IMAZAPYR (267)

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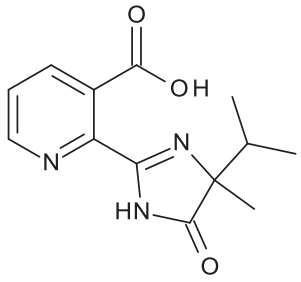
EXPLANATION

Residue and analytical aspects of imazapyr were considered for the first time by the present Meeting. The residue evaluation was scheduled for the 2013 JMPR by the Forty-fourth Session of the CCPR.

Imazapyr is a broad-spectrum herbicide in the imidazolinone family. Its primary use is as a post-emergence herbicide which is particularly effective on hard-to-control perennial grasses. It is non-selective, absorbed by foliage and rapidly translocated. The mode of action of imidazolinone herbicides is the inhibition of the enzyme acetohydroxy acid synthase (AHAS) which is a critical enzyme for the biosynthesis of branched chain amino acids necessary for cell growth and protein synthesis. The Meeting received information on identity, animal and plant metabolism, environmental fates in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fates of residues in processing.

IDENTITY

Common name	Imazapyr
Chemical name	
IUPAC:	2-[(*RS*)-4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl] nicotinic acid
CAS:	2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1H-imidazol-2-yl]-3-pyridinecarboxylic acid
CAS Registry No:	81334-34-1
CIPAC No:	530
Synonyms:	BAS 693 H, CL 243,997
Structural formula:	



Molecular formula:	$C_{13}H_{15}N_3O_3$
Molecular weight:	261.3

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Results	Reference
Appearance (colour, physical state, odour)	Dirty white/ egg-shell white powder with a faint non-specific odour (99.6% purity)	Werle *et al.,* 2001 2001/1024893
Vapour pressure	$< 2.7 \times 10^{-5}$ Pa at 45 °C	Anonymous 1985

Property	Results	Reference
		IZ-301-016
Melting point	170.2–172.0 °C (99.6% purity)	Werle *et al.,* 2001
Boiling point	Imazapyr decomposes prior to boiling (99.6% purity)	2001/1024893
Octanol/water partition coefficient	log Pow = 0.11 at 22 °C (99% purity)	Reichert, 1983 IZ-315-001
	log P_{OW} = 0.04 deionized water at 20 °C log P_{OW} = -0.39 at pH 4 at 20 °C log P_{OW} = -3.96 at pH 7 at 20 °C log P_{OW} = -3.97 at pH 10 at 20 °C (99.6% purity)	Daum, 2001 IZ-315-002
Solubility in water	9.74 g/L in distilled water at 15 °C 11.3 g/L in distilled water at 25 °C 13.5 g/L in distilled water at 35 °C (99% purity)	Anonymous 1985 IZ-311-001
Relative density	1.36 g/cm^3 at 20 °C (99.6% purity)	Werle *et al.,* 2001 2001/1024893
Hydrolysis	In distilled water, pH 5, pH 7 buffers there was no detectable degradation, thus a half-life could not be calculated because of the stability of the compound. At pH 9 the half-life was calculated to be 325 days.	Mangels, 1990 1990/7001950
Photolysis	DT_{50} = 2.7 days in distilled water DT_{50} = 2.7 days at pH 5 DT_{50} = 1.3 days at pH 9	Mangels, 1990 1990/7001949
Dissociation constant	pKa = 3.8 (99.9% purity)	Anonymous 1985 IZ-301-016
	pKa1 = 1.7, pKa2 = 3.5, pKa3 = 11.1 at 20 °C (99.6% purity)	Daum, 2001 IZ-390-005

Technical material

Property	Results	Reference
Appearance (colour, physical state, odour)	White to tan solid with a slightly pungent odour	Anonymous 1985 IZ-301-016
Vapour pressure	< 1.3 × 10^{-5} Pa at 60 °C (97.6% purity)	Mangels, 1986 IZ-306-001
Melting point	168–172 °C	Anonymous 1985 IZ-301-016
Solubility in water	11.1 g/L in distilled water at 25 °C	Anonymous 1985 IZ-301-016
Solubility in organic solvents	Hexane 0.0095 g/L at 25 °C	Teeter, 1990

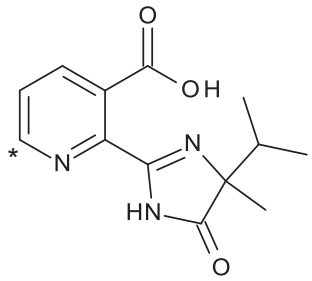
Property	Results		Reference
(98.6% purity)	Toluene	1.80 g/L at 25 °C	IZ-312-001
	Acetone	33.9 g/L at 25 °C	
	Dichloromethane	87.2 g/L at 25 °C	
	Methanol	105 g/L at 25 °C	
	Dimethyl sulfoxide	471 g/L at 25 °C	
Stability	Imazapyr is physically stable for 2 years. (95% purity)		Peevey, 1989 1989/7002428

Formulations

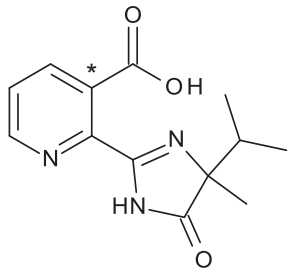
- Water soluble liquid (SL)
- Water dispersible granule (WG)
- Water soluble granule (SG)
- Emulsifiable concentrate (EC)
- Dustable powder (DP)

METABOLISM AND ENVIRONMENTAL FATE

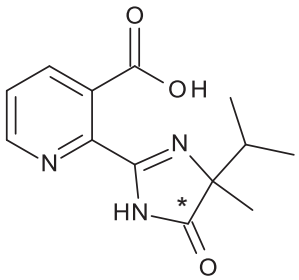
The metabolism, distribution of imazapyr has been investigated in animals and plants. The fate and behaviour of imazapyr in animals, plants and the environment was investigated using the [^{14}C] labelled test materials shown in Figure 1.



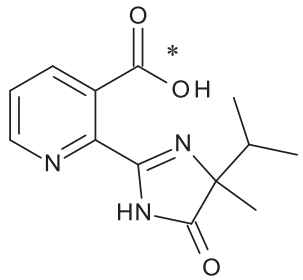
[pyridine-6-^{14}C]-imazapyr



[pyridine-3-^{14}C]-imazapyr



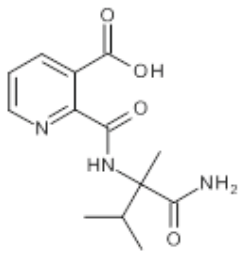
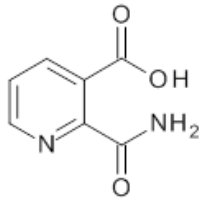
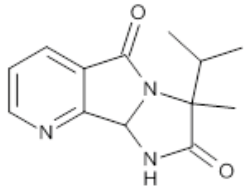
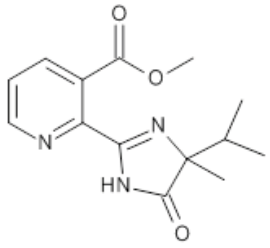
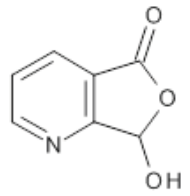
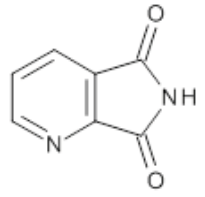
[imidazole-5-^{14}C]-imazapyr

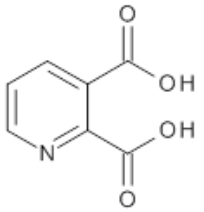
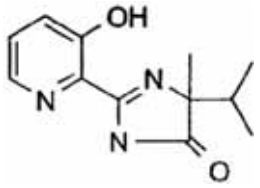


[carboxyl-^{14}C]-imazapyr

Figure 1 [^{14}C]-Labelled test materials used in animals, plants metabolism studies, and the environmental fate studies

The chemical structures of the major degradation compounds from the metabolism of imazapyr are provided below.

Compound name		Structure	Found in metabolism studies
CL 252,974	2-[(1-carbamoyl-1,2- dimethylpropyl) carbamoyl] nicotinic acid		Rat, Plants, Soil, Water
CL 60,032	2-carbamoyl-nicotinic acid		Rat, Soil
CL 247,087	5*H*-imidazo [1',2':1,2] pyrrolo [3,4-b] pyridine -2(3*H*),5-dione, 1.9b α(β)-dihydro-3α- isopropyl-3-ethyl-		Plants, Soil
240,000	2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-3 -carboxymethyl pyridine		Plants, Soil
CL 119,060	Furo[3,4-h]pyridine-5(7*H*)-one, 7-hydroxy-		Plants
CL 17,226	Pyridine 2,3-dicarboximide		Plants

Compound name		Structure	Found in metabolism studies
PDC	Pyridine 2,3-dicarboxylic acid		Plants, Soil
CL 288,247	2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-3-hydroxy pyridine		Soil

Animal metabolism

The Meeting received studies on the metabolism of imazapyr in rats, lactating goat and laying hens. The study on rats was evaluated by the WHO Core Assessment Group of the 2013 JMPR. A summary of the rat metabolism is given in this section.

Rats

The metabolism studies performed on rats indicated that imazapyr was quickly and extensively absorbed following administration. There were no substantial sex differences in the absorption, elimination or distribution of radioactivity in rats receiving an oral dose of radiolabelled imazapyr. The majority of the administered doses were excreted in urine (68–95%) and, to a lesser degree, in faeces (5.5–33%). Most elimination occurred within the first 24 hours after dosing (57–91% in urine; 3–24% in faeces). The half-life of imazapyr in the rat was less than 1 day. Imazapyr was excreted mostly unchanged. Trace levels of polar and nonpolar metabolites were formed and excreted in urine and faeces. Only trace amount of tissue residues were detected in the liver and kidneys on the high dose group, indicating no bioaccumulation.

Lactating goat

Study 1

The metabolism of imazapyr by the lactating dairy goats was studied by Zdybak (1992: IZ-440-002). [pyridine-6-^{14}C]-imazapyr was orally administered in gelatin capsules to lactating goats. Three lactating goats, each weighing from 44 to 67 kg, were used as model ruminants in the study. Each goat received a single daily oral dosage equivalent to a dietary level of 0 ppm (control), 17.7 ppm, or 42.5 ppm, respectively, for seven consecutive days. Based on actual mean feed consumption of 0.859 kg and 1.08 kg per day for low and high dosed goats during the test period, the mean actual daily dietary dosages of imazapyr were 17.7 ppm and 42.5 ppm for low and high dose goats, respectively.

Urine and faeces were collected daily and analysed for radioactivity content. During the treatment period, blood and milk samples were also collected and analysed for total radioactive residues (TRR). Goats were sacrificed ca. 22 hours after the seventh and final doses. Blood and edible tissues (liver, kidneys, leg muscle, loin muscle and omental fat) were collected.

Orally-administered radioactivity was mainly excreted in the urine (65.3% and 60.4% of the doses in the 17.7 ppm and 42.5 ppm dosed goats, respectively). Faecal elimination accounted for 16.1% and 19.0% of the doses in the 17.7 ppm and the 42.5 ppm dosed goats, respectively.

At both treatment levels, radiocarbon content in blood was less than the validated detection limit of 0.05 mg/kg. The TRR levels were < 0.01–0.01 mg/kg and 0.01–0.02 mg/kg in the milk samples of the 17.7 ppm and 42.5 ppm dosed goats, respectively. TRR levels for leg and loin muscles, liver, and fat were all less than the validated limit of detection of 0.05 mg/kg. Detectable residues were found in the kidneys namely 0.08 mg/kg (17.7 ppm dose) and 0.11 mg/kg (42.5 ppm dose).

Milk (day-7) and kidney samples from the 42.5 ppm dosed goat were subjected to solvent extraction followed by chromatographic analysis. Organosoluble milk residues distributed in hexane and ethyl acetate amounted to 17.7% and 6.4% of the TRR (or < 0.01 mg/kg), respectively. Water soluble residues accounted for approximately 49% (0.01 mg/kg) of the TRR, whereas the remainder was unextractable (26.6%, 0.01 mg/kg). Kidney residues were quantitatively extractable, with the majority (95.5%) isolated in a methanol/water fraction.

Table 1 Total radioactive residue (TRR) in goat milk and tissues

Sample	TRR (mg imazapyr equivalent/kg)		
	Control	Low dose (17.7 ppm)	High dose (42.5 ppm)
Milk (Day 1)	< 0.01	< 0.01	0.01
Milk (Day 2)	< 0.01	0.01	0.02
Milk (day 3)	< 0.01	< 0.01	0.01
Milk (Day 4)	< 0.01	< 0.01	0.01
Milk (Day 5)	< 0.01	0.01	0.01
Milk (Day 6)	< 0.01	0.01	0.02
Milk (Day 7)	< 0.01	0.01	0.02
Leg Muscle	< 0.05	< 0.05	< 0.05
Loin Muscle	< 0.05	< 0.05	< 0.05
Fat	< 0.05	< 0.05	< 0.05
Liver	< 0.05	< 0.05	< 0.05
Kidney	< 0.05	0.08	0.11
Blood (Day 0)	< 0.05	< 0.05	< 0.05
Blood (Day 1)	< 0.05	< 0.05	< 0.05
Blood (Day 3)	< 0.05	< 0.05	< 0.05
Blood (Day 7)	< 0.05	< 0.05	< 0.05

The extracted ^{14}C residue in the milk (0.01 mg/kg) was identified as unchanged parent compound by thin layer chromatography (TLC) and high performance liquid chromatography (HPLC) analyses. Similarly, the ^{14}C residue in the kidney was identified as unchanged parent compound. Chromatographic analyses of the urine showed a single radioactive residue component identical to that found in the kidney and milk.

Study 2

The purpose of this study was to determine the concentration of the total ^{14}C residues and the nature of these residues in kidneys and milk of the lactating goats following administration of daily oral doses of [imidazole-5-^{14}C]-imazapyr (Tsalta, 2000: IZ-440-005). [^{14}C] imazapyr or blank gelatin capsules were administered orally to lactating goats weighing between 49 and 50 kg (at treatment) that were less than 5 years of age. The two goats were dosed orally by capsule for seven consecutive days with the actual dietary equivalent of 0 ppm and 47 ppm [^{14}C] imazapyr as groups A (control) and B (treated), respectively, to determine total [^{14}C] imazapyr derived residues in daily milk, urine and faeces, and in the kidneys at sacrifice. The dose rate of 47 ppm for the treated goat was slightly higher than the rate of 42.5 ppm used for a goat study (IZ-440-002). That study showed that kidney and milk were the edible matrices that contained detectable residues following oral dosing of 42.5 ppm for 7 days. The study was to provide tissues (kidney and milk) with high enough residues so that they could be used to conduct an extraction efficiency/accountability study.

The TRR in the milk and the urine samples were determined by direct scintillation counting (LSC). The TRR in the kidneys and faeces were determined by combustion followed by LSC. Radioactivity in the extracts of the kidney and in liquid chromatographic fractions was determined by LSC. Radioactivity in the post extraction solids (PES) generated after kidney and milk extraction was determined by combustion followed by LSC. The specific radioactivity afforded a theoretical detection limit of 0.006 mg/kg imazapyr equivalents in the kidney, milk, urine and faeces when 0.5 g or 0.5 mL (nominal) sample aliquots were analysed by combustion and LSC or by direct LSC. The validated detection limit was 0.006 mg/kg for the milk and urine and 0.007 mg/kg for kidney and faeces.

During this study, milk, urine and faeces samples were collected daily. At sacrifice (approximately 22 hours after the last dose) only kidney samples were collected. The TRR levels in all control samples and pre-treatment samples were non-detectable (< 0.006 mg/kg). The TRR levels for the dosed goat were 0.014–0.015 mg/kg for milk and 0.074 mg/kg for kidney. The rate of elimination for the goat treated daily for 7 days with an oral dose of [^{14}C] imazapyr was determined by analysing the daily samples of urine and faeces. After seven days, 58.7% and 34.4% of the administered radioactive doses were excreted in the urine and faeces, respectively.

Radioactive residues were extracted from kidneys using acetone: water (3:1, v/v) as solvent. The overall recovery in the extractable portion was 93.5% and the unextractable (PES) was 6.5%. The concentrated extract was analysed by HPLC to determine the nature of the radioactive residue. The parent compound (imazapyr) was the predominant radioactive residue (81.9% of TRR, 0.061 mg/kg) in the kidney. Minor polar unknowns (total 11.6% of TRR) were also present in the kidney extract. Since these fractions contained multiple components (at least three), each less than 0.004 mg/kg, no further characterization was attempted.

The Day-7 treated goat milk was extracted by a procedure involving sequential extractions with acetonitrile: water, ethyl acetate and hexane. The extractable radioactivity was 82% and the unextractable (PES) was 18% of the TRR. The major component in the milk extract (65.6% of TRR, 0.01 mg/kg) was the parent compound (imazapyr). Minor polar unknowns (total 14.7% of TRR) were also present in the milk extract. Since these fractions were equivalent to 0.002 mg/kg and contained multiple components, no further characterization was attempted.

Table 2 Characterization and identification of radioactive residues in kidney and milk

Compound	Kidney		Milk, Day 7			
	% TRR	mg/kg [a]	% TRR		mg/kg [a]	
			Aqueous	Ethyl acetate	Aqueous	Ethyl acetate
Extract	93.5	0.07	46.3	35.8	0.006	0.005
Imazapyr	81.9	0.061	33.4	32.2	0.005	0.005
Polar unknowns	11.6	0.009	11.3	3.35	0.002	< 0.001
Unextracted	6.5	0.005	18.0			
TRR		0.074			0.014	

[a] mg imazapyr equivalent/kg

The results of this lactating goat study showed that orally administered [^{14}C] imazapyr was rapidly absorbed and eliminated in urine and faeces. At the end of the 7-day dosing period, 93.1% of the administered cumulative dose was recovered in the urine and faeces of the treated goat. The kidney and the milk of the treated goat contained 0.074 and 0.014–0.016 mg/kg, respectively, of TRR following seven days of oral treatment at approximately 47 ppm. The parent compound (imazapyr) was the predominant radioactive residue in the kidney (81.9% of TRR or 0.061 mg/kg) and in the milk (65.6% of TRR or 0.01 mg/kg). The concentration of the remaining radioactive components in the kidney and the milk were below 0.01 mg/kg.

Laying hens

The elimination, distribution and metabolic fate of imazapyr by egg laying hens were studied by Tsalta (1995: IZ-440-003). Three groups consisting of eight Dekalb XL white leghorn hens, each hen about 20 weeks old and weighing approximately 2.0 kg were used as test animals in this metabolism study. The three groups of hens were dosed orally by capsule for seven consecutive days with the nominal dietary equivalent of 0 ppm, 2 ppm, and 10 ppm [^{14}C]-labelled imazapyr as groups A (control), B (low dose), and C (high dose), respectively. The hens were used to determine total imazapyr-derived residues in the eggs (daily), in the excreta and in the blood and edible tissues at sacrifice. [pyridine-6-^{14}C]-imazapyr was used in the study. The specific radioactivity afforded a validated detection limit of 0.01 mg/kg imazapyr equivalents in the egg and hen tissues when 0.5 g sample aliquots were analysed.

The TRR in the daily egg samples were determined by LSC. The TRR in the blood, tissues and excreta were determined by combustion followed by LSC. Radioactivity in the extracts of excreta and in liquid chromatographic fractions was determined by combustion and by LSC; radioactivity in the post extraction solids of excreta was determined by combustion followed by LSC. The metabolite profile of imazapyr was defined by analysing acetone extracts of the excreta using high performance liquid chromatography. The identity of the main residue component imazapyr was determined by GC-MS.

Elimination of ^{14}C via the excreta accounted for 90.5 and 91.7% of the total doses for the low and high doses, respectively. During treatment, TRR in the egg samples was less than the validated detection limit of 0.01 mg/kg. The only significant component of the excreted ^{14}C residue was defined as imazapyr by HPLC analysis and by mass spectrometry.

Residues in blood, skin with adhering fat, muscle, liver and kidney tissues taken at 22 hours after the last dose were all less than the validated detection limit of 0.01 mg/kg. The results of this study clearly show that orally administered imazapyr was rapidly eliminated by the hen as unchanged parent compound and that imazapyr-related residues do not accumulate in eggs or the edible tissues of poultry at 108× the potential maximal dietary exposure level. These data demonstrate that there is no reasonable expectation of finite residues of imazapyr occurring in poultry tissue or eggs as a result of the herbicide use on corn.

Table 3 Total radioactive residues (TRR) in egg and tissues of hens

Sample	TRR (mg imazapyr equivalent/kg) [a]		
	Control	Low dose (1.98 ppm)	High dose (9.72 ppm)
Excreta (Day 1)	< 0.01	1.98	9.52
Excreta (Day 2)	< 0.01	1.57	9.85
Excreta (day 3)	< 0.01	1.62	9.90
Excreta (Day 4)	< 0.01	1.70	8.66
Excreta (Day 5)	< 0.01	1.78	9.73
Excreta (Day 6)	< 0.01	1.54	9.91
Excreta (Day 7)	< 0.01	1.77	9.45
Eggs (Day 1)	< 0.01	< 0.01	< 0.01
Eggs (Day 2)	< 0.01	< 0.01	< 0.01
Eggs (day 3)	< 0.01	< 0.01	< 0.01
Eggs (Day 4)	< 0.01	< 0.01	< 0.01
Eggs (Day 5)	< 0.01	< 0.01	< 0.01
Eggs (Day 6)	< 0.01	< 0.01	< 0.01
Eggs (Day 7)	< 0.01	< 0.01	< 0.01
Blood	< 0.01	< 0.01	< 0.01
Muscle	< 0.01	< 0.01	< 0.01
Skin with adhering fat	< 0.01	< 0.01	< 0.01
Liver	< 0.01	< 0.01	< 0.01
Kidney	< 0.01	< 0.01	< 0.01

[a] Excreta: mg/kg calculated on a fresh weight basis. Excreta sample from all hens (eight) from each group were combined for each day and analysed.

Blood and tissues: Values are the average of eight laying hens

The results of this laying hen study conducted at the actual dose levels of 1.98 and 9.72 ppm feed equivalents of imazapyr, showed that the parent compound or derived residues are excreted without retention or accumulation in eggs and edible poultry tissues. Thus, poultry exposed to diets of agricultural commodities containing even highly exaggerated levels of imazapyr residues would not contain or accumulate imazapyr-derived residues in the edible tissues and eggs.

Summary of animal metabolism

The metabolism of ^{14}C-labelled imazapyr has been studied in lactating goat and laying hens. In both studies, imazapyr was mainly eliminated and metabolised to several polar compounds. Imazapyr was the most important component in milk and kidney for lactating goat.

Plant metabolism

Plant metabolism studies were performed on soya bean, maize, sugarcane, oil palm, clover and Bermuda grass with imazapyr ^{14}C-labelled at the 3 or 6-position pyridine ring to track metabolites. Metabolites were identified using multiple chromatographic systems and authentic standards.

Soya beans

A metabolism study was conducted with [pyridine-3-^{14}C]-imazapyr in transgenic soya bean variety BPS-CV127-9 in which the csr1-2 gene encoding an altered AtAHSL protein was inserted to make the host tolerant to imidazolinone herbicide (Dohn, 2012: 2012/7000103). [^{14}C] imazapyr was formulated (a soluble liquid (SL)) and applied once to the above ground portion of soya beans plants at BBCH growth stage 65 with a hand operated pump sprayer. The application rate was 107 g/ha which was 149% of the maximum anticipate use rate of 72 g/ha. The test plots were located outdoors, and consisted of two plots, one control and one treated. The forage was harvested approximately one hour after application and the hay was harvested 35 days after application. Soya bean straw, pods and seeds were harvested when mature at 98 days after treatment.

All crop samples were processed, subjected to combustion analysis, extracted and analysed by reverse phase HPLC. The TRR of soya bean forage from combustion was 0.655 mg/kg, soya bean hay was 0.247 mg/kg, soya bean seed was 0.062 mg/kg, soya bean straw was 0.079 mg/kg and soya bean pod was 0.146 mg/kg. The extraction procedure released 72.5% to 98.7% of the TRR. The unextracted residues were small, 0.008 mg/kg (1.3% TRR) in forage, 0.028 mg/kg (11.1% TRR) in hay, 0.011 mg/kg (15.9% TRR) in seeds, 0.016 mg/kg (21.6% TRR) in straw, and 0.039 mg/kg (27.5% TRR) in pods (see Table 4).

The parent compound imazapyr was identified by co-chromatography with a reference standard and by LC-MS analysis of an isolated fraction from hay. Imazapyr was detected in all matrices and was the most abundant component of the residue in soya bean forage (0.599 mg/kg, 93.6% TRR), hay (0.094 mg/kg, 37.3% TRR), and seed (0.0236 mg/kg, 34.2% TRR). Imazapyr was present in straw at 0.006 mg/kg (8.1% TRR) and pods at 0.018 mg/kg (12.7% TRR).

Table 4 Extractability of Soya bean matrices and Total Radioactive Residues

Matrix	DAT	Distribution of radioactive residues						Total extracted		Unextracted		TRR (calculated)	TRR (combustion)
		Methanol extract		Aqueous extract		Acidic methanol							
		mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	mg/kg
Forage	0	0.591	92.3	0.035	5.5	0.006	0.9	0.632	98.7	0.008	1.3	0.640	0.655
Hay	35	0.182	72.2	0.036	14.3	0.006	2.4	0.224	88.9	0.028	11.1	0.252	0.247
Seeds	98	0.022	31.9	0.014	20.3	0.022	31.9	0.058	84.1	0.011	15.9	0.069	0.062
Straw	98	0.040	54.1	0.014	18.9	0.004	5.4	0.058	78.4	0.016	21.6	0.074	0.079
Pods	98	0.031	21.8	0.036	25.4	0.036	25.4	0.103	72.5	0.039	27.5	0.142	0.146

A polar component M3 (not retained on reverse phase HPLC) was the most abundant component in the pods (0.041 mg/kg, 28.9% TRR). This component was also present in hay

(0.028 mg/kg, 11.1% TRR), straw (0.009 mg/kg, 12.2% TRR) and seeds (0.0161 mg/kg, 23.3% TRR), but was not detected in forage. The polar peak was isolated from the soya bean seeds, and shown to consist of multiple components, present at ≤ 0.004 mg/kg. A polar component was observed in seed, hay, straw and pod and shown to be comprised of multiple components. Imazapyr was degraded in soya bean to multiple products present in relatively small concentrations.

The remainder of the residues consisted of multiple components individually present in small concentrations. No metabolites were present at levels exceeding 10% TRR in any crop matrices except for M19 in the straw. M19 was present in the straw at 0.013 mg/kg (17.6% TRR) and was also found in hay at 0.022 mg/kg (8.7% TRR).

Table 5 Nature of the radioactive residues in soya bean matrices

Components	Forage		Hay		Seeds		Straw		Pods	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Extract	0.632	98.7	0.224	88.9	0.058	84.1	0.058	78.4	0.103	72.5
Imazapyr	0.599	93.6	0.094	37.3	0.0236	34.2	0.006	8.1	0.018	12.7
M3 [a]	ND		0.028	11.1	0.0161	23.3	0.009	12.2	0.041	28.9
M4-5	ND		0.013	5.2	0.0008	1.2	0.002	2.7	0.010	7.0
M8	ND		0.008	3.2	ND		ND		0.006	4.2
M19	ND		0.022	8.7	ND		0.013	17.6	0.003	2.1
M28	ND		ND		ND		0.003	4.1	ND	
M31	ND		0.011	4.4	0.0026	3.8	0.005	6.8	0.011	7.7
M45.5	ND		0.024	9.5	0.0016	2.3	0.004	5.4	0.004	2.8
M52	0.027	4.2	0.004	1.6	ND		ND		ND	
Unknowns	0.006	0.9	0.020	8.0	0.0136	19.7	0.016	21.6	0.010	7.0
Unextracted	0.008	1.3	0.028	11.1	0.011	15.9	0.016	21.6	0.039	27.5
Total	0.640	100	0.252	100	0.069	100	0.074	100	0.142	100

[a] Components code with a retention time of LC-MS

Imazapyr underwent significant metabolism, however, the parent molecule was the most abundant component of the residue in all matrices except straw and pods. The concentration of imazapyr declined from 0.599 mg/kg (93.6% TRR) in forage collected approximately 1 hour after application to 0.094 mg/kg in the hay sample (37.3% TRR) collected 35 days after application, and then 0.006 mg/kg (8.1% of the TRR) in the straw sample collected 97 days after application. The imazapyr concentrations were 0.0236 mg/kg (34.2% TRR) in seed, and 0.018 mg/kg (12.7% TRR) in pods (see Table 5).

Imazapyr was degraded to a number of components in soya bean commodities. In seed, multiple metabolites are present with concentrations at ≤ 0.004 mg/kg (5.8% TRR), and parent was the main component. The very polar peak M3 that was not retained on reverse phase HPLC was isolated from the seed extract. Analysis using a Hypercarb column showed the peak to be comprised of multiple components, each present at ≤ 0.004 mg/kg (5.8%TRR). In straw, component M19, a component of intermediate polarity with a retention time of about 19 minutes, was present at amounts > 10% TRR with concentration of 0.013 mg/kg (17.6% TRR). The component was also present in hay but at levels < 10% TRR. Due to the presence over 10% TRR in only straw which is an animal feed item, the component M19 was not further analysed. In addition to minor components such as M31 and M45.5, a large number of components that did not exceed 0.002 mg/kg were present in the matrices of hay, straw and pod. As was demonstrated with the polar peak isolated from the seed extract, the polar peak in the hay, straw and pod was assumed to be comprised of a large number of low-level components.

Maize

A metabolism study was conducted in imidazolinone-resistant maize (Pioneer hybrid 3245 IR) to determine the total ^{14}C residues following a post-emergence treatment of maize plants at the 3 to 4 leaf growth stage with [pyridine-6-^{14}C]-imazapyr at treatment rates of 0.028 kg ai/ha (plot A) and 0.080 kg ai/ha (plot B), and to determine the nature of any significant imazapyr-derived residues in

the green plants, forage, fodder (stalks/husks/cobs), and grain (Zulalian, 1995: IZ-640-003). The treatment rates used in this study correspond to 1× and 2.8× the maximum label rate (0.027 kg ai/ha) for the use of imazapyr on imidazolinone-resistant maize. Soil core samples were taken prior to application of the test substance (–1 DAT), within two hours following application of the test substance (0 DAT), and one day after final harvest (115 DAT) of the mature maize plants. Samples of maize plants were harvested at 0 DAT (green plant), 14 DAT (green plant), 30 DAT (early forage), and 62 DAT (late forage). At maturity (114 DAT), the stalks, husks, and cobs with grain were collected.

The TRR in all plant and soil samples were determined by combustion and liquid scintillation counting (LSC). The TRR in soil (0–3 inches) at 0 DAT was 0.012 mg/kg and 0.047 mg/kg from plots A and B, respectively. At 115 DAT (1 day after maize harvest), the TRR in the soil (0–3 inches) was 0.006 mg/kg and 0.016 mg/kg from plots A and B, respectively. Residues were 0.002 to 0.007 mg/kg in soil at 7.6–15 cm, < 0.002 to 0.003 mg/kg at 15–30 cm and < 0.002 at 30–46 cm.

The green plant and forage samples were extracted with methanol. The fodder was extracted with methanol: water: hydrochloric acid and the grain were extracted with hexane followed by methanol: water: hydrochloric acid.

For the 0 DAT maize plant from plot A, 96.3% (2.37 mg/kg) of the TRR was extracted and 3.7% (0.093 mg/kg) remained in the post extracted solids (PES). The 0 DAT maize plant from plot B was not extracted. For the 14 DAT maize plants from plot A and B, 78.6 to 81.9% (0.046 to 0.126 mg/kg) was extracted and 18.1 to 21.4% (0.012 to 0.028 mg/kg) remained in the PES. For the 30 DAT samples (early forage) from plots A and B, 84.2 to 85.8% (0.008 to 0.022 mg/kg) of the TRR was extracted and 14.2 to 15.7% (0.002 to 0.004 mg/kg) remained in the PES. For the 62 DAT samples (late forage) from plot B, 86.8% (0.022 mg/kg) of the TRR was extracted and 13.3% (0.003 mg/kg) remained in the PES. For the 114 DAT fodder from plot B, 69.9% (0.020 mg/kg) of the TRR was extracted and 30.1% (0.008 mg/kg) remained in the PES. The late forage and fodder samples from plot A were not extracted because the TRR was < 0.01 mg/kg. For the 114 DAT maize grain from plots A and B, 0.8 to 1.5% (< 0.002 mg/kg) was extracted with hexane, 80.0 to 88.8% (0.023 to 0.076 mg/kg) was extracted with methanol: water: hydrochloric acid, and 10.3 to 18.5% (0.005 to 0.009 mg/kg) remained in the PES (see Table 6).

Table 6 Extractability of residues from maize matrices and Total Radioactive Residues

Matrix	DAT	1× Treatment rate (0.028 kg ai/ha: Plot A)					2.8× Treatment rate (0.080 kg ai/ha: Plot B)				
		TRR	Extracted		Unextracted		TRR	Extracted		Unextracted	
		mg/kg	mg/kg	%TRR	mg/kg	%TRR	mg/kg	mg/kg	%TRR	mg/kg	%TRR
Green plant	0	2.471	2.379	96.3	0.093	3.7	8.711	n.p.			
Green plant	14	0.058	0.046	78.6	0.012	21.4	0.153	0.126	81.9	0.028	18.1
Early forage	30	0.010	0.008	84.2	0.002	15.7	0.026	0.022	85.8	0.004	14.2
Late forage	62	0.004	n.p.				0.025	0.022	86.8	0.003	13.3
Fodder [a]	114	0.009	n.p.				0.028	0.020	69.9	0.008	30.1
Grain	114	0.029	0.023	81.5	0.005	18.5	0.086	0.077	89.6	0.009	10.3

n.p. = not performed

[a] Stalks/husks/cobs, combined

The imazapyr-derived residues in the grain did not concentrate in the maize oil since no residues (< 0.002 mg/kg) were detected in the hexane extract of the maize grain from the 1× and 2.8× treatments.

HPLC analyses of the radioactivity extracted with methanol and methanol: water: hydrochloric acid was conducted on a C18 reverse phase column. The radioactivity profiles of the extracts of the green plant, forage, fodder, and grain were similar qualitatively. There was one major component and a minimum of sixteen minor radiolabelled components that were separated and detected by C18 reverse-phase HPLC and LSC. Parent imazapyr constituted the major component of the extractable residue in the green plant, forage, fodder, and grain (12.0 to 80.8% TRR, 0.003 to 1.997 mg/kg). The residue levels of the minor components in the 30 DAT to 114 DAT samples were

all < 0.01 mg/kg. A number of minor components of the TRR exhibited the same HPLC retention times as PDC, CL 60,032, CL 263,078, CL 252,974, CL 252,663 and CL 271,045 (see Table 7).

Table 7 Distribution of extracted ^{14}C residue in the maize matrices following foliar application of [^{14}C] imazapyr

	1× Treatment rate (0.028 kg ai/ha)						2.8× Treatment rate (0.080 kg ai/ha)									
	0 DAT (green plant)		14 DAT (green plant)		114 DAT (grain)		14 DAT (green plant)		30 DAT (early forage)		62 DAT (late forage)		114 DAT (fodder)		114 DAT (grain)	
	mg/kg [a]	% [b]	mg/kg [a]	% [b]	mg/kg [a]	% [b]	mg/kg [a]	% [b]	mg/kg [a]	% [b]	mg/kg [a]	% [b]	mg/kg [a]	% [b]	mg/kg [a]	% [b]
Imazapyr	1.997	84.0	0.029	63.7	0.012	51.0	0.082	64.8	0.014	65.0	0.013	61.5	0.003	16.8	0.055	71.9
PDC	< 0.001	< 0.1	0.001	2.5	0.001	2.4	0.001	0.8	< 0.001	1.3	< 0.001	1.4	0.002	9.6	< 0.001	0.5
CL60,032	0.002	0.1	0.001	1.4	< 0.001	1.1	0.001	1.1	< 0.001	0.9	< 0.001	0.7	< 0.001	1.1	0.0	0.0
CL263,078	0.025	1.1	0.001	2.7	0.001	4.0	0.004	2.8	0.001	2.5	0.001	3.8	0.002	8.9	0.003	3.7
CL252,974	0.026	1.1	0.002	3.7	0.001	3.6	0.004	2.8	0.001	3.6	0.001	2.9	0.001	4.6	0.003	3.7
CL252,663	0.078	3.3	0.001	2.1	0.001	6.3	0.004	3.3	0.001	3.0	0.001	2.9	0.001	3.2	0.006	8.1
CL271,045	0.042	1.8	0.001	2.2	0.001	2.2	0.002	1.7	< 0.001	1.6	< 0.001	1.3	< 0.001	1.1	0.001	1.0
Unknowns	0.209	8.8	0.011	21.9	0.007	29.5	0.028	22.9	0.005	22.0	0.006	25.6	0.011	54.6	0.009	11.2
Total extracted	2.379	100	0.046	100	0.021	100	0.126	100	0.020	100	0.020	100	0.018	100	0.075	100

[a] mg/kg TRR

[b] Relative percent of extracted TRR

Imazapyr was isolated from the extracts of the 62 DAT forage and 114 DAT grain from plot B by affinity chromatography using an anti-imidazolinone antibody and identified by GC-MS analysis.

Imazapyr is the only significant component of the residue in imidazolinone-resistant maize after application of imazapyr at the maximum intended label rate. All components of the residue in the raw agricultural commodities were < 0.01 mg/kg with the exception of the grain in which imazapyr accounted for only 0.012 mg/kg.

Sugarcane

The metabolism of [^{14}C] imazapyr in sugarcane variety CP 65-357 was investigated (Mangels and Lucas, 1987: IZ-640-001). [pyridine-6-^{14}C]-imazapyr was formulated and applied to the soil surface once as a pre-emergence application at a rate equivalent to 0.28 kg ai/ha using a hand-sprayer. Stalk samples were taken at maturity approximately 14 months after the pre-emergence treatment. The sugarcanes (without outer leaves) were homogenized and subjected to combustion analysis.

There were no detectable residues in the treated samples, which gave counts that were equivalent to both the control samples and the background levels of the cocktails and the equipment. The TRR of the samples was below the sensitivity of the method (< 0.005 mg/kg). No further investigations and storage stability analyses were carried out.

Oil palm

A metabolism study was conducted with [pyridine-6-^{14}C]-imazapyr in oil palm (Mallipudi, Taylor and Paniagua, 1986: IZ-640-002). [^{14}C] imazapyr was formulated as the isopropylamine salt and applied to the ground beneath an actively fruiting oil palm. The application rate was 1.0 kg ai/ha. Out of three palms selected as test plots, two were treated with [^{14}C] imazapyr and one was used as control. Fruit samples were collected from the oil palm as they ripened at 0 hour, 7, 30 and 62 days after treatment.

Ripe fruits were collected from untreated palms on the same day as from the treated palms. Palm oil was extracted from the fruits using a hexane: water mixture (3:1, v/v).

Residual radioactivity levels expressed as mg/kg equivalents of imazapyr were below the validated sensitivity of the method (< 0.03 mg/kg) at any given time in the palm oil, aqueous phase, fruit marc, kernel shell and kernel nut. There was no detectable radiocarbon in palm oil and other fruit fractions from the control palm.

The radioactive residues derived from [^{14}C] imazapyr were present in the soil samples (0.30–1.70 mg/kg in the 0–7.6 cm depth) at each sampling interval of oil palm fruits. The results indicate that imazapyr derived residues will not accumulate in the palm oil of an actively fruiting oil palm after application to the ground beneath the palm.

Clover

A metabolism study was conducted with [pyridine-6-^{14}C]-imazapyr (Wu, 1997: IZ-640-007). [^{14}C] imazapyr was applied to clover at a rate of 1.68 kg ai/ha (acid equivalents) as a 240 g/L water soluble formulation. Soil cores were collected to a depth of 45.7 cm before application (–1 day), after application (0 day), and at final sampling (21 day). Clover foliage was collected at 0, 4, 10, 15 and 21 days after treatment (DAT) and analysed for metabolite identification. The field study was conducted outdoors in two separate plots; plot A received the test material spray treatment, and plot B (control) received the blank spray treatment. Phytotoxic effects were apparent four days after application of the test material to the clover in the test plot. The clover in the untreated control plot appeared healthy. The phytotoxicity remained apparent until the final collection 21 days after treatment.

The soil cores and clover foliage from the control and treated plots were analysed for total radioactive residue (TRR) content. The clover foliage from the treated plot at each sampling interval also was extracted with acidified methanol and analysed for metabolites.

The TRR levels (expressed as mg/kg imazapyr equivalent) in soil were low. For example, levels were 0.084 mg/kg at 0–7.62 cm, 0.017 mg/kg at 7.62–15.2 cm at 0 DAT and < 0.01 to 0.144 mg/kg at different depths at 21 DAT. The level of radioactivity in soil increased due to wash-off by water and release of radioactivity by dying plants. Soil cores at all other sampling times and depths had only background levels (< 0.01 mg/kg).

TRR levels in foliage were 43.0 mg/kg (0 DAT), 37.4 mg/kg (4 DAT), 23.4 mg/kg (10 DAT), 41.6 mg/kg (15 DAT) and 49.2 mg/kg (21 DAT). The high radioactive residues in foliage at 0 DAT resulted from direct application of [^{14}C] imazapyr to clover. The TRR in foliage declined from the time of application to 10 DAT. The increased TRR in 15 and 21 DAT samples was probably attributed to the loss of water from plants as the plants died. TRR values in control soil and plant samples were all less than the minimum quantifiable limit of detection (< 0.01 mg/kg). Control clover from 10 DAT, however, was 0.011 mg/kg.

Each clover sample was extracted by blending with methanol/hydrochloric acid (100/0.75, v/v). The acidified methanol extracted 97.6%, 98.9%, 96.9%, 93.7% and 89.4% of TRR from the 0, 4, 10, 15 and 21 DAT samples, respectively. Post extraction solids (PES-1) were hydrolysed with protease in a phosphate buffer (pH approximately 7.5), followed by cellulase in an acetate buffer (pH approximately 5). The enzyme hydrolysates were combined and concentrated for metabolite analysis. The PES-3 fractions (PES after protease and cellulase hydrolyses of PES-1) from 0, 10, 15 and 21 DAT samples were further hydrolysed using 6 N HCl (Aqueous-3).

The acidified methanol extracts were analysed by HPLC and TLC. The majority of the radioactivity was unchanged imazapyr, ranging from approximately 66% to 99% of TRR. The other major metabolites were CL 247,087 plus CL 240,000 (approximately 0–18% of TRR, 80–90% of the total was estimated to be contributed by CL 247,087) and PDC (approximately 0–5% of TRR). There also appeared to be trace amounts of CL 252,974 (0–3% of TRR) and several other unknown metabolites.

Analysis of the enzyme hydrolysate from PES indicated that parent imazapyr was still the major component identified (28.5–44.2%), other metabolites detected were CL 240,000/CL 247,087 (2.74–12.5%) and PDC (0–17.6%), unknown A (R_t approximately 20–21 min, 0–20.7%), unknown B (R_t approximately 32 min, 5.63–58.3%), and CL 252,974 (0–13.9%). The AQ-3 fractions of the 15 and 21 DAT samples (i.e., hydrolysates after 6 N HCl hydrolysis of PES-3) were analysed by HPLC. The 15 and 21 DAT samples showed a major peak at R_t approximately 8 min, this retention time was very close to that of PDC. In addition, two-dimensional TLC analysis indicated that this peak was very likely PDC.

Table 8 Distribution of metabolites in clover after application of [^{14}C] imazapyr

Components	0 DAT		4 DAT		10 DAT		15 DAT		21 DAT	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Imazapyr	42.23	98.2	37.06	99.2	20.17	86.1	31.81	76.4	33.23	67.5
PDC	0.09	0.21	nd		0.11	0.48	2.73	6.56	1.20	2.43
CL 240000 + CL 247087 [a]	0.03	0.07	0.007	0.02	1.86	7.93	2.97	7.14	9.05	18.4
CL 252974	0.05	0.12	nd		0.045	0.19	0.60	1.45	1.77	3.60
Unknown A	0.07	0.17	nd		0.81	3.47	1.20	2.89	0.39	0.79
Unknown B	0.06	0.14	0.18	0.47	0.023	0.10	0.14	0.34	0.12	0.25
Other Unknowns	nd		nd		0.068	0.29	1.27	3.05	1.63	3.32
Precipitate	0.28	0.64	0.08	0.22	0.18	0.77	0.73	1.77	1.52	3.09
Aqueous-3	0.19	0.43	n.a.	n.a.	0.13	0.54	n.a.	n.a.	n.a.	n.a.
Unextracted (PES)	0.01	0.02	0.03	0.08	0.04	0.94	0.15	0.37	0.31	0.62
Total	43.01	100	37.35	100	23.44	100	41.62	100	49.22	100

DAT = days after treatment

nd = not detected

n.a. = not applicable

[a] Majority of the total (80 to 90%) was estimated to be contributed by CL 247087.

In conclusion, imazapyr remained mostly unchanged when applied to clover at 1.68 kg ai/ha. Overall, approximately 91.6–99.2% of the incurred residue had been defined in the study. The unchanged imazapyr accounted for 67.5–99.2% of the total residue. A number of minor metabolites, including CL 240,000/CL 247,087, CL 252,974 and PDC were present at 0.02–18.4% (the estimated ratio of CL 240,000/CL 247,087 was in the range of 1:5 to 1:10), 0–3.60% and 0–6.56% of TRR, respectively.

Bermuda grass

A metabolism study was conducted with [pyridine-6-^{14}C]-imazapyr (Wu, 1997: IZ-640-006). [^{14}C] imazapyr was applied to Bermuda grass at a rate of 1.68 kg ai/ha (acid equivalents) as a 240 g/L water soluble formulation. Soil cores were collected to a depth of 45.7 cm before application (-1 day), after application (0 day), and at the final sampling (21 day). Bermuda grass foliage was collected at 0, 4, 10, 15 and 21 days after treatment (DAT) and analysed for metabolite identification. The field study was conducted outdoors in two separate plots; plot A received the test material spray treatment, and plot B (control) received the blank spray treatment. Phytotoxic effects were apparent four days after application of the test material to the Bermuda grass in the test plot. The Bermuda grass in the untreated control plot appeared healthy. The phytotoxicity remained apparent until the final collection 21 days after treatment.

The soil cores and grass foliage from the control and treated plots were analysed for total radioactive residue (TRR) content. The grass foliage from the treated plot at each sampling interval also was extracted with acidified methanol and analysed for metabolites.

The TRR levels (expressed as mg/kg imazapyr equivalent) in soil were low. For example, levels were 0.041 mg/kg (0 day) at 0–7.62 cm and ranged from 0.048 to 0.131 mg/kg (21 day) at different depths (up to 30.5 cm). The level of radioactivity in soil increased due to wash-off by water

and release of radioactivity by dying plant. Soil cores at all other sampling times and depths had only background levels.

TRR levels in foliage were the highest at 0 DAT (64.3 mg/kg); they then decreased to 17.5 mg/kg at 4 DAT; 22.0 mg/kg at 10 DAT, 24.9 mg/kg at 15 DAT and 47.7 mg/kg at 21 DAT. The high radioactive residues in foliage at 0 DAT resulted from direct application of [^{14}C] imazapyr to Bermuda grass. The increased TRR in 21 DAT compared to the 15 DAT samples was probably attributed to the loss of water as the plants died. TRR values in control soil and plant samples were all less than the minimum quantifiable limit of detection (< 0.01 mg/kg).

Each grass sample was extracted by blending with methanol/hydrochloric acid (100/0.75, v/v). The acidified methanol extracted 97.3%, 97.1%, 94.5%, 88.1% and 85.8% of TRR from the 0, 4, 10, 15 and 21 DAT samples, respectively. Post extraction solids (PES) were hydrolysed with protease in a phosphate buffer (pH approximately 7.5), followed by cellulase in an acetate buffer (pH approximately 5). The enzyme hydrolysates were combined and concentrated for metabolite analysis. The PES-3 fractions from 0, 4, 10, 15 and 21 DAT samples were further hydrolysed using 6 N HCl, yielding AQ-3 and PES-4 fractions. The PES-4 fractions from the 10 and 15 DAT samples were further hydrolysed by 1 N NaOH.

The acidified methanol extracts were analysed by HPLC and TLC. The majority of the radioactivity was unchanged imazapyr, ranging from 74.4% to 94.7% of TRR. Three other identified radio components were PDC, CL 247,087 cyclized product and CL 240,000. The amount of PDC in the acidified methanol fraction increased with time from 0% at 0 DAT to 7.23% of TRR at 21 DAT. The amount of CL 247,087 and CL 240,000 in the acidified methanol fraction was highly variable, ranging from 0% to 10.1% of TRR.

It is known that imazapyr forms methyl ester (CL 240,000) in methanol. Additional experiments were conducted to find out whether the CL 240,000 detected was formed metabolically or as a result of chemical reaction. Extraction of a separate subsample of 0 and 21 DAT grass samples with methanol/HCl or acetone followed by chromatographic analysis showed that CL 240,000 was present in both acetone and acidified methanol extract. The acidified methanol fraction showed higher amounts of CL 240,000; however, acetone had much lower extraction efficiency for the radioactivity. The peak height of CL 240,000 increased after one month storage of acidified methanol extract in the refrigerator. The results indicate that small amounts of CL 240,000 are metabolite generated by the grass; the variability may be a result of the storage stability property.

Analysis of the enzyme hydrolysate from PES indicated that parent imazapyr was still the major component identified (1.10–4.55% of TRR), other metabolites detected were CL 240,000 and/or CL 247,087 (0–0.54% of TRR), PDC (0.08–1.79% of TRR) and unknown C (R_t approximately 20–20.5 min, 0–0.45%). The PES-3 fractions from the 0, 4, 10, 15 and 21 DAT samples were further hydrolysed by 6 N HCl. The hydrolysates were analysed by HPLC. All of the samples showed a single peak at R_t approximately 5 min; this retention time was very close to that of PDC. In addition, two-dimensional TLC analysis indicated that this peak was very likely PDC. The PES-4 samples from the 10 and 15 DAT grass were again hydrolysed with 1 N NaOH, however, the hydrolysates (AQ-4) were not further analysed due to the presence of low radioactive residue levels.

Table 9 Distribution of metabolites in Bermuda grass after application of [^{14}C] imazapyr

Components	0 DAT		4 DAT		10 DAT		15 DAT		21 DAT	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Imazapyr	62.19	96.7	16.52	94.4	20.01	90.8	19.45	78.1	38.15	80.0
PDC	0.21	0.32	0.23	1.32	0.91	4.12	1.22	4.91	6.12	12.8
CL 240000 + CL 247087 [a]	0.19	0.30	nd		0.044	0.20	2.62	10.5	1.44	3.01
Unknown A	nd		nd		0.093	0.42	nd		nd	
Unknown B	nd		0.38	2.18	nd		0.12	0.48	nd	
Unknown C	0.45	0.70	0.28	1.60	0.007	0.03	0.27	1.07	0.50	1.04
Unknown D	nd		nd		0.19	0.87	0.22	0.88	nd	
Unknown E	0.41	0.64	nd		0.079	0.36	nd		nd	
Unknown F	0.78	1.22	nd		0.34	1.54	nd		nd	

Components	0 DAT		4 DAT		10 DAT		15 DAT		21 DAT	
	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR
Precipitate	0.045	0.07	0.039	0.22	0.12	0.56	0.24	0.95	0.76	1.60
Aqueous-4	n.a.	n.a.	n.a.	n.a.	0.22	1.00	0.70	2.81	n.a.	n.a.
Unextracted (PES)	0.019	0.03	0.047	0.27	0.013	0.06	0.065	0.26	0.72	1.50
Total	64.30	100	17.50	100	22.02	100	24.89	100	47.68	100

DAT = days after treatment

[a] The estimated contribution of CL 247087 was about 50% of the total. The estimation was based on extraction of new 0 DAT and 21 DAT subsamples using acetone.

nd = not detected

n.a. = not applicable

In conclusion, imazapyr remained mostly unchanged when applied to Bermuda grass at 1.68 kg ai/ha. Overall, approximately 94–97% of the incurred residue had been defined in the study. The unchanged imazapyr accounted for 78–97% of the total residue. A number of metabolites, including PDC and CL 247,087 plus CL 240,000 were present at 0.32–12.8% and 0–10.5% of TRR, respectively.

Summary of plant metabolism

Metabolism of [^{14}C] imazapyr labelled in pyridine ring has been studied in soya bean (imidazolinone-resistant), maize (imidazolinone-resistant), sugarcane, oil palm, clover and Bermuda grass, which are suitable to cover the crop groups of pulses, cereals, oilseeds and animal feed. Imazapyr is the major component of the residues found in soya bean, maize, clover, and Bermuda grass. PDC is also the major components of the residues in clover and Bermuda grass. The following metabolic pathways were speculated in the plant metabolism studies available.

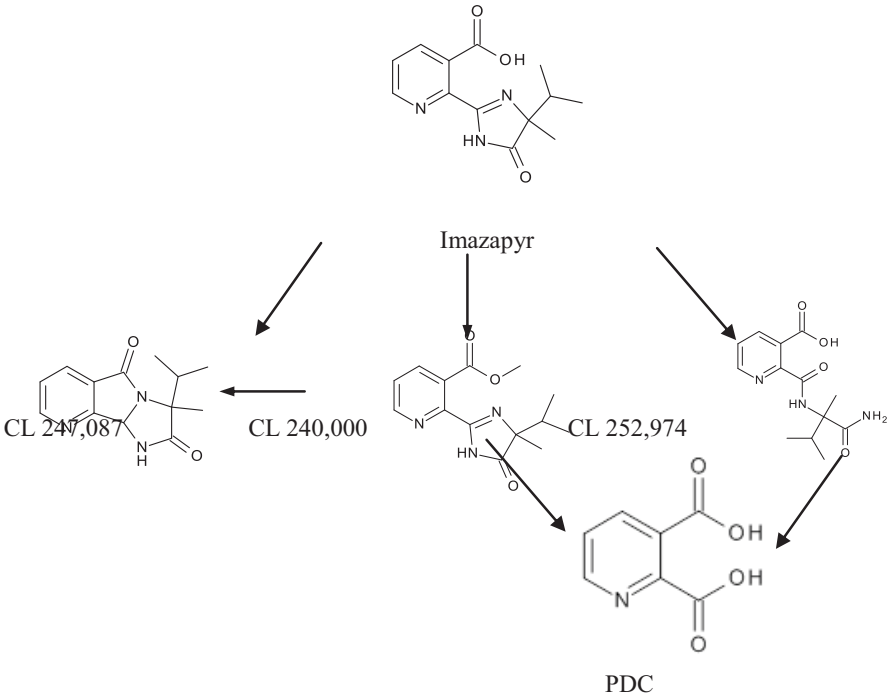


Figure 2 Metabolic Pathway of Imazapyr in Plants (clover and Bermuda grass)

Environmental fate in soil

The Meeting received information on aerobic degradation in soil, photolysis on soil surface, rotational crop and hydrolytic degradation study. Because imazapyr is intend for use as foliar and soil treatment, aerobic degradation, soil photolysis, rotational crop and hydrolytic degradation study relevant to the current evaluations were reported below (FAO Manual 2009).

The fate and behaviour of imazapyr in soils were investigated using [^{14}C-carboxyl] and [^{14}C-pyridine] labelled compounds.

Aerobic degradation

Study 1

The aerobic soil metabolism of [carboxyl-^{14}C]-imazapyr was studied in Princeton sandy loam soil. Imazapyr was applied to the soil at the rate of 1.12 kg ai/ha and kept under aerobic conditions in the dark (Mallipudi, 1983: IZ-620-001). At sampling intervals of 1, 2, 4, 6, 9 and 12 months, [^{14}C] carbon dioxide collected was 2.0, 4.0, 7.2, 9.8, 11.2 and 13.6% of the applied dose, respectively. Aerobic microbes present in the soil slowly decarboxylated the compound imazapyr. No other ^{14}C volatile materials were collected at any given interval during the course of the experiment, indicating volatilization of the parent compound or metabolites had not occurred.

Table 10 Soil characteristics

Parameters	Soil (Princeton)
pH (water)	5.3
Organic matter (%)	1.8
Cation exchange capacity (meq/100 g soil)	8.5
Soil type (USDA) Clay (%) Silt (%) Sand (%)	Sandy loam 10.4 30.8 58.8
Moisture at 1/3 bar (%)	14.4
Bulk density on dry soil (g/cc)	1.3

At sampling intervals of 1, 2, 4, 6, 9 and 12 months, the ^{14}C residues remaining in the soil were 96.4, 95.8, 95.2, 90.8, 89.3 and 86.0% of the applied dose, respectively. The rate constant and the half-life of imazapyr under laboratory conditions were calculated as 0.041/month and 17 months, respectively. The extractable soil ^{14}C residue at 1 to 12 months intervals was characterized as imazapyr by TLC and mass spectrometry. The unextractable soil-bound radioactive residues varied from 6.6 to 14.0% of the total soil radioactive residue during the 12-month period. ^{14}C labelled CO_2 was the only major metabolite detected using [carboxyl-^{14}C]-imazapyr. No marked changes were found in the total bacterial and mould counts on untreated and treated soil with imazapyr, indicating that soil aerobic microflora was active throughout the experimental period and imazapyr has no lethal effects on soil aerobic microflora.

Study 2

The aerobic soil metabolism of [carboxyl-^{14}C]-imazapyr was studied in North Dakota clay loam soil. Imazapyr was applied to the soil at the rate of 1.12 kg ai/ha and kept under aerobic conditions at 22 to 24 °C in the dark (Mallipudi, 1985: IZ-620-002). At sampling intervals of 1, 2, 4, 6, 9 and 12 months, the ^{14}C residues remaining in the soil were 95.1, 91.3, 94.5, 79.0, 83.8 and 79.8%, while [^{14}C] carbon dioxide collected was 2.8, 3.2, 7.3, 13.4, 14.5 and 15.4% of the applied dose, respectively. No other ^{14}C volatile materials were collected at any given interval during the course of the experiment, indicating volatilization of the parent compound or metabolites had not occurred.

Table 11 Soil characteristics

Parameters	Soil (North Dakota)
pH (water)	7.5
Organic matter (%)	2.2
Cation exchange capacity (meq/100 g soil)	50.7
Soil type (USDA) Clay (%) Silt (%) Sand (%)	Clay loam 34.0 42.8 23.2
Moisture at 1/3 bar (%)	46.6
Bulk density on dry soil (g/cc)	1.01

The carboxyl [^{14}C] imazapyr found in soil at the end of the 12-month interval was 60.5% of the applied radioactive dose. At the same time interval, some minor metabolites were identified as PDC (0.3%), CL 60,032 (0.6%) and CL 252,974 (5.4%). There were about six other unknowns comprising 3.0% of the applied dose. No attempt was made to identify these minor unknowns. The unextractable soil-bound radioactive residues varied from 6.0 to 11.2% of the total applied dose during the 12-month period. The amount of ^{14}C residues remaining in the aqueous phase and humic acid were very small at all time intervals. Based on the recovered imazapyr in the soil extract, the rate constant and the half-life of imazapyr under laboratory conditions were calculated as 0.019/month and 37.2 months, respectively, which is longer than the sandy loam soil half-life of 17 months.

Table 12 Distribution of radioactivity in the NaOH trap and in the soil fraction

Time (months)	% of Total Applied Dose		
	NaOH trap	Soil	Total
1	2.8	95.1	97.9
2	3.2	91.3	94.5
4	7.3	94.5	101.8
6	13.4	79.0	92.4
9	14.5	83.8	98.3
12	15.4	79.8	95.2

Table 13 Distribution of [^{14}C] Imazapyr and Metabolites in the soil extracts

Time (months)	% of Total Applied Radioactivity				
	Imazapyr	CL 252,974	CL 60,032	PDC	Others
1	77.6	4.9	0.4	–	2.1 (4) [a]
2	71.8	4.6	0.3	–	1.7 (3)
4	73.9	5.0	0.7	–	1.9 (3)
6	64.4	3.4	0.4	–	1.6 (4)
9	69.0	4.4	0.3	0.2	1.1 (3)
12	60.5	5.4	0.6	0.3	3.0 (6)

[a] number of unknowns metabolites

– = Not detected

Study 3

The aerobic soil metabolism of [^{14}C] imazapyr was studied in Princeton sandy loam soil. The [^{14}C] imazapyr labelled at the pyridine ring was dissolved in acetonitrile and applied to the soil at the rate of 1.68 kg ai/ha and kept under aerobic conditions at 35 °C in the dark (Mangles, 1988: IZ-620-036). Water was added to the soil to bring the moisture of the soil to 75% of field capacity. At intervals of 0, 30, 60, 122, 181, 272 and 365 days after treatment of soil aerobically with [^{14}C] Imazapyr were extracted. At the same intervals, the ethylene glycol, sulphuric acid and potassium hydroxide traps were assayed for the ^{14}C-volatile materials. The soil was extracted with 0.1–0.5 N aqueous sodium hydroxide and the humic acid was removed by acid precipitation. The [^{14}C] imazapyr from the

aqueous phase was extracted with dichloromethane. The solvent was concentrated and analysed by TLC. The soil was combusted to determine the bound residues.

Table 14 Soil characteristics

Parameters	Soil (Princeton)
pH (water)	5.3
Organic matter (%)	2.0
Cation exchange capacity (meq/100 g soil)	7.3
Soil type (USDA) Clay (%) Silt (%) Sand (%)	Sandy loam 10.0 22.0 68.0
Moisture at 1/3 bar (%)	9.5
Bulk density on dry soil (g/cc)	1.49

After 12 months of aerobic incubation 89.3% of the initial dose applied remained as parent compound. Two unknown degradates, none of which accounted for greater than 0.8% of the applied dose, were observed intermittently during the study. None of these intermittent products co-chromatographed with the five reference standards used (PDC, CL 60,032, CL 247,087, CL 247,271 and CL 252,974). A half-life of 7.5 years was calculated.

Table 15 Characterization of Non-volatile [^{14}C] Imazapyr

Time	% of Total Applied Radioactivity						
(days)	Imazapyr	UK 1	UK 2	UK 3	UK 4	UK 5	Remainder
0	98.0	–	–	–	–	–	0.4
30	98.7	–	–	–	–	–	0.9
60	95.8	–	–	0.3	0.6	–	2.7
122	94.6	–	–	–	0.8	–	2.7
181	93.9	–	–	–	–	–	2.5
272	92.2	–	–	–	–	–	2.3
365	89.3	–	–	–	–	–	3.0

UK = unknown compound

– = Not detected

Study 4

The aerobic soil metabolism of [^{14}C] imazapyr was studied in Princeton sandy loam soil. The [^{14}C] imazapyr labelled at the pyridine ring was dissolved in acetonitrile and applied to the soil at the rate of 1.68 kg ai/ha and kept under aerobic conditions at 25 °C in the dark (Mangels, 1987: IZ-620-037). Water was added to the soil to bring the moisture of the soil to 75% of field capacity. At intervals of 0, 30, 60, 122, 181, 272 and 365 days after treatment of soil aerobically with [^{14}C] imazapyr were extracted. At the same intervals, the ethylene glycol, sulphuric acid and potassium hydroxide traps were assayed for the [^{14}C] volatile materials. The soil was extracted with 0.1–0.5 N aqueous sodium hydroxide and the humic acid was removed by acid precipitation. The [^{14}C] imazapyr from the aqueous phase was extracted with dichloromethane. The solvent was concentrated and analysed by TLC. The extracted soil was combusted to determine the bound residues.

Table 16 Soil characteristics

Parameters	Soil (Princeton)
pH (water)	5.3
Organic matter (%)	2.0
Cation exchange capacity (meq/100 g soil)	7.3
Soil type (USDA) Clay (%) Silt (%) Sand (%)	Sandy loam 10.0 22.0 68.0
Moisture at 1/3 bar (%)	9.5

Parameters	Soil (Princeton)
Bulk density on dry soil (g/cc)	1.49

After 12 months of aerobic incubation 87.6% of the initial dose applied remained as parent compound. Five unknown degradates, none of which accounted for greater than 1.3% of the applied dose, were observed intermittently during the study. None of these intermittent products co-chromatographed with the five reference standards used (PDC, CL 60,032, CL 247,087, CL 247,271 and CL 252,974). A half-life of 5.9 years was calculated.

Table 17 Characterization of Non-volatile [^{14}C] Imazapyr

Time	% of Total Applied Radioactivity						
(days)	Imazapyr	UK 1	UK 2	UK 3	UK 4	UK 5	Remainder
0	99.2	0.2	0.1	–	–	–	0.5
30	98.8	0.2	0.1	–	–	–	0.6
60	96.7	–	–	–	–	–	3.0
122	93.3	–	–	0.4	0.3	1.3	3.3
181	93.5	–	–	–	–	–	3.6
272	91.8	–	–	–	–	–	3.1
365	87.6	–	–	–	–	–	5.4

UK = unknown compound

– = Not detected

Study 5

The aerobic soil metabolism of [^{14}C] imazapyr was studied in Princeton Sassafras sandy loam soil. [pyridine-6-^{14}C]-imazapyr was dissolved in water and applied to the soil at the rate of 0.25 kg ai/ha and kept under aerobic conditions at 25 °C in the dark (Ta, 1999: IZ-620-053). Water was added to the soil to bring the moisture of the soil to 75% of 1/3 bar. At intervals of 0, 7, 14, 21, 28, 62, 94 and 121 days after treatment of soil aerobically with [^{14}C] imazapyr were extracted. At the same intervals, the ethylene glycol and sodium hydroxide traps were assayed for the ^{14}C-volatile materials. The soil was extracted once with water and three times with 0.5 N aqueous sodium hydroxide. After extraction, the soils were washed with 100 mL of water, followed by 100 mL of methanol. The water and NaOH extracts were cleaned up and concentrated by C_{18} and SCX solid phase extraction then analysed by TLC, HPLC and HPLC-MS. The extracted soil was combusted to determine the bound residues.

Table 18 Soil characteristics

Parameters	Soil (Princeton)
pH (water)	6.0
Organic matter (%)	2.0
Cation exchange capacity (meq/100 g soil)	7.3
Soil type (USDA) Clay (%) Silt (%) Sand (%)	Sandy loam 10.0 22.0 68.0
Moisture at 1/3 bar (%)	14.9

Approximately 26% of the imazapyr was degraded after 121 days of aerobic incubation. The first-order degradation half-life was calculated to be 313 days. The rate of degradation of imazapyr decreased after 1 month of incubation and appeared to be a biphasic pattern with transition point at approximately 28 days. Using the first month degradation data only, the half-life of imazapyr in soil was calculated to be 117 days. The half-life in the second phase was 438 days. Degradation of imazapyr proceeded via the opening of the imidazolinone ring to yield the metabolite CL 252,974 which accounted for approximately 2% of the applied dose at 4 months of incubation. Degradation of imazapyr can also proceed via the conversion of the carboxylic acid group at the 3 position in the

pyridine ring to form the hydroxyl metabolite (CL 288,247) which reached approximately 6% of the applied dose at 4 months of incubation. The metabolites were identified by co-chromatography (TLC and HPLC) with authentic standard and by mass spectrometry analysis.

Table 19 Distribution of Imazapyr and metabolites in soil (analysed by HPLC)

Time (days)	% of Total Applied Radioactivity							
	Imazapyr	CL288,247	CL252,974	CL240,000	CO_2	Others [a]	Bound Residue	Total
0	94.4	0.5	1.6	1.1	0.0	2.7	0.1	100.4
7	86.4	1.3	3.1	2.1	0.8	3.5	2.1	99.3
14	83.9	1.2	2.4	1.5	1.5	5.2	2.9	98.6
21	81.3	1.7	2.8	1.5	2.1	6.4	3.2	99.0
28	78.9	1.9	2.8	1.3	2.5	6.8	3.8	98.0
62	75.1	4.5	1.5	1.6	4.0	6.7	4.8	98.2
94	70.5	5.7	2.0	1.9	4.9	6.2	5.9	97.1
121	67.6	6.4	2.1	2.3	5.6	6.6	6.1	96.7

[a] Consist of several unknown components

Approximately 5.6% of the total applied radioactivity was evolved as $^{14}CO_2$ over the 4 months of incubation indicating that the pyridine ring was mineralized. No organic volatiles were detected in the glycol traps. Results showed that imazapyr is aerobically degraded in soil and is ultimately mineralized to CO_2.

Soil photolysis

The photolysis of ^{14}C labelled imazapyr was studied on sandy loam soil. [pyridine-6-^{14}C]-imazapyr was dissolved in acetonitrile and applied to the soil surface at a rate equivalent to 1.68 kg ai/ha, then exposed continuously for 28 days to light from a borosilicate-filtered Xenon-arc lamp in a custom-made environmental chamber (Mangels, 1986: IZ-620-010). The soils were irradiated approximately 80 cm from the light, which was operated continuously at 6000 watts and 25 ^{o}C. The light produced by the lamp is comparable to summer sunlight in Chicago, Illinois.

Table 20 Recoveries of radioactivity (% of total applied dose)

Time (days)	Sample	% Recovery [a]
0		100.0
7	Irradiated	98.7
	Control	101.0
14	Irradiated	104.5
	Control	106.5
21	Irradiated	106.4
	Control	105.5
28	Irradiated	99.5
	Control	105.2

[a] Mean of two replicates

Table 21 Distribution of radioactivity (% of total applied dose)

Time (days)	sample	% of total applied dose [a]		
		Imazapyr (organic phase)	Imazapyr (aqueous phase)	Total
0		95.3	2.5	97.6
7	Irradiated	92.1	2.4	94.3
	Control	93.3	2.5	95.3
14	Irradiated	86.3	2.1	87.9
	Control	93.8	1.7	95.2

Time (days)	sample	% of total applied dose [a]		
		Imazapyr (organic phase)	Imazapyr (aqueous phase)	Total
21	Irradiated	85.1	2.0	86.7
	Control	93.5	1.8	94.9
28	Irradiated	84.7	2.4	86.5
	Control	94.7	2.2	96.5

[a] Mean of two replicates

There was 11% degradation of imazapyr over the 28 days of continuous irradiation. There were at least five degradation products formed, none of which accounted for > 10% of the applied dose. The photodegradation half-life of imazapyr under the conditions of this test was calculated to be 149 days.

Residues in rotational crops

Confined rotational crop studies

Study 1

The field phase of the confined rotational crop study was conducted in North Carolina and initiated in the summer of 1993 (Zulalian, 1995: IZ-640-004). The study consisted of one treated and one control plot. For the treated plot, pyridine-6-^{14}C-labeled imazapyr was mixed with [pyridine-6-^{13}C]-imazapyr to afford a final specific activity of 20.8 µCi/mg. The radioactive test substance, formulated as an aqueous ammonium salt solution, was applied as a post-emergence application to IMI-Maize plants (Pioneer hybrid 3245 IR) at the 6-leaf stage at a rate of 0.028 kg ai/ha. The IMI-Maize plants in the control plot were treated with the blank formulation. The final specific activity of the radiolabelled imazapyr afforded a detection limit of 0.002 mg/kg in the radio-assay of soil and plant samples. Soil core samples were taken prior to application (–1 DAT), within two hours following application (0 DAT) and at final harvest (86 DAT) of the mature maize plants.

Winter wheat (cereal grain) was planted 120 DAT. Soya beans (legume), lettuce (leafy vegetable), and radishes (root crop) were planted 271 DAT. Lettuce and radishes were also planted 420 DAT. Samples were collected from the respective plots at immature and mature stages of crop development. The total radioactive residues (TRR) were determined by combustion and liquid scintillation counting. The TRR in winter wheat forage, straw and grain; lettuce; radish foliage and root; and soya bean forage, hay, and seed were all < 0.002 mg/kg, the limit of detection of the radioassay.

Table 22 Total radioactive residues (TRRs) found in crops for confined rotational crop study

Sample		Planting Interval (DAT)	Harvest Interval (DAT)	TRR (mg/kg)
Wheat (Pioneer 2548)	Mid Harvest Forage	120	278	< 0.002
	Final Harvest Straw		337	< 0.002
	Final Harvest Grain		337	< 0.002
Lettuce (Black Seeded Simpson)	Mid Harvest	271	316	< 0.002
	Final Harvest		328	< 0.002
Lettuce (Buttercrunch)	Mid Harvest	420	457	< 0.002
	Final Harvest		474	< 0.002
Radish (White Icicle)	Mid Harvest Foliage	271	306	< 0.002
	Mid Harvest Root		306	< 0.002
	Final Harvest Foliage		321	< 0.002
	Final Harvest Root		321	< 0.002
Radish (White Icicle)	Mid Harvest Foliage	420	449	< 0.002
	Mid Harvest Root		449	< 0.002
	Final Harvest Foliage		461	< 0.002
	Final Harvest Root		461	< 0.002
Soya bean	Mid Harvest Forage	271	356	< 0.002

Sample		Planting Interval (DAT)	Harvest Interval (DAT)	TRR (mg/kg)
(Hartz 6686)	Final Harvest Hay		484	< 0.002
	Final Harvest Seed		484	< 0.002

DAT = days after treatment

The TRRs in soil (0–8 cm) at 0 DAT was 0.013 mg/kg. At the time of maize harvest (86 DAT) the residue in the soil (0–8 cm) was 0.004 mg/kg. Residues were < 0.002 mg/kg in deeper soil layers (8–15 cm, 15–30 cm and 15–46 cm).

In summary, the results of this study indicate that residues in rotational crops would be expected to be < 0.01 mg/kg at the recommended planting intervals following a post-emergence application of imazapyr on IMI-Maize at a maximum label rate of approximately 0.028 kg ai/ha.

Study 2

An outdoor confined rotational crop study with ^{14}C-labelled imazapyr was conducted at Lucama, North Carolina during the year 1997–1999 (Mallipudi, 2000: IZ-640-008). The study was designed to determine the nature and amount of imazapyr-related residue uptake in rotational crops. A leafy vegetable, root crop and small grain were planted at various time intervals after a single application of [^{14}C] imazapyr to the soil at a rate of 0.885 kg ai/ha. [pyridine-6-^{14}C]-imazapyr was used in the study. The position of the ^{14}C label was considered metabolically stable to allow determination of the metabolic profile in the confined rotational crops. The radiolabelled imazapyr-spray mixture was formulated as a liquid end-use product in water. The control plot was treated with a blank formulation and water mixture without test substance.

Five months after treatment with the test substance, the test and control plots were planted with annual weeds and maintained the plots with weeds until the designated rotational crop planting time. The treated test plot and control plot were planted with a series of rotational crops, namely carrot and lettuce were planted at 330 and 540 days after treatment (DAT), winter wheat planted at 359 DAT, spring wheat at 520 DAT. Confined rotational crop samples were collected from the respective plants at immature and mature stages of crop development. Soil core samples were taken to a depth of approximately 30 to 46 cm prior to test substance application, immediately following application, and at each rotational crop replant and harvest. No soil samples were taken for carrot and lettuce planted at 330 DAT.

Before analysis, individual crop samples were homogenized by grinding with dry ice. Soil samples were air dried and homogenized by grinding. Total radioactive residues (TRR) in all plant parts and in soil were determined by combustion of aliquots to yield $^{14}CO_2$. LSC counting was used to analyse trapped $^{14}CO_2$. The LSC techniques were used to analyse fractions from HPLC. The identity of the extractable residue component was determined by chromatography with unknown reference compounds using HPLC.

TRR levels were less than 0.01 to 0.02 mg/kg in different crop samples at various plant-back intervals. TRR values in control samples were all less than the validated minimum quantifiable limit of detection of 0.005 mg/kg. A summary of the TRR values in the treated crop samples is shown below.

Table 23 Total radioactive residues (TRRs) found in crops for confined rotational crop study

Sample		Planting Interval (DAT)	Harvest Interval (DAT)	TRR (mg/kg)
Winter wheat	forage	359	514	< 0.005
	hay		582	0.005
	straw		596	0.006
	grain		596	0.007
Spring wheat	forage	520	574	< 0.005
	hay		590	0.018
	straw		623	0.015

Sample		Planting Interval (DAT)	Harvest Interval (DAT)	TRR (mg/kg)
	grain		623	0.016
Carrot	mature top	330	427	< 0.005
	mature root			< 0.005
Carrot	mature top	540	646	0.007
	mature root			< 0.005
Lettuce	mature	330	391	< 0.005
Lettuce	mature	540	623	0.009

Significant levels of soil radioactive residues were present throughout the experiment period. TRR in the top zero to 8 cm soil layer were 0.472 mg/kg at 0 days after treatment (0 DAT). Residues in the top 8 cm of soil ranged from 0.022 to 0.028 mg/kg up to 646 DAT during which time various crops were grown. The soil TRR ranged between 0.016 to 0.023 mg/kg in the 8 to 15 cm soil core during the same period. During the same time interval, the soil TRRs were 0.029 to 0.051 mg/kg in 15 to 30 cm soil layer and 0.018 to 0.036 mg/kg in 15 to 46 cm soil layer. TRR levels in the control (untreated) soil core samples were all less than the validated limit of detection (0.005 mg/kg).

The radioactivity in the various rotational crop matrices was moderately extractable (ranging from about 23.6% for the winter wheat hay to 51.1% for lettuce) by aqueous acetone. An additional amount of radioactivity (about 5.0% for the spring wheat grain to 15.0% for the winter wheat grain) was extracted with 2% acidified aqueous acetone. Exhaustive extraction and digestion procedure using acetone: water: HCl (3%), 6 N aqueous HCl and 6 N aqueous NaOH yielded additional 3.1% to 18.7% of TRR (< 0.001 to 0.002 mg/kg) in various fractions from different crops. The unextractable radiocarbon in various solids after solvent extraction of crop samples ranged from 12.7% to 71.2% of TRR (0.001 to 0.004 mg/kg).

Table 24 Extraction efficiency of residues of imazapyr in rotational crops

Sample		Harvest DAT	TRR (mg/kg)	Extractable (%TRR)			Unextracted (PES)	
				Aqueous Acetone [a]	Acetone: H_2O:HCl [b]	Total	mg/kg	%TRR
Winter wheat	hay	582	0.005	23.6	7.0	30.6	0.004	71.2
	grain	596	0.007	31.0	15.0	46.0	0.006	80.1
Spring wheat	hay	590	0.018	37.8	10.9	48.7	0.009	51.1
	straw	623	0.015	28.0	5.5	33.5	0.010	67.2
	grain	623	0.016	47.9	5.0	52.9	0.007	46.2
Carrot	mature top	646	0.007	49.8	7.7	57.5	0.003	45.2
Lettuce	mature	623	0.009	51.1	9.9	61.0	0.004	48.1

DAT = days after treatment

[a:] extracted with acetone:H_2O (1:1)

[b:] extracted with 2% HCl (acetone:H_2O.HCl 49:49:2)

Table 25 Distribution of radioactivity in fractions extracted from post extraction solid (PES) of rotational crops

Sample	Harvest DAT	TRR (mg/kg)	Residue in crop PES		Residue Fraction	Residue in crop	
			mg/kg	%TRR		mg/kg	%TRR
Winter wheat grain	596	0.007	0.006	80.1	6 N aqueous HCl	0.001	12.7
					6 N aqueous NaOH	0.001	18.7
					PES (unextractable)	0.001	19.3
Spring wheat straw	623	0.015	0.010	67.2	Aqueous:Acetone:HCl (3%)	0.002	15.0
					6 N aqueous HCl	0.002	15.1
					6 N aqueous NaOH	0.002	12.1
					PES (unextractable)	0.003	19.2
Spring wheat grain	623	0.016	0.007	46.2	Aqueous:Acetone:HCl (3%)	0.002	9.8
					6 N aqueous HCl	0.002	13.2
					6 N aqueous NaOH	0.001	8.4

Sample	Harvest DAT	TRR (mg/kg)	Residue in crop PES		Residue Fraction	Residue in crop	
			mg/kg	%TRR		mg/kg	%TRR
					PES (unextractable)	0.002	12.7
Lettuce	623	0.009	0.004	48.1	Aqueous:Acetone:HCl (3%)	0.001	7.6
					6 N aqueous HCl	0.001	13.2
					6 N aqueous NaOH	< 0.001	3.1
					PES (unextractable)	0.002	22.4

DAT = days after treatment

Analysis of the aqueous acetone and aqueous acetone: HCl (2%) fractions from various crop parts by HPLC was possible. The results showed that the major fraction comprised of unchanged imazapyr, which ranged from < 0.001 to 0.003 mg/kg in various crops. Chromatographic region of interests corresponded to CL 247,087, CL 252,974, CL 17,226 and CL 119,060 were each at less than 0.001 mg/kg by calculations.

When [pyridine-6-^{14}C]-imazapyr was applied to bare sandy loam soil at a use rate of 0.885 kg ai/ha (acid equivalents) radioactivity in soil was present throughout the experiment period. A trace amount of imazapyr-related residue in soil was transported into leafy vegetable, root crop and small grain. Total radioactive residues in follow crops were at < 0.01 to 0.02 mg/kg at the various plant back intervals: Winter wheat at 359 days, spring wheat at 520 days, and carrot and lettuce at 330 and 540 days.

Attempts to elucidate the metabolic profile of imazapyr in rotational crops showed terminal residue in the rotational crops included the unchanged imazapyr which ranged from < 0.001 to 0.003 mg/kg. Metabolites CL 247,087, CL 252,974, CL 17,226, and CL 119,060, each at 0.001 mg/kg or less were present. Thus, it appeared that imazapyr was metabolized in the rotational crops by hydrolysis of imidazolinone ring and cyclization of the carboxyl group and imidazolinyl nitrogen.

Environmental fate in water systems

Hydrolysis

Study1

The [puridine-6-^{14}C]-imazapyr was dissolved in water and applied in to the water layer at an application rate of 1.5 mg/kg (Mangels, 1990: 1990/7001955). Samples were incubated at 25 °C under aerobic conditions in the dark. After various time intervals (0, 1, 2, 3 and 4 weeks), the water samples were directly, analysed by LSC and by TLC two-dimensional TLC upon comparison with authentic standards. Since there was less than 2% of the radioactivity presented in the sediment, no extraction was performed and the radioactivity in the sediment was determined by combustion.

Greater than 98% of the applied radioactivity (AR) was recovered. A total of 1.12% AR mineralized to $^{14}CO_2$ during four weeks of aging and no volatile organic compounds were detected. Greater than 95% of the applied imazapyr remained in the water layer. Less than 2% of the applied radioactivity was detected in the sediment. No measurable degradation occurred during the 1 month experiment indicating imazapyr is stable under aerobic aquatic conditions. In conclusion, imazapyr is stable under aerobic aquatic conditions.

Study 2

The [carboxyl-^{14}C]-imazapyr was dissolved in water to give stock solutions containing 50 mg/L of [^{14}C] imazapyr (Mangels, 1990: 1990/7001950). Aliquots were placed into flasks and diluted in aqueous buffer solutions of pH 5 (0.01 M citrate), pH 5.2 (Distilled water), pH 7 (0.01 M phosphate) and pH 9 (0.01 M borate). Thereafter, aliquots duplicate samples containing each approximately 50 µg/mL were placed into test tubes and incubated in the dark at a controlled temperature of at 25 °C for 30 days. After various time intervals (0, 2, 12, 19, 26 and 30 days), the samples were directly, analysed by LSC and by TLC two-dimensional TLC upon comparison with authentic standards.

The recovery of radioactivity in all samples was between 92.3% and 107.0% of the total applied radioactivity. There was no detectable degradation of imazapyr in distilled water, pH 5 or pH 7 buffers. In pH 9 buffer, there was a slow degradation to a single product which reached a maximum level of 6.9% of the applied dose 30 days after treatment. The degradate was identified as CL 252,974.

Half-life =

pH 5	pH 7	pH 9	Distilled water
NDD	NDD	325 days	NDD

NDD = No Detectable Degradation after 30 days

In conclusion, imazapyr is hydrolytically stable in distilled water (pH 5.2) pH 5, 7 and 9.

RESIDUE ANALYSIS

Analytical methods

Descriptions of analytical methods together with validation data for residues of imazapyr in plant and animal matrices were submitted to the Meeting. The methods rely on an initial extraction, usually with acetonitrile/water. After solvent partition and column cleanup, the imazapyr and metabolites residues are prepared for LC analysis. Imazapyr residues can be measured either by UV detector or mass selective detector (MS), typically to an LOQ of 0.01 mg/kg. Determinations for the metabolites were conducted using HPLC with mass spectrometric detector (MS/MS). The LOQ for the metabolites were typically 0.01 mg/kg. Since the methods use standard extraction solvents and standard detection techniques, they have the potential to be incorporated into existing multi-residue methods.

Brief descriptions of all these analytical methods are presented below.

Plant matrices

Imidazolinone-tolerant sunflower and rape (seeds, meal and refined oil) (2008/7019225, 2008/7019227)

Analyte:	Imazapyr (m/z 262→217 for quantification, 262→149 for confirmation)	LC-MS/MS	M 3519 modified
LOQ:	0.05 mg/kg		
Description	Samples are extracted with acidic aqueous methanol. An aliquot of the extract is diluted with methanol then passed through a SCX cartridge, which retains the analyte. The SCX cartridge is washed with methanol to remove co-extractives then the residues are selectively eluted with a water-methanol solution. The eluate is evaporated to dryness and the residues are dissolved in acidic water for analysis. Residue of imazapyr is determined by LC-MS/MS.		

Soya bean (grain, flaked, meal, toasted meal and oil) (2010/1033332)

Analyte:	Imazapyr (m/z 262→217 for quantification, 262→149 for confirmation)	LC-MS/MS	SOP-PA.0288
LOQ:	0.01 mg/kg		

Description 5 g ± 0.1 of sample are weighed and 50 mL of extraction solution (methanol/water/HCl 1 mol/L, 60/39/1, v/v/v) is added. A mechanical shaker is used for 10 minutes to extract the analytes. The extract is centrifuged for 5 minutes at 3000 rpm and an aliquot of 1 mL is taken into a 5 mL volumetric flask. The volume is filled to the mark with solution 1. The solution is filtered, when necessary, and injected in the liquid chromatographic system, mass/mass spectrometer (LC-MS/MS).

Solution 1: 0.1% formic acid in water/0.1% formic acid in methanol (1/1, v/v)

Soya bean (grain, flaked, meal, toasted meal and oil) (2010/1090461)

Analyte:	Imazapyr (m/z 262→217 for quantification, 262→149 for confirmation)	LC-MS/MS	SOP-PA-0288_E

LOQ: 0.01 mg/kg

Description The residues of imidazolinones and its metabolites are extracted from samples using extraction solution (methanol/water/1 M HCl 60/39/1, v/v/v) in a homogenizer for 5 minutes. The addition of HCl provides additional extraction capability for the analytes with acidic properties. Centrifugation is done whenever it is necessary for 5 minutes at 2000 rpm. For sunflower (seed or oil) shaking is to be done in a mechanical shaker for 10 minutes. A 1 mL aliquot is taken and complete to the mark with solution 1 in a 5 mL volumetric flask. Filter it in a filter unit, when necessary. The residues of imidazolinones and its metabolites are performed by LC-MS/MS and quantified by directive comparison with external standards.

Solution 1: 0.1% formic acid in water/0.1% formic acid in methanol (1/1, v/v)

Sunflower (seed, meal and refined oil) (2002/5004111)

Analyte:	Imazapyr	LC-MS

LOQ: 0.05 mg/kg

Description Residues of imazapyr are extracted from sunflower seed and meal samples with an acidified methanol/water solution, filtered, and concentrated. The residues are purified on a C_{18} solid phase extraction (SPE) column eluted with 25% MeOH in 0.05 M aqueous ammonium acetate and brought to final volume with water. Residues in refined oil samples are diluted in hexane and extracted by shaking with acidified acetonitrile/water solution. Following phase separation, the lower aqueous ACN layer is collected and adjusted to final volume with water. The final chromatography analysis of imazapyr residues is performed using LC-MS.

Sunflower (seed, flower) (2002/3000641)

Analyte:	Imazapyr	LC-UV (254 nm)	SOP-PA.0200, Rev.02-RE.01

LOQ: 0.05 mg/kg

Description: Residues of imazapyr are extracted from sunflower seed and flower samples with acidic aqueous methanol. The extract is filtered, and an aliquot is taken and concentrated. After adding water and acetate, the pH is adjusted to 6.2 with 1 N NaOH solution. Following centrifugation, an aliquot of the upper layer is taken and the pH is then adjusted to 2.1 with 1 N HCl solution. The sample is then partitioned four times with dichloromethane (DCM). The DCM layers are combined and concentrated to dryness. The residue is re-dissolved in methanol-water and passed through an SCX cartridge. The SCX cartridge is washed with methanol to remove co-extractives, and then the residue is selectively eluted with KCl in methanol. The residue is then evaporated to dryness and re-dissolved in acidic water (pH 2.5). The samples are partitioned again four times with DCM and concentrated to dryness. The final extract is dissolved in water for HPLC analysis. A liquid chromatograph (HPLC) equipped with a diode array detector (DAD) set at 254 nm is used for detection.

Soya bean (seed) (2010/1149746), Rice (grain) (2010/1141978)

Analyte:	Imazapyr (m/z 262→217 for quantification, 262→149 for confirmation)	LC-MS/MS	SOP-PA.0249
LOQ:	0.05 mg/kg		
Description:	The sample is weighed into an extraction container. 100 mL of 1 N methanol/water/HCl (60:39:1 v/v/v) are added and extracted in the homogenizer for 5 minutes or in the extraction bath for 20 minutes. The extract is centrifuged for 20 minutes at 2000 rpm and an aliquot of 20 mL is transferred to a concentration flask. The volume is reduced to approximately 7 mL. The extract is transferred to a volumetric flask and filled up to the mark with Milli-Q water. The pH is adjusted to 2.1 with HCl 1 N solution. An aliquot of 2 mL is transferred to a centrifuge tube. 10 mL of dichloromethane are added. Partitioning is performed for 1 minute, followed by centrifugation for 5 minutes to separate the phases. An aliquot of 4 mL of the organic phase (lower phase) is evaporated to dryness. The residue is dissolved in 2 mL of mobile phase A / mobile phase B (1/1, v/v) and injected into the chromatographic system, MS/MS detector. Mobile phase A: 0.1% formic acid in water, Mobile phase B: 0.1% formic acid in methanol		

Maize (grain, forage and fodder) (IZ-731-001, IZ-244-006, IZ-244-005)

Analyte:	Imazapyr	GC-MS	M 2468
LOQ:	0.05 mg/kg		
Description:	The sample is weighed into an extraction flask and 200 mL of acidic acetone/water (1:3 v/v) is added. The extract solution is allowed to soak for 15 minutes, and then blended at medium speed for approximately 5 minutes using an Omni mixer. After mixing, 10 g Celite 545AW are added to the extract and the solution is swirled. The extract is then vacuum-filtered using a glass fibre filter on a Buchner funnel. A 2 mL aliquot of the extract is transferred into a flask and evaporated to near dryness on a rotary evaporator. The extract is then cleaned-up by solid-phase extraction on a Varian Bond Elut C18 cartridge using Milli-Q water, hexane and methylene chloride as wash solvents. After evaporation to dryness, the residue is re-dissolved in 1 mL methanol and sonicated, then transferred to an auto-injector vial for GC-MS analysis. Determination is done at m/z 289 in the negative ion monitoring mode.		

Maize (grain) (IZ-731-021, 1997/7004635 and 1999/7004025)

Analyte:	Imazapyr	LC-UV (254 nm)	M-LAADL R0001
LOQ:	0.05 mg/kg		
Description:	Residues are extracted with acidic aqueous methanol using a Polytron ultrasonic extractor. Residues are partitioned with methylene chloride from concentrated aqueous acid solution. The methylene chloride is evaporated and the remaining material is reconstituted in 50% methanol in pH 2.5 for SPE SCX—Benzensulfonic acid (Strong Cation Exchange) clean-up. The residue is eluted from the SCX with a saturated KCl in methanol, partitioned with methylene chloride, evaporated to dryness and diluted in 2 mL of Milli-Q water for HPLC analysis.		

Sugarcane (juice and bagasse) (IZ-790-015 and IZ-790-017)

Analyte:	Imazapyr	LC-UV (254 nm)	M-LAADL R0002
LOQ:	0.05 mg/kg		

Description: Residues are extracted from sugar cane or sugar cane juice with acetone/methanol/water (1/1/1, v/v/v). The extracts are subjected to suitable clean-up involving solid phase extraction (Ag 1-X8 and SCX cartridge). The determination of imazapyr residues in the final extracts is performed by HPLC-UV (juice) or HPLC-DAD (cane) at 254 nm. Residue concentrations are calculated by direct comparison to external standards.

Rape (seed) (1998/1008995)

Analyte:	Imazapyr	LC-UV (254 nm)	L741/1

LOQ: 0.05 mg/kg

Description: Residues are extracted from samples with methanol/HCl/water (60:1:39, v/v/v). The extract is partitioned with dichloromethane after adjusting the pH to 2 with HCl. Subsequently, the extract is evaporated to dryness and the residue dissolved in methanol/water (1:1, v/v). Following SCX cartridge clean-up, separation and quantitation of imazapyr is accomplished by reverse phase high performance liquid chromatography (HPLC).

Maize (grain, forage, fodder) (IZ-244-009)

Analyte:	Imazapyr	GC-MS

LOQ: 0.05 mg/kg

Description: After soaking for 15 minutes imazapyr is extracted from the sample with 200 mL of acetone/water/HCl (25:74:1 v/v/v) at medium speed in an Omni-Mixer for 5 minutes. The extract is mixed with 10 g of Celite 545 AW and filtered. A 2 mL aliquot of the filtrate is taken, the acetone is evaporated and the remaining extract is transferred to a C18-cartridge. Imazapyr is eluted from the cartridge with CH_2Cl_2. Subsequently, CH_2Cl_2 is evaporated and the residue is dissolved in 1 mL CH_3OH and sonicated for 30 seconds. Of the final extract 0.5 mL are mixed with 50 µL of 0.1 M trimethyl anilinium hydroxide. The determination is performed by GC-MS using ion m/z 289.

Maize (grain, forage, fodder) (IZ-244-008)

Analyte:	Imazapyr	Capillary Electrophoresis	M 2657

LOQ: 0.05 mg/kg

Description: The sample is weighed into an extraction flask and 200 mL of acidic acetone/water (1:4 v/v) are added. The extract solution is allowed to soak for 15 minutes, and then blended using an Omni mixer. After mixing, 10 g Celite 545AW are added to the extract and the solution is swirled. The extract is then vacuum-filtered. An aliquot of the extract is transferred into a separatory funnel. Methylene chloride is added and shaken for 15 seconds. The lower, methylene chloride, layer is drained into a beaker. The aqueous, upper, fraction is partitioned with another 25 mL of methylene chloride, which is added to the previously drained fraction. Solid-phase extraction is done using an Isolute SCX cartridge, the extract allowed to drain through the cartridge by gravity. The cartridge is then washed with acetone, followed by methanol and saturated KCl/methanol. After evaporation to dryness using a rotary evaporator, the residue is re-dissolved in 1% formic acid, then subjected to further clean-up using Spe-ed RP 102 cartridge and washing with methanol and water. After evaporation to dryness, the sample is re-dissolved in 1 mM Tris/1 mM NaH_2PO_4 solution, sonicated for 15 seconds, then transferred to a sample vial for capillary electrophoresis (CE) analysis. The CE system is equipped with a high sensitivity optical flow cell and a UV detector set at 240 nm.

Maize (1999/7004026)

Analyte:	Imazapyr	LC-UV (254 nm)	M 2020

LOQ: 0.05 mg/kg

Description: The sample is weighed into an extraction container. Then, the extraction solution of methanol/water/1 N HCl (60:39:1, v/v/v) is added and blended using an Omni mixer. The extract is filtered, and then Celite 545AW is added. The extraction jar is washed and filtered with extraction solvent. The filtrate is poured into a mixing cylinder, diluted to 250 mL with methanol, mixed and an aliquot is taken. The methanol is evaporated to approx. 50 mL using a flash evaporator. The sample solution is extracted and filtered again, using acetone and Celite, then evaporated to a volume of 10 mL. 1 N HCl is added, the solution filtered and the filter rinsed using methanol and 0.05 N HCl. The solution is partitioned twice, using methylene chloride, before evaporating to dryness. Further partitioning is done using methanol, acetonitrile and hexane. The hexane layer is discarded, the acetonitrile solution evaporated to dryness and the residue dissolved in methanol and 0.05 N HCl. The extract is cleaned-up by solid-phase extraction on a Bond Elut C18 cartridge with deionized water, 0.05 N and 1 N HCl, hexane and methanol as wash solvents. The eluate is discarded, and the system eluted with saturated KCl in methanol. After evaporation to dryness, the residue is re-dissolved in methanol and 0.05 N HCl, and poured into a separatory funnel with methylene chloride. Three more portions of methylene chloride are added for partitioning, then the solution is evaporated to dryness, and partitioning is repeated with methylating reagent and methanol. After re-evaporation to dryness, the residue is dissolved in methanol in preparation for analysis by LC with a UV detector set at 254 nm.

Validation data for methods on plant matrices are summarized in Table 26.

Table 26 Summary of Recovery Data for imazapyr fortified into plant matrices

Commodity		Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Sunflower, seed (CR)		0.05 0.50 5.0	4 3 5	59–86 71–94 75–78	73 81 76	15.3 14.4 1.8	2008/7019225 M 3519 modified
Sunflower, meal (CR)		0.05 0.50	1 1	79 88	–		
Sunflower, refined oil (CR)		0.05 0.50	1 1	112 109	–		
Lentil, seed (CR)		0.05 5.0	3 3	71–77 68–75	74 71	4.1 5.1	2008/7019226 M 3519 modified
Rape, seed (MV) Dilution solution: water/methanol/formic acid	Mass transition 262→217	0.05 0.50	5 5	80–94 74–98	89 86	6.2 10.8	2008/7019227 M 3519 modified
	Mass transition 262→149	0.05 0.50	5 5	74–100 79–102	87 89	11.6 11.1	
Rape, seed (MV) Dilution solution: water/formic/acid	Mass transition 262→217	0.05 0.50	5 5	73–85 76–88	78 84	5.6 5.5	
	Mass transition 262→149	0.05 0.50	5 5	73–90 78–91	81 87	9.4 6.1	
Rape, refined oil (MV) Dilution solution: water/formic acid	Mass transition 262→217	0.05 0.50	6 5	91–102 96–101	96 99	4.5 1.9	
	Mass transition 262→149	0.05 0.50	6 5	92–106 93–105	98 101	5.9 4.9	
Rape, seed (CR) Dilution solution: water/methanol/formic acid		0.05 0.50	4 4	70–89 74–86	79 80	10.6 6.6	
Rape, seed (CR) Dilution solution: water/formic acid		0.05	2	68, 71	69		
Rape, meal (CR) Dilution solution: water/formic acid		0.05 0.50	2 2	81, 83 79, 83	82 81		
Rape, refined oil (CR) Dilution solution: water/formic acid		0.05 0.50	2 2	103, 109 103, 106	106 104		
Soya bean, seed (MV)	Mass transition 262→217	0.01 1.0	5 5	111–119 98–104	114 102	2.8 2.5	2010/1033332 SOP-PA.0288
	Mass transition 262→149	0.01 1.0	5 5	108–119 96–102	114 100	4.1 2.5	

Commodity		Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Soya bean, oil (MV)	Mass transition 262→217	0.01 1.0	5 5	81–90 94–102	86 97	4.2 3.4	
	Mass transition 262→149	0.01 1.0	5 5	86–89 91–104	87 99	1.5 5.3	
Soya bean, flaked (MV)	Mass transition 262→217	0.01 1.0	5 5	107–115 95–102	112 98	2.9 2.7	
	Mass transition 262→149	0.01 1.0	5 5	110–119 97–106	116 100	3.0 3.8	
Soya bean, meal (MV)	Mass transition 262→217	0.01 1.0	5 5	110–120 88–92	113 90	3.4 1.7	
	Mass transition 262→149	0.01 1.0	5 5	93–106 78–89	99 86	4.9 5.1	
Soya bean, toasted meal (MV)	Mass transition 262→217	0.01 1.0	5 5	113–120 82–89	116 86	2.8 3.2	
	Mass transition 262→149	0.01 1.0	5 5	85–111 78–90	98 83	10.3 5.4	
Soya bean, seed (ILV)		0.01 0.50	5 5	76–99 83–96	84 91	11.1 5.9	2010/1090461 2012/7000359
Soya bean, oil (ILV)		0.01 0.10	5 5	91–99 79–91	94 88	3.4 5.8	SOP-PA.0288
Soya bean, hulls (ILV)		0.01 2.0	5 5	72–91 92–99	79 95	10.1 2.9	
Soya bean, seed (CR)		0.01 0.10 1.0 4.0	2 1 2 2	98, 100 105 98, 102 98, 109	99 – 100 104		2012/3000423 SOP-PA.0288
Sunflower, seed (MV)		0.05 0.10 0.50	3 3 3	74.3–91.1 76.9–79.3 84.9–85.6	81.3 78.2 85.3	10.8 1.56 0.44	2002/5004111
Sunflower, refined oil (MV)		0.05 0.10 0.50	3 3 3	95.2–98.2 96.8–99.6 105–108	96.7 97.8 106	1.55 1.57 1.44	
Sunflower, meal (MV)		0.05 0.10 0.50	3 3 3	70.9–77.8 70.8–78.6 77.4–78.9	75.0 75.8 78.0	4.82 5.70 1.00	
Sunflower, seed (CR)		0.05 0.50	1 1	96 86	–		2002/3000641 SOP-PA.0200, Rev.02-RE.01
Sunflower, flower (CR)		0.05 0.50	1 1	92 88	–		
Soya bean, seed (MV)	Mass transition 262→217	0.05 5.0	5 5	88–100 84–98	94 91	4.6 6.3	2010/1149746 SOP-PA.0249
	Mass transition 262→149	0.05 5.0	5 5	88–102 82–100	94 91	5.9 8.0	
Rice, grain (MV)	Mass transition 262→217	0.05 0.05 0.05 5.0 5.0	5 9 1 1 14	76–92 90–100 96 82 92–108	85 95 – – 102	7.6 3.7 4.8	2010/1141978 SOP-PA.0249
	Mass transition 262→149	0.05 0.05 0.05 5.0 5.0	5 9 1 1 14	76–92 88–98 96 82 90–108	85 94 – – 101	7.0 4.0 4.8	
Maize, grain		0.05 0.50 1.0	4 1 1	89–101 99 80	96	5.8	IZ-731-001 M 2468
Maize, grain		0.05 0.10 1.0	2 2 2	119, 119 93, 105 93, 99	119 99 96		IZ-244-005 M 2468

Commodity	Fortification mg/kg	N	Range Recovery (%)	Mean recovery (%)	% RSD	Reference Method
Maize, forage	0.05 0.10 1.0	2 2 2	87, 96 113, 119 99, 102	92 116 101		
Maize, fodder	0.05 0.10 1.0	2 2 2	114, 114 103, 110 103, 105	114 107 104		
Rape, seed	0.5	2	78, 91	85		1998/1008995
Rape, straw	0.05 0.5	2 2	93, 97 81, 82	95 82		L 741/1
Maize, grain	0.05 0.10 1.0	5 3 2		106 97 96		IZ-244-009 () Stout S.J. *et al,*.
Maize, forage	0.05 0.10 1.0	10 3 2		96 106 101		1996
Maize, fodder	0.05 0.10 1.0	3 2 2		108 107 104		
Maize, grain	0.05 0.10 1.0	2 2 2	88, 102 79, 93 90, 95	95 86 93		IZ-244-008 M 2657
Maize, forage	0.05 0.10 1.0	2 2 2	64, 73 75, 80 83, 84	69 78 84		
Maize, fodder	0.05 0.10 1.0	2 2 2	79, 91 83, 84 80, 85	85 84 83		
Maize, plant	0.05	3	78–80	79	1.3	1999/7004026 M 2020

CR: Concurrent Recovery, MV: Method Validation, ILV: Independent Laboratory Validation

Animal matrices

Milk (IZ-245-003, IZ-245-009 and IZ-245-010)

Analyte:	Imazapyr	Capillary Electrophoresis	M 3075
LOQ:	0.01 mg/kg		
Description	Residues are extracted from milk with 1 M hydrochloric acid. The imazapyr residues are subjected to suitable clean-up involving solvent partitioning (methylene chloride and acetone) and solid phase extraction (SCX and C18 cartridge). Measurement of imazapyr is accomplished by capillary electrophoresis (CE) with UV detection (240 nm) and results are calculated using external standards.		

Muscle, Liver, Kidney and Fat (IZ-245-004)

Analyte:	Imazapyr	Capillary Electrophoresis	M 3184
LOQ:	0.05 mg/kg		
Description	Residues are extracted from bovine tissues with an acidic water/acetone solution (3:1, v/v). The mixture is centrifuged and then filtered through celite. An aliquot of the filtrate containing the imazapyr residues is subjected to partitioning with methylene chloride. The extract is purified by solid phase extraction. Quantitation of imazapyr is accomplished by capillary electrophoresis (CE) using UV detection (265 nm). The results are calculated by direct comparison to external standards.		

Milk fat (IZ-245-007)

Analyte:	Imazapyr	Capillary Electrophoresis	M 3223
LOQ:	0.01 mg/kg		
Description	Residues are extracted from milk fat (5 g) with water (19 mL), acetonitrile (50 mL) and hexane (100 mL). The mixture is filtered and the filtrate containing the imazapyr residues is subjected to solvent partitioning (dichloromethane) and solid phase extraction (SCX and C18 cartridge). Quantitation of imazapyr is accomplished by capillary electrophoresis (CE) using UV detection (265 nm), results are calculated by direct comparison to external standards.		

Validation data for methods on animal matrices are summarized in Table 27.

Table 27 Summary of Recovery Data for imazapyr fortified into animal matrices

Commodity	Fortification mg/kg	N	Range of Recovery (%)	Mean recovery (%)	% RSD	Reference
Bovine milk (ILV)	0.01 0.10 0.50	2 2 2	81, 85 92, 96 92, 93	83 94 93		IZ-245-009 M 3075
Bovine muscle (ILV)	0.05 0.25 0.50	2 2 2	71, 78 80, 81 82, 85	75 81 84		IZ-245-004 M 3184
Bovine liver (ILV)	0.05 0.25 0.50	2 2 2	72, 78 79, 85 81, 82	75 82 82		
Bovine kidney (ILV)	0.05 0.25 0.50	2 2 2	71, 77 78, 86 80, 84	74 82 82		
Bovine fat (ILV)	0.05 0.25 0.50	2 2 2	71, 78 87, 87 84, 84	75 87 84		
Bovine milk fat (ILV)	0.01 0.05 0.10	2 2 2	67, 81 73, 86 70, 91	74 80 81		IZ-245-007 M 3223

Stability of pesticide residues in stored analytical samples

The Meeting received data on the stability of imazapyr residues in maize (grain, forage and fodder) and soya bean (seeds and processed fractions) samples for plant and animal commodities stored frozen.

The freezer stability study of imazapyr was conducted on maize grain, forage and fodder (Fletcher, 1997: IZ-326-004). Samples of control maize grain, forage and fodder were fortified with imazapyr at 1.0 mg/kg and stored frozen at -5 to -26 °C. Samples were analysed after storage using GC/MS Method M 2468. The LOQ was 0.05 mg/kg.

Table 28 Recovery of imazapyr from stored fortified samples of maize

Storage interval	Recovery (%) [1.0 mg/kg fortification]		
	Procedural	% remaining	Mean
Grain			
9 months	92	81, 93	87
12 months	105	95, 104	100
18 months	92	95, 102	99
27 months	88	87, 90	89
Forage			
9 months	98	76, 79	78
12 months	89	94, 97	96

Storage interval	Recovery (%) [1.0 mg/kg fortification]		
	Procedural	% remaining	Mean
18 months	96	88, 94	91
27 months	96	87, 87	87
Fodder			
9 months	106	73, 79	76
12 months	91	83, 101	92
18 months	96	77, 92	85
27 months	106	78, 81	80

The storage stability of imazapyr was investigated under deep frozen conditions over a time period of about 10 months for soya bean (seeds) and over a time period of about 3 months for soya bean processed fractions (oil, laminated soya bean, meal and toasted meal), all at –20 °C or lower (Leite, 2011: 2011/1207286). Soya bean samples were spiked with the test items at a concentration level of 0.10 mg/kg. The storage conditions correspond to the usual storage conditions for field samples. The samples were analysed using Method SOP-PA.0288.

Table 29 Recovery of imazapyr from stored fortified samples of soya bean

Storage interval	Recovery (%) [0.10 mg/kg fortification]		
	Procedural	% remaining	Mean
Seed			
0 month	109, 109		
1 month	100, 108	100, 102	101
2 months	91, 97	92, 104	98
3 months	98, 100	88, 97	93
7 months	89, 90	62, 69	66
10 months	141, 145	133, 135	134
Laminated soya bean			
0 month	95, 96		
1 month	109, 113	101, 107	104
3 months	98, 101	101, 101	101
Meal			
0 month	100, 106		
1 month	100, 104	97, 99	98
3 months	100, 101	97, 101	99
Toasted meal			
0 month	101, 110		
1 month	108, 110	111, 113	112
3 months	97, 98	93, 94	94
Oil			
0 month	100, 104		
1 month	95, 102	91, 97	94
3 months	101, 105	99, 105	102

The stability of imazapyr was investigated in bovine milk during freezer storage (Khunachak, 1999: IZ-326-010). The storage conditions chosen were equivalent to the storage conditions used for residue samples awaiting analysis. Milk samples were spiked with the test items at a concentration level of 0.1 mg/kg. The samples were analysed using Method M 3075.

Table 30 Recovery of imazapyr from stored fortified samples of bovine milk

Storage interval	Recovery (%) [0.1 mg/kg fortification]		
	Procedural	% remaining	Mean
0 months	86	85, 87	86
3 months	76	73, 75	74
6 months	95	79, 84	82

The stability of imazapyr was investigated in bovine tissues (muscle and liver) during freezer storage (Khunachak, 1999: IZ-326-011). The storage conditions chosen were equivalent to the storage conditions used for residue samples awaiting analysis. Samples were spiked with the test items at a concentration level of 0.1 mg/kg. The samples were analysed using Method M 3184.

Table 31 Recovery of imazapyr from stored fortified samples of bovine tissues

Storage interval	Recovery (%) [0.1 mg/kg fortification]		
	Procedural	% remaining	Mean
Muscle			
0 month	86	84, 84	84
3 month	84	80, 83	82
8 months	76	80, 82	81
Liver			
0 month	83	81, 81	81
3 month	80	81, 82	82
8 months	75	77, 77	77

USE PATTERN

Imazapyr is registered for the control of broad leaf and grassy weeds on pulses, cereals and oilseed etc. The Meeting received labels from Argentina, Australia, Brazil and the USA. The information available to the Meeting on registered uses of imazapyr is summarized in the table below.

Table 32 Registered uses of imazapyr for crops

Crop	Country	Formulation		Application				PHI, days or Application timing
		Type	Conc. of imazapyr	Method	Rate kg ai/ha	Volume L/ha	No. max	
Grasses for sugar or syrup production								
Sugarcane	Argentina	SL	250 g/L	Spray onto weeds	0.5	150–200	2	30–45 days before planting
Sugar cane	Brazil	SL	266.3 g/L	Spray onto weeds	0.125–0.5	100–400	1	not required when used as directed

Table 33 Registered uses of imazapyr for imidazolinone tolerant crops

Crop	Country	Formulation		Application				PHI, days and/or Application timing
		Type	Conc. of imazapyr	Method	Rate kg ai/ha	Volume L/ha	No. max	
Pulses								
Lentil	Canada	SL	15 g/L	Foliar	0.00906	50–100	1	60
Cereals								
Maize	Argentina	WG	175 g/kg	Foliar	0.025	150–200 (ground)	1	before reaching 6th fully developed leaf status
Maize	Argentina	WG	175 g/kg	Foliar	0.0252	100–150 (ground)	1	before reaching 6th fully developed leaf status
Maize	Australia	WG	175 g/kg	Foliar	0.0175–0.0219	> 50	1	crops in the 2–6 leaf stage [a]
Maize	Brazil	WG	175 g/kg	Foliar	0.0175	100–250 (ground) 40–50 (aerial)	1	96
Maize	USA	WG	175 g/kg	Foliar	0.0157	> 94 (ground) > 47 (aerial)	1	45
Maize	USA		40 g/kg	Foliar	0.0157	> 94 (ground) > 47 (aerial)	1	45 before crop height is 20 inches or crop has 6 leaf collars

Crop	Country	Formulation		Application				PHI, days and/or Application timing
		Type	Conc. of imazapyr	Method	Rate kg ai/ha	Volume L/ha	No. max	
Rice	Brazil	WG	175 g/kg	Foliar	0.0735	100–200 (ground) 40–50 (aerial)	2	60
Wheat	Australia	WG	175 g/kg	Foliar	0.0035–0.0070	> 70	1	crops in the 2–6 leaf stage [a]
Wheat	Australia	EC	7.3 g/L	Foliar	0.0066	> 50	1	crops in the 4 leaf to commencement of flag leaf stage (BBCH 14–37) [a]
Oilseeds								
Rape	Australia	SL	15 g/L	Foliar	0.0045–0.0113	> 70	1	[b]
Rape	Australia	WG	175 g/kg	Foliar	0.0035–0.0096	> 70	1	[c]
Rape	Canada	SL	15 g/L	Foliar	0.00906	50–100	1	60
Sunflower	Argentina	WG	175 g/kg	Foliar	0.025	150–200 (ground)	1	before reaching 6th fully developed leaf status
Sunflower	Argentina	SL	240 g/L	Foliar	0.080	120–150	1	early post emergence
Sunflower	Argentina	WG	800 g/kg	Foliar	0.080	120–150	1	early post emergence
Sunflower	Argentina	SL	15 g/L	Foliar	0.0225–0.030	150–200	1	early post emergence
Sunflower	Uruguay	SL	240 g/L	Foliar	0.080	120–150	1	

[a] Do not graze or cut for stock food for 4 weeks after application, not required to harvest for grains when used directed.

[b] Do not graze or cut for stock food for 5 weeks, not required for grains when used as directed

[c] Do not graze or cut for stock food for 6 weeks, not required for grains when used as directed

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

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

Group	Commodity	Table
Pulses	Lentil (dry)	Table 34
	Soya bean (dry)	Table 35
Cereal grains	Maize	Table 36
	Rice	Table 37
	Wheat	Table 38
Grasses for sugar or syrup production	Sugar cane	Table 39
Oilseed	Rape seed	Table 40, 41
	Sunflower seed	Table 42
Straw, fodder and forage of cereals	Maize fodder and forage	Table 43
	Wheat straw and forage	Table 44
Miscellaneous fodder and forage crops	Rape straw and forage	Table 45

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

Pulses

Lentil (dry)

The Meeting received five trials on imidazolinone-tolerant lentil which were conducted in Canada (Norris, 2009: 2008/7019226). In each trial, a single broadcast foliar application of a SL formulation (15 g/L imazapyr) was made to lentil targeting 0.009 kg ai/ha of imazapyr. The application was made 58–60 days prior to the normal harvest of mature (dry) seed.

The lentil seed samples were analysed for residues of imazapyr using Method M3519 (modified). The maximum storage interval for field-treated samples was 166 days (5.5 months).

Table 34 Imazapyr residues on imidazolinone-tolerant lentil from supervised trials in Canada

Lentil, seed country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	GS	no.			
GAP, Canada	SL	0.00906	50–100		1	60		
Canada, 2008 Dundurn/SK (Impact)	SL	0.009	100	Stem elongation (7–8 nodes)	1	58	0.06, 0.06 mean 0.06	2008/7019226 Norris, 2009
Canada, 2008 Dundurn/SK (Imperial)	SL	0.009	100	Stem elongation (7–8 nodes)	1	58	0.06, 0.10 mean 0.08	Sampling to analysis: 125–166 days
Canada, 2008 Portage la Prairie/MB (CDC Impact)	SL	0.009	99	Early flower	1 50 60 70 81	40 50 60 70 81	0.08, 0.08 mean 0.08 0.07, 0.08 mean 0.08 0.07, 0.07 mean 0.07 0.07, 0.08 mean 0.08 0.07, 0.07 mean 0.07	
Canada, 2008 Wellwood/MB (CDC Imperial)	SL	0.009	103	Early bud	1	59	0.06, 0.06 mean 0.06	
Canada, 2008 Alvena/MB (Impact)	SL	0.009	100	Stem elongation (6–8 nodes)	1	59	0.06, 0.06 mean 0.06	

Soya bean (dry)

Eight residue trials in soya beans were conducted in different representative growing areas in Brazil (Resende, 2008: 2008/1097472). The 480 g/L SL formulation was applied once at a rate equivalent to 0.072 kg ai/ha in a spray volume of 200 L/ha. Method SOP-PA.0249 rev.03 was used to analyse soya bean seed samples for the residues of imazapyr. Imazapyr was quantified by LC-MS/MS with a LOQ of 0.05 mg/kg.

Eight residue trials in soya beans were conducted in different representative growing areas in Brazil (Resende, 2008: 2008/1097470). The WG formulation containing 525 g/kg imazapyr was applied once at a rate equivalent to 0.0525 kg ai/ha in a spray volume of 200 L/ha. Method SOP-

PA.0249 rev.04 was used to analyse soya bean seed samples for the residues of imazapyr. Imazapyr was quantified by LC-MS/MS with a LOQ of 0.05 mg/kg.

One trial (with five plots) in soya bean was conducted in Brazil (Resende, 2010: 2010/1079212, 2010/1010261). The WG formulation containing 525 g/kg imazapyr was applied once at a rate equivalent to 0.0525 kg ai/ha in a spray volume of 200 L/ha. Method SOP-PA.0288 was used for analysis of imazapyr residues in soya bean quantifying the analyte by LC-MS/MS with a LOQ of 0.01 mg/kg.

Two trials in soya beans were conducted in Brazil (Jones, 2011: 2010/1127505). The WG formulation containing 525 g/kg imazapyr was applied once at a rate equivalent to 0.0525 kg ai/ha in a spray volume of 200 L/ha. Each trial consisted of four plots. Method SOP-PA.0288 was used for analysis of imazapyr residues in soya beans quantifying the analyte by LC-MS/MS with a LOQ of 0.01 mg/kg.

Five trials in soya beans were conducted in Brazil (Jones, 2012: 2012/3000423). The WG formulation containing 525 g/kg imazapyr was applied once at a rate equivalent to 0.0525 kg ai/ha in a spray volume of 200 L/ha. Method SOP-PA.0288 was used for analysis of imazapyr residues in soya beans quantifying the analyte by LC-MS/MS with a LOQ of 0.01 mg/kg.

Table 35 Imazapyr residues on imidazolinone-tolerant soya bean seeds from supervised trials in Brazil

Soya bean, seed country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
GAP, Brazil	SL	0.072		100–200 (ground) 40–50 (aerial)		1	60		
Brazil, 2007 Santo Antônio de Goiás/GO (CV 603)	SL	0.072		200	77 71 66 59 39	1	40 60 80 100 120	1.8 1.7 2.0 < 0.05 < 0.05	2008/1097472 Resende, 2008
Brazil, 2007 Santo Antônio de Goiás/GO (CV 603)	SL	0.072		200	67	1	60	1.4	Sampling to analysis: 46–87 days
Brazil, 2007 Uberaba/MG (CV 603)	SL	0.072		200	78 73 51 29 19	1	40 60 80 100 120	1.7 1.3 1.5 0.05 < 0.05	
Brazil, 2007 Uberaba/MG (CV 603)	SL	0.072		200	73	1	60	2.0	
Brazil, 2007 Brasilia/DF (CV 603)	SL	0.072		200	75	1	60	1.9	
Brazil, 2007 Santo Antônio de Posse/SP (CV 603)	SL	0.072		200	72	1	60	0.92	
Brazil, 2007 Santo Antônio de Posse/SP (CV 603)	SL	0.072		200	29 24 18 15 12	1	40 60 80 100 120	0.06 0.41 0.08 < 0.05 < 0.05	
Brazil, 2007 Londrina/PR (CV 603)	SL	0.072		200	71	1	60	< 0.05	
Brazil, 2007 Uberaba/MG (CV 603)	WG	0.053		200	77 73 51	1	40 60 80	2.3 2.5 0.09	2008/1097470 Resende, 2008

Soya bean, seed country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	GS	no.			
					29 19		100 120	< 0.05 < 0.05	
Brazil, 2007 Uberaba/MG (CV 603)	WG	0.053		200	73	1	60	3.0	Sampling to analysis: 49–65 days
Brazil, 2007 Brazilia/DF (CV 603)	WG	0.053		200	75	1	60	1.3	
Brazil, 2007 Santo Antônio de Posse/SP (CV 603)	WG	0.053		200	78 72 65 53 38	1	40 60 80 100 120	0.85 0.48 0.08 < 0.05 < 0.05	
Brazil, 2007 Santo Antônio de Goiás/GO (CV 603)	WG	0.053		200	77 71 66 59 39	1	40 60 80 100 120	1.4 0.45 0.30 0.07 < 0.05	
Brazil, 2007 Santo Antônio de Goiás/GO (CV 603)	WG	0.053		200	71	1	60	1.3	
Brazil, 2007 Santo Antônio de Posse/SP (CV 603)	WG	0.053		200	24	1	60	0.27	
Brazil, 2007 Londrina/PR (CV 603)	WG	0.053		200	67	1	60	< 0.05	
Brazil, 2008 Santo Antônio de Posse/SP (CV 127)	WG	0.053	0.026	200	79–83 75 66 51 13	1	40 60 80 100 120	0.10 0.07 0.01 < 0.01 < 0.01	2010/1010261 2010/1079212 Sampling to analysis: 613 days
Brazil, 2010 Ponta Grossa /PR (L 08)	WG	0.053	0.026	200	83 75 68 66	1	20 40 60 80	< 0.01 0.07 0.90 1.0	2010/1127505 Jones, 2011
Brazil, 2010 Santo Antônio de Posse/SP (CV 127)	WG	0.053	0.026	200	89 87 77 73	1	20 40 60 80	< 0.01 < 0.01 0.35 0.20	Sampling to analysis: 27–78 days
Brazil, 2011 Ponta Grossa /PR (BRZ 08-200151)	WG	0.053	0.026	200	79 75 73 64 62	1	20 40 60 80 100	< 0.01 < 0.01 0.26 0.83 0.25	2012/3000423 Jones, 2012 Sampling to analysis: 162–273 days
Brazil, 2011 Senador Canedo/PR (BRZ 5384)	WG	0.053	0.026	200	66	1	60	0.11	
Brazil, 2011 Anápolis/GO (BRZ 5384)	WG	0.053	0.026	200	69	1	60	0.07	
Brazil, 2011 Santo Antônio de Posse/SP (BRZ 5384))	WG	0.053	0.026	200	73	1	60	1.3	
Brazil, 2011 Castro/PR (BRZ 08-200151	WG	0.053	0.026	200	71	1	60	0.55	

Cereal grains

Maize

Residue data have been collected from 19 field trials located in the USA. At each trial, one broadcast application was made to the treated plot. The formulations used in the trials were 240 g/kg ASU and 240 g/kg WP formulation. Method M 2468 was used for analysis of imazapyr residues in maize grains quantifying the analyte by gas chromatography/negative ion chemical ionization mass spectrometry (GC/ECNICI) with a limit of quantitation of 0.05 mg/kg.

Six trials in maize were conducted in Australia to determine the residue level of imazapyr in/on maize grains. The SL formulations containing 250 g/L imazapyr was applied once as broadcast foliar application. The applications were made in half of the trials post sowing—pre-emergence and in the second half of the trials at early post-emergence. Imazapyr was analysed in the maize grains using method L 741/1. Separation and quantitation were accomplished with reverse phase high performance liquid chromatography using a UV-VIS detector with a limit of quantitation of 0.05 mg/kg.

Seven trials in maize were conducted in the Argentina to determine the residue level of imazapyr on maize. Method LAADL R0001 was used for analysis of imazapyr residues in maize grains quantifying the analyte by HPLC/UV with a limit of quantitation of 0.05 mg/kg.

Three trials in maize were conducted in Brazil to determine the residue level of imazapyr in/on maize. The WG formulation was applied to the plots as broadcast foliar application in spray volumes of 200 L/ha. Method SOP-PA.0200 rev.02-re.01, based on methods MR0001.01, M-2020 and M-1928, was used for analysis of imazapyr residues in maize grains. Imazapyr was determined by LC-MS/MS (mass transition 262/217). The limit of quantitation for this method in maize grains was 0.05 mg/kg.

Table 36 Imazapyr residues on imidazolinone-tolerant maize grains from supervised trials

Maize grain country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	no.			
GAP, USA		0.0157			1	45		
USA, 1993 Conklin/MI (Pioneer Hybrid 3417 IR)	ASU	0.027	188	14	1	126	< 0.05	IZ-731-001 Mahl, 1995 Sampling to analysis: 456–581 days
USA, 1993 Webster/IA (Pioneer Hybrid 3377 IR)	ASU	0.027	187	14	1	131	< 0.05	IZ-731-002 Mahl, 1995 Sampling to analysis: 513–642 days
USA, 1993 Verona/WI (Pioneer Hybrid 3417 IR)	ASU	0.027	186	14	1	125	< 0.05	IZ-731-003 Mahl, 1995 Sampling to analysis: 520–644 days
USA, 1993 Jamesville/ NC (Pioneer Hybrid 3245 IR)	ASU	0.027	202	14–15	1	103	< 0.05	IZ-731-004 Mahl, 1995 Sampling to analysis: 538–638 days
USA, 1994 Conklin/MI (Pioneer Hybrid 3751 IR)	WP	0.027	191	14	1	124	< 0.05	IZ-731-005 Mahl, 1995 Sampling to analysis: 191–314 days
USA, 1994 New Holland/ OH (Pioneer Hybrid 3245 IR)	WP	0.027	171	14	1	123	< 0.05	IZ-731-006 Mahl, 1995 Sampling to analysis: 209–332 days
USA, 1993 Gary/SD	ASU	0.027	181	14	1	134	< 0.05	IZ-731-007 Mahl, 1995

Maize grain country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	no.			
(Pioneer Hybrid 3417 IR)								Sampling to analysis: 476–609 days
USA, 1994 Snock/TX (Pioneer Hybrid 3162 IR)	WP	0.027	199	15	1	110	< 0.05	IZ-731-008 Mahl, 1995 Sampling to analysis: 244–353 days
USA, 1994 Noblesville /IN (Pioneer Hybrid 3395 IR)	WP	0.027	181	14	1	132	< 0.05	IZ-731-009 Mahl, 1995 Sampling to analysis: 224–354 days
USA, 1994 Carman/IL (Pioneer Hybrid 3417 IR)	WP	0.027	187	14–15	1	126	< 0.05	IZ-731-010 Mahl, 1995 Sampling to analysis: 210–335 days
USA, 1993 York/NE (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14–15	1	116	< 0.05	IZ-731-011 Mahl, 1995 Sampling to analysis: 666–802 days
		0.27	187	14–15	1	116	< 0.05	
USA, 1993 Carman/IL (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14–15	1	117	< 0.05	IZ-731-012 Mahl, 1995 Sampling to analysis: 561–677 days
USA, 1993 Clarence/MO (Pioneer Hybrid 3245 IR)	ASU	0.027	187	14	1	112	< 0.05	IZ-731-013 Mahl, 1995 Sampling to analysis: 490–733 days
USA, 1994 Fisher/MN (Pioneer Hybrid 3751 IR)	WP	0.027	187	14–15	1	146	< 0.05	IZ-731-014 Mahl, 1995 Sampling to analysis: 201–425 days
USA, 1994 Webster City/ IA (Pioneer Hybrid 3417 IR)	WP	0.027	191	14	1	131	< 0.05	IZ-731-015 Mahl, 1995 Sampling to analysis: 212–419 days
USA, 1993 Noblesville/ IN (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14	1	149	< 0.05	IZ-731-016 Mahl, 1995 Sampling to analysis: 581–730 days
USA, 1993 New Holland/ OH (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14	1	127	< 0.05	IZ-731-017 Mahl, 1995 Sampling to analysis: 484–779 days
		0.27	187	14	1	127	0.088	
USA, 1994 York/NE (Pioneer Hybrid 3417 IR)	WP	0.027	189	14	1	122	< 0.05	IZ-731-018 Mahl, 1995 Sampling to analysis: 234–355 days
USA, 1993 Hamburg/PA (Pioneer Hybrid 3245 IR)	ASU	0.027	189	14	1	141	< 0.05	1995/7004189 Mahl, 1995 Sampling to analysis: 488–630 days
GAP, Australia	WG	0.018-0.022	> 50		1	not required		
Australia, 1997 Darlington Point/NSW (62 IT)	SL	0.016	112	Early post emergent	1	109	< 0.05	2000/1023965 Mooney, 2000 Sampling to analysis: 138 days
		0.032	112	Early post emergent	1	109	< 0.05	
		0.048	112	Early post emergent	1	109	< 0.05	
	SL	0.016	112	Post sowing pre- emergent	1	152	< 0.05	Sampling to analysis:

Maize grain country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	no.			
		0.032	112	Post sowing pre- emergent	1	152	< 0.05	93 days
		0.048	112	Post sowing pre- emergent	1	152	< 0.05	
GAP, Argentina	WG	0.025			1	before reaching 6th fully developed leaf status		
GAP, Brazil	WG	0.0175			1	96 day PHI		
Argentina, 1996–1997 Santa Fe (Pioneer 3162 IR)	SL	0.040	170	12–13	1	117	< 0.05	IZ-731-021 Steling, 1998 Sampling to analysis: 530 days
		0.080	170	12–13	1	117	< 0.05	
	WG	0.020	170	12–13	1	117	< 0.05	
		0.030	170	12–13	1	117	< 0.05	
Argentina, 1995–1996 Santa Fe (IR)	SL	0.020		17	1	110	< 0.05	1997/7004635 Steling, 1997 Sampling to analysis: 345 days
		0.040		17	1	110	< 0.05	
Argentina, 1997–1998 San Jeronimo (Pioneer 3162 IR)	SC	0.025	166	14	1	140	< 0.05	1999/7004025 Steling, 1999 Sampling to analysis: 48–395 days
		0.040	166	14	1	140	< 0.05	
		0.050	166	14	1	140	< 0.05	
Argentina, 1997–1998 Santos Unzue (Pioneer 3162 IR)	SC	0.025	195	14	1	121	< 0.05	
		0.040	195	14	1	121	< 0.05	
		0.050	195	14	1	121	< 0.05	
Argentina, 1997–1998 Fauzon (Pioneer 3162 IR)	SC	0.025	195	12	1	141	< 0.05	
		0.040	195	12	1	141	< 0.05	
		0.050	195	12	1	141	< 0.05	
Argentina, 1998–1999 San Jeronimo (Asgrow AX 888 IT)	WG	0.020	180	16	1	116	< 0.05	
		0.040	180	16	1	116	< 0.05	
Argentina, 1995–1996 Fauzon (Funks Capitan IT)	SL	0.040	190	14	1	135	< 0.05	1998/3002021 Steling, 1998 Sampling to analysis: 665 days
		0.080	190	14	1	135	< 0.05	
		0.020	190	14	1	135	< 0.05	
		0.040	190	14	1	135	< 0.05	
Brazil, 2002 Londrina/PR (Clearfield)	WG	0.021	200	83	1	30	< 0.05	2002/3001461 Borges, 2002 Sampling to analysis: 118 days
		0.042	200	83	1	30	0.05	
Brazil, 2002 Santo Antônio de Posse/SP (Clearfield)	WG	0.021	200	89	1	30	< 0.05	2002/3001462 Borges, 2002 Sampling to analysis: 36 days
		0.042	200	89	1	30	< 0.05	
Brazil, 2002 São Gotardo/ MG (Clearfield)	WG	0.021	200	73	1	30	< 0.05	2002/3001463 Borges, 2002 Sampling to analysis: 77 days
		0.042	200	73	1	30	< 0.05	

ASU: Aqueous Solution with urea

NA: not analysed

Rice

Four trials with 11 plots in rice were conducted in Brazil (Dantas, 2003: 2003/3001441). The WG formulation containing 525 g/kg imazapyr was applied twice to each trial with rate equivalents of 0.12 kg ai/ha or 0.24 kg ai/ha for each application. Method SOP-PA.0231 was used for analysis of

residues in rice. Imazapyr was measured by liquid chromatography with a UV detector. The limit of quantitation for this method in rice grain samples was 0.05 mg/kg.

Table 37 Imazapyr residues on imidazolinone-tolerant rice grains from supervised trials in Brazil

Rice grain country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	For m	kg ai/ha	water, L/ha	Growth stage	no.			
GAP, Brazil	WG	0.074	100–200		2	60		
Brazil, 2003 São Vicente do Sul/RS (IRGA 422 CL)	WG	0.12	200	5 leaf stage + Stem elongation Booting Booting Inflorescence emergence flowering	2	50 55 60 65 70	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	2003/3001441 Dantas, 2003 Sampling to analysis: 132–139 days
Brazil, 2003 São Vicente do Sul/RS (XP 701 CL)	WG	0.12	200	4 leaf stage + Booting	2	60	0.05	
		0.24	200	4 leaf stage + Booting	2	60	0.05	
Brazil, 2003 Cachoeirinha/RS (XP 701 CL)	WG	0.12	200	3–4 leaf stage + Booting	2	60	< 0.05	
		0.24	200	3–4 leaf stage + Booting	2	60	< 0.05	
Brazil, 2003 Uruguaiana/ RS (Irga 422 CL)	WG	0.12	200	1–2 leaf stage + tillering	2	60	< 0.05	
		0.24	200	1–2 leaf stage + tillering	2	60	< 0.05	

Wheat

The trials were conducted at six locations in Australia (Mooney, 1999: 1999/1013037). The SL formulation containing 250 g/L imazapyr or the WG formulation containing 750 g/kg imazapyr were applied once as broadcast foliar or band application. Samples were collected 83–104 days after application and stored frozen at or below –18°C until analysis. Imazapyr was analysed in the wheat grains using method L 741/1. In principle the active substance is extracted from the matrix using several extraction and separation steps involving HCl:water (1:39) and DCM (dichloromethane). The extract is then cleaned-up by elution through a SCX cartridge and further extractions with DCM. Separation and quantitation are accomplished with reverse phase high performance liquid chromatography using a UV-VIS detector and external standard calibration procedures. The limit of quantitation for this method in wheat grain samples was 0.05 mg/kg.

Table 38 Imazapyr residues on imidazolinone-tolerant wheat from supervised trials in Australia

Wheat grain country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage	no.			
GAP, Australia	WG	0.004–0.007	> 70		1	not required		
Australia, 1997–1998 Winulta, Yorke Peninsula/SA (IT)	SL	0.007	110	2 node stage	1	88	< 0.05	11999/1013037 Mooney, 1999 Sampling to analysis: 182 days
		0.014	110	2 node stage	1	88	< 0.05	
		0.028	110	2 node stage	1	88	< 0.05	
Australia, 1997–1998 Osbourne/ NSW (IT)	SL	0.007	112	Early post emergent	1	83	< 0.05	Sampling to analysis: 193 days
		0.014	112	Early post emergent	1	83	< 0.05	
		0.028	112	Early post emergent	1	83	< 0.05	
Australia, 1997–1998 Gnarwarre/ Vic (IT)	SL	0.007	50	Early post emergent	1	104	< 0.05	Sampling to analysis: 46 days
		0.015	50	Early post emergent	1	104	< 0.05	

Wheat grain country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage	no.			
		0.028	50	Early post emergent	1	104	< 0.05	
Australia, 1998 Manoora/SA (IT)	SL	0.014	117	Stem elongation stage (1–2 node stage	1	95	< 0.05	Sampling to analysis: 67 days
		0.028	117	Stem elongation stage (1–2 node stage	1	95	< 0.05	
Australia, 1998–1999 Burabadji/WA (IT)	WG	0.014	110	Advanced tillering	1	94	< 0.05	Sampling to analysis: 54–148 days
		0.028	110	Advanced tillering	1	94	< 0.05	

Grasses for sugar or syrup production

Sugar cane

The trials were conducted at two locations in Argentina and Brazil to determine the residue level of imazapyr in/on sugar cane. The SL formulation containing 250 or 500 g/L imazapyr, were applied once at a rate equivalent to 0.18, 0.35 and 0.50 kg ai/ha in a spray volume of 168 and 400 L/ha. Method LAADL R0002 was used for analysis of imazapyr residues in sugar cane. Imazapyr was extracted from the sugar cane with acetone:methanol:water (1:1:1) and then subjugated the extract to suitable cleanup involving solid phase extraction techniques. Measurement of imazapyr was accomplished by high performance liquid chromatography (HPLC). Results were calculated by direct comparison of the sample peak heights to those of external standards. The limit of quantitation of the method is 0.05 mg/kg for sugar cane.

Table 39 Imazapyr residues on sugar cane from supervised trials in Argentina and Brazil

Sugar cane country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage/ Timing	no.			
GAP, Argentina GAP, Brazil	SL SL	0.5 0.125 –0.5	150–200 100–400		2 1	30–45 days before planting Not required when used as directed		
Argentina, 1996–1998 Lules/Tucumán (Famaillá 8116)	SL	0.50	168	38 days before plantation	1	392	< 0.05	IZ-790-015 Steling, 1998 Sampling to analysis: 354 days
Brazil, 1996–1998 Piracicaba/SP (RB-72454)	SL	0.18	400	Pre-emergent	1	243	< 0.05	IZ-790-017 Steling, 1998 Sampling to analysis: 216 days
		0.35	400	Pre-emergent	1	243	< 0.05	

Oilseed

Rape seed

A total of 12 trials were conducted on rape in Canada (Norris, 2009: 2008/7019227). At each test location, a single broadcast foliar application of a SL formulation containing 15 g ai/L was made to rape seed targeting 0.009 kg ai/ha. The applications were made in 98–102 L/ha of water using ground equipment, and an adjuvant (0.5% v/v) was added to the spray mixture for all applications. The rape seed RAC (seed) samples were analysed for residues of imazapyr using Method M 3519, modified. Imazapyr was quantified by liquid chromatography, mass/mass detector (LC-MS/MS) with a limit of quantitation (LOQ) of 0.05 mg/kg.

In the growing seasons 1997/1998, trials were conducted at three locations in Australia to determine the residue level of imazapyr in/on rape seed. The WG formulation containing 175 g/kg imazapyr was applied once as broadcast foliar application. The application was generally made post-emergence. Imazapyr was analysed in the rape matrices using method L 741/1. Quantitation was accomplished with reverse phase high performance liquid chromatography using a UV-VIS detector and external standard calibration procedures. The LOQ of the method is 0.05 mg/kg for rape matrices.

Table 40 Imazapyr residues on imidazolinone-tolerant rape seed from supervised trials in Canada

Rape seed country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	Water, L/ha	GS (BBCH)	no.			
GAP, Canada	SL	0.009			1	60		
Canada, 2008 Portage la Prairie/MB (Clearfield 45H73)	SL	0.009	101	65	1	70	< 0.05, < 0.05 Mean ≤ 0.05	2008/7019227 Norris, 2009
Canada, 2008 Josephberg/AB (Clearfield 45H73)	SL	0.009	101	14	1	49 60 70 80 90	< 0.05, < 0.05 < 0.05, < 0.05 Mean < 0.05 < 0.05, < 0.05 < 0.05, < 0.05 < 0.05, < 0.05	Sampling to analysis: 126-184 days
Canada, 2008 Waldheim/SK (Clearfield 45H72)	SL	0.009	100	16–33	1	70	< 0.05, < 0.05 Mean ≤ 0.05	
Canada, 2008 Wellwood/MB (Clearfield 45H72)	SL	0.009	101	65	1	70	< 0.05, < 0.05 Mean ≤ 0.05	
Canada, 2008 Wellwood/MB (Clearfield 45H72)	SL	0.009	101	65	1	70	< 0.05, < 0.05 Mean < 0.05	
Canada, 2008 Dundern/SK (Clearfield 45H72)	SL	0.009	100	13	1	72	< 0.05, < 0.05 Mean ≤ 0.05	
Canada, 2008 Alvena/SK (Clearfield 45H72)	SL	0.009	100	25–33	1	68	< 0.05, < 0.05 Mean ≤ 0.05	
Canada, 2008 Alvena/SK (Clearfield 45H73)	SL	0.009	99	25–33	1	68	< 0.05, < 0.05 Mean ≤ 0.05	
Canada, 2008 Waldheim/SK (Clearfield 45H73)	SL	0.009	102	16–33	1	70	< 0.05, < 0.05 Mean < 0.05	
Canada, 2008 Josephberg/AB (Clearfield 45H72)	SL	0.009	100	15–16	1	70	< 0.05, < 0.05 Mean ≤ 0.05	
Canada, 2008 Lamont/AB (Clearfield 45H72)	SL	0.009	98	> 30	1	70	< 0.05, < 0.05 Mean ≤ 0.05	
Canada, 2008 Lamont/AB (Clearfield 45H73)	SL	0.010	102	> 30	1	70	< 0.05, < 0.05 Mean < 0.05	

Table 41 Imazapyr residues on imidazolinone-tolerant rape seed from supervised trials in Australia

Rape seed country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage	no.			
GAP, Australia	SL	0.005-0.011	> 70		1	not required		
Australia, 1997 Winula, Yorke Peninsula/SA (IT)	WG	0.007	110	Post emergence	1	66	< 0.05	1998/1008955 Anonymous, 2003
		0.014	110	Post emergence	1	66	< 0.05	
		0.028	110	Post emergence	1	66	< 0.05	

Rape seed country, year (variety)	Application					DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage	no.			
Australia, 1997 Gnarwarre/Vic (IT)	WG	0.007	50	Post emergence	1	106	< 0.05	Sampling to analysis: not specified
		0.015	50	Post emergence	1	106	< 0.05	
		0.028	50	Post emergence	1	106	< 0.05	

Sunflower seed

During the two growing seasons 2007/2008 and 2008/2009, trials were conducted for imidazolinone-resistant sunflower at three locations in Argentina to determine the residue level of imazapyr. The WG formulation containing 800 g/kg imazapyr were applied once at rates of 0.06 and 0.12 kg ai/ha. Method SOP-PA.0288 was used to analyse sunflower seed samples for the residues of imazapyr by liquid chromatography using a mass/mass detector (LC-MS/MS) with a limit of quantitation (LOQ) of 0.01 mg/kg.

During the growing season 2010/2011, four trials were conducted in sunflower in Argentina to determine the residue level of imazapyr. The SL formulation containing 15 g/L imazapyr was applied once at a rate of 0.030 kg ai/ha while on the other 4 plots 0.060 kg ai/ha were applied. Method SOP-PA.0288 was used to analyse sunflower seed samples for the residues of imazapyr by liquid chromatography and mass/mass detector (LC-MS/MS) with a limit of quantitation (LOQ) of 0.01 mg/kg.

Table 42 Imazapyr residues on imidazolinone-tolerant sunflower seed from supervised trials

Sunflower seed country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	kg ai/hL	Growth stage	no.			
GAP, Argentina	WG	0.080	120–150			1	Early post emergent		
Argentina, 2007–2008 Balcarce	WG	0.060	120	0.050	13–14	1	106	< 0.01	2010/1057139 Guimarães, 2010
		0.12	120	0.10	13–14	1	106	< 0.01	
Argentina, 2008–2009 Balcarce	WG	0.060	120	0.050	13–14	1	105	< 0.01	Sampling to analysis: 381–751 days
		0.12	120	0.10	13–14	1	105	< 0.01	
Argentina, 2007–2008 Tandil	WG	0.060	120	0.050	13–14	1	105	0.03	
		0.12	120	0.10	13–14	1	105	0.01	
Argentina, 2008–2009 Tandil	WG	0.060	120	0.050	13–14	1	104	0.01	
		0.12	120	0.10	13–14	1	104	0.02	
Argentina, 2007–2008 Realicó	WG	0.060	120	0.050	13–14	1	98	< 0.01	
		0.12	120	0.10	13–14	1	98	0.02	
Argentina, 2008–2009 Realicó	WG	0.060	120	0.050	13–14	1	109	< 0.01	
Argentina, 2010–2011 Gonzalez Moreno	SL	0.030				1	81	< 0.01	2012/3000521 Jones, 2012
		0.060				1	81	0.03	
Argentina, 2010–2011 Venado Tuerto	SL	0.030				1	98	< 0.01	Sampling to analysis: 380–746 days
		0.060				1	98	< 0.01	
Argentina, 2010–2011 Balcarce	SL	0.030				1	103	< 0.01	
		0.060				1	103	< 0.01	
Argentina, 2010–2011 Yuto	SL	0.030				1	143	< 0.01	
		0.060				1	143	< 0.01	
Argentina, 2001–2002	SL	0.080	170		12–14	1	90	< 0.05	2002/3000641 Borges, 2002

Sunflower seed country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	kg ai/hL	Growth stage	no.			
Balcarce (Clearfield)		0.16	170			1	90	< 0.05	
Argentina, 2002 Miramar (Clearfield)	SL	0.10	170		12–14	1	100	< 0.05	Sampling to analysis: 89–458 days
Uruguay, 2010–2011 Ciudad Del Plata (Neo G-09 CL)	SL	0.080	148.4		14	1	102	< 0.01, < 0.01 Mean < 0.01	2012/7000183 Carringer, 2012
Uruguay, 2010-2011 Canelones (Neo G-09 CL)	SL	0.080	148.3		14	1	103	< 0.01, < 0.01 Mean < 0.01	Sampling to analysis: 321-339 days
Argentina, 2010-2011 Gahan (Paraiso 103 CL)	SL	0.076	142.4		15	1	111	< 0.01, < 0.01 Mean < 0.01	

Straw, fodder and forage of cereals

Maize fodder and forage

Residue data have been collected from 19 field trials located in the USA. Method M 2468 was used for analysis of imazapyr residues in maize samples quantifying the analyte by gas chromatography/negative ion chemical ionization mass spectrometry (GC/ECNICI) with a limit of quantitation of 0.05 mg/kg.

Six trials in maize were conducted in Australia to determine the residue level of imazapyr in/on maize. The SL formulation was applied once as broadcast foliar application. The applications were made in half of the trials post sowing—pre-emergence and in the second half of the trials at early post-emergence. Imazapyr was analysed in the maize matrices using method L 741/1. Separation and quantitation were accomplished with reverse phase high performance liquid chromatography using a UV-VIS detector with a limit of quantitation of 0.05 mg/kg.

Seven trials in maize were conducted in the Argentina. Method LAADL R0001 was used for analysis of imazapyr residues in maize quantifying the analyte by HPLC/UV with a limit of quantitation of 0.05 mg/kg.

Three trials in maize were conducted in Brazil. The WG formulation was applied to the plots as broadcast foliar application in spray volumes of 200 L/ha. Method SOP-PA.0200 rev.02-re.01, based on methods MR0001.01, M-2020 and M-1928, was used for analysis of imazapyr residues in maize samples. Imazapyr was determined by LC-MS/MS (mass transition 262/217). The limit of quantitation for this method in maize samples was 0.05 mg/kg.

Table 43 Imazapyr residues on imidazolinone-tolerant maize from supervised trials

Maize country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	Analytical portion	no.			
GAP, USA		0.016				1	45		
USA, 1993 Conklin/MI (Pioneer Hybrid 3417 IR)	ASU	0.027	188	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 45 60 80 90 126	3.1 0.11 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-001 Mahl, 1995 Sampling to analysis: 456–581 days

Maize country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	Analytical portion	no.			
USA, 1993 Webster/IA (Pioneer Hybrid 3377 IR)	ASU	0.027	187	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 31 46 60 80 90 131	2.6 0.087 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-002 Mahl, 1995 Sampling to analysis: 513–642 days
USA, 1993 Verona/WI (Pioneer Hybrid 3417 IR)	ASU	0.027	186	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 45 60 82 105 125	1.6 0.13 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-003 Mahl, 1995 Sampling to analysis: 520–644 days
USA, 1993 Jamesville/ NC (Pioneer Hybrid 3245 IR)	ASU	0.027	202	14–15	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 29 43 60 81 90 103	1.9 0.14 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-004 Mahl, 1995 Sampling to analysis: 538–638 days
USA, 1994 Conklin/MI (Pioneer Hybrid 3751 IR)	WP	0.027	191	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 45 60 80 98 124	1.8 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-005 Mahl, 1995 Sampling to analysis: 191–314 days
USA, 1994 New Holland/ OH (Pioneer Hybrid 3245 IR)	WP	0.027	171	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 32 45 60 80 89 123	5.4 0.057 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-006 Mahl, 1995 Sampling to analysis: 209–332 days
USA, 1993 Gary/SD (Pioneer Hybrid 3417 IR)	ASU	0.027	181	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 44 61 80 92 134	2.7 0.17 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-007 Mahl, 1995 Sampling to analysis: 476–609 days
USA, 1994 Snock/TX (Pioneer Hybrid 3162 IR)	WP	0.027	199	15	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 8 30 45 60 80 90 110	2.4 0.13 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-008 Mahl, 1995 Sampling to analysis: 244–353 days

Maize country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	Analytical portion	no.			
USA, 1994 Noblesville /IN (Pioneer Hybrid 3395 IR)	WP	0.027	181	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 6 31 48 60 80 90 132	1.7 0.10 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-009 Mahl, 1995 Sampling to analysis: 224–354 days
USA, 1994 Carman/IL (Pioneer Hybrid 3417 IR)	WP	0.027	187	14–15	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 44 61 79 91 126	3.8 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-010 Mahl, 1995 Sampling to analysis: 210–335 days
USA, 1993 York/NE (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14–15	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 31 45 60 80 90 116	4.7 0.095 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-011 Mahl, 1995 Sampling to analysis: 666–802 days
USA, 1993 Carman/IL (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14–15	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 29 46 60 81 91 117	3.2 0.11 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-012 Mahl, 1995 Sampling to analysis: 561–677 days
USA, 1993 Clarence/MO (Pioneer Hybrid 3245 IR)	ASU	0.027	187	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 31 44 62 80 92 112	3.1 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-013 Mahl, 1995 Sampling to analysis: 490–733 days
USA, 1994 Fisher/MN (Pioneer Hybrid 3751 IR)	WP	0.027	187	14–15	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 45 60 80 93 146	3.0 0.073 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-014 Mahl, 1995 Sampling to analysis: 201–425 days
USA, 1994 Webster City/ IA (Pioneer Hybrid 3417 IR)	WP	0.027	191	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 31 45 60 81 90 131	2.4 0.087 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-015 Mahl, 1995 Sampling to analysis: 212–419 days

Maize country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	Analytical portion	no.			
USA, 1993 Noblesville/ IN (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 31 44 59 80 90 149	4.2 < 0.05 < 0.05 NA < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-016 Mahl, 1995 Sampling to analysis: 581–730 days
USA, 1993 New Holland/ OH (Pioneer Hybrid 3417 IR)	ASU	0.027	187	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 8 30 45 59 80 90 127	2.1 0.16 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-017 Mahl, 1995 Sampling to analysis: 484–779 days
USA, 1994 York/NE (Pioneer Hybrid 3417 IR)	WP	0.027	189	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 45 60 80 90 122	2.7 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	IZ-731-018 Mahl, 1995 Sampling to analysis: 234–355 days
USA, 1993 Hamburg/PA (Pioneer Hybrid 3245 IR)	ASU	0.027	189	14	Forage Forage Forage Forage Forage Early Silage Late Silage Fodder	1	0 7 30 46 60 81 91 141	3.3 0.16 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	1995/7004189 Mahl, 1995 Sampling to analysis: 488–630 days
GAP, Australia	WG	0.018 – 0.022	> 50			1	Do not gaze and cut for 4 weeks after application		
Australia, 1997 Darlington Point/NSW (62 IT)	SL	0.016	112	Early post emergent	Forage	1	7 21 37 50	0.08 < 0.05 < 0.05 < 0.05	2000/1023965 Mooney, 2000
		0.032	112	Early post emergent	Forage	1	7 21 37 50	0.09 < 0.05 < 0.05 < 0.05	Sampling to analysis: 197–240 days
		0.048	112	Early post emergent	Forage	1	7 21 37 50	0.19 < 0.05 < 0.05 < 0.05	
	SL	0.016	112	Post sowing pre- emergent	Forage	1	29 43 67	< 0.05 < 0.05 < 0.05	
		0.032	112	Post sowing pre- emergent	Forage	1	29 43 67	< 0.05 < 0.05 < 0.05	
		0.048	112	Post sowing pre- emergent	Forage	1	29 43 67	< 0.05 < 0.05 < 0.05	Sampling to analysis: 179–216 days
GAP, Argentina	WG	0.025				1	before reaching 6th fully developed leaf status		

Maize country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage (BBCH)	Analytical portion	no.			
Argentina, 1997–1998 San Jeronimo (Pioneer 3162 IR)	SC	0.025	166	14	Forage	1	90	< 0.05	1999/7004026 Steling, 1999
		0.040	166	14	Forage	1	90	< 0.05	
		0.050	166	14	Forage	1	90	< 0.05	
Argentina, 1997–1998 Santos Unzue (Pioneer 3162 IR)	SC	0.025	195	14	Forage	1	80	< 0.05	Sampling to analysis: 109–461 days
		0.040	195	14	Forage	1	80	< 0.05	
		0.050	195	14	Forage	1	80	< 0.05	
Argentina, 1997–1998 Fauzon (Pioneer 3162 IR)	SC	0.025	195	12	Forage	1	96	< 0.05	
		0.040	195	12	Forage	1	96	< 0.05	
		0.050	195	12	Forage	1	96	< 0.05	
Argentina, 1998–1999 San Jeronimo (Asgrow AX 888 IT)	WG	0.020	180	16	Forage	1	56	< 0.05	
		0.040	180	16	Forage	1	56	< 0.05	

ASU: Aqueous Solution with urea

Wheat straw and forage

The trials were conducted at six locations in Australia. The SL formulation or the WG formulation were applied once as broadcast foliar or band application. Imazapyr was analysed in the wheat matrices using method L 741/1. Quantitation was accomplished with reverse phase high performance liquid chromatography using a UV-VIS detector and external standard calibration procedures. The limit of quantitation for this method in wheat samples was 0.05 mg/kg.

Table 44 Imazapyr residues on imidazolinone-tolerant wheat from supervised trials in Australia

Wheat country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage	Analytical portion	no.			
GAP, Australia	WG	0.004–0.007	> 70			1	Do not gaze and cut for 4 weeks after application		
Australia, 1997–1998 Winulta, Yorke Peninsula/SA (IT)	SL	0.007	110	2 node stage	Straw	1	88	< 0.05	1999/1013037 Mooney, 1999 Sampling to analysis: 182 days
		0.014	110	2 node stage		1	88	< 0.05	
		0.028	110	2 node stage		1	88	< 0.05	
		0.007	110	2 node stage	Forage	1	0 13 25	0.12 < 0.05 < 0.05	
		0.014	110	2 node stage		1	0 13 25	0.12 < 0.05 < 0.05	Sampling to analysis: 245–273 days
		0.028	110	2 node stage		1	0 13 25	0.22 0.05 < 0.05	
Australia, 1997–1998 Osbourne/ NSW (IT)	SL	0.007	112	Early post emergent	Straw	1	83	< 0.05	Sampling to analysis: 193 days
		0.014	112	Early post emergent		1	83	< 0.05	
		0.028	112	Early post emergent		1	83	< 0.05	
		0.007	112	Early post emergent	Forage	1	0 14 28	0.40 < 0.05 < 0.05	Sampling to analysis: 217–

Wheat country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage	Analytical portion	no.			
		0.014	112	Early post emergent		1	0 14 28	0.73 0.08 < 0.05	245 days
		0.028	112	Early post emergent		1	0 14 28	1.5 0.10 0.05	
Australia, 1997–1998 Gnarwarre/ Vic (IT)	SL	0.007	50	Early post emergent	Straw	1	104	< 0.05	Sampling to analysis: 46 days
		0.015	50	Early post emergent		1	104	< 0.05	
		0.028	50	Early post emergent		1	104	< 0.05	
		0.007	50	Early post emergent	Forage	1	14 29	0.051 < 0.05	Sampling to analysis: 107–122 days
		0.015	50	Early post emergent		1	14 29	0.090 0.087	
		0.028	50	Early post emergent		1	14 29	< 0.05 < 0.05	
Australia, 1998 Rannock/ NSW (IT)	SL	0.014	84	Early to late tillering	Forage	0 1	– 1 15 30	< 0.05 0.11 < 0.05 < 0.05	Sampling to analysis: 21–48 days
		0.028	84	Early to late tillering	Forage	1	1 15 30	0.65 0.05 < 0.05	
Australia, 1998 Manoora/SA (IT)	SL	0.014	117	Stem elongation stage (1–2 node stage)	Straw	1	95	< 0.05	Sampling to analysis: 67 days
		0.028	117	Stem elongation stage (1–2 node stage)		1	95	< 0.05	
		0.014	117	Stem elongation stage (1–2 node stage)	Forage	1	0 13 26 42	0.10 0.08 < 0.05 < 0.05	Sampling to analysis: 91–133 days
		0.028	117	Stem elongation stage (1–2 node stage)	Forage	1	0 13 26 42	0.33 0.11 0.11 < 0.05	
Australia, 1998–1999 Burabadji/WA (IT)	WG	0.014	110	Advanced tillering	Straw	1	94	< 0.05	Sampling to analysis: 54–148 days
		0.028	110	Advanced tillering		1	94	< 0.05	
		0.014	110	Advanced tillering	Forage	1	0 14 28 42	0.37 < 0.05 < 0.05 < 0.05	
		0.028	110	Advanced tillering	Forage	1	0 14 28 42	0.62 0.10 < 0.05 < 0.05	

Miscellaneous fodder and forage crops

Rape straw and forage

In the growing seasons 1997/1998, trials were conducted at 3 locations in Australia. The WG formulation was applied once as broadcast foliar application. Imazapyr was analysed in the rape matrices using method L 741/1. Quantitation was accomplished with reverse phase high performance

liquid chromatography using a UV-VIS detector and external standard calibration procedures. The LOQ of the method is 0.05 mg/kg for rape matrices.

Table 45 Imazapyr residues on imidazolinone-tolerant rape from supervised trials in Australia

Rape country, year (variety)	Application						DALA Days	Residues, mg/kg	Ref
	Form	kg ai/ha	water, L/ha	Growth stage	Analytical portion	no.			
GAP, Australia	SL	0.005 – 0.012	> 70			1	Do not gaze and cut for 5 weeks after application		
Australia, 1997 Winula, Yorke Peninsula/SA (IT)	WG	0.007	110	Post emergence	Forage	1	0 13 25	0.12 < 0.05 < 0.05	1998/1008955 Anonymous, 2003 Sampling to analysis: not specified
		0.014	110	Post emergence	Forage	1	0 13 25	0.14 < 0.05 < 0.05	
		0.028	110	Post emergence	Forage	1	0 13 25	0.22 0.11 < 0.05	
		0.007 0.014 0.028	110	Post emergence	Straw	1 1 1	66 66 66	< 0.05 < 0.05 < 0.05	
Australia, 1997 Gnarwarre/Vic (IT)	WG	0.007 0.015 0.028	50	Post emergence	Forage	1 1 1	29 29 29	< 0.05 < 0.05 < 0.05	
		0.007 0.015 0.028	50	Post emergence	Straw (fodder)	1 1 1	106 106 106	< 0.05 < 0.05 < 0.05	
Australia, 1998 Rannock/NSW (IT)	WG	0.014	84	Post emergence	Forage	1	19 33 47	< 0.05 < 0.05 < 0.05	
		0.028	84	Post emergence	Forage	1	19 33 47	0.05 < 0.05 < 0.05	

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received information on high temperature hydrolysis of imazapyr and the fate of imazapyr residues during the processing of soya bean seeds, maize grains, rape seeds and sunflower seeds.

Soya bean, maize, rape and sunflower of the crops that the Meeting received information on supervised field trials may be processed prior to consumption. Processing factors have been calculated for imazapyr residues in soya bean seeds, maize grains, rape seeds and sunflower seeds.

High temperature hydrolysis

The degradation of [^{14}C] imazapyr was studied under hydrolytic conditions at high temperatures in sterile aqueous buffers at pH 4, 5 and 6 for periods of up to 60 minutes so as to simulate common processing practices (pasteurization, baking/brewing/boiling/, and sterilization) (Osterman, 2012: 2012/7000341). All test samples were prepared in sterile amber bottles to eliminate possible photolysis and all sterile buffers were bubbled with nitrogen to avoid the effects of oxygen on the test systems. The concentration of imazapyr was approximately 10 mg/L. The hydrolysis tests were conducted with [^{14}C] imazapyr in sterile 0.1 M aqueous solutions buffered at pH 4, 5 and 6. The samples were prepared in duplicates for each test system. Samples were analysed immediately at time zero (these samples were not heated). Two additional samples at pH 4 ± 0.1 were placed in an oven and maintained at 90 °C ± 5 °C for 20 minutes, another two samples at pH 5 ± 0.1 were placed in an

oven and maintained at 100 °C ± 5 °C for 60 minutes, and two more samples at pH 6 ± 0.1 were placed in an autoclave and maintained at sterilizing conditions (121 °C) for 20 minutes.

Radiocarbon recoveries ranged from 99.7 to 101.5% for all samples. The test solutions were analysed by reverse phase HPLC. The experiments showed that imazapyr was stable under hydrolytic conditions at high temperatures. No degradates were detected at any of the investigated pH and temperature ranges.

Table 46 Hydrolysis recovery under the conditions for processing simulation

Temperature	Time	pH	Representative process	Recovery (%) [10 mg/L]	
				% remaining	Mean
90 °C ± 5 °C	20 minutes	4 ± 0.1	Pasteurization	100.2, 99.7	100
100 °C ± 5 °C	60 minutes	5 ± 0.1	Baking/brewing/boiling	101.5, 101.2	101
121 °C (actual)	20 minutes	6 ± 0.1	Sterilization	100.1, 100.5	100

Soya bean seeds

Processing studies for soya bean were conducted in Brazil to determine the potential for concentration of residues of imazapyr in the processed fractions of soya bean. The SC formulation containing 480 g/L imazapyr was applied once at exaggerated rates of 0.22 kg ai/ha (3×) or 0.14 kg ai/ha (2×), respectively. The WG formulation containing 525 g/kg imazapyr was applied once at exaggerated rates of 0.16 kg ai/ha or 0.11 kg ai/ha, respectively. Samples of soya bean seeds (RAC) were processed according to simulated commercial procedures into flaked soya bean, oil, meal, toasted meal, defatted meal, toasted defatted meal, laminated soya bean fractions and hulls. For one trial, The WG formulation containing 525 g/kg imazapyr was applied once at a rate equivalent to 0.053 kg ai/ha in a spray volume of 200 L/ha. Soya bean seeds were used for processing and aspirated grain fractions (AGF) were produced. Method SOP-PA.0288 was used for analysis of imazapyr residues in soya bean quantifying the analyte by LC-MS/MS with a LOQ of 0.01 mg/kg.

The soya bean grains were initially dried in a ventilated oven at 60 °C, for approximately 5 hours and after drying they were cleaned. The soya bean grains were rolled in an “expeller” press type. The layers of soya bean presented an average thickness of 2 mm (flaked/laminated). Part of rolled material was submitted to the extraction of the oil in “batch” type equipment, using indirect vapor for heating the solvent n-hexane between 45–50 °C. The extractors were heated with indirect vapour during 20 min to evaporate the residual solvent from the meal. The material was heated with direct vapour for 5 min (defatted meal). The material was submitted under pressure of 0.4 kg/cm^2 during 2 min to finish the toasting process (toasted meal). The first part (oil mixture and solvent), produced after the extraction was collected in the tank and the solvent was removed under indirect heating at 60 °C (oil). The humidity of the meals was reduced by a ventilated oven at 60 °C for 12 hours (meal).

Table 47 Imazapyr residues in processed commodities of soya bean seeds from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg		Ref.
	kg ai/ha	water, L/ha	GS (BBCH)	no.			mg/kg	PF	
Brazil, 2007–2008 Santo Antônio de Posse/SP (CV 127)	0.22	200	75	1	60	seed flaked oil meal toasted meal	0.18 0.28 < 0.01 0.33 0.25	 1.6 < 0.06 1.8 1.4	2009/7000088 Resende, 2009 Processing to analysis: 70–85 days
Brazil, 2007–2008 Londorina/PR (CV 127)	0.14	200	72	1	60	seed flaked oil meal toasted meal	1.56 1.41 < 0.01 1.90 2.14	 0.90 < 0.006 1.2 1.4	

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg		Ref.
	kg ai/ha	water, L/ha	GS (BBCH)	no.			mg/kg	PF	
Brazil, 2007–2008 Santo Antônio de Posse/SP (CV 127)	0.16	200	75	1	60	seed flaked oil meal toasted meal	0.14 0.13 < 0.01 0.21 0.20	 0.93 < 0.07 1.5 1.4	2009/7000089 Resende, 2009 Processing to analysis: 70–87 days
Brazil, 2007–2008 Londorina/PR (CV 127)	0.11	200	72	1	60	seed flaked oil meal toasted meal	1.04 0.60 < 0.01 1.23 0.91	 0.58 < 0.01 1.2 0.88	
Brazil, 2008–2009 Santo Antônio de Posse/SP (CV 127)	0.16	200	67	1	60	seed defatted meal toasted defatted meal oil laminated	2.22 2.79 2.42 < 0.01 0.85	 1.3 1.1 < 0.005 0.38	2012/1044747 Jones, 2012 Sampling to analysis: max. 16 months
						seed meal hulls	1.81 1.65 0.97	 0.91 0.54	
Brazil, 2008–2009 Londorina/PR (CV 127)	0.11	200	69	1	60	seed defatted meal toasted defatted meal oil laminated	1.22 1.63 1.54 < 0.01 0.39	 1.3 1.3 < 0.008 0.32	
						seed meal hulls	0.98 1.01 0.77	 1.0 0.79	
Brazil, 2011 Santo Antônio de Posse/SP (BRZ 5384))	0.053	200	73	1	60	seed AGF	1.27 0.04	 0.031	2012/3000423 Jones, 2012 Sampling to analysis: 169 days

Maize grains

A processing study was conducted on maize in the USA during the 1993 growing season to determine the potential for concentration of residues of imazapyr in maize processed fractions. At the test location, a single broadcast foliar application of the ASU formulation containing 240 g/L imazapyr was made to maize targeting 0.27 kg ai/ha (10×) of imazapyr. Grains for processing were harvested at 136 days after application and were later processed according to simulated commercial procedures into maize processed commodities meal and oil. The maize grain RAC and processed commodity samples (meal and oil) were analysed for residues of imazapyr using Method M 2468 with a LOQ of 0.05 mg/kg. Measurement of imazapyr was accomplished by gas chromatography/negative ion chemical ionization mass spectrometry.

Table 48 Imazapyr residues in processed commodities of maize grains from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg		Ref.
	kg ai/ha	water, L/ha	GS (BBCH)	no.			mg/kg	PF	
USA, 1993 York/NE (Pioneer Hybrid 3417 IR)	0.27	187	14–15	1	136	grain meal oil	0.0995 0.116 < 0.05	 1.2 < 0.50	IZ-731-011 Mahl, 1995 Sampling to analysis: 667 days

Rape seeds

A processing study for rape seed was conducted to determine the potential for concentration of residues of imazapyr in the processed fractions of rape seed. The study was conducted in Canada during the 2008 growing season to determine the potential for concentration of residues of imazapyr in rape seed processed fractions. At the test location, a single broadcast foliar application of a SL formulation containing 15 g/L imazapyr was made to rape targeting 0.045 kg ai/ha. Rape seed bulk samples harvested from the plot treated at an exaggerated rate (5×) were harvested 70 days after treatment and were later processed according to simulated commercial procedures into rape seed meal and refined oil. Method M 3519 was used to analyse rape seed samples for the residues of imazapyr. Imazapyr was quantified by liquid chromatography and mass/mass detector (LC-MS/MS) with a LOQ of 0.05 mg/kg.

Table 49 Imazapyr residues in processed commodities of rape seeds from supervised trials

country, year (variety)	Application				DALADays	Commodity	Residues, mg/kg		Ref.
	kg ai/ha	water, L/ha	GS (BBCH)	no.			mg/kg	PF	
Canada, 2008 Waldheim/SK (Clearfield 45H72)	0.045	100	16–33	1	70	grain meal refined oil	0.07 0.10 < 0.05	 1.4 < 0.71	2008/7019227 Norris, 2009 Sampling to analysis: 126-184 days

Sunflower seed

A processing study for sunflower seed was conducted in the USA in the year 2000 to determine the potential for concentration of residues of imazapyr in the processed fractions of sunflower seeds. The applications were made as broadcast, post-emergence sprays targeting when the plants were 18–30 cm tall. Replicate sunflower seed RAC samples were harvested at normal crop maturity, 88 days after treatment. The whole sunflower seed samples were processed into meal and refined oil according to simulated commercial procedures.

The method used for analysis determines residues of imazapyr using HPLC with MS detection. Residues of imazapyr were extracted from sunflower seed and meal samples with an acidified methanol: water solution, filtered, and concentrated. The residues are then purified on a C18 solid phase extraction (SPE) column and eluted with 25% MeOH in 0.05 M aqueous ammonium acetate. Residues in refined oil samples are diluted in hexane and extracted by shaking with acidified acetonitrile: water solution. Following phase separation, the lower aqueous ACN layer is collected and imazapyr residues are detected by LC-MS with a LOQ of 0.05 mg/kg.

Table 50 Imazapyr residues in processed commodities of sunflower seeds from supervised trials

country, year (variety)	Application				DALA Days	Commodity	Residues, mg/kg		Ref.
	kg ai/ha	water, L/ha	Growth stage	no.			mg/kg	PF	
USA/ND, 2000 (CMS HA425/ RHA 426)	0.022	95	18–30 cm height	1	88	seed meal refined oil	< 0.05 < 0.05 < 0.05		2002/5004111 Johnston, 2003
	0.045	94	18–30 cm height	1	88	seed meal refined oil	< 0.05 0.07 < 0.05	 > 1.4	Processing to analysis: max. 5 months

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received lactating dairy cow feeding studies.

Lactating dairy cow

The study was designed to determine the residues of imazapyr found in milk and tissues following oral administration to dairy cow (Khunachak, 1999: IZ-705-001). Five groups of cows, each group containing three animals, were orally dosed with imazapyr for 28–29 days at dosages of approximately 1.2, 3.6, 12 and 36 g imazapyr per animal per day. This would be equivalent to total dietary residues of approximately 58, 157, 607 and 1680 ppm feed. Dose levels were based on a nominal feed intake of 20 kg (dry matter equivalent) per day for a cow. Gelatin capsules containing imazapyr were used for administration.

Milk samples (composites of the pm and am milking of the following day) were collected from all cows. Milk fat samples were prepared from composite milk samples of all cows in each group at 8, 15 and 22 days of treatment. Within 24 hours of the last dosing, all cows were sacrificed and the edible tissues (muscle, fat, liver and kidney) from each animal were collected. All samples were analysed for imazapyr using Methods M 3075 (milk), M 3184 (tissues, fat) and M 3223 (milk fat). Quantitation in milk, muscle, fat, liver, and kidney was performed using capillary electrophoresis with UV detection, and quantitation in milk fat was performed using LC-MS with single ion monitoring (SIM). The LOQ of the individual methods for imazapyr detection in milk and milk fat was 0.01 mg/kg and for tissues and fat 0.05 mg/kg.

Imazapyr residues in milk samples from cows in group A (control) were less than LOQ. The pre-treatment milk samples from all cows from the treated groups showed imazapyr residues of < 0.01 mg/L. In the three highest dose groups (157, 607, 1.680 ppm feed), residues of imazapyr in milk reached a plateau after about 2–3 days

Table 51 Residues of imazapyr in whole milk

Day	Residues, mg/L		
	Group A (control)	Group B (58 ppm feed)	Group C (157 ppm feed)
-1	< 0.01 (3)	< 0.01 (3)	< 0.01 (3)
2	< 0.01 (3)	< 0.01, < 0.01, 0.013 mean 0.011	0.030, 0.039, 0.036 mean 0.035
3	< 0.01 (3)	< 0.01, < 0.01, 0.012 mean 0.011	0.029, 0.021, 0.024 mean 0.024
6	< 0.01 (3)	< 0.01 (3)	0.022, 0.029, 0.027 mean 0.026
8	< 0.01 (3)	< 0.01 (3)	0.028, 0.028, 0.024 mean 0.027
10	< 0.01 (3)	< 0.01 (3)	0.023, 0.036, 0.028 mean 0.029
13	< 0.01 (3)	< 0.01 (3)	0.023, 0.025, 0.025 mean 0.024
15	< 0.01 (3)	na	na
17	< 0.01 (3)	< 0.01 (3)	0.031,0.033,0.023 mean 0.029
20	< 0.01 (3)	na	na
22	< 0.01 (3)	na	na
24	< 0.01 (3)	< 0.01, < 0.01, 0.013 mean 0.011	0.024, 0.025, 0.034 mean 0.028
27	< 0.01 (3)	< 0.01, < 0.01, 0.012 mean 0.011	0.021, 0.031, 0.027 mean 0.026

Day	Residues, mg/L	
	Group D (607 ppm feed)	Group E (1680 ppm feed)
-1	< 0.01 (3)	< 0.01 (3)
2	0.073, 0.072, 0.12 mean 0.087	0.24, 0.35, 0.35 mean 0.31
3	0.11, 0.094, 0.12 mean 0.11	0.24, 0.27, 0.30 mean 0.27
6	0.090, 0.075, 0.11 mean 0.092	0.21, 0.25, 0.27 mean 0.24
8	0.083, 0.080, 0.13 mean 0.096	0.19, 0.25, 0.23 mean 0.22
10	0.067, 0.057, 0.12 mean 0.082	0.21, 0.29, 0.32 mean 0.27
13	0.077, 0.081, 0.13 mean 0.095	0.18, 0.33, 0.30 mean 0.27
15	na	na
17	0.11, 0.076, 0.10 mean 0.096	0.17, 0.27, 0.23 mean 0.22
20	na	na
22	na	na
24	0.11, 0.071, 0.099 mean 0.094	0.25, 0.30, 0.25 mean 0.27
27	0.083, 0.057, 0.086 mean 0.075	0.18, 0.26, 0.29 mean 0.24

na: not analysed

Imazapyr residues in milk fat samples from cows in group A (control) were < 0.01 mg/kg. Averages imazapyr residues in milk fat samples from cows in treated groups B, C, D and E were < 0.01, 0.013, 0.037 and 0.10 mg/kg, respectively.

Table 52 Residues of imazapyr in milk fat

Group	Residue, mg/kg		
	Day 8	Day 15	Day 22
A (control)	< 0.01	< 0.01	< 0.01
B (58 ppm)	< 0.01	< 0.01	< 0.01
C (157 ppm)	0.012	0.011	0.015
D (607 ppm)	0.039	0.041	0.032
E (1680 ppm)	0.10	0.093	0.11

Imazapyr residues in muscle, fat, kidney and liver samples from cows in group A (control) were all < 0.05 mg/kg. In kidney, imazapyr was detected as predominant residue in all treated groups.

Table 53 Residues of imazapyr in tissues

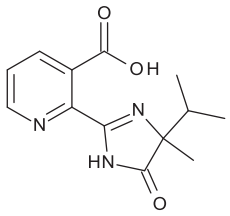
Group	Residues, mg/kg			
	Muscle	Fat	Kidney	Liver
A (control)	< 0.05 (3)	< 0.05 (3)	< 0.05 (3)	< 0.05 (3)
B (58 ppm)	< 0.05 (3)	< 0.05 (3)	0.11, 0.36, 0.28 mean 0.25	< 0.05 (3)
C (157 ppm)	< 0.05 (3)	< 0.05 (3)	0.32, 0.90, 0.34 mean 0.52	< 0.05, 0.070, < 0.05 mean 0.057
D (607 ppm)	0.15, 0.083, 0.064 mean 0.097	0.15, < 0.05, < 0.05 mean 0.083	7.0, 3.9, 2.1 mean 4.4	0.32, 0.39, 0.20 mean 0.30
E (1680 ppm)	0.19, 0.25, 0.27 mean 0.23	0.086, 0.11, 0.080 mean 0.092	7.3, 7.2, 8.0 mean 7.5	0.55, 0.70, 1.2 mean 0.81

APPRAISAL

Residue and analytical aspects of imazapyr were considered for the first time by the present Meeting. The residue evaluation was scheduled for the 2013 JMPR by the Forty-fourth Session of the CCPR.

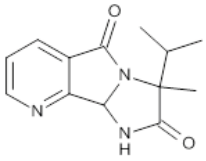
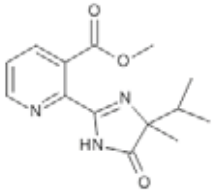
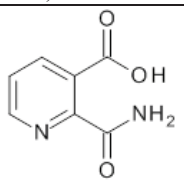
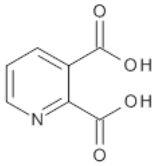
Imazapyr is a broad-spectrum herbicide in the imidazolinone family. Its primary use is as a post-emergence herbicide which is particularly effective on hard-to-control perennial grasses. It is non-selective, absorbed by foliage and rapidly translocated. The mode of action of imidazolinone herbicides is the inhibition of the enzyme acetohydroxy acid synthase (AHAS) which is a critical enzyme for the biosynthesis of branched chain amino acids necessary for cell growth and protein synthesis. The Meeting received information on identity, animal and plant metabolism, environmental fates in soil, rotational crops, analytical methods, storage stability, use patterns, supervised trials, farm animal feeding studies and fates of residues in processing.

2-[(*RS*)-4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl] nicotinic acid



Imazapyr is a 1:1 mixture of the enantiomers.

In this appraisal, the following abbreviated names were used for metabolites.

CL 247, 087	CL 240,000	CL 60,032	PDC
			
5*H*-imidazo [1',2':1,2] pyrrolo [3,4-b] pyridine -2(3*H*),5-dione, 1.9b α(β)-dihydro-3α- isopropyl-3-ethyl-	2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-3 -carboxymethyl pyridine	2-carbamoyl-nicotinic acid	Pyridine 2,3-dicarboxylic acid

Animal metabolism

The Meeting received animal metabolism studies with imazapyr in rats, lactating goats and laying hens. The metabolism and distribution of imazapyr in animals were investigated using the [^{14}C-carboxyl], [^{14}C-6-pyridine] and [^{14}C-5-imidazole]-labelled imazapyr.

Metabolism in rats was summarized and evaluated by the WHO panel of the JMPR in 2013.

Lactating goats were dosed with [pyridine-6-^{14}C]-imazapyr as a single daily oral dosage equivalent to a dietary level of 17.7 or 42.5 ppm for 7 consecutive days. Radioactivity was mainly excreted in the urine (65.3% and 60.4%) and faeces (16.1% and 19.0%) of the dose in the 17.7 ppm and 42.5 ppm dosed goats, respectively).

The TRR levels were < 0.01–0.01 mg equiv/kg and 0.01–0.02 mg equiv/kg in the milk samples of the 17.7 and 42.5 ppm dose goats, respectively. TRR levels for leg and loin muscles, liver, and fat were all less than the LOQ (0.05 mg equiv/kg) and these tissues were not analysed further. Detectable residues were found in the kidneys at 0.08 mg equiv/kg (17.7 ppm dose) and 0.11 mg equiv/kg (42.5 ppm dose).

Kidney residues were quantitatively extractable, with the majority (95.5%) isolated in a methanol/water fraction. The extracted ^{14}C residue in the milk and kidney was identified as unchanged imazapyr.

Lactating goats received [imidazole-5-^{14}C]-imazapyr at a dose equivalent to 47 ppm in the diet once daily for 7 consecutive days. After seven days, 58.7% and 34.4% of the administered radioactive dose were excreted in the urine and faeces, respectively. The TRR levels for the dosed goat were 0.014–0.015 mg equiv/kg for milk and 0.074 mg equiv/kg for kidney.

The major component in the milk (day-7) extract (65.6% of TRR, 0.01 mg/kg) was the parent compound (imazapyr). Polar unknowns (total 14.7% of TRR) were also present in the milk extract. Since these fractions were 0.002 mg equiv/kg and contained multiple components, no further characterization was attempted. Imazapyr was the predominant radioactive residue (81.9% of TRR,

0.061 mg/kg) in the kidney. Polar unknowns (total 11.6% of TRR) were also present in the kidney extract. The concentration of the remaining radioactive components in the kidney and the milk was below 0.01 mg equiv/kg individually.

Laying hens were orally dosed with [pyridine-6-^{14}C]-imazapyr at the actual dietary dose equivalent to 2.0 or 9.7 ppm in the feed for 7 consecutive days. The majority of the dose was rapidly eliminated in the excreta. Elimination of ^{14}C via the excreta accounted for 90.5 and 91.7% of the total dose for the low and high dose, respectively.

During treatment, TRR in the egg samples was less than the LOQ (0.01 mg equiv/kg). Residues in skin with adhering fat, muscle, liver and kidney tissues were all less than the LOQ (0.01 mg equiv/kg). Parent compound or derived residues are excreted without retention or accumulation in eggs and edible poultry tissues.

In animal metabolism studies, imazapyr was the major component in milk and kidney of lactating goat, but no residue was found in eggs and all tissues of laying hens.

Plant metabolism

Imazapyr is used in three different situations:

- Directed sprays for weed control (crop not intentionally treated)
- Use for weed control with a crop (crop treated)
- Selective use in genetically modified imidazolinone-resistant crops (crop treated)

Plant metabolism studies were conducted with imazapyr to investigate these three situations.

The Meeting received plant metabolism studies performed on soya bean, maize, sugarcane, oil palm, clover and Bermuda grass with imazapyr [^{14}C] labelled in two positions ([^{14}C-3-pyridine] and [^{14}C-6-pyridine]).

Directed sprays to weeds

In a sugar cane metabolism study, [pyridine-6-^{14}C]-imazapyr was applied to the soil surface once as a pre-emergence application at a rate equivalent to 0.28 kg ai/ha. Stalk samples were taken at maturity approximately 14 months after the pre-emergence treatment. There were no detectable residues in the treated samples.

In an oil palm metabolism study, [pyridine-6-^{14}C]-imazapyr was applied to the ground beneath an actively fruiting oil palm at an application rate of 1.0 kg ai/ha. Fruit samples were collected from the oil palm as they ripened at 0 hour, 7, 30 and 62 days after treatment. Palm oil was extracted from the fruits using a hexane: water mixture (3:1, v/v). Residual radioactivity levels were below the LOQ (0.03 mg equiv/kg) at any given time in the palm oil, aqueous phase, fruit marc, kernel shell and kernel nut. The results indicate that imazapyr derived residues will not accumulate in the palm oil of an actively fruiting oil palm after application to the ground beneath the palm.

Weed control

In a clover metabolism study, [pyridine-6-^{14}C]-imazapyr was applied to clover at a rate of 1.68 kg ai/ha. Clover foliage was collected at 0, 4, 10, 15 and 21 days after treatment (DAT). Phytotoxic effects were apparent four days after application of the test material to the clover in the test plot. The phytotoxicity remained apparent until the final collection 21 days after treatment. The TRR levels in foliage ranged from 23–49 mg equiv/kg. The majority of the radioactivity was unchanged imazapyr ranging from 68–99% of TRR. The major metabolite was CL 247,087 with CL 240,000 as a minor metabolite (their sum accounting for 0.02–18% of TRR).

In a Bermuda grass metabolism study, [pyridine-6-^{14}C]-imazapyr was applied to Bermuda grass at a rate of 1.68 kg ai/ha. Bermuda grass foliage was collected 0, 4, 10, 15 and 21 days after treatment (DAT). Phytotoxic effects were apparent four days after application of the test material to

the Bermuda grass in the test plot. The phytotoxicity remained apparent until the final collection 21 days after treatment. TRR levels in foliage were the highest at 0 DAT (64 mg equiv/kg), and then were 18 mg equiv/kg (4 DAT), 22 mg equiv/kg (10 DAT), 25 mg equiv/kg (15 DAT) and 48 mg equiv/kg (21 DAT). The majority of the radioactivity was unchanged imazapyr ranging from 78–97% of TRR. Three other identified radio-components were PDC, CL 247,087 and CL 240,000. The amount of PDC increased with time to 13% of TRR at 21 DAT. The amount of CL 247,087 plus CL 240,000 ranged from 0% to 10.5% of TRR between 4 DAT and 21 DAT.

Imidazolinone-resistant crops

In a soya bean metabolism study, [pyridine-3-^{14}C]-imazapyr (SL formulation) was applied once to the above ground portion of transgenic soya bean plants at BBCH growth stage 65 at an application rate of 0.11 kg ai/ha. The forage was harvested approximately one hour after application and the hay was harvested 35 days after application. Soya bean straw, pods and seeds were harvested when mature at 98 days after treatment.

The TRR of soya bean forage was 0.66 mg equiv/kg, soya bean hay was 0.25 mg equiv/kg, soya bean seed was 0.062 mg equiv/kg, soya bean straw was 0.079 mg equiv/kg and soya bean pod was 0.15 mg equiv/kg. Imazapyr was detected in all matrices and was the most abundant component of the residue in soya bean forage (0.60 mg/kg, 93.6% TRR), hay (0.094 mg/kg, 37.3% TRR), and seed (0.024 mg/kg, 34.2% TRR). Imazapyr was present in straw at 0.006 mg/kg (8.1% TRR) and pods at 0.018 mg/kg (12.7% TRR). A polar component M3 was the most abundant component in the pods (0.041 mg equiv/kg, 28.9% TRR). This component was also present in hay (0.028 mg equiv/kg, 11.1% TRR), straw (0.009 mg equiv/kg, 12.2% TRR) and seeds (0.0161 mg equiv/kg, 23.3% TRR), but was not detected in forage. This polar peak was isolated from the soya bean seeds, and shown to consist of multiple components, each present at ≤ 0.004 mg equiv/kg. M19 was present in the straw at 0.013 mg equiv/kg (17.6% TRR) and was also found in hay at 0.022 mg equiv/kg (8.7% TRR). This component had an intermediate polarity with a retention time of about 19 minutes and was not identified.

In a maize metabolism study, [pyridine-6-^{14}C]-imazapyr was applied to imidazolinone-resistant maize at the 3 to 4 leaf growth stage at treatment rates of 0.028 and 0.080 kg ai/ha. Samples of maize plants were harvested at 0 DAT (green plant), 14 DAT (green plant), 30 DAT (early forage), and 62 DAT (late forage). At maturity (114 DAT), the stalks, husks, and cobs with grain were collected.

For the 0 DAT, 96.3% (2.37 mg equiv/kg) of the TRR was extracted. For the 114 DAT fodder, 69.9% (0.020 mg equiv/kg) of the TRR was extracted and 30.1% (0.008 mg equiv/kg) remained in the PES. For the 114 DAT grain, 0.8 to 1.5% (< 0.002 mg equiv/kg) was extracted with hexane, 80.0 to 88.8% (0.023 to 0.076 mg equiv/kg) was extracted with methanol:water:hydrochloric acid (80:18:2, v/v/v), and 10.3 to 18.5% (0.005 to 0.009 mg equiv/kg) remained in the PES. Parent imazapyr constituted the major component of the extractable residue in the green plant, forage, fodder and grain (16.8 to 84.0% of extracted TRR, 0.003 to 2.0 mg/kg). The residue levels of the minor components in the 30 DAT to 114 DAT samples were all < 0.01 mg equiv/kg.

In the plant metabolism studies on soya bean (imidazolinone-resistant), maize (imidazolinone-resistant), sugarcane, oil palm, clover and Bermuda grass, Imazapyr is the major component of the residues found in soya bean, maize, clover, and Bermuda grass. CL 247,087 and PDC were also significant components of the residues in clover and Bermuda grass.

Environmental fate

The Meeting received information on aerobic soil metabolism, soil photolysis, rotational crop and hydrolysis.

In soil under the aerobic conditions, the DT_{50} ranged from 15 months to 7.5 years at 20 °C–35 °C. At 12 months after application, imazapyr remained in soil was 60.5–89.3% of the applied radioactivity. Minor degradates were identified as PDC, CL 60,032 and CL 240,000.

In soil photolysis study, there was 11% degradation of imazapyr over the 28 days of continuous irradiation. There were at least five degradation products formed, none of which accounted for > 10% of the applied dose. The photodegradation half-life of imazapyr was 149 days at 25 °C.

In confined rotational crop study, rotational crops (wheat, radish, lettuce and soya bean) were planted at 120 days after treatment (DAT) for wheat, 271 DAT for radish, lettuce and soya bean, 420 DAT for radish and lettuce. The test substance was applied as a post-emergence application to imidazolinone-resistant maize plants at the 6-leaf stage at a rate of 0.028 kg ai/ha.

The TRR in wheat forage, straw and grain; lettuce; radish foliage and root; and soya bean forage, hay, and seed were all < 0.002 mg/kg, the limit of detection of the radio assay.

A series of rotational crops, namely carrot and lettuce were planted at 330 and 540 DAT, winter wheat planted at 359 DAT, spring wheat at 520 DAT. A single application of [^{14}C] imazapyr was made to the soil at a rate of 0.885 kg ai/ha which is 10 times the highest GAP rate for crops used in rotation.

The TRR in follow crops were at < 0.01 to 0.02 mg equiv/kg at the various plant back intervals. Residue in the rotational crops included the unchanged imazapyr which ranged from < 0.001 to 0.003 mg/kg. Metabolites were not detected (< 0.001 mg/kg). The Meeting noted that residues are not expected on rotational crops.

Imazapyr is used for paddy rice. In a hydrolysis study, imazapyr was stable in water (pH 5, 7 and 9) at 25 °C.

Methods of analysis

The Meeting received description and validation data for analytical methods for residues of imazapyr in plant and animal commodities.

In most of the methods for determination of imazapyr in plants, homogenized samples were extracted with acidic aqueous methanol or acidic aqueous acetone, and the extract was cleaned up with column chromatography using solid phase extraction and/or strong cation exchange cartridges. Residues were determined by HPLC with UV or MS/MS detection. The methods of analysis for a range of substrates were validated with LOQs of the 0.05 mg/kg for imazapyr.

In the methods for animal commodities, homogenized samples were extracted with acidic solvent, and the extract was cleaned up by solvent partition and solid phase extraction. Residue of imazapyr was determined by capillary electrophoresis with UV detection. The methods of analysis were validated with the LOQ of 0.01 mg/kg for milk and milk fat, and 0.05 mg/kg for tissues of cattle.

No multi-residue method was submitted.

Stability of residues in stored analytical samples

The Meeting received information on the freezer storage stability of imazapyr in plant (maize grain, forage and fodder and soya bean seed), their processed (soya bean meal and oil) commodities and animal products.

Storage stability results indicate that imazapyr residue was stable for at least 3 months in soya bean (seed, laminated soya bean, meal and oil), at least 6 months in milk, at least 8 months in muscle and liver, and at least 27 months in maize (grain, forage and fodder).

The periods of storage stability studies generally cover the sample storage intervals of residue trials.

Definition of the residue

In the lactating goat metabolism studies, TRRs in kidney (0.074–0.11 mg equiv/kg) was higher than those in milk (< 0.01–0.02 mg equiv/kg), liver (< 0.05 mg equiv/kg), muscle (< 0.05 mg equiv/kg) and fat (< 0.05 mg equiv/kg). Imazapyr is the major component of the residue in kidney (82% TRR)

and milk (67% TRR). The concentration of the remaining radioactive components in the kidney and milk were below 0.01 mg/kg.

The Meeting decided that imazapyr is suitable analytes for enforcement purposes and dietary risk assessment in animal commodities.

The octanol/water coefficient (log P_{ow}) of imazapyr is –3.96 at 20 °C (pH 7). The Meeting considered the residue of imazapyr is not fat soluble.

In plant metabolism studies, parent imazapyr was a major component (8.1–99% TRR) in soya bean (forage, hay, seed, straw and pods), maize (forage, grain and fodder), clover and Bermuda grass. Imazapyr was a major compound in conventional and tolerant crops. Though PDC was found as a significant compound in Bermuda grass (13% TRR) which is a feed commodity, it may not be necessary to consider this compound for the definition of residue for food commodities.

The Meeting decided that parent imazapyr is a suitable analyte for enforcement purposes and dietary risk assessment in plant commodities.

The Meeting recommended the following residue definition:

For plants and animals the definition of the residue (for compliance with the MRL and for estimation of dietary intake): *Imazapyr*

The residue is not fat soluble

Results of supervised residue trials on crops

The Meeting received supervised trial data for the foliar application of imazapyr on lentil, soya bean, maize, rice, wheat, sugar cane, rape and sunflower. Residue trial data was made available from Argentina, Australia, Brazil, Canada, Uruguay and the USA.

Labels were available from Australia, Canada, Latin American countries and the USA describing the registered uses of imazapyr.

Pulses

Lentil (dry)

Data were available from supervised trials on imidazolinone-tolerant lentils in Canada.

The GAP on imidazolinone-tolerant lentil of Canada is a foliar application at a maximum rate of 0.0091 kg ai/ha with a PHI of 60 days.

Imazapyr residues in lentil seeds from independent trials in Canada matching GAP were (n=4): 0.06 (2) and 0.08 (2) mg/kg.

Based on the trials for lentils in Canada, the Meeting estimated a maximum residue level and an STMR value for imazapyr in lentil seeds of 0.3 and 0.07 mg/kg respectively.

Soya bean (dry)

Data were available from supervised trials on imidazolinone-tolerant soya beans in Brazil.

No GAP of Brazil was available for imidazolinone-tolerant soya beans.

The Meeting agreed that no recommendation could be made for soya beans.

Cereal grains

Maize

Data were available from supervised trials on imidazolinone-tolerant maize in Argentina, Brazil, Australia and the USA.

The GAP on imidazolinone-tolerant maize of Argentina is a foliar application at a maximum rate of 0.025 kg ai/ha with the application timing before reaching 6th fully developed leaf status.

Imazapyr residues in maize grains from trials in Argentina matching GAP were (n=7): < 0.05 (7) mg/kg.

Trials from Australia on maize were reported for a foliar application of a SL formulation (GAP: a foliar application at a rate of 0.018–0.022 kg ai/ha with a PHI not required when used as directed at the application timing of 2–6 leaf stage for crop). Imazapyr residue in maize grains from data in Australia at exaggerated rate of 0.032 kg ai/ha (1.5 × GAP rate) were < 0.05 (2) mg/kg and 0.048 kg ai/ha (2.2 × GAP rate) were < 0.05 (2) mg/kg.

The GAP on imidazolinone-tolerant maize of the USA is a foliar application at a maximum rate of 0.016 kg ai/ha with a PHI of 45 days at the application timing of before 6 leaf stage for crop. However, imazapyr residue trials on maize in the USA did not match the GAP of the USA.

The Meeting decided to use the data of imazapyr residues in maize grain from the trials in Argentina.

Based on the trials for maize in Argentina, the Meeting estimated a maximum residue level, an STMR value for imazapyr in maize of 0.05 (*) and 0.05 mg/kg respectively.

Rice

Data were available from supervised trials on imidazolinone-tolerant paddy rice in Brazil.

The GAP on imidazolinone-tolerant rice of Brazil is two foliar applications at a maximum rate of 0.074 kg ai/ha with a PHI of 60 days.

Trials from Brazil on rice were reported for two foliar applications at a rate of 0.12 kg ai/ha with a PHI of 60 days. Imazapyr residues in rice grains from trials at a rate of 0.12 kg ai/ha in Brazil were (n=4): < 0.05 (3) and 0.05 mg/kg. However, the trials for rice in Brazil were insufficient to estimate a maximum residue level for the commodity.

The Meeting could not estimate a maximum residue level for imazapyr in rice.

Wheat

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

The GAP on imidazolinone-tolerant wheat of Australia is a foliar application at a rate of 0.004–0.007 kg ai/ha with a PHI not required for wheat grains when used as directed.

Imazapyr residues in wheat grains from trials in Australia matching GAP were (n=3): < 0.05 (3) mg/kg. Imazapyr residues in wheat grains from data in Australia at exaggerated rate of 0.014 kg ai/ha (2 × GAP rate) were < 0.05 (5) mg/kg and 0.028 kg ai/ha (4 × GAP rate) were < 0.05 (5) mg/kg.

Based on the trials for wheat in Australia, the Meeting estimated a maximum residue level and an STMR value for imazapyr in wheat grains of 0.05 (*) and 0 mg/kg respectively.

Grasses for sugar or syrup production

Sugar cane

Data were available from supervised trials on sugar cane in Argentina and Brazil.

The GAP on sugar cane of Argentina is two applications with spray to weeds at a maximum rate of 0.5 kg ai/ha with 30–45 days before planting. The GAP on sugar cane of Brazil is an application with spray to weeds of a rate of 0.13–0.5 kg ai/ha with a PHI not required when used as directed.

Imazapyr residue in sugar cane from trials in Argentina matching GAP was (n=1): < 0.05 mg/kg. However, the trial for sugar cane in Argentina was insufficient to estimate a maximum residue level for the commodity.

The Meeting could not estimate a maximum residue level for imazapyr in sugar cane.

Oilseed

Rape seed

Data were available from supervised trials on imidazolinone-tolerant rape in Australia and Canada.

The GAP on imidazolinone-tolerant rape of Canada is a foliar application at a maximum rate of 0.0091 kg ai/ha with a PHI of 60 days.

Imazapyr residues in rape seeds from independent trials in Canada matching GAP were (n=10): < 0.05 (10) mg/kg.

Trials from Australia on rape were reported for a foliar application of a WG formulation (GAP: a foliar application at a rate of 0.0045–0.011 kg ai/ha with a PHI not required when used as directed).

Imazapyr residues in rape seeds from trials in Australia matching GAP were (n=2): < 0.05 (2) mg/kg. Imazapyr residues in rape seeds from data in Australia at an exaggerated rate of 0.028 kg ai/ha (2.3 × GAP rate) were < 0.05 (2) mg/kg.

Based on the trials for rape seeds in Canada and Australia, the Meeting estimated a maximum residue level, an STMR value for imazapyr in rape seed of 0.05 (*) and 0 mg/kg respectively.

Sunflower seed

Data were available from supervised trials on imidazolinone-tolerant sunflower in Argentina and Uruguay.

The GAP on imidazolinone-tolerant sunflower of Argentina and Uruguay is a foliar application at a maximum rate of 0.080 kg ai/ha with the application timing of early post emergence.

Imazapyr residues in sunflower seeds from trials in Argentina and Uruguay approximating GAP were (n=15): < 0.01 (10), 0.01, 0.03 (2) and < 0.05 (2) mg/kg.

Based on the trials for sunflower in Argentina and Uruguay, the Meeting estimated a maximum residue level and an STMR value for imazapyr in sunflower seed of 0.08 and 0.01 mg/kg respectively.

Animal feedstuffs

Maize fodder and forage

Data were available from supervised trials on imidazolinone-tolerant maize in Argentina, Australia and the USA.

Trials from Argentina on maize forage were reported for the foliar application of SC or WG formulation (GAP: a foliar application of a maximum rate of 0.025 kg ai/ha, the application timing before reaching 6th fully developed leaf status).

Trials from Australia on maize forage were reported for the foliar application of a SL formulation (GAP: a foliar application of a rate of 0.018–0.022 kg ai/ha at the application timing of 2–6 leaf stage for crop, do not graze or cut for stock food for 4 weeks after application and not required to harvest for grains when used directed).

Trials from the USA on maize fodder and forage were reported for the foliar application of ASU or WP formulation at a rate of 0.027 kg ai/ha (GAP: a foliar application of a maximum rate of 0.016 kg ai/ha, PHI of 45 days at the application timing of before 6 leaf stage for crop).

Maize fodder

In the residue trials for imazapyr on maize fodder in the USA, no samples were collected on 45 days after application. The Meeting could not estimate a maximum residue level for imazapyr in maize fodder.

Maize forage

Imazapyr residues in maize forage from trials in Argentina matching GAP were (n=4): < 0.05 (4) mg/kg as received basis. Imazapyr residues in maize forage from data in Argentina at exaggerated rate of 0.040 kg ai/ha (1.6 × GAP rate) were < 0.05 (4) mg/kg and 0.050 kg ai/ha (2 × GAP rate) were < 0.05 (4) mg/kg.

Imazapyr residues in maize forage from data in Australia at exaggerated rate of 0.032 kg ai/ha (1.5 × GAP rate) were < 0.05 (2) mg/kg and 0.048 kg ai/ha (2.2 × GAP rate) were < 0.05 (2) mg/kg.

Imazapyr residues in maize forage from trials in the USA at exaggerated rate of 0.027 kg ai/ha (1.7 × GAP rate) were < 0.05 (18) mg/kg as received basis.

Based on the residues in maize forage from trials in Argentina, Australia and the USA, the Meeting estimated a median residue value and a highest residue value for imazapyr in maize forage both at 0 mg/kg.

Wheat straw and forage

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

Trials from Australia on wheat were reported for the foliar application of a SL formulation (GAP: a foliar application of a rate of 0.004–0.007 kg ai/ha, do not graze or cut for stock food for 4 weeks after application and not required to harvest for grains when used directed).

Wheat straw

Imazapyr residues in wheat straw from trials in Australia matching GAP were (n=3): < 0.05 (3) mg/kg as received basis. Imazapyr residues in wheat straw from data in Australia at exaggerated rate of 0.014 kg ai/ha (2 × GAP rate) were < 0.05 (5) mg/kg and 0.028 kg ai/ha (4 × GAP rate) were < 0.05 (5) mg/kg.

Based on the residues in wheat straw from trials in Australia, the Meeting estimated a maximum residue level, a median residue value and a highest residue value for imazapyr in wheat straw and fodder of 0.05 (*), 0 and 0 mg/kg respectively.

Wheat forage

Imazapyr residues in wheat forage from trials in Australia matching GAP were (n=3): < 0.05 (3) mg/kg as received basis. Imazapyr residues in wheat forage from data in Australia at exaggerated rates of 0.014 kg ai/ha (2 × GAP rate) were < 0.05 (5) and 0.087 mg/kg, and 0.028 kg ai/ha (4 × GAP rate) were < 0.05 (5) and 0.11 mg/kg as received basis.

Based on the residues in wheat forage from trials in Australia, the Meeting estimated a median residue value and a highest residue value for imazapyr in wheat forage both at 0.05 mg/kg.

Rape forage

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

Trials from Australia on rape were reported for the foliar application of a WG formulation (GAP: a foliar application of a rate of 0.005–0.012 kg ai/ha, do not graze or cut for 5 weeks).

Imazapyr residues in rape forage from trials in Australia matching GAP were (n=1): < 0.05 mg/kg. Imazapyr residues in rape forage from data in Australia at an exaggerated rate of 0.028 kg ai/ha (2.3 × GAP rate) or at DALA 25–29 days were < 0.05 (4) mg/kg.

Based on the residues in rape forage from trials in Australia, the Meeting estimated a median residue value and a highest residue value for imazapyr in rape forage both at 0 mg/kg.

Fate of residues during processing

High temperature hydrolysis

The degradation of [^{14}C] imazapyr was studied under hydrolytic conditions at high temperatures in sterile aqueous buffers at pH 4, 5 and 6 for periods of up to 60 minutes so as to simulate common processing practice (pasteurization, baking/brewing/boiling, and sterilization). No degradates were detected at any of the investigated pH and temperature ranges. Imazapyr is stable under hydrolytic conditions at high temperatures.

Residues in processed commodities

The fate of imazapyr residues has been examined in maize grains, rape seeds and sunflower seeds processing studies. Based on the results of processing studies conducted in Canada and the USA in combination with the residues from supervised trials, the Meeting concluded that no residues are expected in processed rape and sunflower commodities. Estimated processing factors and the derived STMR-Ps are summarized in the Table below.

Processing factors, STMR-P and HR-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)
Maize grain	Meal	1.2	1.2	0.05	0.06
	Crude oil	< 0.50	< 0.50		0.025

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

Residue in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of imazapyr in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual 2009. Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed in a dry weight basis.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Appendix IX of the FAO manual. The calculations were made according to the animal diets from US-Canada, EU, Australia and Japan in the Table (Appendix IX of the FAO manual).

Livestock dietary burden, imazapyr, ppm of dry matter diet								
	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	0.063	0.063	0.085	0.085	0.20 [a]	0.20 [b]	0.061	0.061
Dairy cattle	0.083	0.083	0.057	0.057	0.15	0.15 [c]	0.045	0.045
Poultry—broiler	0.056d	0.056e	0.040	0.040	0.014	0.014	0.040	0.040
Poultry—layer	0.056	0.056	0.073 [d]	0.073 [e]	0.014	0.014	0.045	0.045

[a] Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian meat, fat, edible offal and milk

[b] Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian meat, fat and edible offal

[c] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk

[d] Highest maximum broiler poultry dietary burden suitable for MRL estimates for poultry meat, fat, edible offal and eggs

[e] Highest mean broiler poultry dietary burden suitable for STMR estimates for poultry meat, fat, edible offal and eggs

Farm animal feeding studies

The Meeting received a lactating dairy cow feeding studies using imazapyr, which provided information on likely residues resulting in animal commodities and milk from imazapyr residues in the animal diet.

A poultry feeding study was not submitted as the expected residues of imazapyr in poultry feed were extremely low. A poultry metabolism study at a dose rate of 9.7 ppm imazapyr in feed demonstrated that there was very low transfer to eggs and tissues with all residues of imazapyr less than 0.01 mg/kg.

Lactating dairy cows

Lactating dairy cows were dosed with imazapyr for 28–29 days at the dose equivalent to 58, 157, 607 and 1680 ppm in the diet. Residues of imazapyr were at or less than the LOQ (0.01 mg/kg) in whole milk at 58 ppm of feeding level. In the three higher dose groups (157, 607 and 1680 ppm feed), imazapyr residues in milk reached a plateau after 2–3 days. In kidney, imazapyr was detected as the highest concentrations among all tissues and milk in all treated groups.

Animal commodities maximum residue levels

For MRL estimation, the residue in the animal commodities is imazapyr.

The maximum dietary burden for beef and dairy cattle is 0.20 and is lower than the dose level in the lactating goat metabolism study of 18 ppm and the lactating cow feeding study of 58 ppm. In the metabolism study, in which imazapyr equivalent to 18 ppm in the diet was dosed to lactating goats for 7 consecutive days, residues of imazapyr were detected at 0.01 mg/kg in milk and 0.08 mg/kg in kidney. The maximum dietary burden for beef and dairy cattle is 1% of the dose rate in feed of the metabolism study.

The Meeting estimated a maximum residue level of 0.01 (*) mg/kg and an STMR value of 0 mg/kg in milk.

The Meeting estimated a maximum residue level of 0.05 (*) mg/kg, an STMR value of 0 mg/kg and an HR value of 0 mg/kg in mammalian meat and fat.

The Meeting estimated a maximum residue level of 0.05 (*) mg/kg, an STMR value and an HR value of 0.0008 in mammalian edible offal.

The maximum dietary burden for broiler and layer poultry is 0.073 and is lower than the dose level in the laying hen metabolism study of 9.7 ppm. In the metabolism study, in which imazapyr equivalent to 9.7 ppm in the diet was dosed to laying hens for 7 consecutive days, no residues of imazapyr exceed 0.01 mg/kg were detected in tissues and eggs.

The Meeting estimated a maximum residue level of 0.01 (*) mg/kg, an STMR value of 0 mg/kg and an HR value of 0 mg/kg in poultry meat, fat, edible offal and eggs.

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 and IESTI assessment.

Plant and animal commodities:

Definition of the residue for plant commodities (for compliance with the MRL and for estimation of dietary intake): *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		
MO 0105	Edible offal (Mammalian)	0.05*	0.0008	
PE 0112	Eggs	0.01*	0	
VD 0533	Lentil (dry)	0.3	0.07	
GC 0645	Maize	0.05*	0.05	
MF 0100	Mammalian fats (except milk fats)	0.05*	0	
MM 0095	Meat (from mammals other than marine mammals)	0.05*	0	
ML 0106	Milks	0.01*	0	
PO 0111	Poultry, Edible offal of	0.01*	0	
PF 0111	Poultry fats	0.01*	0	
PM 0110	Poultry meat	0.01*	0	
SO 0495	Rape seed	0.05*	0	
SO 0702	Sunflower seed	0.08	0.01	
GC 0654	Wheat	0.05*	0	
AS 0654	Wheat straw and fodder, dry	0.05*	0	0

* At or about the LOQ.

Commodity Name	STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
Maize forage	0	0
Maize meal	0.06	
Maize oil	0.025	
Rape forage	0	0
Wheat forage	0.05	0.05

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDIs) of imazapyr were calculated for the 13 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (Annex 3). 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 intakes of residues of imazapyr, resulting from the uses considered by current JMPR, are unlikely to present a public health concern.

Short-term intake

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

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IZ-731-007	Mahl, T	1995	CL 243,997 (Imazapyr): Crop residue study with AC 243,997 on field corn in South Dakota 1993, American Cyanamid Co., Princeton NJ, USA, GLP, Unpublished
IZ-731-008	Mahl, T.	1995	CL 243,997 (Imazapyr): Crop residue study with AC 243,997 on field corn in Texas 1994, American Cyanamid Co., Princeton NJ, USA, GLP, Unpublished
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IZ-731-021	Steling, C	1998	CL 243,997: Determination of CL 243,997 residues in corn samples from Argentina, LAADL—Latin America Agricultural Development Laboratory, Rio de Janeiro, Brazil, GLP, Unpublished
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