IMAZAMOX (276)

The first draft was prepared by Professor Mi-Gyung Lee, Andong National University, Republic of Korea

EXPLANATION

Imazamox is an imidazolinone herbicide used for the control of a wide spectrum of grassy and broadleaf weeds. The mode of action of imazamox is the inhibition of acetohydroxy acid synthase (AHAS), an enzyme involved in the synthesis of essential amino acids (leucine, isoleucine and valine) for the development of the plant.

At the 45th session of the CCPR (2013), it was scheduled for evaluation as a new compound by the current JMPR. Imazamox has been registered in various countries worldwide.

The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials and processing studies.

IDENTITY

ISO common name: Imazamox

Chemical name

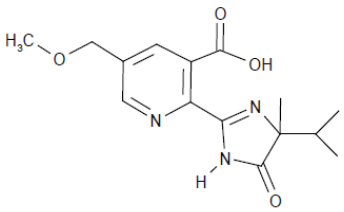
IUPAC: *(RS)*-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-5-methoxymethylnicotinic acid

CAS: 2-[4,5-dihydro-4-methyl-4-(1-methylethyl)-5-oxo-1*H*-imidazol-2-yl]-5-(methoxymethyl)-3-pyridinecarboxylic acid

CAS Registry No.: 114311-32-9

CIPAC No.: 619

Structural formula



Molecular formula $C_{15}H_{19}N_3O_4$

Molecular mass: 305.3

PHYSICAL AND CHEMICAL PROPERTIES

Purified active ingredient

Property	Result	Method	References
Melting point:	165.5–167.2 °C	OECD 102	ID-303-001 Coover, 1994a
Relative density:	1.39 at 20 °C	EEC Method A3	ID-301-001 Patel, 1993a
Vapour pressure:	$< 1.33 \times 10^{-5}$ Pa at 25 °C or $< 1.0 \times 10^{-7}$ Torr at 25 °C	US EPA 63-9	ID-306-001 Morelli, 1994a
Volatility:	Henry's Law Constant (k_H) at 25 °C: $< 9.76 \times 10^{-7}$ Pa m^3 mol^{-1}	Calculation	ID-306-002 Martin, 1997a
Physical state:	Powdered solid at 24.5 °C	Visual inspection, conforming to U.S. EPA registration requirements	ID-301-001 Patel, 1993a

Property	Result	Method	References
		under 40CFR 63-3	
Colour:	White.	ASTM D1535-68	ID-301-001 Patel, 1993a
Odour:	Odourless	ASTM D1292-86	ID-301-001 Patel, 1993a
Solubility in water including effect of pH:	at 25 °C: pH 5: 114 g/L pH 7: > 643 g/L pH 9: > 652 g/L at 10 °C, deionized water: 4.011 g/L at 20 °C, deionized water: 4.413 g/L at 30 °C, deionized water: 4.488 g/L	EEC A.6	ID-310-001 Coover, 1994b
Solubility in organic solvents:	at 25 °C: hexane: 0.0006 g/100 mL solvent methanol: 6.68 g/100 mL solvent acetonitrile: 1.85 g/100 mL solvent toluene: 0.21 g/100 mL solvent acetone: 2.93 g/100 mL solvent dichloromethane: 14.3 g/100 mL solvent ethyl acetate: 1.02 g/100 mL solvent	EEC A.6	ID-310-001 Coover, 1994b
Partition Coefficient:	K_{ow} = 5.36 at 25 °C log K_{ow} = 0.73 at 25 °C	US EPA 63-11	ID-315-001 Morelli, 1994b
Hydrolysis rate:	No hydrolysis was observed at pH 4 and pH 7 after 30 days at 50 °C. at pH 9: 70 °C: $t_{1/2}$ = 1.70 d 60 °C, $t_{1/2}$ = 4.17 d 50 °C: $t_{1/2}$ = 11.9 d 25 °C: $t_{1/2}$ = 192 d (extrapolated)	EEC C.7	ID-322-002 Holman, 1997b
Photochemical degradation:	at 25 °C: pH 5: $t_{1/2}$ = 6.8 h pH 7: $t_{1/2}$ = 6.7 h pH 9: $t_{1/2}$ = 7.1 h	US EPA 161-2	ID-324-002 An & Ta, 1995a
Dissociation Constant:	pK_a = 2.3, 3.3, 10.8 Active substance is the free acid.	US EPA 63-10	ID-320-001 Melcer, 1993a

Technical active ingredient

Property	Result	Method	References
Melting point	166.0–166.7 °C	OECD 102	ID-303-001 Coover, 1994a
Decomposition or sublimation temperature	Decomposition commences ca. 160 °C.	Accelerating rate calorimetry over the temperature range of 50–350 °C	ID-334-001 Patel, 1994a
Physical state:	Powdered solid at 24.5 °C	Visual inspection, conforming to U.S. EPA registration requirements under 40CFR 63-3	ID-301-001 Patel, 1993a
Colour:	Off—white	ASTM D1535-68	ID-301-001 Patel, 1993a
Odour:	Odourless	ASTM D1292-86	ID-301-001 Patel, 1993a
Solubility in water including effect of	at 25 °C: pH 5: 116 g/L	EEC A.6	ID-310-001 Coover,

Property	Result	Method	References
pH:	pH 7: > 626 g/L pH 9: > 628 g/L at 10 °C, deionized water: 3.795 g/L at 20 °C, deionized water: 4.160 g/L at 30 °C, deionized water: 4.193 g/L		1994b
Solubility in organic solvents:	at 25 °C: hexane: 0.0007 g/100 mL solvent methanol: 6.75 g/100 mL solvent acetonitrile: 1.90 g/100 mL solvent toluene: 0.22 g/100 mL solvent acetone: 3.09 g/100 mL solvent dichloromethane: 21.8 g/100 mL solvent ethyl acetate: 1.05 g/100 mL solvent	EEC A.6	ID-310-001 Coover, 1994b

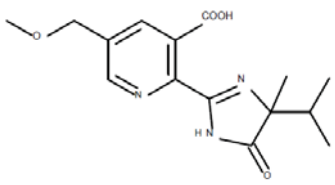
Formulations

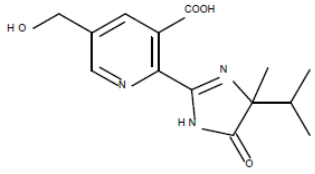
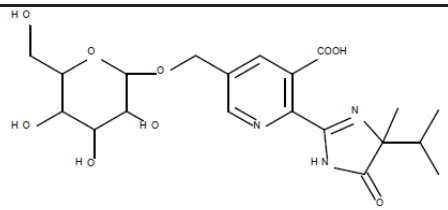
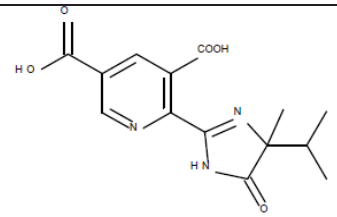
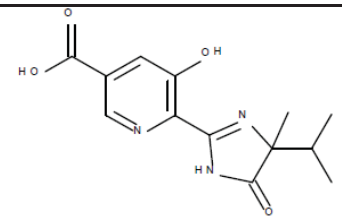
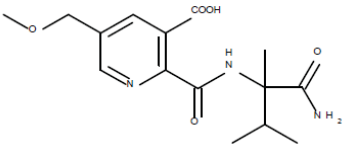
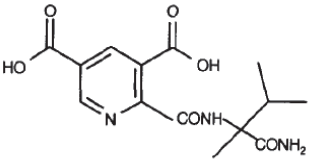
The following formulations are commercially available:

Formulation type	Content of imazamox	Other active substances
Aqueous suspension concentrate (SC):	20 g ai/L	560 g ai/L 2,4-D
	17.5 g ai/L	375 g ai/L metazachlor
	25 g ai/L	375 g ai/L metazachlor
	17.5 g ai/L	375 g ai/L metazachlor, 100 g ai/L quinmerac
	6.25 g ai/L	375 g ai/L metazachlor, 125 g ai/L quinmerac
Emulsifiable concentrate (EC):	16. 7 g ai/L	250 g ai/L pendimethalin
Soluble concentrate (SL):	40 g ai/L	
	120 g ai/L	
	0.805%	
	33 g ai/L	15 g ai/L imazapyr
	28 g ai/L	600 g ai/L bentazone
	22.4 g ai/L	480 g ai/L bentazone
	20 g ai/L	429 g ai/L bentazone
Water dispersible granule (WG):	70%	
Water dispersible granule (WG):	35%	35% imazethapyr

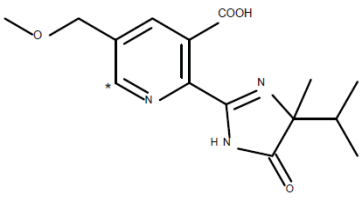
METABOLISM AND ENVIRONMENTAL FATE

The following links code number and structure or description of the compound appearing in the various metabolism and environmental fate studies.

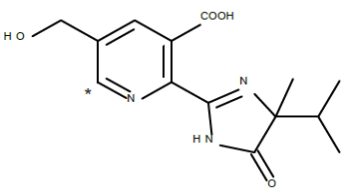
Code (MW)	Synonyms	IUPAC chemical name	Structure	Found in
CL 299263 (305)	Imazamox; BAS 720 H	(RS)-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-5-methoxymethylnicotinic acid		rat, goat, hen, oilseed rape, soya bean, pea, alfalfa, maize, wheat, soil, water

Code (MW)	Synonyms	IUPAC chemical name	Structure	Found in
CL 263284 (291)	Reg. No. 4110773; M715H001	5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazazolin-2-yl) nicotinic acid		oilseed rape, soya bean, pea, alfalfa, maize, wheat
CL 189215 (453.5)	Reg. No. 4110445; M715H002	5-[(β-glucopyranosyl oxy) methyl]-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl) nicotinic acid		oilseed rape, soya bean, pea, alfalfa, maize, wheat
CL 312622 (305)	Reg. No. 4110542; M720H002	2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-3,5-pyridine-dicarboxylic acid		oilseed rape, soya bean, pea, alfalfa, maize, wheat, soil
CL 354825 (277)	–	5-hydroxy-6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-nicotinic acid		maize, soil
CL 336554 (323)	–	2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-5-(methoxymethyl)-nicotinic acid		hydrolysis product at pH 9
CL 152795		2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-3,5-pyridinedicarboxylic acid		impurity present in dosing solution in animal metabolism study

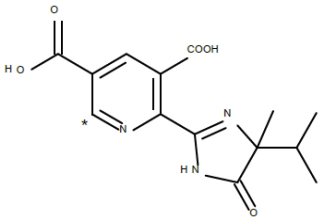
The metabolism and distribution of imazamox in animals and plants and the fate of imazamox in the environment were investigated using the following radiolabelled compounds (* indicates position of radiolabelling).



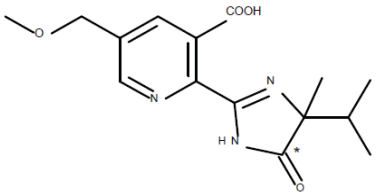
[pyridine-6-^{14}C]-imazamox



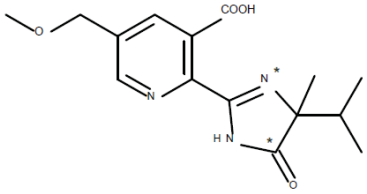
[pyridine-6-^{14}C]-CL 263284



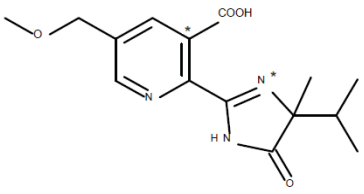
[pyridine-6-^{14}C]-CL 312622



[imidazolinone-5-^{14}C]-imazamox



[imidazolinone-5-^{14}C, 3-^{15}N]-imazamox



[pyridine-3-^{14}C, imidazolinone-3-^{15}N]-imazamox

Animal metabolism

The Meeting received information on the metabolism of imazamox in ruminants (lactating goats), poultry (laying hens) and laboratory animals (rat). Metabolism studies on rats were reviewed in the framework of toxicological evaluation by the current JMPR and relevant information is summarized below.

Rat

Imazamox administered to rats orally was primarily eliminated as unchanged test compound via the urine and secondly via the faeces. Biliary excretion was not an important route of elimination. Elimination was very efficient and occurred rapidly within 6–48 hours post-dose. In addition, the ratio of enantiomers did not change, demonstrating a similar absorption and excretion rate for the individual enantiomers of imazamox in the rat [Chiu 1995a, ID-440-003; Chiu 1996a, ID-440-004; Thiaener & Lutz 2012a, 2011/1080475].

Lactating goats

Imazamox

Lactating goats were orally given single daily doses of [pyridine-6-^{14}C]-imazamox for seven consecutive days at a level of either 2.08 or 11.6 ppm in the feed [Johnson 1994a, ID-440-002]. Radiolabelled imazamox was administered orally in gelatin capsules with lactose carrier to lactating goats. Mean feed consumption was 1890 and 1721 g per day for the low and high dose goats during the treatments. The milk, urine, faeces, and blood were collected daily starting one day before the first dosing for each animal group and continued throughout the dosing period. Kidney, liver, leg and loin muscle, and omental fat samples were collected approximately 20 hours after the last dosing. All samples were analysed for radioactivity content.

Samples were analysed by LSC or combustion followed by LSC. The validated detection limit of the radioassay method was approximately 0.01 mg eq/kg for milk, blood, and all tissues. Radioactivity in kidney and urine extracts was characterized using HPLC. Aliquots of the radioactive components isolated from kidney and urine were analysed by GC-MS for confirmation.

During treatment, the total radioactive residues (TRR) in the daily blood and milk samples were less than 0.01 mg eq/kg regardless of the treatment dose levels. The TRR in liver, leg and loin muscle, and omental fat were less than 0.01 mg eq/kg regardless of dose level. The imazamox-derived TRR in the kidney was 0.02 mg eq/kg from the goat dosed at 2.08 ppm and 0.06 ppm from the goat dosed at 11.6 ppm.

Elimination of ^{14}C radioactivity in the urine accounted for 91.2% and 64.8% of the total dose for the low and high doses, respectively. The radioactivity excreted in the faeces represented 14.8% and 24.0% of the total dose for the low and high doses, respectively. Total recovery of radioactivity in urine and faeces was 106.0% and 88.8% for the low and high doses, respectively.

Radioactive residues from kidney of the high dose goat were extractable (99%) with methanol/water (80:20, v/v). Analysis of the extract by reverse-phase HPLC showed that 89% of the

extractable TRR was attributable to imazamox. The identity of this compound was confirmed by GC-MS. The other radioactivity detected was distributed throughout the chromatogram at levels near background.

That imazamox parent is the only significant compound in tissues is supported by results for urine. Reverse-phase HPLC of goat urine showed 91% of radioactivity was imazamox (confirmed by GC-MS). A radioactive peak containing approximately 5% of the radioactivity recovered from the HPLC column occurred at a shorter retention time than imazamox and the remaining radioactivity occurred as minor components (< 0.5% of radioactivity). The component with a shorter retention time was isolated using HPLC fraction collection, partitioned from the mobile phase using dichloromethane, and reanalysed using HPLC. Of the radioactivity recovered from the HPLC column, 70% was imazamox (confirmed by GC-MS). The most likely explanation for this is that imazamox formed a weak complex with a component in the urine matrix, causing the shorter retention time. This then dissociated during isolation, giving imazamox.

In summary, orally administered imazamox in the goat is mainly excreted in urine. At highly exaggerated dose rates, there were no detectable imazamox-derived residues in milk during and after treatment, nor in tissues except kidney 20 hours after the last dose. The kidney contained 0.02 and 0.06 mg eq/kg total radioactive residue from the low and high dose ^{14}C-treated goats, respectively. This was mostly imazamox. Unaltered parent accounted for most of the excreted residue.

CL 263284

A ruminant metabolism study was conducted with [pyridine-6-^{14}C]-CL 263284 [Kao 1994a, IA-440-001]. Dose levels for the goats, administered orally in gelatin capsules, were 0, 2.33 or 14.5 mg/kg feed daily for seven days. Samples of blood, milk and excreta were collected daily. After seven days of dosing, the goats were sacrificed (approximately 20 hours after the last dose) and the tissues kidney, liver, muscle and fat were collected. Samples were analysed by LSC or combustion followed by LSC. The validated detection limit of the radioassay method was approximately 0.01 mg eq/kg. Kidney, urine and faeces samples were additionally analysed via HPLC-MS.

TRR levels in most control samples and most tissue samples (muscle, fat and liver) from the low and high dose treated goat and from all milk samples were non-detectable (< 0.01 mg eq/kg). The control sample from kidney showed a residue of 0.01 mg eq/kg and the kidney sample from the high dose goat was at 0.03 mg eq/kg.

Elimination of ^{14}C radioactivity via urine accounted for 14.6% and 18.2% of the total cumulative dose at low dose and high dose, respectively (81.7% and 67.8% via faeces). The recovery of radioactivity in urine and faeces totalled 96.3% and 86.0% at low dose and high dose, respectively.

All milk and all tissue samples contained less than 0.01 mg eq/kg of TRR, the detection limit, except the kidney samples from goat treated at the high dose rate.

Radioactive residues were extracted from kidney using methanol as solvent. The main radioactivity was observed in the extract (91%) and only minor amounts were detected in the PES (9%). The kidney extract was analysed by HPLC to determine the nature of the radioactive residue. The administered compound (CL 263284) was the minor radioactive residue (9% TRR, < 0.01 mg/kg). One further peak was present in the chromatogram (M1; 78% TRR, 0.02 mg eq/kg). M1 was further characterized by treatment with β-glucuronidase, sulfatase or buffer alone. M1 showed to be a very labile component and was easily degraded to CL 263284 under non-enzymatic or enzymatic hydrolysis. Because M1 was converted to CL 263284 in buffer (0.2 M potassium phosphate, pH 6.8) alone, it is likely that M1 was the parent compound weakly coupled with kidney endogenous components. It is suspected that M1 is a salt or conjugate of CL 263284 that is readily dissociated in aqueous solutions such as buffer.

HPLC analysis of the faeces extract of goat treated at high dose showed that 96% of the radioactivity had a retention tune corresponding to that of CL 263284 reference standard. HPLC analysis of the faeces extract of goat treated at low dose showed that the major component had a retention time at 9 minutes which is the same as the M1 component found in the high dose kidney.

The M1 component was very labile and was easily converted to CL 263284 under aqueous, acidic conditions or incubated with β-glucuronidase and sulfatase.

The results of this lactating goat study showed that the administered compound was excreted without retention or accumulation in milk. Of the edible tissues only kidney showed detectable residues and these were identified as the administered compound CL 263284 and a labile metabolite that converted to CL 263284 in aqueous solution. This shows a very low transfer of CL 263284-derived residues from animal feed into tissues and milk of lactating ruminants.

CL 312622

Lactating goats were orally treated with a mixture of [pyridine-6-^{13}C] and [pyrindine-6-^{14}C]-radiolabelled imazamox metabolite (CL 312622) [Tsalta 1999a, ID-440-006]. The three goats were dosed for five consecutive days with the nominal dietary equivalent of 0 ppm (control), 3.1 ppm (low dose) and 33 ppm (high dose) in the feed. Milk, urine and faeces samples were collected daily and edible tissues (muscle, fat, liver and kidney) were collected at sacrifice (approximately 22 hours after the last dose). Samples were analysed by LSC or combustion followed by LSC. The specific radioactivity afforded a validated detection limit of 0.006 mg eq/kg CL 312622 equivalents in the tissues, milk, urine and faeces.

TRR levels in all tissue and milk samples were non-detected (< 0.006 mg eq/kg), except kidney (0.025 mg eq/kg) and liver (0.009 mg eq/kg) of goats treated at high dose (33.4 mg/kg feed). Elimination of ^{14}C radioactivity via urine accounted for 8.5% and 7.0% of the total cumulative dose at low dose and high dose, respectively (90.4% and 90.0% via faeces). The recovery of radioactivity in urine and faeces totalled 98.9% and 97.0% at low dose and high dose, respectively.

Radioactive residues were extracted from kidney using acetone/water (3:1, v/v) as solvent. The overall recovery in the extractable portion was > 100% (by comparison with the TRR obtained by combustion); no radioactivity was observed in the PES. The kidney extract was analysed by HPLC to determine the nature of the radioactive residue. The administered CL 312622 was the predominant radioactive residue (60.0%). The impurity known to be present in the dosing solution (CL 152795) was also found in the extract at 11.0% of TRR.

Minor polar unknowns (total 10.3% of TRR) and non-polar unknowns (total 11.9% of TRR) were also present in the kidney extract. Since these fractions were equivalent to 0.003 mg eq/kg and contained multiple components, no further characterization was attempted.

Radioactive residues were extracted from liver according to a modification of the kidney extraction procedure. The percent extractable radioactivity was 98.7% and the percent TRR in the PES by combustion/LSC analysis yielded residues less than the validated detection limit.

The concentrated extract was analysed by HPLC after solid phase extraction (SPE) clean-up. The administered CL 312622 was present at 38.2% of TRR in the liver. The impurity known to be present in the dosing solution (CL 152795) was also found in the liver extract at 19.0% of the TRR as determined by HPLC. Polar radioactive components were present in the liver extract at 31.2% of the TRR. The majority of that fraction (22.9%) was not retained by the SPE cartridge, indicating it to be very polar material. Some non-polar metabolites were also present at a total concentration of 11.3% of the TRR. Since these fractions were equivalent to 0.003 mg eq/kg or lower and contained multiple components, no further characterization was attempted.

Data on the distribution of the recovered radioactivity from the kidney and the liver extract, as determined by HPLC, are summarized in table below.

Table 1 Distribution of radioactivity in high dose goat kidney and liver goat treated with [^{14}C]-CL 312622 (ID-440-006)

Compound	Kidney (0.025 mg eq/kg)		Liver (0.009 mg eq/kg)	
	% TRR	mg eq/kg	% TRR	mg eq/kg
CL 312622	60.0	0.015	38.2	0.003
CL 152795	11.0	0.003	19.0	0.002

Polar unknowns	10.3	0.003	31.2	0.003
Non-polar unknowns	11.9	0.003	11.3	0.001

Kidney and liver of goat administered with 33.4 mg/kg feed
CL 152795: impurity present in dosing solution

The results of this CL 312622 study showed that the administered compound (CL 312622) and the CL 152795 compound (present as an impurity in the dosing solution) were excreted with minimal retention by the kidney and the liver at the exaggerated dose of 33.4 ppm in the feed. There was no accumulation in any other edible goat tissue or in the milk. There were no residues found in the tissues or milk of the low dose treated goat.

Laying hens

Imazamox

Laying hens were orally dosed with [pyridine-6-^{14}C]-imazamox [Johnson 1994b, Johnson 1994b, (IA-440-002)]. Feeding levels for each group consisting of eight white Leghorn hens were 0, 2.11 and 10.2 ppm in the feed daily by gelatin capsule for seven consecutive days. Eggs were collected twice daily and blood and the tissues (liver, kidney, muscle, and skin with adhering fat) were collected for analysis approximately 22 hours after the last dose. Samples were analysed by combustion followed by LSC. The validated detection limit of the radioassay method was approximately 0.01 mg eq/kg for eggs, blood and all tissues.

Residues in eggs, blood, skin with adhering fat, muscle, liver, and kidney tissues were all less than 0.01 mg eq/kg. Recovery of ^{14}C in excreta collected over the 7 day treatment period averaged 85.5% of the total administered dose at 2.11 ppm feeding level and 84.8% at 10.2 ppm feeding level.

The results of this study indicate that imazamox-related residues do not accumulate in eggs or edible tissues of poultry. Orally administered imazamox was mainly eliminated from the hen through excreta. No detectable (< 0.01 mg eq/kg) [^{14}C]imazamox-derived residues in eggs or edible tissue were found.

CL 263284

A poultry metabolism study was conducted with [pyridine-6-^{14}C]-radiolabelled imazamox metabolite CL 263284 [Afzal 1994a, IA-440-002]. Hens of each group containing eight animals were dosed orally by gelatin capsules at 0, 2.14 and 10.9 ppm in the feed daily for seven days. Eggs and excreta were collected daily. After seven days of dosing, the hens were sacrificed and the tissues (liver, kidney, muscle and skin with adhering fat) and blood were collected for analysis approximately 22 hours after the last dose. Samples were analysed by combustion followed by LSC. The validated detection limit of the radioassay method was 0.01 mg eq/kg CL 263284 equivalents in the tissues, eggs and excreta.

Total recovery of radioactivity in excreta was 85.3% and 88.6% for the low and high dose, respectively. Residues in all tissues, blood and eggs were less than 0.01 mg eq/kg, the validated detection limit.

The results of this study indicate that the administered CL 263284-related residues do not accumulate in eggs or edible tissues of poultry. Orally administered imazamox was mainly eliminated from the hen through excreta. No detectable (< 0.01 mg eq/kg) ^{14}C-CL 263284-derived residues in eggs or edible tissue were found.

CL 312622

Laying hens were orally treated with a mixture of [pyridine-6-^{13}C] and [pyrindine-6-^{14}C]-radiolabelled imazamox metabolite CL 312622 [Afzal 1999a, ID-440-005]. The eight hens (Hy Line W-36, White Leghorn) by each group were dosed for five consecutive days with 0 ppm feed (control), 0.13 ppm feed (low dose) and 10.5 ppm feed (high dose). Eggs were collected twice daily and bile and the edible tissues (muscle, liver and skin with adhering fat) were collected at sacrifice approximately 22

hours after the last dose. Samples were analysed by combustion followed by LSC. The validated detection limit of the radioassay method was 0.01 mg/kg CL 312622 equivalents in the tissues, eggs and excreta. Detection of CL 312622-related residues in excreta extract was accomplished by HPLC-UV. The radioactivity was quantitated by LSC.

With the exception of high dose composite bile sample (0.018 mg eq/kg), residue in tissues (liver, muscle and skin with adhering fat), eggs and bile were all less than or equal to the validated detection limit (0.006 mg/kg) of the radioassay. Recovery of ^{14}C in excreta collected over the five day treatment period averaged 87.5% and 90.9% of the total administered dose from the low dose (0.13 ppm feed) and high dose (10.5 ppm feed), respectively. These data showed that the administered CL 312622 or derived residues as well as CL 152795 (present as an impurity in the dosing solution) were excreted without retention or accumulation in eggs and edible poultry tissues.

The extremely low residues in eggs and edible tissues precluded further characterization. The high dose day-2 excreta sample was extracted with methanol. The extractability of ^{14}C residue was 91.2%. The extracted ^{14}C residue was analysed by HPLC. The major component of the extractable residue was unchanged CL 312622, accounting for 76.7% of the extractable radioactive residue. The polar dosing solution impurity (CL 152795) accounted for approximately 6.5% of the ERR. All other components were insignificant and no single component exceeded 5% of the TRR.

Table 2 Distribution of radioactivity in high dose hen excreta of day 2 treated with [^{14}C]-CL 312622 (ID-440-005)

Compound	Excreta (9.2 mg eq/kg)	
	% TRR	mg eq/kg
CL 312622	70.0	7.0
CL 152795	5.9	0.6
Non-polar unknowns / Methyl ester	2.0	0.2

Excreta of hen administered with 10.5 ppm.

Plant metabolism

The Meeting received information on the fate of imazamox in oilseed rape, soya bean, pea, maize, wheat and alfalfa.

Oilseed rape

Study 1

A metabolism study was conducted to investigate the amount of imazamox residues and the nature of its degradation products in summer oilseed rape after foliar application [Radzom 2013a, 2011/1281377]. Oilseed rape (imidazolinone herbicide-tolerant variety: Salsa CL) was sown into ten plastic containers (total test area, external size = 2.4 m^2) filled with sandy loam soil, which were located in climatic chambers (phytotrons). The phytotrons simulated the natural climatic conditions of a typical rapeseed-growing area.

Under the indoor condition, the crop was treated once with [imidazolinone-5-^{14}C, 3-^{15}N]-imazamox at growth stage BBCH 10–18 with a rate of 0.075 kg ai/ha. Labelled ^{14}C, ^{15}N-imazamox and unlabelled ^{12}C-imazamox were mixed at a ratio of 2:1 (^{14}C, ^{15}N-imazamox: ^{12}C-imazamox). Blank formulation (40 g ai/kg blank formulation), adjuvant BAS 160 00 S (1 L/ha) and water was added. The application solution was monitored for purity by HPLC and the isotope pattern was determined by mass spectrometry. The formulation in a spray volume of 216 L/ha was applied with an automatic spray track. Forage samples were taken 12 days after treatment (BBCH 39). At harvest (90 DAT, BBCH 89), mature oilseed rape pods and straw were cut off with scissors. Hull and seed were separated using a thresher. All samples were stored in a freezer at approximately –18 °C or below. The storage conditions stayed the same until analysis

started and during the whole period of the metabolism study. Extracts were stored in a refrigerator or, for longer periods, in a freezer.

The plant material (forage, straw, hull and seed) were extracted with methanol and water. The combined extracts were measured by LSC. The residual radioactive residues after solvent extraction were combusted. On the other hand, a sequential solubilisation procedure was applied for the PES of rape straw, hull and seed. The residues were extracted with aqueous ammonia solution (1%) and subsequently by treatment with protease (only seed) and with cellulase and macerozyme. The extracts and residues were analysed by LSC.

Methanol extracts of rape forage, straw and hull were directly investigated by LC-MS. Peak assignment in the other samples was done by comparing the metabolite patterns with those of the extracts investigated by MS-analysis and by comparison of the retention times of the identified components (CL 263284, CL 312622) with the ^{14}C-signals of the quantitative and confirmatory HPLC analyses. Additionally, a co-chromatography experiment with the reference items CL 312622 and CL 263284 was performed with the water extract of rape straw. In order to investigate, if cleavage of the imazamox ring systems occurs, reference item dimethylhydantoin was studied with respect to its chromatographic properties to check for similar degradation products. According to retention time comparison, in the HPLC chromatograms of oilseed rape matrices no corresponding signals were detected and therefore no formation of cleavage products was observed.

The TRR of rape forage was 1.004 mg eq/kg, straw 1.100 mg eq/kg, hull 2.448 mg eq/kg, seed 0.149 mg eq/kg, when determined by direct combustion.

Table 3 Total radioactive residues (TRR) in oilseed rape samples following foliar application of [^{14}C, ^{15}N]-imazamox (2011/1281377)

Radioactive residues in treated oilseed rape			
Matrix	Days after treatment	TRR determined by direct combustion (mg eq/kg)	TRR calc. (mg eq/kg)
Forage	12	1.004	0.889
Straw	90	1.100	1.134
Hull	90	2.488	2.527
Seed	90	0.149	0.152

The extractability of rape forage with methanol and water was 97.7% TRR (0.868 mg eq/kg) with the major part of the residues extracted with methanol (0.823 mg eq/kg, 92.6% TRR). The extractability of rape straw and hull with methanol and water was 77.6% TRR (0.879 mg eq/kg) and 78.8% TRR (1.991 mg eq/kg), respectively. The major part of the residues was also extracted with methanol (50.5% TRR in straw and 57.7% TRR in hull). From rape seed, 58.3% TRR (0.089 mg eq/kg) was extracted by solvent extraction (methanol and water), with more radioactivity was extracted with water (34.3% TRR) than with methanol (24.0% TRR).

Table 4 Distribution of residues of [^{14}C, ^{15}N]-imazamox in oilseed rape samples (2011/1281377)

			Extracts						PES	
Matrix	Days after treatment	TRR calc .[a]	methanol extract		aqueous extract		total			
		mg eq/kg	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Forage	12	0.889	0.823	92.6	0.045	5.0	0.868	97.7	0.021	2.3
Straw	90	1.134	0.573	50.5	0.307	27.1	0.879	77.6	0.254	22.4
Hull	90	2.527	1.458	57.7	0.533	21.1	1.991	78.8	0.536	21.2
Seed	90	0.152	0.036	24.0	0.052	34.3	0.089	58.3	0.063	41.7

[a] TRR calc.: sum of extracted + PES

Table 5 Metabolites detected in oilseed rape matrices following foliar application of [^{14}C, ^{15}N]-imazamox (2011/1281377)

Components	Forage		Straw		Hull		Seed	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	0.373	41.9	n.d.		n.d.		n.d.	
CL 263284	0.476	53.5	0.496	43.7	1.776	70.3	0.047	31.0
CL 312622	0.076	8.6	0.296	26.2	0.137	5.4	0.004	2.8
Total identified from extracts	0.925	104.0	0.792	69.9	1.913	75.7	0.051	33.8
Total characterized from extracts	0.063	7.1	0.230	20.3	0.261	10.3	0.037	24.2
Total identified and/or characterized from extracts	0.988	111.2	1.022	90.1	2.174	86.0	0.088	58.0
Unextracted (PES)	0.021	2.3	0.131	11.6	0.323	12.8	0.040	26.5
Grand total	1.009	113.5	1.153	101.7	2.496	98.8	0.146	96.0

Analysis of the methanol extract of rape forage resulted in a pattern of two major and four minor peaks, of which three were identified by direct LC-MS analysis of the methanol extract. One of the two prominent peaks was identified as metabolite CL 263284 and accounted for 0.476 mg eq/kg (53.5% TRR). Parent compound imazamox was the second most abundant component and accounted for 0.373 mg/kg (41.9%). Metabolite CL 312622 was identified at 8.6% TRR. The remaining components were detected at levels below 1.1% TRR and characterised by their chromatographic properties. The same methanol extract was additionally analysed using HPLC that confirmed the metabolite pattern of the quantitative analysis. The respective water extract and the residue obtained after solvent extraction were not further investigated due to their low amounts of radioactive residues. In the extracts, 104.0% of the total radioactive residues were identified and 7.1% were characterised by HPLC analysis or their extractability with water.

Analysis of the methanol extract of rape straw resulted in a pattern of one main peak and seven minor peaks. The main peak was identified as metabolite CL 263284 and accounted for 0.398 mg eq/kg (35.1% TRR). The metabolite CL 312622 was identified at a level of 0.071 mg eq/kg (6.3% TRR). Parent compound imazamox was not detected. The remaining components were detected at levels below 3.7% TRR. HPLC analysis of the water extract of rape straw yielded a pattern of three peaks. The most abundant peak corresponded to the metabolite CL 312622 (0.212 mg eq/kg, 8.7% TRR). The second most abundant component was metabolite CL 263284, which accounted for 0.066 mg eq/kg (5.8% TRR). The PES was further solubilised with ammonia, and a mixture of macerozyme and cellulase. The first solubilisation step with aqueous ammonia solubilised a portion of 0.069 mg eq/kg (6.1% TRR) of the radioactive residues, which may have been incompletely extracted with methanol and water or weakly associated with insoluble plant material. In the subsequent solubilisation step, the NH_4OH residue was incubated with a mixture of macerozyme and cellulase which released 0.045 mg eq/kg (4.0% TRR). Analysis of the ammonia extract resulted in the identification of CL 263284 and metabolite CL 312622, which accounted for up to 1.5% of the TRR. In the supernatant obtained after treatment with macerozyme and cellulase, CL 263284 (1.3% TRR) was identified. In the extracts of rape straw, 66.0% of the total radioactive residues were identified and 14.4% TRR were characterised by HPLC. In the PES, additional 3.9% TRR were identified and 5.9% TRR were characterised by their HPLC elution behaviour.

Analysis of the methanol extract of rape seed hull resulted in a pattern of one main peak and five minor peaks. The main peak was identified as metabolite CL 263284 and accounted for 1.321 mg eq/kg (52.3% TRR). The metabolite CL 312622 was identified at a level of 0.084 mg eq/kg (3.3% TRR). The remaining components were detected at levels below 1.4% TRR and characterised by their chromatographic properties. Likewise, in the water extract again the main constituent was CL 263284 at 0.348 mg eq/kg (13.8% TRR) detected. CL 312622 was identified at 0.049 mg eq/kg (1.6% TRR). The remaining five minor peaks were detected at levels at or below 2.0% TRR and characterised by their chromatographic properties. The residue after solvent extraction was further solubilised with ammonia, and a mixture of macerozyme and cellulase. The first solubilisation step with aqueous ammonia solubilised a portion of 0.114 mg/kg (4.5% TRR) and subsequent solubilisation step with macerozyme and cellulase released 0.082 mg eq/kg (3.2% TRR). Analysis of

the ammonia extract resulted in the identification of CL 263284 and metabolite CL 312622, which accounted for up to 2.4% of the TRR. In the supernatant obtained after treatment with macerozyme and cellulase, CL 263284 (1.8% TRR) was identified. In the extracts of rape hull, 71.0% of the total radioactive residues were identified and 8.3% TRR were characterised by HPLC. In the PES, additional 4.7% TRR were identified and 2.0% TRR were characterised by their HPLC elution behaviour.

Analysis of the methanol extract of rape seed resulted in a pattern of two peaks. The peak at 24.8 min was identified as metabolite CL 263284 and accounted for 0.020 mg eq/kg (13.3% TRR). One polar component eluted at 3.4 min and was detected at 0.017 mg eq/kg (11.1% TRR). In the confirmatory HPLC chromatogram the peak eluting in the polar region was split up into three components each below or equal to 0.009 mg eq/kg (5.9% TRR). For further investigation, the polar fraction was isolated by SPE fractionation and analysed by HPLC. Both chromatograms confirmed that the polar fraction consist of at least two components. In the water extract the main constituent was identified as CL 263284 at 0.027 mg eq/kg (17.8% TRR). CL 312622 was identified at 0.004 mg eq/kg (2.8% TRR). The remaining non-identified components were detected at levels of up to 0.007 mg eq/kg (4.3% TRR). The residue after solvent extraction was further solubilised with ammonia, protease and a mixture of macerozyme and cellulase. The first solubilisation step with aqueous ammonia solubilised a portion of 0.015 mg eq/kg (9.8% TRR) and solubilisation with protease and macerozyme/cellulase released 11.9% TRR and 4.8% TRR, respectively. An aliquot of the concentrated protease solubilisate was analysed and some minor components below or equal to 0.003 mg eq/kg (1.9% TRR) eluting in the polar region were detected. In the ERR, 33.8% of the total radioactive residues were identified and 24.2% were characterised by HPLC. In the PES, additional 9.4% TRR were characterised by HPLC analysis of the protease supernatant. In addition, 14.6% TRR were characterised by solubilisation with aqueous ammonia and a mixture of macerozyme and cellulase.

In order to analyse whether one enantiomer of imazamox and CL 263284 was preferably metabolised in oilseed rape, enantiomer-specific analyses were performed in all four matrices (forage, straw, hull and seed). For the test item and the unlabelled reference item CL 263284 the ratio of enantiomer 1 to enantiomer 2 was found to be approximately 50:50. For the determination of the enantiomer ratio in the different matrices, the parent compound imazamox and metabolite CL 263284 were isolated from the methanol extracts and analysed using HPLC. The enantiomer ratio of imazamox in forage was found to be about 30:70 and the enantiomer ratio of its metabolite CL 263284 was found to be between approximately 40:50 in forage and straw and about 60:40 in hull and seed.

Study 2

A metabolism study was conducted with [pyridine-6-^{14}C]-imazamox and [pyridine-6-^{13}C]-imazamox [McDonnell 1995a, ID-640-003]. The in-life phase of the study was conducted by the EnviroQuest facilities in Minto, Manitoba, Canada in imidazolinone-resistant oilseed rape variety NS 1471. The oilseed rape was maintained under normal outdoor agronomic practices for the region. The test substance was applied as a single post-emergence broadcast spray to a 5.6 m^2 plot of established oilseed rape seedlings (3- to 4-leaf stage). The application rate was 0.020 kg ai/ha. Plants samples were collected after application on the day of treatment (0 DAT). Seed was collected at maturity (82 DAT). All samples were frozen within 3 hours of the time of sampling and were kept until assay in a freezer, at approximately –20 °C. The crop samples were processed, extracted and analysed within 14 to 42 days of harvest. Plant samples were combusted using an oxidizer and the radioactive residues were assayed by LSC.

The immature oilseed rape foliage sample (0 DAT) was extracted with water/methanol (2:8, v/v). The extract was subjected to radioassay and HPLC analysis. The harvest seeds were processed to oilseed rape oil and the resultant meal. A finely ground seed sample was extracted twice with hexane. The hexane extract was evaporated and the remaining oily residue was presumed to be rape oil, while the air dried PES (post extraction solid) was presumed to be the meal. Radioactive residues in the oil and seed were assayed by LSC.

The TRR in canola foliage (0 DAT) was 2.14 mg eq/kg. The presence of [^{14}C]imazamox (91.9% TRR) was confirmed by comparison of retention time with that of known analytical standard. There were no peaks of radioactivity comprising greater than 2% impurity in the radio-profile.

In the seed, the TRR was below the detectable amount of radioactivity, 0.002 mg eq/kg. The TRR in the canola oil was below the detectable amount of radioactivity (< 0.004 mg eq/kg). No detectable TRR was present in the oil meal.

Study 3

A small plot field study on the metabolic fate of imazamox in oilseed rape was conducted during the oilseed rape-growing season in 1998 [Roman 1999b, ID-640-009]. The field phase of this study was conducted in outdoor fenced-off research plots at EnviroQuest, Minto, Manitoba, Canada where imidazolinone-tolerant oilseed rape variety 45A71 received a single post-emergence treatment with [pyridine-6-^{14}C]-imazamox. Plot A was treated at a rate of 0.051 kg ai/ha. Plot B was treated at a rate of 0.089 kg ai/ha. The test solution was sprayed evenly over the entire plot of the oilseed rape at the 3 to 4-leaf stage. The spray volume of 280.5 L/ha and the application timing simulated the actual field use conditions.

Oilseed rape plant specimens were sampled directly after the application (0 DAT), foliage was sampled at 22 DAT and the last sampling of seeds and straw took place at 78 DAT. Green plants (0 and 22 DAT) were cut into small pieces and then ground into a fine powder with dry ice. Straw collected at later sampling intervals was first homogenized in dry ice. The seed was manually cleaned of any remaining chaff. The resulting chaff was combined with straw and processed as part of the straw sample. The seed samples were ground into a fine powder. All processed samples were stored frozen until used. The crop samples were processed, extracted, and analysed within 9 to 401 days of harvest. Rape plant and soil samples were combusted and radioactive residues were quantitated by LSC.

Finely ground oilseed rape samples were extracted three times with methanol/water (80:20, v/v) and then once with a solvent mixture of methanol/acetone/water (1:1:1, v/v/v). The radioactive residues in the combined extracts (neutral organo-extractable) were assayed by LSC. The extract was analysed for the radioactive components by HPLC. Oilseed rape seed from plot treated at a 0.089 kg ai/ha was extracted with hexane. The resulting extracts were assayed by LSC.

The remaining non-neutral organo-extractable residues in all crop samples were extracted once with 2% HCl in methanol/water (4:1). The remaining residues in straw samples were further treated with Penicillium cellulase and subsequently by 6 N HCl reflux. The extracts and hydrolysates were analysed by combustion.

The TRR in the rape foliage taken at 0 DAT was 2.208 mg eq/kg at a rate of 0.051 kg ai/ha (low rate) and 3.943 mg eq/kg at a rate of 0.089 kg ai/ha (high rate). The level of the radioactive residues, at the low and high rate, declined to 0.040 mg eq/kg and 0.130 mg eq/kg at 22 DAT but increased to 0.088 mg eq/kg and 0.178 mg eq/kg in the straw presumably due to dehydration. The concentration of the TRR in the mature seed (78 DAT) was relatively low as 0.004 mg eq/kg at a low rate and 0.006 mg eq/kg at a high rate.

Table 6 Total radioactive residues (TRRs) in oilseed rape plant, foliage, straw and seed samples after application of [^{14}C]-imazamox

TRRs in treated oilseed rape plant, foliage, straw, seed and soil			
Matrix	Days after treatment	TRR (mg eq/kg)	
		0.051 kg ai/ha	0.089 kg ai/ha
Plant	0	2.208	3.943
Foliage	22	0.040	0.130
Straw	78	0.088	0.178
Seed	78	0.004	0.006

Table 7 Distribution of residues of [^{14}C]-imazamox in rape samples (ID-640-009)

			Extracts						PES	
Matrix	DAT	TRR calc.	neutral organo-extractable		2% HCl in methanol		total			
		(mg eq /kg)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)
0.051 kg ai/ha										
Plant	0	2.208	2.142	97.00	0.026	1.17	2.168	98.17	0.040	1.83
Foliage	22	0.040	0.037	92.11	0.001	2.56	0.038	94.67	0.002	5.34
Straw	78	0.088	0.067	75.89	0.004	4.60	0.071	80.49	0.017	19.51
Seed	78	0.004	n.d.		n.d.		n.d.			
0.089 kg ai/ha										
Plant	0	3.943	3.840	97.38	0.049	1.24	3.889	98.62	0.055	1.39
Foliage	22	0.130	0.120	92.04	0.006	4.51	0.126	96.55	0.004	3.45
Straw	78	0.178	0.140	78.42	0.007	4.13	0.147	82.55	0.031	17.44
Seed	78	0.006	0.003	56.96	0.000	5.17	0.003	62.13	0.002	37.86

TRR calc.: sum of extracted + PES
Neutral organo-extractable: the extraction was done with 20% aqueous methanol three times followed by methanol/acetone/water once.
n.d.: Not detected, < 0.001 mg eq/kg

In plant, forage, straw and seed, 62.1% to 98.6% of the TRR were extracted with aqueous organic solvent mixtures. The majority (57.0–97.4% TRR) was extracted with 80% aqueous methanol three times and with methanol/acetone/water (1:1:1, v/v/v). Increase in extractability by acidic organic solvent (2% HCl in methanol) was small (1.17–5.17% TRR). The unextracted radioactivity from rape plant, foliage and straw accounted for 1.4 % to 19.5% of the TRR. In the seed, the extractability of the residues was 62.1% (0.003 mg eq/kg) of the TRR. The radioactivity remaining in seed accounted for 37.9% (0.002 mg/kg) of the TRR. In the dry straw, Penicillin cellulose and 6 N HCl reflux released 2.0–2.5% TRR and 7.4–9.4% TRR, respectively. A small extractability by enzyme treatment suggested that the presence of glycosidic linkages of the residue with endocons in the oilseed rape samples is insignificant. Due to the very low residue levels (0.002–0.004 mg eq/kg) in the enzyme hydrolysates of straw sample, no attempts were made to further characterize the nature of the residues.

In rape plant at 0 DAT, residue level of the unchanged imazamox was 1.726 mg /kg or 3.068 mg /kg (78% TRR in both). Imazamox accounted for 21% or 16% TRR in forage (22 DAT), 2% TRR in straw (78 DAT). Imazamox was extensively metabolized in the late growing and matured oilseed rape plants.

Each of the metabolites (CL 189215, CL 263284 and CL 312622) was found in rape at 0 DAT as a minor with none exceeding 2.6% of the TRR. At 22 DAT and 78 DAT, the hydroxymethyl metabolite CL 263284 accounted for 37.4% or 36.0% in forage and 49.6% or 46.4% in straw. Its glucoside CL 189215 and di-acid metabolite CL 312622 were present in very low level (below 8.6% TRR, 0.003 mg eq/kg).

Table 8 Metabolites detected in oilseed rape matrices following foliar application of 0.051 kg ai/ha [^{14}C]-imazamox

Components	Plant (0 DAT)		Foliage (22 DAT)		Straw (78 DAT)		Seed (78 DAT)	
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)
Imazamox	1.726	78.2	0.008	21.3	0.002	2.1	n.d.	
CL 189215	0.028	1.3	0.001	3.1	0.002	1.7	n.d.	
CL 263284	0.053	2.4	0.014	37.4	0.044	49.6	n.d.	
CL 312622	0.045	2.0	0.003	8.6	0.001	1.4	n.d.	
Total identified from extracts	1.852	83.9	0.026	70.4	0.049	54.8	n.d.	
Total characterized from extracts	0.290	13.1	0.009	21.7	0.019	21.2	n.d.	
Total identified and/or characterized from extracts	2.142	97.0	0.035	92.1	0.068	76.0	n.d.	

Components	Plant (0 DAT)		Foliage (22 DAT)		Straw (78 DAT)		Seed (78 DAT)	
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)
2% HCl in aqueous MeOH (unidentified compounds)	0.026	1.17	0.001	2.56	0.004	4.60	n.d.	
Enzyme-released (cellulase)	n.a.	n.a.	n.a.	n.a.	0.002	2.46	n.d.	
6 N HCl Reflux	n.a.	n.a.	n.a.	n.a.	0.008	9.37	n.d.	
Unextracted (PES)	0.040	1.83	0.002	5.34	0.006	6.43	n.d.	
Grand total	2.208	100	0.038	100	0.0088	98.9	0.004	100

n.d.: Not detected
n.a.: Not analysed

Table 9 Metabolites detected in oilseed rape matrices following foliar application of 0.089 kg ai/ha [^{14}C]-imazamox

Components	Plant (0 DAT)		Foliage (22 DAT)		Straw (78 DAT)		Seed (78 DAT)	
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)
Imazamox	3.068	77.8	0.020	15.5	0.003	1.7	0.001	12.6
CL 189215	0.045	1.2	0.002	1.7	0.002	1.1	0.001	20.5
CL 263284	0.104	2.6	0.047	36.0	0.083	46.4	0.000	4.6
CL 312622	0.061	1.6	0.004	3.2	0.001	0.8	0.000	2.1
Total identified from extracts	3.278	83.2	0.073	56.4	0.089	50.0	0.002	39.8
Total characterized from extracts	0.561	14.2	0.046	35.7	0.050	28.4	0.002	17.4
Total identified and/or characterized from extracts	3.839	97.4	0.119	92.1	0.139	78.4	0.004	57.2
2% HCl in aqueous MeOH (unidentified compounds)	0.049	1.24	0.006	4.51	0.007	4.13	0.000	5.17
Enzyme-released (cellulase)	n.a.	n.a.	n.a.	n.a.	0.004	2.01	n.a.	n.a.
6 N HCl Reflux	n.a.	n.a.	n.a.	n.a.	0.013	7.40	n.a.	n.a.
Unextracted (PES)	0.055	1.39	0.004	3.45	0.010	5.67	0.002	37.86
Grand total	3.943	100	0.130	100	0.173	97.6	0.006	100

n.a.: not analysed

Soya bean

A small-plot field study was conducted in an outdoor, fenced-off research area during the growing season of soya bean [Chiu 2000b, ID-640-010]. [pyridine-6-^{14}C]-imazamox was applied as a single post-emergence on soya bean plants var., Hartz 5566 at the three-to-four trifoliate stage. The [pyridine-6-^{14}C]-imazamox was diluted with non-radiolabelled imazamox and [pyridine-6-^{13}C]-imazamox. The radiolabelled imazamox was formulated for application as the ammonium salt in aqueous solution and was sprayed at a rate of 0.39 kg ai/ha. Crop samples were collected at 0, 29, 60, and 153 days after treatment. Plant samples were stored frozen from sampling until analysis for a maximum period of 448 days.

Soya bean matrices were ground with dry ice. The TRR were determined by direct LSC or combustion for all needed samples (plant tissues, non-extractable residues, extracts, fractions from HPLC). The validated detection limit of the radioassay was 0.002 mg eq/kg.

Finely ground soya bean samples were extracted with methanol/water (80:20, v/v) and then partitioned three times with dichloromethane. The unextractable solids were blended with methanol, and the methanol rinse was analysed separately. To determine the potential for residues to accumulate in soya bean oil, the 153 DAT soya bean seed sample was blended with hexane and fractionated. Both fractions, hexane and residues, were assayed by oxidative combustion and LSC analysis.

The PES from soya bean straw and seed (after solvent extraction) were subjected to acid hydrolysis. The PES was stirred in the presence of 2% HCl in methanol/water (1:1) solution. The mixture was filtered. The filtrate was extracted with ethyl acetate if needed (> 0.01 mg eq/kg). The PES was subjected to reflux in the presence of 6 N HCL and subsequently of 1 N NaOH. If needed, the basic filtrate was extracted with ethyl acetate.

The amount of ^{14}C radioactivity present in the extracts and in the post-extraction solids was determined either by direct counting in a liquid scintillation counter or by oxidative combustions of aliquots. Distribution of radioactivity in the extractable fractions of soya bean samples was analysed by HPLC-UV at 254 nm and TLC.

Table 10 Distribution of residues of [^{14}C]-imazamox in soya bean samples (ID-640-010)

			Extracts						PES	
Matrix	DAT	TRR calc.	dichloromethane / methanol extract [a]		aqueous extract [b]		total			
		me eq/kg	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Forage	0	54.90	46.78	85.21	6.62	12.05	53.40	97.26	1.50	2.74
Forage	29	0.56	0.03	5.20	0.47	83.34	0.50	88.54	0.06	11.46
Forage	60	0.13	< 0.01	4.67	0.11	81.25	0.11	85.92	0.02	14.08
Straw	153	0.08	< 0.01	5.22	0.03	35.77	0.04	40.99	0.04	59.01
Seed	153	0.02	< 0.01	5.91	< 0.01	29.39	< 0.01	35.30	0.02	64.70

DAT: Days after treatment

TRR calc: sum of extracted + PES, equal to TRR determined by direct combustion.

[a] Residues from three times extraction with dichloromethane, after extraction with aqueous methanol solution (methanol/water, 80:20, v/v). Residue in the methanol rinse (washing of the remaining solids after extracting with the aqueous methanol) was added.

[b] Residues in the aqueous layer from extraction of three times with dichloromethane

The largest amount of [pyridine-6-^{14}C]-imazamox-derived radioactivity appeared in the forage (54.9 mg eq/kg at 0 DAT, 0.56 mg eq/kg at 29 DAT and 0.13 mg/kg at 60 DAT), whereas smaller amounts of residues were noted in the harvest straw and seed, 0.08 mg eq/kg and 0.02 mg eq/kg, respectively.

The TRR in soya bean seed oil extracted with hexane was detectable at 0.002 mg eq/kg. No additional analysis was carried out due to the extremely low radiocarbon content.

Although the radioactive residue in the 0, 29 and 60 DAT forage samples was readily extractable, the extractability of the 153 DAT samples had decreased significantly. By the 153 DAT sampling, the extractability had decreased to 40.99% for straw and 35.30% for seed (8% extractability for seed when hexane was used, < 0.01 mg eq/kg).

The majority of radioactive residues in 0 DAT treated soya bean was extracted into dichloromethane (85.21% of the TRR). In the other samples, smaller amounts of residues were extracted into dichloromethane (excluding residues from methanol rinse): 4.09% TRR in 29 DAT forage, 3.06% TRR in 60 DAT forage, 1.56% TRR in straw and 4.28% TRR in seed.

The unextracted radioactivity in the PES from soya bean straw and seed were 59.01% (0.04 mg eq/kg) and 64.70% of TRR (0.02 mg eq/kg), respectively. By acid and base digestion, 52.96% TRR (0.03 mg eq/kg) and 56.79% TRR (0.01 mg eq/kg) were released from the straw and seed marc, respectively.

Table 11 Metabolites detected in soya bean matrices following foliar application of [^{14}C]-imazamox (ID-640-010)

Components	Forage (0 DAT)		Forage (29 DAT)		Forage (60 DAT)		Straw (153 DAT)		Seeds (153 DAT)	
	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg)	%TRR
Imazamox	41.81	76.16	0.06	10.69	0.01	5.89	< 0.01	0.44	n/a	n/a
CL 263284	0.28	0.51	0.05	10.07	0.01	7.99	0.01	12.24	n/a	n/a
CL 312622	0.18	0.33	0.04	8.35	0.01	11.07	< 0.01	3.81	n/a	n/a
CL 189215	0.05	0.09	0.20	35.84	0.04	31.87	< 0.01	3.33	n/a	n/a
Total identified from extracts	42.32	77.09	0.35	64.95	0.07	56.82	0.02	19.82	n/a	n/a
Non-polar unknowns	4.42	8.06	0.02	4.71	0.01	6.63	< 0.01	0.43	n/a	n/a
Polar unknowns	0.04	0.07	0.10	17.79	0.03	17.81	< 0.01	5.10	n/a	n/a

Components	Forage (0 DAT)		Forage (29 DAT)		Forage (60 DAT)		Straw (153 DAT)		Seeds (153 DAT)	
	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg)	%TRR
Total characterized from ERR	4.46	8.13	0.12	22.50	0.04	24.44	< 0.01	5.53	n/a	n/a
Total identified and/or characterized from extracts	46.78	85.22	0.47	87.45	0.11	81.26	0.02	25.35	n/a	n/a
Solvent extract (unidentified compounds)	6.62	12.05	0.01	1.11	< 0.01	4.67	0.05	68.6	n/a	n/a
Unextracted (PES)	1.50	2.74	0.06	11.46	0.02	14.08	0.01	6.05	< 0.01	7.91
Grand total	54.9	100	0.54	100	0.13	100	0.08	100	0.02	100

n/a: Not applicable because some fractions were not analysed due to low residue level (< 0.01 mg/kg)

The parent imazamox declined from 41.81 mg/kg at 0 DAT to 0.06 mg/kg (10.69% TRR) at 29 DAT in forage. The glucoside metabolite CL 189215 increased from 0.05 mg eq/kg (0.09% TRR) at 0 DAT to 0.20 mg eq/kg (35.84% TRR) at 29 DAT. However, CL 263284 and CL 312622, showed a decrease in concentration from 0 DAT to 29 DAT in forage, that is, 0.28 mg eq/kg (0.51%) to 0.05 mg eq/kg (10.07% TRR) for CL 263284 and 0.18 mg eq/kg (0.33% TRR) to 0.04 mg eq/kg (8.35% TRR) for CL 312622, respectively.

In forage at 60 DAT, residue levels of the three components (imazamox, CL 263284 and CL 312622) were very low at 0.01 mg eq/kg (5.89–11.07% TRR) and CL 189215 was found only at 0.04 mg eq/kg, although it accounted for 31.87% TRR. Three non-polar and three polar unknowns were detected but none exceeded 0.01 mg eq/kg or 10% of TRR individually.

In straw and seed at 153 DAT, no or little residues of parent imazamox and the metabolites were found.

Pea

Field peas (variety: Express) were treated post-emergence at the three to four node stage (thirty days after planting) with [pyridine-6-^{14}C]-imazamox formulated as the ammonium salt at 0.040 kg ai/ha [Chiu 1995c, ID-640-004]. The in-life phase of this study was conducted outdoors in a sandy loam soil. The plots were equipped with removable plastic-covered "All" farms to exclude excessive rainfall, when required.

The whole plants were taken at 0 and 20 DAT. At 61 DAT, foliage/vine, pea, pea shell and pea pod at immature state were sampled. Mature pea and hay were harvested at 84 DAT. All samples were stored in a freezer at approximately –20 °C until used. The crop samples were analysed within 12 to 188 days of harvest. TRR for all samples were analysed by combustion and LSC.

A subsample of the 0 DAT treated plant tissue was extracted twice with methanol/water (8:2, v/v). The PES was air-dried and combusted prior to radioanalysis. Similarly a subsample of the 0 DAT control plant tissue treated with the blank formulation was spiked with [^{14}C]imazamox and extracted as above.

A subsample of 20 DAT immature pea plant tissue was extracted twice with methanol/water (8:2, v/v). The PES was subjected to microwave extractions with 20% aqueous methanol solution and then 2% HCl in 20% aqueous methanol. Subsequently, reflux with 0.2 N NaOH in 20% aqueous methanol was made.

A subsample of 61 DAT immature pea sample was extracted twice with methanol/water (8:2, v/v). The combined filtrate was concentrated and stored in the refrigerator overnight. A precipitate which formed was removed. The filtrate was further concentrated, followed by precipitation with an equal volume of acetonitrile/methanol (1:1).

A subsample of harvest pea sample (84 DAT) was extracted twice with methanol/water (8:2, v/v). The combined filtrate was concentrated resulting in a thick oily residue. This residue was

washed with acetonitrile/methanol (1:1), resulting in some precipitation. After the acetonitrile/methanol-soluble phase was decanted, the precipitate was reconstituted in water. The water-soluble fraction was concentrated, adjusted to pH 3, and then introduced onto a solid phase extraction (SPE) column.

A subsample of the harvest pea hay sample (84 DAT) was extracted twice with methanol/water (8:2, v/v). The PES was subjected to microwave extractions with 2% HCl in 20% aqueous methanol, and then 0.2 N NaOH in 20% aqueous methanol.

The extracts were assayed by combustion and LSC. HPLC and TLC analysis was made for characterization and identification.

The TRR in whole plant sampled at 0 DAT was determined to be 1.1 mg eq/kg. The TRR in immature pea plants at 20 DAT had decreased to 0.035 mg eq/kg, most probably associated with growth dilution. No detectable residues above the validated limit of the radioanalysis method (0.01 mg eq/kg) were seen in any plant tissues (foliage, pea, shell, and immature pea pod) sampled at 61 DAT. At harvest (84 DAT), the dry pea and pea hay contained small but detectable TRR, 0.01 mg eq/kg and 0.05 mg eq/kg, respectively.

Table 12 TRR in various pea matrices after application of [^{14}C]imazamox

Matrix	Days after treatment	TRR determined by direct combustion (mg eq/kg)
Whole plant	0	1.12
Whole plant	20	0.035
Foliage/vine	61	< 0.01
Immature pea	61	< 0.01
Immature pea shell	61	< 0.01
Immature pea pod	61	< 0.01
Harvest pea	84	0.01
Harvest pea hay	84	0.05

The radioactive residues from 0 DAT whole pea plant samples with the aqueous methanol solution extracted up to 99% (1.1 mg eq/kg) of the TRR. In whole pea plant fortified with a predetermined amount of [^{14}C]imazamox, nearly 100% of the TRR was recovered.

From immature green peas (61 DAT) and harvested pea samples (84 DAT), approximately 61% of the TRR (< 0.01 mg eq/kg) and 39.2% of the TRR (0.004 mg eq/kg) were extracted, respectively. No further extractions of the PES were pursued due to the low radioactive residue.

From 20 DAT pea plants and 84 DAT hay samples, 65.4% of the TRR (0.023 mg eq/kg) and 58.4% (0.029 mg eq/kg) of the TRR were extracted respectively. More extractions under acidic and basic condition released nearly all of the remaining residues in the post extraction solids.

Table 13 Distribution of residues of [^{14}C]imazamox in pea samples

Matrix	DAT	TRR calc.	Extracts, organo-extractable		PES	
		(mg eq/kg)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Whole plant	20	0.035	0.023	65.4	0.012	34.6
Hay	84	0.05	0.029	58.4	0.021	41.6
Peas	84	0.01	0.004	39.2	0.006	60.8

TRR calc: sum of extracted + PES

Organo-extractable means the residues extracted with methanol/water (8:2, v/v).

In all pea samples, the parent imazamox, CL 263284, and CL 189215 were found at or below the limit of detection (0.01 mg eq/kg), except hay (0.02 mg eq/kg, 66.3% TRR).

Table 14 Metabolites detected in pea matrices following foliar application of [^{14}C]imazamox

Components	Plant (20 DAT)		Pea (61 DAT)		Pea (84 DAT)		Hay (84 DAT)	
	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Imazamox	< 0.01	10.18	< 0.01	30.05	< 0.01	7.82	0.02	66.31
CL 189215	< 0.01	10.98	< 0.01	6.28	< 0.01	8.34	< 0.01	3.02
CL 263284	0.01	43.91	< 0.01	39.91	< 0.01	5.85	< 0.01	13.24
Polar unknown	< 0.01	14.77	< 0.01	5.16	< 0.01	8.57	< 0.01	
Total identified and characterized	0.01	79.84	< 0.01	81.4	< 0.01	30.58	< 0.01	89.57

Alfalfa

The [pyridine-6-^{14}C]-imazamox was applied once post-emergence to alfalfa (variety, WL 414) [Wu 1998b, ID-640-008]. The test substance (formulated as an ammonium salt) was applied once at a rate of 0.13 kg ai/ha onto four plots: plot B (established crop, breaking dormancy, at the 3rd trifoliate stage), plot D (newly seeded crop, at the 3rd trifoliate stage), plot F (newly seeded crop, late treatment at 5 days after the 1st cut) and plot H (newly seeded crop, late treatment at early bud stage).

The alfalfa forage and hay samples were taken from all four plots at the day of the application (0 DAT), 26 DAT (plots B, D and F), 45 or 74 DAT (1st cut; plots B and D, respectively); 87, 111 or 25 DAT (2nd cut, plots B, D and F, respectively) and 87, 157 or 53 DAT (3rd cut, plots B, D and F, respectively). Mature alfalfa seeds were sampled 74 DAT from plot H.

Forage and hay samples were taken from the three plots (B, D and F). In plot H, forage and seed were sampled at 0 DAT and 74 DAT, respectively. In the all plots, forage was sampled at 0–157 DAT and hay was takes 2–3 days later. All samples were stored frozen at approximately –20 °C.

The TRR in green plants (forage), hay and seeds was determined by combustion and LSC except forage sample collected at 0 DAT. The 0 DAT forage was determined by solvent extraction followed by combustion due to very small size sample and the expected high radioactivity.

Ground alfalfa samples were extracted with methanol/acetone/water (1:1:1, v/v/v). The seed extract was additionally subjected to hexane partition. The PES were re-extracted three times with the acidic extraction solvent (methanol/water/HCl, 40:9:1, v/v/v), except several alfalfa samples due to high extractability and a very low residue level (0.003 mg eq/kg). The residual solids derived from the acidic methanol/water extraction (forage, hay or seed) were treated with cellulase and by 6 N HCl reflux.

The methanol/acetone/water extract of the alfalfa hay was treated with β-glucosidase or with 1 N NaOH for base hydrolysis.

The extracts were assayed by direct LSC or by combustion and characterized by HPLC. Structural characterization of the parent compound and the major metabolites was performed by LC-MS.

The TRR in forage at 0 DAT were 15.28 mg eq/kg (plot B), 10.28 mg eq/kg (plot F), 14.00 mg eq/kg (plot D) and 6.51 mg eq/kg (plot H). At 26 DAT, the radioactive residues declined significantly to 0.34 mg eq/kg (plot B) and 0.67 mg eq/kg (plot D), respectively. The TRR in forage of 1st, 2nd and 3rd cut from the all plots were found at relatively low levels, < 0.01 mg eq/kg to 0.21 mg eq/kg. The TRR levels in hay were three to five times higher than those of forage.

In forage and hay samples, the extracted residues from solvent extraction ranged from 74.9 to 98.8% of the TRR. The most (65.3–98.8%) was derived from methanol/acetone/water solvent extraction. The acidified aqueous methanol (2% HCl in 80% methanol) extracted an additional 1.2 to 14.2% of the TRR (0.001–0.090 mg eq/kg) in the forage and hay. As the result, the residual radioactive residues in PES were 0.001 to 0.252 mg eq/kg. In alfalfa seed, 72.7% TRR was extracted from the organic solvent extractions (15% TRR from acidified aqueous methanol extraction).

The radioactive residues in PES were released by enzyme treatment and acid hydrolyses. The results suggested the presence of glycosidic linkages of the residue with endocons in the alfalfa samples. After cellulase hydrolysis, the PES (greater than 0.05 mg eq/kg) were subjected to acid hydrolysis (6 N HCl reflux). The acid hydrolysis step released about 60% of the radioactive residues (equivalent to 8–9% of TRR or 0.033–0.056 mg eq/kg) while about 40% (equivalent to 6% of TRR or 0.022–0.039 mg eq/kg) remained bound. The results indicated that the labelled carbon has been incorporated into undefined plant constituents.

Table 15 Distribution of TRR following solvent partitioning and extraction of residues of [^{14}C]imazamox in alfalfa matrices

				Extracts						PES	
Plot	Matrix	DAT	TRR (combusted)	methanol/ acetone/ water		acidic methanol/ water		total			
			mg eq/kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg	% TRR	mg eq /kg
Plot B	Forage	0	15.28	98.8	15.10	n.e.	n.e.	98.8	15.10	1.2	0.183
	Forage	26	0.34	89.3	0.304	1.2	0.004	90.5	0.308	9.5	0.032
	Forage	45 (1st cut)	0.21	94.9	0.199	n.e.	n.e.	94.9	0.199	5.1	0.011
	Hay	48 (1st cut)	0.63	91.5	0.576	3.9	0.025	95.4	0.601	4.6	0.029
	Forage	87 (2nd cut)	0.03	91.4	0.027	n.e.	n.e.	91.4	0.027	8.6	0.003
	Hay	89 (2nd cut)	0.12	88.6	0.106	5.0	0.006	93.6	0.112	6.4	0.008
	Forage	129 (3rd cut)	0.02	96.4	0.019	n.e.	n.e.	96.4	0.019	3.6	0.001
	Hay	131 (3rd cut)	0.06	86.7	0.052	7.3	0.004	94.0	0.056	6.0	0.004
Plot D	Forage	0	14.00	98.2	13.75	n.e.	n.e.	98.2	13.75	1.8	0.252
	Forage	26	0.67	77.3	0.518	6.4	0.043	83.4	0.559	16.6	0.111
	Forage	76 (1st cut)	0.07	74.1	0.052	9.7	0.007	83.8	0.059	16.2	0.011
	Hay	78 (1st cut)	0.35	64.7	0.226	13.8	0.048	78.5	0.274	21.5	0.075
	Forage	111 (2nd cut)	0.01	74.9	0.007	n.e.	n.e.	74.9	0.007	25.1	0.003
	Hay	113 (2nd cut)	0.04	70.5	0.028	10.7	0.004	81.2	0.032	18.8	0.008
	Forage	157 (3rd cut)	< 0.01	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.	n.e.
	Hay	160 (3rd cut)	0.02	68.5	0.014	11.1	0.002	79.6	0.016	20.4	0.004
Plot F	Forage	0	10.28	98.8	10.16	n.e.	n.e.	98.8	10.16	1.2	0.123
	Forage	26 (2nd cut)	0.18	85.9	0.155	5.0	0.009	90.9	0.164	9.1	0.016
	Hay	28 (2nd cut)	0.83	80.7	0.670	10.8	0.090	91.5	0.760	8.5	0.071
	Forage	53 (3rd cut)	0.02	80.2	0.016	5.9	0.001	86.1	0.017	13.9	0.003
	Hay	55 (3rd cut)	0.08	65.3	0.052	14.2	0.011	78.8	0.063	21.2	0.017
Plot H	Forage	0	6.51	98.2	6.393	n.e.	n.e.	98.2	6.393	1.8	0.117
	Seed	74	0.02	57.7	0.012	15.0	0.003	72.7	0.015	30.0	0.006

Plot B:Breaking dormancy, at 3rd trifoliate stage
Plot D: Newly seeded crop, at 3rd trifoliate stage
Plot F: Newly seeded crop, late treatment at 5 days after 1st cut
Plot H: Newly seeded crop, late treatment at early bud stage
PES: Post extraction solid resulted from methanol/acetone/water extraction followed by acidic methanol/water extraction
n.e.: not extracted

At 0 DAT, the unchanged imazamox parent accounted for the majority of the TRR (79.7–86.6%, 5.64–12.98 mg/kg) in forage from all plots. All other components at 0 DAT, including CL 189215, CL 263284 and CL 312622 were found to be minor (each at < 2% of the total residue). Imazamox was extensively metabolized in alfalfa and accounted for only 3–6% TRR (0.019–0.023 mg/kg) in forage of plot B and D (only data available) at 26 DAT. The metabolites included

CL 189215 (14–20% TRR, 0.048–0.132 mg eq/kg), CL 263284 (14–23% TRR, 0.079–0.095 mg eq/kg) and CL 312622 (18–22% TRR, 0.075–0.119 mg eq/kg) at 26 DAT.

In the 1st and 2nd cut hay (plot B, D and F), imazamox was found at very low level of 2–6% TRR (0.001–0.036 mg/kg). Relatively, the residue levels of the three main metabolites were high: CL 189215 12–31% TRR (0.005–0.208 mg eq/kg), CL 263284 12–15% TRR (0.006–0.105 mg eq/kg) and CL 312622 4–26% TRR (0.002–0.186 mg eq/kg). In seed collected at 74 DAT, residue levels of parent and the three main metabolites were very low at < 0.001 mg eq/kg or 0.001 mg eq/kg.

Table 16 Metabolites detected in alfalfa matrices following foliar application of [14C]imazamox

Plot	Matrix	DAT	TRR (mg/kg)	Components							
				CL 189215		CL 263284		CL 312622		Imazamox	
				% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plot B	Forage	0	15.28	1.24	0.189	0.94	0.144	1.60	0.244	84.96	12.98
	Forage	26	0.34	13.98	0.048	23.15	0.079	22.11	0.075	5.52	0.019
	Forage	45 (1st cut)	0.21	25.66	0.054	18.26	0.038	26.67	0.056	2.65	0.006
	Hay	48 (1st cut)	0.63	23.85	0.150	14.48	0.091	26.05	0.164	5.52	0.034
	Forage	87 (2nd cut)	0.03	25.84	0.008	21.08	0.006	13.79	0.004	4.51	0.001
	Hay	89 (2nd cut)	0.12	30.81	0.037	11.28	0.014	21.67	0.026	1.53	0.002
	Forage	129 (3rd cut)	0.02	20.15	0.004	24.67	0.005	13.14	0.003	6.40	0.001
	Hay	131 (3rd cut)	0.06	21.90	0.013	25.06	0.015	20.64	0.012	2.63	0.002
Plot D	Forage	0	14.00	0.93	0.130	1.23	0.172	1.56	0.218	82.92	11.61
	Forage	26	0.67	19.80	0.132	14.11	0.095	17.80	0.119	3.46	0.023
	Forage	76 (1st cut)	0.07	16.96	0.012	9.49	0.007	13.91	0.010	4.36	0.003
	Hay	78 (1st cut)	0.35	19.62	0.069	14.73	0.051	20.13	0.070	3.60	0.013
	Forage	111 (2nd cut)	0.01	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Hay	113 (2ndcut)	0.04	11.83	0.005	14.90	0.006	3.96	0.002	2.73	0.001
	Forage	157 (3rd cut)	< 0.01	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Hay	160 (3rd cut)	0.02	16.80	0.003	15.89	0.003	0.71	< 0.001	2.43	< 0.001
Plot	Forage	0	10.28	1.50	0.154	1.93	0.198	1.97	0.203	79.66	8.189
F	Forage	26 (2nd cut)	0.18	25.84	0.047	14.24	0.026	17.98	0.032	4.20	0.008
	Hay	28 (2nd cut)	0.83	25.02	0.208	12.72	0.105	22.48	0.186	4.36	0.036
	Forage	53 (3rd cut)	0.02	15.69	0.003	16.53	0.003	7.03	0.001	1.57	< 0.001
	Hay	57 (3rd cut)	0.08	14.07	0.011	18.53	0.015	7.81	0.007	3.62	0.003
Plot	Forage	0	6.51	0.70	0.046	1.02	0.066	0.67	0.044	86.63	5.640
H	Seed	74	0.02	3.00	0.001	7.21	0.001	2.05	< 0.001	2.32	< 0.001

Plot B:Breaking dormancy, at 3rd trifoliate stage
Plot D: Newly seeded crop, at 3rd trifoliate stage
Plot F: Newly seeded crop, late treatment at 5 days after 1st cut
Plot H: Newly seeded crop, late treatment at early bud stage

Table 17 Balance of identified, characterized and unextractable radioactive residues in alfalfa matrices following foliar application of [^{14}C]-imazamox

Plot	Matrix	DAT	Total Identified		Total Characterized		Total Identified and/or Characterized		Unextracted (PES)		Grand Total	
			% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
Plot	Forage	0	88.74	13.56	12.95	1.981	101.7	15.54	1.2	0.183	102.9	15.72
B	Forage	26	64.76	0.221	28.74	0.097	93.5	0.318	6.5	0.022	100.0	0.340

Plot	Matrix	DAT	Total Identified		Total Characterized		Total Identified and/or Characterized		Unextracted (PES)		Grand Total	
			% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
	Forage	45 (1st cut)	73.24	0.154	21.66	0.046	94.9	0.200	5.1	0.011	100.0	0.211
	Hay	48 (1st cut)	69.90	0.439	25.50	0.160	95.4	0.599	4.6	0.029	100.0	0.628
	Forage	87 (2nd cut)	65.22	0.019	26.18	0.008	91.4	0.027	8.6	0.003	100.0	0.030
	Hay	89 (2nd cut)	65.29	0.079	28.31	0.034	93.6	0.113	6.4	0.008	100.0	0.121
	Forage	129 (3rd cut)	64.36	0.013	32.04	0.007	96.4	0.020	3.6	0.001	100.0	0.021
	Hay	131-3rd cut	70.23	0.042	23.76	0.014	94.0	0.056	6.0	0.004	100.0	0.060
Plot	Forage	0	86.64	12.13	11.55	1.617	98.2	13.75	1.8	0.252	100.0	13.40
D	Forage	26	55.17	0.369	39.02	0.262	94.2	0.631	5.8	0.039	100.0	0.670
	Forage	76 (1st cut)	44.71	0.032	43.88	0.030	88.6	0.062	11.4	0.008	100.0	0.070
	Hay	78 (1st cut)	58.08	0.203	35.53	0.125	94.3	0.328	6.4	0.022	100.7	0.350
	Forage	111 (2nd cut)	n.a.	n.a.	74.9	0.007	74.9	0.007	25.1	0.003	100.0	0.010
	Hay	113 (2ndcut)	33.42	0.014	50.19	0.020	83.6	0.034	16.4	0.007	100.0	0.041
	Forage	157 (3rd cut)	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
	Hay	160-3rd cut	35.83	0.008	44.78	0.009	80.6	0.017	19.4	0.004	100.0	0.021
Plot	Forage	0	85.66	8.744	13.75	1.414	99.4	10.16	1.2	0.123	100.6	10.28
F	Forage	26 (2nd cut)	62.26	0.113	28.64	0.052	90.9	0.165	9.1	0.016	100.0	0.181
	Hay	28 (2nd cut)	64.58	0.535	30.00	0.249	94.6	0.784	5.4	0.045	100.0	0.829
	Forage	53 (3rd cut)	40.82	0.008	45.28	0.009	86.1	0.017	13.9	0.003	100.0	0.020
	Hay	57 (3rd cut)	44.03	0.036	41.86	0.034	85.9	0.070	14.1	0.011	100.0	0.081
Plot	Forage	0	89.02	5.796	9.19	0.598	98.2	6.394	1.8	0.117	100.0	6.511
H	Seed	74	14.58	0.004	56.11	0.012	70.7	0.016	25.9	0.005	96.6	0.021

Plot B:Breaking dormancy, at 3rd trifoliate stage
Plot D: Newly seeded crop, at 3rd trifoliate stage
Plot F: Newly seeded crop, late treatment at 5 days after 1st cut
Plot H: Newly seeded crop, late treatment at early bud stage

The metabolite profile of the alfalfa hay based on HPLC analysis shows that the incurred [^{14}C]imazamox-derived residues in the alfalfa hay are stable when stored frozen for about 13 months. The quantitative distribution of imazamox and metabolites CL 189215, CL 263284, CL 312622 and the very polar components peak in the sample analysed within one month after sampling is similar to those found in the sample analysed at about 13 months after sampling.

Maize

Study 1

A metabolism study of imazamox in maize (imidazolinone-tolerant variety, Pioneer hybrid 3571 IR) was conducted in an outdoor, fenced-off research area on sandy loam soil in Lucama, North Carolina, during the growing season 1996 [Zulalian 1997c, ID-640-006]. The radiolabelled imazamox was a mixture prepared from [pyridine-6-^{14}C]-imazamox, [pyridine-6-^{13}C]-imazamox and non-isotopically labelled imazamox. The radioactive test substance formulated as an aqueous ammonium salt solution was applied as a post-emergence broadcast application 0.13 kg ai/ha to maize plants at the 4–8 leaf growth stage. Samples of maize plants were harvested at 0 DAT (immature maize plant), 14 DAT (immature plant), 30 DAT (early forage), and 62 DAT (late forage). At maturity (100 DAT), collected field samples were separated with fodder (stalks, husks, cobs) and grain. The maize samples were ground with dry ice and stored frozen. The samples were subjected to oxidative combustion and LSC. All control samples were < 0.004 mg eq/kg. The crop samples were analysed within 44 days.

Green plant, forage and fodder samples were extracted three times with methanol/water (80:20, v/v). The PES was rinsed with methanol and the rinsate was combined with the extracts. A grain sample was extracted three times with hexane. The PES was extracted three times with methanol/water (80:20, v/v).

The methanol/water extracts were partitioned three times with dichloromethane. The dichloromethane and aqueous fractions were separated. For the 14 DAT, 30 DAT and 62 DAT forage and 100 DAT fodder samples, the PES (after partitioning with dichloromethane) were extracted once with methanol/water/hydrochloric acid (130:18:2, v/v/v).

TRR in extracts and residual radioactive residues were analysed by combustion and LSC. Distribution of radioactivity in the extracts was analysed by HPLC-UV at 254 nm. Imazamox and the metabolite CL 263284 were isolated and identified by mass spectral analysis. The other components of the residue were identified by comparison of HPLC retention time.

The TRR in immature maize plant at 0 DAT was 8.2 mg eq/kg and rapidly decreased to 0.41 mg eq/kg (14 DAT). At 30 DAT and 62 DAT, the TRR in early and late forage were 0.087 mg eq/kg and 0.078 mg eq/kg, respectively. In maize fodder and grain at maturity (100 DAT), the TRR were 0.047 mg eq/kg and 0.021 mg eq/kg, respectively.

Of the TRR in the various maize substrates, between 48% and 98% (0.014–8.0 mg eq/kg) was extracted with methanol/water (80:20, v/v) and between 2% and 52% (0.006–0.17 mg eq/kg) remained unextracted. Negligible radioactivity (0.4–0.6% of the TRR, < 0.001 mg eq/kg) was extracted from the grain with hexane indicating the imazamox-derived residues did not concentrate in the maize oil.

The additional extraction with acidic extraction solvent (methanol/water/HCl, 80:18:2) released more residues in maize samples up to approximately 56% to 98% of the TRR (0.013–8.0 mg eq/kg). Approximately 2% to 44% (0.006–0.021 mg eq/kg [^{14}C]imazamox equivalents) remained in the post extraction solids.

Table 18 Distribution of TRR following solvent partitioning and extraction of residues of [^{14}C]imazamox in maize samples

Matrix	DAT	TRR calc.	Extracts								PES	
			dichloromethane extract		aqueous extract		methanol/water/ HCl extract		total			
		(mg eq /kg)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)
Immature plant	0	8.199	3.126	38.1	4.902	59.8			8.028	97.9	0.171	2.1
Immature plant	14	0.409	0.081	19.8	0.281	68.8	0.012	2.9	0.374	91.5	0.035	8.5
Early forage	30	0.087	0.012	14.3	0.061	69.9	0.006	7.0	0.079	91.2	0.008	8.8
Late forage	62	0.078	0.003	4.3	0.055	70.7	0.004	5.6	0.062	80.6	0.015	19.5[3]
Fodder	100	0.047	0.001	2.4	0.021	45.6	0.004	8.2	0.026	56.2	0.021	43.8
Grain	100	0.021	0.004	18.8	0.011	53.1			0.015	71.9 [a]	0.006	27.6
Grain	100	0.021	0.001	4.9	0.012	59.2			0.013	64.1 [b]	0.007	35.3

After extraction with methanol/water (80:20, v/v), followed by partitioning with dichloromethane
TRR calc.: sum of extracted + PES, equal to TRR determined by direct combustion.
Additional radioactivity was released from the PES at 62 DAT as follows. The PES was extracted at elevated temperature and pressure in an ASE 200 Accelerated Solvent Extractor to recover 0.016 mg/kg [^{14}C]imazamox equivalents (20.5% of the TRR) and only 0.003 mg/kg [^{14}C]imazamox equivalents (3.8% of the TRR) remained in the PES following this more vigorous procedure. This indicated there were no significant bound residues in the maize forage.
[a] Replicate 1, 0.4% (< 0.004 mg/kg) was hexane-soluble.
[b] Replicate 2, 0.6% (< 0.004 mg/kg) was hexane-soluble.

The unchanged parent imazamox amounted to 5.3 mg/kg (65% TRR) at 0 DAT plant, 0.073 mg/kg (18% TRR) at 14 DAT plant and less than 0.01 mg/kg (2–9% of the TRR) thereafter.

CL 263284 was identified as the major metabolite in maize, accounting for 0.24 mg eq/kg (3% TRR) at 0 DAT plant, but only 0.007 mg eq/kg (15% TRR) in maize fodder and only 0.004 mg eq/kg (20% TRR) in grain. The residue levels of glucose conjugate (CL 189215), the di-acid (CL 312622) and the hydroxy-acid (CL 354825) were < 0.001–0.003 mg eq/kg (2–5% TRR) in fodder and grain. Many polar and nonpolar compounds (minimum 4 polar and 3 nonpolar) were found at 0.012 mg eq/kg or less (1–14% TRR) individually since 30 DAT.

Table 19 Metabolites detected in maize matrices following post-emergence foliar application of [^{14}C]-imazamox

Components	Plant (0 DAT)		Plant (14 DAT)		Forage (30 DAT)		Forage (62 DAT)		Fodder (100 DAT)		Grain (100 DAT)	
	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Imazamox	5.305	64.7	0.073	17.8	0.008	9.2	0.003	3.8	0.001	2.2	0.001	5.0
CL 263284	0.237	2.9	0.093	22.7	0.025	28.7	0.032	41.0	0.007	15.2	0.004	20.0
CL 312622	0.562	6.9	0.029	7.1	0.007	8.0	0.001	1.3	0.001	2.2	0.001	5.0
CL 189215	0.198	2.4	0.024	5.9	0.004	4.6	< 0.001	< 1	0.003	6.5	0.003	15.0
CL 354825	0.486	5.9	0.009	2.2	0.003	3.4	0.002	2.6	< 0.001	< 1	< 0.001	< 1
Total identified	6.788	82.8	0.228	55.7	0.047	53.9	0.038	48.7	0.013	26.1	0.009	45.0
Min. of 4 polar and 3 nonpolar compounds	1.242 (0.055–0.281)	15.2 (0.7–4.4)	0.135 (0.003–0.059)	33.0 (0.7–14.4)	0.024 (0.001–0.012)	27.3 (1.1–13.8)	0.017 (< 0.001–0.009)	21.8 (< 1–11.5)	0.009 (< 0.001–0.004)	19.6 (< 1–8.7)	0.002 (< 0.001–0.001)	10.0 (< 1–5.0)
Total identified and/or characterized	8.030	97.9	0.359	88.8	0.071	81.6	0.055	70.5	0.021	45.7	0.011	55.0

Study 2

The metabolism study of imazamox in maize (Pioneer hybrid 3571 IR, Imidazolinone-tolerant variety) was conducted in an outdoor, fenced-off research area on sandy loam soil in Lucama, North Carolina, during the 1996 growing season [Zulalian 1997d, ID-640-007]. The radio impurity amounting to approximately 3% was identified as the di-acid (CL 312622). The radiolabelled imazamox was a mixture prepared from [pyridine-6-^{14}C]-imazamox, [pyridine-6-^{13}C]-imazamox and non-isotopically labelled imazamox. The radioactive test substance was formulated as an aqueous ammonium salt solution and applied as a pre-emergence broadcast treatment (one day after seeding) at a rate of 0.14 kg ai/ha. Samples of maize plants were taken at 14 DAT (immature maize plant), 30 DAT (early maize forage) and 62 DAT (late maize forage). At maturity (112 DAT), the fodder (stalks/husks/cobs) and grain were collected. The samples were ground with dry ice and stored frozen. TRR was analysed by oxidative combustion and LSC. The detection limit was < 0.004 mg eq/kg. The crop samples were analysed within 24 days.

The extraction method with methanol/water (80:20, v/v) and dichloromethane was the same with that of above study. The PES of immature maize plant (14 DAT) was extracted once with methanol/water/hydrochloric acid (80:18:2, v/v/v). A grain sample was extracted three times with hexane. The PES was extracted three times with methanol/water (80:20, v/v). The extracts and post extraction solids were analysed by combustion and LSC. Distribution of radioactivity in the extractable fractions of maize samples was analysed by HPLC-UV detector set at 254 nm. Imazamox and the metabolite CL 263284 were isolated and identified by mass spectral analysis. The other components of the residue were identified by retention times by HPLC.

The TRR in immature maize at 14 DAT was 0.16 mg eq/kg and decreased to 0.027 mg eq/kg in early forage (30 DAT) and further to 0.011 mg eq/kg in late forage (62 DAT). In harvest fodder and grain (112 DAT), the TRR were 0.012 mg eq/kg and 0.010 mg eq/kg, respectively.

In the various maize substrates, 40% to 89% (0.004–0.13 mg eq/kg) of the TRR was extracted with methanol/water, and 17% to 60% (0.005–0.028 mg eq/kg) remained in the post extraction solid

(for 14 DAT maize, including 7% TRR released from acidic solvent extraction). Negligible radioactivity (2.1% of the TRR, < 0.001 mg eq/kg) was extracted from the grain with hexane indicating the imazamox-derived residues did not concentrate in the maize oil.

Since the TRR in the extracts of the 62 DAT and 112 DAT maize samples were insufficient for identification/characterization, the extracts were not carried through the solvent partitioning procedures.

Table 20 Distribution of TRR following solvent partitioning and extraction of residues of [^{14}C]imazamox in maize samples

Matrix	DAT	TRR calc.	Extracts								PES	
			dichloromethane extract		aqueous extract		methanol/water/ HCl extract		total			
		(mg eq/kg)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)	(mg eq /kg)	(%TRR)
Green plant	14	0.158	0.044	28.0	0.075	47.2	0.011	6.9	0.13	82.1	0.028	17.9
Early forage	30	0.027	0.007	25.5	0.015	57.1			0.022	82.6	0.005	17.3
Late forage	62	0.011							0.009	88.8	0.002	21.2
Fodder	112	0.012							0.005	40.3	0.007	59.7
Grain	112	0.010							0.004	43.6	0.006	56.4

TRR calc.: sum of extracted + PES, equal to TRR determined by direct combustion.
Extracts of grain includes hexane extract, approximately 2% TRR (< 0.001 mg eq/kg).

The unchanged parent imazamox amounted to 0.049 mg/kg (31% TRR) at 14 DAT maize plant and 0.010 mg/kg (37% TRR) at 30 DAT forage. CL 263284 was identified as the major metabolite in maize, accounting for 12% TRR (0.019 mg eq/kg) at 14 DAT plant and 19% TRR (0.005 mg eq/kg) at 30 DAT forage. The identification of imazamox and CL 263284 was confirmed by LC-MS analysis. The residue levels of the metabolites (identified by HPLC retention time) glucose conjugate (CL 189215), the di-acid (CL 312622) and the hydroxy-acid (CL 354825) were between 0.003 (2% TRR) and 0.008 mg eq/kg (5% TRR) at 14 DAT and below < 0.001 mg eq/kg (less than 4% of the TRR) at 30 DAT. The remaining radioactive residue was characterized as multiple polar and nonpolar water-soluble metabolites (0.001–0.016 mg eq/kg, 0.6–10% TRR individually). The only significant components of the residue in maize were imazamox and CL 263284.

Table 21 Metabolites detected in maize immature plant and forage following pre-emergence foliar application of [^{14}C]imazamox

Components	14 DAT plant		30 DAT forage	
	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Imazamox	0.049	31.0	0.010	37.0
CL 263284	0.019	12.0	0.005	18.5
CL 312622	0.008	5.1	0.001	3.7
CL 354825	0.005	3.2	0.001	3.7
CL 189215	0.003	1.9	0.001	3.7
Total identified	0.084	53.2	0.018	66.6
Min. of 4 polar and 3 nonpolar compounds	0.034 (0.001–0.016)	21.4 (0.6–10.1)	0.008 (< 0.001–0.002)	13.41 (< 0.1–7.41)
Total identified and/or characterized	0.118	74.6	0.026	80.01

Wheat

Study 1

Spring wheat (imidazolinone-tolerant variety: AP603 CL) was sowed into ten plastic containers filled with loamy sand soil, which was conducted in climatic chambers, the phytotrons simulated the natural climatic conditions for a typical spring, [Grosshans *et al.,* 2012b, 2012/1064722]. The crop was treated once with [imidazolinone-5-14C, 3-15N]-imazamox at a rate of 0.76 kg ai/ha at growth stage BBCH 13–24. The application formulation (including the test item: unlabelled imazamox (2:1 ratio), blank formulation and adjuvant Dash EC) was applied with an automatic spray track at a spray volume of 219 L/ha. Samples of wheat forage and hay were taken 8 days (BBCH 39) after application. Harvest of wheat plants and sampling of straw and grain was accomplished 62 days after application. Eighty wheat plants were thinned out. One half of the plants was minced and frozen as forage, the other half was minced and dried at room temperature. Wheat ears and straw (GS 89) were cut off and the straw was minced. Afterwards, ears were separated into chaff and grain using a thresher. The chaff was mixed to the straw. All samples were stored in a freezer at –18 °C or below. The samples were stored in a refrigerator or in a freezer. Radioassay was performed by combustion and LSC.

The samples were extracted with methanol and water. The residual radioactive residues were extracted twice with 1% ammonia, and then treated with the enzymes in order of macerozyme/cellulase, α-amylase/β-amylase/amyloglucosidase, and tyrosinase/lactase. After the tyrosinase incubation the PES were extracted once with 1 N HCl, thereafter refluxed with 6 N HCl. The extracts and PES were assayed by LSC. The residues in the extracts were characterized or identified by HPLC. For enantiomer-specific analysis, an aliquot of wheat forage, hay or grain was extracted with methanol and water and then the methanol extract was extracted three times with ethyl acetate, subsequently the ethyl acetate phase partitioned with water (acidified with formic acid to pH 2). The water phase was fractionated using HPLC and the fractions were subjected to enantiomer-specific analysis using HPLC.

There were no major differences between measured TRR and calculated TRR (ERR plus PES). Here, the calculated TRR values were set to 100% TRR for all matrices. For the wheat forage, hay, straw and grain, the TRR were 1.6 mg eq/kg, 8.0 mg eq/kg, 3.2 mg eq/kg and 1.4 mg eq/kg, respectively.

Table 22 Distribution of residues of [^{14}C]imazamox in wheat samples (2012/1064722)

				Extracts						PES	
Matrix	DAT	TRR	TRR cal.	combined methanol extract		combined aqueous extract		total			
		(mg eq/kg)	(mg eq/kg)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)	(%TRR)
Forage	8	1.819	1.602	1.427	89.0	0.055	3.4	1.482	92.5	0.121	7.5
Hay	8	10.09	8.011	6.008	75.0	1.329	16.6	7.337	91.6	0.674	8.4
Straw	62	3.493	3.199	1.962	61.3	0.586	18.3	2.548	79.6	0.651	20.4
Grain	62	1.374	1.412	0.618	43.8	0.608	43.1	1.227	86.9	0.185	13.1

TRR cal.: sum of extracts + PES

The extractability of wheat forage and hay with methanol and water was very high and accounted for 92.5% and 91.6% of the TRR. The major part of the residues was extracted with methanol (forage: 89.0% TRR, hay: 75.0% TRR). The extractability of wheat straw and grain with methanol and water was high and accounted for 79.6% and 86.9% of the TRR. For straw, the major part of the residues was extracted with methanol (61.3% TRR), whereas for grain virtually equal amounts were extracted with methanol and water (methanol 43.8% TRR; water 43.1% TRR).

In wheat forage, analysis of the methanol extract using HPLC resulted in a pattern of six peaks, of which four were identified. The main peak was identified as the parent compound imazamox and accounted for 1.046 mg/kg (65.3% TRR). Metabolite CL 263284 was the second most abundant component and accounted for 0.286 mg eq/kg (17.9% TRR). The metabolites CL 312622 and

CL 189215 were identified at levels of up to 6.7% TRR. In the water extract, the parent compound imazamox was the main constituent at 0.033 mg/kg (2.1% TRR). The remaining components CL 263284, CL 312622 and CL 189215 were identified at levels of up to 0.6% TRR. No signals corresponding to the reference items Reg. No. 60967 (5,5-dimethylimidazolidine-2,4-dione [or 5,5 dimethylhydantoin]; CAS Nr 77-71-4) and Reg. No. 199553 (5-isopropyl-5-methyl-2,4-imidazolidinedione [or iPr-Me-Hydantoin]; CAS-N 5455-35-6)—putative cleavage products of imazamox, imidazoline moiety- were detected in the solvent extracts using HPLC. The residue after solvent extraction was further solubilized. Analysis of the ammonia solubilizate identified imazamox and metabolite CL 263284, which accounted for up to 0.4% of the TRR. In the ERR, 94.5% of the total radioactive residues were identified and 4.1% were characterized. In the PES, additional 3.9% of the TRR were identified and characterized.

In wheat hay, analysis of the methanol extract resulted in a pattern of five peaks, of which four were identified. The parent compound imazamox accounted for 4.056 mg/kg (50.6% TRR). The second most abundant component was metabolite CL 263284, which accounted for 1.689 mg eq/kg (21.1%). The metabolites CL 312622 and CL 189215 were identified at levels of up to 4.2% TRR. In the water extract the same components as in the methanol extract were identified. The parent compound imazamox accounted for 0.910 mg/kg (11.4% TRR). The remaining components CL 263284, CL 312622 and CL 189215 were identified at levels of up to 2.8% TRR. No signals corresponding to the reference items Reg. No. 60967 and Reg. No. 199553 (putative cleavage products of imazamox, imidazoline moiety) were detected in the solvent extracts using HPLC. The ammonia solubilizate contained imazamox and metabolite CL 263284, which accounted for up to 0.4% of the TRR. In the ERR, 96.0% of the total radioactive residues were identified and 1.0% were characterized. In the PES, additional 6.5% of the TRR were identified and characterized.

In wheat straw, the methanol extract showed a pattern of eleven peaks, of which four were identified. The metabolite CL 263284 accounted for 1.041 mg/kg (32.6% TRR). CL 189215 and CL 312622 were 0.394 mg/kg (12.3% TRR) and 0.396 mg/kg (12.4% TRR), respectively. The parent compound imazamox accounted for 6.1% TRR. In the water extract, the same components as in the methanol extract were identified. Likewise, the main constituent was CL 263284 at 0.172 mg eq/kg (5.4% TRR). The remaining components imazamox, CL 189215 and CL 312622 were identified at levels of up to 4.6% TRR. In the solvent extracts up to four non-identified peaks (up to approximately 1% TRR) eluted prior to metabolite CL 189215. It was not possible to assign the peaks to putative cleavage products of imazamox (imidazoline moiety) by HPLC-MS experiments. The ammonia solubilizate contained imazamox, metabolite CL 263284 and CL 312622 at up to 1.2% of the TRR. In the ERR, 78.0% of the TRR were identified and 10.5% were characterized. In the PES, additional 13.7% of the TRR were identified and characterized.

In wheat grain, analysis of the methanol extract of wheat grain resulted in a pattern of eleven peaks, of which three were identified. The parent compound imazamox accounted for 0.577 mg/kg (40.8% TRR) and the metabolites CL 263284 and CL 189215 were identified at levels of up to 3.5% TRR. In the water extract the same components as in the methanol extract were identified. The parent compound imazamox accounted for 0.473 mg/kg (33.5% TRR). The remaining components CL 263284 and CL 189215 were identified at levels of up to 3.0% TRR. In the solvent extracts up to four non-identified peaks (up to approximately 1% TRR) eluted prior to metabolite CL 189215 using HPLC. It was not possible to assign similar peaks to putative cleavage products of imazamox (imidazoline moiety) by HPLC-MS experiments. In the ammonia solubilizate the parent compound imazamox accounted for 0.026 mg/kg (1.8% TRR). In the ERR, 83.4% of the TRR were identified and 5.5% were characterized. In the PES, additional 10.1% of the TRR were identified and characterized.

The ratio of enantiomer 1 to enantiomer 2 for the test item was approximately 1:1. For the parent compound and metabolite CL 263284 isolated from methanol extracts of four matrices (forage, hay, straw and grain), the enantiomer ratio of each compound was found to be approximately 1:1 in all matrices, thus remained unchanged during metabolism of imazamox in wheat.

Table 23 Metabolites detected in wheat matrices following foliar application of [^{14}C]imazamox (2012/1064722)

Components [a]	Forage (8 DAT)		Hay (8 DAT)		Straw (62 DAT)		Grain (62 DAT)	
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	1.079	67.3	4.966	62.0	0.251	7.8	1.050	74.4
CL 189215	0.027	1.7	0.411	5.1	0.486	15.2	0.036	2.5
CL 263284	0.295	18.4	1.914	23.9	1.213	37.9	0.092	6.5
CL 312622	0.113	7.0	0.399	5.0	0.544	17.0	n.d.	
Total identified from extracts	1.514	94.5	7.690	96.0	2.494	78.0	1.177	83.4
Total characterized from extracts	0.066	4.1	0.077	1.0	0.337	10.5	0.077	5.5
Total identified and/or characterized from extracts	1.580	98.6	7.767	97.0	2.831	88.5	1.255	88.9
Unextracted (PES)	0.062	3.9	0.523	6.5	0.439	13.7	0.143	10.1
Grand total	1.688	105	8.392	105	3.336	104	1.416	100

[a] The extracts (methanol + aqueous) for these samples were combined and analysed by HPLC
n.d.: Not detected

The radioactive residues in wheat samples were initially extracted with methanol and water and analysed by HPLC at102–126 days (forage), 102–129 days (hay), 45–75 days (straw) and 47–160 days (grain), after sampling. After 324 days (forage), 323 days (hay), 326 days (straw) and 327 days (grain), re-analysis with the methanol and water extract confirmed the initial metabolic patterns. Otherwise, re-extraction from straw and grain was performed 362 days and 336 days, respectively after sampling. The metabolic patterns of the initial analyses remained. During the storage of sample extracts or straw and grain samples over approximately one year, no changes in the metabolic patterns were found.

Study 2

In another wheat study, [pyridine-6-^{14}C]-imazamox was applied post-emergence to imidazolinone-tolerant wheat (variety: Fidel selection number 2) at a rate of 0.14 kg ai/ha [Johnson 1996b, ID-640-005]. Wheat seeds were planted in a field test site under outdoor condition. Emergence was noted seven days after sowing. The wheat forage, forage of hay stage, straw (including chaff), and grain were harvested 28, 45, 70, and 70 days after treatment, respectively. Additionally, plant samples were taken the same day of treatment (0 DAT). The samples were stored in a freezer (–20 °C). Extracts were stored in a refrigerator or in a freezer. Total radioactive residues were analysed by combustion and LSC

For forage at 0 DAT, 28 and 45 DAT, the radioactive residues were extracted three times with methanol/water (8:2, v/v). The aqueous phase was partitioned three times with methylene chloride.

For wheat grain and straw, residues in the samples were extracted three times with methanol/water (8:2, v/v) and further extracted three times with a mixture of methanol/water/concentrated HCl (8:2:0.2, v/v/v). The PES were extracted with 0.5% t-octylphenoxy-polyethoxyethanol (Triton X-100) and then by 6 N HCl reflux.

Extracts were concentrated, assayed by LSC and characterized/identified by HPLC. Further, imazamox, CL 263284 and CL 312622 were identified by gas chromatography-negative ion chemical ionization mass spectrometric (GC-NICIMS) analysis.

The TRR found in 0 DAT (plant), 28 DAT (forage), 45 DAT (hay stage), straw (70 DAT) and grain (70 DAT) were 5.57 mg eq/kg, 0.102 mg eq/kg, 0.087 mg eq/kg, 0.157 mg eq/kg and 0.067 mg eq/kg, respectively. Most likely plant growth dilution resulted in the rapid decline in residue from the time of application.

The radioactive residues in the 0 DAT, 28 DAT and 45 DAT forage as well as the harvest wheat grain were readily extractable, with 97.4%, 82.3%, 75.6 and 75.2% of the TRR extracted into

methanol/water, respectively. However, only approximately 46% of TRR was extractable from the harvest wheat straw sample. For residual radioactive residues in PES from 28 DAT/45 DAT forage and wheat grain, no further characterization was made (< 0.05 mg eq/kg, 17.7–24.8% TRR).

In straw sample, 91.5% TRR was totally extracted: 46% with aqueous methanol, 6.5% with acidic aqueous methanol, 1% with Triton X-100 and 38% by 6 N HCl reflux. The PES contained only 5.0% (0.008 mg eq/kg) of the TRR.

Table 24 Distribution of residues of [^{14}C]imazamox in wheat samples (ID-640-005)

			Extracts						PES	
Matrix	DAT	TRR [c]	organic extract		aqueous extract		total			
		mg eq/kg	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Plant	0	5.570	5.150	92.5	0.270	4.90	5.42	97.4	0.14	2.6
Forage	28	0.102	0.026	25.6	0.058	56.7	1.084	82.3	0.018	17.7
Forage	45	0.087	0.017	19.6	0.049	56.0	0.066	75.6	0.021	24.4
Grain	70	0.067	0.050 [d]	75.2**	n.d.		0.050	75.2	0.017	24.8
Straw	70	0.157	0.083 [a]	52.5 [a]	0.062 [b]	39.0 [b]	0.145	91.5	0.008	5.0

[a] Sum of extracted residues with aqueous methanol (46% TRR) and acidic aqueous methanol (6.5% TRR)

[b] Sum of extracted residues with X-100 (1% TRR) and 6 N HCl reflux (38% TRR)

[c] TRR was determined after combustion.

[d] Aqueous methanol

In forage at 0 DAT, the unaltered parent imazamox accounted for 91.8% (5.11 mg/kg) of the TRR. No known metabolites (CL 263284, CL 312622, CL 189215) were detected.

In 28 DAT forage, the concentration of the parent imazamox was 0.042 mg/kg (41.6% TRR) and present in both organic and aqueous phases. There was only one major radioactive peak (imazamox) in the organic phase. In the aqueous phase, there were four peaks (imazamox, CL 263284, CL 312622 and CL 189215). CL 263284 was present at 0.010 mg eq/kg (9.8% TRR). The other two metabolites identified (CL 312622 and CL 189215) were both less than 0.01 mg eq/kg (each < 10% TRR). Several minor components were detected at near background radioactivity.

In 45 DAT forage, the parent imazamox was present at 0.035 mg/kg (39.9% TRR). There was only one major radioactive peak (imazamox) in the organic phase. In the aqueous phase, there were four peaks (imazamox, CL 263284, CL 312622 and CL 189215). The three metabolites were all less than 0.01 mg eq/kg (each < 10% of TRR). Several minor components were detected at near background radioactivity.

In 70 DAT grain, imazamox was present at 0.027 mg/kg (40.3% TRR). Two other components (CL 263284 and CL 189215) were also present at less than 0.01 mg eq/kg (< 10% TRR) each; the di-acid metabolite (CL 312622) was not observed. Several minor components were detected at near background radioactivity.

In 70 DAT straw, the metabolite CL 263284 was present in the largest quantity, at 0.020 mg eq/kg (13.0% TRR), followed by imazamox at 0.015 mg/kg (9.5% TRR), and CL 312622 and CL 189215 at less than 0.01 mg eq/kg (< 10% TRR) each. There were many unknown minor components present at near background levels of radioactivity (< 0.01 mg eq/kg, < 10% TRR each). From extractions with the acidic aqueous methanol, X-100 surfactant and 6 N HCL reflux, the radioactive residues were found at 0.01 mg eq/kg (6.3% TRR), < 0.01 mg eq/kg (1.0% TRR) and 0.06 mg eq/kg (38.0% TRR), respectively. HPLC analysis of the each extract (except surfactant extract due to low radioactivity) showed several minor components at near background radioactivity.

Table 25 Metabolites detected in wheat matrices following foliar application of [^{14}C]imazamox (ID-640-005)

Components	Forage						Grain (70 DAT)		Straw (70 DAT)	
	0 DAT		28 DAT		45 DAT					
	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	5.11	91.8	0.042	41.6	0.035	39.9	0.027	40.3	0.015	9.5
CL 263284	n.d.		0.010	9.8	0.008	9.7	0.007	10.3	0.020	13.0
CL 312622	n.d.		0.006	6.2	0.004	5.1	n.d.		0.009	5.7
CL 189215	n.d.		0.004	3.5	0.002	2.3	0.002	3.70	0.004	2.8
Total identified from extracts	5.11	91.8	0.062	61.1	0.049	57.0	0.036	54.3	0.048	31.0
Total characterized from extracts	0.31	5.6	0.021	21.4	0.015	18.6	0.014	20.9	0.096	60.5 [a]
Total identified and/or characterized from extracts	5.41	97.4	0.083	82.5	0.064	75.6	0.05	75.2	0.144	91.5
Unextracted (PES)	0.14	2.6	0.018	17.7	0.021	24.4	0.017	24.8	0.008	5.0
Grand total	5.55	100	0.101	100.2	0.085	100	0.067	100	0.152	96.5

[a] Sum of the radioactivity not accounted for by the identified compounds in the initial extract and the radioactivity in the dilute acid, X-100 and 6 N acid extracts

PES after extraction with solvents like methanol and water

n.d: not detected

After 4 to 39 days after sampling, the all samples were removed from the combustion and HPLC analyses, except the 0 DAT plant sample. The 0 DAT sample was analysed by HPLC 423 days after sampling. The parent compound imazamox was only significant component in the 0 DAT sample. This indicated that the residue component in the sample was stable for more than 14 months.

Proposed metabolic pathway of imazamox in plants

The metabolism of imazamox was studied in oilseed rape, soya bean, pea, alfalfa, maize and wheat. Application was made on imidazolinone-tolerant varieties, except pea and soya bean. In all the studied crops, [pyridine-6-^{14}C]-imazamox was treated with post-emergence application (for maize, additionally pre-emergence treatment). In oilseed rape and wheat studies, imidazolinone-labelled imazamox, [imidazolinone-5-^{14}C, 3-^{15}N]-imazamox, was also used.

The key step of the metabolism of imazamox was the cleavage of the methyl ether group (demethylation) resulting in metabolite CL 263284. Subsequently, oxidation of the hydroxyl group of CL 263284 generated the dicarboxylic acid metabolite CL 312622, while glycosylation led to the glucose conjugate CL 189215. Both metabolites were formed mostly in very minor amounts. In oilseed rape, soya bean, alfalfa and maize, several polar metabolites were also present. A number of nonpolar metabolites and possibly the hydroxy acid (CL 354825) were found in maize in addition.

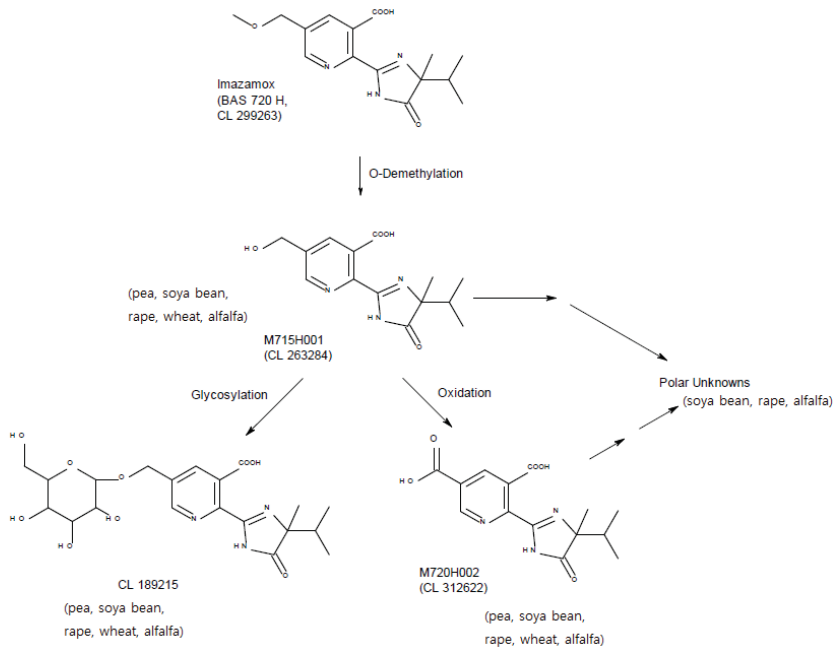


Figure 1 Proposed metabolic pathway of imazamox in pea, soya bean, oilseed rape, wheat and alfalfa

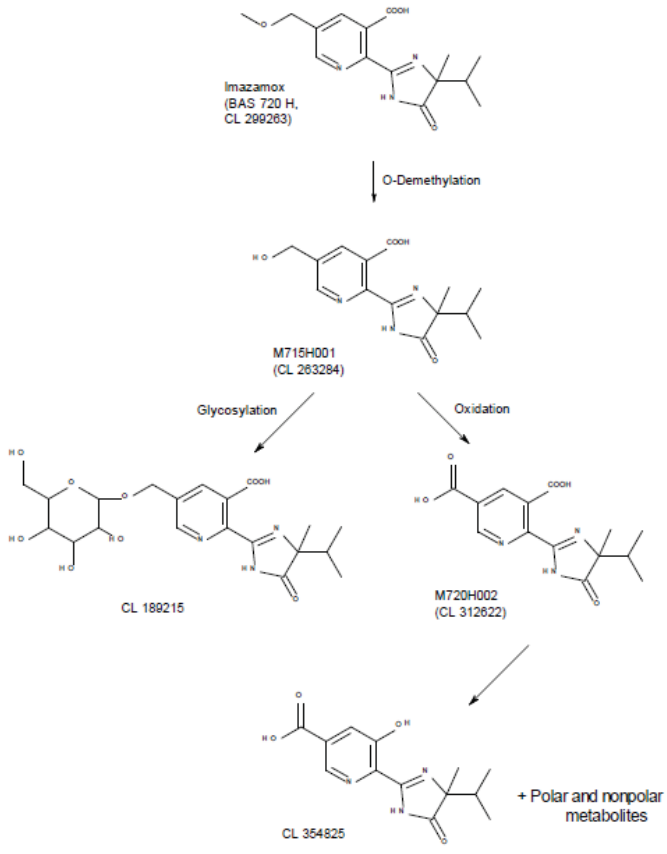


Figure 2 Proposed metabolic pathway of imazamox in maize plant

Environmental in soil

The Meeting received information on aerobic soil degradation, photolysis on soil, hydrolysis, and confined rotational crop studies. The fate and behaviour of imazamox in the environment was investigated using [pyridine-6-^{14}C]-imazamox, [Imidazolinone-5-^{14}C]-imazamox, (see Figure 1).

Aerobic degradation in soil—laboratory studies

Study 1

The study was conducted using [pyridine-6-^{14}C]-imazamox on a New Jersey (sandy loam) soil [Ta 1997a, ID-620-018]. The test compound was applied at a rate of 0.10 kg ai/ha. The soil was maintained aerobically in the dark at 25 ± 1 °C for up to 12 months at 75% of 1/3 bar moisture. The soil characteristics were as follows: % sand, 65; % silt, 22; % clay, 13; CEC (meq/100 g), 9.1; % moisture at 1/3 bar, 14.1; % organic carbon, 1.5; pH in soil:$CaCl_2$ (1:1), 6.8.

The ethylene glycol and NaOH traps were radioassayed and the foam plugs were extracted with ethyl acetate before being radioassayed. The extracts, after clean up by SPE using C-18 OH and SCX cartridges, were analysed by HPLC and TLC.

Total recoveries of radioactivity ranged from 95.0% to 102% of the total applied radioactivity throughout 1 year of incubation. Imazamox degraded rapidly in soil with an initial half-life of 31 days. The parent compound was initially oxidized to CL 312622 (the di-acid metabolite). The CL 312622 was then converted to a second metabolite CL 354825 (the hydroxy acid metabolite). There were no other metabolites > 5% TAR observed. Imazamox and both metabolites were further mineralized to $^{14}CO_2$ (48.1% TAR over the year) or bound into soil (7.1% TAR at the end of 1 year incubation).

Table 26 Distribution of parent and metabolites (as % TAR) in soil treated with [^{14}C]imazamox (ID-620-018)

Time	Parent	CL 312622	CL 354825	CO_2	Bound residue	Others	Total
0 Time	96.3	2.3	ND	ND	1.0	0.0	99.6
1 Day	92.6	4.3	ND	0.8	1.8	0.0	99.5
3 Days	85.4	11.9	ND	1.4	2.3	0.0	101.0
7 Days	71.0	24.0	1.6	2.0	3.0	0.0	101.6
14 Days	59.9	24.3	7.7	3.1	3.4	0.0	98.4
21 Days	59.2	22.0	7.9	4.5	4.3	0.0	97.9
28 Days	50.1	26.2	9.5	6.0	5.3	0.0	97.1
60 Days	33.9	16.8	24.3	12.8	6.2	1.0	95.0
90 Days	29.5	6.6	27.1	21.3	7.3	3.87	95.5
120 Days	20.7	4.2	29.6	27.6	6.7	6.7	95.5
180 Days	17.3	1.3	32.5	33.2	8.9	4.2	97.4
270 Days	15.3	1.1	25.6	42.6	7.9	4.4	96.9
365 Days	15.2	0.2	22.2	48.1	7.1	5.1	97.9

Study 2

The study was conducted using [pyridine-6-^{14}C]-imazamox on a New Jersey (sandy loam) soil at 25 ± 1 °C for up to 12 months at 75% of 1/3 bar moisture [Ta 1995a, ID-620-008]. The test material was applied at a rate of 0.10 kg ai/ha. The soil characteristics were as follows: % sand, 53.2; % silt, 34.4; % clay, 12.4; CEC (MEQ/100 g), 5.8; % moisture at 1/3 bar, 14.1; % organic carbon, 1.7; pH in soil:$CaCl_2$ (1:1), 6.6. EDTA (ethylene diamine tetracetic acid-disodium salt) extraction and subsequently followed 0.5 N NaOH extraction recovered the major part of radioactivity in soil.

Total recoveries of radioactivity ranged from 90.5% to 104% of the time0 dose, through 86 days of incubation, and then slowly decreased to 81% of the TAR after 365 days.

Approximately 60% of imazamox was degraded during the first month, 69% by two months, and 77% by 365 days of aerobic incubation. The degradation pattern was biphasic, with an initial half-life of 28 days over the first two months. The DT_{90} was greater than 365 days.

Imazamox was initially oxidized to CL 312,622 (identified by TLC, HPLC, MS and NMR). The di-acid reached a maximum concentration of approximately 44% of the applied dose at 4–6 weeks after dosing. It then decreased over the remainder of the study and accounted for less than 5% of the applied dose from 6 to 12 months. A second metabolite, CL 354,825 (identified by TLC, HPLC, MS and NMR) increased in concentration over time, and accounted for approximately 55% of the applied dose from 6 to 12 months. Ultimately imazamox and its degradates were mineralized to CO_2.

Table 27 Distribution of parent and metabolites (as % TAR) in soil treated with [^{14}C]imazamox (ID-620-008)

Time, days	% Parent	% AC 312622	% AC 354825
0	89.9	2.9	1.0
2	93.2	11.9	2.8
4	75.4	18.2	3.0
7	55.7	31.2	6.4
14	45.1	40.8	13.0
21	38.5	42.5	12.0
90	30.5	43.6	16.2
44	24.7	40.6	22.1
58	21.4	33.7	26.0
86	16.3	27.3	35.8
114	16.1	13.8	44.8
177	13.5	3.3	53.5
267	11.8	3.4	55.2
365	12.5	2.9	54.0

Study 3

The rate of degradation of [pyridine-6-^{14}C]-imazamox was studied on three EU soils (two silt loams [Ta and Lewis 1997a, ID-620-027]. The test material was applied to soil at a rate of 0.075 kg ai/ha. The soil moistures were maintained at approximately 45% of the maximum water holding capacity (MWHC). Soil samples were extracted three times with 0.5 M NaOH solution. The extracts were subjected to acid precipitation. After centrifugation, the humic acid fraction (precipitate) was re-dissolved in 0.5 N NaOH. The supernatant (fulvic acid fraction) was passed through a C-18 cartridge and the radioactivity was eluted with methanol:water (90:10 v/v). The extracts of one of the silt loam soils (Soil A) were further cleaned-up using a silica column. All samples were concentrated prior to chromatography by HPLC and TLC. Unextracted soil residues (humin fraction) were dried and aliquots combusted.

Table 28 Characteristics of soils used (ID-620-027)

Location	Limours (FR) Soil A	Boissy (FR) Soil B	Ponfaverger (FR) Soil C
% Sand	4.7	7.9	14.3
% Silt	79.4	74.7	60.7
% Clay	15.8	17.4	25.1
USDA Textural Class	Silt loam	Silt loam	Silty clay loam
CEC (meq/100 g)	13	12.6	18.3
% Moisture at 1/3 bar	20.4	19.4	22.2
% Organic Carbon	0.8	0.8	1.1
pH in 1:1 soil:water ratio	5.8	6.5	8.1

CEC: cation exchange capacity

Mass balance at 122 days was ≥ 96% (≥ 95% at all earlier time points).

In the silty clay loam (soil C), at 20 °C, the amount of AC 299,263 decreased from 97% to 4% of applied radioactivity during the 122 day incubation period. CL 312,622, was a major metabolite at especially 14 days and 27 days (42%) but then decreased to 2% at 122 days. CL 354,825 increased to 40% at 66 days then remained at about this level for the remainder of the incubation period.

In one of the silt loams (soil B) at 20 °C, the amount of imazamox decreased from 95% of applied radioactivity to 61% at 27 days. The main degradate was CL 354,825, which increased to 33% at 66 days. CL 312,622 was a minor metabolite reaching its maximum concentration of 8% at 14 days.

In the other silt loam (soil A) at 20 °C, degradation of imazamox was much slower than either of the other soils at the same temperature. Imazamox decreased from 91% TAR at 0 day to 73% TAR at 122 days, CL 354,825 increased to 12% TAR at 122 days but amounts of CL 312,622 were < 1% throughout the incubation period.

In the silty clay loam (soil C) at 10 °C, the amount of imazamox declined from 97% TAR to 12% TAR at 122 days. CL 312,622 was the principal metabolite and increased to 45% TAR at 66 days, then remained at this level to the end of the study (42% at 122 days). Levels of CL 354,825 increased during the study and accounted for 24% at 122 days.

In the silt loam (soil B, Boissy, FR) at 10 °C, the amount of imazamox declined from 96% TAR to 42% TAR at 122 days. CL 312,622 and CL 354,825 were both formed, and accounted for 22% and 19%, respectively after 122 days.

Several minor unidentified degradation products were detected but in total accounted for < 6% of applied radioactivity at any time point. Small amounts of radioactivity were also detected in the non-absorbed fraction from the C-18 cartridges (≤ 5%) and the humic acid fractions (≤ 4%), but these were not characterized.

The calculated first-order DT_{50} and DT_{90} values are summarized as follows:

Soil	Temperature (°C)	DT_{50} (Days)	DT_{90} (Days)
C (Silty clay loam)	20	12	39
B (Silt loam)	20	44	147
A (Silt loam)	20	207	687
C (Silty clay loam)	10	42	140
B (Silt loam)	10	113	375

Imazamox was degraded in this laboratory study at rates dependent upon the soil type and temperature. In most incubation groups two major metabolites (CL 312622 and CL 354825) were present. Radiolabelled residues were also incorporated into soil bound residues (especially the humin fraction, values up to 17%). Up to 24% (1–24%) of the radioactivity was evolved as volatiles, mainly carbon dioxide, demonstrating eventual mineralization of imazamox.

Study 4

The aerobic metabolism of imazamox in soils was conducted using [Imidazolinone-5-^{14}C]-imazamox [Ta 2012a, 2011/7002438]. Two soils of different textures: a sandy loam soil (Limbergerhof, Rheinland-Pfalz, Germany) and a loam soil (Hunterdon County in New Jersey, USA) were used. The test material was applied at the rate of 0.050 kg ai/ha (0.066 mg ai/kg) and the soil moistures were maintained to approximately 50% MWHC. The soil samples were extracted once with methanol, followed by 50% methanol in water, twice. Soils were further extracted with 0.5 N NaOH solution and then the extract was subjected to acid precipitation.

Table 29 Characteristics of soils used (2011/7002438)

Location	Bruch West (DE)	New Jersey (US)
% Sand	65.7	28
% Silt	23.1	49

Location	Bruch West (DE)	New Jersey (US)
% Clay	11.2	23
USDA Textural Class	Sandy loam	Loam
CEC (meq/100 g)	12.6	7.6
Maximum water holding capacity %	31.5	37.4
% Organic Carbon	1.38	1.16
pH in 1:1 soil:water ratio	8.1	7.1

The average total recovery of the radiolabelled material was 95.5% of the TAR for the entire samples from two soils.

From both soils, organic solvent extractable [^{14}C]-residues decreased from approximately 94.2% to 96.8% TAR at 0 days after treatment to approximately 23.4% to 40.5% TAR at 120 DAT. NaOH extracts increased from 3.2% to 3.4% at 0 DAT to 35.8% to 49.5% TAR at 120 DAT and non-extractable [^{14}C]-residues increased from approximately 0.8% to 1.6% TAR at 0 DAT to 17.7% to 22.1% TAR at 120 DAT. At 120 DAT, the total evolved $^{14}CO_2$ was 2.5 to 6.8% TAR.

The concentration of [imidazolinone-5-^{14}C]-imazamox decreased to approximately 28.4% to 38.8% TAR at 120 DAT. There were two major (> 5% TAR) transformation products, CL 312622 and CL 354825 at maximum values of 14.8% to 30.7% TAR, and 18.1% to 28% TAR, respectively. Several minor transformation products were also observed and none of them reached concentration of > 5% TAR. Additional investigations on the potential shift of the isomer ratio of imazamox and its metabolites during soil incubation were performed. The results clearly show that the isomer ratios were almost constant over time.

The DT_{50} values of imazamox were calculated to be 38.1 days and 23.1 days in sandy loam soil (Bruch West, Germany) and loam soil (New Jersey, USA), respectively. Mineralization was observed with levels of CO_2 reaching values of 2.5% to 6.8% TAR after 120 days of incubation.

Proposed degradation pathway of imazamox in soil under aerobic condition

Imazamox was rapidly degraded in soil. Half-life of imazamox was 28–38 days or 12–207 days (depending on soil types and temperatures). In soil, two major metabolites, CL 312622 (di-acid) and CL 354825 (hydroxyl acid) were present. Imazamox was initially oxidized to CL 312,622 (the di-acid metabolite). The CL 312622 was then converted to a second metabolite CL 354,825 (the hydroxy acid metabolite). There were no other metabolites > 5% TAR observed. Parent and both metabolites were further mineralized to $^{14}CO_2$ or bound into soil.

Photodegradation on soil surface

Study 1

The photolysis of imazamox was conducted using [pyridine-6-^{14}C]-imazamox [Ta 1995b, ID-620-004]. The test compound was applied to New Jersey (sandy loam) soil (53.2% sand; 34.4 silt: 12.4% clay: 1.7% organic carbon; pH 6.6; CEC 5.8 meq/100 g; 14.1% soil moisture at 1/3 bar) at a rate equivalent to 0.10 kg/ha (40 ppm on a soil weight basis) in photolysis chamber. Samples were irradiated continuously for up to 30 days by light from a xenon-arc lamp (filtered to remove wavelengths less than 290 nm) and maintained at 25 ^{o}C and 75% of 1/3 bar moisture during the course of the study. Soil sample was extracted two times with 0.5 M NaOH, then the extract was combusted and analysed by LSC. TLC (normal and reverse phase) and HPLC were used for identification.

Total radioactivity recovered from the irradiated samples ranged between 97.7% TAR and 101.3% TAR (105.5–109.0% TAR in control samples) throughout the experiment. These recoveries demonstrated that volatile degradates were not formed.

The 0.5 M NaOH extracts contained 96% or greater of the radioactivity initially applied to the soil. The residual radioactivity remaining in the soil after extraction ranged from 0.5% TAR to 1.3% TAR in the irradiated samples (0.8–1.2 % TAR in the control samples).

TLC analyses of the soil extracts showed that approximately 68.6% (normal phase TLC) to 73.7% (reverse phase TLC), averaging 71% of the parent compound remained intact at the end of the 30 days irradiation. The control samples were stable throughout the course of the study. The half-life was calculated to be 65 days of continuous irradiation.

There was one major product formed, CL 312,622 (the di-acid metabolite) which accounted for approximately 11.9% TAR (normal phase TLC) to 15.4% TAR (reverse phase TLC), averaging 14% TAR after 30 days. This metabolite was also found in the dark control sample, indicating that this metabolite is not photolytic specific degradate. There were at least ten other unidentified radiolabelled components, each of which represented less than 6% TAR.

Study 2

The photolysis of imazamox was conducted using [pyridine-6-^{14}C]-imazamox or [imidazolinone-5-^{14}C]-imazamox [McCall and Blood 2013a, 2012/7003612]. The test compound was applied to a loam soil from New Jersey at a rate of 0.050 kg/ha (0.33 ppm based on an assumed bulk density of 1.5 g/cm^3 and 1 cm depth). Samples were irradiated continuously for up to 15 days at 20 °C by light from a xenon-arc lamp filtered to remove wavelengths less than 290 nm. Total irradiance during the duration of the study was 528.56 W/m^{-2} which is comparable to the natural sunlight intensity in the spring at 40 ° N latitude. The loam soil characteristics were as follows: 28% sand; 48% silt: 24% clay: 1.3% organic carbon; pH 6.9; CEC 8.9 meq/100 g; 49% a maximum of water holding capacity.

Volatile residues were collected for the irradiated and the dark control samples and analysed at the soil sampling intervals. Each sample was extracted once with 40 mL methanol and two times with 40 mL methanol:water (1:1; v:v) and finally with 40 mL 0.5 N NaOH at each sampling point. The extracts were analysed by HPLC. Reference standards were used for co-chromatography for characterization in conjunction with MS/MS analysis of select samples.

The mass balance for both irradiated and dark controls for the imidazolinone label ranged from 89.3% TRR to 101.2% TAR. The cumulative volatile ^{14}C-residues from the irradiated and dark control samples were 10.7 %TAR for the light and for the dark controls, 1.2% of the TAR. The mass balance for both irradiated and dark controls for the pyridine label ranged from 98.0% to 102.5% TAR.

In overall, extractabilities of radioactive residues were in the range of 70.7–99.9% TRR.

The DT_{50} value of imazamox in the irradiated samples was calculated to be approximately 28.4 days (both labels, n=4) and found to be 19.7 days in the dark control samples. One minor product (< 10% TAR) observed in the irradiated samples was the di-acid CL 312,622. It accounted for a maximum level of approximately 8% TAR through the study period in both labels. In the control samples, CL 312622 accounted for a maximum of 41% TRR in the imidazolinone label, and a maximum of 25% TRR in pyridine label through the study period. There were no unique photoproducts of imazamox in soil at 20 °C.

These studies indicated that imazamox on soil surface is photodegraded and any specific metabolite is not formed under irradiation. The half-life of imazamox was 65 days or 28 days and it was slightly longer in dark control condition. One metabolite CL 312622 was present in irradiated soil as a major or minor component; however, it was not specific for photolysis.

Hydrolysis

Test solution containing imazamox at concentration of 8 μg/mL was prepared in pH 4, 7 and 9 buffers and incubated at 50 °C [Holman 1997a, ID-322-002]. The test was conducted for 5 days in the dark under sterile conditions. Additional samples buffered at pH 9 were incubated for up to 24 days at 50 °C, 10 days at 60 °C, and 4 days at 70 °C. Aliquots of the samples were analysed by HPLC.

No degradation of imazamox occurred during the test period of 5 days at 50 °C in pH 4 and pH 7 buffers, indicating that the test compound is stable to hydrolysis in these buffers.

In the sample at pH 9, considerable loss of the compound was observed. For samples at pH 9 and 50 °C, the half-life of imazamox was calculated to be 11.9 days; at 60 °C, 4.17 days; and at 70 °C, 1.70 days. Extrapolating to 25 °C, the half-life is 192 days.

There was one major hydrolysis product confirmed by LC-MS. This was identified as the carbamoyl compound CL 336554, formed from the ring opening at the nitrogen bond.

The study indicated that imazamox is hydrolytically stable at pH 4 and pH 7. However, at pH 9 at 25 °C, it hydrolyses with a half-life of 192 days, forming the hydrolysis product, carbamoyl compound CL 336554.

Metabolites and distribution in succeeding crops

Confined Rotational Crop Study

Study 1

Soya beans (variety, Hutcheson) were treated at the 4–6 leaf stage (sandy loam soil) with [pyridine-6-^{14}C]-imazamox at a rate of 0.072 kg ai/ha (ammonium salt formulation) [Gatterdam 1994b, ID-640-002]. At 100 days after treatment (100 DAT), the soya bean crop was harvested and winter wheat (variety, Pioneer 2548) was seeded in subplot 1. At 268 DAT, maize (variety, Pioneer 3165), radish (variety, White Icicle) and lettuce (variety, Black-Seeded Simpson) were seeded (subplots 2–4), respectively. Radish and lettuce were planted in subplots 5 and 6 at 420 DAT.

Soya bean foliage was sampled at the day of treatment; fodder and seed were taken at harvest, 100 DAT. The rotational crop commodities wheat forage, maize forage (stalk, ear foliage and cob), radish leaves and lettuce plants were sampled at mid-maturity, 238, 357, 302 and 311 DAT, respectively. Wheat grain, maize seed, radish roots and lettuce plants were taken at harvest, 331, 420, 311 and 335 DAT, respectively. The study was terminated before harvesting the 420 DAT samples (radish and lettuce), thus these samples were not analysed for radioactive residues. Soil samples were taken before treatment, immediately after application, at rotational crop planting and at crop sampling (mid-maturity and harvest). Soil samples were 46 cm in depth (sections of 0–8, 8–15, 15–30, and 30–46 cm) except the pre-treatment and 0 DAT cores which were 30 cm in depth. Plant tissue and soil samples were combusted and ^{14}C was determined by LSC.

The soil extract was analysed by HPLC-UV. The column was eluted isocratically with a mobile system consisting of acetonitrile/ tetrahydrofuran/formic acid/water (14:2:1:83, v/v/v/v). A liquid scintillation cocktail was added for LSC. The retention time of imazamox analytical standard was compared to radiochromatograms of soil extracts to verify identification of the radioactivity.

Soya bean foliage at 0 DAT contained total radioactive residues averaging 1.70 mg eq/kg. The plants at harvest (100 DAT) were collected but not analysed.

Rotational crops planted at 100 DAT (wheat) and 268 DAT (maize, radish and lettuce) showed TRRs below the limit of detection of 0.01 mg/kg at both mid-maturity and harvest intervals. Further analysis for radish and lettuce planted at 420 DAT was not performed.

Radioactive residues in treated soil (0–8 cm) at 0 DAT averaged 0.03 mg/kg. At the 100 DAT, 268 DAT and 420 DAT rotational crop intervals, there were no detectable residues in soil (< 0.01 mg eq/kg TRR).

Plant and soil samples were stored frozen from sampling until analysis. Sampling to final analysis ranged from 14 to 71 days for crops and from 8 to 147 days for soil.

The stability of the treatment solution was verified in a soil extract sample (0 DAT). Recovery of radioactivity by extraction showed 92.8% was extracted and 7.2% was unextracted. Cartridge clean-up (C_{18} SPE) of the soil extract yielded two carbon-14 containing residues. The major fraction (approximately 90% of the cleaned up residue) showed one peak by HPLC constituting 97.3% of the recovered radioactivity. This radioactive peak coincided with imazamox on the HPLC-

UV chromatogram. The minor fraction (approximately 10% of the cleaned up residue) was not analysed due to low radioactivity.

Table 30 Total radioactive residues in crops after treatment with [^{14}C]imazamox (ID-640-002)

Matrix (Days after sowing /planting, DAP)	TRR (mg eq/kg)
Soya bean (0 DAT)	1.70
Soya bean (100 DAT)	n.a.
Plant back interval: 100 DAT, seeded	
Wheat forage (138 DAP, 238 DAT)	< 0.01
Wheat grain (231 DAP, 331 DAT)	< 0.01
Plant back interval: 268 DAT, seeded	
Maize forage (89 DAP, 357 DAT)	< 0.01
Maize seed (152 DAP, 420 DAT)	< 0.01
Radish leaves (34 DAP, 302 DAT)	n.a.
Radish roots (43 DAP, 311 DAT)	< 0.01
Lettuce plants (43 DAP, 311 DAT)	< 0.01
Lettuce plants (67 DAP, 335 DAT)	< 0.01
Plant back interval: 420 DAT, planted	
Radish	n.a.
Lettuce	n.a.

DAT: Days after treatment
n.a.: not analysed

Table 31 Total radioactive residues in soil samples following treatment with [^{14}C]imazamox (ID-640-002)

Soil Samples (Days after treatment, DAT)	TRR determined by direct combustion (mg eq/kg)
(0 DAT)	0.03 (0–8 cm); < 0.01 (8–46 cm)
(100 DAT)	< 0.01
Plant back interval: 100 DAT	
Wheat (100, 238, 331 DAT)	< 0.01
Plant back interval: 268 DAT	
Maize (268, 357, 420 DAT)	< 0.01
Radish (268, 302, 311 DAT)	< 0.01
Lettuce (268, 311, 335 DAT)	< 0.01
Plant back interval: 420 DAT	
Radish (420 DAT)	< 0.01
Lettuce (420 DAT)	< 0.01

Study 2

A confined rotational crop study was conducted with two radiolabels-[imidazolinone 5-^{14}C, 3-^{15}N]-imazamox and [pyridine-3-^{14}C, imidazolinone-3-^{15}N]-imazamox [Funk and Possienke 2013b, 2013/1085609]. The active substance was applied to bare sandy loam soil (USDA scheme) in plastic containers at a rate of 0.075 kg ai/ha using an automatic spray track system. The nature and the level of radioactive residues were investigated in spinach (variety, Corvette F1; immature and mature), white radish (variety, April Cross; root and top) and spring wheat (variety, Thassos; forage, hay, straw and grain). For the imidazolinone label samples from the plant back intervals 31, 119 and 364 days were investigated, while for the pyridine label plant back intervals of 29, 123 and 365 days were used.

Plant samples were harvested at maturity and additional immature spinach samples as well as spring wheat forage samples (in part dried to hay) were taken 23 to 33 days and 50 to 75 days after sowing, respectively. Mature and immature spinach leaves were sampled and the roots remained in the soil. Ripe white radishes were pulled from the soil and separated into the edible parts (root) and the remaining green parts (top). Soil samples were taken after ploughing at the individual plant back intervals and after harvest of the mature crops.

All samples were stored in a freezer at –18 °C or below. Prior to extraction and determination of the TRR, sample material was homogenized. Plant matrices with a sufficient level of radioactivity were extracted with solvents, while for samples with low concentrations of radiolabelled compounds the amount of the TRR was obtained only by combustion analysis. Soil samples were directly subjected to combustion.

Aliquots of homogenized plant material were extracted three times with methanol. The residue was further extracted in the same way with water (twice). The TRR included in some spring wheat hay and straw samples radioactive residue of pendimethalin, which was identified as a contamination in this study. The obtained methanol extracts of spring wheat matrices were partitioned three times with ethyl acetate for forage, hay and grain, with cyclohexane for wheat grain. For grain, further partition for the obtained ethyl acetate phase was conducted with n-hexane.

The radioactive residues in the PES (after solvent extraction with methanol and water) were subsequently extracted twice with ammonia. After ammonia extraction, the residues were dried and subsequently solubilized with different enzymes (β-glucosidase and hesperidinase; macerozyme R-10 and cellulase Onozuka R-10; α-amylase, β-amylase and amyloglucosidase). For straw, the macerozyme solubilizates were treated with yeast (fermentation). For grain, water extract derived from the solvent extraction with water two times was precipitated with acetone. The precipitate was treated with protease. The extracts were analysed by LSC and if needed, HPLC.

The calculated TRR of all extracted rotational crop matrices showed no major differences to the TRR values obtained by combustion.

At all plant back intervals, the highest TRR levels were found in spring wheat hay, straw and grain. The concentration of residues in grain decreased significantly after a plant back interval of approximately 365 days: in imidazolinone/pyridine label, respectively, 0.032/0.053 mg eq/kg at one month PBI and 0.002/0.004 mg eq/kg at one year PBI. For spinach and white radish, much lower TRR values (less than 0.01 mg eq/kg) were found in all PBI intervals and both labels.

Table 32 Total radioactive residues in crops after treatment with [^{14}C]imazamox (2013/1085609)

Matrix	TRR Combusted (mg eq/kg)		TRR Calc. (mg eq/kg)	
(Days After Sowing /Planting, DAP)	Imidazolinone label	Pyridine label	Imidazolinone label	Pyridine label
Plant back interval days	31	29	31	29
Spinach (immature) (25/26)	0.006	0.009	0.005	0.008
Spinach (mature) (43/41)	0.003	0.005	0.003	0.004
White radish top (64/67)	0.005	0.006	0.006	0.005
White radish root (64/67)	0.003	0.003	0.003	0.003
Spring wheat forage (52/52)	0.008	0.014	0.008	0.012
Spring wheat hay (52/52)	0.046	0.078	0.042	0.074 [b]
Spring wheat straw (incl. chaff) (115/108)	0.044	0.077	0.040 [b]	0.080 [b]
Spring wheat grain (115/108)	0.032	0.053	0.030	0.051
Plant back interval days	119	123	119	123
Spinach (immature) (26/23)	0.005	0.002	0.004	0.002
Spinach (mature) (42/39)	0.005	0.002	0.004	0.002
White radish top (60/65)	0.009	0.003	0.007	0.002
White radish root (60/65)	0.002	0.002	0.001	0.001
Spring wheat forage (53/50)	0.012	0.004	0.011	0.004
Spring wheat hay (53/50)	0.050	0.023	0.043	0.020
Spring wheat straw (incl. chaff) (97/108)	0.132	0.026	0.111 [b]	0.025
Spring wheat grain (97/108)	0.035	0.019	0.036	0.019
Plant back interval days	364	365	364	365

Matrix	TRR Combusted (mg eq/kg)		TRR Calc. (mg eq/kg)	
(Days After Sowing /Planting, DAP)	Imidazolinone label	Pyridine label	Imidazolinone label	Pyridine label
Spinach (immature) (33/33)	< 0.001	0.004	< 0.001 [a]	0.004
Spinach (mature) (46/48)	< 0.001	0.005	< 0.001 [a]	0.004
White radish top (60/76)	< 0.001	0.006	< 0.001 [a]	0.006
White radish root (60/76)	< 0.001	0.001	< 0.001 [a]	0.001
Spring wheat forage (55/75)	< 0.001	0.008	< 0.001 [a]	0.008
Spring wheat hay (55/75)	0.002	0.052	0.002 [a]	0.051
Spring wheat straw (incl. chaff) (137/129)	0.003	0.034	0.003 [a]	0.033
Spring wheat grain (137/129)	0.002	0.004	0.002 [a]	0.005

DAT: days after treatment
[a] TRR combusted. TRR calculated was not determined because of low amount of radioactivity.
[b] Including a contamination of pendimethalin

Soil samples (imidazolinone label/pyridine label) were taken at 0/0 day DAT. On the days after ploughing at 31/29, 119/123 and 364/365 DAT (PBIs) and on the days after harvest of mature crops, the each labelled soil samples were taken at 74, 161, 410 DATs in spinach, 95, 179, 424 DATs in white radish and 146, 216, 501 DATs in spring wheat.

In both labels, the residue concentration in the top soil layer decreased rapidly after aging and ploughing, 0.670 mg eq/kg (0 DAT) to 0.019 mg eq/kg (31 DAT) in imidazolinone label and 0.694 mg eq/kg (0 DAT) to 0.050 mg eq/kg (29 DAT) in pyridine label. Then, the level decreased relatively in small ratio: imidazolinone label 0.013 mg eq/kg at 364 DAT and pyridine label 0.018 mg eq/kg at 365 DAT. After harvest of the mature crops, the residue levels in soil were 0.009–0.019 mg eq/kg for the all PBIs of both labels, being remained more or less stable.

The extractability of the radioactive residues with methanol and water ranged for rotational crop matrices from 17.1% to 77.1% TRR in the imidazolinone label and from 17.8% to 77.9% TRR for the pyridine label. In spring wheat grain, low extractability with both solvents was observed (less than 30% TRR). Besides, extractabilities for some forage and hay sample (pyridine label, 123 DTA) were low (less than 30% TRR). The major portions of the radioactive residues were generally extracted with methanol, except for spring wheat grain (all PBIs, both labels), hay (123 DAT, pyridine label) and straw (365 DAT, pyridine label) where similar portions were extracted with methanol and water.

In most cases, higher portions of the radioactive residues extracted with methanol were water soluble, and lower portions were found in the organic fractions (ethyl acetate phase or cyclohexane phase). In some cases, comparable portions were found in the organic phases and in the water phase.

HPLC analysis for all spring wheat matrices and plant back intervals showed a metabolite pattern with only a few peaks. Parent imazamox was identified very small in three matrices: 31 DAT grain (0.002 mg/kg, 6.2% TRR) in imidazolinone label and 29 DAT hay (0.007 mg/kg, 9.1% TRR) and 29 DAT straw (0.002 mg/kg, 2.1%) in pyridine label. The metabolite CL 263284 was found in only two matrices-29 DAT hay (0.002 mg/kg, 2.3% TRR) and 123 DAT straw (0.001 mg/kg, 5.5% TRR) in pyridine label. CL 263284 is characterized by a hydroxyl group, derived from the cleavage of the methyl ether group (demethylation) of the parent. There were degradation products in minor concentrations. These were characterized by HPLC and/or liquid-liquid partition.

Table 33 Distribution of radioactive residues in rotational crops—imidazolinone label (2013/1085609)

		Extracts						PES	
DAT	TRR calc.	Methanol extract		Water extract		Total			
	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Spinach (immature)									
31	0.005	0.003	50.1	< 0.001	6.0	0.003	56.1	0.002	43.0
119	0.004	0.003	74.7	< 0.001	2.4	0.003	77.1	0.001	22.9
Spinach (mature)									
31	0.003	0.002	52.7	< 0.001	8.2	0.002	60.9	0.001	39.1
119	0.004	0.002	64.7	< 0.001	3.4	0.002	68.1	0.001	31.9
White radish top									
31	0.006	0.003	53.8	0.001	11.1	0.004	64.9	0.002	35.1
119	0.007	0.004	63.0	0.001	9.7	0.005	72.7	0.002	27.3
White radish root									
31	0.003	0.002	60.3	< 0.001	4.3	0.002	64.6	0.001	35.4
119	0.001	0.001	56.0	< 0.001	0.0	0.001	56.0	0.001	44.0
Spring wheat forage									
31	0.008	0.003 n.a.[a] n.a.[b]	35.5 n.a.[a] n.a.[b]	< 0.001	5.2	0.003	40.7	0.005	59.3
119	0.011	0.005 0.001[a] 0.004[b]	48.7 13.9[a] 33.5[b]	0.001	6.3	0.006	55.0	0.005	45.0
Spring wheat hay									
31	0.042	0.017 0.007[a] 0.008[b]	39.4 16.4[a] 19.9[b]	0.001	2.9	0.018	42.3	0.024	57.7
119	0.043	0.018 0.007[a] 0.012[b]	42.0 16.3[a] 28.7[b]	0.006	14.0	0.024	56.0	0.019	44.0
Spring wheat straw									
31	0.040	0.016 0.011[a] 0.007[b]	41.2 28.0[a] 17.6[b]	0.003	7.9	0.019	49.2	0.020	50.8
119	0.111	0.056 0.035[a] 0.020[b]	50.0 31.7[a] 17.6[b]	0.018	16.1	0.074	66.7	0.037	33.3
Spring wheat grain									
31	0.030	0.004 0.001[a] 0.003[b]	12.6 3.5[a] 8.8[b]	0.004	13.0	0.008	25.5	0.022	74.5
119	0.036	0.004 0.002[a] 0.002[b]	10.7 5.4[a] 5.3[b]	0.002	6.4	0.006	17.1	0.030	82.9

TRR calc.: sum of extracts + PES

[a] Organosoluble: ethyl acetate used as organic solvent for forage, hay and straw extracts; cyclohexane used as organic solvent for grain extracts.

[b] Water soluble: ethyl acetate used as organic solvent for forage, hay and straw extracts; cyclohexane used as organic solvent for grain extracts.

Table 34 Distribution of radioactive residues in rotational crops—pyridine label (2013/1085609)

		Extracts						PES	
DAT	TRR calc.	Methanol extract		Water extract		total			
	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Spinach (immature)									
29	0.008	0.004	50.7	0.001	6.2	0.005	56.8	0.004	43.2
123	0.002	0.001	54.0	< 0.001	9.9	0.001	63.9	0.001	36.1
365	0.004	0.003	72.2	< 0.001	5.7	0.003	77.9	0.001	22.1
Spinach (mature)									

		Extracts						PES	
DAT	TRR calc.	Methanol extract		Water extract		total			
	mg eq/kg	% TRR	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
29	0.004	0.002	42.4	< 0.001	8.2	0.002	50.6	0.002	49.4
123	0.002	0.001	45.1	< 0.001	9.8	0.001	54.9	0.001	45.1
365	0.004	0.002	59.6	< 0.001	7.1	0.002	66.7	0.001	33.3
White radish top									
29	0.005	0.003	49.4	0.001	10.0	0.003	59.4	0.002	40.6
123	0.002	0.001	42.5	< 0.001	12.4	0.001	54.9	0.001	45.1
365	0.006	0.004	62.1	0.001	11.4	0.004	73.5	0.002	26.5
White radish root									
29	0.003	0.002	69.6	< 0.001	2.9	0.002	72.5	0.001	27.5
123	0.001	0.001	67.4	< 0.001	2.6	0.001	70.0	< 0.001	30.0
365	0.001	0.001	66.8	< 0.001	4.0	0.001	70.8	< 0.001	29.2
Spring wheat forage									
29	0.012	0.005 0.002 [a] 0.003 [b]	41.2 13.1 [a] 24.8 [b]	< 0.001	3.8	0.005	45.0	0.006	55.0
123	0.004	0.001 n.a.[a] n.a.[b]	24.0 n.a.[a] n.a.[b]	< 0.001	4.8	0.001	28.8	0.003	71.2
365	0.008	0.004 n.a.[a] n.a.[b]	44.4 n.a.[a] n.a.[b]	< 0.001	6.0	0.004	50.4	0.004	49.6
Spring wheat hay									
29	0.074	0.025 0.010[a] 0.014[b]	33.7 13.3[a] 19.4[b]	0.007	9.8	0.032	43.4	0.042	56.6
123	0.020	0.002 0.001[a] 0.004[bc]	9.1[a] 5.6[b] 18.0	0.002	8.7	0.004	17.8	0.014	67.8
365	0.051	0.020 0.006[a] 0.011[b]	39.1 12.5[a] 21.1[b]	0.011	21.2	0.030	60.3	0.020	39.7
Spring wheat straw									
29	0.080	0.039 0.026[ad] 0.012[b]	48.6 32.3[a] 15.3[b]	0.010	12.6	0.049	61.3	0.031	38.7
123	0.025	0.006 0.003[a] 0.003[b]	23.6 10.1[a] 12.3[b]	0.003	11.4	0.009	35.0	0.016	65.0
365	0.033	0.007 0.002[a] 0.004[b]	21.5 6.7[a] 12.4[b]	0.007	20.5	0.014	42.0	0.019	58.0
Spring wheat grain									
29	0.051	0.008 0.002[a] 0.006[b]	15.9 3.9[a] 11.3[b]	0.007	13.4	0.015	29.3	0.036	70.7
123	0.019	0.002 0.001[a] 0.001[b]	8.7 2.9[a] 4.4[b]	0.002	9.3	0.003	18.0	0.015	82.0
365	0.005	0.001 n.a.[a] n.a.[b]	11.8 n.a.[a] n.a.[b]	0.001	16.0	0.001	27.8	0.003	72.2

TRR calc.: sum of extracts + PES

[a] Organosoluble: ethyl acetate used as organic solvent for forage, hay and straw extracts; cyclohexane used as organic solvent for grain extracts.

[b] Water soluble: ethyl acetate used as organic solvent for forage, hay and straw extracts; cyclohexane used as organic solvent for grain extracts.

[c] Radioactive residues in methanol extract presumably underestimated (low dpm values in LSC measurement).

[d] Organosoluble as sum of water phase and n-hexane phase derived from ethyl acetate phase

Table 35 Identified or characterized residues in wheat samples—imidazolinone label (2013/1085609)

Designation	Wheat forage 119 DAT		Wheat hay 31 DAT		Wheat hay 119 DAT		Wheat straw 31 DAT		Wheat straw 119 DAT		Wheat grain 31 DAT		Wheat grain 119 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Imazamox								12.1			0.002	6.2		
Total characterized from extracts	0.006	53.7	0.018	42.0	0.025	59.0	0.017	44.1	0.072	64.4	0.005	17.1	0.006	16.5
Total characterized from PES			0.014	33.5	0.009	20.6	0.010	24.1	0.020	18.0	0.020	67.6	0.025	69.5
Total identified and /or characterized			0.032	75.5	0.034	79.6	0.032[a]	80.0[a]	0.092[b]	82.4[b]	0.027	90.9	0.031	86.0
Final residue	0.005		0.006	14.5	0.006	14.7	0.009	23.4	0.017	15.6	0.003	9.1	0.004	9.8
Grand total	0.011	100.0	0.038	90.0	0.040	94.3	0.041	103.6	0.109	98.0	0.030	99.9	0.035	95.8

[a] For wheat straw (31 DAT), the value included residue concentration of pendimethalin (contaminant), detected at 0.005 mg/kg (12.1% TRR).

[b] For wheat straw (119 DAT), the value included residue concentration of pendimethalin (contaminant), detected at 0.035 mg/kg (31.7% TRR).

Table 36 Identified or characterized residues in rotational crops (wheat forage, hay and straw)—pyridine label (2013/1085609)

Designation	Wheat forage 29 DAT		Wheat hay 29 DAT		Wheat hay 123 DAT		Wheat hay 365 DAT		Wheat straw 29 DAT		Wheat straw 123 DAT		Wheat straw 365 DAT	
	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Imazamox			0.007	9.1	0.001	4.0			0.002	2.1				
CL 263284			0.002	2.3							0.001	5.5		
Total characterized from ERR	0.005	41.7	0.021	28.9	0.006	28.2	0.027	53.7	0.038	46.8	0.007	28.3	0.013	39.6
Total characterized from PES			0.024	31.9	0.006	29.8	0.010	20.7	0.012	14.5	0.005	20.3	0.007	21.5
Total identified and /or characterized			0.055	74.3[a]	0.013	62.0	0.038	74.4	0.059[a]	73.5[2]	0.013	54.2	0.020	61.1
Final residue	0.006	55.0	0.014	18.7	0.007	31.8	0.007	14.6	0.014	17.7	0.009	36.7	0.012	35.7
Grand total	0.011	96.7	0.069	93.0[a]	0.019	93.8	0.045	89.0	0.073[a]	91.2[a]	0.022	90.9	0.032	96.9

[a] Residue value (0.002 mg/kg, 2.2% TRR) of contaminant pendimethalin was included.

Table 37 Identified or characterized residues in rotational crop (wheat grain)—pyridine label (2013/1085609)

Designation	Wheat grain 29 DAT		Wheat grain 123 DAT	
	mg eq/kg	%TRR	mg eq/kg	%TRR
Imazamox	0.005	10.1	0.001	2.7
CL 263284	0.001	1.4		
Total characterized from extracts	0.004	8.5	0.003	15.0
Total characterized from PES	0.026	50.7	0.010	52.7
Total identified and /or characterized	0.039	77.6	0.013	70.5
Final residue	0.005	10.6	0.002	13.3
Grand total	0.045[a]	88.2[a]	0.016[b]	83.8[b]

[a] Loss of 8.8% TRR during ammonia solubilisation for solid residue was not significant due to low amount of radioactive residues (0.004 mg/kg).

[b] Loss of 16.7% TRR during ammonia solubilisation for solid residue was not significant due to low amount of radioactive residues (0.003 mg/kg).

Methanol and water extracts of rotational crop matrices were prepared within periods of 12 to 243 days after sampling for the imidazolinone label and 223 to 463 days after sampling for the pyridine label. For partition phases of the methanol extract, concentrated water extracts and supernatants from water extract incubations (after acetone precipitation and protease treatment, HPLC analysis was conducted within 28 to 458 days after extraction for imidazolinone label and 7 to 329 days after extraction for pyridine label.

With spring wheat grain (imidazolinone label PBI 31 DAT and pyridine label PBI 29 DAT samples), storage stability tests were performed for the water phase of the methanol extract.

PBI 31 DAT wheat grain sample was extracted at 176 days after sampling. The stored water phase of the methanol extract was analysed by HPLC at 220 days after extraction and re-analysed at 647 days (14 months) after extraction. For PBI 29 DAT wheat grain sample, extraction was conducted at 387 days after sampling. The water phase of the methanol extract was analysed by HPLC at 54 days after extraction and re-analysed at 344 days (9 months) after extraction. As results, water phase of the methanol extract was stable for at least 14 months.

PBI 31 DAT and 29 DAT wheat grain samples were re-extracted at 817 days (2.2 years) after sampling and 729 days (2 years) after sampling, respectively. Similar amounts of radioactive residues were extracted compared to the initial extractions (176 days after sampling for PBI 31 DAT and 387 days after sampling for PBI 29 DAT). Further, HPLC analysis for the water phase of the methanol extract showed similar HPLC patterns. Thus, residues in wheat grain frozen stored were stable for about 2 years. From these results, residues in wheat grain over the period of the study were shown to be stable.

In summary, the confined rotational crop studies (radish, lettuce, spinach, maize and wheat) showed that imazamox-derived residues from a previous use or soil treatment do not accumulate in rotational crops; only low levels of radioactive residues were observed in all rotation crop matrices at all analysed plant back intervals.

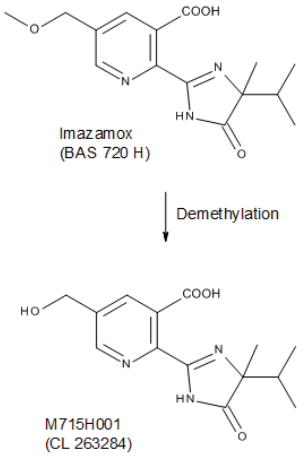


Figure 3 Proposed metabolic pathway of imazamox in rotational crops

Field rotational studies

No data submitted.

RESIDUE ANALYSIS

The Meeting received descriptions and validation data for analytical methods for imazamox and its metabolites in plant or animal matrices.

Good linearity was observed in the standard solutions with linear correlations with coefficients > 0.99. Overall, recovery values were within an acceptable level (70–120%), where the

relative standard deviations were mostly less than 20%. A brief description of the methods is given below.

Table 38 Summary of analytical methods used for the determination of imazamox and its metabolites in various matrices

Method No.	Matrix	Analyte(s)	Detection	LOQ (mg/kg)	Reference
L0188/01	Grapes Green beans Peas (dry) Rice (grain) Sunflower (seed) Rice (straw) Rice (whole plant)	Imazamox CL 263284 CL 189215 CL 312622	LC-MS/MS	0.01	2012/1294678 (validation) 2013/1177533 (validation) 2013/1249356 (ILV)
CEM-236/001	Peas French beans (pods) French beans (whole plant) Maize (grain) Maize (ears, whole plant)	Imazamox CL 263284	HPLC-UV	0.05	ID-244-018 (validation) ID-244-015 (validation) ID-244-016 (validation) ID-244-017 (validation)
M 3076	Oilseed rape (seed)	Imazamox	CE	0.05	ID-244-020 (ILV)
M 2460	Peas (whole pod) Peas (dry seed) Soya bean (seed) Oilseed rape (seed) Peas (vine, hay, straw) Soya bean (forage, straw)	Imazamox CL 263284 combined imazamox + CL 263284	GC-NPD	0.05	ID-244-005 (ILV)
M 3519	Lentil (seed) Lentil (forage)	Imazamox CL 263284 CL 189215 CL 312622	LC-MS, LC-MS/MS	0.05	2004/5000274 (validation) 2002/5004302 (ILV)
M 3515	Orange Grapes Peas Maize (grain) Wheat (grain) In ILV, grapes, maize (grain), and wheat (grain)	Imazamox CL 263284	LC-MS	0.05 for orange, grapes, peas and maize (grain) 0.01 for wheat (grain)	2002/7006089 (ID-244-029) (validation) 2002/5002749 (ILV)
M 3178	Alfalfa (forage + hay) Alfalfa (seed)	Imazamox CL 263284 CL 189215 CL 312622	LC-MS LC-MS/MS	0.1	ID-244-022 (ILV)
M 3098	Wheat (grain) Wheat (straw + hay) Wheat (forage)	Imazamox CL 263284	CE-UV	0.05	ID-244-026 (ILV)
M 2248, M 2248.01	Soya bean (seed) Peanut (nut) Soya bean (foliage) Peanut (foliage) In ILV, soya bean (seed) only	Imazamox	HPLC-UV	0.05	M 2248 ID-244-002 (validation) M 2248.01 ID-244-001 (validation) ID-244-007 (validation) ID-244-009 (validation) ID-244-008 (validation) ID-244-010 (validation) ID-244-004 (ILV of M 2248.01)
M 2333 (confirmatory method for M 2248)	Soya bean (seed)	Imazamox	LC-MS	0.05	ID-244-003 (validation)
ABC Report 46324	Sunflower (seed) Sunflower (refined oil) Sunflower (meal)	Imazamox CL 263284 CL 312622	LC-MS	0.05	2002/5004111 (validation)
SOP-PA.0288	Sunflower (seed)	Imazamox CL 263284 CL 189215	LC-MS/MS	0.01	2010/1090461 (validation) 2010/1057139 (validation)

Method No.	Matrix	Analyte(s)	Detection	LOQ (mg/kg)	Reference
D0303	Bovine muscle Bovine fat Bovine kidney + liver Bovine milk Poultry egg (validated only in reference 2013/7002842)	Imazamox CL 263284	LC-MS/MS	0.01	2003/5000116 (validation) 2013/7002842 (validation)

Analytical methods for plant matrices

L0188/01

Imazamox and its metabolites CL 312622, CL 263284 and CL 189215 were extracted from plant matrices (grapes, green beans, dry peas, rice grain, sunflower seed, rice straw, rice whole plant) with methanol/water/1 N HCl (60:39:1, v/v/v). In case of high protein samples (e.g. peas and beans), ammonium sulphate was added to the extract. A portion of the extract was centrifuged for 5 min at 4000 rpm, filtered through a disposable syringe filter and an aliquot of the filtrate was diluted to obtain the final volume for LC-MS/MS determination against matrix matched standards. In all matrices tested the mean recovery values were between 80% and 108% and the RSDs were below 12% except one value (23% in 0.01 mg/kg fortified sunflower seed). The LOQ for each analyte was 0.01 mg/kg in all matrices tested [Lehmann 2013a, 2012/1294678; Lehmann 2013b, 2013/1177533].

Table 39 Recovery results of imazamox, CL 312622, CL 263284 and CL 189215 from plant matrices using the method L0188/01

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)		RSD (%)	
Transition				1:00 AM	2	1:00 AM	2
Imazamox	Rice (whole plant)	0.01	5	101.3	107.4	9.1	6.3
		0.1	5	92.3	91	2.5	2.7
	Rice (grain)	0.01	5	95.4	94.4	1.5	2.5
		0.1	5	94.4	95	1.9	0.9
	Rice (straw)	0.01	5	102.5	100.3	6.5	7.8
		0.1	5	87.2	86.9	4	4.2
	Green beans	0.01	5	96.9	95	1.8	1.6
		0.1	5	92.8	92.6	0.5	1.3
	Sunflower (seeds)	0.01	5	95.4	94.8	2.5	4.3
		0.1	5	91.1	92.3	2.8	3.4
	Peas (dry)	0.01	5	89.1	89.5	11.7	12
		0.1	5	86.8	90.5	4.2	7.4
	Grapes	0.01	5	94.1	93.6	3.1	6.7
		0.1	5	85.7	85.5	4.4	4.3
Transition				1:00 AM	2	1:00 AM	2
CL 312622	Rice (whole plant)	0.01	5	98.7	105.5	9	9.1
		0.1	5	92.2	90.8	1.4	1.7
	Rice (grain)	0.01	5	96.7	93.9	1.6	4.9
		0.1	5	94.8	95.1	2.2	1.6
	Rice (straw)	0.01	5	96	100.8	5.3	7.6
		0.1	5	85.7	84.4	3.1	3.8
	Green beans	0.01	5	93.6	91	2.1	4.6
		0.1	5	91.9	92.5	1.5	1.5
	Sunflower (seeds)	0.01	5	92.6	92	6.9	4.4
		0.1	5	89	87.7	3	3.8
	Peas (dry)	0.01	5	92	92	4.5	10.2
		0.1	5	89.7	92.2	4.9	1.5
	Grapes	0.01	5	94.7	101.9	9.1	6.8
		0.1	5	86.6	88.9	3.9	4.5

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)		RSD (%)	
Transition				1	2:00 AM	1	2:00 AM
CL 263284	Rice (whole plant)	0.01	5	95.1	98.8	9.4	8.8
		0.1	5	94.1	93.8	2.5	2.5
	Rice (grain)	0.01	5	99.8	99.5	4.5	3.9
		0.1	5	97.9	96.9	2.9	2.6
	Rice (straw)	0.01	5	101.4	103.2	8.6	9.2
		0.1	5	89.4	89.2	3.2	1.9
	Green beans	0.01	5	94.1	92.4	4.1	1.8
		0.1	5	91.3	91.2	1.6	1.4
	Sunflower (seeds)	0.01	5	89.4	88.4	2.8	3.7
		0.1	5	85	84.7	2.5	3
	Peas (dry)	0.01	5	93.2	94.8	11.8	4.6
		0.1	5	97.3	95.3	2.2	1.9
	Grapes	0.01	5	97.1	93.4	4.4	3.8
		0.1	5	87.8	87.3	3.2	2.9
Transition				1:00 AM	2	1:00 AM	2
CL 189215	Rice (whole plant)	0.01	5	98.6	98.1	8.5	10
		0.1	5	94.7	93.7	1.1	2.6
	Rice (grain)	0.01	5	103.1	107.7	3.3	7.3
		0.1	5	100	98.9	2.8	2.2
	Rice (straw)	0.01	5	101.8	93.5	8.9	8.5
		0.1	5	91.8	90.3	1.4	2.9
	Green beans	0.01	5	89	90.6	2.2	5.4
		0.1	5	89.1	88.7	3.1	3.4
	Sunflower (seeds)	0.01	5	87.7	80.1	2.1	23.3
		0.1	5	88.3	88	1.8	3.6
	Peas (dry)	0.01	5	92.7	89.9	12.2	14.1
		0.1	5	95.8	95.8	4.7	3
	Grapes	0.01	5	102	94.3	8.2	8.2
		0.1	5	88	91.6	2.9	4.3

Transition 1: imazamox, 306→261 m/z; CL 312622, 306→261 m/z; CL 263284, 292→232m/z; CL 189215, 454→292 m/z

Transition 2: imazamox, 306→193 m/z; CL 312622, 306→69 m/z; CL 263284, 292→247 m/z; CL 189215, 454→86 m/z

[a] Proposed for quantification

An independent laboratory validation study (ILV) of method L0188/01 for the determination of imazamox, CL 312622, CL 263284 and CL 189215 in plant matrices (grapes, green beans, dry peas, rice grain, sunflower seed, rice straw and rice whole plant) was conducted. In all matrices tested, the mean recovery values were between 76% and 110% (RSD, < 18%). The LOQ for each analyte was 0.01 mg/kg in all matrices tested [Mewis A., 2013a, 2013/1249356].

Table 40 Recovery results of imazamox, CL 312622, CL 263284 and CL 189215 from plant matrices using the Method L0188/01 (independent laboratory validation study)

Test Substance	Crop	Fortification Level (mg/kg)	No. of Tests	Average Recovery (%)		RSD (%)	
Transition				1:00 AM	2	1:00 AM	2
Imazamox	Rice (whole plant)	0.01	5	106	102	8	4
		0.1	5	78	78	12	12
	Rice (grain)	0.01	5	101	97	3	2
		0.1	5	85	84	6	6
	Rice (straw)	0.01	5	99	102	3	1
		0.1	5	86	85	16	16
	Green beans	0.01	5	106	106	7	4
		0.1	5	86	86	8	8

Test Substance	Crop	Fortification Level (mg/kg)	No. of Tests	Average Recovery (%)		RSD (%)	
	Sunflower (seeds)	0.01	5	97	93	5	6
		0.1	5	85	83	6	7
	Peas (dry)	0.01	5	100	99	3	5
		0.1	5	83	84	4	3
	Grapes	0.01	5	108	106	6	4
		0.1	5	92	90	7	7
Transition				1:00 AM	2	1:00 AM	2
CL 312622	Rice (whole plant)	0.01	5	106	96	6	15
		0.1	5	80	77	11	14
	Rice (grain)	0.01	5	103	101	4	3
		0.1	5	84	88	5	3
	Rice (straw)	0.01	5	103	101	4	3
		0.1	5	82	81	16	16
	Green beans	0.01	5	106	106	5	4
		0.1	5	84	87	7	7
	Sunflower (seeds)	0.01	5	98	94	6	11
		0.1	5	86	83	6	6
	Peas (dry)	0.01	5	102	106	6	3
		0.1	5	83	83	3	4
	Grapes	0.01	5	103	100	7	9
		0.1	5	92	92	6	8
Transition				1:00 AM	2	1:00 AM	2
CL 263284	Rice (whole plant)	0.01	5	109	108	11	6
		0.1	5	80	78	13	11
	Rice (grain)	0.01	5	104	107	3	4
		0.1	5	84	84	8	5
	Rice (straw)	0.01	5	104	101	6	3
		0.1	5	82	81	17	16
	Green beans	0.01	5	102	106	9	8
		0.1	5	83	82	8	8
	Sunflower (seeds)	0.01	5	96	100	11	7
		0.1	5	85	84	8	6
	Peas (dry)	0.01	5	99	106	2	3
		0.1	5	83	84	2	3
	Grapes	0.01	5	108	108	7	5
		0.1	5	94	92	6	7
Transition				1:00 AM	2	1:00 AM	2
CL 189215	Rice (whole plant)	0.01	5	108	94	10	11
		0.1	5	77	76	13	11
	Rice (grain)	0.01	5	104	100	6	9
		0.1	5	86	87	7	10
	Rice (straw)	0.01	5	106	101	8	12
		0.1	5	86	85	15	16
	Green beans	0.01	5	107	107	5	18
		0.1	5	82	82	7	7
	Sunflower (seeds)	0.01	5	99	100	9	11
		0.1	5	81	83	6	5
	Peas (dry)	0.01	5	105	100	2	9
		0.1	5	85	82	4	5
	Grapes	0.01	5	106	110	5	10
		0.1	5	90	89	6	7

Transition 1: imazamox, 306→261 m/z; CL 312622, 306→261 m/z; CL 263284, 292→232m/z; CL 189215, 454→292 m/z

Transition 2: imazamox, 306→193 m/z; CL 312622, 306→69 m/z; CL 263284, 292→247 m/z; CL 189215, 454→86 m/z

[a] Proposed for quantification

This method was used in the field residue trials for beans, rice and sunflower seeds, where the procedural recoveries and LOQ levels were acceptable.

CEM-236/001

Imazamox and CL 263284 are extracted from plant matrices (peas, beans pods, whole bean, maize grain, maize ears and maize whole plant) by homogenization with methanol/water/1 M hydrochloric acid (60:39:1, v/v/v). Clean-up of the extract was achieved using liquid/liquid partition and solid phase extraction using a Bond Elut SCX cartridge. Final quantitative determination of residues was by HPLC-UV detection at 254 nm. In all tested matrices, the recovery values were in the range of 72% to 110%. The LOQ for each analyte was 0.01 mg/kg in all matrices tested [Robbins 1996a, ID-244-018; Robbins 1997a, ID-244-015; Robbins 1997b, ID-244-016; Robbins 1997c, ID-244-017].

Table 41 Recovery results of imazamox and CL 263284 from plant matrices using the Method CEM-236/001

Substance	Crop	Fortification level (mg/kg)	No. tests	Range (mg/kg)	Average recovery (%)	RSD (%)
Imazamox	Peas (fresh)	0.05–1.0	5	94–109	101	5.7
	French beans (pods)	0.05–1.0	5	73–99	88	12.5
	French beans (whole plant)	0.05–1.0	5	79–98	90	8
	Maize (grain)	0.05–1.0	5	80–107	93	13.1
	Maize (ears)	0.05–1.0	5	87–104	94	6
	Maize (whole plant)	0.05–1.0	5	82–97	89	6.5
CL 263284	Peas (fresh)	0.05–1.0	5	76–98	89	10.2
	French beans (pods)	0.05–1.0	5	72–82	77	6
	French beans (whole plant)	0.05–1.0	5	72–89	79	10
	Maize (grain)	0.05–1.0	5	91–110	102	6.9
	Maize (ears)	0.05–1.0	5	82–90	86	3.5
	Maize (whole plant)	0.05–1.0	5	75–87	82	5.5

Recovery test was conducted with one test per each of five fortification levels (0.05, 0.10, 0.25, 0.50 and 1.0 mg/kg).

In the field residue trials for beans, peas and rice, this method was used with acceptable procedural recoveries and LOQ levels.

M 3076

An independent laboratory validation study of method M 3076 for the determination of residues of imazamox in canola seed was performed. Residues of imazamox were extracted from oilseed rape seed (canola) with acidic aqueous methanol. Clean-up of the extract was achieved by evaporation, liquid/liquid partition and solid phase extraction using an SCX cartridge. Final quantitative determination of residues was by capillary electrophoresis (CE). The recovery values were in the range of 74% to 85%. The LOQ for imazamox was 0.05 mg/kg in rape seed [Sweeney and Gross 1998a, ID-244-020].

Table 42 Recovery results of imazamox from rape seed using the method M 3076 (independent laboratory validation study)

Crop	Fortification level (mg/kg)	No. of tests	Recovery (%)	Average recovery (%)	RSD (%)
Oilseed rape (seeds)	0.05	2	84, 85	85	
	0.10	2	74, 85	80	
	0.50	2	79, 83	81	

M 2460

Residues of imazamox and CL 263284 are extracted from the plant samples (pea whole pod/dry seed, soya bean seed/forage/straw, rape seed, and pea vine/hay/straw) with acidic methanol-water. The extracts are subjected to suitable clean-up involving solvent partitioning and solid phase extraction. Both compounds are converted to a common methylated product and measured as one chromatographic peak representing a total imazamox-related residue. Measurement of the total imazamox-related residues is accomplished using on-column methylation and GC-NPD. Results were calculated as total imazamox-related residues by the direct comparison of peak heights to those of external standards. In all matrices tested, the recovery values were in the range of 65% to 131%. The LOQ was 0.05 mg/kg for each compound as well as for combined imazamox and CL 263284 in all matrices tested [Safarpour 1995a, ID-244-005].

Table 43 Recovery results of imazamox and CL 263284 from plant matrices using the method M 2460

Test substance	Crop	Fortification level (mg/kg)	No. of Tests	Recovery (%)	Average recovery (%)	RSD (%)
Imazamox + CL 263284	Oilseed rape (seeds)	0.05	2	85, 105	95	
		0.50	2	120, 121	121	
	Pea (vines)	0.05	2	91, 131	111	
		0.50	2	106, 116	111	
	Pea (hay)	0.05	2	90, 91	91	
	Pea (dry seed)	0.05	2	99, 102	101	
	Pea (straw)	0.05	2	65, 103	84	
	Pea (whole pod)	0.05	2	85, 100	93	
	Soya bean (forage)	0.05	2	70, 83	77	
	Soya bean (seed)	0.05	2	82, 84	83	
	Soya bean (straw)	0.05	1	98	98	
		0.50	1	105	105	
Imazamox	Oilseed rape (seeds)	0.05	2	114, 121	118	
	Soya bean (straw)	0.05	2	104, 110	107	
CL 263284	Oilseed rape (seeds)	0.05	2	124, 122	123	
	Soya bean (straw)	0.05	2	91, 94	93	

This method was used in the field residue trials for peas, soya bean and rape seeds, where the procedural recoveries and LOQ levels were acceptable.

M 3519

Imazamox, CL 263284, CL 189215 and CL 312622 were extracted from lentil seed and forage samples using an acidic methanol-water solution. An aliquot of the seed or forage sample extract was diluted with methanol and then passed through an SCX cartridge, which retained the analytes. The SCX cartridge was washed with methanol to remove co-extractives; then the residues were selectively eluted with a water-methanol solution. The eluate was evaporated to dryness and the residues were dissolved in acidic water for analysis. Measurement of the residues was accomplished by LC-MS or LC-MS/MS. In both matrices tested, the mean recovery values were between 75% and 99% (RSD, < 4%). The LOQ for each analyte was 0.05 mg/kg in lentil seed and forage [Nejad 2004a, 2004/5000274].

Table 44 Recovery results of imazamox, CL 263284, CL 189215 and CL 312622 from lentils using the method M 3519

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Lentil (seed)	0.05	5	93	4
		0.5	5	95	2
	Lentil (forage)	0.05	5	94	2
		0.5	5	92	1
CL 263284	Lentil (seed)	0.05	5	98	4
		0.5	5	95	2
	Lentil (forage)	0.05	5	94	2
		0.5	5	93	1
CL 189215	Lentil (seed)	0.05	5	89	3
		0.5	5	99	2
	Lentil (forage)	0.05	5	92	3
		0.5	5	96	3
CL 312622	Lentil (seed)	0.05	5	86	4
		0.5	5	75	2
	Lentil (forage)	0.05	5	95	4
		0.5	5	87	3

An independent laboratory validation of method M 3519 for the determination of imazamox, CL 263284, CL 189215 and CL 312622 residues in lentils (seed and forage) was performed. In both matrices tested, the mean recovery values were between 84% and 109% (RSD, < 19%). The LOQ for each analyte was 0.05 mg/kg in lentil seed and forage [Jordan J.M., 2003a, 2002/5004302].

Table 45 Recovery results of imazamox, CL 263284, CL 189215 and CL 312622 from lentils using the method M 3519 (independent laboratory validation study)

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Lentil (seed)	0.05	5	95	16
		0.50	5	87	7
	Lentil (forage)	0.05	5	84	15
		0.50	5	88	5
CL 263284	Lentil (seed)	0.05	5	109	19
		0.50	5	99	4
	Lentil (forage)	0.05	5	100	9
		0.50	5	87	14
CL 189215	Lentil (seed)	0.05	5	86	7
		0.50	5	93	7
	Lentil (forage)	0.05	5	84	2
		0.50	5	89	6
CL 312622	Lentil (seed)	0.05	5	86	10
		0.50	5	88	6
	Lentil (forage)	0.05	5	85	11
		0.50	5	87	13

This method was used in the field residue trials for lentil, rape seeds and sunflower seeds, where the procedural recoveries and LOQ levels were acceptable.

Homogenised subsamples of straw and seed were additionally extracted according to method M 3519, QuEChERS method and DFG S19 method. For straw samples the extractability with method No. M 3519 (80.2% TRR) and QuEChERS method (64.8% TRR) was comparable to the extractability with methanol and water (77.6% TRR). For seed, method M 3519 extracted about one half (31.0% TRR) and QuEChERS method one third (17.2% TRR) of the radioactive residues extracted with methanol and water (58.3% TRR). For both matrices the DFG S19 method extracted the lowest amounts of radioactive residues with 16.9% TRR for straw and 8.0% TRR for seed. HPLC analyses of the straw and seed extracts obtained from extraction with method M 3519, QuEChERS and DFG S19

method (for straw only) showed a similar metabolite pattern compared to methanol extracts used for metabolism investigation, with CL 263284 as the main component. This data demonstrates that among the tested residues methods method M 3519 has the capability to extract the relevant residues of imazamox.

M 3515

Imazamox and CL 263284 were extracted from plant matrices (orange, grape, pea, maize grain and wheat grain) using an acidic methanol-water solution. For wheat grain, an aliquot of the sample extract was evaporated to dryness, the residue dissolved in acidic water and then passed through a C-18 cartridge, which retained the analytes. The cartridge was washed with water to remove co-extractives; then the residues were downloaded with methanol onto an SCX cartridge, which retained the analytes. For all other plant matrices, an aliquot of the sample extract was diluted with methanol then passed through an SCX cartridge, which retained the analytes. For all plant matrix samples, the SCX cartridge was washed with methanol to remove co-extractives then the residues were eluted with a water-methanol solution. The eluate was evaporated to dryness and the residues were dissolved in water for analysis. Measurement of the residues was accomplished by LC-MS. In all matrices tested, the mean recovery values were between 83% and 109% (RSD, < 12%). The LOQ for each analyte was 0.01 mg/kg in wheat grain and 0.05 mg/kg in all other matrices [Fletcher 2002a, ID-244-029].

Table 46 Recovery results of imazamox and CL 263284 from plant matrices using the method M 3515

Test substance	Crop	Fortification level (mg/kg)	No. of Tests	Average recovery (%)	RSD (%)
imazamox	Peas	0.05	5	94	6.5
		0.5	5	96	3.8
	Grapes	0.05	5	94	5.4
		0.5	5	88	2.0
	Orange	0.05	5	97	2.5
		0.5	5	89	3.7
	Maize (grain)	0.05	5	89	2.7
		0.5	5	91	9.1
	Wheat (grain)	0.01	5	83	3.1
		0.1	5	77	6.3
L 263284	Peas	0.05	5	95	3.4
		0.5	5	91	6.1
	Grapes	0.05	5	95	4.9
		0.5	5	86	3.1
	Orange	0.05	5	106	2.5
		0.5	5	90	3.6
	Maize (grain)	0.05	5	109	3.5
		0.5	5	94	11.6
	Wheat (grain)	0.01	5	90	6.7
		0.1	5	71	4.5

An independent laboratory validation study of method M 3515 for the determination of imazamox and CL 263284 residues in plant matrices (grape, maize grain and wheat grain) was performed. In all matrices tested, the mean recovery values were between 77% and 97% (RSD, < 19%). The LOQ for each analyte was 0.01 mg/kg in wheat grain and 0.05 mg/kg in grape and maize grain [Malinsky 2002a, 2002/5002749].

Table 47 Recovery results of imazamox and CL 263284 from plant matrices using the method M 3515 (Independent laboratory validation study)

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Grapes	0.05	5	86	9
		0.50	5	89	6
	Maize (grain)	0.05	5	93	4

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
		0.50	5	91	3
	Wheat (grain)	0.01	5	97	2
		0.10	5	90	6
CL 263284	Grapes	0.05	5	78	10
		0.50	5	79	5
	Maize (grain)	0.05	5	94	4
		0.50	5	84	4
	Wheat (grain)	0.01	5	88, 96 [a]	19, 5 [a]
		0.10	5	77	11
		overall	9	85	14

[a] When one value of 55% was excluded, the average (n = 4) and the RSD

In the field residue trials for lentil and sunflower seeds, this method was used with acceptable procedural recoveries and LOQ levels.

M 3178

An independent laboratory validation study of the method M 3178 was conducted. Imazamox, CL 263284, CL 189215 and CL 312622 in alfalfa (forage, hay and seeds) were extracted from the samples with acidic water-methanol followed by filtration. For imazamox, CL 263284 and CL 189215, the extracts were cleaned up with precipitation, centrifugation and solid phase extraction (SPE) techniques using RP-102 and SCX columns. For CL 312622, the extracts were cleaned up by filtration with an ISOLUTE filtration column and SPE (C_{18}, QMA and SCX columns). Measurements of the residues were accomplished by reverse-phase HPLC with mass spectrometric detection (LC-MS) and monitoring product ions (LC-MS/MS). LC-MS/MS was used for the analysis of imazamox, CL 263284, CL 189215 and CL 312622 in alfalfa seed samples. The recovery values were between 68% and 109%, except for alfalfa seed at 10 mg/kg fortification level (59–75%). The LOQ for each analyte was 0.1 mg/kg in the all alfalfa matrices [Wickremesinhe and Safarpour 1998a, ID-244-022].

Table 48 Recovery results of imazamox, CL 263284, CL 189215 and CL 312622 from alfalfa using the method M 3178 (independent laboratory validation study)

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Recovery range (%)	Average recovery (%)	RSD (%)
Imazamox	Alfalfa (forage)	0.1–10	8	73–101	85	12
	Alfalfa (hay)	0.1–10	8	82–102	92	7.5
	Alfalfa (seeds)	0.1–10	8	59 [a], 84–88	83	12
CL 263284	Alfalfa (forage)	0.1–10	8	80–102	87	11
	Alfalfa (hay)	0.1–10	8	76–88	84	4.9
	Alfalfa (seeds)	0.1–10	8	68 [a], 84–109	91	13
CL 189215	Alfalfa (forage)	0.1–10	8	74–96	82	11
	Alfalfa (hay)	0.1–10	8	68, 68, 71–82	76	7.9
	Alfalfa (seeds)	0.1–10	8	59 [a], 70–96	75	13
CL 312622	Alfalfa (forage)	0.1–10	8	77–91	86	5.6
	Alfalfa (hay)	0.1–10	8	72–84	80	5.0
	Alfalfa (seeds)	0.1–10	8	75 [a]–85	78	4.0

Recovery test was conducted with two tests per each of four fortification levels (0.1, 0.2, 0.5 and 10 mg/kg).
[a] At a fortification level of 10 mg/kg

This method was used in the residue trials for soya bean and alfalfa with acceptable procedural recoveries and LOQ levels.

M 3098

An independent laboratory validation study of the method M 3098 for the determination of residues of CL 299263 and CL 263284 in wheat hay, forage, straw and grain was conducted. Imazamox and CL 263284 were extracted from wheat forage, hay, straw and grain samples with acidic water-

methanol. Samples were further purified using solid phase extraction (SPE) and liquid-liquid partitioning techniques. Measurement of imazamox and CL 263284 residues was accomplished by capillary electrophoresis (CE) using a high sensitivity- flow cell and a ultra-violet absorbance detector set at 240 nm. In all matrices tested, the recovery values were between 69% and 97%. The LOQ of each analyte was 0.05 mg/kg in wheat matrices [Xu and Nejad 1999a, ID-244-026].

Table 49 Recovery results of imazamox and CL 263284 from wheat using the method M 3098 (independent laboratory validation study)

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Range of recovery (%)	Average recovery (%)	RSD (%)
Imazamox	Wheat (forage)	0.05, 0.50	4	87–93	90	2.8
	Wheat (hay)	0.05, 0.50	4	75–89	82	7.8
	Wheat (straw)	0.05, 0.50	4	69–89	80	12
	Wheat (grain)	0.05, 0.50	4	72–87	82	8.8
CL 263284	Wheat (forage)	0.05, 0.50	4	85–88	85	2.5
	Wheat (hay)	0.05, 0.50	4	71–97	79	9.8
	Wheat (straw)	0.05, 0.50	4	74–88	81	7.5
	Wheat (grain)	0.05, 0.50	4	72–83	79	6.5

Recovery test was conducted with two tests per each of two fortification levels.

This method was used in residue trials for wheat with acceptable procedural recoveries and LOQ levels.

M 2248, M 2333 (Confirmatory method for M 2248)

M 2248: Imazamox (CL 299263) was extracted from soya bean seeds with acidic aqueous methanol using a Polytron ultrasonic extractor. The extract was cleaned up by liquid/liquid partitioning with methylene chloride, gel permeation chromatography using a Biobead S-X3 column and solid phase extraction using an SCX column. Measurement of the residue was performed by HPLC-UV at 254 nm (HPLC-UV). The mean recovery values were between 82% and 88% (RSD, < 11%). The LOQ was 0.05 mg/kg for all matrices tested [Witkonton 1994b, 1994/7003428; Witkonton 1993a, ID-244-002].

Soya bean seed samples showing positive (> 0.05 mg/kg) residues of imazamox by method M 2248 above were confirmed by LC-MS (method M 2333). The two same extracts prepared in method M 2248 were directly amenable to analysis by LC-MS. The $(M+H)^+$ ion of imazamox at m/z 306^+ was monitored for confirmation [Witkonton *et al*., 1994a, ID-244-003]. The recovery values were 76% and 79% and the LOQ was 0.05 mg/kg.

Table 50 Recovery results of imazamox from soya bean using the method M 2248 and M 2333

Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)	Reference
Soya bean (seeds)	0.05	3	88	6.8	ID-244-002 M 2248
	0.10	3	82	11	
	0.50	3	86	10	
	0.05	2	78 (mean of 76 and 79)		ID-244-003 M 2333

M 2248.01

Imazamox (CL 299263) was extracted from the samples (soya bean seed/foliage and peanut/peanut foliage) with acidic aqueous methanol using a Polytron ultrasonic extractor. The extract was cleaned up by liquid/liquid partitioning with methylene chloride and solid phase extraction using an SCX column. Measurement of the residue was performed by HPLC-UV at 254 nm. There was a major modification to the method M 2248.01: exclusion of a sample clean up procedure by gel-permeation chromatography (GPC). The mean recovery values were between 81–115%. The LOQ was

0.05 mg/kg for each matrix [[Minoura and Ohba 1996a, ID-244-007; Minoura and Ohba 1996b, ID-244-008; Minoura and Ohba 1996c, ID-244-009; Minoura and Ohba 1996d, ID-244-010].

Table 51 Recovery results of imazamox from soya bean using the method M 2248.01

Crop	Fortification level (mg/kg)	No. of tests	Range of recovery (%)	Average recovery (%)	RSD (%)	Reference
Soya bean (seeds)	0.05, 0.2	4	100.3–112.4	105.0	5.0	ID-244-007
Soya bean (foliage)	0.05, 0.1	4	87.2–115.4	101.4	11	ID-244-008
Peanut (nut in shell)	0.05, 0.2	4	90.0–109.9	101.8	9.5	ID-244-009
Peanut (foliage)	0.05, 0.1	4	80.6–104.7	94.9	12	ID-244-010

Recovery test was conducted with two tests per each of two fortification levels

An independent laboratory validation study of method M 2248.01 for the determination of imazamox residues in soya bean seed was performed. GPC clean up procedure was included in this study. The recovery values were 75% between 90% and the LOQ (0.05 mg/kg) for soya bean seed was confirmed [Witkonton 1994a, ID-244-004].

Table 66 Recovery results of imazamox from soya bean seed using the method M 2248.01 (independent laboratory validation study)

Crop	Fortification level (mg/kg)	No. of tests	Recovery (%)
Soya bean (seeds)	0.05	2	75, 81
	0.10	2	76, 86
	0.40	2	88, 90

In the field residue trials for peanut, this method was used with acceptable procedural recoveries and LOQ levels.

ABC Report 46324

Imazamox and metabolites CL 263284 and CL 312622 were extracted from sunflower seed and meal samples with an acidified methanol/water solution, filtered and concentrated. The residues were purified on a C_{18} SPE column eluted with methanol in aqueous ammonium acetate. Residues in refined oil samples were diluted in hexane and extracted by shaking with acidified acetonitrile/water solution. Following phase separation, the lower aqueous acetonitrile layer was collected. The final chromatography analysis of imazamox residues was performed using LC-MS. The mean recovery values were between 75–109% (RSD, < 13%). The LOQ for each analyte was 0.05 mg/kg in sunflower matrices [Johnston 2003a, 2002/500411].

Table 52 Recovery results of imazamox, CL 263284 and CL 312622 from sunflower using the method ABC Report 46324

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
Imazamox	Sunflower (seed)	0.05	3	92	3.3
		0.1	3	90.1	0.8
		0.5	3	92.7	0.5
	Sunflower (refined oil)	0.05	3	94.5	0.7
		0.1	3	96.5	0.8
		0.5	3	107	0.5
	Sunflower (meal)	0.05	3	102	5.1
		0.1	3	87.7	1.9
		0.5	3	79.8	3.5
CL 263284	Sunflower (seed)	0.05	3	90.7	6
		0.1	3	87.9	1.4
		0.5	3	92	0.3
	Sunflower (refined oil)	0.05	3	97.3	0.9
		0.1	3	96.8	0.3

Test Substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)	RSD (%)
		0.5	3	108	1.1
	Sunflower (meal)	0.05	3	89.8	5.9
		0.1	3	82	2.9
		0.5	3	79.6	3.7
CL 312622	Sunflower (seed)	0.05	3	95.4	8.9
		0.1	3	77.8	2.3
		0.5	3	74.6	0.7
	Sunflower (refined oil)	0.05	3	93.6	1.6
		0.1	3	97.3	0.5
		0.5	3	109	0.9
	Sunflower (meal)	0.05	3	88.2	10
		0.1	3	81.2	1
		0.5	3	75.3	5.3

SOP-PA.0288

Imazamox and its metabolites CL 263284 and CL 189215 were extracted from sunflower samples using extraction solution methanol/water/1 M HCl (60:39:l, v/v/v). Final determination was performed using LC-MS/MS determination. The mean recovery values were between 76% and 106% (RSD, < 15%). The LOQ for each analyte was 0.01 mg/kg in sunflower seed [Freitas 2010a, 2010/1057139; Leite 2008a, 2010/1090461]

Table 53 Recovery results of imazamox, CL 263284 and CL 189215 from sunflower using the method SOP-PA.0288

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery (%)		RSD (%)	
Transition				1	2	1	2
Imazamox	Sunflower (seeds)	0.01	6	85	76	8	5
		1.0	5	100	103	4	6
Transition				1	2	1	2
CL 263284	Sunflower (seeds)	0.01	5	82	82	7	9
		1.0	6	106	101	6	8
Transition				1	2	1	2
CL 189215	Sunflower (seeds)	0.01	6	98	94	15	11
		1.0	6	105	106	5	6

Transition 1 (for quantification): imazamox, 306→246 m/z; CL 263284, 292→247m/z; CL 189215, 454→179 m/z

Transition 2 (for confirmation): imazamox, 306→193 m/z; CL 263284, 292→179 m/z; CL 189215, 454→292 m/z

Analytical methods for animal matrices

D0303

Imazamox and CL 263284 were extracted from bovine tissues (liver, kidney and muscle) with acidic methanol-water solution. Following centrifugation, an aliquot of the extract was diluted with methanol and then passed through an SCX cartridge, which retained the analytes. The SCX cartridge was washed with methanol to remove co-extractives; then the residues were selectively eluted with a water-methanol solution. The eluate was evaporated to dryness and the residues were dissolved in water for analysis. Residues from milk and fat were extracted with acetonitrile in hexane. Following solvent partitioning, an aliquot of the acetonitrile extract was diluted with methanol and passed through the SCX cartridge using the same procedure as described for the tissue samples. The final detection was accomplished by LC-MS/MS. In all matrices tested, the mean recovery values were

between 69% and 121% (RSD, 25%). The LOQ for each analyte was 0.01 mg/kg for bovine matrices [Stewart 2003a, 2003/5000116; Gooding 2013a, 2013/7002842].

Table 54 Recovery results of imazamox and CL 263284 from bovine matrices using the method D0303

Test substance	Crop	Fortification level (mg/kg)	No. of tests	Average recovery [a] (%)	RSD (%)
Imazamox	Liver	0.01	5	92	25
		0.10	5	93	9
	Kidney	0.01	4	82	7
		0.10	5	73	7
	Muscle	0.01	5	81	6
		0.10	5	87	7
	Fat	0.01	5	95	9
		0.10	4	69	7
	Milk	0.01	5	103	17
		0.10	5	86	8
CL 263284	Liver	0.01	5	116	16
		0.10	5	102	10
	Kidney	0.01	4	106	10
		0.10	5	78	8
	Muscle	0.01	5	98	17
		0.10	5	83	17
	Fat	0.01	5	121 (110, 114, 116, 122 and 144%)	12
		0.10	4	79	19
	Milk	0.01	5	93	6
		0.10	5	93	4

[a] Recoveries have been corrected for mean recoveries in control samples. The detected residue concentrations in two control samples were: for imazamox, 0.004 and 0.005 mg/kg in liver, 0.005 and 0.003 mg/kg in kidney, 0.004 and 0.006 mg/kg in muscle, 0.004 and 0.003 mg/kg in fat, and 0.004 and 0.000 mg/kg in milk; for CL 263284, 0.000 and 0.003 mg/kg in liver.

Another validation study for the method D0303 was performed for bovine matrices and egg commodity. In all matrices tested, the mean recovery values were between 70% and 111% (RSD, < 22%).

Table 55 Recovery results of imazamox and CL 263284 from bovine matrices and egg using the method D0303

Test Substance	Crop	Fortification level (mg/kg)	No. of Tests	Recovery range (%)	Average recovery (%)	RSD (%)	No. of tests	Recovery range (%)	Average recovery (%)	RSD (%)
Transition				1				2		
Imazamox	Muscle	0.01	5	71–87	79	9	5	67, 68, 70–84	72	10
		0.1	5	77–84	81	4	5	75–78	77	1
	Kidney	0.01	5	73–90	78	9	5	71–82	76	6
		0.1	5	75–82	79	4	5	70–85	77	9
	Liver	0.01	5	65, 90–107	90	17	5	62, 77–93	78	14
		0.1	5	71–106	89	15	5	68, 81–94	85	13
	Fat	0.01	5	100–117, 126	110	10	5	89–108, 125	105	13
		0.1	5	88–94	89	5	5	82–94	89	5
	Milk	0.01	5	89–109. 126	107	12	5	92–120	106	13
		0.1	5	81–120	93	17	5	84–105	93	9
	Egg	0.01	5	101–111	107	4	5	97–111	104	5
		0.1	5	83–96	89	6	5	79–99	87	9
Transition				1					2	
CL 263284	Muscle	0.01	5	69, 72–86	77	9	5	71–84	80	7

Test Substance	Crop	Fortification level (mg/kg)	No. of Tests	Recovery range (%)	Average recovery (%)	RSD (%)	No. of tests	Recovery range (%)	Average recovery (%)	RSD (%)
		0.1	5	77–88	82	6	5	75–79	77	2
	Kidney	0.01	5	72–82	76	6	5	71–84	77	7
		0.1	5	80–82	81	2	5	79–85	81	3
	Liver	0.01	5	63, 75–103	80	18	5	54, 66, 66, 68, 95	70	22
		0.1	5	73–103	92	13	5	95–101	90	14
	Fat	0.01	5	91–103	96	6	5	89–108	97	7
		0.1	5	84–91	87	3	5	83–90	86	3
	Milk	0.01	5	96–118	108	10	5	101–115, 134	111	12
		0.1	5	72–88	82	8	5	77–97	86	8
	Egg	0.01	5	87–117	100	13	5	92–111, 127	106	13
		0.1	5	93–112	99	9	5	88–115	99	10

Transition 1 used for quantification: imazamox, 306→261 m/z; CL 263284, 292→247m/z
Transition 2 used for confirmation: imazamox, 306→86 m/z; CL 263284, 292→179 m/z

M 2503, M 2463, SOP CEM-236/002, M 2379 and M 3178.01

The methods used for storage stability studies are summarized briefly below.

M 2503:The samples were analysed by method M 2503 which determines the analytes by means of CE-UV. Residues of a soya bean sample aliquot were extracted with acidic water methanol. After filtration of the samples, one aliquot was subjected to solid phase extraction and liquid-liquid portioning techniques. The final quantitation was performed by CE-UV (240 nm). The method has a limit of quantitation of 0.5 mg/kg for each compound in soya bean.

M 2463: The samples were analysed by Method M 2463 which determines the analytes by means of CE-UV. The validated LOQ of the method is 0.1 mg/kg per compound. Residues of the test compounds were extracted with acidic water-methanol followed by clean-up steps which included precipitation, centrifugation and solid-phase extraction techniques. Measurement of CL 263284 was accomplished by capillary electrophoresis (CE) equipped with a high sensitivity flow cell and a UV detector set at 240 nm.

SOP CEM-236/002: The samples were analysed by CEMAS SOP CEM-236/002 which determines the analytes by means of HPLC-UV. Residues of imazamox and CL 263284 were extracted by maceration with extraction solution (methanol/water/1 M hydrochloric acid). After filtration, extracts were re-suspended in methanol and hydrochloric acid before they were partitioned into dichloromethane. After evaporation to dryness, the residue was reconstituted in methanol and acetonitrile before being extracted into hexane. The hexane layer was evaporated to dryness and the residuum was re-dissolved into 0.2% hydrochloric acid. The extracts were cleaned up by solid phase extraction. The quantitation of residues was performed by HPLC-UV at 254 nm. The method has a limit of quantitation of 0.05 mg/kg for each compound in maize.

M 2379: The samples were analysed by Method M 2379 which determines the analytes by means of CE-UV. Residues of the test compounds were extracted with 1 N HCl/water/methanol solution (1:39:60, v/v/v).

M 3178.01: The samples were analysed by Method M 3178.01 which determines the analytes by means of HPLC-MS. The validated LOQ of the method is 0.1 mg/kg per compound. Residues of the test compounds were extracted with acetone/water/methanol solution (1:1:1, v/v/v) and purified by SPE techniques. Measurement of imazamox, CL 263284, CL 189215 and CL 312622 was accomplished by LC-MS.

RLA 12539, HPLC-MSD and CEM-236

The methods used for determination of incurred residues in supervised field trial samples are summarized briefly below.

RLA 12539 (wheat, ID-730-019): The whole wheat specimens were analysed for residues of imazamox using method RLA 12539 (CE-UV). The validated limit of quantitation was 0.05 mg/kg. The samples were extracted with methanol:water:HCl 1 mol/L (60:39:1, v/v/v). After clean-up by solid phase extraction, the specimen was partitioned with dichloromethane and evaporated to dryness and reconstituted in water. The final determination was performed with CE-UV at 240 nm. Mean recovery of imazamox from fortified untreated wheat whole plant, wheat grain and wheat straw, over the range 0.05 to 1.00 mg/kg, was 87%, 88% and 84%, respectively. Mean recovery of CL 263284 from fortified untreated wheat whole plant, wheat grain and wheat straw, over the range 0.05 to 1.00 mg/kg, was 81%, 77%, and 76%, respectively.

HPLC-MSD method (sunflower, sunflower seed processing, 2002/5004111): Residues of imazamox were extracted from sunflower seed and meal samples with an acidified methanol:water solution, filtered and concentrated. The residues are purified on a C_{18} SEP 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 acetonitrile layer is collected and adjusted to final volume with water. The final analysis of imazamox residues is performed using LC-MSD. At fortification levels of 0.05, 0.1 and 0.5 mg/kg for sunflower seed and 0.05 mg/kg for refined oil and meal, recoveries were in the range of 70.8% and 113% (RSDs, 2–13%).

CEM-236 (alfalfa, ID-731-001): The specimens were analysed for imazamox and its metabolite CL 263284 with method CEM-236 quantifying each relevant analyte with a limit of quantitation of 0.05 mg/kg. For the analysis, alfalfa samples (green matter and hay) were extracted with an acidified methanol/water mixture. After clean-up of the extract by liquid/liquid partition and solid phase extraction residues were determined by UV detection after liquid chromatographic separation (HPLC-UV). In whole plant samples fortified at 0.05 mg/kg with imazamox and CL 263284 each, recovery was 82% and 72%, respectively.

Stability of residues in stored analytical samples

The Meeting received information on freezer storage stability of imazamox and its metabolites in plant commodities. Storage stability study on animal commodity was not submitted. Residues in plant commodities were generally stable for the duration of the studies.

Frozen samples were homogenized and fortified with test compound. The fortified homogenate was stored in deep freezer. After each specified period, a portion of sample was analysed for test compound. The stability results are expressed as average percentage of the nominal fortification and are not corrected for the procedural recoveries. In order to account for possible variations over the time investigated, the mean procedural recovery results are additionally given. The storage conditions, storage periods and percent remaining after each period were summarized in the table below.

Table 56 Stability of imazamox and its metabolites in frozen plant matrices

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
Soya bean matrices fortified at 0.1 mg/kg, stored at –20 °C and analysed with Method SOP-PA.0288 [Leite and Alves 2011(a), 2011/1207286]									
Grain	0			107	–	107	–		
	1			101	103	94	108		
	2			92	99	88	106		
	3			104	103	99	107		
	7			75	89	94	103		
	10			111	123	100	124		

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
Laminated bean	0			95	–	90			
	1			98	108	86	111		
	2			–	–	–	–		
	3			93	104	99	89		
	7			–	–	–	–		
	10			–	–	–	–		
Defatted meal	0			101	–	92	–		
	1			105	107	85	126		
	2			–	–	–	–		
	3			107	97	97	100		
	7			–	–	–	–		
	10			–	–	–	–		
Toasted defatted meal	0			114	–	98	–		
	1			112	104	88	126		
	2			–	–	–	–		
	3			96	103	111	90		
	7			–	–	–	–		
	10			–	–	–	–		
Oil	0			111	–	106	–		
	1			86	102	83	100		
	2			–	–	–	–		
	3			100	110	93	116		
	7			–	–	–	–		
	10			–	–	–	–		
Soya bean matrices fortified at 0.5 mg/kg, stored at ≤ –15 °C and analysed with method M 2503 [Bixler and Safarpour 2000(a), ID-720-070]									
Seed	32	119	78	80	74				
	35	134	87	91	79				
	38	124	90	86	85				
	44	124	88	80	79				
Forage	32	91	79	61	74				
	35	109	89	73	74				
	38	105	90	70	81				
	44	100	85	66	77				
Hay	32	92	87	57	75				
	35	112	93	64	79				
	38	106	90	69	83				
	44	107	86	73	84				
Wheat matrices fortified at 0.5 mg/kg, stored at ≤ –15 °C and analysed with method M 3098 Bibo X. 2002(a), 2002/5004279].									
Grain	0	82	95	71	84				
	3	101	102	83	82				
	6	108	103	97	85				
	12	109	100	89	88				
	18	98	92	81	75				
	24	110	108	98	94				
	37	87	92	88	84				
	48	84	84	68	72				
Straw	0	83	87	70	75				
	3	92	94	78	74				
	6	95	93	81	82				
	12	99	92	79	75				
	18	76	88	62	73				
	24	91	99	81	89				
	37	80	98	84	85				
	48	81	82	61	66				
Hay	0	88)	93	78	80				
	3	95	90	77	78				

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
	6	75	91	62	82				
	12	76	98	61	85				
	18	64 (65)	98	57	82				
	24	93	95	76	92				
	37	74	88	70	79				
	48	65 (80)	81	57	75				
Forage	0	80	86	70	77				
	3	90	94	76	71				
	6	90	108	75	85				
	12	88	93	75	83				
	18	79	90	69	82				
	24	88	106	82	91				
	37	91	99	82	95				
	48	72	83	62	68				
Wheat matrices fortified at 0.5 mg/kg, stored at ≤ –10 °C and analysed with method M 2463 [Nejad 1999(a), IA-730-011].									
Grain	14			91	91	83	90		
	18			102	104	82	98		
	24			112	106	85	103		
Straw	14			92	91	79	78		
	18			93	98	80	89		
	24			92	92	87	77		
Hay	14			82	92	83	92		
	18			75	89	77	95		
	24			84	108	88	93		
Forage	14			79	89	79	85		
	18			97	87	84	95		
	24			70	87	71	90		
Maize matrices fortified at 0.5 mg/kg, stored at ≤ –18 °C and analysed with method SOP CEM-236/002 [Rawle 2003(a), 2003/1030079]									
Grain	0	86	82	75	72				
	3	85	82	75	71				
	6	80	78	74	76				
	12	71	75	71	71				
	18	87	89	83	90				
	24	83	88	79	92				
Ear	0	86	89	77	75				
	3	84	90	75	88				
	6	95	93	92	93				
	12	69	75	64	74				
	18	79	89	80	88				
	24	86	94	80	90				
Immature plant	0	80	100	75	87				
	3	83	72	77	76				
	6	86	78	81	76				
	12	85	81	80	74				
	18	80	76	77	78				
	24	87	87	80	87				
Rape seed fortified at 0.5 mg/kg, stored at ≤ –18 °C and analysed with method M 2460 [Bixler and Khunachak 2000 (a), ID-326-022]									
Rape seed	0	74	97						
	3	72	99						
	6	64	87						
	7.5	75	72						
	12	87	108						
	18	67	99						
Peanut matrices fortified at 1 mg/kg, stored at –5 to –25 °C and analysed with method M 2379 [Nejad and Xu 2000 (a), IA-740-023]									
Hull	1			87	78	81	78		
	5			122	113	84	79		
	6			86	88	97	96		

Matrix	Storage period (month)	Imazamox % remaining	Proc. rec.	CL 263284 % remaining	Proc. rec.	CL 189215 % remaining	Proc. rec.	CL 312622 % remaining	Proc. rec.
	12			81	84	84	86		
	18			68	85	74	85		
	24			83	93	83	96		
Nutmeat	1			87	98	80	87		
	5			83	92	84	91		
	6			76	85	76	86		
	12			75	93	78	96		
	18			62	84	66	83		
	24			85	81	82	77		
Alfalfa matrices fortified at 1.0 mg/kg, stored at –10 to –35 °C and analysed with method M 3178.01 [Fletcher 2001 (a), ID-326-024]									
Hay	0	79	81	76		69	87	70	68
	1	76	89	76		70	72	65	93
	3	71	94	68		65	78	67	86
	6	70	81	69		60	64	69	83
	12	75	85	82		63	98	79	93
	18	75	92	84		80	91	101	93
Forage	0	59	67	67	82	66	77	81	85
	1	78	85	82	88	80	81	83	94
	3	74	82	75	86	78	82	81	87
	6	78	80	75	74	66	80	77	79
	12	75	81	73	78	68	71	72	84
	18	76	87	78	91	75	75	76	84
Seed	0	58	59	76	78	55	80	71	82
	1	65	72	76	82	61	80	68	79
	3	68	83	79	87	57	81	66	88
	6	74	79	79	87	61	68	75	79
	12	76	90	63	85	59	88	77	91
	18	84	89	74	72	61	63	77	82

Stability of residues in stored analytical samples was investigated for imazamox, CL 263284, CL 189215 and CL 312622.

In acidic methanol water extract and final volume solution (for LC-MS/MS), the four compounds were stable for 3 days and 7 days, respectively in the matrices of rice (whole plant, grain and straw), grapes and sunflower seeds at refrigeration conditions [Lehmann 2013a, 2012/1294678; Lehmann 2013b, 2013/1177533].

Stability of calibration solutions prepared with 1% acetic acid in water was tested at 4 °C, where the four compounds were stable for 27 days [Jordan 2003a, 2002/5004302]. The mixed fortification standard solution in 10% methanol/water was stable up to one month at 4 °C with no more than 8% difference between the response of the one month old and the new fortification solution [Wickremesinhe and Safarpour 1998a, ID-244-022].

Imazamox and CL 263284 in methanol or acidified water (calibration solution) were stable for 56 days and 28 days, respectively when stored under refrigeration [Stewart 2003a, 2003/5000116; Gooding 2013a, 2013/7002842]. In the study, extracts of livestock matrices in acidic methanol-water were stable for the time period tested 2 to 6 days.

USE PATTERNS

Imazamox is a systemic herbicide used for the control of grassy and broadleaf weeds. It is formulated as a liquid or granular product either a solo product or in combination with other active substances for use on legume vegetables, pulses, cereal grains, oilseeds and legume animal feeds. It is registered in a number of counties for crops with pre-emergence and post-emergence applications.

The authorized uses relevant to the supervised trials data submitted to the current Meeting and described in English are summarized in the table below.

Table 57 Registered use of imazamox relevant to the residue evaluation by the current Meeting

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Bean	Brazil	WG 700 g/kg	Post-emergence	1	30–50 100–300	0.028–0.042	43	Add non-ionic surfactant in 0.25% v/v of mixture.
Bean(dry)	Canada	WG 700 g/kg	Early post-emergence BBCH 12–18	1	100	0.020	60	Apply with a nitrogen source and EC surfactant (0.5%, v/v).
Bean (dry)	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence	1	100	0.020	60	Apply with a nitrogen source. Do not graze the treated crop or cut for hay.
Bean	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Bean (common)	Costa Rica	WG 700 g/kg	Early post-emergence	1	200–300	0.040	43	Add a non-ionic surfactant at 0.25%.
Bean (Winter and spring Faba)	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence (within 5 days after sowing); pre-emergence of the weeds	1 (max every 2 yrs)	100–500	0.075	90	Applied on the soil. Do not roll after treatment.
Bean (Green)	Israel	EC 40 g/L	2–3 leaves			0.024		
Bean (Spring and winter field)	UK	EC 16.7 g/L (pendimethalin, 250 g/L)	Before weed emergence after sowing (pre-emergence of the crop)	1	200–300	0.075		Do not apply once the plumule is less than 13 mm from the soil surface.
Bean (Lima , succulent)	USA	SL 120 g/L as free acid (ammonium sulphate)	Early post-emergence (1^{st}-2^{nd} trifoliate leaf stage)	1		0.035		A non-ionic surfactant must be added to the spray solution.
Bean, snap	USA	SL 120 g/L as free acid (amm. sulphate)	Post-emergence (at least 1 fully expanded trifoliate leaf and before the bloom stage)	1		0.035		A non-ionic surfactant must be added to the spray solution.
Bean (dry)	USA	SL 120 g/L as free acid (amm. sulfate)	Post-emergence (prior to bloom stage but after dry beans have at least 3 pairs of leaves)			0.035		An adjuvant (a surfactant or a crop oil concentrate) must be added to the spray solution.
Lentil (Clearfield)	Canada	WG 700 g/kg	Early post-emergence (2–6 leaf stage of the crop) BBCH 12–18	1	100	0.015–0.020	60	Apply with EC surfactant (0.5% v/v). Do not graze the treated crop or cut for hay within 20 days of application.
Lentil (Clearfield)	Canada	WG 35% (imazethapyr, 35%)	Early post-emergence (the 2–6 leaf stage of the crop)	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Do not harvest forage or cut hay for forage.

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Lentil (Clearfield)	Canada	SL 33 g/L (imazapyr, 15 g/L)	Early post-emergence (1–9 node stage) BBCH 12–17	1	50–100	0.020	60	Apply with EC surfactant (0.5% v/v).
Lentil	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence post-sowing of the crop	1	100–500	0.037	63	
Lentil (Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence (2–leaf to prior to flower bud formation)	1		0.035–0.053		A non-ionic surfactant and nitrogen-based fertilizer must be added to the spray solution.
Pea (Field)	AUS	WG 700 g/kg	Early post-emergence	1	min 50	0.032	not req.	WHP: 6 weeks (grazing). Spray adjuvant and liquid ammonium sulfate should be added.
Pea (Succulent shelled peas)	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence (3 to 6 node stage)	1	100	0.020	40	
Pea (Field)	Canada	WG 350 g/kg (imazethapyr, 35%)	BBCH 11–16	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Field peas treated may be fed to livestock 30 days after application. Do not harvest forage or cut hay for forage.
Pea (Succulent)	Canada	WG 700 g/kg	Early post-emergence (3–6 node stage)	1	100	0.020	40	Must be applied in tank mix combination with bentazone (429 g/ha) and nitrogen source (2 L/ha). Do not graze the treated crop or cut for hay.
Pea (Field)	Canada	WG 700 g/kg	Early post-emergence (3–6 node stage)	1	100	0.020	60	The same with the above
Pea (Field)	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence (3–6 node stage)	1	100	0.020	60	Apply with a nitrogen source. Do not graze the treated crops or cut for hay.
Pea	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		–
Pea (canning)	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence (within 3–4 days after sowing)	1 (max every 2 years)	100–500	0.075	63	Applied on the soil. Do not roll after treatment.
Pea (Winter and spring protein)	France	EC 16.7 g/L (pendimethalin, 250 g/L)	Pre-emergence post-sowing (preferred) or post-emergence (2–3 leaf stage of the culture) BBCH 12–13	1 (max every 2 years)	100–500	0.075	63	
Pea (Combining and vining pea)	UK, Ireland	EC 16.7 g/L	Before weed emergence after	1	200–300	0.075		Do not apply once the plumule is less than 13

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
		(pendimethalin, 250 g/L)	sowing					mm from the soil surface.
Pea	Israel	EC 40 g/L	2–3 leaves			0.024		
Pea (English)	USA	SL 120 g/L as free acid (amm. sulfate)	Early post-emergence	1		0.026		A non-ionic surfactant must be added to the spray solution.
Pea (dry) (field pea, sourthern pea (cow pea))	USA	SL 120 g/L as free acid (amm. sulfate)	Early post-emergence prior to bloom stage but after dry peas have at leaves 3 pairs of leaves	1		0.035		A non-ionic surfactant (or crop oil concentrate) must be added to the spray solution.
Peanut	AUS	WG 700 g/kg	Early post-emergence	1	min 50	0.035		WHP: 4 weeks (grazing) A spray adjuvant and liquid ammonium sulfate should be used. Do not graze or cut for stock food for 4 weeks after application.
Peanut	Israel	EC 40 g/L	3–4 true leaves			0.032		
Peanut	South Africa	SL 120 g/L	Post-emergence	1	200	0.048	50	This must only be used with a non-ionic surfactant (0.2% v/v) and ammonium sulfate solution.
Peanut	Zimbabwe	SL 120 g/L	Early post-emergence	1	200	0.036		Add an adjuvant and ammonium sulfate solution. Do not graze the treated crops or cut for hay within 20 days of application.
Rape (Clearfield winter rape)	UK	SC 17.5 g/L (metazachlor, 375 g/L)	Post-emergence BBCH 10–18	1	100	0.035		EC spreader may be used.
Rape seed (Clearfield canola)	AUS	SL 33 g/L (imazapyr, 15 g/L)	Early post-emergence (2–6 leaf stage) BBCH 12–16	1	Min 70–100	0.0099–0.025		Do not graze or cut for stock food for 5 weeks after application.
Rape (Clearfield canola, Clearfield canola quality *Brassica juncea*))	Canada	SL 33 g/L (imazapyr, 15 g/L)	Early post-emergence (2–6 leaf stage)	1	50–100	0.020	60	Apply with EC surfactant (0.5% v/v).
Rape (Clearfield canola, Clearfield canola quality *Brassica juncea*)	Canada	WG 350 g/kg (imazethapyr, 35%)	Early post-emergence BBCH 12–16	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Do not graze treated crops or cut for hay. Do not harvest forage or cut for hay.
Rape (Clearfield canola, Clearfield canola quality *Brassica juncea*)	Canada	WG 700 g/kg	Early post-emergence BBCH 12–16	1	100	0.020	60	Apply with EC surfactant (0.5% v/v). Do not graze treated crops or cut for hay within 20 days of

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
								application.
Rape (Clearfield)	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Rape (Clearfield canola)	South Africa	SL 40 g/L	Post-emergence (after the 5-leaf stage)	1	200	0.048	30	
Rape (Clearfield canola)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence	1		0.035		An adjuvant and a nitrogen fertilizer must be added to the spray solution.
Rice (Red, Clearfield)	COL	SL 33 g/L (imazapyr 15 g/L)	Early post-emergence; 1st application on red rice with 3 leaves	2 Interval, 8–10 day	min 150	0.050	45	Apply always among co-adjuvant (spreader penetrant).
Rice (Red, Clearfield)	COL	WG 700 g/kg	Early post-emergence; 1st application on red rice with 3 leaves	2 Interval, 8–10	min 150	0.098	45	Apply always it among silicone co-adjuvants 1%.
Rice (Asian, Clearfield)	Costa Rica	WG 700 g/kg	Post-emergence (two leaf stage of the crop) BBCH 12–14	2 Interval, 14 day	200–300	0.070	50	Add EC spreader 37.5 (methyl oleate/palmitate).
Rice (Asian, Clearfield)	DOM	WG 700 g/kg	Early post-emergence; BBCH 12–14 (1st)	2 Interval, 14 day	200–300	0.081	50	Add EC spreader 37.5 (methyl oleate/palmitate).
Rice (Clearfield)	Greece	SL 40 g/L as free acid (amm. salt)	Post-emergence (two leaf stage of the crop) BBCH 12–22	1	200–400	0.050		Add EC spreader.
Rice (Clearfield)	Greece	SL 40 g/L as free acid (amm. salt)	BBCH 11–14 3–4 leaves at 1st appl.; BBCH 15–22 at 2nd appl.	2 Interval, 21 day	200–400	0.035		Add EC spreader.
Rice (Clearfield)	Spain	SL 40 g/L	Post-emergence	2 Interval, 14–21 days	200–300	0.035		Rice plant in water or dry planted rice
Rice (Clearfield, Clearfield rice hybrids)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence (4-leaf rice to rice panicle initiation plus 14 days for Clearfield rice; 4-leaf rice up to rice panicle initiation for Clearfield rice hybrids)	1 or 2		0.035–0.053 (0.087 /season) G or A		For water-seeded and dry/drill-seeded rice. Do not apply to Clearfield rice hybrids after panicle initiation. A crop oil concentrate must be added to the spray solution (1% v/v).
Soya bean	AUS	WG 700 g/kg	Post-emergence	1	min 50	0.035		WHP: 4 weeks (grazing) A spray adjuvant and liquid ammonium sulphate should be used. Do not graze or cut for stock food for 4 weeks after application.

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Soya bean	Brazil	WG 700 g/kg	Post-emergence	1	30–50 in aerial appl; 100–300 in ground appl	0.042–0.049	70	Use non-ionic adjuvant at 0.25% v/v of mixture. Aerial spraying
Soya bean	Canada	WG 700 g/kg	BBCH 10–14	1	200	0.025	60	Apply with a non-ionic surfactant at a rate of 0.25% v/v and a fertilizer solution. Do not graze the treated crops or cut for hay within 20 days of application.
Soya bean	Canada	SL 20 g/L (bentazone, 429 g/L)	Early post-emergence (cotyledon to 4-leaf stage)	1	100	0.020	60	Apply with a nitrogen source. Do not graze the treated crops or cut for hay within 20 days of application.
Soya bean	Canada	WG 35% (imazethapyr, 35%)	BBCH 11–13 (1–3 true leaf stage after weeds have emerged)	1	50–100	0.015	85	Apply with EC surfactant (0.5% v/v). Do not graze the treated crops or cut for hay. Do not harvest forage or cut hay for forage.
Soya bean	China	SL 40 g/L	Early post-emergence	1	225–450	0.045–0.050		
Soya bean	Costa Rica	WG 700 g/kg	Early post-emergence	1	200–300	0.040	70	Add a non-ionic surfactant (0.25%).
Soya bean	France	SL 40 g/L	Post-emergence (1 trifoliate leaf up to 5–6 leaf stage of the culture BBCH 12–14	1	100–300	0.040–0.050	90	
Soya bean	France	SL 40 g/L	BBCH 12–14	2 Interval, 8–10 day	100–300	0.025	90	An adjuvant is used.
Soya bean	South Africa	SL 120 g/L	Post-emergence	1	min. 200	0.048	64	This must be used with a non-ionic surfactant (0.2% v/v). Grazing of crop 64 days after application.
Soya bean	USA	SL 120 g/L as free acid (amm. sulfate)	Early post-emergence; before bloom stage	1		0.035–0.045		
Soya bean	ZIM	SL 120 g/L	Early post-emergence in soya beans up to but before flowering	1	200	0.036		Add an adjuvant and ammonium sulphate to the spray solution. Do not graze the treated crops or cut for hay within 20 days of application.
Sunflower (Clearfield)	Canada	WG 700 g/kg	Early post-emergence (2–8 leaf)	1	100	0.015–0.020	70	Apply with EC surfactant (0.5% v/v). Do not graze treated

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
			BBCH 12–18					crop or cut for straw.
Sunflower (Clearfield)	Canada	WG 350 g/kg (imazethapyr, 35%)	Early post-emergence (2–8 leaves) BBCH 12–18	1	50–100	0.015	60	Apply with EC surfactant (0.5% v/v). Do not harvest forage or cut hay for forage.
Sunflower (Merigold, Clearfield)	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Sunflower (Clearfield)	France	SL 40 g/L	Post emergence (2–6 leaf) BBCH 12–18	1	100–300	0.050	90	Do not add any adjuvant at greater than 0.050 kg ai/ha. At fractionation, 0.025 kg ai/ha with adjuvant for each 1st and 2nd application.
Sunflower (Clearfield)	Greece	SL 40 g/L	Post-emergence BBCH 14–16	1	200–300	0.040		+ 0.5 L/ha of EC spreader
Sunflower (Clearfield)	South Africa	SL 33 g/L (imazapyr, 15 g/L)	2–6 leaf BBCH 12–16		150–250	0.033	70	
Sunflower (Clearfield)	Spain	SL 40 g/L	Post-emergence (4–8 pairs of leaves)	1		0.040		+ 0.5 L/ha of EC spreader
Sunflower (Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Early post-emergence (2–8 leaf) BBCH 12–18	1		0.035		A non-ionic surfactant and nitrogen-based fertilizer must be added to the spray solution.
Wheat (Clearfield)	ARG	WG 700 g/kg	Early post-emergence	1	100–200	0.049–0.070		Add a non-ionic surfactant of 0.25% of the main ingredient into the total volume.
Wheat (Clearfield Plus)	AUS	SL 33 g/L (amm. salt; imazapyr, 15 g/L)	Early post-emergence (3 leaf-1st node stage) BBCH 13–31,	1	min 70–100	0.012–0.025	not required	Do not graze or cut for stock food for 4 weeks after application. Do not use on CL STL and CL JNZ wheat variety.
Wheat (Clearfield Plus)	Canada	EC 20 g/L (2-4 D of 2-ethyl ester, 560 g/L)	Early post-emergence (3 to 6-leaf stage; prior to flag leaf emergence) BBCH 13–16	1	100	0.020	79	Apply with non-ionic surfactant at a rate of 0.25% v/v. Do not graze the treated crop within 4 days of application.
Wheat (Clearfield)	Canada	SC 120 g/L (amm. salt)	Early post-emergence (2–6 leaf) BBCH 12–16	1	50–100	0.015–0.020	79 (grain, straw)	Apply with non-ionic surfactant at a rate of 0.25% v/v. Do not graze the treated crop within 14 days of application or cut for hay within 42 days of application.
Wheat (Clearfield)	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056		
Wheat (Spring, Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Post-emergence (4 leaf to prior-to jointing)	1		0.13		A molded jug pack contains ammonium salt of imazamox and 2-ethylhexyl ester of MCPA (67.9%). A surfactant and nitrogen-

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
								based fertilizer must be added to the spray solution. Do not forage or graze meat animals on treated areas within 7 days of slaughter. Do not forage or graze dairy animals on treated areas within 7 days after treatment.
Wheat (Winter, Clearfield)	USA	SL 120 g/L as fee acid (amm. salt)	Post-emergence at tiller initiation but prior-to jointing	1		0.11–0.16		the same with remarks above
Wheat (Spring, Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Early post-emergence (4-leaf to prior-to jointing)	1		0.035–0.044		Add a non-ionic surfactant and nitrogen-based fertilizer. COC or MSO may be substituted for the non-ionic surfactant.
Wheat (Winter, Clearfield)	USA	SL 120 g/L as free acid (amm. salt)	Early post-emergence (after tiller initiation but prior to jointing)	1 or 2		0.035–0.053 (0.070 kg ai/ha/season)		Add a non-ionic surfactant and nitrogen-based fertilizer. COC or MSO may be substituted for the non-ionic surfactant in 2-gene winter wheat. There are no restrictions for feeding or grazing of wheat forage and hay.
Alfalfa (Lucerne)	AUS	WG 700 g/kg	Post-emergence (2 trifoliate leaf; apply as following cutting or grazing)	1	min 50	0.032–0.035		WHP: 7 days (grazing). Do not graze or cut for stock food for 7 days after application. A spray adjuvant and liquid ammonium sulfate should be added. Do not use the Hasten or Kwickin mix (oil adjuvant) on seedling lucerne.
Alfalfa	Chile	WG 700 g/kg	Early post-emergence	1	150–200	0.042–0.056	n.a.	
Alfalfa	France	EC [a] 16.7 g/L (pendimethalin, 250 g/L)	Post-emergence [b]	1	100–500	0.033 or 0.067 [b]	30	
Alfalfa (Medicago sativa)	Greece	SL 40 g/L (4.22% amm. salt)	[c]	1	300	0.050	28	+ 4 kg/ha ammonium sulfate
Alfalfa (established)	Saudi Arabia	SL 40 g/L	[d]	1	300	0.048 (newly sown) or 0.060 (established)	35	Addition of nitrogen fertilizer (urea) to spray solution at a rate of 2 kg/ha.

Crop	Country	Formulation	Application					Remarks
			Growth stage & season (crop)	No.	Water L/ha	Rate, kg ai/ha	PHI, days	
Alfalfa (Lucerne)	South Africa	SL 120 g/L	post-emergence	1	min. 200	0.036–0.048	22	This must only be used in a tank mixture with a non-ionic surfactant (0.2% v/v) and ammonium sulfate (2.0% v/v).
Alfalfa	South Africa	SL 40 g/L	Post-emergence	1 or 2	200	0.048	22	Two applications can be made on lucerne per growing season. If necessary, the 2nd application can be made immediately after any of the early to mid-season cuttings.
Alfalfa	Spain	SL 40 g/L	Early post-emergence (4 true leaves)			0.050		
Alfalfa	USA	SL 120 g/L as free acid (amm. sulfate)	[e]	1		0.035–0.053		

In Costa Rica, Guatemala, El Salvador, Nicaragua, and Panama, the same use is approved for common bean.

As well as UK, the same use for field bean is approved in Ireland.

In Cota Rica, Guatemala, El Salvador, Nicaragua, and Panama, the same use is approved for soya bean.

In Cota Rica and El Salvador, the same use is approved for Asian rice (Clearfield).

[a] Seed-bearing fodder legumes (violet clover, white clover and crimson clover and of seed-bearing alfalfa: a) on set-up cultures at 0.067 kg ai/ha, in a single application and making sure that the application is carried out at most every 2 years. B) on a young plant culture at 0.033 kg ai/ha, between the 2 leaf and 4–5 trifoliate leaf stages. A second 0.033 kg ai/ha application may prove to be useful in certain cases. Do not exceed a maximum of two applications every 2 years.

[b] Applied in post-emergence of the alfalfa: for young cultures, 0.033 kg ai/ha from the 2 trifoliate leaf stage of alfalfa; for cultures set up, a) end of autumn/beginning of winter: 0.033 kg ai/ha b) out of winter (resumption of vegetation growth): 0.033–0.067 kg ai/ha. If an application was carried out at 0.033 kg ai/ha at the end of autumn/beginning of winter, resumption is possible at the end of winter at a dosage of 0.033 kg ai/ha if this is necessary. Notes: Do not apply if the culture has been milled beforehand. At the end of the season between the last cut and the potential application, observe a min. 15 days period

[c] 1) New planting: one application after germination or after the 1st cut, when the crop has 2–5 leaves 2) Established planting (from 2nd year): one application during dormancy of the crop or after the 1st cut (as soon as the alfalfa acquires 2–5 new leaves)

[d] 1) Newly sown: 2–3 leaf stage 2) Established: after dormant season or 4–5 days after cutting

[e] Early post-emergence 1) Seedling: application in the 2nd trifoliate stage or larger. For alfalfa grown for seed, before bud formation. 2) Established: application in the fall, winter, or in the spring to dormant or semi dormant alfalfa, or between cuttings. Any application should be made before significant alfalfa growth or regrowth (3 inches).

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received residue data from supervised field trials conducted on legume vegetables, pulses, cereal grains, oilseed and animal feeds.

Application rates and residue concentrations were reported as imazamox. Residue concentrations were recorded unadjusted for recoveries or for residue values in control samples. Where multiple samples were taken from a single plot, the calculated mean concentration is reported and used for estimation of maximum residue level. When trials were conducted in the same location, with the same or similar varieties, same or similar formulations, and same equipment, and at the same or similar timing, they are not regarded as independent and only one result from these trials was chosen for the estimation of a maximum residue level.

Analytical methods used for determination of imazamox-related residues in field trial samples were well validated through procedural recovery tests or method validation conducted previously.

Concentration of the metabolite CL 263284 was expressed as parent equivalents using conversion factor of 1.048. When calculating sum of parent and CL 263284, for values below LOQ, the LOQ was used for each analyte.

In many of the following trials, plants resistant to imidazolinone pesticides were used. That is expressed as "CL" in their variety name, which means non-GM plants showing the tolerance due to the mutation(s) of endogenous AHAS gene from conventional breeding technique.

Commodity group	Commodity	Table No.
Legume vegetables	Beans	58
Pulses	Peas	59
	Beans (dry)	60
	Peas (dry)	61
	Lentil (dry), imidazolinone-tolerant	62
	Soya bean (dry)	63
Cereals	Rice, imidazolinone-tolerant	64
	Wheat, imidazolinone-tolerant	65
Oilseed crop	Peanut	66
	Rape seed, imidazolinone-tolerant	67
	Sunflower seed, imidazolinone-tolerant	68
Legume animal feeds	Alfalfa	69
	Pea forage and fodder	70
	Soya bean forage and fodder	71
Forage and fodder of cereal grains and grasses	Rice forage and straw, imidazolinone-tolerant	72
	Wheat forage and straw, imidazolinone-tolerant	73, 74
Miscellaneous fodder and forage crops	Rape seed forage, imidazolinone-tolerant	75

Legume vegetables

Beans

Residue field trials on beans were conducted in the European countries and USA during the growing season of 1996, 2011 and 2012. SL or EC formulation was used at various rates of 0.028–0.075 kg ai/ha with a pre- or post-emergence application. Bean seeds or other bean fractions were analysed for the determination of imazamox and the metabolites.

Table 58 Residues of imazamox in beans from supervised field trials

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Denmark Røjleskowej 2011 (Flagrano) 404371// L110253 2012/1221518	SL 22.4	0.028	0.027	1	(22-51)	0 29 29 62 62 62 62	w. plant pod w/ seed rest of plant pod w/ seed pod w/o seed rest of plant seeds	1.70 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	1.71 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Germany Palatinate 2011 (Primel) 404371// L110251 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 27 27 41 41 41 41	w. plant pod w/ seed rest of plant pod w/ seed pod w/o seed rest of plant seeds	0.48 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.49 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
Germany Baden-Württemberg 2011 (Flageolet) 404371// L110254 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 30 30 51 51 51 51	w. plant pod w/ seed rest of plant pod w/ seed pod w/o seed rest of plant seeds	2.10 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.02 < 0.01 < 0.01 < 0.01 < 0.01	2.11 < 0.02 0.03 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 0.07 < 0.01 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
N-France Loiret 2011 (Nagano) 404371// L110252 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 28 28 35 35	w. plant pod w/seed rest of plant pod w/seed rest of plant	3.70 < 0.01 < 0.01 < 0.01 < 0.01	0.03 < 0.01 < 0.01 < 0.01 < 0.01	3.73 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 0.02 < 0.01 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01
N-France Hebuterine 2012 (Flageolet bean: Flambean) 421932// L120366 2013/1361329	EC 16.7	0.075	0.038	1	Pre-emergence	46 74 74	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
UK Haddenham 2012 (Flageolet bean: Safari) 421932// L120365 2013/1361329	EC 16.7	0.075	0.038	1	Pre-emergence	48 73 73	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
S-France Tarnet Garonne 2011 (Proton) 404371// L110255 2012/1221518	SL 22.4	0.028	0.028	1	(25, 60)	0 28 28	w. plant pod w/seed rest of plant	1.60 < 0.01 < 0.01	< 0.01 < 0.01 0.03	1.61 < 0.02 0.04	< 0.01 0.02 0.08	< 0.01 < 0.01 < 0.01
S-France Tarnet Garonne 2011 (Proton) 404371// L110256 2012/1221518	SL 22.4	0.039	0.039	1	(25, 60)	0 28 28	w. plant pod w/seed rest of plant	0.80 < 0.01 < 0.01	< 0.01 < 0.01 0.01	0.81 < 0.02 0.02	< 0.01 0.02 0.05	< 0.01 < 0.01 < 0.01
S-France Pusignan 2012	EC 16.7	0.075	0.038	1	Pre-emergence	36 64 64	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(Flageolet bean: Flajoly) 421932// L120368 2013/1361329												
Greece Thessalonikj 2011 (Etna) 404371// L110257 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 29 29 29 29 39 39 39 39	w. plant pod w/ seed pod w/o seed rest of plant seeds pod w/seed pod w/o seed rest of plant seeds	0.79 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.06 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.85 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 0.01 < 0.01 < 0.01 0.02 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
Italy Mincrbio (French bean : n.r.) 1994 94072//1 ID-720-044	SL 120	0.050	0.010	1	Post-emergence	53	pods	< 0.05	< 0.05	< 0.10		
Italy Sasso Morelli-Imola 1994 (French bean: n.r.) 94072//2 ID-720-044	SL 120	0.050	0.010	1	Post-emergence	58	pods	< 0.05	< 0.05	< 0.10		
Italy Sabbiuno 1994 (French bean: Bronco) 94072//3 ID-720-044	SL 120	0.050	0.010	1	Post-emergence	28	pods	< 0.05	< 0.05	< 0.10		
Italy Granarolo Faentino 1994 (French bean: Masai) 94072//4 ID-720-044	SL 120	0.050	0.010	1	Post-emergence	29	pods	< 0.05	< 0.05	< 0.10		
Italy Sasso Morelli 1996 (French bean: Twiggi) ID-IT-96-601// 96-601-01 ID-720-053	SL 40	0.050	0.010	1	Post-emergence (13)	42 51 61	whole plant whole plant pod w/ seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.10 < 0.10 < 0.10		
Italy Gaiba 1996 (French bean: Masai) ID-IT-96-	SL 40	0.050	0.010	1	Post-emergence (13)	26 35 45	whole plant whole plant pod w/ seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.10 < 0.10 < 0.10		

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
601// 96-601-02 ID-720-053												
Italy Roncoferraro 2012 (Flageolet bean : Borlotto Lingua di Fuoco) 421932// L120367 2013/1361329	EC 16.7	0.075	0.038	1	Pre-emergence	32 74 74	w. plant pod w/ seed rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain La Rioja, 2011 (Beronia) 404371// L110258 2012/1221518	SL 22.4	0.028	0.028	1	(25)	0 27 27 35 35 35 35	w. plant pod w/ seed rest of plant pod w/ seed pod w/o seed rest of plant seeds	0.81 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.83 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01 < 0.01
USA Freeville, NY 1998 (Snap bean: Labrador) 06663// 98-NY23 ID-720-073	SL 11.5%, w/w	0.050		1	Post-emergence	35	pod w/seed	< 0.05				
USA Arlington, WI 1998 (Snap bean: Hystyle) 06663// 98-WI30 ID-720-073	SL 11.5%, w/w	0.050		1	Post-emergence	30	pod w/seed	< 0.05				
USA East Lansing, MI 1998 (Snap bean: Strike) 06663// 98-MI40 ID-720-073	SL 11.5%, w/w	0.050		1	Post-emergence	41	pod w/seed	< 0.05				
USA Clinton, NC 1998 (Snap bean: Provider) 06663// 98-NC21 ID-720-073	SL 11.5%, w/w	0.050		1	Post-emergence	50	pod w/seed	< 0.05				
USA Gainesville, FL	SL 11.5%, w/w	0.050		1	Post-emergence	37	pod w/seed	< 0.05				

Location Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in beans, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
1998 (Snap bean: Podsquad) 06663// 98-FL61 ID-720-073												
USA Kimberly, ID 1998 (Snap bean: Idelite Garden) 06663// 98-ID15 ID-720-073	SL 11.5%,w/w	0.040	0.022	1	Post-emergence	44	pod w/seed	< 0.05				

Mix formulation: SL 22.4 g/L (bentazone 480 g/L); EC 16.7 g (pendimethalin 250 g/L)
w. plant: whole plant without roots

Peas

Residue trials on peas were conducted in EU countries and USA during the growing season of 1995–1998 and 2002–2008. SL or EC formulation was used at various rates of 0.028–0.075 kg ai/ha with a pre- or post-emergence application. Pea seeds or other pea fractions were analysed for the determination of imazamox and the metabolites.

Table 59 Residues of imazamox in peas from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
Germany Ostrau (Ambassador) 2007/2008 B42930// A/GE/H/07/136 2007/1023133	SL 22.4	0.028	0.014	1	Post-emergence (18)	40	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.013	< 0.02 < 0.02 0.02
N-France Caillouet (Solara) 1996 ID-FR-96-610//96-610-319 ID-720-048	EC 16.7	0.075	0.021	1	Pre-emergence (05–08)	88	seeds (green)	< 0.05	< 0.05	< 0.1
N-France Bourgogne (Baccara) 1996 ID-FR-96-610//96-610-422 ID-720-048	EC 16.7	0.075	0.031	1	Pre-emergence (00)	105	seeds (green)	< 0.05	< 0.05	< 0.1
N-France Fains (Solara) 1996 ID-FR-96-611//96-	EC 16.7	0.075	0.021	1	Pre-emergence (05–08)	95	seeds (green)	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
611-320 ID-720-051										
N-France Fains (Solara) 1996 ID-FR-96-611//96-610-423 ID-720-051	EC 16.7	0.075	0.031	1	Pre-emergence (00)	110	seeds (green)	< 0.05	< 0.05	< 0.1
N-France Viesly Nord Pas de Calais (Arabelle) 2007/2008 B42930// A/NF/H/07/133 2007/1023133	SL 22.4	0.029	0.007	1	Post-emergence (51)	43 43 43	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02
N-France Wassigny (Ultimo) 2007/2008 B42930// A/NF/H/07/134 2007/1023133	SL 22.4	0.028	0.007	1	Post-emergence (51)	44 44 44	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02
UK Evesham (Ambassador) 2007/2008 B42930// A/UK/H/07/135 2007/1023133	SL 22.4	0.028	0.007	1	Post-emergence (16–17)	51 48 48	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.010	< 0.02 < 0.02 0.02
S-France Nerac (Carrera) 1996 ID-FR-96-660//96-660-293 ID-720-049	EC 16.7	0.075	0.023	1	Pre-emergence (00-01)	96	seeds (green)	< 0.05	< 0.05	< 0.1
S-France Chateaurenard (Douce Provence) 2007/2008 B42930// A/SF/H/07/137 2007/102333	SL 22.4	0.029	0.007	1	Post-emergence (12)	37 39 39	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.015	< 0.02 < 0.02 0.02
S-France Lagnes (Douce Provence) 2007/2008 B42930// A/SF/H/07/140 2007/102333	SL 22.4	0.027	0.007	1	Post-emergence (14)	29 31 31	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.026	< 0.02 < 0.02 0.04
Italy Emilia-Romagna 1994 (n.r.) 94071//1 ID-720-047	SC 22.7	0.068	0.014	1	Pre-emergence	78	seeds	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	SL 120	0.050	0.010	1	Post-emergence	59	seeds	< 0.05	< 0.05	< 0.1
Italy Emilia-Romagna (Hunter) 1994 94071//2 ID-720-047	SC 22.7	0.068	0.014	1	Pre-emergence	77	seeds	< 0.05	< 0.05	< 0.1
	SL 120	0.050	0.010	1	Post-emergence	34	seeds	< 0.05	< 0.05	< 0.1
Italy Emilia-Romagna (Hunter) 1994 94071//3 ID-720-047	SC 22.7	0.068	0.014	1	Pre-emergence	83	seeds	< 0.05	< 0.05	< 0.1
	SL 120	0.050	0.010	1	Post-emergence	38	seeds	< 0.05	< 0.05	< 0.1
Italy Emilia-Romagna (n.r.) 1994 94071//4 ID-720-047	SC 22.7	0.068	0.014	1	Pre-emergence	70	seeds	< 0.05	< 0.05	< 0.1
	SL 120	0.050	0.010	1	Post-emergence	53	seeds	< 0.05	< 0.05	< 0.1
Italy Torello (Zaffiro) 1996 ID-IT-96-608// 96-608-01 ID-720-050	SL 40	0.050	0.010	1	Post-emergence (13)	52	seeds (green)	< 0.05	< 0.05	< 0.1
	SL 40	0.075	0.016	1	Post-emergence (13)	52	seeds (green)	< 0.05	< 0.05	< 0.1
Italy Comacchio (Zaffiro) 1995 ID-IT-95-012// 95-012-01 ID-720-052	SL 120	0.050	0.01	1	Post-emergence (21–25)	40	seeds (green)	< 0.05	< 0.05	< 0.1
Italy Masi Torello (Java) 1995 ID-IT-95-012// 95-012-02 ID-720-052	SL 120	0.050	0.01	1	Post-emergence (12–13)	63	seeds (green)	< 0.05	< 0.05	< 0.1
Italy Caleppio di Settala (Pactol) 2007/2008 B42930// A/IT/H/07/138 2007/1023133	SL 22.4	0.028	0.007	1	Post-emergence (24)	72 72 72	seed (green) pods plant w/o pods	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.029	< 0.02 < 0.02 0.039
Italy Marcallo con	SL 22.4	0.027	0.009	1	Post-emergence	62 62	seed (green) pods	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
Castone (Starter) 2007/2008 B42930// A/IT/H/07/139 2007/1023133					(25)	62	plant w/o pods	< 0.01	< 0.01	< 0.02
Spain Sarifiena-Huesca (Puyet) 1996 ID-SP-96-609 //96-609-27 ID-720-054	SL 40	0.052	0.017	1	Post-emergence (14–15)	64	seeds	< 0.05	< 0.05	< 0.1
	SL 40	0.073	0.024	1	Post-emergence (14–15)	64	seeds	< 0.05	< 0.05	< 0.1
USA Freeville (Bolero) 1998 06664//98-NY12 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	59	seeds	< 0.05		
USA Salinas (Maestro) 1998 06664//98-CA*38 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	44	seeds	< 0.05		
USA Salinas (Oregon Sugar Pod II 1998 06664//98-CA*39 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	36	pods with seeds	< 0.05		
USA Aurora (Knight) 1998 06664//98-OR19 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	65	seeds	< 0.05		
USA Arlington (Rally) 1998 06664//98-WI16 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	44	seeds	< 0.05		
USA Arlington (Dual) 1998 06664//98-WI17 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	48	seeds	< 0.05		
USA[a] East Lansing (Utrillo) 1998 06664//98-MI24 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	57	seeds	< 0.05		
USA[a] East Lansing	SL 11.5%,	0.050		1	Post-emergence	57	seeds	< 0.05		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage at application (BBCH)	DAT	Portion analysed	Residue in peas, mg/kg		
								Parent	CL 263284	Parent + CL 263284
(Utrillo) 1998 06664//98-MI25 ID-720-075	w/w									
USA Charleston (Wando) 1998 06664//98-SC*03 ID-720-075	SL 11.5%,w/w	0.050		1	Post-emergence	49	seeds	< 0.05		

Mix formulation: SC 22.7 g/L (pendimethalin of 341 g/L); SL 22.4 g/L (bentazone of 480 g/L); EC 16.7 g/L (pendimethalin of 250 g/L)
wp: Whole plant
[a] Not independent trials

Pulses

Bean (dry)

Residue field trials on dry beans were conducted in the European countries and USA during the growing season of 1998, 2011 and 2012. SL or EC formulation was used at various rates of 0.028–0.075 kg ai/ha with a pre- or post-emergence application. Bean seeds or other bean fractions were analysed for the determination of imazamox and the metabolites.

Table 60 Residues of imazamox in bean (dry) from supervised field trials

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Hebuterine 2012 (Flageolet bean : Flam bean) 421932 //L120366 2013/1361329	EC 16.7	0.075	0.038	1	Pre-emergence	108 108 108 108	pod w/seed pod w/o seed rest of plant seeds	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
GermanyHalen 2011 (Broad bean: Fuego) 407230 //L110308 2012/1299407	SL 40	0.040	0.020	1	16	0 116 116	w. plant seeds rest of plant	3.07 < 0.01 < 0.01	0.04 < 0.01 < 0.01	3.11 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Netherlands Vlagtwedde 2011 (Broad bean: Fuego) 407230 2012/1299407	SL 40	0.040	0.020	1	16	0 115 115	wp w/o root seeds rest of plant	1.58 < 0.01 < 0.01	0.02 < 0.01 < 0.01	1.60 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
UK Haddenham 2012 (Flageolet bean	EC 16.7	0.075	0.038	1	Pre-emergence	97 97 97 97	pod w/seed pod w/o seed rest of plant seeds	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
: Safari) 421932 //L120365 2013/1361329												
UK Warwickshire 2011 (Broad bean: Wizard) 407230 //L110309 2012/1299407	SL	0.040	0.020	1	16	0 117 117	w. plant seeds rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
UK Banbury 2011 (Broad bean: Wizard) 407230 //L110310 2012/1299407	SL 40	0.040	0.020	1	16	0 139 139	w. plant seeds rest of plant	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01	< 0.02 < 0.02 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
S-France Pusignan 2012 (Flageolet bean: Flajoly) 421932 //L120368 2013/1361329	EC 16.7	0.075	0.038	1	Pre-emergence	91 91 91 91	pod w/seed pod w/o seed rest of plant seeds	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
S-France Tarn Et Garonne 2011 (Irene) 4042844// L110245 2011/1112577	SL 22.4	0.031	0.030	1	25	0 55 55	w. plant seeds pods+ rest5	1.2 < 0.01 < 0.01	< 0.01 0.01 0.05	1.2 0.02 0.06	< 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01
S-France Tarn Et Garonne 2011 (Castel) 4042844// L110247 2011/1112577	SL 22.4	0.033	0.030	1	24-25	0 57 57	w. plant seeds pods+ rest5	0.62 < 0.01 < 0.01	< 0.01 < 0.01 0.01	0.63 < 0.02 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Italy Roncoferraro 2012 (Flageolet bean : Borlotto Lingua di Fluco) 421932 //L120367 2013/1361329	EC 16.7	0.075	0.038	1	Pre-emergence	92 92 92 92	pod w/seed pod w/o seed rest of plant seeds	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Italy Vaccolino 2011 (Broad bean: Aqua Dulce) 407230 //L110314 2012/1299407	SL 40	0.040	0.020	1	16	0 51 51	w. plant seeds rest of plant	2.05 < 0.01 < 0.01	0.04 < 0.01 < 0.01	2.09 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Greece Thessalonikl 2011 (Aqua Dulce) 4042844// L110248 2011/1112577	SL 22.4	0.028	0.03	1	25	0 62 62	w. plant seeds pods+rest 5	1.8 < 0.01 < 0.01	0.03 < 0.01 0.02	1.8 < 0.02 0.03	< 0.01 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain Zafarraya 2011 (Broad bean: Muchamiel) 407230 //L110313 2012/1299407	SL 40	0.040	0.020	1	16	0 91 91	w. plant seeds rest of plant	2.15 < 0.01 < 0.01	0.45 < 0.01 < 0.01	2.60 < 0.02 < 0.02	0.02 < 0.01 < 0.01	0.02 < 0.01 < 0.01
Spain Malaga 2011 (Broad bean: Muchamiel) Broa407230 //L110315 2012/1299407	SL 40	0.040	0.020	1	16	0 90 90	w. plant seeds rest of plant	1.34 < 0.01 < 0.01	0.29 < 0.01 < 0.01	1.63 < 0.02 < 0.02	0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01
USA Ereeville, NY 1998 (Red kidney bean: Horizon Light) 06820// 98-NY11 ID-720-071	SL 11.5%	0.050	0017–0.035	1	Post-emergence 1st trifoliate stage	76	dry seeds	< 0.05				
USA[a] Prosser 1998 (Othello Pinto) 06820// 98-WA*41 ID-720-071	SL 11.5%	0.050	0.017	1	Post-emergence 1st trifoliate stage	59	dry seeds	< 0.05				
USA[a] Prosser 1998 (Othello Pinto) 06820// 98-WA*42 1998 ID-720-071	SL 11.5%	0.050	0.017	1	Post-emergence 1st trifoliate stage	59	dry seeds	< 0.05				
USA Parlier 1998 (UC 6 Blackeye) 6820//98-CA37 ID-720-071	SL 11.5%	0.050	0.035	1	Post-emergence 1st trifoliate stage	84	dry seeds	< 0.05				
USA[b] Freemont 1998 (Avanti Navy) 06820// 98-OH*09 ID-720-071	SL 11.5%	0.050	0.035	1	Post-emergence 1st trifoliate stage	75	dry seeds	< 0.05				
USA[b]	SL	0.050	0.035	1	Post-	70	dry seeds	< 0.05				

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Freemont 1998 (Avanti Navy) 06820// 98-OH*10 1998 ID-720-071	11.5%				emergence 1st trifoliate stage							
USA[b] Freemont 1998 (Avanti Navy) 06820// 98-OH*11 ID-720-071	SL 11.5%	0.050	0017–0.035	1	Post-emergence 1st trifoliate stage	68	dry seeds	< 0.05				
USA[c] Scottsbluff 1998 (Berly Great Northern) 06820//98-NE01 ID-720-071	SL 11.5%	0.050	0017–0.035	1	Post-emergence 1st trifoliate stage	78	dry seeds	< 0.05				
USA[c] Scottsbluff 1998 (Berly Great Northern) 06820// 98- NE02 ID-720-071	SL 11.5%	0.050	0017–0.035	1	Post-emergence 1st trifoliate stage	78	dry seeds	< 0.05				
USA[c] Scottsbluff 1998 (Berly Great Northern) 06820// 98- NE03 ID-720-071	SL 11.5%	0.050	0017–0.035	1	Post-emergence 1st trifoliate stage	82	dry seeds	< 0.05				
USA Prosser 1998 (Lima bean: Baby) 06659// 98-WA*40 1998 ID-720--072	SL 11.5%	0.050		1	Post-emergence 1st -2nd trifoliate stage	76	seeds	< 0.05				
USA[d] Tifton 1998 (Lima bean: Cangreen Bush) 06659// 98-GA*11 ID-720--072	SL 11.5%	0.050		1	Post-emergence 1st -2nd trifoliate stage	64	seeds	< 0.05				
USA[d] Tifton 1998 (Lima bean: Cangreen Bush 06659// 98-GA*12 ID-720--072	SL 11.5%	0.050		1	Post-emergence 1st -2nd trifoliate stage	64	seeds	< 0.05				

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
USA Fermont 1998 (Lima bean: Nemagreen) 06659// 98-OH*08 ID-720--072	SL 11.5%	0.050		1	Post-emergence 1st -2nd trifoliate stage	74	seeds	< 0.05				
USA Parlier 1998 (Lima bean: Henderson) 06659// 98-CA35 ID-720-072	SL 11.5%	0.070		1	Post-emergence 1st -2nd trifoliate stage	61	seeds	< 0.05				
USA Parlier 1998 (Lima bean: Mezcla) 06659// 98-CA36 ID-720--072	SL 11.5%	0.070		1	Post-emergence 1st -2nd trifoliate stage	98	seeds	< 0.05				

Mix formulation: SL 22.4 g/L (bentazone of 480 g/L); EC 16.7 g/L (pendimethalin of 250 g/L)
w. plant: Whole plant without roots
n.a.: Not available; a default LOQ (0.05 mg/kg) for CL263284 was used in calculating sum of imazamox and CL 263284 residues
[a] Not independent trials
[b] Not independent trials
[c] Not independent trials
[d] Not independent trials

Pea (dry)

Residue field trials on dry peas were conducted in France during the growing season of 1994 and 1996. SL or EC formulation was used at rates of 0.071–0.075 kg ai/ha with a pre-emergence application. Pea seeds or other pea fractions were analysed for the determination of imazamox and the metabolites.

Table 61 Residues of imazamox in pea (dry) from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in pea (dry), mg/kg		
								Parent	CL 263284	Total
N-France Serez 1994 (Solara) 94092// R03.94/F-370 ID-720-042	SL 120	0.075	0.019	1	Pre-emergence (06–07)	127 127	grain straw			< 0.05 [a] < 0.05 [a]
N-France Bataille 1994 (Solara) 94092// R03.94/F-371 ID-720-042	SL 120	0.075	0.019	1	Pre-emergence (06–07)	126 126	grain straw			< 0.05 [a] < 0.05 [a]

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in pea (dry), mg/kg		
								Parent	CL 263284	Total
N-France Bourgogne 1994 (Messire) 94092// R03.94/F-449 ID-720-042	SL 120	0.075	0.025	1	Pre-emergence (06)	117	grain straw			< 0.05 [a] < 0.05 [a]
N-France Marne 1994 (Montana) 94092// R03.94/F-535 ID-720-042	SL 120	0.075	0.023	1	Pre-emergence (00–05)	124	grain straw			< 0.05 [a] < 0.05 [a]
N-France Varennes 1994 (Mesire) 94092// R03.94/F-536 ID-720-042	SL 120	0.075	0.0 23	1	Pre-emergence (00–02)	116	grain straw			< 0.05 [a] < 0.05 [a]
N-France Caillouet-Orgeville 1996 (Solara) ID FR 96 610 //96-610-319 ID-720-048	EC 16.7	0.075	0.021–0.031	1	Pre-emergence (05–08)	106 106	pods+haulm seeds (dried)	< 0.05 < 0.05	0.17 < 0.05	0.22 < 0.1
N-France Bourgogne 1996 (Baccara) ID-FR-96-610 //96-610-422 ID-720-048	EC 16.7	0.075	0.031	1	Pre-emergence (00)	126 126	pods+haulm seeds (dried)	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
N-France Fains 1996 (Solara) ID FR 96 611 //96-611-320 ID-720-051	EC 16.7	0.075	0.021	1	Pre-emergence (05–08)	117 117	seeds (dried) pods+haulm	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
N-France Pomacle 1996 (Carrera) ID FR 96 611 //96-610-423 ID-720-051	EC 16.7	0.071	0.031	1	Pre-emergence (00)	129 129	seeds (dried) pods+haulm	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
S-France Nerac 1996 (Carrera) ID FR 96 660 //96-660-293 ID-720-049	EC 16.7	0.075	0.023	1	Pre-emergence (00–01)	120 120	seeds (dried) pods+haulm	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)

[a] Imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

Lentil

Residue trials were conducted with imidazolinone-tolerant lentils in Canada and USA during the growing season of 2001–2008. SL or WG formulation was used at rates of 0.015–0.021 kg ai/ha with a post-emergence application. Lentil seeds were analysed for the determination of imazamox and the metabolites.

Table 62 Residues of imazamox in imidazolinone-tolerant lentils from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in lentil, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Canada Dundurn 2008 (Impact) 327307//R080103 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Stem elongation, 7–8 nodes	58	seed	< 0.05	< 0.05	< 0.1		
Canada Portage la Prairie 2008 (CDC Impact) 327307//R080104 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Early flower	40 50 60 70 81	seed seed seed seed seed	0.08 0.07 < 0.05 0.06 0.06	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.13 0.11 < 0.1 0.11 0.11		
Canada Dundurn 2008 (Imperial) 327307//R080105 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Stem elongation, 7–8 node	58	seed	< 0.05	< 0.05	< 0.1		
Canada Wellwood 2008 (CDC Impact) 327307//R080106 2008/7019226	SL 31.3	0.020	0.019 – 0.020	1	Early bud	59	seed	< 0.05	< 0.05	< 0.1		
Canada Alvena 2008 (Impact) 327307//R080107 2008/7019226	SL 31.3 g/L	0.020	0.020	1	Stem elongation, 6–8 nodes	59	seed	0.06	< 0.05	0.11		
Canada Elm Creek 2001 (RH 44) 66880//2001926 2002-5004457	WG 360	0.015	0.015	1	6 nodes	78	seed	< 0.05	< 0.05	< 0.10	< 0.05	< 0.05
Canada Schuler 2001 (RH 44) 66880//2001927 2002-5004457	WG 360	0.020	0.020	1	3–6 leaf	55	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Richmond 2001 (RH 44) 66880//2001928 2002-5004457	WG 360	0.015	0.015	1	6 nodes	48	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Delisle 2001 (RH 44) 66880//2001929 2002-5004457	WG 360	0.020	0.020	1	4–6 nodes	62 69	seed seed	< 0.05 0.056	< 0.05 < 0.05	< 0.1 0.11	< 0.05 < 0.05	< 0.05 < 0.05

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in lentil, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Canada Paradise Valley 2001 (RH 44) 66880/2001930 2002-5004457	WG 360	0.015	0.015	1	5–6 nodes	64	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Minto 2001 (RH 44) 66880//2001931 2002-5004457	WG 360	0.015	0.015	1	6 nodes	98	seed	< 0.05	< 0.05	< 0.1	< 0.05	< 0.05
Canada Hepburn, SK 2005 (CDC 2464) 138146//R05083 2006/7006733	WG 700	0.020	0.010	1	Full flowering (50% flowers Open)	60	seed	< 0.05	< 0.05	< 0.1		
Canada Hepburn, SK 2006 (Smart) 258736//R06475 2006/7011065	WG 700	0.021	0.010	1	Crop 25 in tall (51–62)	60	seed	< 0.05	< 0.05	< 0.1		
USA Pepin, WI 2005 (CDC 2464) 138146//R05080 2006/7006733	WG 700	0.020	0.011	1	Inflorescence or flower buds visible	60	seed	0.12	< 0.05	0.17		
USA Stutsman, MD 2005 (CDC 2464) 138146//R05081 2006/7006733	WG 700	0.021	0.017	1	First flower petals visible (in petalled form)	60	seed	0.12	< 0.05	0.17		
USA Brown, SD 2005 (CDC 2464) 138146//R05082 2006/7006733	WG 700	0.020	0.017	1	First flowers open	60	seed	0.065	< 0.05	0.11		

Mix formulation: SL 31.3 g/L (imazapyr of 14.2 g/L); WG 360 g/kg (imazethapyr of 350 g/kg)

Soya bean (dry)

Residue trials on soya beans were conducted in EU countries and Canada during the growing season of 1994 and 2007–2011. SL or WG formulation was used at various rates of 0.035–0.071 kg ai/ha with a pre- or post-emergence application. Soya bean seeds were analysed for the determination of imazamox and the metabolites.

Table 63 Residues of imazamox in soya bean (dry) from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Piney 2011 (Sigalia)	SC 22.4	0.042	0.021	1	(25–55)	104	seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
402845// L110243 2011/1112578												
N-France Cigogné 2011 (Protina) 402845// L110244 2011/1112578	SC 22.4	0.042	0.021	1	(69)	69	seed	< 0.01	< 0.01	< 0.02	0.04	< 0.01
N-France [a] Burgundy 2008 (Essor) B89774// A/NF/H/08/48 2008/1034456	SC 22.4	0.043	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
N-France [a] Burgundy 2008 (Essor) B89774// A/NF/H/08/49 2008/1034457	SC 22.4 + Dash	0.043	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
Germany Limburgerhof 2011 (Merlin) 402845// L110241 2011/1112578	SC 22.4	0.042	0.021	1	(25)	78	seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Germany Lentzke 2011 (Merlin) 402845// L110242 2011/1112578	SC 22.4	0.042	0.021	1	(21–51)	98	seed	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
S-France Saint-Paul-Les-Romans 2007 (Quito) B42941// A/SF/H/07/142 2007/1023134	SC 22.4	0.042	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
S-France Saint-Paul-Les-Romans 2007 (Quito) B44111// A/SF/H/07/165 2007/1028359	SC 22.4 + Dash	0.045	0.021	1	(13)	90	seed	< 0.01	< 0.01	< 0.02		
S-France Bevons 2007 (Deka Big) B42941// A/SF/H/07/143 2007/1023134	SC 22.4	0.040	0.021	1	(13)	104	seed	< 0.01	< 0.01	< 0.02		
S-France Bevons 2007	SC 22.4	0.042	0.021	1	(13)	104	seed	< 0.01	< 0.01	< 0.02		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean (dry), mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(Deka Big) B4411// A/SF/H/07/166 2007/1023134	+ Dash											
Italy Gorgonzola 2007 (M10, Pioneer) B42941// A/IT/H/07/144 2007/1023134	SC 22.4	0.043	0.011	1	(23–24)	88	seed	< 0.01	< 0.01	< 0.02		
Italy Gorgonzola 2007 (M10, Pioneer) B44111// A/IT/H/07/67 2007/1028359	SC 22.4 + Dash	0.042	0.011	1	(23–24)	88	seed	< 0.01	< 0.01	< 0.02		
Italy Liscate 2007 (B92, Pioneer) B42941// A/IT/H/07/145 2007/1023134	SC 22.4	0.042	0.011	1	(24)	85	seed	< 0.01	< 0.01	< 0.02		
Italy Liscate 2007 (B92, Pioneer) B44111// A/IT/H/07/168 2007/1028359	SC 22.4 + Dash	0.042	0.011	1	(24)	85	seed	< 0.01	< 0.01	< 0.02		
Canada Wallacetown 1994 (n.r.) RES 95-157// 101355 ID-720-015	WG 700	0.035		1	Pre-emergence	119	bean			< 0.05		
		0.070		1	Pre-emergence	119	bean			< 0.05		
		0.035		1	Post-emergence	112	bean			< 0.05		
		0.070		1	Post-emergence	112	bean			< 0.05		
Canada Georgetown 1994 (AC Bravor) RES 95-048// XP94CN07 ID-720-020	WG 700	0.071		1	Post-emergence	106	dry seed			< 0.05		

Mix formulation: SC 22.4 g/L (bentazone of 480 g/L)

In the trials conducted in Canada, imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

[a] Not independent trials

Cereals

Rice

Residue trials were conducted with rice plant water (imidazolinone-tolerance variety) in Italy in 2004–2012. SL formulation was used at various rates of 0.035–0.075 kg ai/ha at BBCH 13–31. In the all trial, the flooding was interrupted a few days before treatment. Applications were carried out on humid soil but without water in the paddy rice. Rice grain was analysed for the determination of imazamox and the metabolites.

Table 64 Residues of imazamox in imidazolinone-tolerant rice from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Italy 2004 (n.r.) A-49-04-30//n.r. 2004/1025824 2004-1026636	SL 40	0.070	n.r.	1	25–30	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
	SL 40 + Dash	0.050	n.r.	1	25–30	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
	SL 40	0.035	n.r.	2	13–14 25–31 Interval, 15–25 days	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
	SL 40 + Dash	0.035	n.r.	2	13–14 25–31 Interval, 15–25 days	n.r.	paddy rice brown rice milled rice	< 0.05 < 0.05 < 0.05				
Italy Salussola 2011 (Sirio CL) 407231//L110320 2012/1278352	SL 40	0.075	0.038	1	24–25	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.075	0.038	1	24–25	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13–14 24–25 Interval, 13 days	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Italy Zeme 2011 (Sirio CL) 407231// L110321 2012/1278352	SL 40	0.075	0.038	1	25	78	grain	0.01	0.04	0.05	0.02	< 0.01
	SL 40 + Dash	0.075	0.038	1	25	78	grain	0.02	0.05	0.07	0.03	< 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	SL 40 + Dash	0.038	0.019	2	13–14 25 Interval, 31 days	78	grain	< 0.01	0.03	0.04	0.02	< 0.01
Italy Zibido 2012 (CL 71) 429716// L120440 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	85	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 37 days	85	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Italy Salussola 2012 (CL 71) 429716// L120441 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 37 days	90	grain	< 0.01	< 0.01	< 0.02	< 0.01	< 0.01
Spain [a] Los Palacios 2011 (Sirio) 407231// L110322 2012/1278352	SL 40	0.075	0.038	1	25	84	grain	< 0.01	0.03	0.04	0.02	< 0.01
	SL 40 + Dash	0.075	0.038	1	25	84	grain	< 0.01	0.03	0.04	0.01	< 0.01
	SL 40 + Dash	0.038	0.019	2	13–14, 25 Interval, 18 days	84	grain	< 0.01	0.03	0.04	0.01	< 0.01
Spain [a] Los Palacios 2011 (Sirio) 407231// L110323 2012/1278352	SL 40	0.075	0.038	1	25	84	grain	< 0.01	0.02	0.03	0.01	< 0.01
	SL 40 + Dash	0.075	0.038	1	25	84	grain	< 0.01	0.04	0.05	0.02	< 0.01
	SL 40 + Dash	0.038	0.019	2	13–14, 25 Interval, 18 days	84	grain	< 0.01	0.01	0.02	0.01	< 0.01
Spain [b] Los Palacios 2012	SL 40	0.075	0.038	1	25	77	grain	0.01	0.05	0.06	0.02	< 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(Sirio) 429716// L120442 2012/1272623	+ Dash											
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 20 days	77	grain	< 0.01	0.02	0.03	< 0.01	< 0.01
Spain [b] Los Palacios 2012 (Sirio) 429716 2012/1272623 L120443	SL 40 + Dash	0.075	0.038	1	25	84	grain	0.01	0.04	0.05	0.02	< 0.01
	SL 40 + Dash	0.038	0.019	2	13, 25 Interval, 17 days	84	grain	< 0.01	0.01	0.02	< 0.01	< 0.01

n.r.: Not reported
w. plant: Whole plant without roots
[a] Not independent trials
[b] Not independent trials

Wheat

During the growing season of 1995–1998, residue trials were conducted with wheat of imidazolinone-tolerant variety in France, Italy, Canada and USA. Treatments were made post-emergent with various formulations at rates of 0.020–0.075 kg ai/ha. Wheat grain samples were analysed for the determination of imazamox and the metabolites.

Table 65 Residues of imazamox in imidazolinone-tolerant wheat from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Les Mureaux 1996 (Fidel) ID-FR-96-617// 96-617-323 ID-730-023	SL 40	0.075	0.021	1	25 5 tillers detectable	129	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	25 5 tillers detectable	129	grain	< 0.05	< 0.05	< 0.1
N-France Pignicourt 1996 (Fidel) ID-FR-96-617// 96-617-465 ID-730-023	SL 40	0.075	0.025	1	25–29 5 to 9 or more tillers detectable	125	grain	< 0.05	< 0.05	< 0.1
	EC	0.067	0.022	1	25–29	125	grain	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	16.7				5 tillers detectable-beginning of stem elongation					
N-France Cassignas 1995 (Fidel) ID-FR-95-005// ID-730-034	SL 120	0.075	0.019	1	31 first node detectable	118	grain	< 0.05	< 0.05	< 0.1
N-France Montchauvet 1996 (Fidel) ID-FR-96-616// 96-616-322 ID-730-035	SL 40	0.075	0.021	1	23–29 3 tillers detectable – end of tillering	121	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.028	1	23–29	121	grain	< 0.05	< 0.05	< 0.1
N-France Tilloy-Bellay 1996 (Fidel) ID-FR-96-616// 96-616-464 ID-730-035	SL 40	0.075	0.032	1	25–27 5–7 tillers detectable	146	grain	< 0.05	< 0.05	< 0.10
	EC 16.7	0.067	0.018	1	25–27	146	grain	< 0.05	< 0.05	< 0.1
S-France[a] Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-291 ID-730-022	SL 40	0.075	0.020	1	26–28 6–8 tillers detectable	131	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	26–28 6–8 tillers detectable	131	grain	< 0.05	< 0.05	< 0.1
S-France[a] Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-292 ID-730-022	SL 40 g/L	0.075	0.020	1	27–29 7 tillers detectable-end of tillering	118	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	27–29 7 tillers detectable-end of tillering	118	grain	< 0.05	< 0.05	< 0.1
S-France Montchauvet 1995 (Fidel) ID-FR-95-005// 95-005-318 ID-730-034	SL 120	0.075	0.019-0.025	1	30 beginning of stem elongation	140	grain	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
S-France Bourgogne 1995 (Fidel) ID-FR-95-005// 95-005-484 ID-730-034	SL 120	0.075	0.019-0.025	1	30	135	grain	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-618// 96-618-01 ID-730-019	SL 40	0.075	0.016	1	25 (5 tillers detectable)	102	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	102	grain	< 0.05	< 0.05	< 0.1
Italy Pisa 1996 (Fidel) ID-IT-96-618// 96-618-02 ID-730-019	SL 40 g/L	0.075	0.016	1	25	103	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	103	grain	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-615// 96-615-01 ID-730-020	SL 40	0.075	0.016	1	25 5 tillers detectable	95	grain	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25 5 tillers detectable	95	grain	< 0.05	< 0.05	< 0.1
Italy Bologna 1997 (Fidel) ID-IT-97-808// 97-808-01 ID-730-021	SL 40	0.075	0.016	1	29 end of tillering	98	grain	< 0.05	< 0.05	< 0.1
Canada Minto 1997 (SWP 965-001) RES 98-001// XP97CN01 ID-730-015	WG 700 + Merge	0.020	0.020	1	5–6 leaf stage	89	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040	0.040	1	5–6 leaf stage	89	grain	< 0.05	< 0.05	< 0.1
Canada Aberdeen 1997 (SWP 965-001) RES 98-005// XP97CN05 ID-730-016	WG 700 + Merge	0.020		1	3–6 leaf stage	72	grain	< 0.05	< 0.05	< 0.1
	WG	0.040		1	3–6 leaf	72	grain	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	700 + Merge				stage					
Canada Saskatoon 1997 (SWP 965-001) RES 98-002// XP97CN02 ID-730-017	WG 700 + Merge	0.020		1	4–6 leaf stage	79	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	79	grain	< 0.05	< 0.05	< 0.1
Canada Barnwell 1997 (SWP 965-001) RES 98-003// XP97CN03 ID-730-018	WG 700 + Merge	0.020		1	4–6 leaf stage	69	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	69	grain	< 0.05	< 0.05	< 0.1
Canada Nisku 1997 (SWP 965-001) RES 98-006// XP97CN06 ID-730-028	WG 700 + Merge	0.020		1	6 leaf stage	90	grain	< 0.05	< 0.05	< 0.1
	WG 700 + Merge	0.040		1	6 leaf stage	90	grain	< 0.05	< 0.05	< 0.1
USA Northwood, ND 1998 (NDGRDN01) RES 99-094// XP98ND04 ID-730-038	SC 11.5% + X-77	0.067	0.067	1	5 leaf stage	82	grain	< 0.05	< 0.05	< 0.1
	SC 11.5% + X-77	0.20	0.20	1	5 leaf stage	82	grain	< 0.05	< 0.05	< 0.1
	SC 11.5% + X-77	0.32	0.32	1	5 leaf stage	82	grain	0.053	< 0.05	0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)
PE: pre-treatment
[a] Not independent trials

Oilseed crop

Peanut

Residue trials were conducted with peanut in Australia and Brazil. Treatments were made with various formulations at rates of 0.056–0.072 kg ai/ha. Peanut samples were analysed for the residues of imazamox and the metabolites.

Table 66 Residues of imazamox in peanut from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in peanut, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
AUS Kingaroy 1994 (n.r.) TTR-96-010// ID-720-034	100A	0.072	0.072	1	n.r.	130	nut in shell	< 0.05				
AUS Kingaroy 1994 (n.r.) TTR-96-011// ID-720-035	WG 700	0.072	0.072	1	n.r.	130	nut in shell	< 0.05				
AUS Norwin 1993/1994 (n.r.) TTR-96-012//n.r. ID-720-036	SL 120	0.072	0.072	1	n.r.	171	nut in shell	< 0.05				
Brazil Jaboticabal 2011/2012 (IAC 505) 382673// G100726 2013/3000444	SL 28	0.056	0.028	1	81 79 79	36 43 50	seeds seeds seeds	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	0.01 0.02 0.05	
Brazil Candido Rodrigues 2011/2012 (IAC 505) 382673// G100727 2013/3000444	SL 28	0.056	0.028	1	81 77 77	36 43 50	seeds seeds seeds	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	
Brazil Santo Antônio de Posse 2011/2012 (Tatu) 382673// G100730 2013/3000444	SL 28	0.056	0.028	1	79	43	seeds	< 0.01	< 0.01	< 0.02	0.07	
Brazil Uberlândia 2011/2012 (Tatu) 382673// G100733	SL 28	0.056	0.028	1	79 75 71	36 43 50	seeds seeds seeds	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.02 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in peanut, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
2013/3000444												
Brazil Senador Canedo 2012 (Ranner) 382673// G100734 2013/3000444	SL 28	0.056	0.028	1	75	43	seeds	< 0.01	< 0.01	< 0.02	< 0.01	

Mix formulation: SL 28 g/L (bentazone of 600 g/L)
n.r.: Not reported

Rape seed

Residue trials were conducted with rape plant of imidazolinone-tolerant variety (Imi-res.) in European countries, Argentina and Canada in 1994–1998 and 2005–2009. Treatments were made post-emergent with SL, SC, or WG formulations at various rates of 0.020–0.080 kg ai/ha. Rape seed samples were analysed for residues of imazamox and the metabolites.

Table 67 Residues of imazamox in imidazolinone-tolerant rape seed from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Boisset-Les-Prevanches 1996/97 (Imi-res., NW 4602) ID-FR-97-667// 97-667-301 ID-750-009	SL 40	0.073		1	(16–17)	236	seed			< 0.05 [c]		
N-France Mousseaux- Neuville 1996/97 (Imi-res., NW4602) ID-FR-97-667// 97-667-302 ID-750-009	SL 40	0.075		1	(17–18)	221	seed			< 0.05 [c]		
N-France Caillouet-Orgeville 1996 (Imi-res., Pioneer 46A72) ID FR 96 663// 96-663-385 ID-750-006	SL 40	0.075		1	(13–14)	74	seed			< 0.05 [c]		
N-France Herpy 1996 (Imi-res., 46A72) ID FR 96 663// 96-663-469 ID-750-006	SL 40	0.075		1	(12–14)	87	seed			< 0.05 [c]		
Germany Uetza 2005/2006	SC 25	0.050		1	(51)	101	seed	< 0.05	< 0.05	< 0.1		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(PS22-1A-VH) 211921// AF/10114/BA/3 2007/1007939												
Germany Uetza 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/3 2005/2006 2007/1007963	SC 25 + Dash	0.050		1	(51)	101	seed	< 0.05	< 0.05	< 0.1		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/4 2007/1007939	SC 25	0.050		1	(51)	100	seed	< 0.05	< 0.05	< 0.1		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/4 2007/1007963	SC 25 + Dash	0.050		1	(51)	100	seed	< 0.05	< 0.05	< 0.1		
Germany Euskirchen-Dom Esch 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-01 ID-750-017	SL 40	0.075	0.019	1	(18) 8 leaves unfolded	254	seed			< 0.05 [c]		
Germany Billingsdorf Flurnummer 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-02 1997/98 ID-750-017	SL 40	0.077	0.025	1	(17) 7 leaves unfolded	262	seed			< 0.05 [c]		
UK[a] Sowerby, Thirsk 1996 (Imi-res., 45A71) ID UK 96 661// 96-661-01 ID-750-004	SL 40	0.075		1	(13) 3 leaves unfolded	93	seed			< 0.05 [c]		
UK[b] Sessay, Thirsk 1996 (Imi-res., 45A71) ID UK 96 661// 96-661-04 ID-750-004	SL 40	0.075		1	(14) 4 leaves unfolded	93	seed			< 0.05 [c]		
UK[a] Sowerby, Thirsk	SL 40	0.075		1	(13)	93	seed	< 0.05	< 0.05	< 0.05 [c]		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
1996 (Imi-res., 45A71) ID UK 96 662// 96-662-01 ID-750-005												
UK[b] Sessay, Thirsk 1996 (Imi-res., 45A71) ID UK 96 662// 96-662-04 ID-750-005	SL 40	0.075		1	(14)	93	seed	< 0.05	< 0.05	< 0.05 [c]		
UK Northamptonshire 1996/97 (Imi-res., Pioneer 4601) ID-UK-97-681 97-681-01 ID-750-008	SL 40	0.078		1	(13–16) 3–6 leaves	240	seed			< 0.05 [c]		
	SL 40 + amm. sulfate	0.078		1	(13–16)	240	seed			< 0.05 [c]		
UK Suffolk 1996/97 (Imi-res., Pioneer 4601) ID-UK-97-681// 97-681-02 ID-750-008	SL 40 g/L	0.079		1	(13–16) 3–6 leaves	238	seed			< 0.05 [c]		
	SL 40 + amm. sulfate	0.079		1	(13–16)	238	seed			< 0.05 [c]		
S-France La francaise 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/1 2007/1007939	SC 25	0.050		1	(61)	78	seed	< 0.05	< 0.05	< 0.1		
S-France La francaise 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/1 2007/1007939	SC 25 + Dash	0.050		1	(61)	78	seed	< 0.05	< 0.05	< 0.1		
S-France Montauban 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/2 2007/1007939	SC 25	0.050		1	(53)	83	seed	< 0.05	< 0.05	< 0.1		
S-France	SC	0.050		1	(53)	83	seed	< 0.05	< 0.05	< 0.1		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Montauban 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/2 2007/1007939	25 + Dash											
S-France Saint-Brice 1997/98 (Imi-res., NW 4200) ID-FR-98-401// 98-401-297 ID-750-016	SL 40 g	0.075		1	(17–18) 7–8 leaves unfolded	246	seed			< 0.05 [c]		
S-France Martres 1997/98 (Imi.-res., NW 4200) ID-FR-98-401// 98-401-298 ID-750-016	SL 40	0.075		1	(14–17) leaves unfolded	243	seed			< 0.05 [c]		
S-France Yvrac 1997/98 (Imi-re s., NW 4200) ID-FR-98-401// 98-401-299 1997/98 ID-750-016	SL 40	0.075		1	(18–19)	244	seed			< 0.05 [c]		
S-France Martres 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-294 ID-750-007	SL 40	0.075		1	(16–17)	220	seed			< 0.05 [c]		
S-France Cassignas 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-296 ID-750-007	SL 40	0.075		1	(16–18)	217	seed			< 0.05 [c]		
S-France Laroque Timbaut 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-297 ID-750-007	SL 40	0.075		1	(16–17)	212	seed			< 0.05 [c]		
S-France St Quentin De Baron 1996/97 (Imi-res., NW4602) ID-FR-97-683// 97-683-298 ID-750-007	SL 40	0.075		1	(17–18)	210	seed			< 0.05 [c]		
S-France Frespech 1996/97 (Imi-res., NW4602)	SL 40	0.075		1	(16–17)	210	seed			< 0.05 [c]		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
ID-FR-97-683// 97-683-299 ID-750-007												
Argentina Venado Tuerto 2009 (CL rape: Advanta) 420295// G110284 2012/3001643	WG 700	0.050		1		84	seed	< 0.01	< 0.01	< 0.02	< 0.01	
	WG 700	0.10		1		84	seed	< 0.01	< 0.01	< 0.02	< 0.01	
Argentina San Jeronimo 2009 (CL rape: Advanta) 420295// G110285 2012/3001643	WG 700 g/kg	0.050		1	(14–17)	83	seed	< 0.01	< 0.01	< 0.02	< 0.01	
	WG 700	0.10		1		83	seed	< 0.01	< 0.01	< 0.02	< 0.01	
Canada Estlin 19994 (n.r.) RES 95-113// 101086 ID-750-003	WG 700	0.020		1		83	seed			< 0.05 [c]		
	WG 700	0.040		1		83	seed			< 0.05 [c]		
	WG 700	0.080		1		83	seed			< 0.05 [c]		
	WG 700	0.20		1	(18–19)	83	seed			< 0.05 [c]		
Canada Stirling 1994 (n.r.) RES 95-113// 101088 ID-750-003	WG 700	0.020		1		93	seed			< 0.05 [c]		
	WG 700	0.040		1		93	seed			< 0.05 [c]		
	WG 700	0.080		1		93						
Canada Nisku 1994 (n.r.) RES 95-113// 101089 ID-750-003	WG 700	0.020		1		95	seed			< 0.05 [c]		
	WG 700	0.040		1	Post-emergence	95	seed			< 0.05 [c]		
	WG	0.080		1	Post-emerg.	95	seed			< 0.05 [c]		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	700											
Canada Rosthern 1994 (n.r.) RES 95-113// 101057 ID-750-003	WG 700	0.020		1	Post-emergence	86	seed			< 0.05 [c]		
Canada Carman 1994 (n.r.) RES 95-113// 50128 ID-750-003	WG 700	0.020		1	Post-emergence	70	seed			< 0.05 [c]		
	WG 700	0.040		1	Post-emergence	70	seed			< 0.05 [c]		

w. plant: Whole plant without roots
[a] Not independent trials
[b] Not independent trials
[c] Imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

Sunflower seed

Residue trials on sunflower seed (imidazolinone-tolerance variety) were conducted in European countries, Turkey, Argentina, Canada and USA in 2002–2012. Foliar application was made post-emergent with SL, SC or WG formulation at various rates of 0.015–0.081 kg ai/ha. Rape seed samples were analysed for residues of imazamox and the metabolites.

Table 68 Residues of imazamox in imidazolinone-tolerant sunflower from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Seebach 2003 (IMA-tol., DK 3900 CL) 166093//FAN/28/03 2004/1000754	SL 40	0.049	0.017	1	(14)	16 38 99	w. plant w. plant seed	0.16 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.21 < 0.1 < 0.1		
N-France Thorée les Pins 2003 (IMA-tol., CMS425xRHA426 SF02) 166093//FBM/18/03 2004/1000754	SL 40	0.051	0.017	1	(18)	8 37 98	w. plant w. plant seed	0.09 < 0.05 < 0.05	0.07 < 0.05 < 0.05	0.16 < 0.1 < 0.1		
N-France Pays de la Loire 2012 (Paraiso 1000) 417820//L120331 2013/1003728	SL 25	0.050		1	(18)	0 121 121	w. plant r. plant seed	3.30 < 0.01 < 0.01	0.01 < 0.01 0.02	3.31 < 0.02 0.03	< 0.01 < 0.01 0.07	0.01 < 0.01 < 0.01
	SL	0.050		1	(18)	0	w. plant	2.40	< 0.01	2.41	< 0.01	0.02

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	40 + Dash					121 121	r. plant seed	< 0.01 < 0.01	< 0.01 0.01	< 0.02 0.02	< 0.01 0.05	< 0.01 < 0.01
Germany Halen 2011 (IMA-tol., LN 11180CL plus) 407229//L110316 2012/1084183	SL 40	0.050	0.03	1	(18)	0 112 112	w. plant r. plant seed	1.74 < 0.01 < 0.01	< 0.01 < 0.01 0.02	1.75 < 0.02 0.03	< 0.01 < 0.01 0.03	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 112 112	w. plant r. plant seed	1.60 < 0.01 < 0.01	< 0.01 < 0.01 0.02	1.61 < 0.02 0.03	< 0.01 < 0.01 0.03	< 0.01 < 0.01 < 0.01
Germany Manker Brandenburg 2012 (Paraiso 1000) 417820//L120330 2013/1003728	SL 25	0.050		1	(18)	0 118 118	w. plant r. plant seed	2.40 < 0.01 < 0.01	0.01 < 0.01 0.07	2.41 < 0.02 0.08	< 0.01 < 0.01 0.12	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 118 118	w. plant r. plant seed	2.60 < 0.01 < 0.01	0.01 < 0.01 0.07	2.61 < 0.02 0.08	< 0.01 < 0.01 0.13	< 0.01 < 0.01 < 0.01
UK Banbury 2011 (IMA-tol., LN 11180 CL plus) 407229//L110317 2012/1084183	SL 40	0.050	0.03	1	(18)	0 117 117	w. plant r. plant seed	4.32 < 0.01 < 0.01	0.01 < 0.01 0.01	4.33 < 0.02 0.02	< 0.01 < 0.01 0.02	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050	0.03	1	(18)	0 117 117	w. plant r. plant seed	4.34 < 0.01 < 0.01	< 0.01 < 0.01 0.02	4.35 < 0.02 0.03	< 0.01 < 0.01 0.02	0.01 < 0.01 < 0.01
S-France Tourtres 201 (IMA-tol., LN 11180 CL plus) 407229//L110318 2012/1084183	SL 40	0.050	0.03	1	(18)	0 111 111	w. plant r. plant seed	2.78 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.79 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050	0.03	1	(18)	0 111 111	w. plant r. plant seed	2.47 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.48 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Italy Chiesuol Del Fosso 2011 (IMA-tol., LN 11180 CL plus) 407229//L110319 2012/1084183	SL 40	0.050	0.03	1	(18)	0 117 117	w. plant r. plant seed	2.96 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.97 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 117 117	w. plant r. plant seed	3.20 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	3.21 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01
Italy Altedo Emilia Romagna 2012 (Paraiso 1001) 417820//L120333 2013/1003728	SL 25	0.050		1	(18)	0 114 114	w. plant r. plant seed	4.80 < 0.01 < 0.01	0.01 < 0.01 < 0.01	4.81 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	SL 40 + Dash	0.050		1	(18)	0 114 114	w. plant r. plant seed	2.90 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.91 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain Utrera Andalucia 2012 (Paraiso 1001) 417820//L120332 2013/1003728	SL 25	0.050		1	(18)	0 107 107	w. plant r. plant seed	6.60 < 0.01 < 0.01	< 0.01 < 0.01 0.02	6.61 < 0.02 0.03	< 0.01 < 0.01 0.05	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.050		1	(18)	0 107 107	w. plant r. plant seed	2.70 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.71 < 0.02 < 0.02	< 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01
Spain Los Palacios 2002 (Mycogene X 81359) 142855//ALO/22/02 2002/1012087	SL 40	0.040	0.013	1	(14)	17 46 103	w. plant w. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Spain Utrera 2002 (Mycogene X 81359) 142855//ALO/23/02 2002/1012087	SL 40	0.040	0.013	1	(16)	17 46 110	w. plant r. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Spain El Coroni 2002 (Mycogene X 81359) 142855//AYE/16/02 2002/1012087	SL 40	0.040	0.013	1	(15)	14 43 91	w. plant r. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Spain Alcala del Rio 2002 (Mycogene X 81359) 142855//AYE/17/02 2002/1012087	SL 40	0.040	0.013	1	(16)	20 36 83	w. plant r. plant seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
Turkey n.r. (n.r.) 148288// 2002/1020471	SC 33	0.041		1	4-leaf stage	n.r. n.r. n.r.	seed seed seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
		0.041		1	8-leaf stage	n.r. n.r. n.r.	seed seed seed	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1		
		0.081		1	4-leaf stage	n.r. n.r. n.r.	seed seed seed	0.15 0.11 0.10	0.10 0.07 0.08	0.25 0.18 0.18		
		0.081		1	8-leaf stage	n.r. n.r. n.r.	seed seed seed	0.13 0.08 0.11	0.10 < 0.05 0.07	0.23 0.13 0.18		
Argentina Balcarce Buenos Aires 2007/2008 (f) 2008/2009 [d]	WG 700	0.040		1	V3–V4	106[c] 105[d]	seed [c] seed [d]	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
(n.r.) 310642//G100057 2010/1057139												
	WG 700	0.080		1	V3–V4	106[c] 105[d]	seed [c] seed [d]	< 0.01 < 0.01	0.02 0.02	0.03 0.03	0.03 0.04	
Argentina Tandil, Buenos Aires 2007/2008[c] 2008/2009[d] (n.r.) 310642//G100062 2010/1057139	WG 700	0.040		1	V3–V4	106[c] 105[d]	seed [c] seed [d]	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 0.01	
	WG 700	0.080		1	V3–V4	106[c] 105[d]	seed [c] seed [d]	< 0.01 < 0.01	< 0.01 < 0.01	< 0.02 < 0.02	< 0.01 < 0.01	
Argentina Realicó, Lapampa 2007/2008 [c] 2008/2009 [d] (n.r.) 310642//G100063 2010/1057139	WG 700	0.040		1	V3–V4	98 [c] 109[d]	seed [c] seed [d]	0.02 < 0.01	0.04 < 0.01	0.06 < 0.02	0.09 0.02	
	WG 700	0.080		1	V3–V4	98 [c] 109[d]	seed [c] seed [d]	0.03	0.09	0.12	0.20	
Canada Grey, MB 2007 (Jaguar) 261967// R070268 2008/7008101	WG 350 + Merge	0.015		1	(61–65)	59	seed	0.07	0.06	0.13		
Canada Grant, SK 2007 (Viper) 261967//R070270 2008/7008101	WG 350 + Merge	0.015		1	(65)	58	seed	0.14	0.16	0.30		
Canada St. Claude, MB 2008 (NuSun 8N358) 327308//R080098 2008/7019225	SL 33	0.019		1	Flowering declining (67)	68	seed	0.05	0.19	0.25		
Canada Portage la Prairie, MB 2008 (Viper) 327308// R080099 2008/7019225	SL 33	0.019		1	Full flowering (65)	49 59 70 82 92	seed seed seed seed seed	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	0.06 0.09 0.06 0.09 0.10	0.11 0.14 0.11 0.14 0.15		
Canada Dundern, SK 2008 (Viper) 327308// R080100	SL 33	0.020		1	Stem elongation (33–39)	70	seed	< 0.05	< 0.05	< 0.1		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
2008/7019225												
Canada Wellwood, MB 2008 (Jaguar) 327308//R080101 2008/7019225	SL 33	0.020		1	End of flowering (69)	70	seed	< 0.05	0.06	0.11		
Canada Wellwood, MB 2008 (Viper) 327308//R080102 2008/7019225	SL 33	0.019		1	End of flowering (69)	70	seed	< 0.05	0.06	0.11		
USA Pepin, WI 2007 (8H419CL) 261967//R070269 2008/7008101	WG 350 + Merge	0.017		1	(65)	60	seed	0.05	0.17	0.22		
USA McHenry, ND 2007 (DKF38-80CL) 261967//R070271 2008/7008101	WG 350 + Merge	0.017		1	Late flowering	59	seed	< 0.05	< 0.05	< 0.1		
USA Cass, ND 2007 (Jaguar) 261967//R070272 2008/7008101	WG 350 + Merge	0.034		1	Early bud	40 50 60 69 79	seed seed seed seed seed	0.21 0.14 0.085 0.060 0.095	0.10 0.05 0.07 0.06 0.12	0.31 0.19 0.16 0.12 0.21		
USA Fargo, ND 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//ND02 2002/5004111	SL 11.78% + MSO [b]	0.056		1	vegetative ,18–30 cm	100	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA[a] Minot, ND 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//ND03 2002/5004111	SL 11.78% + MSO [b]	0.056		1	vegetative ,18–30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA[a] Minot, ND 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//ND04 2002/5004111	SL 11.78% + MSO [b]	0.056		1	Vegetative,33 cm	90	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA Scottsbluff, NE 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//NE01 2002/5004111	SL 11.78% + MSO [b]	0.056		1	Vegetative, 33 cm	77	seed	< 0.05	< 0.05	< 0.1		< 0.05

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in sunflower, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
USA Scottsbluff, NE 2000 (IMI sunflower hybrid, CMS HA425/RHA426) 07219//NE02 2002/5004111	SL 11.78% + MSO [b]	0.056		1	Vegetative, 33 cm	76	seed	< 0.05	< 0.05	< 0.1		< 0.05
USA Fort Collins, CO 2000 (IMI sunflower hybrid, CMS 425/RHA426) 07219//CO02 2002/5004111	SL 11.78% + MSO [b]	0.056		1	vegetative ,30–46 cm	72	seed	< 0.05	< 0.05	< 0.1		< 0.05

Mix formulation: SC or SL 33 g/L (imazapyr of 15 g/L); WG 350 g/kg (imazethapyr 35%)
w. plant: whole plant without roots
r. plant: rest of plant without roots
MSO: methylated seed oil
[a] Not independent trials due to the same trial location and close application time two days separate
[b] Tank mix with imazapyr
[c] Grown in 2007/2008
[d] Grown in 2008/2009

Legume animal feeds

Alfalfa

Residue trials on alfalfa were conducted in Greece, Spain and USA in 1996–1999. Foliar application was made post-emergent with SL, SC or WG formulation at rates of 0.067–0.075 kg ai/ha. The forage and hay samples were collected and analysed for residues of imazamox and the metabolites.

Table 69 Residues of imazamox in alfalfa from supervised residue trials

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Greece Partheni, Thessaloniki 1999 (Ipati) ID-HE-99-12// 99-12-01 ID-731-023	SL 40	0.075		1	(19)	21 29 42	w. p. [a] w. p. [a] w. p. [a]	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1
Spain Lerida 1996 (Aragon) ID-SP-96-613// 96-613-24 ID-731-001	SL 40	0.070	0.023	1	4–6 true leaves (14–16)	35 49 56 63 63	w. p. [a] w. p. [a] w. p. [a] w. p. [a] w. p. [b]	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1		
Spain Basanova,	SL 40	0.073		1	5–7 true leaves	7 21	w. p. [a] w. p. [a]	< 0.1 < 0.1	0.27 < 0.1	0.37 < 0.2	0.28 < 0.1	0.68 < 0.1

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Almenar 1997(Aragon) ID-SP-97-805// 97-805-04 ID-731-002					(15–17)	28 35 35	w. p. [a] w. p. [a] w. p. [b]	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1
Spain Basanova, Almenar 1996 (Aragon) ID-SP-96-612// 96-612-25 ID-731-003	SL 40	0.070		1	4–6 true leaves (14–16)	40 40	w. p. [a] w. p. [b]	< 0.1 < 0.1	< 0.1 < 0.1	< 0.2 < 0.2	< 0.1 < 0.1	< 0.1 < 0.1
Spain Castellsera, Lerida 1996 (Aragon) ID-SP-96-612// 96-612-25 ID-731-003	SL 40	0.070		1	5–7 true leaves (15–17)	40 40	w. p. [a] w. p. [b]	< 0.1 < 0.1	< 0.1 < 0.1	< 0.2 < 0.2	< 0.1 < 0.1	< 0.1 < 0.1
USA Northwood, ND 1997 (Vernal) RES 99-006// XP97ND05 ID-731-004	WG 700	0.067		1	10–15 cm early season regrowth	27 27 56 56 99 99	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1
USA Kerman, CA 1998 (Cuff) RES 99-048// XP98CA02 ID-731-007	SL 11.5%	0.067		1	post-emergence 25–36 cm plant height	PE PE 0 0 7 7 14 14 20 20 50 50 79 79	forage hay forage hay forage hay forage hay forage hay forage hay forage hay	< 0.1 < 0.1 3.9 6.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 0.32 3.3 0.13 0.48 < 0.1 0.28 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 4.6 10 0.25 0.60 < 0.2 0.42 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 0.28 < 0.1 0.15 < 0.1 0.14 < 0.1 0.10 < 0.1 < 0.1 < 0.1 < 0.1	< 0.10 < 0.10 0.21 2.9 0.22 0.73 < 0.1 0.36 < 0.1 0.16 < 0.1 < 0.1 < 0.1 < 0.1
USA Barnard, SD 1998 (DeKalb DK 127) RES 99-088// XP98SD01 ID731-009	WG 70.7%	0.068		1	10–20 cm plant height regrowth	26 26 56 56 96 96	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1
USA Noblesville, IN 1998 (Blazer) RES 99-086// XP98IN02 ID-731-010	WG 70.7%	0.067		1	5 cm size regrowth	36 36 92 92 155 155	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1
USA New Holland,	SL 11.5%	0.067		1	13 cm plant height	22 22	forage hay	< 0.1 < 0.1	< 0.1 < 0.1	< 0.20 < 0.20	< 0.1 < 0.1	< 0.1 0.13

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
OH 1998 (Mustang) RES 99-087// XP98OH02 ID-731-013						48 48 80 80	forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1	< 0.20 < 0.20 < 0.20 < 0.20	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1
USA Ephrata, WA 1997 (89-31 WL 323 Foundation) RES 99-092// XP97WA03 ID-731-014	SL 11.9%	0.071		1	4^{th} –5^{th} trifoliate regrowth stage	30 30 66 66 105 105	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.14 < 0.1 < 0.1 < 0.1 < 0.1
USA Germansville, PA 1997 (WL-322 HQ) RES 98-237// XP97PA01 ID-731-017	SL 11.9%	0.067		1	10–15 cm regrowth	27 27 57 57 89 89	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.29 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 0.41 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 0.58 < 0.1 < 0.1 < 0.1 < 0.1	0.33 0.92 < 0.1 < 0.1 < 0.1 < 0.1
USA Hawkinsville, GA 1997 (Alfagraze) RES 99-093// XP97GA01 ID-731-022	WG 700	0.067		1	6^{th}–8^{th} trifoliate stage	27 27 62 62 125 125	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.19 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 0.32 < 0.2 < 0.2 < 0.2 < 0.2	0.16 0.36 < 0.1 < 0.1 < 0.1 < 0.1	0.30 0.49 < 0.1 < 0.1 < 0.1 < 0.1
USA Ephrata, WA 1998 (WH 80) RES 99-049// XP98WA01 ID-731-005	SL 11.5%	0.067		1	early bloom	78	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Five Points, CA 1998 (Pioneer N58258) RES 99-058// XP98CA04 ID-731-006	WG 700	0.068		1	late bud/early bloom	68	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Mendota, CA 1998 (SW 14) RES 99-057// XP98CA03 ID-731-008	SL 11.5%	0.067		1	early bud stage, 71 cm tall	75	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Noblesville, IN 1998 (Pioneer 5454) RES 99-085// XP98IN01 ID-731-011	SL 11.5%	0.067		1	15–18 cm plant post dormancy regrowth	20 20 73 73 165 165	forage hay forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	0.43 0.89 < 0.1 < 0.1 < 0.1 < 0.1	0.56 1.02 < 0.2 < 0.2 < 0.2 < 0.2	0.45 0.80 < 0.1 < 0.1 < 0.1 < 0.1	0.82 1.6 < 0.1 < 0.1 < 0.1 < 0.1
USA Ephrata, WA 1997 (WH 80)	SL 11.9%	0.067		1	Early bud	74	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
RES 98-234// XP97WA01 ID-731-012												
USA Fort Dodge, IA 1997 (Cenex/LOL 740) RES 99-041// XP97IA02 ID-731-015	WG 700	0.067		1	3 leaf	21 21 52 52	forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 0.13 < 0.1 < 0.1
USA Kerman, CA 1997 (Cuff 101) RES 99-069// XP97CA01 ID-731-016	SL 11.9%	0.067		1	10–18 cm plant height after regrowth	PE PE 0 0 7 7 14 14 18 18 48 48	forage hay forage hay forage hay forage hay forage hay forage hay	< 0.1 < 0.1 2.2 6.4 < 0.1 0.24 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 0.59 3.3 0.11 0.48 < 0.1 0.11 < 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 2.9 12 0.21 0.81 < 0.2 0.22 < 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 0.82 0.13 0.30 < 0.1 0.23 < 0.1 0.11 < 0.1 < 0.1	< 0.1 < 0.1 0.32 2.9 0.22 0.70 0.10 0.37 < 0.1 0.21 < 0.1 < 0.1
USA Delta, CO 1998 (Vernema) RES 99-083// XP98CO01 ID-731-018	SL 11.5%	0.067		1	1–8 cm plant height, third trifoliate stage	39 39 102 102	forage hay forage hay	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1
USA Payette, ID 1997 (Vernema) RES 98-233// XP97ID01 ID-731-019	WG 700	0.069		1	post-emergence, early bud stage	79-80	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Payette, ID 1998 (Vernema) RES 99-099// XP98ID02 ID-731-020	WG 700	0.067		1	post-emergence, early bud stage	73	seed	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1
USA Carlyle, IL 1998 (77917) RES 99-084// XP98IL01 ID-731-021	WG 700	0.067		1	10–25 cm plant height	PE PE 0 0 7 7 14 14 20 20 52 52 80	forage hay forage hay forage hay forage hay forage hay forage hay forage	< 0.1 < 0.1 3.7 5.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 0.11 1.6 0.56 2.0 0.25 0.70 0.15 0.31 < 0.1 < 0.1 < 0.1	< 0.2 < 0.2 3.8 7.4 0.93 2.2 0.38 0.82 0.25 0.46 < 0.2 < 0.2 < 0.2	< 0.1 < 0.1 < 0.1 0.52 0.20 0.43 0.20 0.43 0.17 0.20 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 2.6 0.73 2.2 0.54 1.3 0.36 0.64 < 0.1 < 0.1 < 0.1

Location, (Variety) Year Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in alfalfa, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
						80	hay	< 0.1	< 0.1	< 0.2	< 0.1	< 0.1

[a] w. p : Whole plant, green
[b] w. p.: Whole plant, dried
PE: Pre-treatment

Pea forage and fodder

Residue trials on pea forage or fodder were conducted in France, Spain and Italy in 1994, 1996 and 2011. EC or SL formulation was used at rates of 0.050–0.075 kg ai/ha.

Table 70 Residues of imazamox in pea forage and fodder from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in pea forage and fodder, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Caillouet (Solara) 1996 ID-FR-96-610//96-610-319 ID-720-048	EC 16.7	0.075	0.021	1	Pre-emergence (05–08)	88 106	pods+haulm pods+haulm	< 0.05 < 0.05	< 0.05 0.17	< 0.1 0.22
N-France Bourgogne (Baccara) 1996 ID-FR-96-610//96-610-422 ID-720-048	EC 16.7	0.075	0.031	1	Pre-emergence (00)	105 126	pods+haulm pods+haulm	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
N-France Fains (Solara) 1996 ID-FR-96-611//96-611-320 ID-720-051	EC 16.7	0.075	0.021	1	Pre-emergence (05–08)	75 85 95	whole plant whole plant pods+haulm	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
N-France Fains (Solara) 1996 ID-FR-96-611//96-610-423 ID-720-051	EC 16.7	0.075	0.031	1	Pre-emergence (00)	91 100 110 129	whole plant whole plant pods+haulm pods+haulm	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1 < 0.1
S-France Nerac (Carrera) 1996 ID-FR-96-660//96-660-293 ID-720-049	EC 16.7	0.075	0.023	1	Pre-emergence (00–01)	77 86 96 120	whole plant whole plant pods+haulm pods+haulm	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.10 < 0.10
Italy Torello (Zaffiro) 1996 ID-IT-96-608// 96-608-01 ID-720-050	SL 40	0.050	0.010	1	Post-emergence (13)	33 42 52	whole plant whole plant pods+haulm	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	SL	0.075	0.016	1	Post-	52	pods+haulm	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai/hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in pea forage and fodder, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	40				emergence (13)					
Spain Sarifiena-Huesca (Puyet) 1996 ID-SP-96-609 //96-609-27 ID-720-054	SL 40	0.052	0.017	1	Post-emergence (14–15)	44 53 64	whole plant whole plant pods+haulm	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	SL 40	0.073	0.024	1	Post-emergence (14–15)	44 53 64	whole plant whole plant pods+haulm	< 0.05 < 0.05 < 0.05	< 0.05 0.06 < 0.05	< 0.1 0.11 < 0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)

Soya bean forage and fodder

Residue trials on soya bean forage or fodder were conducted in France, Germany and Canada in 1994 and 2011. SC or WG formulation was used at rates of 0.035–0.071 kg ai/ha.

Table 71 Residues of imazamox in soya bean forage and fodder from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean forage and fodder, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
N-France Piney 2011 (Sigalia) 402845// L110243 2011/1112578	SC 22.4	0.042	0.021	1	(25–55)	0 28 104	w. plant w. plant rest	1.5 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	1.5 < 0.02 < 0.02	< 0.01 0.04 < 0.01	< 0.01 < 0.01 < 0.01
N-France Cigogné 2011 (Protina) 402845// L110244 2011/1112578	SC 22.4	0.042	0.021	1	(69)	0 28 69	w. plant w. plant rest	2.1 < 0.01 < 0.01	< 0.01 0.01 0.05	2.11 0.02 0.06	< 0.01 0.14 0.08	< 0.01 < 0.01 < 0.01
Germany Limburgerhof 2011 (Merlin) 402845// L110241 2011/1112578	SC 22.4	0.042	0.021	1	(25)	0 27 78	w. plant w. plant rest	2.2 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	2.2 < 0.02 < 0.02	< 0.01 0.05 0.01	< 0.01 < 0.01 < 0.01
Germany Lentzke 2011 (Merlin) 402845// L110242 2011/1112578	SC 22.4	0.042	0.021	1	(21–51)	0 27 98	w. plant w. plant rest	3.8 < 0.01 < 0.01	< 0.01 < 0.01 0.01	3.9 < 0.02 0.02	< 0.01 0.07 < 0.01	< 0.01 < 0.01 < 0.01
Canada Guelph 1994 (Galt) RES 95-049// XP94CN08	WG 700	0.035		1	Post-emergence	PE 0 17 31 42	forage forage forage forage forage			< 0.05 1.9 < 0.05 < 0.05 < 0.05		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in soya bean forage and fodder, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
ID-720-019						54	forage			< 0.05		
		0.070		1	Post-emergence	PE 0 17 31 42 54	forage forage forage forage forage forage			< 0.05 6.6 < 0.05 < 0.05 < 0.05 < 0.05		
Canada Georgetown 1994 (AC Bravor) RES 95-048// XP94CN07 ID-720-020	WG 700	0.035		1	Post-emergence	PE 0 14 31 42 64 106	forage forage forage forage forage forage straw			< 0.05 3.7 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05		
	WG 700	0.071		1	Post-emergence	PE 0 14 31 42 64 106	forage forage forage forage forage forage straw			< 0.05 11 0.23 < 0.05 < 0.05 < 0.05 0.06		

Mix formulation: SC 22.4 g/L (bentazone of 480 g/L)
w. plant: whole plant
PE: pre-treatment

Forage and fodder of cereal grains and grasses

Rice forage and straw

Residue trials were conducted with imidazolinone-tolerant rice in Italy and Spain in 2011 and 2012. SL formulation was used at rates of 0.038–0.075 kg ai/ha. The forage and straw were collected and analysed for residues of imazamox and the metabolites.

Table 72 Residues of imazamox in imidazolinone-tolerant rice forage and straw from supervised field trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice forage and straw, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Italy Salussola 2011 (Sirio CL) 407231//L110320 2012/1278352	SL 40	0.075	0.038	1	24–25	0 42 90	w. plant w. plant straw	3.74 < 0.01 < 0.01	0.03 < 0.01 < 0.01	3.77 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01
	SL 40 + Dash	0.075	0.038	1	24–25	0 42 90	w. plant w. plant straw	3.42 < 0.01 < 0.01	0.05 < 0.01 < 0.01	3.47 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	0.02 < 0.01 < 0.01
	SL 40 +	0.038	0.019	2	24–25	0 42 90	w. plant w. plant straw	1.91 < 0.01 < 0.01	0.04 < 0.01 < 0.01	1.95 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice forage and straw, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
	Dash											
Italy Zeme 2011 (Sirio CL) 407231// L110321 2012/1278352	SL 40	0.075	0.038	1	25	0 25 78	w. plant w. plant straw	1.74 0.01 < 0.01	< 0.01 0.02 0.02	1.75 0.03 0.03	< 0.01 < 0.01 0.01	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.075	0.038	1	25	0 25 78	w. plant w. plant straw	1.68 0.03 < 0.01	0.01 0.04 0.03	1.69 0.07 0.04	< 0.01 < 0.01 0.02	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 25 78	w. plant w. plant straw	1.06 0.01 < 0.01	0.02 0.03 0.02	1.08 0.04 0.03	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01
Italy Zibido 2012 (CL 71) 429716// L120440 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	0 39 85	w. plant w. plant straw	2.1 < 0.01 < 0.01	0.02 < 0.01 < 0.01	2.1 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 39 85	w. plant w. plant straw	1.8 0.01 < 0.01	0.02 0.08 < 0.01	1.8 0.09 < 0.02	< 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01
Italy Salussola 2012 (CL 71) 429716// L120441 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	0 41 90	w. plant w. plant straw	3.6 0.01 < 0.01	0.04 0.08 < 0.01	3.6 0.09 < 0.02	< 0.01 0.02 < 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 41 90	w. plant w. plant straw	1.5 < 0.01 < 0.01	0.02 < 0.01 < 0.01	1.5 < 0.02 < 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain[a] Los Palacios 2011 (Sirio) 407231// L110322 2012/1278352	SL 40	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	3.75 < 0.01 < 0.01	0.02 0.02 < 0.01	3.77 0.03 0.02	< 0.01 < 0.01 < 0.01	0.01 < 0.01 < 0.01
	SL 40 + Dash	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	2.22 0.01 < 0.01	0.02 0.03 0.01	2.24 0.04 0.02	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 36 84	w. plant w. plant straw	2.00 < 0.01 < 0.01	0.02 0.02 0.01	2.02 0.03 0.02	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
Spain[a] Los Palacios 2011 (Sirio)	SL 40	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	2.91 0.01 < 0.01	0.01 0.04 0.02	2.92 0.05 0.03	< 0.01 0.02 0.01	0.01 < 0.01 < 0.01

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue in rice forage and straw, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
407231// L110323 2012/1278352												
	SL 40 + Dash	0.075	0.038	1	25	0 36 84	w. plant w. plant straw	2.40 0.01 < 0.01	0.02 0.04 0.02	2.42 0.05 0.03	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 36 84	w. plant w. plant straw	1.44 < 0.01 < 0.01	0.02 0.02 < 0.01	1.46 0.03 0.02	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01
Spain[b] Los Palacios 2012 (Sirio) 429716// L120442 2012/1272623	SL 40 + Dash	0.075	0.038	1	25	0 31 77	w. plant w. plant straw	5.1 0.02 < 0.01	0.04 0.04 0.06	5.1 0.06 0.07	< 0.01 < 0.01 0.03	< 0.01 < 0.01 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 31 77	w. plant w. plant straw	1.7 < 0.01 < 0.01	0.02 0.02 0.02	1.7 0.03 0.03	< 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01
Spain[b] Los Palacios 2012 (Sirio) 429716 2012/1272623 L120443	SL 40 + Dash	0.075	0.038	1	25	0 38 84	w. plant w. plant straw	2.9 < 0.01 < 0.01	0.04 0.02 0.04	2.9 0.03 0.05	< 0.01 < 0.01 0.02	< 0.01 < 0.01 0.01
	SL 40 + Dash	0.038	0.019	2	25	0 38 84	w. plant w. plant straw	1.2 < 0.01 < 0.01	0.02 < 0.01 0.02	1.2 < 0.02 0.03	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01

n.r.: Not reported
w. plant: Whole plant without roots
[a] Not independent trials
[b] Not independent trials

Wheat forage and straw

Residue trials were conducted with imidazolinone-tolerant wheat in France, Italy, Canada and USA. Treatments were made with various formulations at rates of 0.020–0.075 kg ai/ha. The forage and straw were analysed for residues of imazamox and metabolites.

Table 73 Residues of imazamox in imidazolinone-tolerant wheat forage from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Les Mureaux 1996 (Fidel) ID-FR-96-617//	SL 40	0.075	0.021	1	25 5 tillers detectable	71 91	whole plant whole plant	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
96-617-323 ID-730-023										
	EC 16.7	0.067	0.018	1	25 5 tillers detectable	71 91	whole plant whole plant	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1
N-France Pignicourt 1996 (Fidel) ID-FR-96-617// 96-617-465 ID-730-023	SL 40	0.075	0.025	1	25–29 5 to 9 or more tillers detectable	0 65 83	whole plant whole plant whole plant	5.12 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	5.17 < 0.1 < 0.1
	EC 16.7	0.067	0.022	1	25–29 5 tillers detectable-beginning of stem elongation	0 65 83	whole plant whole plant whole plant	4.47 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	4.52 < 0.1 < 0.1
N-France Montchauvet 1996 (Fidel) ID-FR-96-616// 96-616-322 ID-730-035	SL 40	0.075	0.021	1	23–29 3 tillers detectable – end of tillering	83	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.028	1	23–29	83	whole plant	< 0.05	< 0.05	< 0.1
N-France Tilloy-Bellay 1996 (Fidel) ID-FR-96-616// 96-616-464 ID-730-035	SL 40	0.075	0.032	1	25–27 5–7 tillers detectable	110	whole plant	< 0.05	< 0.05	< 0.10
	EC 16.7	0.067	0.018	1	25–27	110	whole plant	< 0.05	< 0.05	< 0.1
S-France[a] Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-291 ID-730-022	SL 40	0.075	0.020	1	26–28 6–8 tillers detectable	96	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	26–28 6–8 tillers detectable	96	whole plant	< 0.05	< 0.05	< 0.1 <
S-France[a] Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-292 ID-730-022	SL 40 g/L	0.075	0.020	1	27–29 7 tillers detectable-end of tillering	83	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	27–29 7 tillers detectable-end of tillering	83	whole plant	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
Italy Bologna 1996 (Fidel) ID-IT-96-618// 96-618-01 ID-730-019	SL 40	0.075	0.016	1	25 (5 tillers detectable)	0 53 74	whole plant whole plant whole plant	3.34 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	3.39 < 0.1 < 0.1
	EC 16.7	0.067	0.016	1	25	0 53 74	whole plant whole plant whole plant	2.74 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	2.79 < 0.1 < 0.1
Italy Pisa 1996 (. Fidel) ID-IT-96-618// 96-618-02 ID-730-019	SL 40 g/L	0.075	0.016	1	25	0 50 71	whole plant whole plant whole plant	1.61 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	1.66 < 0.1 < 0.1
	EC 16.7	0.067	0.016	1	25	0 50 71	whole plant whole plant whole plant			
Italy Bologna 1996 (Fidel) ID-IT-96-615// 96-615-01 ID-730-020	SL 40	0.075	0.016	1	25 5 tillers detectable	69	whole plant	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25 5 tillers detectable	69	whole plant	< 0.05	< 0.05	< 0.1
Italy Bologna 1997 (Fidel) ID-IT-97-808// 97-808-01 ID-730-021	SL 40	0.075	0.016	1	29 end of tillering	70	whole plant	< 0.05	< 0.05	< 0.1
Canada Minto 1997 (SWP 965-001) RES 98-001// XP97CN01 ID-730-015	WG 700 + Merge	0.020	0.020	1	5–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage forage	< 0.05 1.60 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 1.65 0.10 < 0.1 < 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040	0.040	1	5–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage forage	< 0.05 3.51 0.10 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 3.56 0.15 < 0.1 < 0.1 < 0.1 < 0.1
Canada Aberdeen 1997 (SWP 965-001) RES 98-005// XP97CN05 ID-730-016	WG 700 + Merge	0.020		1	3–6 leaf stage	0 14 28	forage forage forage	1.19 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	1.28 < 0.1 < 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat forage, mg/kg		
								Parent	CL 263284	Parent + CL 263284
	WG 700 + Merge	0.040		1	3–6 leaf stage	0 14 28	forage forage forage	1.94 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	2.07 < 0.1 < 0.1
Canada Saskatoon 1997 (SWP 965-001) RES 98-002// XP97CN02 ID-730-017	WG 700 + Merge	0.020		1	4–6 leaf stage	7 29 56	forage forage forage	0.13 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.20 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	7 29 56	forage forage forage	0.24 < 0.05 < 0.05	0.056 < 0.05 < 0.05	0.36 < 0.1 < 0.1
Canada Barnwell 1997 (SWP 965-001) RES 98-003// XP97CN03 ID-730-018	WG 700 + Merge	0.020		1	4–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage forage	< 0.05 1.6 0.15 0.080 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.1 1.7 0.23 0.13 < 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	PE 0 7 14 28 42 56	forage forage forage forage forage forage forage	< 0.05 3.2 0.40 0.18 0.055 < 0.05 < 0.05	< 0.05 < 0.05 0.11 0.083 < 0.05 < 0.05 < 0.05	< 0.1 3.3 0.52 0.26 0.11 < 0.1 < 0.1
Canada Nisku 1997 (SWP 965-001) RES 98-006// XP97CN06 ID-730-028	WG 700 + Merge	0.020		1	6 leaf stage	0 14 41	forage forage forage	0.68 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.78 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	6 leaf stage	0 14 41	forage forage forage	1.2 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	1.3 < 0.1 < 0.1
USA Northwood, ND 1998 (NDGRDN01) RES 99-094// XP98ND04 ID-730-038	SL 11.5% + X-77	0.067	0.067	1	5 leaf stage	13 47	forage forage	0.12 < 0.05	< 0.05 < 0.05	0.13 < 0.1

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)
PE: Pre-treatment
[a] Not independent trials

Table 74 Residues of imazamox in imidazolinone-tolerant wheat straw from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
N-France Les Mureaux 1996 (Fidel) ID-FR-96-617// 96-617-323 ID-730-023	SL 40	0.075	0.021	1	25 5 tillers detectable	129	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	25 5 tillers detectable	129	straw	< 0.05	< 0.05	< 0.1
N-France Pignicourt 1996 (Fidel) ID-FR-96-617// 96-617-465 ID-730-023	SL 40	0.075	0.025	1	25–29 5 to 9 or more tillers detectable	125	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.022	1	25–29 5 tillers detectable-beginning of stem elongation	125	straw	< 0.05	< 0.05	< 0.1
N-France Cassignas 1995 (Fidel) ID-FR-95-005// ID-730-034	SL 120	0.075	0.019	1	31 first node detectable	118	straw	< 0.05	< 0.05	< 0.1
N-France Montchauvet 1996 (Fidel) ID-FR-96-616// 96-616-322 ID-730-035	SL 40	0.075	0.021	1	23–29 3 tillers detectable – end of tillering	121	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.028	1	23–29	121	straw	< 0.05	< 0.05	< 0.1
N-France Tilloy-Bellay 1996 (Fidel) ID-FR-96-616// 96-616-464 ID-730-035	SL 40	0.075	0.032	1	25–27 5–7 tillers detectable	146	straw	< 0.05	< 0.05	< 0.10
	EC 16.7	0.067	0.018	1	25–27	146	straw	< 0.05	< 0.05	< 0.1
S-France[a] Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-291 ID-730-022	SL 40	0.075	0.020	1	26–28 6–8 tillers detectable	131	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	26–28 6–8 tillers detectable	131	straw	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
S-France[a] Cassignas 1996 (Fidel) ID-FR-96-619// 96-619-292 ID-730-022	SL 40 g/L	0.075	0.020	1	27–29 7 tillers detectable–end of tillering	118	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.018	1	27–29 7 tillers detectable–end of tillering	118	straw	< 0.05	< 0.05	< 0.1
S-France Montchauvet 1995 (Fidel) ID-FR-95-005// 95-005-318 ID-730-034	SL 120	0.075	0.019-0.025	1	30 beginning of stem elongation	140	straw	< 0.05	< 0.05	< 0.1
S-France Bourgogne 1995 (Fidel) ID-FR-95-005// 95-005-484 ID-730-034	SL 120	0.075	0.019-0.025	1	30	135	straw	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-618// 96-618-01 ID-730-019	SL 40	0.075	0.016	1	25 5 tillers detectable	102	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	102	straw	< 0.05	< 0.05	< 0.1
Italy Pisa 1996 (. Fidel) ID-IT-96-618// 96-618-02 ID-730-019	SL 40 g/L	0.075	0.016	1	25	103	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25	103	straw	< 0.05	< 0.05	< 0.1
Italy Bologna 1996 (Fidel) ID-IT-96-615// 96-615-01 ID-730-020	SL 40	0.075	0.016	1	25 5 tillers detectable	95	straw	< 0.05	< 0.05	< 0.1
	EC 16.7	0.067	0.016	1	25 5 tillers detectable	95	straw	< 0.05	< 0.05	< 0.1
Italy Bologna 1997 (Fidel) ID-IT-97-808// 97-808-01	SL 40	0.075	0.016	1	29 end of tillering	98	straw	< 0.05	< 0.05	< 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
ID-730-021										
Canada Minto 1997 (SWP 965-001) RES 98-001// XP97CN01 ID-730-015	WG 700 + Merge	0.020	0.020	1	5–6 leaf stage	42 56 89	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040	0.040	1	5–6 leaf stage	42 56 89	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
Canada Aberdeen 1997 (SWP 965-001) RES 98-005// XP97CN05 ID-730-016	WG 700 + Merge	0.020		1	3–6 leaf stage	28 36 72	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	3–6 leaf stage	28 36 72	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
Canada Saskatoon 1997 (SWP 965-001) RES 98-002// XP97CN02 ID-730-017	WG 700 + Merge	0.020		1	4–6 leaf stage	42 56 79	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	42 56 79	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
Canada Barnwell 1997 (SWP 965-001) RES 98-003// XP97CN03 ID-730-018	WG 700 + Merge	0.020		1	4–6 leaf stage	42 56 69	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	4–6 leaf stage	42 56 69	hay hay straw	< 0.05 < 0.05 < 0.05	0.098 0.056 < 0.05	0.17 0.11 < 0.1
Canada Nisku 1997 (SWP 965-001) RES 98-006// XP97CN06 ID-730-028	WG 700 + Merge	0.020		1	6 leaf stage	41 56 90	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
	WG 700 + Merge	0.040		1	6 leaf stage	41 56 90	hay hay straw	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.1 < 0.1 < 0.1
USA Northwood, ND 1998	SL 11.5% +	0.067	0.067	1	5 leaf stage	47 82	hay straw	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue in wheat straw, mg/kg		
								Parent	CL 263284	Parent + CL 263284
(NDGRDN01) RES 99-094// XP98ND04 ID-730-038	X-77									

Mix formulation: EC 16.7 g/L (pendimethalin of 250 g/L)
PE: Pre-treatment
[a] Not independent trials

Miscellaneous Fodder and Forage crops

Rape seed forage

Residue trials were conducted with imidazolinone-tolerant rape plant in Germany, UK and France. Treatments were made with SC or SL formulation at rates of 0.050–0.077 kg ai/ha. The plant was analysed for residues of imazamox and metabolites.

Table 75 Residues of imazamox in imidazolinone-tolerant rape seed forage from supervised residue trials

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed forage, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Germany Uetza 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/3 2007/1007939	SC 25	0.050		1	(51)	0 13	w. plant r. plant	0.53 < 0.05	< 0.05 < 0.05	0.58 < 0.1		
Germany Uetza 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/3 2005/2006 2007/1007963	SC 25 + Dash	0.050		1	(51)	0 13	w. plant r. plant	2.20 0.19	< 0.05 0.52	2.25 0.71		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/4 2007/1007939	SC 25	0.050		1	(51)	0 12	w. plant r. plant	0.26 < 0.05	< 0.05 < 0.05	0.31 < 0.1		
Germany Schwienau/Melzingen 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/4 2007/1007963	SC 25 + Dash	0.050		1	(51)	0 12	w. plant r. plant	0.22 0.05	< 0.05 < 0.05	0.27 0.10		
Germany Euskirchen-Dom	SL 40	0.075	0.019	1	(18) 8 leaves	0 145	whole plant whole plant			< 0.05 [a] < 0.05 [a]		

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	Spray conc., kg ai /hL	No.	Crop growth stage (BBCH) at appl.	DAT	Portion analysed	Residue in rape seed forage, mg/kg				
								Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
Esch 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-01 ID-750-017					unfolded	230	whole plant			< 0.05 [a]		
Germany Billingsdorf Flurnummer 1997/98 (Imi-res., NW 4200) ID-GE-98-403// 98-403-02 1997/98 ID-750-017	SL 40	0.077	0.025	1	(17) 7 leaves unfolded	0 154 232	whole plant whole plant whole plant			< 0.05 [a] < 0.05 [a] < 0.05 [a]		
UK Sowerby, Thirsk 1996 (Imi-res., 45A71) ID UK 96 662// 96-662-01 ID-750-005	SL 40	0.075		1	(13)	0 51 63	whole plant whole plant whole plant	3.97 < 0.05 < 0.05	0.06 < 0.05 < 0.05	4.03 < 0.1 < 0.1		
UK Sessay, Thirsk 1996 (Imi-res., 45A71) ID UK 96 662// 96-662-04 ID-750-005	SL 40	0.075		1	(14)	0 51 63	whole plant whole plant whole plant	3.13 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	3.18 < 0.1 < 0.1		
S-France La francaise 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/1 2007/1007939	SC 25	0.050		1	(61)	0 15	w. plant r. plant	0.68 < 0.05	< 0.05 < 0.05	0.73 < 0.1		
S-France La francaise 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/1 2007/1007939	SC 25 + Dash	0.050		1	(61)	0 15	w. plant r. plant	0.97 0.12	< 0.05 0.21	1.02 0.33		
S-France Montauban 2005/2006 (PS22-1A-VH) 211921// AF/10114/BA/2 2007/1007939	SC 25	0.050		1	(53)	0 19	w. plant r. plant	0.44 < 0.05	< 0.05 < 0.05	0.49 < 0.1		
S-France Montauban 2005/2006 (PS22-1A-VH) 251536// AF/10475/BA/2 2007/1007939	SC 25 + Dash	0.050		1	(53)	0 19	w. plant r. plant	1.50 0.14	< 0.05 0.28	1.6 0.42		

w. plant: Whole plant without roots
r. plant: Rest of plant without roots that seeds were taken

[a] Imazamox and CL 263284 were measured as one chromatographic peak representing a total imazamox-related residue.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

The Meeting received information on effects of heating in water and processing on imazamox residues in soya bean, wheat and sunflower seed.

The processing factors (residue processed commodity divided by residue RAC) were calculated based on the total imazamox residues (sum of imazamox and CL 263284, expressed as imazamox). When the total imazamox residue value of the RAC was below limit of quantification, no processing factor was derived. When the total imazamox residue value of the processed commodity was below limit of quantification, the calculated processing factor is reported with a "less than" (<) symbol.

Effects on the nature of residue

To estimate the degradation behaviour of [pyridine-3-^{14}C, imidazolinone-3-^{15}N]-imazamox during industrial processing or household preparation, different processes (pasteurization, baking, brewing, boiling and sterilization) were simulated [Hassink 2012(a), 2011/1286214].

The test item was suspended in aqueous buffer solutions of different pH-values, to give a final concentration of ca. 0.92 µg/mL. Pasteurization was simulated by incubating the test solutions (pH 4) in a rotated round-bottom flask in a water bath for 20 min at 90 °C. For baking, brewing and boiling, the test solutions (pH 5) were treated in an Erlemeyer flask under reflux at 100 °C for 60 min. To avoid an influence of light, the glassware was wrapped. Sterilization was performed by autoclaving the test solution (pH 6) at about 120 °C for 20 min. The total radioactivity present was determined by LSC. Characterization of the radioactivity in the buffered samples after incubation was performed by HPLC.

The recovery of total applied radioactivity (TAR) was 98.3–103.5%. Only in sterilization test, one replicate test solution showed a breakdown product of 5.8% TAR, the peak split into at least two peaks. Imazamox was stable under the simulation conditions of pasteurization, baking, boiling, brewing and sterilization.

Effects on the residue level

No residues were observed in trials with soya beans treated at exaggerated rates (0.13 kg ai/ha or 0.22 kg ai/ha) and no processing of the raw commodity was undertaken [ID-720-005, ID-720-006].

Wheat

During the 1998 growing season, a field trial was carried out in North Dakota, United States [Rodriguez 1999a, ID-730-038]. Wheat was treated with SL formulation (11.5%) and 0.25% adjuvant X-77. Trial plot received one post-emergence broadcast foliar application at a rate of either 0.20 kg ai/ha or 0.32 kg ai/ha, respectively. The applications were made when the plants were in the 5 leaf growth stage. Replicate wheat straw and grain samples were collected at crop maturity for typical wheat harvest (82 DAT). Wheat grain samples treated with 0.20 kg ai/ha were analysed for residues of imazamox and CL 263284 according to method M 3098 (CE-UV). The validated limit of quantitation was 0.05 mg/kg for imazamox and CL 263284 each. Wheat grain treated with 0.32 kg ai/ha was analysed for imazamox and CL 263284 residues according to method M 3252 (HPLC-MS; method M 3098). The validated limit of quantitation was 0.01 mg/kg for each analyte. Samples were stored frozen for a maximum of 5 months until analysis.

Table 76 Residues of imazamox and its metabolites in processed commodities of wheat

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at application	DAT	Analysed portion	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
USA Northwood, ND 1998 (NDGRDN01) RES 99-094// XP98ND04 ID-730-038	SL 11.5% + X-77	0.20	1	5 leaf stage	82	grain	< 0.05	< 0.05	< 0.1
	SL 11.5% + X-77	0.32	1	5 leaf stage	82	Grain [a]	0.053	< 0.05	0.1
						pre-processing grain	0.028 [b]	< 0.01	0.038
						germ	0.075 Pf=2.7	< 0.01	0.085 Pf=2.2
						bran	0.11 Pf=3.9	0.020	0.13 Pf=3.4
						middlings	0.046 Pf=1.6	< 0.01	0.056 Pf=1.5
						shorts	0.090 Pf=3.2	0.013	0.104 Pf=2.6
						flour	0.034 Pf=1.2	< 0.01	0.044 Pf=1.2
						asp. grain fr.	0.029 Pf=1.0	< 0.01	0.039 Pf=1.0

[a] Field trial sample

[b] Mean residue for five subsamples (0.031, 0.030, 0.027, 0.027, 0.026 mg/kg)

Sunflower seed

During the 2000 growing season, one field trial was carried out in Minot, ND, US. Sunflowers were treated with SL formulation (11.78%) and a formulation containing imazapyr. Trial plot received one foliar tank-mix application at a rate of either 0.056 kg ai/ha or 0.11 kg ai/ha of imazamox. Another one plot received a single foliar application of imazamox alone at 0.14 kg ai/ha. All spray solutions included methylated seed oil (1.0% v/v). The applications were made post-emergence when the plants were 30 to 46 cm tall (6 to 8 leaf growth stage). Replicate sunflower seed RAC samples were harvested at normal crop maturity, 88 days after treatment and processed according to simulated commercial practices into meal and refined oil. Sunflower samples were analysed for residues of imazamox, CL 263284 and CL 312622 using a developed HPLC-MSD method. The validated limit of quantitation was 0.05 mg/kg for each analyte. Samples were kept in frozen storage from collection until analysis for a maximum period of 7 months.

Processing factors were not derived as residue concentrations of sunflower seed (RAC) were all < LOQ.

Table 77 Residues of imazamox and its metabolites in processed commodities of sunflower seeds (2000)

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at application	DAT	Portion analysed	Residue, mg/kg				
							Parent	CL 263284	Parent + CL 263284	CL 189215	CL 312622
USA Minot, ND 2000 (IMI sunflower hybrid, cmS HA425/RHA426) 07219//ND03 2002/5004111	SL 11.78 % + MSO [a]	0.056	1	vegetative ,18–30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
						meal	< 0.05	< 0.05	< 0.1		< 0.05
						refined oil	< 0.05	< 0.05	< 0.1		< 0.05
	SL 11.78 % + MSO [a]	0.11	1	vegetative ,18–30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
						meal	0.07	0.07	0.14		< 0.05
						refined oil	< 0.05	< 0.05	< 0.1		< 0.05
	SL 11.78 % + MSO	0.14	1	vegetative ,18–30 cm	88	seed	< 0.05	< 0.05	< 0.1		< 0.05
						meal	0.05	0.06	0.11		< 0.05
						refined oil	< 0.05	< 0.05	< 0.1		< 0.05

[a] tank mix with imazapyr

During the 2008 growing season, one field trial was carried out in SK, Canada [Norris 2009(a), 2008/7019225]. Clearfield Sunflowers were treated with a single foliar application of SL formulation (33% imazamox and 15% imazapyr) at 0.10 kg ai/ha. The applications were made broadcast post-emergence targeting stem elongation (BBCH 33-39) to late flowering growth stages (BBCH 69), at 70 days prior to the harvest of mature (dry) seed. The sunflower samples were analysed for residues of imazamox and CL 263284 using method M 3519 (LC-MS/MS) with a limit of quantitation of 0.05 mg/kg. Samples were stored after harvest for a maximum of 157 days prior to analysis.

Table 78 Residues of imazamox and its metabolites in processed commodities of sunflower seeds (2008)

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at appl.	DALA	Portion analysed	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
Canada Dundern, SK 2008 (Viper) 327308//	SL 33	0.10	1	(33–39)	70	seed	0.20 0.26 mean: 0.23	0.24 0.35 mean: 0.30	0.45 0.63 mean: 0.54

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at appl.	DALA	Portion analysed	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
R080100 2008/7019225									
						meal	0.53 0.51 mean: 0.52 Pf=2.3	1.0 1.0 mean: 1.0	1.6 1.5 mean: 1.6 Pf=3.0
						refined oil	< 0.05 < 0.05 Pf=< 0.2	< 0.05 < 0.05	< 0.05 < 0.05 Pf=< 0.2

Residues from sampled two bulks
Mix formulation: SL 33 g/L (imazapyr of 15 g/L).

During the 2007 growing season, one field trial was carried out in ND, USA [Johnston 2009a, 2008/7008101]. Clearfield sunflowers were treated with a single foliar application of WG formulation (35% imazamox and 35% imazethapyr) at 0.075 kg ai/ha of imazamox. The applications were made post-emergence when the sunflowers were at flower buds visible to late flowering growth stages, at 59 days prior to the harvest of mature (dry) seed. Sunflower samples were analysed for residues of imazamox and CL 263284 using method SOP-PA.0288 (LC-MS/MS) with a limit of quantitation of 0.05 mg/kg. Seed samples were stored frozen after harvest or fraction collection for a maximum of 239 days prior to analysis.

Table 79 Residues of imazamox and its metabolites in processed commodities of sunflower seeds (2007)

Location, Year (Variety) Study code// Trial No. Doc. ID	FL, g/L or g/kg	Rate, kg ai/ha	No.	Crop growth stage (BBCH) at appl.	DALA	Portion analysed	Residue, mg/kg		
							Parent	CL 263284	Parent + CL 263284
USA McHenry, ND 2007 (DKF38-80CL) 261967// 2008/7008101	WG 350 + Merge	0.075	1	Late flowering	59	seed	< 0.05 < 0.05 mean: < 0.05	0.16 0.11 mean: 0.14	0.21 0.17 mean: 0.19
						meal	< 0.05 < 0.05	0.24 0.34 mean: 0.29	0.30 0.41 mean: 0.36 Pf=1.9
						refined oil	< 0.05 < 0.05	< 0.05 < 0.05	< 0.1 < 0.1 Pf=< 0.5

Mix formulation: WG 350 g/kg (imazethapyr 35%)

Table 80 Summary of processing factors for sunflower seed processing

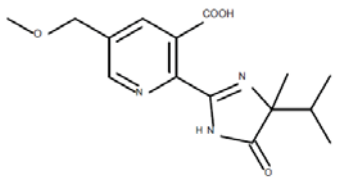
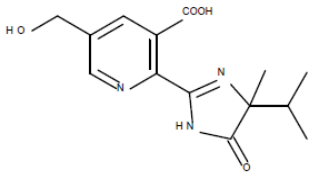
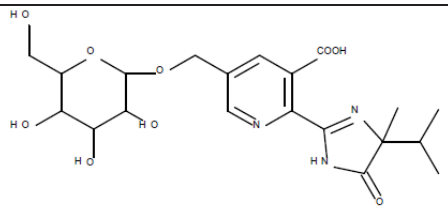
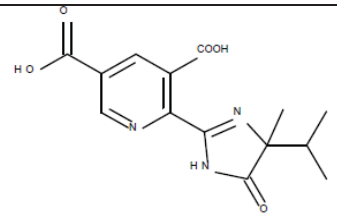
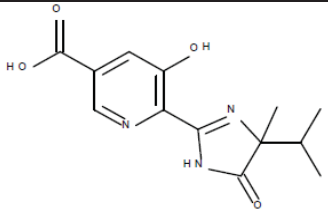
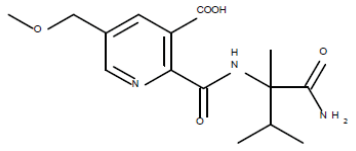
Processed commodity	Imazamox processing factor	Mean or best estimate for imazamox Pf	Sum of imazamox and CL 263284	Mean or best estimate for sum residue Pf
Meal	2.3	2.3	1.9, 3.0	2.5
Refined oil	< 0.2	< 0.2	< 0.2, < 0.5	< 0.5

APPRAISAL

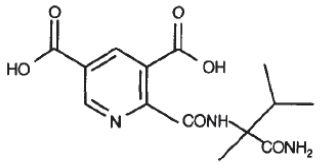
Imazamox is an imidazolinone herbicide registered in many countries to control a wide spectrum of grass and broadleaf weeds. At the Forty-fourth Session of the CCPR (2012)[3], it was scheduled for evaluation as a new compound by the 2014 JMPR.

The Meeting received information on the physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use patterns, supervised trials and processing studies.

The following abbreviated names were used for the metabolites discussed below.

Code (MW)	IUPAC chemical name	Structure
CL 299263 (305) Imazamox; BAS 720 H	*(RS)*-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-5-methoxymethylnicotinic acid	
CL 263284 (291) M715H001	5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazazolin-2-yl) nicotinic acid	
CL 189215 (453.5) M715H002	5-[(β-glucopyranosyl oxy) methyl]-2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl) nicotinic acid	
CL 312622 (305) M720H002	2-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-3,5-pyridine-dicarboxylic acid	
CL 354825 (277)	5-hydroxy-6-(4-isopropyl-4-methyl-5-oxo-2-imidazolin-2-yl)-nicotinic acid	
CL 336554 (323)	2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-5-(methoxymethyl)-nicotinic acid	

[3] REP12/PR-Appendix XIII

Code (MW)	IUPAC chemical name	Structure
CL 152795	2-[(1-carbamoyl-1,2-dimethylpropyl) carbamoyl]-3,5-pyridinedicarboxylic acid	

Animal metabolism

The Meeting received metabolism studies on imazamox, CL 263284 (hydroxymethyl) and CL 312622 (dicarboxylic acid) orally administered to lactating goats and laying hens.

Imazamox

Lactating goats were orally administered single daily doses of [pyridine-6-^{14}C]-imazamox, via gelatin capsules, for seven consecutive days at either 2.1 or 12 ppm. Kidney, liver, leg and loin muscle, and omental fat samples were collected approximately 20 hours after the last dosing.

In the goat the imazamox was mainly excreted in urine (92% of the low dose and 65% of the high dose) and via faeces (14.8% of the low dose and 24.0% of the high dose). Unaltered parent accounted for most of the excreted residue. The total radioactive residues in the daily blood and milk samples and tissues (liver, leg, loin, omental fat) were less than 0.01 mg eq./kg regardless of the treatment dose levels. In the kidney, the TRR was 0.02 mg eq./kg at a dose level of 2.1 ppm and 0.06 mg eq./kg at a dose level of 12 ppm. This was mostly imazamox (89% TRR).

CL 263284

A ruminant metabolism study was conducted with [pyridine-6-^{14}C]-CL 263284. Dose levels for the goats, administered orally in gelatin capsules, were either 2.3 or 15 ppm daily for seven days. Samples of blood, milk and excreta were collected daily. After seven days of dosing, the goats were sacrificed (approximately 20 hours after the last dose) and the tissues kidney, liver, muscle and fat were collected.

Elimination of ^{14}C radioactivity via faeces accounted for 82% and 68% of the total cumulative dose at the low dose and high dose, respectively. TRR levels in most tissue samples (muscle, fat, liver) from the low and high dose treated goat and all milk samples were < 0.01 mg eq./kg. The kidney showed a residue of 0.03 mg eq./kg at the 15 ppm dose, which comprised minor component CL 263284 (9% TRR, < 0.01 mg/kg) and very labile component M1(salt or conjugate of CL 263284; 78% TRR, 0.02 mg eq./kg).

CL 312622

Lactating goats were orally treated with a mixture of [pyridine-6-^{14}C, ^{13}C]-CL 312622. The three goats were dosed for five consecutive days with either 3.1 or 33 ppm. Milk, urine and faeces samples were collected daily. Edible tissues (muscle, fat, liver and kidney) were collected at sacrifice (approximately 22 hours after the last dose).

About 90% of the total cumulative dose was excreted via the faeces. TRR levels in all tissues and milk samples were < 0.006 mg eq./kg, except kidney (0.025 mg eq./kg) and liver (0.009 mg eq./kg) of goats treated at the high dose level (33 ppm).

In the kidney sample, CL 312622 was the predominant radioactive residue (60% TRR). The impurity (CL 152795) present in the dosing solution was found at 11% of TRR. Minor polar unknowns (total 10% of TRR) and non-polar unknowns (total 12% of TRR) were also present in the kidney extract, composed with fractions of 0.003 mg eq./kg with multiple components. In the liver, CL 312622 was present at 38% of TRR (impurity, CL 152795, 19% TRR) and polar and nonpolar (31% and 11% TRR, respectively) fractions were equivalent to 0.003 mg eq./kg or lower, containing multiple components.

In summary imazamox and its metabolites (CL 263284 and CL 312622) each was mainly excreted unchanged through urine and faeces in lactating goats. There was no accumulation in any other edible goat tissue or in the milk (< 0.01 mg eq./kg) except kidney. Kidney mostly contained the unchanged administered compound.

Metabolism of imazamox in laying hens

Imazamox

Laying hens were orally dosed with [pyridine-6-^{14}C]-imazamox at a dose level of either 2.1 or 10 ppm for seven consecutive days. Eggs were collected twice daily and blood and the tissues (liver, kidney, muscle, and skin with adhering fat) were collected for analysis approximately 22 hours after the last dose. About 85% of the total dose administered at the low and high dose was in the excreta. Residues in eggs, skin with adhering fat, muscle, liver and kidney tissues were all less than 0.01 mg eq./kg.

CL 263284

A poultry metabolism study was conducted with [pyridine-6-^{14}C]-CL 263284. Hens were dosed orally at a dose level of either 2.1 or 11 ppm by gelatin capsules for seven days. Eggs and excreta were collected daily. The hens were sacrificed and the tissues (liver, kidney, muscle and skin with adhering fat) were collected approximately 22 hours after the last dose. About 87% of the total dose administered at the low and high dose was eliminated via excreta. Residues in all tissues and eggs were less than 0.01 mg eq./kg.

CL 312622

Laying hens were orally treated with a mixture of [pyridine-6-^{13}C]-CL 312622 and [pyrindine-6-^{14}C]-CL 312622. Hens were dosed for five consecutive days with a dose level of either 0.13 or 10.5 ppm. Eggs were collected twice daily and the edible tissues (muscle, liver and skin with adhering fat) were collected at sacrifice approximately 22 hours after the last dose.

About 89% of the total dose administered at the low dose and high dose was eliminated via the excreta. Residues in tissues (liver, muscle and skin with adhering fat) and eggs were all 0.006 mg eq./kg or below. There was no retention or accumulation in eggs or edible poultry tissues.

In summary, in laying hens, imazamox, its metabolites (CL 263284 and CL 312622) were mainly eliminated unchanged through excreta. No residues (< 0.01 mg eq./kg) in eggs or edible tissues were found.

Plant metabolism

The Meeting received information on the fate of imazamox in oilseed rape, soya bean, pea, maize, wheat and alfalfa.

The metabolic fate of imazamox in imidazolinone-tolerant oilseed rape was investigated in one indoor and two outdoor studies. In one of the outdoor studies, a mixture of [pyridine-6-^{14}C]-imazamox and [pyridine-6-^{13}C]-imazamox was post-emergence treated at 0.02 kg ai/ha. Residues in plant at 0 DAT and seed at 82 DAT were 2.1 mg eq./kg and < 0.002 mg eq./kg, respectively (not further identified for component in plant and seed).

In the other two studies (indoor or outdoor), either [pyridine-6-^{14}C]-imazamox or [imidazolinone-5-^{14}C, 3-^{15}N]-imazamox was applied post-emergence at rates of 0.051–0.089 kg ai/ha (outdoor) or 0.075 kg ai/ha (indoor), respectively. TRR in plant at 0 DAT were 2.2–3.9 mg eq./kg. Residues in forage (12–22 DAT) ranged from 0.04 mg eq./kg to 0.89 mg eq./kg. In straw and seed (78–90 DAT), residues were 0.088–1.1 mg eq./kg and 0.004–0.15 mg eq./kg, respectively. The majority of residues (80–99% TRR) in forage and straw samples were extracted with aqueous methanol or acidic aqueous methanol. Extractability in seeds was relatively low, 58–80% TRR.

In both labels, major components of the residues in forage were imazamox (16–42% TRR) and CL 263284 (36–54% TRR). In straw, major components were CL 263284 (44–50% TRR) and CL

312622 (up to 26% TRR); imazamox, < 2% TRR. In rape seed, major components of the residues were CL 263284 (up to 31%) or CL 189215 (up to 21%); imazamox, present at up to 13%.

The metabolic fate of [pyridine-6-^{14}C]-imazamox in soya bean under outdoor field was studied with post-emergence treatment at a rate of 0.39 kg ai/ha. TRR in plant at 0 DAT was 55 mg eq./kg. Residues in forage (29–60 DAT) ranged from 0.13 mg eq./kg to 0.56 mg eq./kg. In straw and seed (153 DAT), residues were 0.08 mg eq./kg and 0.02 mg eq./kg, respectively. The majority of residues in forage (86–97%) were extracted with aqueous methanol and dichloromethane. Acidic and/or basic digestion for straw and seed samples extracted up to 53% and 57% of residues, respectively.

A major component of the residue in soya bean forage (29–60 DAT) was CL 189215 (32–36% TRR); imazamox, CL 263284 and CL 312622 each, present at 6–11% TRR. In soya bean seed, CL 263284 was a major component, representing 12% TRR.

Field peas were treated post-emergent, under outdoor growing conditions, with [pyridine-6-^{14}C]-imazamox at a rate of 0.040 kg ai/ha. TRRs in whole plant at 0 DAT and 20 DAT were 1.1 mg eq./kg and 0.035 mg eq./kg, respectively. At 61 DAT, residues in all of the samples (pea foliage, immature peas, pea shells and pea pods) were < 0.01 mg eq./kg. In pea and pea hay (84 DAT), residues were 0.01 mg eq./kg and 0.05 mg eq./kg, respectively. The aqueous methanol extracted 61–99% of the TRR in whole plant. From the hay and peas, 58% and 39% of the TRR was extracted, respectively.

In pea hay, imazamox was present at 0.02 mg eq./kg (66% TRR) and other components (CL 263284, CL 189215) were negligible (≤ 0.01 mg eq./kg).

Alfalfa was treated, post-emergent, in plots at different ages of crop and timing of application under outdoor conditions with [pyridine-6-^{14}C]-imazamox at a rate of 0.13 kg ai/ha. TRR in alfalfa plants at 0 DAT was 6.5–15 mg eq./kg. Residues in forage (26 DAT) ranged from 0.18 mg eq./kg to 0.67 mg eq./kg. Residues in forage (1st, 2nd, 3rd cut at 26–111 DAT) and hay (1st, 2nd, 3rd cut at 28–160 DAT) were < 0.01–0.21 mg eq./kg and 0.02–0.83 mg eq./kg, respectively. The majority (65–99% TRR) of residues in forage and hay were extracted with a solvent mixture of methanol/acetone/water.

Residue components in alfalfa forage (26 DAT) were: imazamox, 3–6% TRR and CL 263284, CL 189215 and CL 312622, 14–26% TRR. For forage and hay samples, 1st, 2nd, 3rd cut, composition of the compounds were similar to those in forage at 26 DAT.

The metabolic fate of imazamox on imidazolinone-tolerant maize was investigated in two outdoor studies. Radiolabelled imazamox, either [pyridine-6-^{14}C]-imazamox or a mixture of [pyridine-6-^{14}C]-imazamox and [pyridine-6-^{13}C]-imazamox was post- or pre-emergence (one day after seedling) treated at rates of 0.13–0.14 kg ai/ha. Residues in forage (14, 30 and 62 DAT) ranged from 0.011 mg eq./kg to 0.41 mg eq./kg. In fodder and grain (100–112 DAT), TRRs were 0.012–0.047 mg eq./kg and 0.010–0.01 mg eq./kg, respectively. 40–98% of the TRR in maize samples were extracted with aqueous methanol (40% TRR in fodder).

Major components of the residues in maize forage were imazamox (18–31% TRR) and CL 263284, (12–23% TRR). In fodder, CL 263284 was present at 15% TRR, < 1–7% TRR in the other components. In grain, major components were CL 263284 (20% TRR) and CL 189215 (15% TRR) with imazamox at low level, < 5% TRR.

The metabolic fate of imazamox on imidazolinone-tolerant spring wheat was investigated under indoor and outdoor conditions. Radiolabelled imazamox, [pyridine-6-^{14}C]-imazamox or [imidazolinone-5-^{14}C, 3-^{15}N]-imazamox was post-emergence applied at rates of 0.14 or 0.76 kg ai/ha. Residues in forage (8–28 DAT) ranged from 0.10 mg eq./kg to 1.6 mg eq./kg. In straw and grain (62–70 DAT), residues were ranged in 0.16–3.2 mg eq./kg and 0.067–1.4 mg eq./kg, respectively. The 53–93% TRR in all wheat samples was extracted with methanol and water or acidic aqueous methanol (53% in straw).

Major components of the residues in spring wheat forage were imazamox (42–67% TRR) and CL 263284 (10–18% TRR). In straw, CL 263284 was most abundant (13–38% TRR). The other

components, imazamox (8–10% TRR), CL 189215 (3–15% TRR) and CL 312622 (6–17% TRR) were found at lower levels. In wheat grain, imazamox was most abundant component (40–70% TRR, 0.027–1.1 mg eq./kg); CL 263284 (7–10% TRR) and CL 189215 (3–4% TRR) were present at relatively low levels and CL 312622 was not detected.

The key step of the metabolism of imazamox in plant was the cleavage of the methyl ether group (demethylation) resulting in metabolite CL 263284. Subsequently, oxidation of the hydroxyl group of CL 263284 generated the dicarboxylic acid metabolite CL 312622, while glycosylation led to the glucose conjugate CL 189215.

In conclusion, in the edible portions of most treated food crops harvested at maturity, no or a very small residue of imazamox or its metabolites are expected to be found. However, for certain crops such as wheat, it is considered that the residues may be detected in wheat grain. In animal feed crops, imazamox, CL 263284, CL 189215 and CL 312622 are expected to be found above the LOQ.

Environmental fate

The Meeting received information on aerobic soil metabolism, photodegradation on the soil surface, hydrolysis and residues in succeeding crops.

Aerobic soil metabolism

Imazamox applied in sandy loam soil degraded rapidly. In three studies, half-life of imazamox was 28–38 days in sandy loam soil treated with imazamox at 0.050–0.10 kg ai/ha. However, in another study, half-life of imazamox treated in combinations of soil type (silty clay loam and silt loam) and temperature (10 °C and 20 °C) was in a range of 12–207 days (DT_{90}, 39–687 days). Imazamox was shown to be moderately persistent in aerobic soil. In all the studies, dicarboxylic acid (CL 312622) and hydroxy acid (CL 354825) metabolites were the major residue components. The parent and both metabolites were ultimately mineralized to CO_2.

Residues in succeeding crops

In a confined rotational crop study, [pyridine-6-^{14}C]-imazamox was applied once to soya beans at the 4–6 leaf stage, in a sandy loam soil, at a rate of 0.072 kg ai/ha. One-hundred days after treatment, the soya bean crop was harvested. On the day of harvest, winter wheat was seeded into a subplot (100-day plant-back interval; PBI) of the treated field. Maize, radish, and lettuce were seeded into separate subplots at a 268-day PBI. Total radioactive residues were < 0.01 mg eq./kg in all rotational crops.

In another study, radiolabelled imazamox, either [imidazolinone-5-^{14}C, 3-^{15}N]-imazamox or [pyridine-3-^{14}C, imidazolinone-3-^{15}N]-imazamox were applied to bare sandy loam soil in plastic container followed by sowing spinach, white radish and spring wheat at PBIs of 1 month, 4 month and 1 year.

In spinach and white radish, residues were < 0.01 mg eq./kg at any PBIs. However, in spring wheat samples (forage, hay, straw, grain), total residues (TRR) were: 0.008–0.078 mg eq./kg at one month PBI (grain, 0.032–0.053 mg eq./kg), 0.004–0.132 mg eq./kg at four month PBI (grain 0.019–0.035 mg eq./kg) and < 0.001–0.052 mg eq./kg at one year PBI (grain, 0.002–0.004 mg eq./kg). In wheat grain at one month PBI, residues of imazamox and CL 263284 were 0.001–0.005 mg eq./kg.

In conclusion, residues of imazamox and the metabolites are expected to be less than 0.01 mg eq./kg in succeeding crops.

Photodegradation

Soil photolysis of [pyridine-6-^{14}C]-imazamox was studied in a sandy loam soil, surface treated at a rate equivalent to 0.10 kg ai/ha and exposed for 30 days to artificial sunlight. The 92% imazamox at time-0 decreased to 74% imazamox at the end of the 30 days irradiation. A half-life of imazamox was calculated to be 65 days. A degradate, dicarboxylic acid CL 312622 (2% TAR in dark control), was formed at about 14% of the total residues after 30 days irradiation.

Imazamox is photodegraded with a half-life of 65 days on the surface of soil. Any degradate specific for photolysis was not found.

Hydrolysis

Imazamox is used for rice production. In the hydrolysis study under different pH conditions, imazamox was stable at pH 4, pH 7 and pH 9.

Methods of analysis

The Meeting received description and validation data for analytical methods for residue analysis of imazamox and its metabolites in various plant and animal commodities.

In general, the methods involved extraction of residues with acidic methanol-water solution, clean-up procedure by mainly solvent partitioning and solid phase extraction, and determination by HPLC-UV or LC-MS/MS. In the case of milk and fat, acetonitrile in hexane is used for the extraction step. Some methods employ capillary electrophoresis-UV or GC-NPD, in case of use of GC-NPD, where combined determination for imazamox-related residues is possible by converting to a common methylated product.

A number of specific methods for plant matrices were found suitable for analysis of imazamox, CL 263284, CL 189215 and CL 312622 with LOQ ranging 0.01–0.05 mg/kg for these analytes except that it was 0.1 mg/kg for alfalfa.

For animal matrices, one method determined by LC-MS/MS was submitted and found suitable for analysis of imazamox and CL 263284 with LOQs of 0.01 mg/kg for bovine matrices and poultry egg.

No multi-residue methods were submitted.

Stability of residues in stored analytical samples

The stability of imazamox and its metabolites during frozen storage of samples was investigated in a range of plant matrices for which supervised residue trials were submitted.

Compounds tested were imazamox, CL 263284, CL 189215 and CL 312622. Each compound was spiked to matrices at 0.1 to 1 mg/kg.

All of the compounds tested were found to be stable (> 70% remaining) at least during the storage periods tested: for imazamox, 3.6 years in soya bean (seed, forage and hay), 4 years in wheat (grain, straw, forage and hay), 2 years in maize (grain, ear and immature plant), 1.5 years in rape seed and 1.5 years in alfalfa (hay, forage and seed); for CL 263284, 3.6 years in soya bean (seed, forage and hay), 10 months in processed soya bean products, 4 years in wheat (grain, straw, forage and hay), 2 years in maize (grain, ear and immature plant) and peanut (hull and nutmeat) and 1.5 years in alfalfa (hay, forage and seed); for CL 189215, 10 months in soya bean seed and processed soya bean products, 2 years in peanut (hull and nutmeat) and 1.5 years in alfalfa (hay, forage and seed); and for CL 312622, 1.5 years in alfalfa (hay, forage and seed).

Definition of the residue

In metabolism studies of imazamox and the metabolites (CL 263284, CL 312622) in goats and hens, the compounds were mostly excreted (85–106%) unchanged through urine and faeces. In hen, no detectable residues were found in eggs or edible tissue. In goat, there were no detectable residues in milk and tissues except kidney. Residue in the kidney was predominantly the administered parent. Therefore, no residues of imazamox and related compounds are expected in livestock tissues, milk or eggs at expected livestock dietary burdens of less than 3 ppm.

The log P_{ow} for imazamox is 0.73 suggesting imazamox residues are not fat soluble.

In confined crop rotation studies, any imazamox related residues in succeeding crops were not detected above more than 0.01 mg/kg.

In primary crops, imazamox is either not found or found at very low levels (< 30% TRR, < 0.01 mg eq./kg) in non-tolerant food commodities and imidazolinone-tolerant food commodities (tolerant rape seeds, soya bean seeds, pea seeds, alfalfa, tolerant maize seeds). Imidazolinone-tolerant wheat grain, is the only food commodity, where parent is found at significant levels (40–74% TRR, 0.027–1.1 mg eq./kg). Among the metabolites found, CL 263284 is the most predominant metabolite, although present at low levels in food commodities (up to 31% TRR, maximum 0.092 mg eq./kg). In supervised trials imazamox and CL 263284 are found at levels above the LOQ in food commodities (lentil seeds, sunflower seeds). In feed commodities (tolerant rape forage or straw, soya forage or straw, pea forage or hay, alfalfa forage or hay, tolerant maize forage or fodder, wheat forage, hay or straw), imazamox (1.5–67% TRR, < 0.01–5.0 mg eq./kg) and CL 263284 are more prominent (8–54% TRR, < 0.01–1.9 mg eq./kg).

Parent and CL 263284 are the only two compounds expected to be found in food commodities. Metabolite CL 263284 can also arise in plant commodities as a result of treatment with imazapic, another imidazolinone herbicide. Since CL 263284 cannot be seen as a marker for imazamox, the Meeting decided to define the residue for enforcement/monitoring as parent only.

Parent and CL 263284 are considered relevant for dietary risk assessment as the toxicity of CL 263284 is similar to that of imazamox in rat. Therefore, the Meeting decided to define the residue for dietary risk assessment as the sum of parent and CL 263284, expressed as imazamox.

The Meeting agreed the following residue definitions:

Definition of the residue for plant and animal commodities for compliance with the MRL: imazamox.

Definition of the residue for plant and animal commodities for the estimation of dietary intake: sum of imazamox and 5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazazolin-2-yl) nicotinic acid (CL 263284), expressed as imazamox.

Residue is not fat-soluble.

Should imazamox be used on crops other than cereals, pulses and oilseeds, the residue definition may need to be revised.

Results of supervised residue trials on crops

The Meeting received supervised trial data for imazamox on legume vegetables (beans and peas), pulses (bean, pea, lentil and soya bean), cereals (rice and wheat), oil seed crops (peanut, rape and sunflower) and alfalfa.

In below, a maximum residue level was estimated based on residue concentration of imazamox. Total residue means sum of parent compound and CL 263284. Concentration of CL 263284 was expressed as parent equivalents (conversion factor, 1.048).

Legume vegetables

Beans

Residue trials on beans were performed in Denmark, Germany, France, the UK, Greece, Italy, Spain and the USA. The GAP for beans in Chile is a single early post-emergent application at 0.056 kg ai/ha with no PHI specified.

In six trials on French beans conducted in Italy (1×0.050 kg ai/ha at post-emergence) matching the Chile GAP, residues in bean pods with seeds were: parent < 0.05 (6) mg/kg and metabolite < 0.05 (6) mg/kg. Total residues in the bean pods with seeds were (n=6): < 0.1 (6) mg/kg.

In other six trials from USA (1×0.050 kg ai/ha at post-emergence) matching the Chile GAP, residue concentrations of imazamox (only measured) were < 0.05 (6) mg/kg in pods with seeds of snap beans.

Three trials conducted in France, the UK and Italy involved an earlier application timing and a higher rate (1×0.075 kg ai/ha at pre-emergence). Residues in pods with seeds of common bean (flageolet bean) were: parent < 0.01 (3) mg/kg, metabolite < 0.01 (3) mg/kg. Total residues in the bean pods with seeds were < 0.02 (3) mg/kg.

The Meeting considered that there was no expectation of residues above LOQ in bean pods with seeds. The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0 mg/kg for beans, except broad bean and soya bean (green pods and immature seeds).

Peas

Residue trials on peas were performed in Italy, France, Spain, the UK, Germany and the USA. The GAP for peas in France is for a single pre-emergent application post sowing per every 2 years, treating on soil at 0.075 kg ai/ha with a PHI of 63 days.

In nine trials conducted in Italy and France approximating the French GAP (1×0.068–0.075 kg ai/ha at pre-emergence, 70–110 day PHI), residues in pea seeds were: parent < 0.05 (9) mg/kg and metabolite < 0.05 (9) mg/kg. Total residues in pea seeds were (n=9): < 0.1 (9) mg/kg.

Another ten trials were conducted in Italy and Spain with post-emergence treatments at 0.050–0.052 kg ai/ha in eight trials and 0.073–0.075 kg ai/ha in two trials. The total residues were all < 0.1 (10) mg/kg.

The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0 mg/kg for peas, shelled (succulent seeds).

Pulses

Bean (dry)

Residue trials on dry beans were performed in France, Germany, Netherlands, the UK, Italy, Greece, Spain and the USA. The GAP for dry bean in the UK is a single pre-emergence application to soil at a rate of 0.075 kg ai/ha.

In four trials from France, the UK and Italy (1×0.075 kg ai/ha at pre-emergence, 91–108 day PHIs), approximating the UK GAP, residues in common bean seeds were: parent < 0.01 (4) mg/kg and metabolite < 0.01 (4) mg/kg. Total residues in seed of common beans were < 0.02 (4) mg/kg. In these trials, the total residues in whole plants harvested early (PHIs of 32–48 days) were < 0.02 mg/kg.

In two trials from the USA applied at a post-emergence timing and at a rate comparable to the UK GAP (1×0.070 kg ai/ha, 61–98 day PHIs), residue concentrations of the parent compound were also below LOQ, < 0.05 (2) mg/kg.

Pea (dry)

The GAP for dry peas in France is a single pre- or post-emergence application every 2 years at a rate of 0.075 kg ai/ha with a PHI of 63 days. A total of ten trials were conducted in France at the GAP rate (1×0.075 kg ai/ha at pre-emergence) but with longer PHIs (106–129 days). The residues measured for each compound or combined total residue were: parent < 0.05 (5) mg/kg, metabolite < 0.05 (5) mg/kg and total residue < 0.1(10) mg/kg.

Lentil (dry)

Residue trials for lentil were available from Canada and the USA. In Canada, imazamox is approved for use in imidazolinone-tolerant lentils with a single early post-emergent application at a rate of 0.020 kg ai/ha (plus adjuvant) with a PHI of 60 days.

In a total of fourteen trials (1×0.015–0.021 kg ai/ha at post- or early post-emergent timings and a 60 day, or shorter, PHI (without use of an adjuvant), residues in imidazolinone-tolerant lentils

were: imazamox < 0.05 (9), 0.056, 0.060, 0.065, 0.12, 0.12 mg/kg and metabolite < 0.05 (14) mg/kg. Total residues were: < 0.1 (9), 0.11 (3), 0.17, 0.17 mg/kg.

Soya bean (dry)

Residue trials on soya bean were performed in France, Germany, Italy and Canada. The GAP for soya bean in Brazil is for a single post-emergence application at a rate of 0.049 kg ai/ha (plus adjuvant) with a PHI of 70 days.

In nine trials conducted in France, Germany and Italy (1×0.042–0.045 kg ai/ha at post- or early post-emergence, 69–104 days of PHI, with or without adjuvant) approximating the Brazilian GAP, residues in soya bean seeds were: imazamox < 0.01 (7) mg/kg and metabolite < 0.01 (7) mg/kg. Total residues were < 0.02 (7) mg/kg. Of which five trials showed no effect of adjuvant on the level of residue in dry seed.

In two trials conducted in Canada at an exaggerated rate of 0.070–0.071 kg ai/ha (single post-emergence application, 106–112 day PHIs), total residues were all < 0.05 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0 mg/kg for bean and pea in dry, respectively.

For lentil (dry), the Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.1 mg/kg.

For soya bean (dry), the Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0 mg/kg.

Cereals

Rice

Residue trials on imidazolinone-tolerant rice were performed in Italy and Spain. The use pattern in Spain is twice post-emergence treatment at a rate of 0.035 kg ai/ha with an interval of 14–21 days and no PHI, applying on imidazolinone-tolerant rice plant in water or dry planted.

In a total of six trials on imidazolinone-tolerant rice (2×0.035–0.038 kg ai/ha at BBCH 13–14 and BBCH 25–31, 10–12 day interval) from Italy and Spain matching the Spain GAP, residues in rice grain were: imazamox < 0.01 (6) mg/kg and metabolite < 0.01 (3), 0.02, 0.03 (2) mg/kg. Total residues were: < 0.02 (3), 0.030, 0.040 (2) mg/kg.

The Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0.025 mg/kg for rice.

Wheat

Residue trials on imidazolinone-tolerant wheat were performed in France, Italy, Canada and the USA. The GAP for wheat (imidazolinone-tolerant) in Argentina is a single early post-emergence application at 0.070 kg ai/ha with no PHI.

In a total of twelve trials from France (eight trials), Italy (four trials) and USA (one trial) (1×0.067–0.075 kg ai/ha at post-emergence) matching the GAP, residues in imidazolinone-tolerant wheat grain were: imazamox < 0.05 (13) mg/kg and metabolite < 0.05 (13) mg/kg. Total residues were < 0.1 (13) mg/kg.

The Meeting considered that there is no expectation of residues above the LOQ for wheat grain and agreed to estimate a maximum level of 0.05* mg/kg and an STMR of 0.1 mg/kg for wheat.

Oilseeds

Peanuts

Five trials were conducted in Brazil with a single post-emergence application (BBCH 71–79) at a rate of 0.056 kg ai/ha with a PHI of 43–50 days without use of an adjuvant, matching the GAP for peanut in South Africa (1×0.048 kg ai/ha plus adjuvant) at post-emergence with a PHI 50 days. The residues in peanut kernels were: imazamox < 0.01 (5) mg/kg and metabolite < 0.01 (5) mg/kg. Total residues were < 0.02 (5) mg/kg.

Although only a relatively small number of trials were available, there is no expectation of residues above the LOQ in peanuts. The Meeting estimated a maximum residue level of 0.01* mg/kg and an STMR of 0 mg/kg.

Rape seed

Residue trials on imidazolinone-tolerant oilseed rape were conducted in France, Germany, the UK and Canada and Argentina. The GAP for imidazolinone-tolerant rape in Chile is a single early post-emergence application at a rate of 0.056 kg ai/ha with no PHI. In six trials from Germany, France and Argentina (1×0.050 kg ai/ha at post-emergence) matching the Chile GAP, residues in rape seed (imidazolinone-tolerant variety) were: imazamox < 0.01 (2), < 0.05 (4) mg/kg and metabolite < 0.01 (2), < 0.05 (4) mg/kg. Total residues were < 0.02 (2), < 0.1 (4) mg/kg.

In another eighteen trials from France, Germany and the UK, an exaggerated rate of 0.073–0.079 kg ai/ha did not lead to above LOQ (< 0.05 mg/kg) for total residues in seeds of imidazolinone-tolerant oilseed rape.

The Meeting considered that there is no expectation of residues above the LOQ in rape seeds.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR of 0 mg/kg for rape seed.

Sunflower seed

Residue trials on imidazolinone-tolerant sunflower were conducted in France, Germany, UK, Italy, Spain, Argentina, Canada and USA. In Canada, critical GAP for sunflower (imidazolinone-tolerant) is a single early post-emergence application at a rate of 0.015 kg ai/ha with a specified PHI of 60 days and an addition of adjuvant.

A total of six trials conducted in Canada and USA (1×0.015–0.019 kg ai/ha at PHIs of 58–68 days with or without adjuvant) approximated the Canadian GAP. The residues in seeds of imidazolinone-tolerant sunflower were: imazamox < 0.05 (2), 0.05 (2), 0.07, 0.14 mg/kg and metabolite < 0.05, 0.06, 0.10, 0.16, 0.17, 0.19 mg/kg. Total residues were: < 0.1, 0.13, 0.15, 0.22, 0.25, 0.30 mg/kg.

The Meeting estimated a maximum residue level of 0.3 mg/kg and an STMR of 0.19 mg/kg for sunflower seed.

Legume animal feeds

Alfalfa

Residue trials on alfalfa were performed in Greece, Spain and USA. The GAP for alfalfa in France is a single post-emergence application at a rate of 0.067 kg ai/ha with a PHI of 30 days.

In a total of seven trials from Greece, Spain and USA (1×0.067–0.075 kg ai/ha at PHIs of 26–30 days) matching the French GAP, residues in alfalfa forage were as received basis: imazamox < 0.1 (7) mg/kg and metabolite < 0.1 (7) mg/kg. Total residues in alfalfa forage were < 0.2 (7) mg/kg. For alfalfa hay, residues were as received basis: imazamox < 0.1 (5) mg/kg and metabolite < 0.1 (3), 0.19, 0.29 mg/kg. Total residues were < 0.2 (3), 0.32, 0.41 mg/kg in hay.

The Meeting estimated a median residue of 0 mg/kg and a highest residue of 0.2 mg/kg for alfalfa forage.

For alfalfa hay, the Meeting estimated a maximum residue level of 0.1* mg/kg, a median residue of 0.20 mg/kg and a highest residue of 0.41 mg/kg.

Pea vines (green) and pea fodder

Five trials approximating the French GAP for pea (single pre-emergence post-sowing per every 2 years at 0.075 kg ai/ha) were conducted in France with PHIs of 88–110 days. Residues in pea vines, (pods + haulm) on an as received basis, were: imazamox < 0.05 (5) mg/kg and metabolite < 0.05 (5) mg/kg. Total residues in pea vines were < 0.1 mg/kg.

Ten trials approximating the French GAP for field pea (single pre- or post-emergence application per every 2 years at a rate of 0.075 kg ai/ha) were conducted in France with PHIs of 106–120 days. Residues in pea fodder (straw or pods + haulm) on an as received basis, were: imazamox < 0.05 (5) mg/kg and metabolite < 0.05 (4), 0.17 mg/kg. Total residues were < 0.05 (5), < 0.1(4) and 0.22 mg/kg.

The Meeting estimated a median residue of 0.1 mg/kg and a highest residue of 0.1 mg/kg for pea vines. For pea fodder, the Meeting estimated a maximum residue level of 0.05* mg/kg, a median reside of 0.075 mg/kg and a highest residue of 0.22 mg/kg.

Soya bean, forage and fodder

Four trials were conducted in France and Germany approximating the Brazil GAP for soya bean (1×0.049 kg ai/ha). Residues in soya bean forage harvested at 27–28 PHIs were as received basis: imazamox < 0.01 (4) mg/kg and metabolite < 0.01 (4) mg/kg. Total residues were < 0.02 (4) mg/kg. For fodder, residues were as received basis: imazamox < 0.01 (4) mg/kg and metabolite < 0.01 (2), 0.01, 0.05 mg/kg. Totals residues were < 0.02 (2), 0.02, 0.06 mg/kg in soya bean fodder.

For soya bean forage, the Meeting estimated a median residue of 0.02 mg/kg and a highest residue of 0.02 mg/kg.

For soya bean fodder, the Meeting estimated a maximum residue level of 0.01* mg/kg, a median residue of 0.02 mg/kg and a highest residue of 0.06 mg/kg.

Forage and fodder of cereal grains and grasses

Rice straw and fodder

In six trials conducted in Italy and Spain matching the Spain GAP (post-emergence treatment on imidazolinone-tolerant rice plant, water or dry planted, 2× 0.035 kg ai/ha, 14–21 day interval), residues in imidazolinone-tolerant rice straw (77–90 day PHIs) were as received basis: imazamox < 0.01 (6); metabolite < 0.01 (3), 0.01, 0.02 (2) mg/kg. Total residues were < 0.02 (3), 0.02, 0.03 (2) mg/kg in straw.

The Meeting estimated a maximum residue level of 0.01* mg/kg, a median residue of 0.02 mg/kg and an highest residue of 0.03 mg/kg in rice straw.

Wheat forage and straw

Trials on imidazolinone-tolerant wheat were conducted in Canada approximating the GAP for imidazolinone-tolerant wheat in Canada (single early post-emergence application at 0.020 kg ai/ha). In Canada, grazing and cutting for hay are not permitted within 4 days and 42 days of application, respectively. Five trials for forage (7, 14 day PHIs) and four trials (42, 56 day PHIs) for hay and five trials (72–90 PHIs) for straw matched the Canadian GAP on feedstuffs.

Residues in imidazolinone-tolerant wheat forage were as received basis: imazamox < 0.05 (2), 0.05, 0.13, 0.15 mg/kg; metabolite < 0.05 (5) mg/kg. Total residues were < 0.1 (2), 0.10, 0.20, 0.23 mg/kg.

Residues in imidazolinone-tolerant wheat hay were as received basis: imazamox < 0.05 (4) mg/kg; metabolite < 0.05 (4) mg/kg. Total residues were < 0.1 (4) mg/kg.

Residues in imidazolinone-tolerant wheat straw were as received basis: imazamox < 0.05 (5) mg/kg; metabolite < 0.05 (5) mg/kg. Total residues were < 0.1 (5) mg/kg.

For wheat forage, the Meeting estimated a median residue of 0.1 mg/kg and a highest residue of 0.23 mg/kg.

For wheat hay and straw, the Meeting estimated a maximum residue level of 0.05* mg/kg, a median residue of 0 mg/kg and a highest residue of 0.1 mg/kg.

Miscellaneous Fodder and Forage crops

Rape seed forage

In four residue trials on imidazolinone-tolerant oilseed rape conducted in France and Germany ,matching the GAP of Chile for oilseed rape (imidazolinone-tolerant rape, a single early post-emergence application, 0.056 kg ai/ha), residues in whole plants without roots (12–19 day PHIs) on an as received basis were: imazamox, 0.05, 0.12, 0.14 0.19mg/kg; metabolite 0.52, < 0.05, 0.21, 0.28 mg/kg. Total residues were: 0.10, 0.33, 0.42 and 0.71, mg/kg.

The Meeting estimated a median residue of 0.38 mg/kg and an highest residue of 0.71 mg/kg for rape forage.

Fate of residues during processing

High temperature hydrolysis

The hydrolysis of [pyridine-3-^{14}C, imidazolinone-3-^{15}N]-imazamox was investigated in aqueous buffer solutions. After pasteurization (20 minutes at 90 °C, pH 4), baking/brewing/boiling (60 minutes at 100 °C, pH 5) and sterilization (20 minutes at 120 °C, pH 6), 98–104% of the applied radioactivity remained in the test solutions, where the detectable radioactive component was unchanged imazamox only. Imazamox was stable under such simulated processing conditions.

Processing

Information on processing of wheat and sunflower seed were available for an estimation of maximum residue level and STMR-P for the processed product. Estimated processing factors, maximum residue levels and STMR-Ps are summarized below. A maximum residue level and STMR-P value for processed product were calculated with imazamox residue and STMR value (total residue of imazamox and CL 263284), respectively.

RAC and processed product	RAC		Pf, best estimate (individual Pf)		Processed product	
	Estimated maximum residue level (mg imazamox/kg)	STMR (mg total/kg)	Imazamox	Total residue	Estimated maximum residue level (mg imazamox/kg)	STMR-P total residue (mg total/kg)
Wheat	0.05*	0.1				
Wheat germ			2.7	2.2	0.1	0.22
Wheat bran, unprocessed			3.9	3.4	0.2	0.34
Wheat flour			1.2	1.2	0.06	0.12
Wheat asp. grain fraction			1.0	1.0		0.10
Wheat by products (middling, shorts)			2.4 (1.6, 3.2)	2.1 (1.5, 2.6)		0.21
Sunflower seed	0.3	0.19				
Sunflower seed meal			2.3	2.5 (1.9, 3.0)		0.48
Sunflower refined oil				< 0.5 (< 0.2, < 0.5)		0.095

The Meeting estimated maximum residue levels of 0.1 mg/kg for wheat germ and 0.2 mg/kg for wheat bran, unprocessed.

Residues in animal commodities

Farm animal feeding studies

Information on farm animal feeding studies was not submitted.

Estimation of dietary burdens

Dietary burden calculations for beef cattle and dairy cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2009 edition of the FAO Manual.

Summary of livestock dietary burden (ppm of dry matter diet)

	US-Canada		EU		Australia		Japan	
	max	mean	max	mean	max	mean	max	mean
Beef cattle	0.230	0.178	0.821	0.463	2.37[a]	1.27[c]	0.217	0.193
Dairy cattle	0.670	0.404	0.0.749	0.0433	1.50[b]	0.765[d]	0.256	0.197
Poultry broiler	0.278	0.278	0.179	0.179	0.200	0.200	0.052	0.023
Poultry layer	0.278	0.278	0.75[e]	0.478[f]	0.188	0.188	0.072	0.072

[a] Highest maximum beef cattle dietary burden suitable for MRL estimates for mammalian meat
[b] Highest maximum dairy cattle dietary burden suitable for MRL estimates for milk
[c] Highest mean beef cattle dietary burden suitable for STMR estimates for mammalian meat
[d] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk
[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.
[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs

Animal commodity maximum residue levels

Maximum estimated dietary burdens for beef cattle and dairy cattle were 1.4 and 1.1 ppm, respectively. These dietary burdens were similar to the dose rates in the metabolism studies in lactating goats (< 0.01 mg eq./kg at 2.1 ppm for imazamox; < 0.01 mg eq./kg at 2.3 ppm for CL 263284). As no residues in cattle meat and milk are expected, the Meeting estimated a maximum residue level of 0.01* mg/kg and HR and STMR of 0 in each animal commodity: milk, meat (mammalian except marine mammals) and edible offal and fat.

In poultry, the maximum dietary burden, 0.75 ppm was 3 times lower than dose rates in metabolism studies in laying hens (< 0.01 mg eq./kg at 2.1 ppm for imazamox and CL 263284 each). The Meeting estimated a maximum residue level of 0.01* mg/kg and HR and STMR of 0 for poultry meat, poultry edible offal, poultry fat and eggs.

RECOMMENDATIONS

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

Definition of the residue for plant and animal commodities for compliance with the MRL: *imazamox*

Definition of the residue for plant and animal commodities for estimation of dietary intake: *sum of imazamox and 5-(hydroxymethyl)-2-(4-isopropyl-4-methyl-5-oxo-2-imazazolin-2-yl) nicotinic acid (CL 263284), expressed as imazamox*

Residue is not fat soluble.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
VP 0061	Beans, except broad bean and soya bean (green pods and immature seeds)	0.05*		0	
VP 0064	Peas, shelled (succulent seeds)	0.05*		0	
VD 0071	Bean (dry)	0.05*		0	
VD 0072	Pea (dry)	0.05*		0	
VD 0533	Lentil (dry)	0.2		0.10	
VD 0541	Soya bean (dry)	0.01*		0	
GC 0649	Rice	0.01*		0.025	
GC 0654	Wheat	0.05*		0.1	
CF 1210	Wheat germ	0.1		0.22	
CM 0654	Wheat bran, unprocessed	0.2		0.34	
CF 1211	Wheat flour			0.12	
SO 0697	Peanut	0.01*		0	
SO 0495	Rape seed	0.05*		0	
SO 0702	Sunflower seed	0.3		0.19	
AL 1020	Alfalfa fodder	0.1*		0.20	0.41
AL 0072	Pea hay or fodder	0.05*		0.075	0.22
AL 0541	Soya bean fodder	0.01*		0.02	0.06
AS 0649	Rice straw and fodder, dry	0.01*		0.02	0.03
AS 0654	Wheat straw and fodder, dry	0.05*		0	0.1
MO 0105	Edible offal (mammalian)	0.01*		0	0
PE 0112	Eggs	0.01*		0	0
MM 0095	Meat (from mammals other than marine mammals)	0.01*		0	0
MF 0100	Mammalian fats (except milk fats)	0.01*		0	0
ML 0106	Milks	0.01*		0	0
PM 0110	Poultry meat	0.01*		0	0
PF 0111	Poultry fats	0.01*		0	
PO 0111	Poultry, Edible offal of	0.01*		0	0
	Sunflower seed refined oil			0.095	

For calculating animal dietary burden

Commodity		Median residue or STMR-P (mg/kg)	Highest residue or HR-P (mg/kg)
CCN	Name		
AL 1021	Alfalfa forage (green)	0	0.2
AL 0528	Pea vines (green)	0.1	0.1
AL 1265	Soya bean forage (green)	0.02	0.02
	Wheat forage	0.1	0.23
AV 0495	Rape seed forage	0.38	0.71
	Wheat aspirated grain fraction		0.10
	Wheat by products (middling, shorts)		0.21
	Sunflower seed meal		0.48

DIETARY RISK ASSESSMENT

Long-term intake

The ADI for imazamox is 0–3 mg/kg bw. The International Estimated Daily Intakes (IEDIs) for imazamox were estimated for the 17 GEMS/Food Consumption Cluster Diets using the STMR or STMR-P values estimated by the present JMPR. The results are shown in Annex 3 of the 2014 JMPR Report. The IEDIs were 0% of the maximum ADI. The Meeting concluded that the long-term intake

of residues of imazamox from uses considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD for imazamox is 3 mg/kg bw. The International Estimate of Short Term Intakes (IESTIs) for imazamox were calculated for the food commodities for which STMRs or HRs were estimated by the present Meeting and for which consumption data were available. The results are shown in Annex 4. The IESTIs were 0% of the ARfD for children and the general population.

The Meeting concluded that the short-term intake of residues of imazamox from uses considered by the present Meeting are unlikely to present a public health concern.

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2013/3000444	Dantas, C & Cardoso Almeida, B de	2013 a	Estudo de residuos de Bentazon e Imazamox em amendoim (graos), apos tratamento com Amplo, em condicoes de campo no Brasil BASF SA, Guaratingueta, Brazil Unpublished
2004/1025824	Dubey, L	2004 a	Determination of Imazamox residues in rice samples after application of Imazamox on rice fields in Italy Battelle, Carouge, Switzerland Unpublished
2004/1026636	Dubey, L	2004 b	Amendment no. 1 to the final report: Determination of Imazamox residues in rice samples after application of Imazamox on rice fields in Italy Battelle, Carouge, Switzerland Unpublished
2013/1003728	Erdmann, HP	2013 a	Study on the residue behaviour of BAS 720 H (Imazamox) in sunflower after application of either BAS 720 AM H or BAS 720 06 H under field condition in Northern France, Italy, Spain and Germany, 2012 Agro-Check Dr. Teresiak & Erdmann GbR, Lentzke, Germany Fed.Rep. Unpublished

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2012/1044751	Fabian, E & Landsiedel, R	2012 a	^{14}C-BAS 720 H—Study on the kinetics in rats BASF SE, Ludwigshafen/Rhein, Germany Fed.Rep. Unpublished
2012/1044751	Fabian, E & Landsiedel, R	2012 b	^{14}C-BAS 720 H—Study on the kinetics in rats BASF SE, Ludwigshafen/Rhein, Germany Fed.Rep. Unpublished
ID-411-001	Fischer, J	1995 a	Oral LD50 study in albino rats with AC 299, 263 technical—Amendment 1 included American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-412-001	Fischer, JE	1995 a	Dermal LD50 study in albino rabbits with AC 299, 263 technical—Amendment 1 and 2 included American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-415-001	Fischer, JE	1995 b	Skin irritation study in albino rabbits with AC 299, 263 technical—Amendment 1 included American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-415-002	Fischer, JE	1996 a	Eye irritation study in albino rabbits with AC 299, 263 technical—Amendment 2 included American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-420-002	Fischer, JE	1996 b	A 28-day dietary toxicity study in albino rats with AC 299, 263 American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-425-001	Fischer, JE	1995 c	AC 299, 263: 13-week dietary toxicity study in the albino rat—Amendment 1 at begin of the document included American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-427-002	Fischer, JE & Hess, FG	1995 b	Chronic dietary toxicity and oncogenicity study with AC 299,263 in the albino rat (Volume I–VI) American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-029	Fletcher, JS	2002 a	BAS 720 H (CL 299263): Laboratory validation of LC/MS determinative and LC/MS/MS confirmatory method M 3515 for the determination of BAS 720 H and CL 263284 residues in plant matrices BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-326-024	Fletcher, JS	2001 a	CL 299263 (Imazamox): Freezer storage stability of CL 299263, CL 263284 , CL 189215 and CL 312622 residues in alfalfa seed, forage and hay BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-790-014	Fletcher, JS	2001 b	CL 299263 (Imazamox): Freezer storage stability of CL 299263, (and its metabolites) CL 263284, CL 189215 and CL 312622 residues in alfalfa seed, forage and hay—Report amendment No. 01 BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-432-001	Foss, JA	1994 b	An oral developmental toxicity (embryo-fetal toxicity / teratogenicity) study with AC 299, 263 in rats Argus Research Laboratories Inc., Horsham PA, United States of America Unpublished
2010/1057139	Freitas & Guimaraes, S	2010 a	Study of Imazapyr and Imazamox residues in sunflower (seed), after treatment with Sweeper 70 DG and Clearsol DF, under field conditions in Argentina BASF SA, Guaratingueta, Brazil Unpublished
2010/1057139	Freitas & Guimaraes, S	2010 a	Study of Imazapyr and Imazamox residues in sunflower (seed), after treatment with Sweeper 70 DG and Clearsol DF, under field conditions in Argentina BASF SA, Guaratingueta, Brazil Unpublished
2013/1085609	Funk, D & Possienke, M	2013 b	Nachbaustudie mit ^{14}C-BAS 720 H—Confined rotational crop study with ^{14}C-BAS 720 H BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2013/1233155	Funk, D & Taraschewski, I	2013 a	Comparative in-vitro-metabolism with ^{14}C-BAS 720 H BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
ID-452-001	Futagawa, H	1995 a	AC 299263 technical: General pharmacological study The Institute of Environmental Toxicology, Kodaira-shi Tokyo 187, Japan Unpublished
2012/1084183	Gabriel, EJ	2013 b	Study on the residue behaviour of Imazamox in sunflower after treatment with BAS 720 06 H and BAS 160 00 S under field conditions in Germany, the United Kingdom, southern France and Italy, 2011 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. Unpublished
2012/1272623	Gabriel, EJ	2013 c	Study on the residue behaviour of Imazamox (BAS 720 H) in rice after treatment with BAS 720 06 H, applied with the adjuvant Dash (BAS 160 00 S) under field conditions in Italy and Spain, 2012 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. Unpublished
2012/1278352	Gabriel, EJ	2013 a	Study on the residue behaviour of BAS 720 H (Imazamox) in rice after treatment with BAS 720 06 H under field conditions in Italy and Spain, 2011 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep.

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			Unpublished
ID-640-002	Gatterdam, PE	1994 b	Confined accumulation study of carbon-14 labelled CL 299, 263 using radishes, corn, lettuce and wheat as rotational crops American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-416-001	Glaza, SM	1992 a	Dermal sensitization study with AC 299, 263 in guinea pig Hazleton Laboratories America Inc., Madison WI, United States of America Unpublished
ID-416-002	Glaza, SM	1996 a	Dermal sensitization study of AC 299, 263 in guinea pigs—Maximization test Corning Hazleton Inc., Madison WI, United States of America Unpublished
2012/7004251	Gooding, R	2013 a	Validation of BASF analytical method D1102: Method for determination of BAS 720 H and Its metabolites CL 312622 and CL 354825 residues in soil using LC-MS/MS BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
2013/7002842	Gooding, RF	2013 a	Validation of BASF analytical method D0303: Method for the determination of BAS 720 H (CL 299263) and its metabolite CL 263284 in animal matrices using LC-MS/MS BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
2012/1064722	Grosshans, F *et al.*	2012 b	Metabolism of ^{14}C-Imazamox in wheat BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
ID-731-015	Hallman, DC	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 70DG herbicide in Iowa American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-016	Hallman, DC	1999 b	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 1AS herbicide in California American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-017	Hallman, DC	1999 c	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after early season treatment with AC 299263 1AS herbicide in Pennsylvania American Cyanamid Co., Princeton NJ, United States of America Unpublished
2011/1286214	Hassink, J	2012 a	Imazamox: Hydrolysis at 90 °C, 100 °C and 120 °C BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
ID-731-014	Higham, JW	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 263284, CL 189215 and CL 312632 residues in newly-seeded alfalfa after treatment with AC 299263 1AS herbicide in Washington State American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-022	Higham, JW	1999 b	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in newly-seeded alfalfa after treatment with AC 299263 (70DG) herbicide in Georgia American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-432-002	Hoberman, AM	1995 c	An oral developmental toxicity (embryo-fetal toxicity / teratogenicity) definitive study with AC 299, 263 in rabbits Argus Research Laboratories Inc., Horsham PA, United States of America Unpublished
ID-413-001	Hoffman, GM	1994 a	Acute inhalation toxicity study with AC 299, 263 in rats Bio/dynamics Inc., East Millstone NJ, United States of America Unpublished
ID-322-002	Holman, J	1997 a	Hydrolysis of AC 299, 263 American Cyanamid Co., Ewing NJ, United States of America Unpublished
ID-322-002	Holman, J	1997 b	Hydrolysis of AC 299, 263 American Cyanamid Co., Ewing NJ, United States of America Unpublished
ID-360-001	Humphries, K	1996 a	CL 299, 263—Spectral database (Report amendment 2) American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-440-001	Johnson, DH	1994 b	Metabolic fate of carbon-14 labelled CL 299263 in tissues and eggs of the laying hen American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-440-002	Johnson, DH	1994 a	Metabolic fate of carbon-14 labelled CL 299263 in the milk and edible tissues of the lactating goat American Cyanamid Co., Princeton NJ, United States of America Unpublished

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ID-640-005	Johnson, DH	1996 b	CL 299263: Metabolism of carbon-14 labelled CL 299263 in wheat under field conditions—Report amendment number 1 (Amendment number 1 included—in the first place) American Cyanamid Co., Princeton NJ, United States of America Unpublished
2002/5004111	Johnston, R	2003 a	The magnitude of Imazamox (BAS 720 H) and Imazapyr (BAS 693H) residues in sunflower and sunflower processed fractions BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2002/5004111	Johnston, R	2003 a	The magnitude of Imazamox (BAS 720 H) and Imazapyr (BAS 693H) residues in sunflower and sunflower processed fractions BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2002/5004111	Johnston, R	2003 a	The magnitude of Imazamox (BAS 720 H) and Imazapyr (BAS 693H) residues in sunflower and sunflower processed fractions BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2008/7008101	Johnston, RL	2009 a	Magnitude of Imazamox and Imazethapyr residues in sunflower RAC and processed fractions following applications of BAS 724 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2008/7008101	Johnston, RL	2009 a	Magnitude of Imazamox and Imazethapyr residues in sunflower RAC and processed fractions following applications of BAS 724 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2002/5004302	Jordan, JM	2003 a	Independent method validation of BASF analytical method M 3519 entitled BAS 720 H and BAS 685 H: Determination and confirmation of BAS 720 H, CL 263284, CL 189215, CL 312622, BAS 685 H, CL 288511 and CL 182704 residues in lentils BASF Agro Research RTP, Research Triangle Park NC, United States of America Unpublished
2002/5004302	Jordan, JM	2003 a	Independent method validation of BASF analytical method M 3519 entitled BAS 720 H and BAS 685 H: Determination and confirmation of BAS 720 H, CL 263284, CL 189215, CL 312622, BAS 685 H, CL 288511 and CL 182704 residues in lentils BASF Agro Research RTP, Research Triangle Park NC, United States of America Unpublished
IA-440-001	Kao, LM	1994 a	CL 263,284: Metabolism of [^{14}C] CL 263,284 in the lactating goat American Cyanamid Co., Princeton NJ, United States of America Unpublished
2013/1235040	Kapp, MD,& Landsiedel, R	2013 a	Reg.No. 4110542 (metabolite of BAS 720 H, Imazamox)—In vitro gene mutation test in CHO cells (HPRT locus assay) BASF SE, Ludwigshafen/Rhein, Germany Fed.Rep. Unpublished
ID-425-002	Kelly, CM	1994 b	90-day dietary toxicity study with AC 299, 263 in purebred beagle dogs Pharmaco LSR Inc., East Millstone NJ, United States of America Unpublished
ID-427-001	Kelly, CM	1995 c	One-year dietary toxicity study with AC 299, 263 in purebred beagle dogs (Volume I and II) Pharmaco LSR Inc., East Millstone NJ, United States of America Unpublished
ID-428-001	Kelly, CM	1995 d	An oncogenicity study with AC 299, 263 in mice (Volume I–V) Pharmaco LSR Inc., East Millstone NJ, United States of America Unpublished
ID-731-009	Kenny, S	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in newly seeded alfalfa after treatment with AC 299263 70DG herbicide in South Dakota, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-004	Kenny, SM	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after early season treatment with AC 299263 70DG herbicide in North Dakota American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-005	Kenny, SM	1999 b	CL 299263 (Imazamox): CL 299263, CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 1AS herbicide in Washington American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-006	Kenny, SM	1999 c	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 70DG herbicide in California, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished

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ID-731-007	Kenny, SM	1999 d	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues and their decline over time in established alfalfa after treatment with AC 299263 1AS herbicide in California, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-008	Kenny, SM	1999 e	CL 299263 (Imaxamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 1AS herbicide in California, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-010	Kenny, SM	1999 f	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 70DG herbicide in Indiana, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-011	Kenny, SM	1999 g	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after early season treatment with AC 299263 1AS herbicide in Indiana, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-013	Kenny, SM	1999 h	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in established alfalfa after treatment with AC 299263 1AS herbicide in Ohio, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-018	Kenny, SM	1999 i	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in newly seeded alfalfa after treatment with AC 299263 1AS herbicide in Colorado, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-020	Kenny, SM	1999 j	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 70DG herbicide in Idaho, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-021	Kenny, SM	1999 k	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues and their decline over time in established alfalfa after early season treatment with AC 299263 70DG herbicide in Illinois, USA American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-324-003	Knoch, E	1996 a	Determination of the direct phototransformation of AC299263 in a buffered medium at pH 7 Institut Fresenius Chemische und Biologische Laboratorien GmbH, Herten, Germany Fed. Rep. Unpublished
2007/1023133	Kreke, N	2009 a	Residues at harvest of Imazamox and Bentazone in green pea (RAC seed, pods, and plant without pods) following one treatment with BAS 762 01 H (22.4/480 g/L) from eight open field trials in Northern and Southern Europe in 2007/2008 Harlan Laboratories Ltd., Itingen, Switzerland Unpublished
ID-731-012	Kukel, C	1999 a	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 1AS herbicide in Washington American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-731-019	Kukel, C	1999 b	CL 299263 (Imazamox): CL 299263, (and its metabolites) CL 189215, CL 312622 and CL 263284 residues in alfalfa grown for seed production after treatment with AC 299263 70DG herbicide in Idaho American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-435-004	Kumaroo, VP	1994 b	AC 299, 263: Test for chemical induction of chromosome aberration in cultured Chinese hamster ovary (CHO) cells with and without metabolic activation SITEK Research Laboratories, Rockville MD, United States of America Unpublished
2012/1294678	Lehmann, A	2013 a	Validation of BASF method no. L0188/01: Method for the determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2012/1294678	Lehmann, A	2013 a	Validation of BASF method no. L0188/01: Method for the determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished

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2013/1177533	Lehmann, A	2013 b	Amendment No. 1: Validation BASF No. L0188/01—Method for determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2013/1177533	Lehmann, A	2013 b	Amendment No. 1: Validation BASF No. L0188/01—Method for determination of Imazamox (BAS 720 H, Reg.No. 4096483) and its metabolites Reg.No. 4110542 (CL312622), Reg.No. 4110773 (CL263284) and Reg.No. 4110445 (CL189215) in plant matrices BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2010/1090461	Leite, R	2008 a	SOP-PA.0288_E Rev.03—Determination of Imidazolinones residues and its metabolites in several matrices and processed fractions by LC/MS/MS BASF SA, Guaratingueta, Brazil Unpublished
2011/1207286	Leite, R.,& Alves, M	2011 a	Investigation study of the storage stability of Imazapyr (BAS 693 H), Imazapic (BAS 715 H) and its metabolites CL 263,284 and CL 189,215 in soya bean and processed fractions BASF SA, Guaratingueta, Brazil Unpublished
2002/5004457	Leonard, R	2003 a	Magnitude of residue of BAS 720 H and BAS 685 H and their related metabolites residues in lentils after treatment with BAS 724 00H BASF Corp. Agricultural Products Center, Research Triangle Park NC, United States of America Unpublished
2002/5002749	Malinsky, DS	2002 a	Independent method validation of BASF analytical method M3515 entitled: BAS 720 H (CL 299263): LC/MS determinative and LC/MS/MS confirmatory method for the determination and confirmation of BAS 720 H and CL 263284 residues in plant matrices BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
ID-324-001	Mangels, G	1994 a	AC 299,263: Estimation of the photochemical oxidation rate in the atmosphere American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-306-002	Martin, C	1997 a	Henry's Law constant calculation for Imazamox (AC 299, 263) American Cyanamid Co., Princeton NJ, United States of America Unpublished
2012/7003612	McCall, WS & Blood, A	2013 b	BAS 720 H Imazamox: Soil photolysis BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
ID-640-003	McDonnell, RJ	1995 a	CL 299263: Metabolism of carbon-14 labelled CL 299263 in field grown canola American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-320-001	Melcer, M	1993 a	AC 299, 263: Determination of the dissociation constants American Cyanamid Co., Princeton NJ, United States of America Unpublished
2013/1249356	Mewis, A	2013 a	Independent laboratory validation (ILV) of an analytical method L0188/01 for the determination of BAS 720 H and 3 metabolites in plant matrices Eurofins Agroscience Services GmbH, Niefern-Oeschelbronn, Germany Fed.Rep. Unpublished
2012/1299407	Meyer, M	2013 a	Study on the residue behaviour of Imazamox in broad beans after treatment with BAS 720 06 H under field conditions in Germany, the United Kingdom, The Netherlands, Italy and Spain, 2011 SGS Institut Fresenius GmbH, Taunusstein, Germany Fed. Rep. Unpublished
ID-244-007	Minoura, M & Ohba, K	1996 a	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in soya bean, seed Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-244-008	Minoura, M & Ohba, K	1996 b	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in soya bean, foliage Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-244-009	Minoura, M & Ohba, K	1996 c	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in peanut, nut in shell Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-244-010	Minoura, M & Ohba, K	1996 d	CL 299263 (Imazamox): Validation of HPLC method M 2248.01 for the determination of CL 299263 residues in peanut, foliage Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-720-034	Minoura, M & Ohba, K	1996 a	CL 299263 100A: Residues of CL 299263 in peanut, nut in shell / Kingaroy, Queensland, Australia Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-720-035	Minoura, M &	1996 b	CL 299263 700SG: Residues of CL 299263 in peanut, nut in shell /

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	Ohba, K		Kingaroy, Queensland, Australia Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-720-036	Minoura, M & Ohba, K	1996 c	CL 299263 120A: Residues of CL 299263 in peanut, nut in shell / Norwin, Queensland, Australia Cyanamid Japan Ltd., Tahara Aichi 441-34, Japan Unpublished
ID-306-001	Morelli, D	1994 a	AC 299, 263—Determination of the vapor pressure American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-315-001	Morelli, D	1994 b	AC 299, 263—Determination of the N-octanol/water partition coefficient American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-435-001	Mulligan, E	1995 a	Evaluation of CL 299, 263 in a bacterial / microsome mutagenicity assay American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-470-003	Mulligan, E	1995 c	Microbial mutagenicity plate incorporation assay of CL 263,284 American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-470-004	Mulligan, E	1995 d	Microbial mutagenicity plate incorporation assay of CL 189,215 American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-470-005	Mulligan, E	1995 b	Microbial mutagenicity plate incorporation assay of CL 312,622 American Cyanamid Co., Princeton NJ, United States of America Unpublished
2004/5000274	Nejad, H	2004 a	Validation of BASF analytical method M 3519 (2002) entitled BAS 720 H and BAS 685 H: LC/MS determinative and LC/MS/MS confirmatory method for BAS 720 H, CL 263284, CL 189215, CL 312622, BAS 685 H, CL 288511 CL 182704 in lentils seed, forage BASF Corp. Agro Research, Princeton NJ, United States of America Unpublished
IA-730-011	Nejad, H	1999 a	CL 263,222 (Imazapic): Freezer stability of residues of CL 263,222, CL 263,284 and CL 189,215 in wheat green forage, wheat hay, wheat straw, and wheat grain Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished
IA-740-023	Nejad, H & Xu, B	2000 a	CL 263,222 (Imazapic): Freezer stability of residues of CL 263,222, CL 263,284 and CL 189,215 in peanut hull and nutmeat Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished
2008/7019226	Norris, F	2009 a	The magnitude of Imazamox and Imazapyr residues in Clearfield lentils following application of BAS 723 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2008/7019225	Norris, FA	2009 a	The magnitude of Imazamox and Imazapyr residues in Clearfield sunflower and Clearfield sunflower processed fractions following application of BAS 723 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2008/7019225	Norris, FA	2009 a	The magnitude of Imazamox and Imazapyr residues in Clearfield sunflower and Clearfield sunflower processed fractions following application of BAS 723 00 H BASF Agricultural Research Center, Research Triangle Park NC, United States of America Unpublished
2007/1007939	North, L	2007 a	Study on the residue behaviour of Imazamox, Metazachlor and Quinmerac in oilseed rape following foliar applications under field conditions in Northern and Southern Europe during 2005–2006 Agrisearch UK Ltd., Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
2007/1007963	North, L	2007 b	Study on the residue behaviour of Imazamox, Metazachlor and Quinmerac in oilseed rape following foliar applications under field conditions in Northern and Southern Europe during 2005–2006 Agrisearch UK Ltd., Melbourne Derbyshire DE73 8AG, United Kingdom Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-301-001	Patel, JA	1993 a	Technical CL 299263—Color, physical state, odor, bulk density, relative density, pH, oxidising / reducing properties American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-334-001	Patel, JA	1994 a	Technical CL 299, 263—Explodability American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-334-001	Patel, JA	1994 a	Technical CL 299, 263—Explodability American Cyanamid Co., Princeton

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2011/1281377	Radzom, M	2013 a	Metabolism of ^{14}C-Imazamox in rapeseed BASF SE, Limburgerhof, Germany Fed.Rep. Unpublished
2003/1030079	Rawle, NW	2003 a	Freezer stability of AC 299263 and CL 263284 in maize grain, ear and immature whole plant samples CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-015	Robbins, AJ	1997 a	Validation of CEM Analytical Services Standard Operating Procedure CEM-236 for the determination of residues of AC 299263 and CL 263284 in French bean whole plant samples CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-016	Robbins, AJ	1997 b	Validation of CEM analytical services standard operating procedure CEM-236 for the determination of residues of AC 299263 and CL 263284 in maize grain samples CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-017	Robbins, AJ	1997 c	Validation of CEM analytical services standard operating procedure CEM-236 for the analysis of residues of AC 299263 and CL 263284 in maize ears and immature whole plant CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-244-018	Robbins, AJ	1996 a	Standard Operating Procedure—The determination of residues of AC 299263 and AC 263284 in crops using HPLC CEMAS—CEM Analytical Services Ltd., North Ascot Berkshire SL5 8JB, United Kingdom Unpublished
ID-730-038	Rodriguez, D	1999 a	CL 299263 (Imazamox): CL 299263 and CL 263, 284 residues in imidazolinone-tolerant spring wheat racs and processed commodities after post-emergence treatment with AC 299263 (1AS) herbicide in North Dakota American Cyanamid Co., Princeton NJ, United States of America Unpublished
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ID-640-009	Roman, Y	1999 b	AC 299263: Metabolism of carbon-14 labelled AC 299263 in field grown oil seed rape/canola American Cyanamid Co., Princeton NJ, United States of America Unpublished
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ID-435-003	Sharma, R	1993 b	Evaluation of CL 299, 263 in the in vivo micronucleus assay in mouse bone marrow cells American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-435-002	Sharma, RK	1993 b	Evaluation of CL 299, 263 in the mammalian cell CHO/HGPRT mutagenicity assay American Cyanamid Co., Princeton NJ, United States of America Unpublished
2002/1020471	Stewart, J	2002 a	Analysis of Imazamox and Imazapyr residues in sunflower samples treated with BAS 723 00 H from efficacy trials in Turkey, 2002—Analytical report of the analysis of BAS 720 H, CL 263284 and BAS 693 H BASF Corp., Research Triangle Park NC, United States of America Unpublished
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ID-244-020	Sweeney, RA, & Gross, JR	1998 a	CL 299263: Independent laboratory validation of CE method M 3076 for the determination of residues of CL 299263 in canola seed American Cyanamid Co., Princeton NJ, United States of America Unpublished
2011/7002438	Ta, C	2012 a	Aerobic soil metabolism of ^{14}C-Imidazolinone BAS 720 H BASF Crop Protection, Research Triangle Park NC, United States of America Unpublished
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ID-620-027	Ta, CT & Lewis, CJ	1997 a	AC 299263: Soil degradation study Covance Laboratories, Harrogate North Yorkshire HG3 1PY, United Kingdom Unpublished
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ID-440-006	Tsalta, C	1999 a	CL 312622: Metabolism of [^{14}C]-CL 312622 in the lactating goat American Cyanamid Co., Princeton NJ, United States of America Unpublished
ID-244-022	Wickremesinhe, E & Safarpour, H	1998 a	CL 299263 (Imazamox): Independent laboratory validation of HPLC/MSD method M 3178 for the determination of residues of CL 299263, CL 263284, CL 189215 and CL 312622 in alfalfa seed, forage (whole green plant) and hay (whole dried plant) Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished

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ID-244-026	Xu, B & Nejad, H	1999 a	CL 299263 (Imazamox): Independent laboratory validation of CE method M 3098 for the determination of residues of CL 299263 and CL 263284 in wheat hay, forage, straw and grain Centre Analytical Laboratories Inc., State College PA, United States of America Unpublished
ID-640-006	Zulalian, J	1997 c	CL 299, 263: Metabolism of carbon-14 labelled CL 299, 263 in imidazolinone-resistant corn applied post-emergence under field conditions (Amendment number 1 included) American Cyanamid Co., Princeton NJ, United States of America Unpublished
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