

IMAZAPYR (267)

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EXPLANATION

Imazapyr is a broad-spectrum herbicide in the imidazolinone family. It was first evaluated by JMPR in 2013 (T, R). An ADI of 0–3 mg/kg bw was established and an ARfD was unnecessary. The compound was evaluated for residues in 2015. In 2013 the Meeting agreed that the definition of the residue was imazapyr for plant and animal commodities (for compliance with MRLs and for estimation of dietary intake). It was listed by the 48th Session of CCPR (2016) for the evaluation of 2017 JMPR for additional MRLs.

The current Meeting received information on analytical method, use patterns and supervised residue trials to support estimation of a maximum residue level for barley.

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on the analytical method (Method M3098) for the determination of related compound of imazapyr (imazamox and CL 263284) in wheat forage, hay, straw and grain (Xu and Nejad, 1999: ID-244-026). This method was evaluated at the 2014 JMPR.

This method was modified by the performing laboratory to allow simultaneous analysis of imazapyr and imazamox residues and replaced the method of determination from capillary electrophoresis to LC-MS/MS. These changes were validated within the residue study by analyzing untreated control specimens fortified with known amounts of imazapyr and imazamox and determining the percent recovery.

Concurrent recovery results for the method performance on imazapyr in barley from the supervised residue study (Allen, 2009: 2009/1130682) were summarized in Table 1. The method is suitable for the analysis of imazapyr residues in barley forage, straw and grain.

Table 1 Concurrent recovery results of method M3098 for the determination of residues of imazapyr in barley

Commodity	Fortification level (mg/kg)	N	Mean recovery (%)	% RSD
Barley, forage	0.01–2.0	6	88.8	4.2
Barley, grain	0.01–2.0	6	92.4	6.4
Barley, straw	0.01–2.0	6	100.4	5.3

USE PATTERN

The Meeting received labels in Australia. The authorized uses relevant to the supervised residue trials data submitted to the current Meeting are summarized in Table 2.

Table 2 Registered uses of imazapyr relevant to the residue evaluation by the current Meeting

Crop	Country	Formulation		Application				PHI, days
		Type	Conc. of imazapyr	Method	Rate kg ai/ha	Volume L/ha	No. max	
Barley (imidazolinone-tolerant)	Australia	SL	15 g/L	Foliar spray	0.0056-0.011	Min. 70	1	^a ^b

^a Apply to the crops from the 5 leaf stage to 1st node stage. Do not apply after the 1st node stage (Z31).

^b Do not graze or cut for forage and fodder for 4 weeks after application.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

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

Group	Commodity	Table
Cereal grains	Barley	Table 4, 5
Straw, fodder and forage of cereal grains and grasses	Barley forage and straw	Table 6, 7

Imazapyr formulation was applied for foliar treatment. Each of the field trial sites generally consisted of untreated control plot and treated plot. 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.

Cereal grains

Barley

Two field trials on imidazolinone-tolerant barley were conducted in Australia during the 2008 growing season. The SL formulation was applied once as broadcast foliar application when the crop was at the 1-2 node growth stage. A small plot harvester was used to collect the grain. Grain samples were collected at typical harvest stage (59 and 68 days after application). The samples were placed into labelled plastic bags, double bagged and placed into freezers.

The method M 3098 was used for analysis of imazapyr residues in barley grain samples with LC-MS/MS. Average recovery of imazapyr in barley grain was shown in Table 1. The LOQ was 0.01 mg/kg.

Table 3 Imazapyr residues on imidazolinone-tolerant barley grain from supervised trials in Australia (Allen, 2009: 2009/1130682)

Barley country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth Stage	no.			
<i>GAP, Australia</i>	SL	<u>0.0056- 0.011</u>	min. 70	the 5 leaf stage to 1st node stage	1			
Australia, 2008 Murchison/ VIC (Clearfield)	SL	0.011	100	1-2 nodes	1	68	<u>< 0.01</u>	Sampling to analysis: 101 days
		0.022	100	1-2 nodes	1	68	< 0.01	
Australia, 2008 Roseworthy/ SA (Clearfield)	SL	0.011	100	First node	1	59	<u>< 0.01</u>	Sampling to analysis: 122 days
		0.022	100	First node	1	59	< 0.01	

Four field trials on imidazolinone-tolerant barley were conducted in different growing areas in Australia during the 2015 and 2016 growing season to determine the residue level of imazapyr

in/on barley grain. The SL formulation was applied once when the crop was between end of tillering to first node stage (29–32 BBCH). Grain samples were collected at typical harvest stage, 84–140 days after application.

The Method M 2468 was developed for the analysis of imazapyr residues in maize grains. For the analysis of barley grain samples, this method was modified utilizing LC-MS/MS instead of GC-MS because of the greater sensitivity and shorter analysis time. Due to the increased sensitivity, the extra SPE clean-up was not required. Average recoveries of imazapyr were $101 \pm 12\%$ ($n=5$, 0.02 mg/kg fortification level), $107 \pm 3.2\%$ ($n=5$, 0.2 mg/kg fortification level) and 96% ($n=1$, 1 mg/kg fortification level). The LOQ was 0.02 mg/kg.

Table 4 Imazapyr residues on imidazolinone-tolerant barley grain from supervised trials in Australia (Bower, 2016: 2016/8000282)

Barley country, year (variety)	Application					DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	water, L/ha	Growth Stage	no.		as received	as dry weight	
<i>GAP, Australia</i>	SL	0.0056-0.011	min. 70	the 5 leaf stage to 1st node stage	1				
Australia, 2015-2016 Forth/TAS (Scope)	SL	0.011	113	BBCH 31-32	1	84	<u>0.19</u>	Sampling to analysis: 16 days	
		0.023	111	BBCH 31-32	1	84	0.48		
Australia, 2015 Balliang/VIC (Scope)	SL	0.011	99	BBCH 29-31	1	99	<u>0.16</u>	Sampling to analysis: 42 days	
		0.023	101	BBCH 29-31	1	99	0.46		
Australia, 2015 Wasleys/SA (Scope)	SL	0.011	101	BBCH 31	1	88	<u>0.25</u>	Sampling to analysis: 30 days	
		0.023	101	BBCH 31	1	88	0.80		
Australia, 2015 Williams/WA (Scope)	SL	0.011	100	BBCH 30	1	140	<u>0.32</u>	Sampling to analysis: 28 days	
		0.023	100	BBCH 30	1	140	0.84		

Straw, fodder and forage of grasses

Two field trials on imidazolinone-tolerant barley were conducted in Australia during the 2008 growing season. The SL formulation was applied once as broadcast foliar application when the crop was at the 1–2 node growth stage. Forage plants were selected at random from across the plots and cut off at ground level. Samples of forage were collected at 0, 28, 35 and 42 or 49 days after application, from one/two nodes to soft dough stage. A small plot harvester was used to collect the straw specimens. Straw samples were collected at typical harvest stage (59 and 68 days after application). All samples were placed into labelled plastic bags, double bagged and placed into freezers.

The method M 3098 was used for analysis of imazapyr residues in barley forage and straw samples with LC-MS/MS. Average recoveries of imazapyr in barley forage and straw were shown in Table 1. The LOQ was 0.01 mg/kg.

Table 5 Imazapyr residues on imidazolinone-tolerant barley forage and straw from supervised trials in Australia (Allen, 2009: 2009/1130682)

Barley country, year (variety)	Application				Growth Stage at harvest	Portion analysed	DALA Days	Residues, mg/kg		Ref
	Form	kg ai/ha	water, L/ha	no.				as received	as dry weight	
<i>GAP, Australia</i>	SL	0.0056-0.011	min. 70	1	<i>Do not graze or cut for forage and fodder for 4 weeks after application</i>					
Australia, 2008 Murchison/VIC	SL	0.011	100	1	1-2 node	Forage	-0	< 0.01	< 0.01	Sampling to analysis: 101-169
					1-2 node		0	0.32	2.7	
					Milky dough		28	<u>0.04</u>	0.31	
					Ealy dough		35	0.01	0.06	

Barley country, year (variety)	Application				Growth Stage at harvest	Portion analysed	DALA Days	Residues, mg/kg		Ref	
	Form	kg ai/ha	water, L/ha	no.				as received	as dry weight		
(Clearfield)					Soft dough		49	< 0.01	< 0.01	days	
					Grain ripe	Straw	68	< 0.01	< 0.01		
					1-2 node	Forage	-0	< 0.01	< 0.01		
					1-2 node			0	0.97		8.8
					Milky dough			28	0.19		1.5
					Ealy dough			35	0.06		0.40
					Soft dough			49	0.05		0.32
Grain ripe	Straw	68	0.01	0.01							
Australia, 2008 Roseworthy/ SA (Clearfield)	SL	0.011	100	1	Z39 ^a	Forage	-0	< 0.01	< 0.01	Sampling to analysis: 122-181 days	
					Z39			0	0.16		1.2
					Z71 ^b			28	0.02		0.16
					Z73 ^c			35	0.01		0.06
					Z85 ^d			42	< 0.01		< 0.01
	Mature	Straw	59	< 0.01	< 0.01						
	0.022	100	1	Z39	Forage	-0	< 0.01	< 0.01			
				Z39			0	0.45	3.7		
				Z71			28	0.06	0.45		
				Z73			35	0.04	0.22		
				Z85			42	0.02	0.12		
Mature				Straw			59	< 0.01	< 0.01		

^a Z39: Flag leaf ligule just visible

^b Z71: Kernel water ripe (no starch)

^c Z73: Early milk

^d Z85: Soft dough

Four field trials on imidazolinone-tolerant barley were conducted in different growing areas in Australia during the 2015 and 2016 growing season to determine the residue level of imazapyr in/on barley forage and straw. The SL formulation was applied once when the crop was between end of tillering to first node stage (29–32 BBCH). Samples of forage were collected at 13/14, 27–29, 41/42 and 55/56 days after application. Straw samples were collected at typical harvest stage, 84–140 days after application.

The Method M 2468 modified utilizing LC-MS/MS instead of GC-MS was used for the analysis of imazapyr residues in barley forage and straw samples. Average recoveries of imazapyr in barley forage were 103 ± 7.0% (n=6, 0.05 mg/kg fortification level) and 103 ± 2.6% (n=5, 0.5 mg/kg fortification level). Average recoveries of imazapyr in barley straw were 101 ± 9.8% (n=5, 0.05 mg/kg fortification level) and 100 ± 1.4% (n=5, 0.5 mg/kg fortification level). The LOQ was 0.05 mg/kg for barley forage and straw.

Table 6 Imazapyr residues on imidazolinone-tolerant barley forage and straw from supervised trials in Australia (Bower, 2016: 2016/8000282)

Barley country, year (variety)	Application				Growth Stage at harvest	Portion analysed	DALA Days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, L/ha	no.				as received	as dry weight	
GAP, Australia	SL	0.0056-0.011	min. 70	1	<i>Do not graze or cut for forage and fodder for 4 weeks after application</i>					
Australia, 2015-2016 Forth/TAS (Scope)	SL	0.011	113	1	BBCH 41	Forage	14	0.077	0.45	Sampling to analysis: 17-91 days
					BBCH 58-59		28	0.051	0.22	
					BBCH 83		42	< 0.05	0.12	
					BBCH 87		56	< 0.05	0.13	
					BBCH 97-99		Straw	84	< 0.05	
	0.023	111	1	BBCH 41	Forage	14	0.14	0.84		
				BBCH 58-59		28	0.082	0.35		
				BBCH 83		42	0.11	0.36		
				BBCH 87		56	0.071	0.21		
				BBCH 97-99		Straw	84	< 0.05	< 0.05	

Barley country, year (variety)	Application				Growth Stage at harvest	Portion analysed	DALA Days	Residues, mg/kg		Ref.
	Form	kg ai/ha	water, L/ha	no.				as received	as dry weight	
Australia, 2015 Balliang/VIC (Scope)	SL	0.011	99	1	BBCH 29-31	Forage	14	0.16	0.81	Sampling to analysis: 42-100 days
					BBCH 37		27	<u>0.099</u>	0.57	
					BBCH 41		42	< 0.05	0.26	
					BBCH 43		56	< 0.05	0.19	
				BBCH 89	Straw	99	< 0.05	<u>< 0.05</u>		
	0.023	101	1	BBCH 29-31	Forage	14	0.32	1.7		
				BBCH 37		27	0.19	1.2		
				BBCH 41		42	0.10	0.67		
BBCH 43				56		0.15	0.68			
			BBCH 89	Straw	99	0.067	0.13			
Australia, 2015 Wasleys/SA (Scope)	SL	0.011	101	1	BBCH 33-34	Forage	14	0.10	0.57	Sampling to analysis: 30-117 days
					BBCH 45		29	0.074	0.30	
					BBCH 59		41	0.078	0.24	
					BBCH 75		55	<u>0.087</u>	0.20	
				BBCH 99	Straw	88	< 0.05	<u>< 0.05</u>		
	0.023	101	1	BBCH 33-34	Forage	14	0.32	1.8		
				BBCH 45		29	0.21	0.90		
				BBCH 59		41	0.22	0.66		
BBCH 75				55		0.22	0.52			
			BBCH 99	Straw	88	0.058	0.064			
Australia, 2015 Williams/WA (Scope)	SL	0.011	100	1	BBCH 31-32	Forage	13	0.23	1.4	Sampling to analysis: 28-128 days
					BBCH 33-37		27	<u>0.094</u>	0.66	
					BBCH 37-41		41	0.052	0.32	
					BBCH 37-51		55	< 0.05	0.16	
				BBCH 99	Straw	140	< 0.05	<u>< 0.05</u>		
	0.023	100	1	BBCH 31-32	Forage	13	0.37	2.7		
				BBCH 33-37		27	0.19	1.5		
				BBCH 37-41		41	0.088	0.66		
BBCH 37-51				55		0.061	0.38			
			BBCH 99	Straw	140	0.056	0.062			

APPRAISAL

Imazapyr is a broad-spectrum herbicide belonging to the imidazolinone family. It was first evaluated by JMPR in 2013 (T, R). An ADI of 0–3 mg/kg bw was established and an ARfD was unnecessary. The compound was evaluated for residues in 2015. In 2013 the Meeting agreed that the definition of the residue was imazapyr for plant and animal commodities (for compliance with MRLs and for estimation of dietary intake). It was listed by the 48th Session of CCPR (2016) for the evaluation of 2017 JMPR for additional MRLs.

The current Meeting received information on analytical method, use patterns and supervised residue trials to support estimation of a maximum residue level for barley.

Method of analysis

The Meeting received information on the analytical method (Method M3098) for the determination of related compounds, imazamox and its metabolite, in wheat forage, hay, straw and grain. This method was evaluated by the 2014 JMPR and modified to allow simultaneous analysis of imazapyr and imazamox residues. The method is suitable for the analysis of imazapyr residues in barley forage, straw and grain.

Stability of residues in stored analytical samples

Freezer storage stability studies on maize (grain, forage and fodder) samples were evaluated by the 2013 JMPR. Storage stability results indicated that imazapyr residues were stable at -5 to -26 °C for at

least 27 months in maize (grain, forage and fodder). The periods of storage stability studies cover the sample storage intervals of residue trials.

Residues resulting from supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of imazapyr on barley (imidazolinone-tolerant) from Australia.

Barley

The supervised trials were conducted on imidazolinone-tolerant barley in Australia.

The GAP on imidazolinone-tolerant barley in Australia is a foliar application at a maximum rate of 0.011 kg ai/ha from the 5 leaf stage to the 1st node stage.

Imazapyr residues in barley grains from independent trials in Australia matching GAP were (n=6): < 0.01 (2), 0.16, 0.19, 0.25 and 0.32 mg/kg.

Based on the residues for barley grains from trials in Australia, the Meeting estimated a maximum residue level and an STMR value for imazapyr in barley of 0.7 and 0.175 mg/kg respectively.

Animal feedstuffs

Barley forage and straw

Data were available from supervised trials on imidazolinone-tolerant barley in Australia.

The GAP on imidazolinone-tolerant barley in Australia is a foliar application at a maximum rate of 0.011 kg ai/ha from the 5 leaf stage to the 1st node stage and not to graze or cut for forage and fodder for 4 weeks after application.

Imazapyr residues in barley forage (as received) from independent trials in Australia matching GAP were (n=6): 0.02, 0.04, 0.051, 0.087, 0.094 and 0.099 mg/kg.

Based on the residues for barley forage, the Meeting estimated a median residue value and a highest residue value for imazapyr in barley forage of 0.069 and 0.099 mg/kg, respectively on an “as received” basis.

Imazapyr residues in barley straw (dry weight basis) from independent trials in Australia matching GAP were (n=6): < 0.01 (2) and < 0.05 (4) mg/kg.

Based on the residues in barley straw, the Meeting estimated a maximum residue level of 0.05 mg/kg, a median residue value of 0.05 mg/kg and a highest residue value of 0.05 mg/kg for imazapyr in barley straw on a dry weight basis.

Residue in animal commodities

The 2017 JMPR evaluated residues of imazapyr in barley, 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 (18 to 18 ppm for cattle and 0.68 to 0.57 ppm for poultry). 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 below are suitable for estimating maximum residue limits and for IEDI assessment.

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

The residue is not fat soluble.

Commodity		Recommended MRL, mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
GC 0640	Barley	0.7		0.175	
AS 0640	Barley straw and fodder, dry	0.05 (dw)		0.05 (dw)	0.05 (dw)

* at or about the LOQ.

dw – dry weight

Commodity	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
Name		
Barley forage	0.069 (as)	0.099 (as)

as – as received

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes (IEDIs) of imazapyr were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the 2013, 2015 and the current Meeting (Annex 3 to the 2017 Report). The ADI is 0–3 mg/kg bw and the calculated IEDIs were 0% of the maximum ADI (3 mg/kg bw). The Meeting concluded that the long-term dietary exposure to residues of imazapyr, resulting from the uses considered by the JMPR, is unlikely to present a public health concern.

Short-term dietary exposure

The 2013 JMPR decided that an ARfD was unnecessary. The Meeting therefore concluded that the short-term dietary exposure to residues of imazapyr is unlikely to present a public health concern.

REFERENCES

Code	Author	Year	Title, Institution, Report reference
2009/1130682	Allen K.	2009	Determination of residues of Imazamox and Imazapyr in imidazolinone tolerant barley following a single foliar application of Intervix at first node detectable crop stage Peracto Pty Ltd., Devonport Tasmania 7310, Australia 2009/1130682, GLP, Unpublished
2016/8000282	Bower M.	2016	Determination of residues of Imazamox, (metabolite CL263284/4110773) and Imazapyr in Clearfield (Scope) barley Peracto Pty Ltd., Devonport Tasmania 7310, Australia 2016/8000282, GLP, Unpublished