5.20 IMIDACLOPRID (206)

RESIDUE AND ANALYTICAL ASPECTS

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 analyzed 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		

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)
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		
	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*

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.