hulling, the material was passed through an aspirator to separate hull and kernel. The kernel material was flaked and heated. After expansion, the collets were dried and promptly taken for solvent extraction. The material was washed several times with hexane. Then the defatted flakes were ground and screened to produce defatted flour and the crude oil was heated for hexane removal and afterwards alkali refined. The refined oil was bleached and deodorised.

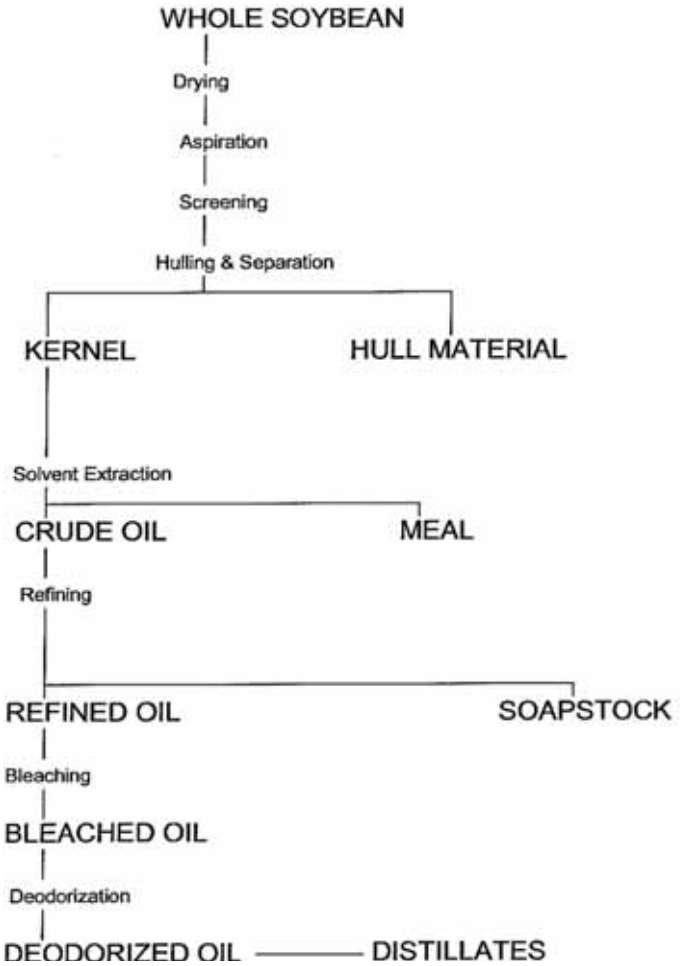

Figure 3 Flow chart for soya bean processing

Table 26 Imidacloprid residues in processed commodities of soya bean seeds from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Total imidacloprid Residues, mg/kg	
	kg ai/ha	water, L/ha	GS (BBCH)	no.			mg/kg	PF
USA, 2004	0.263	160	89	3	20	seed	0.42	
Leland/MS	0.263	170				meal	0.36	0.86
(Pioneer 9492PR)	0.263	162				hulls	0.31	0.72
NT022-04P						refined oil	< 0.10	< 0.24
						defatted flour	0.34	0.80
						aspirated grain fractions	68	160

Tea

Two processing trials were performed in southern India with the imidacloprid 700 g/kg WG formulation. The formulation was applied once at a triple rate of 1.2 kg ai/ha. Tea shoots (two to three leaves and a bud) harvested 7 days after the application were used to manufacture green and black tea. Green and black tea was further processed into infusion using household practices whereas the preparation of instant tea simulated the industrial practice (Manikandan, 2014: RANTN021).

Preparation of infusion

100 g of green or black tea were infused into 5 L of boiling water for approximately 10 min. Infusion solution (liquid part) was separated with a sieve from wastes (infused tea). Wastes were weighed and discarded.

Preparation of instant tea

Two 0.5 L infusion solution subspecimens were collected and deep-frozen (below -18 °C). The Brix degree of the infusion solution was measured. The dry matter of solution determined by its Brix degree was increased with an addition of food additive to obtain between 8 to 9%. The pH of this preparation was measured and corrected with an addition of citric acid to obtain between a pH of 3.0 to 3.2. Afterwards the solution was placed in a vacuum chamber, where the water frozen in the solution was evaporated through sublimation.

All samples were analysed for imidacloprid parent compound and its metabolites and the total residue of imidacloprid according to method 01389. The LOQ was 0.01 mg/kg for imidacloprid in processed commodities (green and black tea infusion and instant green and black tea). The LOQ of the total residue of imidacloprid was 0.05 mg/kg in all sample materials.

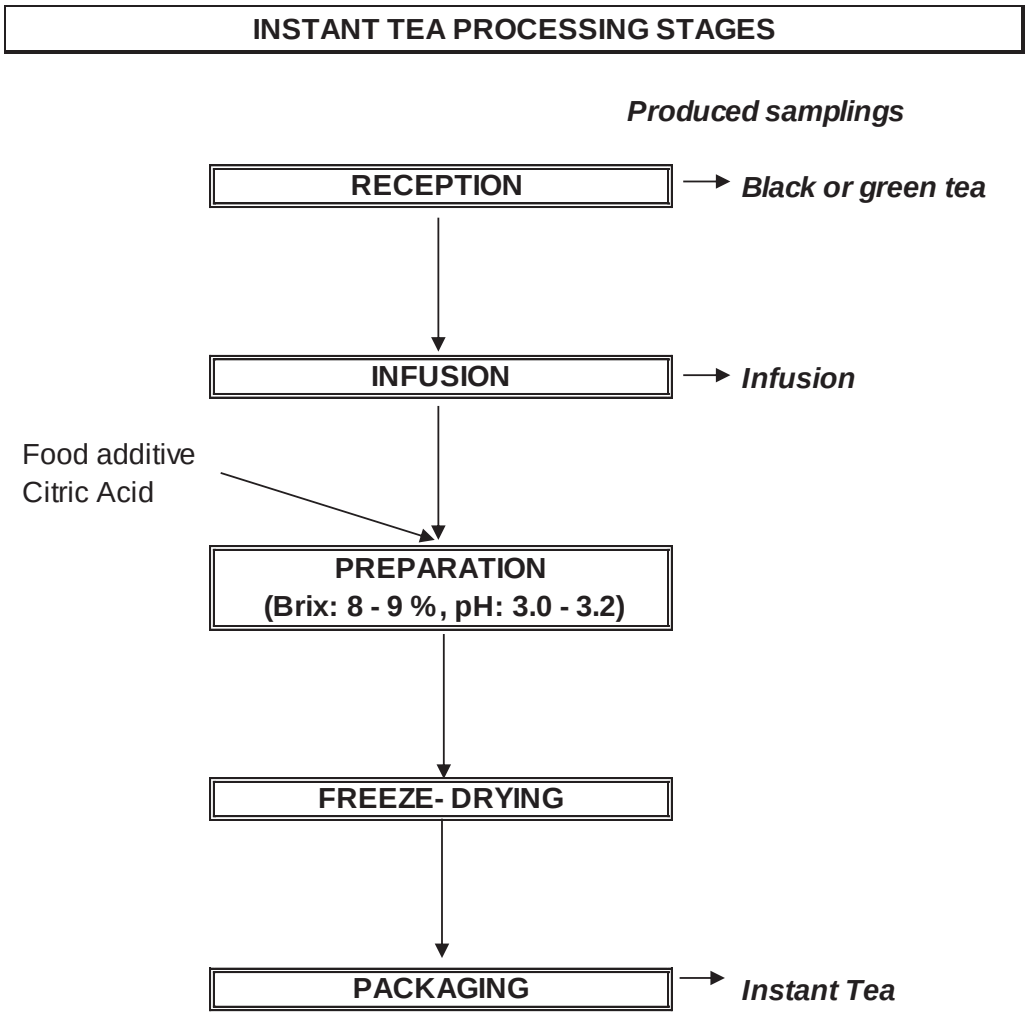

Figure 4 Flow chart for tea processing

Table 27 Imidacloprid residues in processed commodities of tea from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg			
							Parent		Total	
	kg ai/ha	kg ai/hL	L/ha	no.			mg/kg	PF	mg/kg	PF
India, 2012 Coonoor (Assam Jat/ UPASI-9) S3	1.2	0.27	450	1	7	Green tea	16		34	
						Infusion green tea	0.41	0.026	0.81	0.024
						Instant green tea	3.6	0.23	8.0	0.24
						Black tea	14		34	
						Infusion black tea	0.34	0.024	0.57	0.017
						Instant black tea	3.0	0.21	6.4	0.19
India, 2013 Valparai (Assam Jat/ Seedling tea) S4	1.2	0.27	450	1	7	Green tea	4.8		12	
						Infusion green tea	0.12	0.025	0.30	0.025
						Instant green tea	1.1	0.23	3.0	0.25
						Black tea	4.5		13	
						Infusion black tea	0.13	0.029	0.30	0.023
						Instant black tea	1.3	0.29	3.6	0.28

APPRAISAL

Imidacloprid is a systemic insecticide which has been used widely in many crops for years. It was first evaluated by JMPR in 2001 (T) and 2002 (R). An ADI of 0–0.06 mg/kg bw and an ARfD of

0.4 mg/kg bw were established. The compound was evaluated for residues in 2006, 2008 and 2012. In 2002 the Meeting agreed that the residue definition for compliance with MRLs and for estimation of dietary intake for plant and animal commodities should be the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid. It was listed by the Forty-sixth Session of CCPR (2014) for the evaluation of 2015 JMPR for additional MRLs.

The residue studies were submitted by the manufacturer and member countries for additional MRLs for stone fruit, olive, curly kale, soya bean, tea, goji berry (China) and basil (Thailand).

Methods of analysis

The Meeting received information on analytical methods used for the determination of imidacloprid residues in samples derived from supervised trials on olive, kale and soya bean (dry). Samples were fortified with imidacloprid and its metabolites desnitro-imidacloprid and 6-chloronicotinic acid. Imadacloprid and all metabolites containing 6-chloropyridinyl moiety were oxidised with alkaline $KMnO_4$ to yield 6-chloronicotinic acid. The 6-chloronicotinic acid was extracted from the aqueous solution using *tert*-butylmethylether (MTBE) and analysed by HPLC-MS/MS. The LOQ was 0.05 mg/kg (expressed in parent equivalents) for the commodities mentioned above.

The analytical method was developed for the determination of residues of imidacloprid, its 2 metabolites 5-hydroxy imidacloprid and olefin imidacloprid, and for the total residue of imidacloprid determined as 6-chloronicotinic acid in tea. Imidacloprid and its metabolites were extracted from tea (green tea and black tea) with methanol/water (3/1, v/v). For the individual analytes, an aliquot of the extracts was cleaned-up with liquid/liquid SPE. For the common moiety analysis, an aliquot of the extracts was made by alkaline oxidation under reflux and liquid/liquid partition. Final extracts of both branches were subjected to reversed phase HPLC-MS/MS. The LOQ (expressed as imidacloprid equivalents) for the total residue of imidacloprid was 0.05 mg/kg.

The Meeting received information on the analytical method for the determination of imidacloprid residues in fresh and dried goji berries. Imidacloprid was extracted from goji berries with acetonitrile. After adding sodium chloride, an aliquot was concentrated and purified by solid phase extraction using amino cartridges. Imidacloprid residues were analysed by reversed-phase HPLC-UV (275 nm). The LOQ was 0.02 mg/kg for both matrices.

The Meeting received data on the storage stability of imidacloprid, 5-hydroxy imidacloprid and olefin imidacloprid in various plant matrices. Storage stability results indicated that residues of imidacloprid and its metabolites 5-hydroxy imidacloprid and olefin imidacloprid were stable for at least 36 months under freezer conditions at about -18 °C or below in wheat (grain), orange (fruit), tomato (fruit), bean (seed) and rape seed.

Residues resulting from supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of imidacloprid on cherries, plum, peach, olive, kale, goji berry, soya bean, basil and tea. Residue trial data was made available from Canada, China, India, Southern Europe, Thailand and the USA.

Labels were available from China, Italy, Japan, Spain, Thailand and the USA describing the registered uses of imidacloprid.

Stone fruits

The 2002 JMPR evaluated residue supervised trials data for imidacloprid on sweet cherries, plums, peaches and nectarines conducted in southern Europe. New residue data were submitted to the current Meeting for cherries, plums and peaches.

Cherries

Data were available from supervised trials on cherries in the USA.

The GAP of the USA is foliar applications of 0.056-0.11 kg ai/ha at a maximum rate of 0.56 kg ai/ha per year with a PHI of 7 days.

Imidacloprid residues in whole fruits of cherries from independent trials in the USA matching GAP were (n=8): 0.24, 0.36, 0.41, 0.53, 0.57, 0.63, 1.4 and 2.5 mg/kg.

Based on the residues for cherries from trials in the USA, the Meeting estimated a maximum residue level of 4 mg/kg, an STMR value of 0.55 mg/kg and an HR value of 2.5 mg/kg for the cherries subgroup. The Meeting withdrew the previous recommendation for Cherry, Sweet.

Plums

Data were available from supervised trials on plums in the USA.

The GAP of the USA is foliar applications of 0.056–0.11 kg ai/ha at a maximum rate of 0.56 kg ai/ha per year with a PHI of 7 days.

Imidacloprid residues in fruits without stone of plums from independent trials in the USA matching GAP were (n=8): 0.082, 0.095, 0.15, 0.22, 0.34, 0.39, 0.42 and 0.67 mg/kg.

Since the weight of stone does not significantly affect the residue level in plum fruits, the Meeting agreed to use the residues in the edible portion of plums to estimate a maximum residue level.

Based on the residues in the edible portion of plums from trials in the USA, the Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR value of 0.28 mg/kg and an HR value of 0.70 mg/kg (based on a highest residue of duplicate samples) for imidacloprid in the plums (including prunes) subgroup, to replace the previous recommendation for plums (including prunes).

Peaches

Data were available from supervised trials on peaches in the USA.

The GAP in the USA is foliar applications of 0.056-0.11 kg ai/ha at a maximum rate of 0.34 kg ai/ha per year with a PHI of 0 days.

Imidacloprid residues in whole fruit peaches from trials in the USA, matching GAP, were (n=8): 0.10, 0.25, 0.28, 0.34, 0.37, 0.38 (2) and 0.77 mg/kg.

Based on the residues for peaches from trials in the USA, the Meeting estimated a maximum residue level of 1.5 mg/kg, an STMR value of 0.355 mg/kg and an HR value of 0.77 mg/kg for imidacloprid in the Peaches (including nectarine and apricots) subgroup. The Meeting withdrew the previous recommendations for peach, nectarine and apricot.

Olives

Data were available from supervised trials on olives from Southern Europe.

The GAP of Italy is for a foliar application at a maximum concentration of 0.013 kg ai/hL, with a PHI of 28 days. Imidacloprid residues in olives, from trials in Southern Europe matching GAP, were (n=8): 0.12, 0.23, 0.26, 0.28, 0.43, 0.61, 0.77 and 0.81 mg/kg.

The GAP of Spain is a maximum of four foliar applications at a maximum rate of 0.02 kg ai/ha with a PHI of 7 days. Imidacloprid residues in olive from independent trials in Southern Europe matching GAP were (n=8): < 0.05, 0.11, 0.14, 0.22, 0.49, 0.63, 0.71 and 1.1 mg/kg.

Based on the residues for olive from trials with the highest residue levels matching Spanish GAP, the Meeting estimated a maximum residue level of 2 mg/kg, an STMR value of 0.355 mg/kg and an HR value of 1.1 mg/kg for imidacloprid in olives.

Kale

Data were available from supervised trials on curly kale in Italy and Spain.

The GAP of Italy is a maximum two foliar applications at a maximum rate of 0.094 kg ai/ha with a PHI of 7 days.

Imidacloprid residues in curly kale from independent trials in Italy and Spain matching GAP were (n=4): 1.0, 1.1, 1.5 and 2.0mg/kg.

Based on the residues for curly kale from trials in Italy and Spain, the Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 1.3 mg/kg and an HR value of 2.0 mg/kg for imidacloprid in kale.

Goji berry

The GAP of China is a maximum three foliar applications at a maximum concentration of 0.005 kg ai/hL with a PHI of 3 days. Six trials were conducted on goji berries in China in 2010 with foliar treatment by 3 × 0.005 kg ai/hL. Samples were taken at 1–21 days after the last treatment. The data were submitted as separate trials but the analyte was parent imidacloprid only.

As the residue definition of imidacloprid is the sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid, the Meeting could not estimate a maximum residue level for imidacloprid in goji berry.

Soya bean (dry)

Data were available from supervised trials on soya bean in the USA.

The GAP on soya bean of the USA is seed treatment at a maximum rate of 0.125 kg ai/100 kg seed, and/or maximum three foliar applications at a maximum rate of 0.053 kg ai/ha with a PHI of 21 days.

Imidacloprid residues in soya bean seeds from independent trials in the USA matching GAP were (n=20): 0.035, 0.050, 0.052 (2), 0.094, 0.11, 0.18, 0.19, 0.21, 0.38 (2), 0.43, 0.48, 0.61, 0.62, 0.63, 0.67, 0.73 and 1.5 (2) mg/kg.

Based on the residues for soya bean from trials in the USA, the Meeting estimated a maximum residue level of 3 mg/kg and an STMR value of 0.38 mg/kg for imidacloprid in soya bean seed (dry).

Basil

Data were available from supervised trials on basil in Thailand.

The GAP of Thailand is foliar applications when the crop is infested at a maximum concentration of 0.042 kg ai/hL with a PHI of 7 days.

Imidacloprid residues in fresh basil from independent trials in Thailand matching GAP were (n=4): 4.3, 4.9, 5.1 and 6.5 mg/kg.

Based on the residues for basil from trials in Thailand, the Meeting estimated a maximum residue level of 20 mg/kg, an STMR value of 5.0 mg/kg and an HR value of 7.3 mg/kg (based on a highest residue of replicate samples) for imidacloprid in basil.

Tea, Green, Black

Data were available from supervised trials on tea in India.

The GAP on tea of Japan is a foliar application at a maximum concentration of 0.01 kg ai/hL with a PHI of 7 days.

Imidacloprid residues in green tea from independent trials in India matching Japanese GAP were (n=8): 2.9 (2), 3.0, 5.5, 7.3, 11, 12 and 23 mg/kg.

Imidacloprid residues in black tea from independent trials in India matching Japanese GAP were (n=8): 2.7 (2), 3.3, 5.1 (2), 12 (2) and 28 mg/kg.

The samples of green tea and black tea were produced from fresh tea leaves harvested 7 days after application at the same plot.

The Meeting recognized that the residue populations from trials on green tea and black tea were not different according to statistical tests (Mann-Whitney U-test). The Meeting agreed to use highest residues of green tea and black tea samples in each trial to estimate a maximum residue level for tea, green and black.

The residues in green tea and black tea were in rank order (n=8): 2.9, 3.0, 3.3, 5.5, 7.3, 12 (2) and 28 mg/kg.

Based on the residues for green tea and black tea from trials in India, the Meeting estimated a maximum residue level of 50 mg/kg and an STMR value of 6.4 mg/kg for imidacloprid in tea, green and black.

Animal feedstuffs

Soya bean fodder and forage (green)

Data were available from supervised trials on soya bean in the USA.

The GAP on soya bean in the USA isa seed treatment at a maximum rate of 0.125 kg ai/100 kg seed, and/or maximum three foliar applications at a maximum rate of 0.053 kg ai/ha for forage grass for hay.

Imidacloprid residues in soya bean forage from independent trials in the USA matching GAP were (n=21): 1.1, 1.6, 1.8, 2.1 (2), 2.4, 2.6, 2.7, 3.0, 3.1, 3.2, 3.5 (2), 3.8 (2), 3.9, 4.1, 4.2, 4.4, 4.6 and 6.5 mg/kg.

Based on the trials for soya bean forage from trials in the USA, the Meeting estimated a median residue value and a highest residue value for imidacloprid in soya bean forage of 3.2 and 6.5 mg/kg, respectively as received basis.

Imidacloprid residues in soya bean hay from independent trials in the USA matching GAP were (n=21): 4.0, 4.5, 5.7, 6.5, 7.5, 8.5, 9.1, 9.2, 9.4, 9.6, 9.9, 11, 13 (2), 14, 15 (2), 18, 21 (2) and 22 mg/kg.

Based on the residues in soya bean hay from trials in the USA, the Meeting estimated a median residue value of 9.9 mg/kg, a highest residue value of 22 mg/kg on an as received basis and after correction for an average 85% dry matter content, estimated a maximum residue level of 50 mg/kg for imidacloprid in soya bean hay.

Fate of residues during processing

Residues in processed commodities

The fate of imidacloprid residues has been examined in plum, olive, soya bean seeds and tea processing studies. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P for food and feed

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)	RAC HR (mg/kg)	HR-P (mg/kg)
Cherry	Canned fruit	< 0.56, < 0.56, < 0.63 < 0.63	< 0.60	0.55	< 0.33	2.5	<1.5
Plum	Dried (prunes)	3.1	3.1	0.28	0.87	0.70	2.2
Peach	Canned fruit	< 0.38	< 0.38	0.32	< 0.12	0.77	< 0.092
	Jam	< 0.38	< 0.38		< 0.12		
Olive	Crude oil	< 0.19, < 0.36, < 0.23, < 1.0, 0.12	0.12	0.36	0.04		
Soya bean seeds	Refined oil	< 0.24	< 0.24	0.38	< 0.09		

Raw agricultural commodity (RAC)	Processed commodity	Calculated processing factors*	PF (Mean or best estimate)	RAC STMR (mg/kg)	STMR-P (mg/kg)	RAC HR (mg/kg)	HR-P (mg/kg)
	Meal	0.86	0.86		0.33		
	Aspirated grain fractions	160	160		61		
	Hulls	0.72	0.72		0.27		
Green tea	Infusion	0.024, 0.025	0.025	6.4	0.16		
	Instant	0.24, 0.25	0.25		1.6		
Black tea	Infusion	0.017, 0.023	0.02	6.4	0.13		
	Instant	0.19, 0.28	0.24		1.5		

* Each value represents a separate study. The factor is the ratio of the residue in processed commodity divided by the residue in the RAC.

The Meeting estimated a maximum residue level of 5 mg/kg (1.5 × 3.1 = 4.65 mg/kg) for dried plums.

Residue in animal commodities

The 2015 JMPR evaluated residues of imidacloprid in soya bean (dry), which is listed in the OECD feeding table. The Meeting noted that the estimation did not result in a significant change of the dietary burdens of farm animals. The previous recommendations of maximum residue level for animal commodities were maintained.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed in Annex 1 are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Definition of the residue for plant and animal commodities (for compliance with the MRL and for estimation of dietary intake): *Sum of imidacloprid and its metabolites containing the 6-chloropyridinyl moiety, expressed as imidacloprid*

CCN	Commodity	Recommended Maximum residue level (mg/kg)		Recommended Maximum residue level (mg/kg)	HR or HR-P mg/kg
		New	Previous		
FS 0240	Apricot	W	0.5		
HH 0722	Basil	20		5.0	7.3
FS 0013	Cherries	4		0.55	2.5
FS 0244	Cherry, Sweet	W	0.5		
DF 0014	Prunes	5		0.87	2.2
VL 0480	Kale	5		1.3	2.0
FS 0247	Nectarine	W	0.5		
SO 0305	Olives for oil production	2		0.355	1.1
FS 0247	Peach	W	0.5		
FS 2001	Peaches (including nectarines and apricots)	1.5		0.355	0.77
FS 0014	Plums (including Prunes)	1.5	0.2	0.28	0.7
VD 0541	Soya bean (dry)	3		0.38	
AL 0541	Soya bean fodder	50		9.9	22
FT 0305	Table olives	2		0.355	1.1
DT 1114	Tea, Green, Black (black, fermented and dried)	50		6.4	
	Apricot, canned			0.12	0.092
	Apricot jam			0.12	
	Cherries, canned			0.33	1.5
	Nectarine, canned			0.12	0.092

CCN	Commodity	Recommended Maximum residue level (mg/kg)		Recommended Maximum residue level (mg/kg)	HR or HR-P mg/kg
		New	Previous		
	Nectarine, jam			0.12	
OC 0305	Olive oil, virgin oil			0.04	
	Peaches, canned			0.12	0.092
	Peaches, jam			0.12	
OR 0541	Soya bean oil, refined			0.09	
	Tea, infusion			0.16	
	Tea instant			1.6	
AL 1265	Soya bean forage (green)			3.2	6.5
	Soya bean asp gr fn[a]			61	
AB 0541	Soya bean hulls			0.27	
AB 1265	Soya bean meal			0.33	

[a] aspirated grain fractions

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of imidacloprid were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the 2002, 2006, 2008, 2012 and current Meeting (Annex 3). The ADI is 0–0.06 mg/kg bw and the calculated IEDIs were 2–5% of the maximum ADI (0.06 mg/kg bw). The Meeting concluded that the long-term intake of residues of imidacloprid, resulting from the uses considered by the current JMPR, were unlikely to present a public health concern.

Short-term intake

The International Estimated Short-Term Intakes (IESTI) of imidacloprid were calculated for food commodities and their processed commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting (Annex 4). The ARfD is 0.4 mg/kg bw and the calculated IESTIs were a maximum of 10% of the ARfD. The Meeting concluded that the short-term intake of residues of imidacloprid, when used in ways that have been considered by the JMPR, is unlikely to present a public health concern.

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