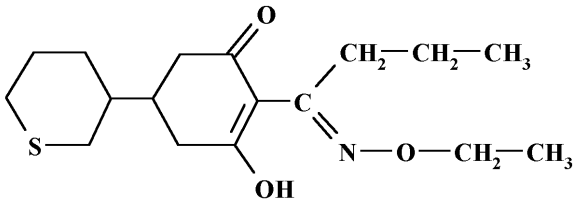
CYCLOXYDIM (179)

First draft prepared by Prof. Eloisa Dutra Caldas, University of Brasilia, Brasilia, Brazil

EXPLANATION

Cycloxydim is a post-emergence cyclohexene oxime herbicide that inhibits the acetylcoenzyme A carboxylase (ACCase) in chloroplasts of sensitive weeds. ACCase catalyses the first step of the *de novo* biosynthesis of fatty acids in plants. Cycloxydim was firstly evaluated by JMPR in 1992 (T, R). In 2009, within the periodic re-evaluation of toxicology, an ADI of 0–0.07 mg/kg bw and an ARfD of 2 mg/kg bw for women of childbearing age were established; ARfD was unnecessary for the general population. Cycloxydim was scheduled for the periodic re-evaluation of residues by the 2012 JMPR. Data on physical and chemical properties of cycloxydim, metabolism in plants and livestock animals, environmental fates and analytical methods were submitted for evaluation. The manufacturer submitted residue supervised trials on pome fruit, stone fruit, grapes, strawberries, potatoes, carrots, celeriac, onions, tomatoes, peppers, cauliflower, Brussels sprouts, head cabbage, curly kale/ Chinese cabbage, lettuce, spinach, green beans and peas, leeks, sugar beet roots, sugar beet tops, dry beans and peas, oilseed rape, sunflower, soya bean, rice and maize, in addition to animal feeding studies and processing studies.

IDENTITY

Common name:	Cycloxydim
IUPAC nomenclature:	(5RS)-2-[(EZ)-1-(ethoxyimino) butyl]-3-hydroxy-5-[(3RS)-thian-3-yl] cyclohex-2-en-1-one
CA nomenclature:	2-[1-(ethoxyimino) butyl]-3-hydroxy-5-(tetrahydro-2H-thiopyran-3-yl)-2-cyclohexen-1-one
CAS:	101 205-02-1
CIPAC:	510
ELINCS:	405-230-9
Molecular formula	$C_{17}H_{27}NO_3S$
Chemical structure	

PHYSICAL AND CHEMICAL PROPERTIES

The pure active ingredient (ai) is a colourless and odourless solid. The technical compound (TC) is a yellow paste with a moderate aromatic odour. Cycloxydim is chemically a weak acid of unpolar nature. It is very soluble in most of the organic solvents. Under neutral and acidic conditions cycloxydim is poorly soluble in water, but of better solubility in a basic environment. Cycloxydim technical is highly flammable but has a high auto-ignition temperature of 295 °C. Cycloxydim is manufactured as cycloxydim TK 42%, a yellow liquid of moderate aromatic odour. The physical and chemical properties of cycloxydim are shown in detail in Table 1.

Table 1 Physical and chemical properties of pure cycloxydim, technical compound (TC) and technical scale (TK)

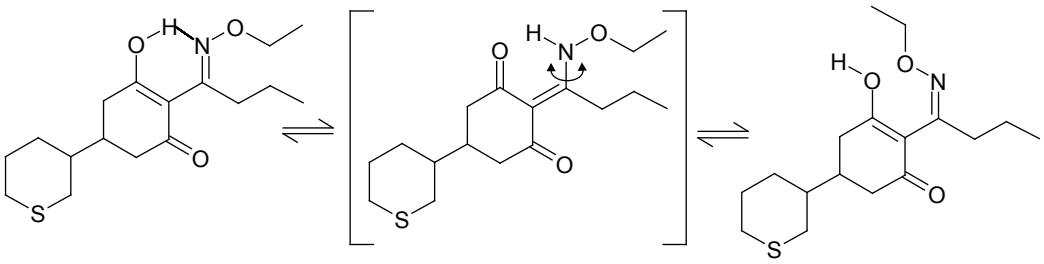
Property	Guideline and method	Findings and comments	Reference
Melting point solidification point	EEC A1. 1.4.4.2, DSC, OECD 102	Pure 99.6%: 37.1–41.2 °C TK, 42.0%: days at 0 °C cause no solidification or sedimentation	Tuerk, 1996a; Daum, 2006a; Kaestel, 1997a
Boiling point	EEC A2. 1.4.7, DSC, OECD 103	Pure 99.6%: endothermic effect other than the melting point.	Tuerk, 1996a,
Temperature of decomposition	EEC A2. 1.4.7, DSC, OECD 103	Pure 99.6%: composition is observed at approx. 200 °C	Tuerk, 1996a,
Relative density	EEC A3. 1.4.4, OECD 109	Pure, 99.6%: 1.165. TK 42.0%: 0.99	Kaestel, 1997b Kaestel, 1997a,
Vapour pressure	EEC A.4	Pure 99.6%: 1.0×10^{-5} Pa at 20 °C.	Kaestel, 1997b
Henry's law constant and volatility	based on vapour pressure, MW and water solubility	Henry's Law constant at 20 °C: $H = 6.1 \times 10^{-8}$ (kPa × m^3/mol.)	Ohnsorge, 2000a,
Colour and physical state	Visual examination	Pure 99.6%: white crystals TC, 92.3%: yellow liquid, a viscous paste TK, 42.0% : yellow liquid	Tuerk, 1996a, Kaestel, 1997c Kaestel, 1997a
Odour	Organoleptic	Pure, 99.6%: odourless TC, 92.3%: moderate aromatic odour. TK, 42%: moderate aromatic odour.	Tuerk, 1996a Kaestel, 1997c Kaestel, 1997a,
UV/VIS, IR, NMR, MS spectra (as)	OECD 101	Pure 99.6%: UV molar extinction coefficient [l× mol^{-1} × cm^{-1}]: 210 nm: 7.2×10^3 227 nm: 4.1×10^3 259 nm: 1.0×10^4 278 nm: 9.3×10^3 290 nm: 7.9×10^3 300 nm: 4.4×10^3 UV molar extinction coefficient under acidic conditions (pH1.4) [l × mol^{-1} × cm^{-1}]: 228 nm: 6753 260 nm: 13788 290 nm: 4199 310 nm: 2930 UV molar extinction coefficient under basic conditions (pH12.1) [l × mol^{-1} × cm^{-1}]: 215 nm :4983 234 nm: 2140 283 nm: 122105 290 nm: 18079	Tuerk, 1996b Kroehl, 2007a Kroehl, 2008a
Solubility in water	EEC A.6.1.4.2, flask method OECD 105	Pure 99.6%, 53 mg/L at 20 °C: The saturated solution is acidic (pH 4.3). 50% w/w of pure ai is soluble as sodium salt at pH 10.7. pH 4: 0.05 g/L (phthalate buffer) pH 7: 0.9 g/L (phosphate buffer) pH 9: 8 g/L (borate buffer)	Tuerk, 1996c Pawliczek, 1988a Class, 2008a
Solubility in organic solvents	Visual classification. CIPAC MT 181 [formerly CIPAC Document No. 3869/M]	Pure 99.6%, in g/100 mL solvent: n-heptane: > 25 toluene: > 25 dichloromethane: > 25 methanol: > 25 acetone: > 25 ethyl acetate > 25	Daum, 1998a, CIPAC MT 181, 1997a
n-octanol/water partition coefficient	OECD 107 flask method	Pure ai, 99.3% log P_{OW} at 25 °C: 3.09 at pH 5,	Redeker, 1988a

Property	Guideline and method	Findings and comments	Reference
		1.36 at pH 7, –0.42 at pH 9	
Hydrolysis rate at pH 4,7 and 9 under sterile conditions in the absence of light	US-EPA, N, § 161-1; OECD 111, 2004, SETAC, March 1995	[^{14}C] ai, > 95%; Half-lives at 25 °C: pH 4: 2.1 days pH 5: 12.2 days pH 7: 264 days (extrapolated) pH 9: 958 days (extrapolated)	Hassink, J., 2009a
Direct photo-transformation	FAO, Rev. 3; US-EPA, Subdivision N, § 161-2	[^{14}C] ai, 92% Half-lives at 22 °C: pH 5: 5.8 h pH 7: 17.6 h pH 9: 22.3 h	Goetz, v N, 2000a
Quantum yield of direct photo-transformation	FAO, Rev. 3; US-EPA, Subdivision N, § 161-2	[^{14}C] ai, 92%quantum yield pH 5: 5.68×10^{-3} pH 7: 1.87×10^{-4} pH 9: 2.02×10^{-4}	Goetz, v N, 2000a
Dissociation constant	OECD 112	Pure, 99.4%: pK_a 4.17 at 20 °C. TK, 42.0%: pH = 4.1; 1% emulsion in CIPAC H_2O D	Redeker, J, 1988b Kaestel, R, 1997a
Photochemical oxidative degradation	BBA guideline part IV, 6-1	T ½ = 6.3 h (Atkinson's method)	Sarafin, R 1991a]
Flammability (upon contact with water)	EEC A.10 EEC A.11 EEC A.12	TC, 92.3%: highly flammable, observed after 31 sec. TK, 42.0%: Not applicable for liquid. TK batch 707: 3 not highly flammable	Loeffler, U, 1997a, Loeffler, U, 1997b Bitterlich, S 2007a
Relative self-ignition temperature	EEC A.15	TC, 92.9%: Auto-ignition at 295 °C TK, 42.0%: Auto-ignition at 360 °C.	Loeffler, U, 2000a Loeffler, U, 1997b
Flash point	EEC A9.1.6.3.2	TC, 92.9%: 89.5 °C TK, 42.0%: 62 °C.	Loeffler, U, 2000a Kaestel, R, 1997a
Explosive properties	EEC A.14 DSC	TC, 92.9%: Not explosive TK, 42.0%: Not applicable TK, batch 7073: not explosive	Loeffler, 2000a; Loeffler, 1997b; Petersen-Thiery, 2006a; Bitterlich, 2007a
Surface tension	EEC A.5	Pure 99.6%: 58.0 mN/m at 0.5% and 57.0 mN/m at 2.0% TC, 92.3%: 56.2 mN/m at 1.0% TK, 42.0%: 52.4 mN/m at 1%	Kaestel, R, 1997b Kaestel, R, 1997c Kaestel, R, 1997a
Oxidizing properties	EEC A.17 EEC A.21	TC, 92.9%: Not applicable TK, 42.0%: Not applicable TK batch 7073: not an oxidizing agent.	Loeffler, U, 2000a Loeffler, U, 1997b Bitterlich, S 2007a

METABOLISM AND ENVIRONMENTAL FATE

E/Z isomerization of cycloxydim at the C=N bond

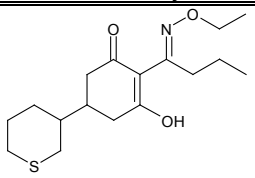
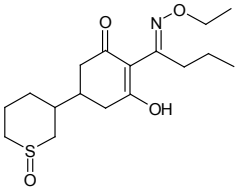
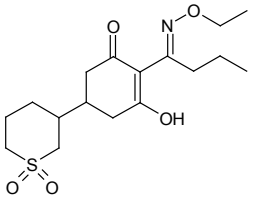
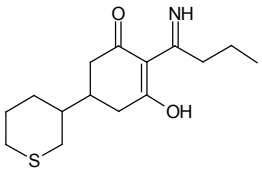
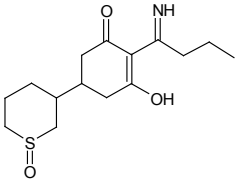
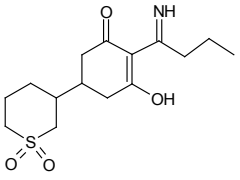
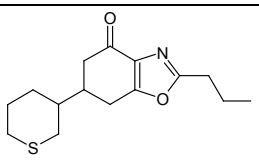
The chemical structure of the active substance cycloxydim contains an ethoxyimino group including a C = N double bond. It has been observed that an E/Z isomerisation occurs frequently and easily. The ratio of isomers depends on the physical state of the compound and the polarity of solvents.

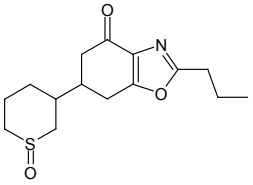
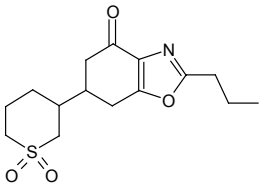
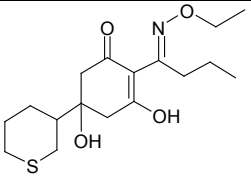
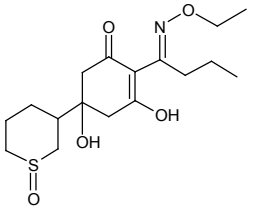
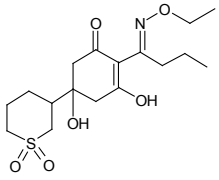
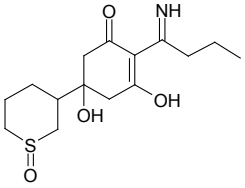
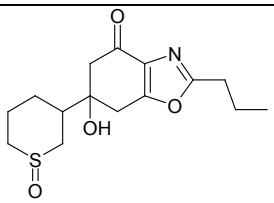
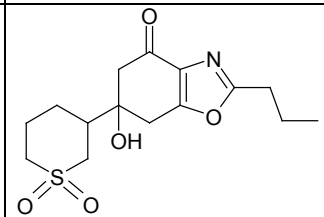
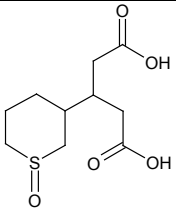


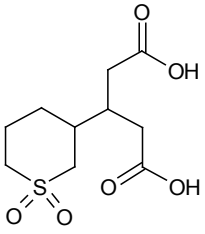
The ^{14}C-labelled cycloxydim, stored in toluene, is almost exclusively present as E-isomer (Grosshans, 2006a). The EC formulation contains a mixture of aromatic hydrocarbons, and cycloxydim is also exclusively present as E-isomer. In the water spray emulsion, the Z-isomer may have been built to a certain extent. The parent compound and all metabolites containing the oxime ether chain can isomerize in its physiological environment in plants and in solution dependent on polarity, temperature and pH and therefore double peaks can occur in the chromatograms. During the course of the metabolism studies, the individual isomer concentration could also be influenced by extraction, solution, or elution solvents used for work-up and analysis. Therefore it is not avoidable that one or the other isomer is detected in higher concentrations which do not exactly represent the E/Z-ratio in the biological matrix.

The metabolites identified in the different metabolism studies are summarized in Table 2.

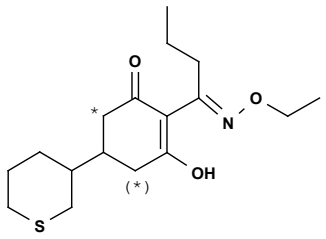
Table 2 Identified cycloxydim and cycloxydim-5-OH-TSO metabolites in animal and plants

Name	Chemical Name	Metabolite Identity	Matrix where found
Cycloxydim	2-[1-(ethoxyimino)butyl]-3-hydroxy-5-(3-thianyl)-2-cyclohexen-1-one		sugar beet soya bean hen goat rat
Cycloxydim-TSO	2-[1-(ethoxyimino)butyl]-3-hydroxy-5-(3-thianyl)-2-cyclohexen-1-one S-oxide		sugar beet soya bean maize hen goat rat rotational crop
Cycloxydim-TSO2	2-[1-(ethoxyimino)butyl]-3-hydroxy-5-(3-thianyl)-2-cyclohexen-1-one S-dioxide		sugar beet soya bean maize hen goat rat rotational crop
Cycloxydim-T1S	2-(1-iminobutyl)-3-hydroxy-5-(3-thianyl)-cyclohex-2-en-1-one		goat (liver) rat
Cycloxydim-T1SO	2-(1-iminobutyl)-3-hydroxy-5-(3-thianyl)-cyclohex-2-en-1-one S-oxide		sugar beet soya bean maize goat (milk, liver, kidney) hen rat rotational crop
Cycloxydim-T1SO2	2-(1-iminobutyl)-3-hydroxy-5-(3-thianyl)-cyclohex-2-en-1-one S-dioxide		sugar beet soya bean maize goat rat
Cycloxydim-T2S	2-propyl-6-(3-thianyl)-4,5,6,7-tetrahydrobenzoxazol-4-one		hen (fat, liver) rat

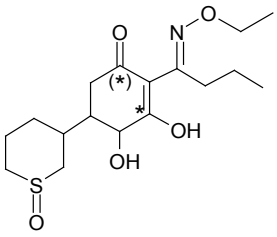
Name	Chemical Name	Metabolite Identity	Matrix where found
Cycloxydim-T2SO	2-propyl-6-(3-thianyl)-4,5,6,7-tetrahydrobenzoxazol-4-one S-oxide		sugar beet soya bean maize goat hen rat rotational crop
Cycloxydim-T2SO2	2-propyl-6-(3-thianyl)-4,5,6,7-tetrahydrobenzoxazol-4-one S-dioxide		sugar beet soya bean goat (milk, liver)
Cycloxydim-5-OH-TS	2-[1-(ethoxyimino)butyl]-3,5-dihydroxy-5-(3-thianyl)-2-cyclohexen-1-one		goat hen
Cycloxydim-5-OH-TSO	2-[1-(ethoxyimino)butyl]-3,5-dihydroxy-5-(3-thianyl)-2-cyclohexen-1-one S-oxide		soya bean
Cycloxydim-5-OH-TSO2	2-[1-(ethoxyimino)butyl]-3,5-dihydroxy-5-(3-thianyl)-2-cyclohexen-1-one S-dioxide		soya bean
Cycloxydim-5-OH-T1SO	2-(1-iminobutyl)-3,5-dihydroxy-5-(3-thianyl)-2-cyclohexen-1-one S-oxide		maize (grain) goat
Cycloxydim-6-OH-T2SO	2-propyl-6-hydroxy-6-(3-thianyl)-4,5,6,7-tetrahydrobenzoxazol-4-one S-oxide		soya bean maize goat
Cycloxydim-6-OH-T2SO2	2-propyl-6-hydroxy-(3-thianyl)-4,5,6,7-tetrahydrobenzoxazol-4-one S-dioxide		soya bean maize
Cycloxydim-TGSO	3-(3-thianyl)-glutaric acid S-oxide		soya bean maize

Name	Chemical Name	Metabolite Identity	Matrix where found
Cycloxydim-TGSO2	3-(3-thianyl)-glutaric acid S-dioxide		sugar beet soya bean maize rotational crop

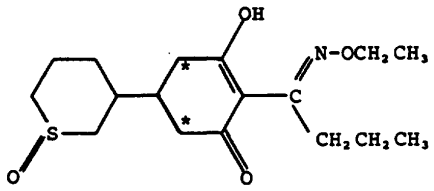
The metabolism and distribution of [^{14}C] cycloxydim and metabolites in animal and or plants was investigated using the test substance labelled in the cyclohexenone ring; * position of the ^{14}C-label (Figure 1).



[^{14}C] Cycloxydim



[^{14}C] Cycloxydim-5-OH-TSO



^{14}C-Cycloxydim-TSO

Figure 1 Labelled cycloxydim and metabolite used in the metabolism studies

Animal metabolism

Lactating goats

[^{14}C] cycloxydim was investigated in two lactating goats following repeated oral administration for 7 days at an actual dose of 15 mg/kg feed or 24 mg/animal/day (Leibold & Hoffmann, 2001a; Hafemann & Knoell, 2003a). Excreta were collected in 24-hour intervals and milk collected in the morning before dosing and in the evening. The portion from the evening was combined with that of the next morning to form the daily pool. Muscle, fat, liver and kidney were pooled from both goats at the day of sacrifice. The total radioactive residues were determined in milk by direct liquid scintillation counting while the other matrices were combusted for this purpose. Milk, liver, and kidney were extracted with methanol and in case of the organs, subsequently with water. Non-extracted residues > 10% TRR were solubilised by pronase treatment. Metabolite patterns were obtained by HPLC analysis.

About 85% of the total administered dose was recovered, mainly in urine (72%) and faeces (12%), and 0.09% in milk. Radioactivity in milk was constant during the 7 days dosing period. More than 96% TRR in milk was extracted with methanol (Table 3). Post-extraction solid contained 64.1%TRR from liver and 49.5%TRR from kidney, most of it solubilised by pronase treatment (57% and 42% of the TRR for liver and kidney, respectively). TRR in muscle and fat were low (0.006 and 0.005 mg/kg, respectively) and further investigations in these matrices were not reported.

Table 3 Extractability of goat matrices after dosing with ^{14}C/^{13}C-cycloxydim

Matrix	TRR	Methanol		Water		ERR [a]		PES [b]		Recovery
	mg/kg	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	mg/kg	% TRR	%
Milk	0.023	0.022	96.5	–	–	0.022	96.5	< 0.001	1.4	97.9
Liver	0.076	0.023	30.5	0.003	4.0	0.026	34.5	0.048	64.1	98.6
Kidney	0.062	0.030	48.8	0.002	3.7	0.032	52.5	0.031	49.5	102.0

[a] ERR = Extractable Radioactive Residue (sum of Methanol and Water)

[b] PES = Post-Extraction Solid

The major metabolites were identified as cycloxydim-T1SO and cycloxydim-TSO in milk, liver and kidney (Table 4). Cycloxydim-T1S, cycloxydim-TSO2 and cycloxydim-T2SO2 were detected as minor metabolites. The post-extraction solids of liver and kidney were solubilised by pronase treatment and showed a complex peak pattern after HPLC analysis with each peak below 0.01 mg/kg eq.. None of these HPLC peaks could be correlated with the metabolite identities found in the organic extracts of liver and kidney.

Table 4 Identified and quantified metabolites in methanol extracts of edible matrices of lactating goats after dosing with $^{14}C/^{13}C$-cycloxydim at a nominal dose level of 12 mg/kg ppm feed

Metabolite	Milk mg/kg (%TRR)	Liver mg/kg (%TRR)	Kidney mg/kg (%TRR)
Cycloxydim	–	0.008 (10.8)	–
cycloxydim-T1SO [a]	0.004 (16.4)	0.001 (1.8)	0.002 (4.1)
cycloxydim-TSO [a]	0.003 (14.8)	0.006 (8.1)	0.008 (12.4)
cycloxydim-T2SO2	< 0.001 (0.5)	0.001 (2.0)	–
cycloxydim-TSO2	–	< 0.001 (0.6)	–
cycloxydim-T1S	–	< 0.001 (0.5)	–
Total idenfified, mg/ kg	0.006	0.018	0.010
Non- idendified residues, mg/kg	0.016	0.005	0.020

[a] Sum of isomers

Storage stability investigations were conducted with milk, liver and faeces. Organic extracts of these matrices were produced at the beginning and at the end of the study. The extracts were further analysed by two different HPLC systems. The storage stability investigations with milk, liver and faeces extracts covered a time period of up to 18 months. Within this time period it could be shown that the metabolite composition was sufficiently stable.

The main route of biotransformation of cycloxydim was the stepwise oxidation of the sulphur of the thiopyrane ring to cycloxydim-TSO (major metabolite) and to cycloxydim-TSO2 (Figure 2). Another metabolic route was the degradation of the oxime ether group by N-de-ethoxylation forming cycloxydim-T1S. Cycloxydim-T1S was further oxidized to the sulfoxide cycloxydim-T1SO. In addition, cycloxydim-TSO was transformed abiotically to the oxazole cycloxydim-T2SO by a Beckmann rearrangement followed by ring closure. This transformation product was further oxidized to the sulphur dioxide cycloxydim-T2SO2. All metabolites containing the intact oxime ether group produced E/Z isomers at the C = N double bond. Metabolites with the sulfoxide group generated additional diastereomers with the sulphur and the 3-yl position as chirality centres.

A lactating goat (50 kg, age 1–2 years) was dosed with gelatine capsules containing [^{14}C] cycloxydim-TSO at 200 mg per day for five days, equivalent to 4 mg/kg body weight (Hawkins *et al.* 1985a). Assuming a standard feed consumption of 2 kg/day, this dose level corresponds to a dietary concentration of 100 mg/kg feed. Urine and faeces were collected separately in 24 hour intervals. The animals were milked twice daily, immediately prior to dosing and at approximately 6 hours after dosing. The treated goat and the control animal were sacrificed 24 hours after the final dose. The livers and kidneys were removed, along with samples of muscle (loin and leg) and omental and back fat. Milk and liver were extracted with methanol. Further samples of liver were also extracted with methanol/water (4:1), ethanol and ethyl acetate and an additional sample incubated with β-glucuronidase/sulphatase and subsequently extracted with methanol. The methanol extracts were analysed by TLC. Metabolites in urine, milk and liver extracts were identified by co-chromatography with reference compounds.

At sacrifice, 89.1% of the administered dose was recovered, mostly in urine (78.5%) and faeces (10%). TRR in milk accounted for 0.11% of the dose. Liver and kidney had the highest residues (Table 5). Mean daily milk concentrations immediately reached a plateau of 0.09–0.12 mg/kg. Concentrations in morning milk were considerably lower than corresponding plasma concentrations.

Table 5 Total radioactive residues after dosing of a lactating goat with [^{14}C] cycloxydim-TSO at a dose level of 100 mg/kg feed

Matrix	% of the dose	TRR, mg/kg
Milk (Day 1–Day 5) [a]	0.11	0.09–0.12
Liver	0.035	0.46
Kidney	0.007	0.52
Muscle	–	0.04
Omental fat	–	0.04
Back fat	–	0.04

[a] Morning and afternoon milk combined

Over 90% of the radioactivity present in milk was extracted in methanol. Extraction of liver with methanol, methanol/water and ethyl acetate removed < 50% of the radioactive residues. After enzyme incubation, three subsequent extractions with methanol removed 63.7% of the liver radioactivity. Unchanged cycloxydim-TSO accounted for more than 50% of radioactivity excreted in urine. In milk and liver extracts cycloxydim-TSO was the major component, followed by cycloxydim-T1SO (Table 6).

Table 6 Summary of metabolites in milk and liver of goats which had received [^{14}C] cycloxydim-TSO at a 100 mg/kg nominal dose level (based on feed intake)

Metabolite	Milk [a] mg/kg (% TRR)	Liver mg/kg (% TRR)
cycloxydim-T1SO	0.06 (22.8)	0.05 (9.9)
cycloxydim-T2SO	0.02 (7.8)	–
cycloxydim-TSO	0.06 (23.1)	0.10 (21.7)
cycloxydim-T1SO2	0.01 (5.0)	–
cycloxydim-TSO2	< 0.01 (2.7)	–
Unidentified	≤0.11 [b] (38.7)	≤ 0.16 [c] (32.1)

[a] Collected 6 hours after the fourth daily dose

[b] 4 TLC peaks in the range of 1.1–23.7% TRR

[c] 5 TLC peaks in the range of 0.5–22.5% TRR

The metabolism and distribution of [^{14}C] cycloxydim-OH-TSO was investigated in two lactating goats following repeated oral administration at one dose level of 12 mg/kg feed on nine consecutive days (Leibold & Ravenzwaay, 2002c; Tilting, 2003a,b). Faeces and urine were collected daily. Milk was sampled in the morning before dose administration and in the afternoon. Animals were sacrificed 23 hours after the last dosing. Liver, kidney, fat, muscle and milk (days 3 to 7) were pooled and analysed. Milk and tissues were extracted with methanol, followed by extraction with water, and clean-up by acetonitrile/iso-hexane partition (milk) or SPE column clean-up. The extraction residue of liver was subjected to enzymatic digestion with a protease (pronase). Characterisation and identification of metabolites was performed by LC-MS.

On average, 97.1% of the administered dose was recovered in the experiment, from which 75% in urine and 15.7% in faeces. In average, 0.11 to 0.12% was recovered in liver and muscle, 0.07% in milk and 0.02% in kidney and fat. TRR ranged from 0.02 mg/kg in milk to 0.26 mg/kg in kidney (Table 7). The radioactivity in milk remained constant, so that the plateau was reached almost immediately. Methanol extracts contained 88.6% to 96.9% TRR except for liver, where a protease treatment was necessary to achieve an addition 11.6% TRR. The non-extracted residues accounted for up to 10.2% TRR in muscle (Table 7).

Table 7 Extractability after dosing lactating goats with [^{14}C] cycloxydim-5-OH-TSO

Matrix	TRR mg/kg	Methanol mg/kg (%TRR)	Water mg/kg (%TRR)	ERR [a] mg/kg (%TRR)	PES [b] mg/kg(%TRR)	Recovery [c] mg/kg(% TRR)
Milk	0.0197	0.018 (91.38)	0.0002 (0.76)	0.018(92.14)	0.0006(3.06)	0.0187(95.2)
Muscle	0.0245	0.023 (94.9)	n.p.	0.023(94.9)	0.0025(10.2)	0.0257(105.1)

Matrix	TRR mg/kg	Methanol mg/kg (%TRR)	Water mg/kg (%TRR)	ERR [a] mg/kg (%TRR)	PES [b] mg/kg(%TRR)	Recovery [c] mg/kg(% TRR)
Liver	0.203	0.159 (78.4)	0.016 (8.0)	0.199(98.0)	0.004(1.7)	0.203(99.7)
Kidney	0.259	0.251 (96.9)	0.004 (1.5)	0.255(97.1)	0.016(6.2)	0.271(103.3)
Fat	0.0237	0.0211 (88.6)	n.p.	0.0211(89.0)	0.00144(6.1)	0.0225(95.1)

[a] ERR = Extractable Radioactive Residue, include 11.6% extracted after protease

[b] PES Post Extraction Solids (residue after all extraction steps)

[c] Sum of all extracts and the final residue

n.p. = Not performed

Over 70% TRR was identified and characterized was in milk and tissues (Table 8). Cycloxydim-5-OH-TSO was the main compound detected, excepted in liver where cycloxydim-5-OH-TS was the main detected. Other metabolites identified were cycloxydim-5-OH-T1SO and cycloxydim-6-OH-T2SO (Table 8). Many minor metabolites, which could not be identified, were formed.

Table 8 Summary of identified and quantified metabolites in edible matrices of lactating goats after dosing with [^{14}C] cycloxydim-5-OH-TSO at a nominal dose level of 12 mg/kg

Metabolite	Milk mg/kg (%TRR)	Muscle mg/kg (%TRR)	Liver mg/kg (%TRR)	Kidney mg/kg (%TRR)	Fat mg/kg (%TRR)
cycloxydim-5-OH-TSO	0.0067 (33.9)	0.0086 (35.1)	0.0216 (10.6)	0.0978 (37.8)	0.0073 (30.9)
cycloxydim-5-OH-TS	0.0008 (3.8)	0.0010 (4.3)	0.0354 (17.4)	0.0654 (25.3)	0.0026 (10.7)
cycloxydim-6-OH-T2SO	0.0012 (6.1)	0.0031 (12.7)	0.0043 (2.1)	0.0080 (3.1)	n.d.
cycloxydim-5-OH-T1SO	0.0016 (8.4)	0.001 (3.7)	n.d.	n.d.	0.0009 (3.8)

n.d.: Not detected

Storage stability investigations were performed in urine and liver. At the beginning and at the end of the study liver samples were extracted and the methanol extracts were analysed by HPLC. Both extraction efficiency and metabolite pattern were absolutely comparable. A decline of cycloxydim-5-OH-TS could be observed probably due to oxidation by air to form cycloxydim-5-OH-TSO.

Figure 2 shows the metabolic pathways of cycloxydim in goats, hens and rats after feeding with cycloxydim and/cycloxydim-TSO and cycloxydim-5-OH-TSO)

Laying hens

The metabolism and distribution of [^{14}C] cycloxydim (^{14}C:^{13}C:^{12}C, 30:50:20) was investigated in laying hens following daily oral administration to a group of ten hens for 10 days at a nominal dose level of 12 mg/kg feed (Leibold & Ravenzwaay, 2002b; Fabian & Knoell, 2003a). Excreta were collected daily and eggs twice a day, in the morning before administration of the test substance and in the afternoon, except for the weekends, where records on egg production were only made once per day. Within 23 hours after the last administration, animals were sacrificed and liver, adipose tissue, chest and leg muscles, and the gastrointestinal (GI) tract (skin and contents) were taken for determination of radioactivity. Eggs and each tissue type were pooled over all animals. Excreta, GI contents and fat were treated with tissue solubiliser after suspending in methanol and freeze-drying. Total radioactive residues in tissues were determined by combustion. Eggs were directly measured after mixing with scintillation fluid. Excreta, muscle and liver samples were extracted with methanol while fat and eggs were extracted with a mixture of methanol/iso-hexane. The extracts were further purified by partition with iso-hexane and in some cases additionally cleaned-up with solid phase extraction (SPE) cartridges or fractionated by HPLC. The extraction residues of liver, muscle, fat, and eggs were subjected to an enzymatic digestion with an unspecific protease (pronase). The purified methanol extracts of eggs and tissues were investigated by radio-HPLC on at least two different

HPLC systems either with synthetic reference substances or metabolite samples identified by LC/MS/MS.

The overall recovery of radioactivity amounted to 80.05% of the administered dose, mostly in the excreta (78.0%), 0.33% in eggs. 0.11% in muscle, 0.07% in liver and 0.02% in fat. Total radioactive residue (TRR) in eggs ranged from 0.052 mg/kg at day 1 to 0.142 mg/kg at day 10 (mean of 0.121 mg/kg), starting to plateau at day 8. In tissues, TRR ranged from 0.051 to 0.281 mg/kg (Table 9). From 11 to 45%TRR was extracted in methanol, and non-extracted residues (post-extraction solids, PES) were in the range of 54 to 91% TRR.

Table 9 Total radioactive residues (TRR) after dosing of laying hens with [^{14}C] cycloxydim

Matrix	TRR	Methanol	Hexane	ERR [a]	PES [b]	Recovery [c]
	mg/kg	mg/kg (% TRR)	mg/kg(% TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Egg, pool	0.121	0.055 (45.0)	0.002 (1.6)	0.057 (46.6)	0.065 (53.9)	100.5
Muscle	0.053	0.006 (11.1)	n.p.	0.006 (11.1)	0.049 (91.0)	102.1
Fat	0.051	0.015 (30.3)	0.001 (2.2)	0.016 (32.5)	0.035 (69.0)	101.5
Liver	0.281	0.075 (26.8)	0.021 (7.6) [d]	0.097 (34.4)	0.201 (71.6)	106.0

[a] ERR = Extracted Radioactive Residue (sum of solvents 1 and 2)

[b] PES = Post-Extraction Solid

[c] Sum of all extracts and the residue

[d] sum of different organic solvents

n.p.: Not performed

Identification and quantification of parent compound and metabolites in the edible matrices of laying hens are shown in Table 10. In all matrixes, the main residue was cycloxydim-TSO (30.9% TRR in eggs). Parent compound represented < 5%TRR and cycloxydim-T2S was present only in fat and liver (≤ 1%TRR). Protease digestion released about 50% TRR in eggs and 66.2%TRR in fat. Unextracted residues in muscle accounted for 4.5% TRR (0.002 mg/kg). Up to eight unknown minor peaks were detected in the matrices, all at levels ≤ 0.001 mg/kg.

Table 10 Summary of identified and quantified metabolites in edible matrices of laying hens after dosing with [^{14}C] cycloxydim at a nominal dose level of 12 mg/kg (based on feed intake)

Metabolite	Eggs mg/kg (% TRR)	Muscle mg/kg (% TRR)	Fat mg/kg (% TRR)	Liver mg/kg (% TRR)
Cycloxydim	0.004 (3.4)	< 0.001 (0.5)	–	0.005 (1.7)
cycloxydim-TSO [a]	0.037 (30.9)	< 0.002 (3.0)	0.008 (18.0)	0.020 (7.4)
cycloxydim-TSO2 [a]	0.008 (6.4)	0.001 (0.9)	< 0.001 (0.4)	0.002 (0.6)
cycloxydim-T2S	–	–	< 0.001 (0.7)	0.003 (1.0)

[a] Sum of diastereomers

All samples used in this study for metabolite identification were extracted and analysed within 16.4 months after sampling. To confirm storage stability, new sub-samples of egg, muscle, fat, and liver were extracted about 16.1 to 16.4 months after sampling and were compared to the HPLC chromatograms obtained at the beginning of the study (within 1.2 months after sampling). For all matrices, the re-extraction of samples showed no significant differences with regard to extractability and metabolite pattern. None of the major peaks were subject to degradation.

The metabolic pathway of cycloxydim in hens was characterized by two main routes: oxidation to cycloxydim-TSO and subsequently to cycloxydim-TSO2 and Beckmann re-arrangement with subsequent ring closure forming the oxazol cycloxydim-T2S.

[^{14}C] Cycloxydim sulfoxide ([^{14}C] cycloxydim-TSO) was administered in gelatine capsules to laying hens once daily for 7 days at a dose level of 5 mg/bird/day, equivalent to an intake of total diet containing residues of 50 mg/kg (Hawkins *et al.* 1986a). The treated animals were sacrificed between 6 and 48 h hours after the last dose administration. Radioactivity was measured in eggs laid during and after the dosing period and in tissues at 6, 24 and 48 hours after the final dose. Excretion of

radioactivity was measured during the dosing period and up to 48 hours after the last dose. Pooled eggs from Group 4 laid on Day 5 were freeze-dried and extracted with methanol. The extract was purified by C_{18} SPE and analysed by TLC. A portion of pooled livers from Group 3 was extracted with methanol and another incubated with glucuronidase/sulphatase, extracted with methanol, purified by SPE and analysed by TLC. TRR in eggs increased rapidly to an apparent plateau value of 0.07–0.10 mg/kg from Day 2 onwards. Radioactive residues in tissues were highest 6 h post dose and declined rapidly with time (Table 11).

Table 11 Mean Total Radioactive Residues in tissues 6 h, 24 h and 48 h after dosing of laying hens with [^{14}C] cycloxydim-TSO

Matrix	TRR, mg/kg		
	Group 2 (6 h Post Dose)	Group 3 (24 h Post Dose)	Group 4 (48 h Post Dose)
Egg (Days 2–7)	–	–	0.07, 0.08, 0.09, 0.08, 010,-0.10
Muscle	0.10	0.06	< 0.01
Fat + skin	0.15	0.15	0.04
Liver	0.57	0.35	0.07
Kidney	0.99	0.62	0.08

Excretion of radioactivity was almost complete by 48 hours after the final dose, with 92.3% of the administered dose recovered in the excreta and a mean of 0.08% in eggs (Group 4). Methanol extraction removed 79.1% TRR from eggs and 76.0% TRR from liver. With preceding enzyme incubation, 90% TRR in liver were extracted with methanol. Cycloxydim-TSO was the major identified component in eggs and liver. Major metabolites in liver were cycloxydim-T2SO and cycloxydim-T1SO. In total, 23.6% TRR of the egg radioactivity remained unidentified (Table 12).

Table 12 Metabolites in eggs and liver of laying hens after dosing with [^{14}C] cycloxydim-TSO

Metabolite	Eggs, group 4, day 5, mg/kg (% TRR)	Liver, group 2, 6 h post sacrifice, mg/kg (% TRR)
cycloxydim-TSO	0.12 (41.4)	0.19 (32.7)
cycloxydim-T2SO	0.02 (5.5)	0.14 (24.3)
cycloxydim-T1SO	–	0.10 (16.6)
cycloxydim-TSO2	0.03 (8.8)	0.03 (5.3)
Unidentified	0.06 [a] (23.6)	0.07 [b] (11.5)

[a] 7 components (0.7–12.1% TRR)

[b] 4 components (1.6–6.8% TRR)

Primary pathways of metabolism of cycloxydim-TSO involved oxidation to the sulfone (cycloxydim-TSO2), de-ethoxylation to yield the imine (cycloxydim-T1SO) and Beckmann rearrangement to yield the cyclic cycloxydim-T2SO.

[^{14}C] cycloxydim-5-OH-TSO was administered to a group of twelve hens daily on 11 consecutive days at a nominal dose level of 12 mg/kg feed (Leibold &Ravenzwaay van, 2002a; Seiferlein, 2003a). Excreta were collected each 24 hours and eggs were sampled twice a day. Animals were sacrificed 23 hours after the last dose and tissues taken for determination of radioactivity. Eggs from Day 2 to Day 11 as well as each tissue type were pooled over all animals. Total radioactive residues in tissues were determined by combustion except for fat, which was treated with tissue solubiliser. Eggs were directly measured after mixing with scintillation fluid. Eggs, muscle and liver were extracted with methanol and fat with methanol/iso-hexane (1:1, v/v). The extracts were further purified by partition with iso-hexane, cleaned-up with SPE and subjected to an enzymatic digestion with protease. Extracts were investigated by radio-HPLC. All samples used in metabolite identification were extracted and analysed within 9 months after sampling, period during which no differences was found in extractability and metabolite pattern.

The radioactivity was rapidly excreted within 24 hours after the last dose, with 92.7% of the applied dose recovered, mainly on excreta (89.7%). Only 0.21% of the dose was found in eggs, with

levels ranging from 0.046 to 0.066 mg/kg from day 1 to 11. The highest concentration of radioactivity was found in the gastrointestinal tract (0.605 mg/kg, 0.56% of the dose), followed by muscle (0.08%, 0.028 mg/kg), liver (0.03%, 0.11 mg/kg) and fat (0.01%, 0.017 mg/kg).

Methanol extracted 88.8% of the TRR present in eggs and 80.5% TRR in fat (Table 13). The final non-extracted residues (post-extraction solids) were in the range of 24 to 38% TRR. Protease released over 80% of the residues in PES.

Table 13 Extractability of edible matrices after dosing of laying hens with [^{14}C] cycloxydim-5-OH-TSO

Matrix	TRR mg/kg	Methanol mg/kg (% TRR)	Hexane mg/kg (% TRR)	ERR [a] mg/kg (% TRR)	PES [b] mg/kg (% TRR)	Recovery [c] mg/kg (% TRR)
Egg	0.066	0.059 (88.8)	n.p.	0.059 (88.8)	0.017 (26.4)	0.076 (115.2)
Muscle	0.028	0.017 (62.8)	n.p.	0.017 (62.8)	0.010 (37.9)	0.028 (100.7)
Fat	0.017	0.014 (80.5)	0.001 (3.8)	0.014 (84.3)	0.004 (24.1)	0.019 (108.4)
Liver	0.110	0.071 (64.6)	n.p.	0.071 (64.6)	0.036 (32.3)	0.107 (96.9)

[a] ERR = Extractable Radioactive Residue (sum of solvents 1 and 2)

[b] PES = Post-Extraction Solid

[c] Sum of all extracts and the residue

n.p.: not performed

Cycloxydim-5-OH-TSO was metabolized to cycloxydim-5-OH-TS in all matrices, accounting with 19.4%TRR in liver to 50.7%TRR in eggs (Table 14). In addition, five to eight unknown minor peaks were seen in the metabolite pattern, each of them in concentrations < 0.01 mg/kg.

Table 14 Summary of identified and quantified metabolites in edible matrices of laying hens after dosing with [^{14}C] cycloxydim-5-OH-TSO at a nominal dose level of 12 mg/kg

Metabolite	Eggs mg/kg (% TRR)	Muscle mg/kg (% TRR)	Fat mg/kg (% TRR)	Liver mg/kg (% TRR)
cycloxydim-5-OH-TSO [a]	0.01 (14.7)	0.007 (23.7)	0.005 (29.0)	0.027 (24.4)
cycloxydim-5-OH-TS	0.034 (50.7)	0.006 (21.9)	0.004 (21.0)	0.021 (19.4)

[a] Sum of Z and E-isomers

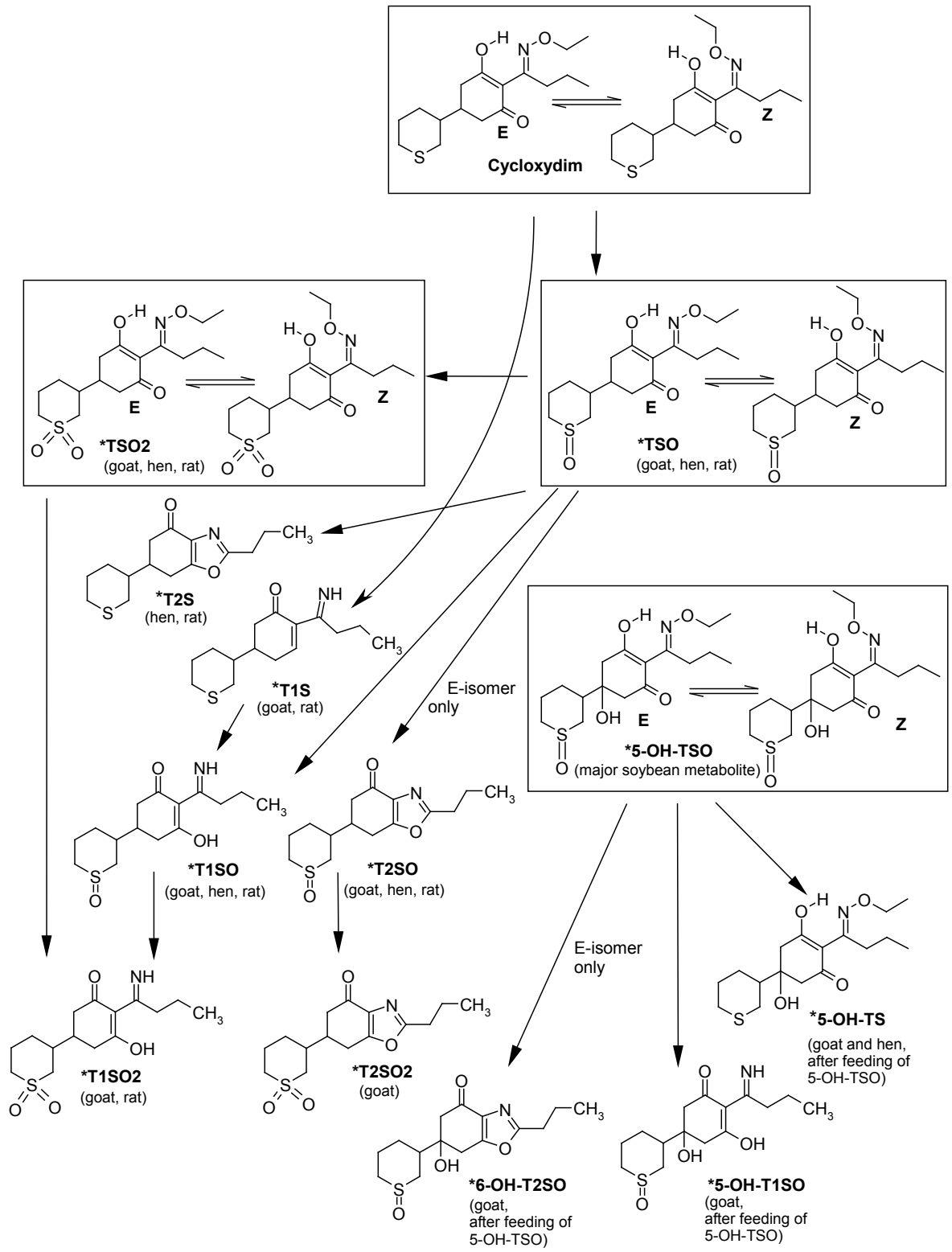


Figure 2 Metabolic pathways of cycloxydim in animals. * cycloxydim

Plant metabolism

Potatoes

A potato study was performed with [^{14}C] cycloxydim EC formulation applied once to potato plants at an exaggerated rate of 2 kg ai/ha (Beutel, 1987a). The plants (variety: Grata-Mittelfruehe) were grown in a mixture of loam/compost and peat under natural Limburgerhof conditions in a glass roof and treated 37 days after sowing. Samples were taken 0 and 24 days after treatment and at crop maturity (73 DAT). Samples were extracted with a mixture of isopropanol and water and the radioactivity determined by LSC. The residual radioactive residue was quantified by combustion analysis. After oxidation with H_2O_2 under alkaline conditions, a methylation step was performed on charcoal. Prior to GC quantitation, the resulting analytes cycloxydim-TDME and cycloxydim-OH-TDME were further purified by liquid/liquid partition and chromatography. No metabolite identification was provided in this report.

TRR accounted for 5.35 mg/kg in potato tubers and 11.54 mg/kg in potato tops (Table 15). Extraction with iso-propanol/water released 96.6% TRR from tubers and 85.5% TRR from tops. The final eluate analysed by GC and quantified using external standard calibration showed cycloxydim-TDME and cycloxydim-OH-TDME at 2.4 and 1.2 mg/kg in the tubers and 2.73 and 1.88 mg/kg, in the tops, respectively.

Table 15 Potatoes: Investigations on extractability and accountability

	Potato Tubers		Potato Tops	
Plant Matrix	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
TRR	5.35	100	11.54	100
Isopropanol/water extract	5.17	96.6	9.87	85.5
Non-extractable residue	0.18	3.4	1.67	14.5
Sum: Waste fractions	0.91	17.0	4.64	42.1
Finale eluate for GC determination	3.59	67.1	4.92	42.6
Overall balance (extractable radioactivity)		87		99

Soya beans, cotton and sugar beet

A study on the uptake and transport of [^{14}C] cycloxydim was performed under controlled climatic conditions in a growth chamber with seedlings of soya beans, cotton and sugar beet (Hamm, 1985a). Two application forms were tested: In one treatment, the plants were cultivated in a nutrient solution containing 5 mg/L of [^{14}C] cycloxydim to evaluate root uptake. To evaluate the uptake by the leaf, 10 µg [^{14}C] cycloxydim was applied to the upper leaf surface of one leaf (soya bean, sugar beet) or one cotyledon (cotton). Samples were taken 3 and 7 days (soya bean and cotton) or 4 and 8 days (sugar beets) after the application, respectively. The plants were divided in two sections, at least, and dried between several layers of filter paper for a week at room temperature. The plant parts were stuck on paper and exposed to X-ray film for about 10 days and the autoradiograms compared with the dried plants. Plant parts were combusted in a sample oxidiser, $^{14}CO_2/CO_2$ absorbed in a liquid scintillation cocktail and radioactivity measured in the liquid scintillation spectrometer. TRRs or metabolite identification were not provided in the report

[^{14}C] cycloxydim was taken up by the soya bean roots and transported acropetally to the cotyledons, stem and the remaining leaves. The highest radioactivity was detected in the primary leaves and roots (Table 16). [^{14}C] cycloxydim was also taken up fast by the cotton and sugar beet roots and transported acropetally to all upper plant parts. The compound was taken up fast within 4 days by the sugar beets roots and transported acropetally to all upper plant parts (Table 16).

Table 16 Translocation behaviour of [^{14}C] cycloxydim after root application, in µg [^{14}C] cycloxydim/ ^{14}C-equiv

Plant Part	Soya bean		Cotton		Sugar Beet	
	3 DAT	7 DAT	3 DAT	7 DAT	4 DAT	8 DAT
Roots	10.8	26.3	8.3	7.8	8.2	8.6
Stem	3.3	3.7	1.2	5.7	–	–
Cotyledons	3.1	4.6	8.4	14.5	2.5	1.6
First pair of leaves	17.8	24.9	–	–	13.7	16.7
Shoot tip/top	4.7	14.6	1.5	1.7	–	–
Balance	39.7	74.1	19.4	29.7	24.4	26.9

Table 17 shows the results for the leaf application. Radioactivity is distributed through the soya bean plant within three days. The preferred translocation is acropetal to the shoot tip. About 23% of the total radioactivity is translocated from the treated primary leaf to the untreated plant parts. A leaf application results in a weak distribution of the labelled compound in the cotton seedlings, with 5% of the applied radioactivity found in untreated plant parts after 3 days. A similar distribution behaviour of the sugar beet seedlings compared to the soya bean and cotton seedlings can be seen.

Table 17 Translocation behaviour of [^{14}C] cycloxydim after leaf application, in µg [^{14}C] cycloxydim/ ^{14}C-equivalents

Plant Part	Soya bean		Cotton		Sugar Beet	
	3 DAT	7 DAT	3 DAT	7 DAT	4 DAT	8 DAT
Treated leaf	6.4	7.3	5.9	6.0	9.7	7.6
Untreated plant part	1.9	2.2	0.3	0.5	0.7	0.9
Stem	0.4	0.4	0.1	0.1		
Shoot tip	0.7	1.1	n.d.	0.1		
Cotyledons	0.2	0.3	–	–	0.1	0.2
Roots	0.3	0.1	0.03	0.02		
Balance	8.3	9.5	6.2	6.5	10.4	8.5
Transport rate %	23	23	5	3	7	11

Sugar beets

Sugar beet plants were cultivated in a growth chamber simulating European climatic conditions (Huber and Schepers, 1986a). At the 3-leaves stage, the seedlings were treated with [^{14}C] cycloxydim at 0.2 kg ai/ha. Samples were taken at 0, 7, 22, 46 and 77 days after treatment and after 119 days the mature plant was harvested. The samples were extracted with aqueous methanol, and radioactivity characterized by liquid/liquid partition using dichloromethane at pH 13 and pH 2. The quantitation of the individual metabolites in tops is based on radio TLC/HPLC of the dichloromethane phases and the metabolites were characterized by mass spectrometry. Due to the low amounts of radioactivity in roots, no further investigations were carried out.

TRRs (extractable and non-extractable) of tops and roots are summarized in Table 18. Immediately after application, 8.48 mg/kg were found in tops and 0.13 mg/kg at harvest. In roots, only small amounts were present at harvest. Extractability with aqueous methanol ranged from 66.2 to 99.3% TRR. Partition of the tops (0 DAT) methanol extracts with dichloromethane at pH 2 yielded 90.2% TRR. At later sampling intervals, the aqueous solubles were predominant.

Table 18 Sugar beet total radioactive residues and extraction behaviour

Plant matrix (DAT)	TRR	Methanol	Residue	DCM (pH 2)	DCM (pH 13)	Aqueous Phase
	mg/kg	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)	mg/kg (%TRR)
Tops (0)	8.48	8.42 (99.3)	0.06 (0.7)	7.65 (90.2)	0.13 (1.5)	0.34 (4.0)
Tops (7)	4.39	4.28 (97.5)	0.11 (2.5)	3.18 (72.4)	0.05 (1.1)	0.89 (20.3)
Tops (22)	1.28	1.20 (93.8)	0.08 (6.3)	0.59 (46.1)	0.11 (8.6)	0.56 (43.8)
Roots (22)	0.31	0.28 (90.3)	0.03 (9.7)	0.11 (35.5)	0.03 (9.7)	0.08 (25.8)
Tops (46)	0.67	0.62 (92.5)	0.05 (7.5)	0.23 (34.3)	0.02 (3.0)	0.40 (59.7)

Plant matrix (DAT)	TRR mg/kg	Methanol mg/kg (%TRR)	Residue mg/kg (%TRR)	DCM (pH 2) mg/kg (%TRR)	DCM (pH 13) mg/kg (%TRR)	Aqueous Phase mg/kg (%TRR)
Roots (46)	0.12	0.10 (83.3)	0.02 (16.7)	0.03 (25.0)	0.01 (8.3)	0.04 (33.3)
Tops (77)	0.27	0.25 (92.6)	0.02 (7.4)	0.07 (25.9)	0.01 (3.7)	0.12 (44.4)
Roots (77)	0.23	0.22 (95.6)	0.01 (4.4)	0.01 (6.1)	0.01 (4.8)	0.02 (8.7)
Tops (119)	0.13	0.086 (66.2)	0.045 (34.6)	0.026 (20.0)	0.004 (3.1)	0.075 (57.7)
Roots (119)	0.015	0.011 (73.3)	0.004 (26.7			

The identification/characterisation of the metabolites present in the dichloromethane phases of tops (0–46 DAT) are shown in Table 19. In samples taken immediately after application and at 7 DAT, only trace amounts of the parent molecule was detected. At early sampling intervals the metabolites cycloxydim-TSO and cycloxydim-TSO2 formed the major part of the radioactivity identified. At later sampling points, the cleavage products cycloxydim-T1SO, cycloxydim-T1SO2 and the oxazole derivatives cycloxydim-T2SO and cycloxydim-T2SO2 resulting from a Beckmann re-arrangement were found. No hydroxylated metabolites have been detected in any sugar beet sample.

Table 19 Identification and characterisation of extractable radioactivity in unripe sugar beet tops

Metabolite	Tops (0 DAT) mg/kg (% TRR)	Tops (7 DAT) mg/kg (% TRR)	Tops (22 DAT) mg/kg (% TRR)	Tops (46 DAT) mg/kg (% TRR)
Cycloxydim	0.32 (3.8)	0.08 (1.8)	n.d.	n.d.
cycloxydim-TSO	6.14 (72.4)	1.47 (33.5)	n.d.	0.023 (3.5)
cycloxydim-TSO2	0.87 (10.3)	0.88 (20.0)	0.18 (13.9)	n.d.
cycloxydim-T1SO	n.d.	n.d.	n.d.	0.014 (2.2)
cycloxydim-T2SO	n.d.	n.d.	0.14 (10.8)	0.005 (0.9)
cycloxydim-T2SO2	n.d.	n.d.	0.007 (5.5)	0.0026 (4.0)
cycloxydim-T1SO2	n.d.	n.d.	n.d.	0.013 (2.0)
Polar	n.d.	n.d.	n.d.	0.022 (3.3)

Sugar beet plants (variety Victoria) were grown in a loamy sand soil under natural Limburgerhof conditions (Veit, 2002a). Cycloxydim was applied in form of the EC formulation to the plants at a rate of 650 g ai/ha 2 months after sowing (BBCH growth stage 17/18, before row closure). Samples were taken 1 day after treatment and at crop maturity (94 DAT). TRR radioactive residues were determined after combustion. The samples were extracted with methanol and water followed by ammonia treatment. For further characterisation of the radioactivity present in the methanol extracts liquid/liquid partition experiments were performed with dichloromethane and ethyl acetate at two different pH values. The mature sugar beet leaf sample was additionally extracted with methanol/aqueous buffer solution for quantitation of cycloxydim-TSO. In sugar beet root (94 DAT) the residual radioactive residues (post extraction solid) were further analysed by treatment with an aqueous ammonia solution. Identification of metabolites was based on LC-MS and HPLC retention time. A very polar peak detected in ripe roots was characterized by yeast digestion.

TRRs and extraction behaviour are summarized in Table 20. One day after application, 24 mg/kg and 4 mg/kg were detected in tops and roots, mostly extracted in methanol. These levels decreased considerably at 94 DAT. Up to 12.2%TRR remained unextracted in both samples at harvest.

Table 20 Sugar beet total radioactive residues and extraction behaviour

Matrix (DAT)	TRR [a]	MeOH Extract		Aqueous Extract		ERR		Residue (RRR)	
	[mg/kg]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
Leaf (1 DAT)	24.0	22.83	95.1	0.572	2.4	23.40	97.5	0.594	2.5
Root (1 DAT)	3.98	3.86	96.8	0.083	2.1	3.94	98.9	0.044	1.1
Leaf (94 DAT)	2.236	1.98	88.6	0.100	4.5	2.081	93.1	0.155	6.9
Root (94 DAT)	0.116	0.093	80.5	0.008	7.3	0.101	87.8	0.014	12.2

[a] TRR calculated: TRR = ERR + RRR

The methanol extracts were further characterized by liquid/liquid partition experiments. One day after treatment, most of the radioactivity was found in the dichloromethane phase (Table 21). At harvest, the amounts of radioactivity in the dichloromethane phase significantly decreased in roots (13.7%); the ethyl acetate phases accounted for approximately 20% of the TRR. The highest concentrations were detected in the aqueous phase (44.7% TRR).

Table 21 Sugar beet: Partition behaviour

Plant Matrix (DAT)	MeOH, mg/kg	DCM, [a] mg/kg (% TRR)	Ethyl acetate (pH 7), mg/kg (% TRR)	Ethyl acetate (pH 2), mg/kg (% TRR)	Water, mg/kg (% TRR)
Leaf (1 DAT)	22.831	15.221 (63.4)	0.982 (4.1)	1.986 (8.3)	4.659 (19.4)
Root (1 DAT)	3.856	3.278 (82.3)	0.124 (3.1)	0.282 (7.1)	0.179 (4.5)
Leaf (94 DAT)	1.981	0.198 (8.8)	0.129 (5.8)	0.311 (13.9)	1.296 (58.0)
Root (94 DAT)	0.093	0.016 (13.7)	0.006 (4.9)	0.018 (15.7)	0.052 (44.7)

[a] Dichloromethane

A summary of the metabolites identified in the extracts is shown in Table 22. Cycloxydim was not detected in any of the samples taken. One day after treatment, residues mainly consisted of the oxidation products cycloxydim-TSO (sulfoxide) and cycloxydim-TSO2 (sulphone). At harvest, both metabolites were still present, but the predominant metabolites were formed by cleavage of the oxime ether side chain. Metabolites as the imines cycloxydim-T1SO, cycloxydim-T1SO2 and cycloxydim-T2SO were present in amounts greater than 10% of the TRR. The subsequent cleavage of the cyclohexenone ring system resulted in the glutaric acid cycloxydim-TGSO2 which was detected in all samples investigated. In the samples harvested at crop maturity, a very polar peak eluting with the void volume from the HPLC column was identified as sugar (most likely as [^{14}C] glucose). No hydroxylation in position 5 of the cyclohexenone ring system was observed.

Table 22 Identification and characterisation of extractable radioactivity in sugar beet samples

Metabolite	Tops (1 DAT)	Roots (1 DAT)	Tops (94 DAT)	Roots (94 DAT)
	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)	mg/kg (% TRR)
cycloxydim-TSO	7.584 (31.6)	2.392 (60.1)	0.049 (2.1)	0.008 (7.3)
cycloxydim-TSO2	3.863 (16.1)	0.710 (17.8)	0.017 (0.7)	0.002 (1.8)
cycloxydim-T1SO	3.327 (13.9)	0.029 (0.7)	0.423 (18.9)	0.017 (14.5)
cycloxydim-T2S	2.746 [a] (11.4)	0.392 [a] (9.8)	0.306 (13.7)	0.001 (1.0)
cycloxydim-T1SO2			0.118 (5.2)	0.005 (4.7)
cycloxydim-TGSO2	1.734 (5.7)	0.034 (0.8)	0.160 (7.1)	0.010 (8.8)
Sugar fraction	n.d.	n.d.	0.002 (0.1)	0.010 (8.8)

[a] Due to peak overlapping in the HPLC chromatograms, the sum is given

n.d.=not detected

Soya beans

The metabolism of [^{14}C] cycloxydim in soya beans was investigated by Huber and Hamm (1986a) using four different treatment groups. In three trials, the EC formulation at 0.2 kg ai/ha was applied to the plant (variety: SRF 450) two to eight weeks after sowing and cultivated in a growth chamber in a mixture of Limburgerhof soil and peat. In one trial, the soya bean plants (variety: Pfitzer) were treated with 1 kg ai/ha two months after sowing and cultivated in a greenhouse located in a mixture of Limburgerhof soil and peat.

Plant and straw samples taken were extracted with aqueous methanol. Seed samples extraction was first performed with hexane (for defatting) followed by methanol extraction. The aqueous methanol extracts were adjusted to pH = 9 and evaporated. The remaining aqueous extracts were partitioned between dichloromethane and water. After acidification (pH = 2) a further partition was performed. The remaining aqueous phase of plant and straw samples was evaporated to dryness

prior to methylation and liquid/liquid partition to convert metabolites as cycloxydim-TGSO2 in the corresponding methyl esters. For the characterisation of the residual radioactive residue after solvent extraction, several procedures were used, including acid and/or base digestion and refluxing and derivatisation with phenyl hydrazine (cellulose, lignin). The remaining aqueous phase from seed samples was concentrated, adjusted to pH 2 and partitioned between ethyl acetate and water. Another aliquot of the aqueous MeOH extract was used for a precipitation reaction with phosphoric acid, selective for proteins. Identification of metabolites was based on radio-TLC analysis of the dichloromethane phase (seed), LC-MS and HPLC.

TRR of the different treatment groups calculated from the extractable residue (ERR) and the non-extractable residue (RRR) are summarized in Table 23. In most plant samples, more than 85% of the TRR were extracted with aqueous methanol. Only minor amounts of radioactivity were soluble in hexane, which was used for removing fats from the seeds. In case of straw, slightly lower amounts were dissolved in aqueous methanol (approximately 70% of the TRR). Organo soluble metabolites were only predominant in plant samples taken 0 DAT. At later sampling intervals, considerable amounts of radioactivity were detected in the aqueous phase. In straw, about 50% of the TRR were found in these phases whereas for seeds about 20% were characterized as aqueous solubles (Table 24).

Table 23 Soya bean total radioactive residues and extraction behaviour

Plant Matrix	TRR [a]	Hexane Extracts	MeOH Extracts		RRR	
(DAT)	[mg/kg]	mg/kg (% TRR)	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
Treatment group 1 (V 43/84): 0.2 kg ai/ha						
Plant (0 DAT)	11.11	n.a.	10.86	97.7	0.25	2.3
Plant (7 DAT)	8.47	n.a.	8.06	95.2	0.41	4.8
Straw (45 DAT)	12.52	n.a.	9.83	78.5	2.69	21.5
Seed (45 DAT)	20.03	0.35 (1.7)	18.31	91.4	1.37	6.8
Treatment group 2 (V 45/84): 0.2 kg ai/ha						
Plant (0 DAT)	43.65	n.a.	42.51	97.4	1.14	2.6
Plant (35 DAT)	3.27	n.a.	2.79	85.3	0.48	14.7
Straw (71 DAT)	0.76	n.a.	0.54	71.1	0.22	28.9
Seed (71 DAT)	2.30	0.12 (5.2)	2.06	89.6	0.12	5.2
Treatment group 3 (V 45/84A): 0.2 kg ai/ha						
Plant (0 DAT)	16.61	n.a.	16.44	99.0	0.17	1.0
Plant (7 DAT)	6.46	n.a.	6.00	92.9	0.46	7.1
Plant (14 DAT)	3.32	n.a.	2.90	87.3	0.42	12.7
Plant (21 DAT)	2.88	n.a.	2.48	86.1	0.40	13.9
Plant (40 DAT)	0.72	n.a.	0.61	84.7	0.11	15.3
Leaves (82 DAT)	5.63	n.a.	4.08	72.5	1.55	27.5
Stalks (82 DAT)	0.31	n.a.	0.22	71.0	0.09	29.0
Pods (82 DAT)	0.53	n.a.	0.36	67.9	0.17	32.1
Seeds (82 DAT)	0.46	0.02 (4.4)	0.39	84.8	0.05	10.9
Treatment group 4 (V 49/84): 1 kg ai/ha						
Straw (69 DAT)	91.00	n.a.	73.10	80.3	17.90	19.7
Seeds (69 DAT)	38.40	0.09 (0.3)	37.62	98.0	0.69	1.8

[a] TRR calculated: TRR = ERR + RRR, ERR: extractable radioactive residue, RRR: residual radioactive residue

n.a. Not applicable

Table 24 Soya bean partition behaviour

Plant Matrix	MeOH	DCM (pH 9)		DCM (pH 2)		Aqueous Phase	
(DAT)	[mg/kg]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
Treatment group 1 (V 43/84): 0.2 kg ai/ha							
Plant (0 DAT)	10.86	0.35	3.2	9.45	85.1	0.42	3.8
Plant (7 DAT)	8.06	0.68	8.0	5.10	60.2	1.74	20.5
Straw (45 DAT)	9.83	1.28	10.2	1.11	8.9	6.64	53.0
Seed (45 DAT)	18.31	8.86	44.2	4.47	22.3	3.59	17.9
Treatment group 2 (V 45/84): 0.2 kg ai/ha							
Plant (0 DAT)	42.51	2.89	6.6	33.58	76.9	1.54	3.5

Plant Matrix	MeOH	DCM (pH 9)		DCM (pH 2)		Aqueous Phase	
(DAT)	[mg/kg]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
Plant (35 DAT)	2.79	0.37	11.3	0.81	24.8	1.36	41.6
Straw (71 DAT)	0.54	0.04	5.3	0.06	7.9	0.41	53.9
Seed (71 DAT)	2.06	0.16	7.0	0.75	32.6	0.35	15.2
Treatment group 3 (V 45/84A): 0.2 kg ai/ha							
Plant (0 DAT)	16.44	1.19	7.2	14.02	84.4	0.66	4.0
Plant (7 DAT)	6.00	0.78	12.1	3.41	52.8	1.58	24.5
Plant (14 DAT)	2.90	0.34	10.2	1.14	34.3	0.97	29.2
Plant (21 DAT)	2.48	0.29	10.1	0.71	24.7	1.09	37.9
Plant (40 DAT)	0.61	0.05	6.9	0.14	19.4	0.40	55.6
Leaves (82 DAT)	4.08	0.67	11.9	0.27	4.8	3.21	57.0
Stalks (82 DAT)	0.22	-	-	0.02	6.5	0.18	58.1
Pods (82 DAT)	0.36	0.05	9.4	0.05	9.4	0.19	35.9
Seeds (82 DAT)	0.39	0.10	21.7	0.13	28.3	0.11	23.9
Treatment group 4 (V 49/84): 1 kg ai/ha							
Straw (69 DAT)	73.10	12.80	14.1	11.80	13.0	48.30	53.1
Seeds (69 DAT)	37.62	2.47	6.4	22.63	58.9	6.75	17.6

Straw from treatment group 4 was used for the characterisation of the non-extracted radioactivity (RRR), which corresponded to 19.7%TRR. Most of the radioactivity released was found in the lignin fractions (67.8%).

The parent molecule was detected only at the day of application (Tables 25–28). It rapidly degraded and formed various metabolites. Hydroxylated metabolites were a major part of the radioactivity identified in seed. For plant and straw, the aqueous phases after partition were predominant at later sampling intervals. In treatment group 4, incorporation into the protein pool occurred to a low extent (2.03 mg/kg, 5.3% TRR).

Table 25 Identification and characterisation of the extractable radioactivity in soya bean samples (treatment group 1, 0.2 kg ai/ha), mg/kg (% TRR)

Metabolite	Plant (0 DAT)	Plant (7 DAT)	Straw (45 DAT)	Seeds (45 DAT)
Cycloxydim	1.40 (12.6)	n.d.	n.d.	n.d.
cycloxydim-TSO	8.03 (72.3)	4.13 (48.7)	1.30 (10.4)	2.38 (11.9)
cycloxydim-TSO2	n.d.	0.32 (3.8)	n.d.	0.24 (1.2)
cycloxydim-T1SO	n.d.	n.d.	0.22 (1.8)	0.65 (3.2)
cycloxydim-T1SO2	0.09 (0.8)	0.27 (3.2)	0.26 (2.0)	0.19 (0.9)
cycloxydim-T2SO	0.05 (0.5)	0.30 (3.5)	0.85 (7.7)	3.70 (18.5)
cycloxydim-T2SO2	n.d.	0.14 (1.7)	0.32 (2.6)	0.95 (4.7)
cycloxydim-5-OH-TSO	n.d.	n.d.	0.26 (2.1)	1.28 (6.4)
cycloxydim-5-OH-TSO2	n.d.	n.d.	n.d.	0.90 (4.5)
cycloxydim-6-OH-T2SO	n.d.	n.d.	n.d.	0.88 (4.4)
cycloxydim-6-OH-T2SO2	n.d.	n.d.	n.d.	0.90 (4.5)
cycloxydim-TGSO [a]	n.p.	0.3 (3.5)	1.63 (13.0)	n.p.
cycloxydim-TGSO2 [a]	n.p.	0.18 (2.1)	0.96 (7.7)	n.p.
Aqueous phase, polar	n.p.	0.77 (9.1)	3.22 (25.7)	n.p.

n.d .= Not detected

n.p .= Further characterisation/methylation not performed

[a] determined as methyl ester.

Table 26 Identification and characterisation of the extractable radioactivity in soya bean samples (treatment group 2, 0.2 kg ai/ha), mg/kg (% TRR)

Metabolite	Plant (0 DAT)	Plant (35 DAT)	Seeds (71 DAT)
Cycloxydim	0.09 (0.2)	n.d.	n.d.
cycloxydim-TSO	35.11 (80.4)	0.70 (21.4)	0.42 (18.3)
cycloxydim-TSO2	n.d.	0.16 (4.9)	0.07 (3.0)
cycloxydim-T1SO	0.41 (0.9)	0.06 (1.8)	0.02 (0.9)
cycloxydim-T1SO2	n.d.	0.04 (1.2)	n.d.

Metabolite	Plant (0 DAT)	Plant (35 DAT)	Seeds (71 DAT)
cycloxydim-T2SO	0.86 (2.0)	0.07 (2.2)	0.11 (4.8)
cycloxydim-5-OH-TSO	n.d.	0.11 (3.4)	0.20 (8.7)
cycloxydim-5-OH-TSO2	n.d.	n.d.	0.26 (11.3)

Table 27 Identification and characterisation of the extractable radioactivity in unripe soya bean plant samples (treatment group 3, 0.2 kg ai/ha), (% TRR)

Metabolite	0 DAT	7 DAT [a]	14 DAT	21 DAT	40 DAT [a]
Cycloxydim	0.11 (0.7)	n.d.	n.d.	n.d.	n.d.
cycloxydim-TSO	14.60 (87.9)	0.10 (1.5)	1.09 (32.8)	0.67 (23.3)	0.14 (19.4)
cycloxydim-TSO2	n.d.	n.d.	0.08 (2.4)	0.06 (2.1)	n.d.
cycloxydim-T1SO	0.15 (0.9)	0.33 (5.1)	0.16 (4.8)	0.11 (3.8)	n.d.
cycloxydim-T1SO2	0.10 (0.6)	0.06 (0.9)	0.02 (0.6)	0.02 (0.7)	n.d.
cycloxydim-T2SO	0.25 (1.5)	0.11 (1.7)	0.06 (1.8)	0.04 (1.4)	n.d.

[a] Only one DCM phase was investigated/reported

Table 28 Identification and characterisation of the extractable radio-activity in ripe soya bean samples

Metabolite	Group 3, 0.2 kg ai/ha, mg/kg (% TRR)		Group 4, 1 kg ai/ha, mg/kg (% TRR)	
	Leaves (82 DAT)	Seeds (82 DAT)	Straw (69 DAT)	Seeds (69 DAT)
cycloxydim-TSO	0.27 (4.8)	0.078 (17.0)	9.67 (10.6)	10.29 (26.8)
cycloxydim-TSO2	n.d.	0.012 (2.6)	3.18 (3.5)	2.75 (7.2)
cycloxydim-T1SO	0.12 (2.1)	n.d.	1.66 (1.8)	0.08 (0.2)
cycloxydim-T1SO2	0.06 (1.1)	0.003 (0.7)	1.62 (1.8)	0.05 (0.1)
cycloxydim-T2SO	0.29 (5.2)	0.029 (6.3)	3.25 (3.6)	0.65 (1.7)
cycloxydim-T2SO2			n.d.	0.30 (0.8)
cycloxydim-5-OH-TSO	n.d.	0.041 (8.9)	n.d.	8.35 (21.8)
cycloxydim-5-OH-TSO2	n.d.	0.055 (12.0)	n.d.	5.80 (15.1)
cycloxydim-6-OH-T2SO			n.d.	0.05 (0.1)
cycloxydim-6-OH-T2SO2			n.d.	0.28 (0.7)
cycloxydim-TGSO			17.3 (19.0)	n.p.
cycloxydim-TGSO2			8.3 (9.1)	n.p.
Remaining aqueous phase			17.3 (19.0)	n.p.
Polar metabolites			23.4 (25.7)	n.p.

For additional characterisation, selected extracts, phases and samples were subjected to a comparable oxidation procedure. The results confirm the findings and the identification of the metabolites (Table 29).

Table 29 Characterisation of the extracted radioactivity by derivatisation (oxidation and methylation), mg/kg (% TRR)

	Plant [a] 0 DAT	Plant [a] 7 DAT	Plant [a] 45 DAT	Seed [b] 69 DAT	Straw [b] 69 DAT
TRR	11.11	8.47	12.52	17.72	91.00
Radioactivity in extract/phase/sample	10.86 (97.6)	8.06 (95.2)	9.83 (78.5)	17.72 (100)	48.3 (53.1)
Radioactivity after oxidation procedure	10.82 (97.2)	8.16 (96.3)	9.65 (77.1)	17.33 (97.8)	48.00 (52.7)
Organo solubles after methylation	9.43 (84.7)	7.52 (88.8)	7.40 (59.1)	13.11 (74.0)	32.1 (35.3)
cycloxydim-T-DME	9.32 (83.7)	6.96 (82.2)	6.66 (53.2)	10.13 (57.2)	26.6 (29.2)
cycloxydim-OH-TDME	n.d.	0.56 (6.6)	0.74 (5.9)	2.44 (13.8)	3.90 (4.3)
Remaining aqueous phase	0.62 (5.6)	0.54 (6.4)	1.18 (10.2)	2.45 (13.8)	11.00 (12.1)

[a] Treatment group 1

[b] Treatment group 4

Tolerant maize

The metabolism of cycloxydim in maize (variety: Scarlet) was investigated using two different use patterns (Hofmann, 1997a,b; Bross, 1998a). In the first study, cycloxydim was applied as an EC formulation at a rate of 0.4 kg ai/ha at BBCH growth stage 22-23 (4–5 leaves unfolded). In the second study the post emergence application was performed with an exaggerated rate (0.8 kg ai/ha) during flowering (BBCH 61-67). In both studies, the maize plants were grown in pots filled with loamy sand in a chamber simulating the climate at 50 ° latitude. At harvest, the plant samples were separated into leaves, straw, flags, cobs and seeds. After homogenisation, TRR was determined by combustion analysis. The sample material was extracted with methanol, water, and mixtures thereof. Prior to methanol extraction, grain was defatted with cyclohexane. The methanol and the aqueous extracts were further characterized by liquid/liquid partition with ethyl acetate at two different pH values (neutral, acid).

The identification and characterisation of extractable radioactivity was based on LC-MS of isolated fractions or methylation products and HPLC retention time. During the study, an accountability experiment was performed using the ethyl acetate phases (neutral, acid) of straw and grain (0.8 kg ai/ha) to determine the amounts of organo soluble radioactivity covered by the common moiety method 407, which includes the oxidation of oxydim and its metabolites to cycloxydim-TGSO2 and cycloxydim-5-OH-TGSO2. Selected aqueous phases were incubated with a mixture of β-glucosidase and hesperidinase. The non-released radioactivity was characterized by ammonia extraction and enzyme treatment. The data are summarized in Table 30. In the normal use rate, residue levels in the harvest samples ranged from 0.060 to 0.168 mg/kg and in the exaggerated rate samples, the TRRs were considerably higher. ERR ranged from 63.7 to 86.1% TRR for the exaggerated rate and higher than 60% TRR only for forage

Table 30 Tolerant maize: Total radioactive residues and extraction behaviour

Plant Matrix	TRR [a]	Hexane	MeOH	Aqueous	ERR		RRR	
(DAT)	[mg/kg]	[mg/kg] (% TRR)	[mg/kg] (% TRR)	[mg/kg] (% TRR)	[mg/kg]	[% TRR]	[mg/kg]	[% TRR]
Normal use rate: 0.4 kg ai/ha								
Forage (72 DAT)	31.381	n.a.	25.0 (79.8)	2.78 (8.9)	27.818	88.7	3.564	11.4
Grain (96 DAT)	0.123	0.007 (6.1)	0.012 (9.2)	0.011 (8.9)	0.030	24.2	0.094	75.9
Straw (96 DAT)	0.168	n.a.	0.087(52.2)	0.011(6.6)	0.098	58.8	0.069	41.1
Husks (96 DAT)	0.118	n.a.	0.033(28.5)	0.007(5.9)	0.040	34.4	0.076	64.6
Cobs (96 DAT)	0.060	n.a.	0.017(29.4)	0.003(3.9)	0.020	33.3	0.040	66.0
Exaggerated use rate: 0.8 kg ai/ha								
Grain (54 DAT)	4.928	0.37 (7.6)	2.54 (51.6)	1.33 (26.9)	4.236	86.1	0.693	14.1
Straw (54 DAT)	13.023	n.a.	8.96 (68.8)	1.63 (12.4)	10.590	81.2	2.433	18.7
Husks (54 DAT) [b]	9.478	n.a.	7.180 (75.8)				2.298	24.2
Cobs (54 DAT) [b]	4.296	n.a.	2.735 (63.7)				1.560	36.3

[a] TRR calculated: TRR = ERR + RRR, ERR: Extractable radioactive residue, RRR: Residual radioactive residue

[b] Extraction was performed with methanol/water (1:1)

n.a.= Not applicable

The methanol and/or aqueous extracts of selected samples were partitioned between water and ethyl acetate at neutral and acid pH. In case of grain and forage, the organo soluble metabolites were predominant whereas for straw most of the radioactivity was found in the aqueous phase (Table 31).

Table 31 Tolerant maize: Partition behaviour

Plant Matrix	Organo Soluble		Aqueous Soluble	
(DAT)	mg/kg	% TRR	mg/kg	% TRR
Normal use rate: 0.4 kg ai/ha				
Forage (72 DAT)	13.912	44.3	11.483	36.6
Grain (96 DAT)	0.020	16.1	0.012	9.1
Straw (96 DAT)	0.028	16.1	0.059	34.9

Plant Matrix	Organo Soluble		Aqueous Soluble	
(DAT)	mg/kg	% TRR	mg/kg	% TRR
Exaggerated use rate: 0.8 kg ai/ha				
Grain (54 DAT)	2.716	55.1	1.588	32.2
Straw (54 DAT)	4.353	33.6	6.186	47.5

In Table 32, the results of the attempts to solubilise the residual radioactive residues (RRR) are summarized. From 4.3 to 8.4% TRR was solubilised by ammonia extraction. HPLC analysis of these extracts showed that they consisted of the same metabolites as the methanol and aqueous extracts. Treatment with the starch cleaving enzymes amyloglucosidase (to glucose) and amylase (to maltose and iso-maltose) 57.0% TRR could be dissolved from the grain RRR. In the case of straw (400 g ai/ha), An additional 22.6% TRR was dissolved by NaOH extraction, specific to lignin and cellulose, from straw. Most of the non-organoextractable radioactivity was associated with carbohydrates. Up to 18.6% TRR still remained unextracted in grain.

Table 32 Characterisation of non-released radioactivity in tolerant maize

	0.4 kg ai/ha, mg/kg (% TRR)			0.8 kg ai/ha, mg/kg (% TRR)	
	Forage	Grain	Straw	Grain	Straw [a]
RRR	3.564 (11.4)	0.094 (75.9)	0.069 (41.1)	0.693 (14.1)	1.580 (17.3)
Ammonia	1.355 (4.3)	0.008 (6.1)	0.014 (8.4)	0.352 (7.2)	0.681 (7.5)
Amyloglucosidase	n.a.	0.043 (34.4)	n.a.	n.a.	n.a.
Amylase	n.a.	0.028 (22.6)	n.a.	n.a.	n.a.
NaOH	n.a.	n.a.	0.038 (22.6)	n.a.	n.a.
Final residue	2.182 (7.0)	0.023 (18.6)	0.016 (9.5)	0.328 (6.7)	0.812 (8.9)

n.a. Not applicable

[a] Additional work-up

The identified metabolites of both treatment groups are shown in Table 33. Cycloxydim was rapidly and extensively metabolized in all samples under investigation and was not detected in any sample. The metabolite patterns were qualitatively similar for the different application rates. In the exaggerated rate grain sample, metabolites cycloxydim-TSO and cycloxydim-TSO2 were the most prominent peaks in the HPLC chromatogram. In all normal use rate samples except forage, the most prominent peak was very polar and eluted with the void volume from the HPLC column. All other metabolites were only present in trace amounts. The metabolites formed were further degraded. It could be shown that incorporation into the carbohydrate pool occurred.

Table 33 Identification and characterisation of the radioactivity in cycloxydim tolerant maize

	0.4 kg ai/ha, mg/kg (% TRR)				0.8 kg ai/ha, mg/kg (% TRR)			
Metabolite	Forage	Grain	Straw	Husks	Grain	Straw	Husks	Cobs
cycloxydim-TSO	1.34 (4.3)	0.001 (0.6)	n.d.	n.d.	0.529 (10.6)	0.288 (1.6)	0.106 (1.1)	0.025 (0.6)
cycloxydim-TSO2	1.13 (3.6)	0.0 (0.4)	n.d.	n.d.	0.352 (7.0)	n.d.	0.076 (0.8)	0.020 (0.5)
cycloxydim-5-OH-T1SO	n.d.	+ T1SO2 0.001 (1.0)	n.d.	n.d.	0.383 (7.7)	n.d.	n.d.	n.d.
cycloxydim-6-OH-T2SO/T2SO2	n.d.	0.001 (0.8)	n.d.	n.d.	0.241 (4.8)	n.d.	0.890 (9.4)	0.324 (7.6)
cycloxydim-T1SO	3.61 (11.5)	0.001(0.5)	0.003(1.8)	n.d.	+ T2SO2 0.712(14.2)	1.33 (7.3)	0.750(7.9)	0.226(5.3)
cycloxydim-T1SO2	2.32 (7.4)	+5-OH-T1SO 0.001(1.0)	0.004(2.5)	n.d.	0.263(5.2)	1.77 (9.8)	0.503(5.3)	0.163(3.8)
cycloxydim-T2SO	1.28 (4.1)	0.000(0.3)	0.002(1.3)	n.d.	0.238(4.8)	0.863(4.8)	1.28 (13.5)	0.217(5.0)
cycloxydim-T2SO2	n.d.	0.000(0.2)	n.d.	n.d.	+ T1SO 0.712(14.2)	n.d.	n.d.	n.d.
cycloxydim-TGSO	2.37 (7.5)	n.d.	0.015(8.7)	n.d.	n.d.	1.25 (6.9)	0.331(3.5)	0.20(4.7)
cycloxydim-TGSO2	1.28 (4.1)	n.d.		n.d.	n.d.	1.09 (6.0)	0.350(3.7)	0.153(3.6)

n.d. Not determined

Cycloxydim is degraded by mainly four key transformation steps:

(a) Oxidation at the sulphur of the thiopyrane ring to the sulfoxide and to the sulphone

(b) Cleavage of the oxime ether group (loss of the alkyl side chain)

(c) Hydroxylation at the 5-position of the cyclohexenone ring system

(d) Oxidative cleavage of the cyclohexenone ring resulting in substituted glutaric acid derivatives.

The combination of these reactions, together with the presumably non-enzymatic Beckmann rearrangement leads to a huge number of metabolites. In addition to the parent molecule, eight of them were present in amounts > 0.050 mg/kg and 10% TRR. The proposed metabolic pathway of cycloxydim in plants is shown in Figure 3.

Environmental fate and behaviour in soil

Degradation under aerobic conditions

The aerobic degradation and metabolism of [^{14}C] cycloxydim was studied in a loamy sand (Speyer standard soil; 84% sand, 8% silt, 6.1% humus, 13.8 mVal/100 g cation exchange capacity (CAC), 6.1 pH, 40% maximum water holding capacity (MWC), 26.7 mg C/100 g dry soil) (Huber, 1987a). A 10 mg/kg dry soil was used, equivalent to a maximum single application rate of about 7.5 kg ai/ha, and to a multiple dose of 0.6 kg ai/ha. The soil was incubated at 22 ± 2 °C for 90 day. The soil samples were extracted with dichloromethane (DCM), water and methanol and the extracts analysed by radio-TLC.

Most of the radioactivity was found in the DCM extracts. The distribution of radioactivity (sum of DCM and methanol extracts) over the total incubation period is shown in Table 34. About 14% of the total radioactive residue (TRR: radioactivity in the extract + residual radioactive residues) was detected as cycloxydim at day 0, decreasing to less than 1%TRR after 14 days. A half-life of 3 days was estimated. Two peak clusters were attributable, one to the series cycloxydim-TSO/ cycloxydim-T1SO/ cycloxydim-T2SO, the other to cycloxydim-T1S/ cycloxydim-T2S/ cycloxydim-TSO2. Mass spectrometry, after HPLC purification, showed that the peak cluster 1 of the 56 day DCM extract consisted mainly of cycloxydim-TSO and cycloxydim-T2SO. Bound residues accounted for about 40% TRR after three months and mineralization rate to CO_2 was 38% TRR.

Table 34 % TRR (total radioactive residue) and distribution of metabolites after application of [^{14}C] cycloxydim to loamy sand soil and incubation under aerobic conditions

DAT, days	$^{14}CO_2$	Cycloxydim	Peak cluster 1 [a] (TSO, T1SO, T2SO)	Peak cluster 2 [b] (T1S, T2S, TSO2)	cycloxydim-T2SO2	TLC-start [b]	H_2O-extract	Bound residues	Total
0	0.0	14.1	31.9	5.8	0.0	10.4	8.0	8.9	79.1
1	0.0	9.5	44.6	6.4	0.0	9.8	7.1	10.7	88.1
3	0.4	4.6	49.1	0.3	0.0	10.9	7.7	12.9	85.9
7	2.6	1.2	49.7	0.0	0.0	10.1	9.5	18.4	91.5
14	8.9	0.9	28.9	2.1	2.3	17.2	11.7	19.9	91.9
28	18.1	0.8	28.7	2.7	4.0	16.2	12.6	23.7	106.8
56	29.5	0.0	16.0	0.0	4.9	13.9	13.6	40.0	117.9
90	38.1	0.0	24.3	0.0	9.7	4.7	9.1	38.8	124.7

[a] Peaks in each cluster could not be separated unambiguously on TLC

[b] Radioactivity remained at start of TLC ($R_f = 0$)

The degradation and mineralisation of [^{14}C] cycloxydim was studied in two freshly collected field soils, a loamy sand (Limburgerhof; 0.52% OC, 14.8 mVal/100 g CAC, 15.9% microbial biomass) and a loam soil (Ruchheim; 1.54% OC, 5.2 mVal/100 g CAC, 85.7% microbial biomass)

(Huber, 1988a). Nominal application rate, study condition and sample extraction procedure were the same as in the previous study (Huber, 1987a).

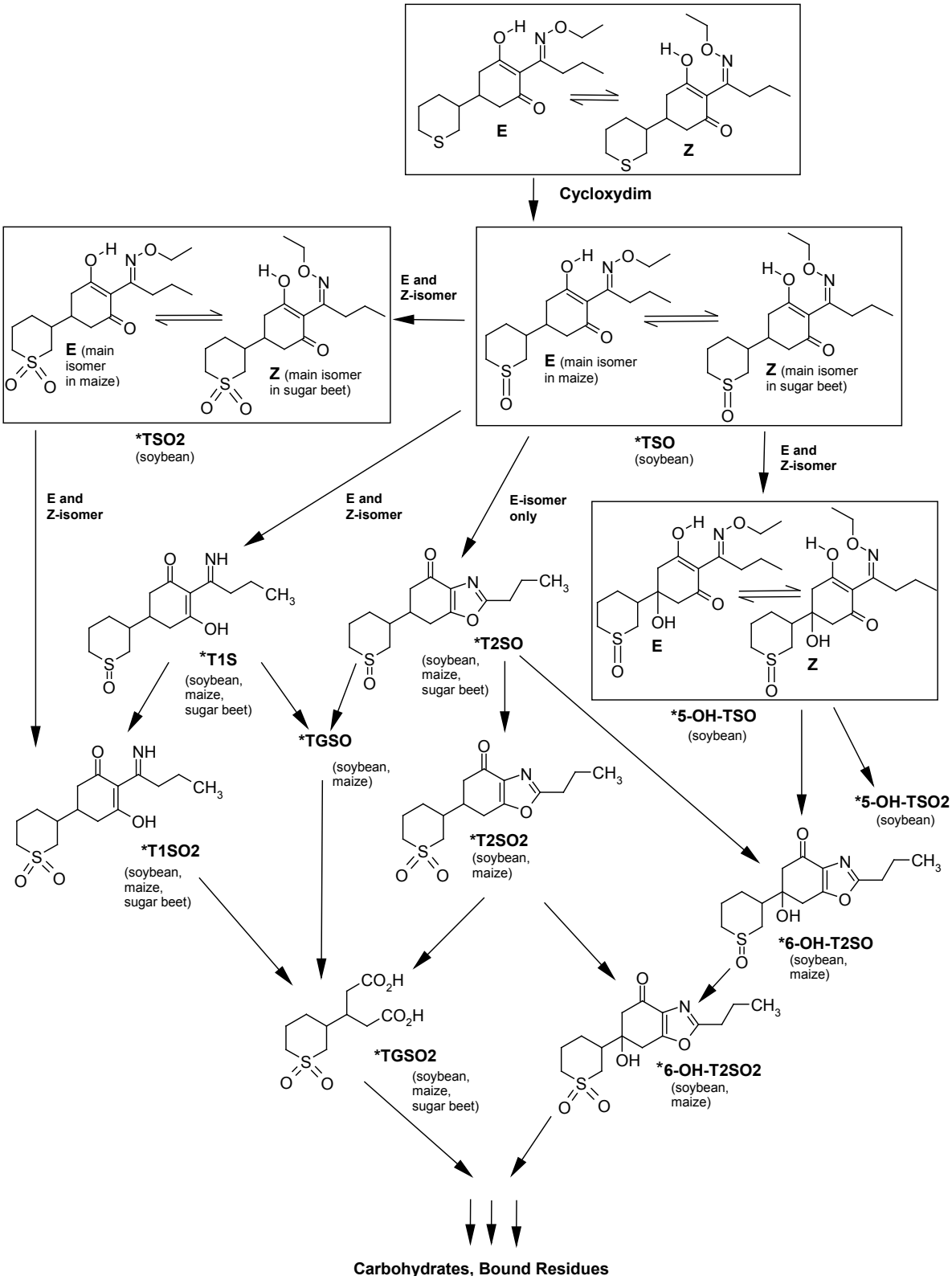


Figure 3 Metabolic pathway of cycloxydim in plants; *cycloxydim

The distribution of radioactivity over the 90 day overall incubation period is shown in Table 35. Only the dichloromethane extracts were analysed by radio-TLC as radioactivity in the water and methanol extracts were low. Cycloxydim accounted for over 90% TRR at day 0, decreasing to 4.6%TRR at day 21 in loamy sand field soil and to 0%TRR in loam soil. To differentiate between cycloxydim-TSO and cycloxydim-T2SO a partition experiment of the DCM residues of the 7 d sample (loamy sand and loam) between water and ethylacetate in alkaline pH was performed. Cycloxydim-TSO is a "vinylogous" acid and remained in the water phase whereas cycloxydim-T2SO would be extracted into the organic phase. By this procedure it could be shown that cluster 1 consisted mainly (≥ 90%) of cycloxydim-TSO.

Table 35 % TRR (total radioactive residue) and distribution of metabolites after application of [^{14}C] cycloxydim to a loamy sand field soil and incubation under aerobic conditions

DAT	$^{14}CO_2$	Cycloxydim	Peak cluster 1 [a] (TSO, T1SO, T2SO)	T1SO2	Peak cluster 2 [a] (TSO2, T2SO2)	Peak cluster 3 [a] (T1S, T2S)	TLC-start [b]	MeOH-extract	H_2O-extract	Bound residues	Total
Loamy sand field soil											
0	0.0	91.7	2.7	0.9	0.0	2.1	1.5	0.5	0.3	0.1	99.8
7	0.4	14.6	39.4	0.0	0.0	0.0	13.4	5.1	7.8	13.9	94.6
21	3.6	4.6	39.5	0.0	2.7	0.0	2.4	10.9	11.7	17.9	93.3
43	16.1	0.0	18.6	0.0	5.5	0.0	9.2	9.0	9.9	23.2	91.5
90	36.2	0.0	11.1	1.1	3.0	1.7	4.2	1.9	7.4	30.7	97.3
Loam field soil											
0	0.0	93.1	3.3	0.0	0.0	2.8	0.0	0.4	0.2	0.2	100.0
7	4.3	0.3	48.2	0.5	3.6	0.0	1.9	4.0	4.2	19.2	86.2
21	23.4	0.0	21.0	1.5	10.5	0.0	0.0	6.1	3.8	28.8	95.1
43	45.7	0.0	3.7	2.0	7.4	0.0	0.0	2.3	5.9	28.6	95.6
90	59.3	0.0	0.9	1.1	1.3	0.4	0.0	0.8	2.3	30.6	96.7

[a] Peaks in each cluster could not be separated unambiguously on TLC

[b] Radioactivity remained at start of TLC ($R_f = 0$)

The aerobic soil degradation and metabolism of [^{14}C] cycloxydim was investigated with various batches of soils treated at 0.8 mg/kg dry soil (Bayer, H 2000a). The soils characteristics are shown in Table 36. The actual application rates (TAR = total applied radioactivity) were calculated as the sum of extractable radioactive residues (ERR) and non-extracted bound residues (RRR = residual radioactive residues) for each soil at day 0. The soils were incubated for 119 days in the dark at 20 °C and 40% maximum water holding capacity. A system with continuous aeration and trapping of volatiles was used. Soil samples were extracted with acetonitrile and acetonitrile/water (1:1) and the extracts analysed by radio-HPLC.

Table 36 Soil characteristics

	LUFA 2.2 F20899 99/736/01	LUFA 2.2 F20899 99/736/02	Bruch West 99/060/01	Bruch West 99/060/02	Li 35 b 99/145/01	Li 35 b 99/145/02	Li 35 b 99/145/04
USDA scheme: (%)	loamy sand	loamy sand	sandy loam	sandy loam	loamy sand	loamy sand	loamy sand
< 2 µm (clay)	3.80	3.84	9.80	7.67	7.68	5.60	7.20
2–50 µm (silt)	16.57	14.98	25.05	29.36	19.31	16.09	20.5
50–2000 µm (sand)	79.62	81.18	65.14	62.97	73.0	78.31	72.2
German scheme: (%)	loamy sand	loamy sand	silty loam	silty loam	loamy sand	loamy sand	loamy sand
< 2 µm (clay)	5.39	7.08	8.07	9.88	6.31	7.29	5.1
2–63 µm (silt)	3.58	4.91	4.28	7.41	4.75	4.49	4.6
63–2000 µm (sand)	91.03	88.02	87.65	82.71	88.93	88.23	90.3
Organic carbon (%)	1.96	1.88	1.63	1.91	1.24	1.16	1.13
NO_3-N (mg/100 g dry soil)	13.5	5.5	0.3	0.6	0.4	0.5	0.9

	LUFA 2.2 F20899 99/736/01	LUFA 2.2 F20899 99/736/02	Bruch West 99/060/01	Bruch West 99/060/02	Li 35 b 99/145/01	Li 35 b 99/145/02	Li 35 b 99/145/04
NH_4-N (mg/100 g dry soil)	0.9	1.5	0.7	0.5	0.7	0.8	0.5
pH ($CaCl_2$)	5.4	5.6	7.2	7.5	6.5	6.5	6.6
CEC (mVal/100 g)	9.8	10.9	12.7	13.9	7.1	7.7	8.5
MWC (g H_2O/100 g dry soil)	43.4	46.4	40.7	44.5	34.0	36.7	34.0
Microbial biomass (mg C/100 g dry soil)	36.0	29.4	27.9	22.4	26.8	18.8	20.2

The peak pattern of the soils Bruch West and Li 35 b was very similar (Table 37). The degradation of cycloxydim was very fast in both soils, with less than 10% of the applied ai found at 0 DAT. The detected metabolites were identified by mass spectrometry and co-chromatography as cycloxydim-TSO, cycloxydim-TSO2, and cycloxydim-T2SO.

Table 37 % TAR (total applied radioactivity) and distribution of metabolites in soil after application of [^{14}C] cycloxydim under aerobic conditions

DAT	Cycloxydim	cycloxydim-TSO [a]	cycloxydim-T2SO	cycloxydim-TSO2	Unknown (3.6 min)	Others [b] (Sum)	Bound residues	Total
sandy loam (Bruch West)								
0	8.5	86.7	0.1	–	1.6	1.3	1.9	100.1
1	0.1	87.3	4.7	–	2.7	3.2	6.2	104.2
3	–	72.3	2.3	4.7	3.0	3.2	6.4	91.9
7	–	49.7	0.6	7.4	4.4	6.7	7.9	76.7
14	–	35.6	6.8	8.9	4.9	2.6	15.1	73.9
30	–	17.0	4.6	7.4	4.8	1.9	19.7	55.4
60	–	6.3	0.8	2.8	4.4	0.8	22.5	37.6
93	–	2.6	0.3	0.9	5.0	0.6	19.2	28.6
119	–	1.9	0.3	0.3	4.7	1.0	22.0	30.2
loamy sand (Li 35 b)								
0	9.9	87.1	0.1	–	0.9	0.9	1.1	100.0
1	–	90.4	3.8	–	2.8	4.3	4.1	105.4
3	–	79.9	4.1	2.9	2.2	2.3	5.4	96.8
7	–	61.7	3.0	4.1	2.8	2.6	10.7	84.9
14	–	31.4	5.3	9.5	5.3	2.9	12.3	66.7
30	–	17.0	2.5	4.1	5.7	2.3	17.4	49.0
60	–	5.4	1.3	1.5	5.6	1.0	21.8	36.6
93	–	2.6	–	0.3	5.7	1.1	17.7	27.4
119	–	1.7	–	0.2	4.6	1.4	21.1	29.0

[a] Sum of isomers

[b] Each individual peak < 5% TAR at any sampling time

Soil Lufa 2.2 showed a slightly different peak pattern, with 17.9% TAR detected as cycloxydim at day 0 (Table 38). Cycloxydim-TSO with its isomer was the major metabolite in all soils. A further study conducted in soil Bruch West under the same conditions indicated that the polar fraction at 3.6 min consisted of several compounds, each of them significantly below 5% TAR (Hassink, 2008a).

Table 38 Recovery of radioactivity in% TAR (total applied radioactivity) and distribution of metabolites after application of [^{14}C] cycloxydim to a loamy sand (Lufa 2.2) and incubation under aerobic conditions

DAT	Cycloxydim	-TSO [a]	-T1SO	-T2SO	-T1SO2	-T2SO2	-T1S	-TSO2	-T2S	Unknown (10.7 min)	Others [b] (Sum)	Bound residues	Total
0	17.9	57.0	2.4	0.3	–	–	0.7	–	0.3	2.4	8.9	1.4	91.3
1	2.4	56.9	3.9	1.9	–	–	0.2	1.1	–	2.9	9.5	5.4	84.2

DAT	Cycloxydim	-TSO [a]	-T1SO	-T2SO	-T1SO2	-T2SO2	-T1S	-TSO2	-T2S	Unknown (10.7 min)	Others [b] (Sum)	Bound residues	Total
3	1.6	50.5	3.2	2.1	–	–	–	2.4	–	3.1	10.7	8.9	82.5
7	1.6	27.1	4.2	4.4	1.2	3.5	0.6	4.5	–	2.8	10.0	10.8	70.7
14	1.3	25.7	2.7	3.8	0.5	1.4	–	2.4	–	2.4	6.9	14.1	61.2
30	–	8.5	2.3	8.1	2.3	8.2	–	3.6	–	2.2	4.4	14.5	54.1
60	–	3.1	2.0	6.9	2.6	9.2	–	1.0	–	2.5	4.1	15.8	47.2
93	–	2.2	2.1	6.1	2.0	9.9	–	0.4	–	1.8	3.0	13.3	40.8
119	–	1.4	2.2	7.3	2.0	9.4	–	0.7	–	1.8	1.8	14.3	40.9

[a] Sum of isomers

[b] each individual peak < 5% TAR at any sampling time

Due to technical problems with the incubation device, the mineralisation rates in the three soils and accordingly the material balances could not be determined properly. Therefore, a second trial was started with the same soils and under the same incubation conditions but with a slightly modified incubation apparatus (Table 39). The mineralisation rates ranged from 42.3 to 53.7% TAR and the material balance from 84.5% to 100% TAR.

Table 39 Mineralisation rate and material balance after application of [^{14}C] cycloxydim [% TAR] to three different soils

	Sandy loam -Bruch West, TAR = 0.779 mg/kg		Loamy sand Li 35 b, TAR = 0.759 mg/kg		Loamy sand Lufa 2.2, TAR = 0.782 mg/kg	
DAT	TRR	CO_2	TRR	CO_2	TRR	CO_2
0	100.0	n.d.	100.0	n.d.	100.0	n.d.
7	83.6	8.1	76.5	14.0	76.2	15.3
14	68.8	21.1	61.1	30.7	65.2	27.2
30	51.5	36.1	45.3	44.1	49.0	35.5
60	43.4	48.0	39.7	50.2	45.1	40.4
93	35.7	52.4	36.0	52.2	43.6	41.9
119	34.5	53.7	34.0	52.7	46.4	42.3

TAR = total applied radioactivity (ERR + RRR 0 days)

TRR = total radioactive residues

n.d. = Not determined

In all three soils both the E-isomer and the Z-isomer of cycloxydim-TSO were detected (Bayer, 2006a). However, only in one soil (Lufa 2.2) the Z-isomer of cycloxydim-TSO could be separated as pure peak by the HPLC system. It was shown that this isomer represented 3.4 to 6.7% TAR and could only be detected within the first 2 weeks after application.

Bayer (2000a) investigated the rate of degradation of cycloxydim and its metabolites in Bruch West, Li 35 b and Lufa 2.2 soils at 20 °C and 40% MWC in the laboratory in the dark. Soils were treated with 0.8 mg/kg dry, corresponding to an application rate of 0.6 kg/ha. Samples were taken at 0, 1, 3, 7, 14, 30, 60, 93 and 119 days after treatment and extracted with acetonitrile and acetonitrile/water. Extractability decreased from > 98% TAR to < 26.2%TAR at the end of the incubation period.

Half-lives were determined using a compartment model given in Figure 4. Degradation was very fast, with half-lives < 9 hours. Mineralization represented > 42%TAR at the end of the incubation period, and up to 22.5%TAR were bound residues. Half-lives of the main metabolites formed are given in Table 40. The metabolite T2SO2 was only observed in one out of three soils. But no degradation rate could be estimated.

Table 40 Degradation rate of cycloxydim metabolites in soil in the dark

$DegT_{50}$	Bruch West	Li 35 b	Lufa 2.2
TSO [d]	10.6	10.1	9.3
TSO2 [d]	12.6	10.8	8.8
T2SO [d]	19.6 [a]	17.9 [a]	291.7 [b]

[a] Derived by conservative fit of the residues from the day of maximum concentration
[b] Not significant

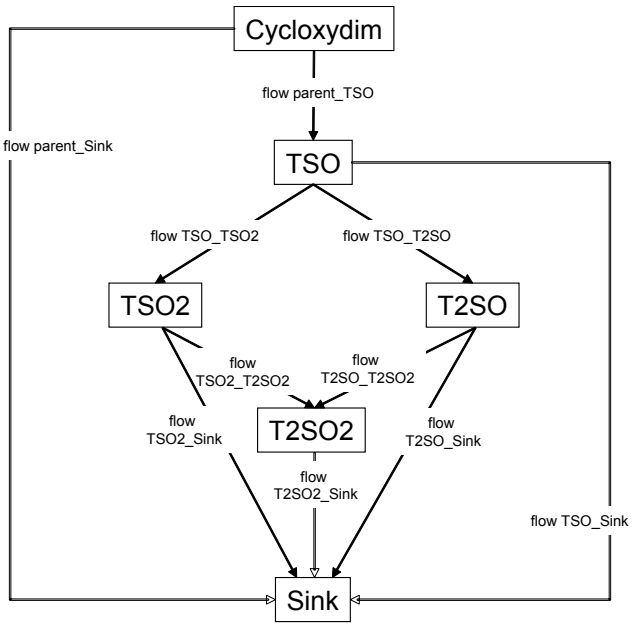


Figure 4 Compartment model considered in the kinetic evaluation

The proposed route of degradation of cycloxydim in aerobic soil considering also the different isomers is shown in Figure 5.

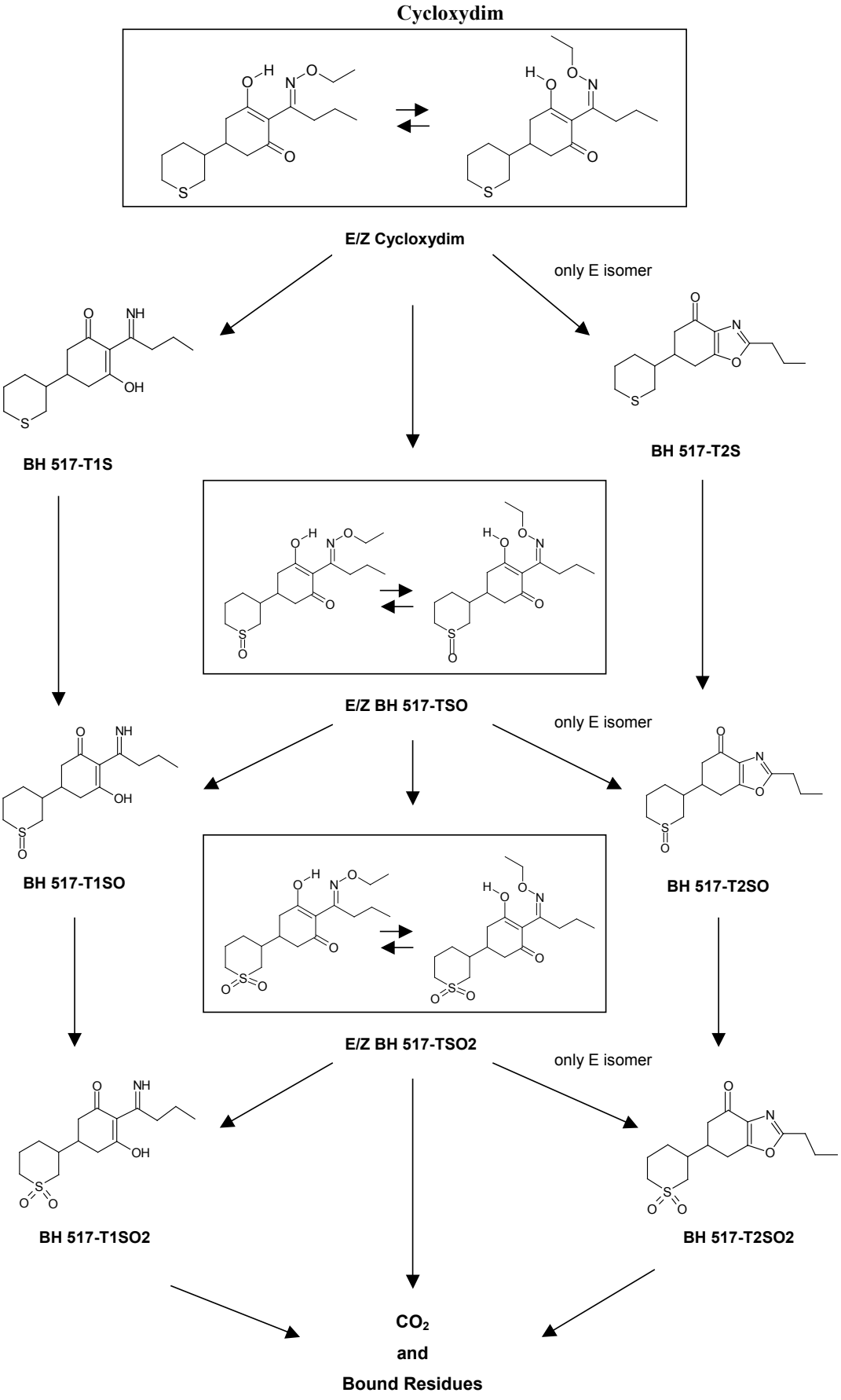


Figure 5 Proposed route of degradation of cycloxydim (BAS 517 H) in aerobic soil

Photolysis on soil surface

The photolytic degradation of cycloxydim on soil surface was studied on a loamy sand soil (Speyer standard soil 2.2) using a Hanau Suntest apparatus equipped with a Xenon burner and a filter system suppressing UV light below 290 nm (Keller, 1985c). The soil was treated with 10 mg [^{14}C] cycloxydim/kg dry, adjusted to 40% MWC and incubated at 30 ± 5 °C for 8 hours. Soil samples were extracted with dichloromethane and water and analysed by radio-TLC.

The formation of bound residues was in the same order of magnitude with and without irradiation (Table 41). Polar degradation products did not accumulate in the course of the study. Radioactive balance was between 86.5 and 108.5% TAR.

Table 41 Distribution of radioactivity in soil photolysis of [^{14}C] cycloxydim [% TAR]

Irradiation time (hours)	Cycloxydim	Peak cluster 1 [a] (-TSO, -T2SO)	Peak cluster 2 [a] (-TSO2, -T2SO2)	cycloxydim-T1S	cycloxydim-T2S	H_2O-extract	Bound residues	Total
0	77.7	17.3	–	–	–	0.8	2.8	98.6
1	33.7	47.4	3.3	–	1.5	6.1	7.0	99.0
2	29.0	44.8	4.5	1.9	5.3	7.4	7.7	100.6
3	4.1	77.4	8.2	–	–	5.4	11.5	106.6
4	1.1	62.0	5.7	–	0.5	5.9	11.3	86.5
6	6.6	63.2	8.1	1.4	1.7	7.5	10.9	99.4
8	2.0	81.4	3.7	–	–	6.5	14.9	108.5
8 (dark control)	15.1	78.8	–	–	–	4.0	8.2	106.1

[a] Peaks in each cluster could not be separated unambiguously on TLC.

The DCM extract of the 8 h irradiated sample was further analysed by HPLC and parent compound, cycloxydim-TSO, cycloxydim-T1SO and cycloxydim-T2SO could be identified. Cycloxydim-TSO and cycloxydim-T2SO contributed each to about 40% TAR. Cycloxydim-TSO and cycloxydim-T2SO were confirmed by MS-analysis.

Residues in rotational crops

Confined Rotational Crop

The residue levels and the nature of the residues in three representative succeeding crops were investigated following application of [^{14}C] cycloxydim at an application rate equivalent to 0.65 kg ai/ha (Veit, 2002b). The treated soil was aged for 30 days (simulating an emergency plant back; 30 DAT, radish and lettuce), 80 days (only wheat), 120 days (simulating a fall plant back; 120 DAT) and 365 days (365 DAT). After 30 and 80 days of treatment, ploughing was simulated by mixing the treated with the untreated soil layers. At the later replant intervals, the soil was mixed after harvest of the ripe crops and before replanting using a spade. For 120 and 365 days, the plants were cultivated using the containers of the 30 DAT replant interval. After mixing, the crops radish, lettuce and wheat were sowed or planted. The aging of the soil and the cultivation of the crops took place in a glass roofed vegetation hall, in phytotrons or in the glass house depending on the climatic conditions outside.

After homogenization, TRR of each sample were determined by combustion analysis. All samples were first extracted three times with methanol followed by an additional water extraction. The extracts were combined and measured by LSC. The identification of the metabolites is based on HPLC. A polar peak eluting with the void volume from the HPLC column was identified/characterized as sugar by yeast digestion. For further characterization of the metabolites, liquid/liquid partitioning of the MeOH extracts was performed using dichloromethane. The residual radioactive residue (RRR) was freeze-dried and subjected to combustion analysis. To release the non-soluble radioactivity, the remaining residual radioactive residues were treated with different solvents

(ammonia, DMSO/water 9:1 and sodium hydroxide) and/or enzymes (cellulase, macerozyme, amylases and/ or amyloglucosidase).

The total radioactive residues reached 0.139 mg/kg in wheat straw (Table 41). After a plant back interval of 30 days, TRR in lettuce and radish samples ranged from 0.032–0.051 mg/kg and decreased to 0.003–0.011 mg/kg after 120 and 365 days. The residue levels in wheat grain ranged from 0.098 mg/kg after a plant back interval of 120 days to 0.039 mg/kg after a plant back interval of 365 days.

The residue levels in the soil decreased from 4.84 mg/kg at 0 DAT to 0.057 mg/kg at 365 DAT (Table 42). After a plant back interval of 30 days and harvest of lettuce and radish, residues in soil decreased significantly compared with those after ploughing, but no difference were observed after harvesting at 365 day plant back interval.

Table 42 Total radioactive residues in crops after treatment with [^{14}C] cycloxydim

Crop Parts (Days after planting, DAP)	TRR in the plant (mg/kg)	TRR in the soil (mg/kg), at 0 day = 4.84	
		After aging/ploughing	After harvesting
Plant back interval: 30 DAT		0.092	
Lettuce Head (67)	0.051		0.054
White Radish Root (86)	0.032		0.046
White Radish Top (86)	0.050		
Plant back interval: 80 DAT		0.136	
Wheat Forage (53)	0.031		0.143
Wheat Straw (118)	0.131		
Wheat Chaff (118)	0.086		
Wheat Grain (118)	0.014		
Plant back interval: 120 DAT		0.083	
Lettuce Head (70)	0.007		0.034
White Radish Root (81)	0.008		0.097
White Radish Top (81)	0.011		
Wheat Forage (57)	0.021		0.023
Wheat Straw (169)	0.139		
Wheat Chaff (169)	0.143		
Wheat Grain (169)	0.098		
Plant back interval: 365 DAT		0.057	
Lettuce Head (59)	0.003		0.061
White Radish Root (82)	0.003		0.046
White Radish Top (82)	0.003		
Wheat Forage (70)	0.008		0.041
Wheat Straw (160)	0.059		
Wheat Chaff (160)	0.044		
Wheat Grain (160)	0.039		

The extractability (ERR) of radioactive residues in rotational crop is shown in Table 43. Most of the residues were recovered in the methanol extracts. Residual radioactive residues (RRR) reached over 85%TRR in wheat grain at plant back interval of 120 or 365 days DAT.

Table 43 Extractability of radioactive residues in rotational crop after [^{14}C] cycloxydim treatment after plant back intervals of 30, 80, 120 and 365 days

Crop Parts (DAP)	TRR (mg/kg)	MeOH		H_2O		ERR [a]		RRR [b]	
		(mg/kg)	(% TRR)	(mg/kg)	(% TRR)	(mg/kg)	(% TRR)	(mg/kg)	(% TRR)
Plant back interval: 30 DAT									
Lettuce Head	0.051	0.032	63.6	0.003	5.0	0.035	68.6	0.014	28.5
Radish Root	0.032	0.020	62.2	0.001	3.2	0.021	65.4	0.011	33.4
Radish Top	0.050	0.025	49.7	0.003	5.7	0.028	55.4	0.015	30.1
Plant back interval: 80 DAT									
Wheat Forage	0.031	0.026	84.3	0.001	2.8	0.027	87.1	0.004	11.5
Wheat Straw	0.131	0.063	48.5	0.030	23.1	0.093	71.6	0.022	16.5

Crop Parts (DAP)	TRR (mg/kg)	MeOH		H_2O		ERR [a]		RRR [b]	
		(mg/kg)	(% TRR)	(mg/kg)	(% TRR)	(mg/kg)	(% TRR)	(mg/kg)	(% TRR)
Wheat Chaff	0.086	0.034	39.3	0.027	31.0	0.061	70.3	0.021	23.9
Wheat Grain	0.014	0.003	20.7	0.003	20.7	0.006	41.4	0.009	62.6
Plant back interval: 120 DAT									
Lettuce Head	0.007	0.003	45.9	< 0.001	4.4	0.004	50.3	0.003	36.9
Radish Root	0.008	0.005	67.0	< 0.001	3.1	0.006	70.1	0.002	26.6
Radish Top	0.011	0.005	48.1	0.001	6.5	0.006	54.6	0.003	31.9
Wheat Forage	0.021	0.010	46.9	0.001	3.3	0.011	50.2	0.008	36.5
Wheat Straw	0.139	0.056	40.5	0.014	10.3	0.070	50.8	0.055	39.9
Wheat Chaff	0.143	0.046	31.9	0.018	12.7	0.064	44.6	0.082	57.5
Wheat Grain	0.098	0.017	17.5	0.007	7.1	0.024	24.6	0.086	88.3
Plant back interval: 365 DAT									
Lettuce Head	0.003	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Radish Root	0.003	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Radish Top	0.003	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Wheat Forage	0.008	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Wheat Straw	0.059	0.015	25.6	0.005	8.8	0.020	34.4	0.023	38.6
Wheat Chaff	0.044	0.010	23.4	0.005	12.2	0.015	35.6	0.029	65.8
Wheat Grain	0.039	0.003	6.4	0.003	7.6	0.006	14.0	0.034	86.3

[a] ERR = extractable radioactive residue

[b] RRR = residual radioactive residue

n.d. = Not determined

Ammonia treatment released 16.4% TRR from wheat grain at 120 DAT and enzyme treatment released from 15 to 24.5% TRR from lettuce and radish at 30 DAT (Table 44).

Table 44 Quantitative distribution of the non-released radioactivity in rotational crops after treatment with [^{14}C] cycloxydim

Crop parts (DAP)	RRR, mg/kg (% TRR)	NH_3 Extract mg/kg (% TRR)	Macerozyme mg/kg (% TRR)	Further treatments, mg/kg (% TRR)	Final Residue
Plant back interval: 30 DAT					
Lettuce Head	0.014 (28.5)	n.p.	0.008 (15.0)	n.p.	0.007 (14.0)
Radish Root	0.011 (33.4)	n.p.	0.008 (24.5)	0.001 (2.6)	0.002 (5.5)
Radish Top	0.015 (30.1)	n.p.	0.010 (19.0)	n.p.	0.006 (11.4)
Plant back interval: 120 DAT					
Wheat Straw	0.055 (39.9)	0.005 (3.4)	0.010 (7.1)	0.022 (15.5)	n.p.
Wheat Chaff	0.082 (57.5)	0.010 (6.9)	n.p.	n.p.	0.070 (48.9)
Wheat Grain	0.086 (88.3)	0.016 (16.4)	0.011 (11.4)	0.036 (36.7)	n.p.

n.p. = Not performed

All the HPLC analysis of extracts of the different matrices showed that parent was not detected anymore and a range of degradation products could be seen depending on the matrix (Tables 45–46). At 30 DAT, cycloxydim-TSO and cycloxydim-TSO2 were the major components of the residues in lettuce head and radish root and top. At 120 DAT, only cycloxydim-TGSO2 could be detected as a single compound (in radish).

Table 45 Summary of major components in lettuce and radish as rotational crops after [^{14}C] cycloxydim treatment and plant back intervals of 30 and 120 days, in mg/kg (% TRR)

	cycloxydim-TSO	cycloxydim-TSO2	cycloxydim-TGSO2	minor components	Polar 'Sugar' Fraction
Plant back interval: 30 DAT					
Lettuce Head	0.008 (16.1)	0.007 (13.1)	nd	3: 0.001–0.004	0.011 (21.9)
Radish Root	0.002 (6.3)	0.001 (2.9)	nd	2: 0.001	0.016 (48.6)
Radish Top	0.006 (12.5)	0.001 (1.9)	nd	4: 0.001–0.003	0.011 (21.0)
Plant back interval: 120 DAT					
Lettuce Head	nd	nd	nd	2: ≤ 0.001	0.002 (25.8)
Radish Root	nd	nd	0.001 (9.8)	3: ≤ 0.001	0.004 (52.7)

	cycloxydim-TSO	cycloxydim-TSO2	cycloxydim-TGSO2	minor components	Polar 'Sugar' Fraction
Radish Top	nd	nd	0.001(10.0)	13: ≤ 0.001	0.001 (7.8)

Cycloxydim-TGSO2 was the major metabolite in wheat forage, but no residues were detected in wheat grain (Table 46).

Table 46 Summary of major components in wheat as rotational crops after [^{14}C] cycloxydim treatment and plant back intervals of 80 to 365 days, in mg/kg (% TRR)

	Cycloxydim-TSO	Cycloxydim-T1SO	Cycloxydim-T2SO	Cycloxydim-TGSO2	Minor components	Unknown	Polar 'Sugar' Fraction
Plant back interval: 80 DAT							
Forage				0.008 (26.5)	1: 0.002	0.016(52.0)	n.d
Straw		0.019 (16.1)	0.026 (20.2)	0.014 (10.6)		0.011 + 0.014	0.006 (5.0)
Chaff			0.005 (5.8)	0.010 (12.1)		0.046 (52.5)	nd
Grain					8: ≤ 0.002		< 0.01 (5.7)
Plant back interval: 120 DAT							
Forage	0.001 (2.4)			0.001 (4.8)	3: ≤ 0.001	0.003 (16.4)	0.003 (13.0)
Straw				0.009 (6.9)	3: 0.005–0.077	0.033 (23.9)	0.010 (7.4)
Chaff					5: 0.002–0.005	0.012 (8.3)	0.033 (23.1)
Grain					6: 0.001–0.003		
Plant back interval: 365 DAT							
Straw				0.003 (5.9)	4: 0.001–0.004		0.009 (15.5)
Chaff					2: 0.001		0.013 (31.6)
Grain					1: 0.001		0.005 (11.5)

The storage stability investigations for lettuce head 30 DAT/ 67 DAP demonstrated that within a storage time of approximately 18.5 month no noticeable changes in the metabolite pattern could be observed. For wheat straw 120 DAT/ 169 DAP, the storage stability investigations demonstrated that within a storage time of 388 days no major changes in the metabolite pattern could be observed. Under the chosen conditions, the radioactive residues were stable in the original matrix.

In general, the metabolic pathway of cycloxydim in rotational plants can be seen in Figure 6. The metabolite cycloxydim-TSO was the oxidation product at the thioether group of the thiopyran ring system. A second oxidation step leads to cycloxydim-TSO2. The metabolites cycloxydim-T2SO and cycloxydim-T1SO were the results of a cleavage of the oxime ether in the side chain and presumably some non-enzymatic Beckmann rearrangements. Subsequent oxidative cleavage of the cyclohexenone ring system leads to glutaric acid derivatives like cycloxydim-1-dicarboxylic acid. The very polar ‘sugar’ fraction, as one of the major components in most of the matrices was identified by yeast fermentation. $^{14}CO_2$ and radiolabelled alcohol were the results of that fermentation process. The glutaric acid derivatives were further metabolized to a small unit, which could be incorporated into the carbohydrate pool of the plants.

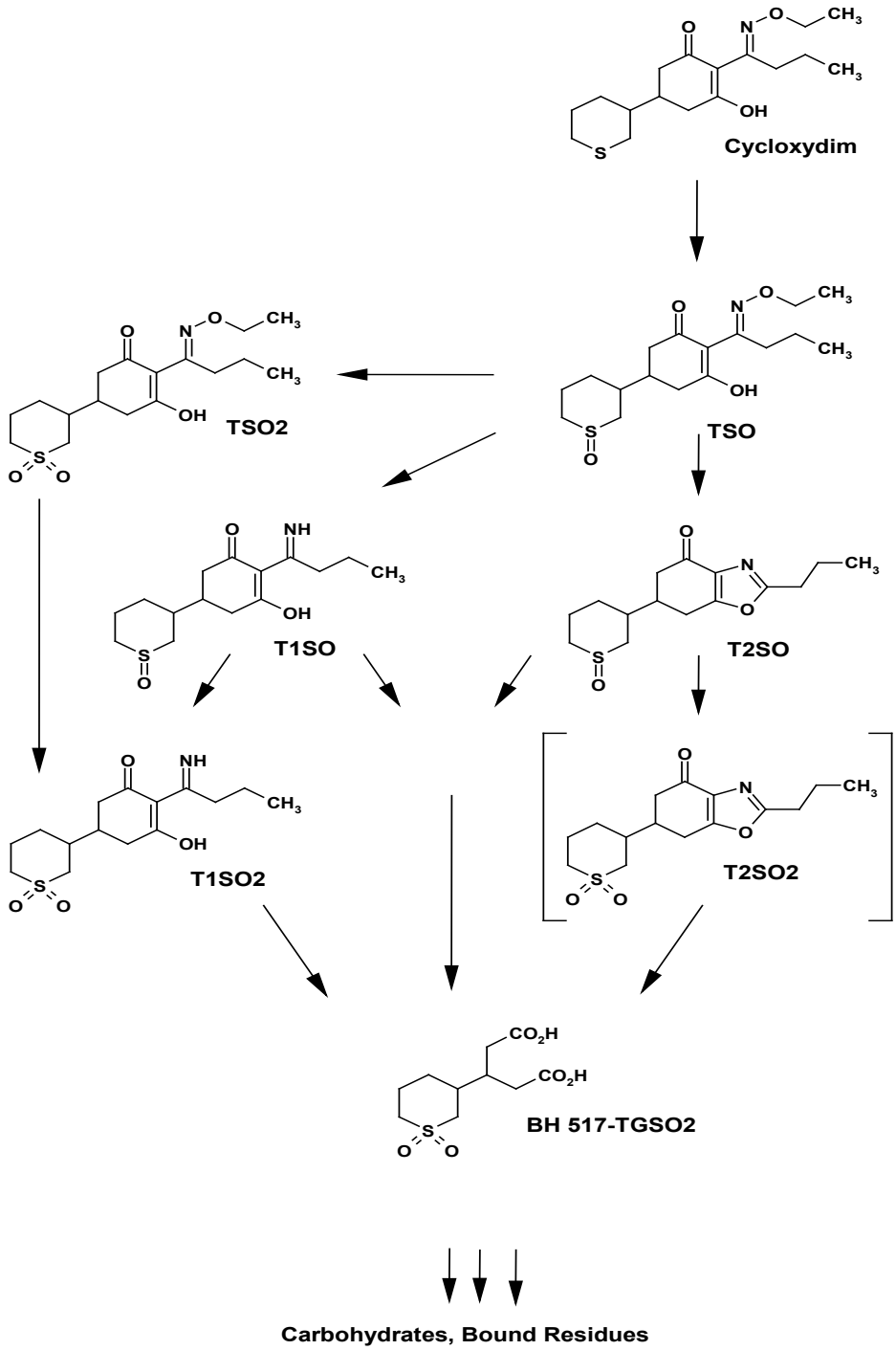


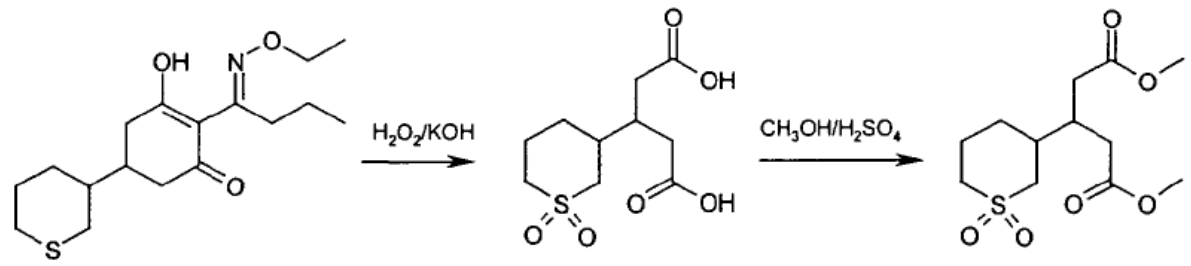
Figure 6 Metabolic pathway of cycloxydim in rotational crops

Residue Analysis

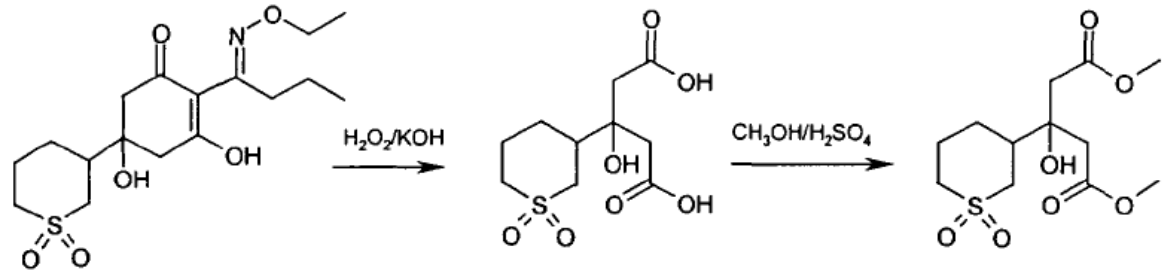
Analytical methods for plant matrices

The residue analytical methods 263 (Beutel, 1987; Beutel, 1988a), 263/1, 263/2 (Herb 1988a) and 263/3 (Tilting, 2002a; Tilting, 2004a) allow the determination of cycloxydim and its metabolite cycloxydim-5-OH-TSO2 in various crop matrices. The residues are extracted with a mixture of isopropanol, water and hydrogen peroxide and oxidized with perhydrol to form the corresponding pentane acids (cycloxydim-TGSO2 and cycloxydim-5-OH-TGSO2; Figure 7). A precipitation step with calcium hydroxide may be introduced before oxidation (Herb, 1988a). After reduction and phase

separation, the acids are isolated from the water phase by activated charcoal adsorption and converted into the dimethyl esters (cycloxydim-TDME and cycloxydim-5-OH-TDME). Subsequently, a clean-up is carried out in a silica gel column and in some cases by an additional HPLC separation. The final determination is made by GC/S-FPD.



Cycloxydim cycloxydim-TGSO2 cycloxydim-TDME



Cycloxydim-5-OH-TSO2 cycloxydim-5-OH-TGSO2 cycloxydim-TDME

Figure 7 Oxidation and methylation steps of cycloxydim and cycloxydim-OH-TSO2 on method 263

Cycloxydim and its metabolite were shown to be stable in standard solution (100 µg/mL in methanol) over a period of 90 days. Good linearity was observed in the range of 10 to 30 ng/mL with a correlation coefficient > 0.99. Interference with the oxidation step leads to low recoveries. Furthermore, the presence of reducing substances and complex formation may require longer reaction times for oxidation. The validation data are shown in Table 47. The method was independently validated by Tillkes (1993a), Schulz (1995a), Schulz (1995b), Bross & Lehmann (2000a) and Grosshans (2009a) in various crops (Table 47).

Table 47 Validation data for analytical method 263 for cycloxydim residues in food of plant origin

		Level	Cycloxydim, %		Cycloxydim-OH-TSO2, %		
Matrix	N	[mg/kg]	Mean recovery	RSD	Mean recovery	RSD	Reference
Sugar beet roots	10	0.05	65.4	13.7	64.7	13.3	Beutel, 1987a
	7	0.5	75.0	21.17	76.7	19.71	
	9	5.0	73.4	7.94	74.2	9.33	
Sugar beet forage	4	0.05	67.7	5.87	54.4	2.05	
Sugar beet young plants	3	0.5	90.7	N/A	83.6	N/A	
	7	5.0	76.1	12.62	76.1	8.43	
Sugar beet leaves	5	0.05	88.1	22.49	66.7	7.16	
	1	0.5	66.1	N/A	66.9	N/A	
	5	5.0	69.5	7.46	72.5	5.85	
Potatoes tubers	8	0.05	83.3	17.73	78.2	14.72	
	4	0.50	79.5	27.70	77.7	21.94	
	4	5.00	85.1	5.80	79.2	2.63	
Potatoes forage	6	0.5	86.3	4.38	87.2	4.23	
	7	5.00	75.7	10.23	76.8	6.88	
Peanuts	8	0.05	86.7	15.34	84.6	17.84	

		Level	Cycloxydim, %		Cycloxydim-OH-TSO2, %		
Matrix	N	[mg/kg]	Mean recovery	RSD	Mean recovery	RSD	Reference
	4	5.00	62.8	11.96	67.5	7.79	
Peanuts forage	5	0.05	95.5	4.84	63.2	19.50	
	4	5.00	67.5	3.54	54.6	7.32	
Rape seed grains	4	0.05	109	7.83	118.9	6.44	
	8	0.5	84.6	8.89	74.5	14.70	
	6	5.00	73.8	7.62	66.4	10.60	
Rape seed forage	2	0.50	82.3	N/A	73.7	N/A	
	1	1.00	76.4	N/A	82.5	N/A	
	5	5.00	67.7	4.70	61.5	3.92	
	2	10.00	82.5	N/A	86.5	N/A	
Rape seed straw	5	0.5	93.2	3.89	86.0	2.65	
	1	5.0	89.7	N/A	82.2	N/A	
Soya beans	4	0.05	101.7	5.97	97.5	5.99	
	1	0.5	101.4	N/A	75.5	N/A	
	4	5.00	83.1	2.34	75.8	3.15	
Soya beans forage	1	0.5	84.7	N/A	71.3	N/A	
	1	5.0	74.3	N/A	70.1	N/A	
Green Beans	4	0.05	80.6	4.87	70.6	8.10	
Green Beans kernels	5	0.05	82.9	8.30	89.8	5.63	
	5	5.00	87.2	3.59	84.9	3.87	
Green Beans forage	2	0.50	72.6	N/A	76.8	N/A	
	1	2.0	65.0	N/A	65.5	N/A	
	2	5.0	67.8	N/A	75.5	N/A	
	1	20.0	82.2-	N/A	63.2	N/A	
Green Beans leaves	5	0.05	56.4	20.48	95.7	7.94	
	5	5.0	76.0	10.86	76.7	16.27	
	2	10.0	73.8	N/A	76.3	N/A	
	2	100.0	71.8	N/A	73.4	N/A	
Peas kernels	4	0.05	74.7	13.61	64.4	21.20	
	4	5.0	78.2	3.82	72.6	3.19	
Peas forage	6	5.0	83.4	9.23	71.0	7.34	
Grass hay	7	0.5	78.2	11.81	80.3	9.37	
	4	5.0	71.1	3.86	66.7	3.44	
Swedish turnips Young plants	4	0.2	64.9	7.02	74.7	13.29	Beutel, 1988a
	4	5.0	84.2	2.52	70.2	2.65	
Swedish turnips leaves	4	0.2	78.3	3.19	65.6	2.64	
	4	5.0	90.2	2.0	78.0	1.96	
Brussels sprouts leaves	4	0.25	97.7	18.37	59.1	14.85	
	4	5.0	97.5	11.94	74.0	10.69	
Brussels sprouts	4	0.25	73.4	9.19	62.6	9.08	
	4	0.5	76.7	10.81	69.5	16.86	
	4	5.0	78.5	3.3	71.3	5.0	
Cotton seed	4	0.05	78.6	6.03	89.8	6.87	
	4	5.0	81.9	1.27	73.3	2.34	
Cotton leaves	4	0.5	59.7	19.55	90.5	9.92	
	4	5.0	82.2	5.36	78.0	5.12	
Carrots	4	0.05	60.7	14.42	73.0	13.22	
	4	5.0	83.4	3.88	75.2	2.89	
Rape seed oil	4	0.05	82.6	3.38	69.4	9.23	
	4	5.0	74.0	5.23	62.0	4.15	
Rape seed meal	4	0.5	82.2	10.26	104.9	9.42	
	4	5.0	89.3	1.24	83.0	2.39	
Lettuce leaves	4	0.05	74.1	8.89	73.3	7.18	
	4	5.0	84.9	7.75	76.0	7.33	
Celeriac roots	4	0.05	87.3	7.54	77.5	9.27	
	4	5.0	78.1	3.97	61.3	3.95	
Pineapple fruit	4	0.05	95.1	3.0	78.9	1.5	Herb, 1988a
	4	5.0	82.9	2.4	66.2	2.1	
Apple fruit	4	0.05	68.2	14.0	69.2	14.2	
	4	5.0	77.1	5.4	67.4	4.5	

		Level	Cycloxydim, %		Cycloxydim-OH-TSO2, %		
Matrix	N	[mg/kg]	Mean recovery	RSD	Mean recovery	RSD	Reference
Pear fruit	4	0.05	71.9	1.7	66.2	2.7	
	4	5.0	71.4	1.7	61.9	3.4	
Cauliflower head	4	0.05	61.7	3.9	74.2	5.1	
	4	5.0	86.6	1.8	67.1	1.6	
Cauliflower plant	4	0.05	88.3	7.9	86.6	9.0	
	4	5.0	86.1	1.0	73.4	4.1	
Beans seeds	4	0.1	82.9	9.6	75.1	5.6	
	4	5.0	83.2	2.6	69.8	2.4	
Citrus fruit	4	0.05	86.3	9.1	68.9	9.3	
	4	5.0	86.6	2.9	71.7	4.0	
Flax seeds	4	0.05	62.3	3.9	70.1	2.9	
	4	5.0	58.5	2.4	65.9	5.0	
Grass hay	4	0.5	90.9	3.9	79.2	5.9	
	4	5.0	76.2	5.2	66.5	4.9	
Potato tuber	4	0.05	71.8	3.4	65.4	3.7	
	4	5.0	83.8	3.3	65.8	5.1	
Pumpkin fruit	4	0.05	68.1	7.2	60.8	7.1	
	4	5.0	72.3	3.1	59.4	2.7	
Leek plant	4	0.05	86.4	3.3	111.5	4.1	
	4	5.0	79.1	3.1	70.8	4.1	
Lucerne green mass	4	0.05	90.7	5.6	80.3	7.6	
	4	5.0	68.9	4.5	61.9	4.7	
Papaya fruit	4	0.05	86.3	3.8	72.9	1.6	
	4	5.0	93.6	4.1	67.7	3.6	
Vine	4	0.05	81.6	2.5	71.0	5.5	
	4	5.0	73.2	7.2	61.4	7.6	
Garlic	4	0.5	76.7	4.0	79.9	5.3	Tilting, 2002a
	4	5.0	81.9	5.5	71.2	6.8	
Eggplant	4	0.05	89.8	2.8	86.9	2.3	
	4	5.0	91.3	4.9	89.8	4.7	
Rice grain	4	0.05	62.8	14.1	56.2	4.3	
	4	5.0	89.7	4.1	66.8	7.4	
Red beet	4	0.5	74.1	8.7	62.9	6.6	
	4	5.0	71.3	6.8	66.6	6.8	
Peas, seeds and pods	4	0.05	58.2	3.0	62.9	16.3	
	4	5.0	82.5	4.9	68.2	4.8	
Melon	5	0.05	58.6	6.5	71.4	5.7	
	5	5.0	73.1	6.6	63.5	5.4	
Peppers	4	0.05	80.7	2.3	71.2	3.6	
	4	5.0	80.9	4.8	68.0	6.1	
Savoy cabbage	4	0.05	86.2	10.5	81.9	13.5	
	4	5.0	84.1	5.9	72.7	5.0	
Celeriac (green plant)	4	0.05	87.7	2.6	84.4	3.1	
	4	5.0	78.8	7.3	74.9	5.7	
Chick pea	4	0.05	64.8	2.4	70.1	3.7	
	4	5.0	69.8	3.9	61.5	4.5	
Spinach	4	0.5	71.6	4.1	62.0	4.4	
	4	5.0	69.6	3.0	62.5	5.0	
Currant	4	0.5	88.0	5.0	63.6	6.2	
	4	5.0	77.7	2.8	66.4	3.6	
Raspberry	4	0.05	72.8	10.9	68.7	11.9	
	4	5.0	75.2	2.9	63.5	2.5	
Rhubarb	4	0.05	67.7	5.5	54.4	5.1	
	4	5.0	70.7	3.5	56.1	2.7	
Horseradish	4	0.5	69.2	13.3	66.4	12.9	
	4	5.0	69.4	7.5	63.0	6.8	
Strawberry (green plant)	4	0.5	83.0	4.5	61.8	4.2	
	5	5.0	74.3	4.4	56.8	8.7	
Carrot (green plant)	4	0.1	85.4	6.8	72.6	6.6	
Maize whole plant	5	0.05	84	6.2	70	20	Beck, 1997a

		Level	Cycloxydim, %		Cycloxydim-OH-TSO2, %		
Matrix	N	[mg/kg]	Mean recovery	RSD	Mean recovery	RSD	Reference
and rest plant without root	2	5.0	87	N/A	63	N/A	
	2	20	91	N/A	75	N/A	
Maize cobs with husk	2	0.05	87	N/A	78	N/A	
	2	5.0	63	N/A	65	N/A	
Maize grain	1	0.05	90	N/A	85	N/A	
	1	5.0	89	N/A	73	N/A	
Maize straw	1	0.05	99	N/A	88	N/A	
	1	5.0	85	N/A	72	N/A	
Maize whole plant	1	0.05	103	N/A	75	N/A	Schulz, 1997a
	1	5.0	91	N/A	87	N/A	
Maize grain	1	0.05	92	N/A	76	N/A	
	1	5.0	77	N/A	63	N/A	
Maize whole plant and rest plant without root	5	0.05	90	8.2	79	6.8	Beck & Schulz, 1997b
	3	5.0	73	8.2	59	8.3	
	1	20	74	N/A	59	N/A	
Maize cobs with husk	2	0.05	90	N/A	79	N/A	
	1	5.0	95	N/A	75	N/A	
Maize grain	1	0.05	98	N/A	86	N/A	
	1	5.0	75	N/A	54	N/A	
Maize straw	1	0.05	127	N/A	109	N/A	
	1	0.1	94	N/A	80	N/A	
	1	5.0	105	N/A	85	N/A	
Maize whole plant	1	0.05	93	N/A	82	N/A	Schulz 1997c
	1	15	85	N/A	71	N/A	
Maize grain	1	0.05	97	N/A	83	N/A	
	1	5.0	70	N/A	60	N/A	

Extractability/accountability investigations were performed to determine the % TRR measured in soya bean and canola matrices by method 263 in plants treated with cycloxydim (Beutel, 1987c; Beutel, 1987b). The radioactivity present in all extracts (including waste fractions) was determined by LSC and residual radioactive quantified by combustion analysis. The results are summarized in Table 48. Extraction with iso-propanol/water released from 66 to 94%TRR. Final eluate containing cycloxydim and cycloxydim-TDME gave similar results either when measured by LSC or GC-FPD.

Table 48 Extractability and accountability of method 263 in soya bean and canola matrices

	Soya bean, mg/kg (%TRR)			Canola, mg/kg (%TRR)	
Plant Matrix	Seeds	Forage	Straw)	Seed	Forage
TRR	2.27 (100)	3.75 (100)	0.89 (100)	3.96 (100)	1.76 (100)
Isopropanol/water extract	2.09 (92.1)	3.22 (85.9)	0.59 (66.3)	2.64 (66.7)	1.65 (93.7)
Finale eluate, by LSC	1.09 (48.0)	1.78 (47.5)	0.29 (32.6)	1.69 (42.7)	1.08 (61.4)
Finale eluate, by GC	1.07 (47.1)	1.44 (38.4)	0.35 (39.9	1.66 (41.9)	119 (67.8)

Method No. 407/0 and 407/1 is a modification of Method No. 263 for onion that allows the determination of cycloxydim, cycloxydim-5-OH-TSO2 and all metabolites that can be oxidized to cycloxydim-TGSO2 (cycloxydim-1) or cycloxydim-5-OH-TGSO2 (cycloxydim-2) with H_2O_2 under alkaline conditions in plant matrices (Sasturain, 1997b; Lehmann and Mackenroth, 2003b; Jones, 2001b). The residues are extracted from the matrices using an isopropanol/distilled water mixture, the fruit acids removed by precipitation using $Ca(OH)_2$. Afterwards the active substance and all relevant metabolites are oxidized using hydrogen peroxide/KOH to yield the two di-acids cycloxydim-TGSO2 and cycloxydim-5-OH-TGSO2, the excess of oxidizing agent is eliminated using a C_{18}-column and after a NH_2-column clean-up, residues are determined by HPLC-MS/MS. The validation data are shown in Tables 49–51. In most of the studies, the validation was performed by fortifying the samples with cycloxydim and cycloxydim-OH-SO2 as representative analytes for the non-hydroxylated and hydroxylated compounds, respectively. In one study (Table 50, Richter, 2011a), validation was performed using other metabolites that could be oxidized to the cycloxydim-1 and cycloxydim-2.

Table 49 Validation data for method 407/0 for the determination of cycloxydim residues (Sasturain, 1997b; Lehmann & Mackenroth, 2003b). N = 5 in each case

	Level	Cycloxydim, %		Cycloxydim-OH-TSO2, %	
Matrix	[mg/kg]	Mean recovery	RSD	Mean recovery	RSD
Onion bulbs	0.05	88.8	11.3	83.0	12.4
	5.0	94.8	6.3	80.0	10.2
Onion stems	0.05	86.8	7.1	68.5	3.0
	5.0	93.2	6.5	78.3	5.7
Maize seed	0.05	100.2	9.1	90.0	4.6
	0.5	103.7	5.0	88.3	4.8
Lettuce	0.05	102.5	4.9	93.3	4.6
	0.5	98.7	3.0	88.7	6.9
Oilseed rape seed	0.05	93.8	5.7	76.2	4.3
	2.5	98.7	2.1	87.5	5.3
Oilseed rape forage	0.05	81.3	5.9	97.0	5.1
	5.0	96.5	5.0	95.3	9.2
Bean	0.05	87.1	9.3	102.6	3.4
	1.0	102.3	3.3	91.7	2.9
Orange	0.05	94.7	9.6	91.6	8.8
	0.5	101.2	5.6	96.6	8.1
Potato tuber	0.05	92.0	10.5	95.8	3.9
	1.0	101.2	5.1	95.3	3.2
Sugar beet leaves	0.05	78.8	9.8	77.2	4.6
	5.0	92.9	3.4	82.3	6.3
Carrot	0.05	90.0	10.4	90.7	7.3
	0.5	93.6	7.3	83.8	7.3
White cabbage	0.05	97.0	9.9	93.5	7.0
	1.0	96.3	5.3	96.2	5.2
Onion	0.05	82.5	6.4	76.1	3.4
	0.5	101.7	3.0	81.5	5.8

Table 50 Recovery data (%) for method 407/1 (L0018/1) for the determination of cycloxydim residues. N = 5 in all case. In parenthesis, the m/z transition in the LC-MS/MS (Richter, 2011a).

	Level,	Lettuce		Rape seed		Orange		Potato		Onion	
Substance	mg/kg	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
cycloxydim-TSO (263/155) [a]	0.05 0.50	104 95.5	1.2 3.3	109 105	4.0 4.0	97.3 93.5	6.5 5.0	103 101	2.3 3.3	101 102	5.6 2.0
cycloxydim-TSO (263/219)	0.05 0.50	107 96.1	4.4 3.1	109 110	3.0 7.4	92.1 96.0	3.6 4.6	100 107	3.3 2.6	97.4 104	2.1 3.0
cycloxydim-T1SO (263/155) [a]	0.05 0.50	86.9 76.2	2.7 4.0	96.2 94.5	5.2 3.2	91.1 75.3	4.7 6.6	89.1 85.0	7.3 4.4	90.7 88.3	4.5 3.4
cycloxydim-T1SO (263/219)	0.05 0.50	89.2 75.3	3.0 6.2	96.0 93.9	3.6 1.7	92.2 71.4	3.3 3.5	94.4 91.9	4.8 7.2	95.0 88.5	3.7 4.0
cycloxydim -T2SO (263/155) [a]	0.05 0.50	84.4 82.5	3.6 6.3			79.9 75.6	7.9 4.8				
cycloxydim -T2SO (263/219)	0.05 0.50	88.3 82.4	1.7 3.0			85.2 82.9	2.2 3.6				
cycloxydim -5-OH-TSO (279/175) [a]	0.05 0.50	95.4 89.1	2.4 2.0	100 94.1	3.9 2.8	84.0 84.4	2.9 3.8	95.7 93.7	3.7 2.9	88.3 93.0	3.0 2.9
cycloxydim-5-OH-TSO (279/217)	0.05 0.50	98.1 87.9	4.6 5.2	94.0 92.6	6.9 11.0	83.4 91.5	2.5 7.7	93.6 91.6	3.4 6.0	90.3 97.5	5.4 6.3

[a] Used for quantification

Table 51 Recovery data (%) for method 407/1(SOP-PA.0271) for the determination of cycloxydim residues. N = 5 in all case. In parenthesis, the m/z transition in the LC-MS/MS (Jones, 2006b).

		cycloxydim (263/155)		cycloxydim (263/219)		cycloxydim-5-OH-TSO2 (279/217)		cycloxydim-5-OH-TSO2 (279/175)	
Crop	Level, mg/kg	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD

		cycloxydim (263/155)		cycloxydim (263/219)		cycloxydim-5-OH-TSO2 (279/217)		cycloxydim-5-OH-TSO2 (279/175)	
Crop	Level, mg/kg	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
Tomato	0.05	96	3	87	9	98	4	94	4
	5	92	14	94	12	78	8	76	11
	25	100	3	100	2	81	5	82	3
Pepper	0.05	88	6	82	7	103	9	100	11
	5	87	4	88	6	78	3	77	3
	25	101	5	100	3	83	5	81	4
Beans	0.05	88	8			103	5	97	6
	5	108	11			81	9	81	8
	50	104	8			81	9	80	8
Grape	0.05	102	4	94	2	76	3	73	4
	25	102	7	102	7	75	6	76	6
Sunflower	0.05	93	4	93	5	72	2	76	3
	25	88	8	91	7	84	15	87	17
Green Peas	0.05	81	8	80	4	76	12	74	6
	25	88	2	85	4	71	2	71	3
Soya bean	0.05	90	5			89	9	92	11
	5	92	3			72	2	73	2
	25	102	5			77	4	81	5
Kale (Leaf)	0.05	83	13			77	3	84	8
	25	93	9			75	5	78	11
Onion	0.05	92	2			73	4	71	3
	5	99	12			80	17	81	15
Carrot	0.05	91	5	100	3	78	3	81	4
	25	102	6	103	5	81	4	81	4
Strawberry	0.05	101	6	102	5	89	3	91	5
	25	102	9	99	8	83	8	84	8
Lettuce (Head)	0.05	81	11	76	10	76	5	76	8
	25	101	4	98	4	76	5	75	4
Spinach	0.05	73	5			74	6	76	13
	25	93	13			87	17	86	15
	80	90	6			78	4	75	7

The ethyl acetate phases of maize grain and straw from the 0.8 kg ai/ha treatment were analysed according to the LC/MS/MS method 407/0 with external calibration. The LSC data/radio-HPLC chromatograms were compared with the data following the common moiety method. Approximately 75% recovery (LSC) was found in grain after applying the residue analytical method (Hofmann, 1997a,b; Bross, 1998a).

Method No. 493/0 was developed to selectively determine cycloxydim-TSO in plant matrices (Bross & Mackenroth, 2003b). Cycloxydim-TSO is extracted from vegetables and fruit with a methanol/water/buffer mixture. After addition of saturated NaCl-solution, a partition with dichloromethane is performed. The aqueous phase is acidified with concentrated formic acid, partitioned with isooctane/dichloromethane mixture and the extract washed with saturated NaCl-solution (pH 3 + 0.1% formic acid). A re-extraction is done with saturated NaCl-solution containing 1% of ammonium hydroxide, the aqueous phase purified by C_{18}SPE and residues determined by HPLC/UV. Validation data is shown in Table 52. The method was independently validated by Schulz (2003b).

Table 52 Validation data method 493/0 for cycloxydim-TSO residues in food of plant origin, N =5 in all cases (Bross & Mackenroth, 2003b)

Matrix	Level [mg/kg]	Means recovery [%]	RSD [%]
Maize seed	0.05	79.5	2.9
	0.5	78.5	1.8
Lettuce	0.05	88.1	1.3
	1.0	80.7	2.3
Rape seed	0.05	74.1	3.9
	5.0	73.4	3.6

Matrix	Level [mg/kg]	Means recovery [%]	RSD [%]
Rape seed forage	0.05	81.9	1.4
	5.0	82.4	1.6
Bean	0.05	84.6	3.9
	2.0	82.5	4.3
Orange	0.05	83.0	1.2
	0.5	77.0	2.8
Potato tuber	0.05	88.9	2.9
	2.0	81.8	1.1
Sugar beet leaves	0.05	91.9	4.7
	5.0	81.2	1.8
Carrot	0.05	85.5	3.3
	0.5	82.2	2.3
White cabbage	0.05	90.7	2.3
	2.0	85.5	1.7
Onion	0.05	83.1	2.6
	1.0	78.1	1.3

Analytical methods for animal matrices

Method No. 982/0 determines cycloxydim, cycloxydim-5-OH-TSO2 and all metabolites that can be oxidized with H_2O_2 under alkaline conditions and methylated to cycloxydim-TDME and cycloxydim-5-OH-TDME in matrices of animal origin (Kampke-Thiel, 1998b). The residues are extracted with isopropanol/water, oxidized with hydrogen peroxide/KOH to yield a substituted glutaric acid. After removing the isopropanol, the analyte is adsorbed on charcoal and methylated by refluxing with methanol/sulphuric acid. Partitioning and silica gel column chromatography are used for clean-up. Quantitation is achieved by GC-MS. Minor modifications to the validation method were made in the independent validation. Validation data are presented in Table 53.

Table 53 Validation data for Method 982/0 for the determination of cycloxydim residues in animal matrices

	Level [mg/kg]	Cycloxydim, %		Cycloxydim-OH-TSO2, %	
Matrix		Mean recovery	RSD	Mean recovery	RSD
Validation Report, N = 6					
Eggs	0.05	87.0	13.9	75.6	12.0
	0.5	80.7	9.0	72.7	12.5
Milk	0.01	105.5	3.3	85.5	8.6
	0.1	99.3	8.9	84.5	4.4
Meat	0.05	74.8	8.7	71.6	7.7
	0.5	78.4	12.9	67.2	13.5
Fat	0.05	81.2	7.5	69.4	17.6
	0.5	81.0	14.7	74.4	20.0
Kidney	0.05	72.9	12.3	76.1	17.4
	0.5	75.2	9.0	77.0	14.8
Liver	0.05	74.7	15.8	71.4	16.1
	0.5	81.6	10.4	83.6	10.1
Independent validation, N = 5					
Eggs	0.05	82.3	5.5	73.6	7.1
	0.5	86.2	10.9	58.0	16.8
Milk	0.01	61.6	11.1	66.5	15.9
	0.1	100.6	9.8	73.1	12.1
Meat	0.05	86.8	4.8	58.5	7.1
	0.5	88.9	5.7	69.8	5.6
Fat	0.05	75.7	6.2	56.0	5.0
	0.5	83.6	4.2	65.2	5.3.
Kidney	0.05	84.2	11.0	62.4	16.7
	0.5	71.8	12.8	58.1	15.7
Liver	0.05	71.7	10.6	59.4	11.6
	0.5	76.0	8.9	67.2	3.7

Method No. 513/0 allows the determination of cycloxydim-TSO, cycloxydim-5-OH-TSO and cycloxydim-5-OH-TS in foodstuffs of animal origin (Grosshans, 2003b). The sample is extracted with acetonitrile/hexane (4:3), the acetonitrile extract acidified with formic acid and a liquid/liquid partition with dichloromethane is carried out. The final determination is performed by HPLC/MSD or LC-MS/MS (independent validation; Class, 2002b). Validation data are presented in Table 54.

Table 54 Validation data for analytical method 513/0 for the determination of cycloxydim residues in animal matrices (Grosshans, 2003b)

		Level	Cycloxydim-TSO, %		Cycloxydim-5-OH-TS cycloxydim-5-OH-TSO, % [a]	
Matrix	N	[mg/kg]	Mean recovery	RSD	Mean recovery	RSD
Eggs	5	0.01	78.5	3.4	70.9	3.1
	5	0.1	93.6	2.8	80.7	4.5
Milk	5	0.003	82.8	5.0	66.5	6.7
	5	0.03	96.5	3.7	89.0	5.7
Meat	5	0.01	87.2	4.5	76.0	3.9
	5	0.1	78.7	8.3	74.8	12.7
Fat	5	0.01	75.4	10.7	61.7	3.0
	5	0.1	93.9	6.2	77.2	11.8
Kidney	5	0.01	103.3	3.6	93.0	4.0
	4	0.1	82.5	8.2	82.6	8.1
Liver	5	0.01	95.3	5.8	89.8	7.0
	5	0.1	84.5	3.6	90.1	9.7
Eggs	5	0.01	91	15	78	8
	5	0.1	78	4	67	5
Milk	5	0.003	101	13	78	7
	5	0.03	91	5	78	6
Meat	5	0.01	105	3	85	8
	5	0.1	93	3	80	4

[a] Sum OH-metabolites

The efficiency of the acetonitrile/hexane (4:3) extraction of cycloxydim and its metabolites used in Method No. 513/0 was investigated within hen metabolism studies (Fabian & Knoell, 2003a, Seiferlein, 2003a). The samples were extracted, the radioactivity determined by LSC counting and analysed by HPLC. The extractabilities achieved with the residue method are compared with the results of the metabolism studies, except for liver, where acetonitrile/hexane only extracts about 50% of the radioactivity (Tables 55 and 56).

Table 55 Extractability of residues in the analytical method vs. hen metabolism study (Fabian & Knoell, 2003a).

Matrix	TRR mg/kg	Methanol (Metabolism Study)[a], mg/kg (% TRR)		ACN/Hexane (Residue Method), mg/kg (% TRR)	
		Total extract	cycloxydim-TSO [b]	Total extract	cycloxydim-TSO [b]
Eggs	0.121	0.056 (46.3)	0.037 (31.0)	0.051 (41.8)	0.031 (25.6)
Muscle	0.053	0.006 (10.8)	0.002 (3.5)	0.004 (8.0)	0.003 (5.4)
Liver	0.281	0.087 (31.1)	0.015 (4.8)	0.041 (14.7)	0.16 .7)

[a] For eggs, extraction was performed with methanol + isohexane and for liver and muscle the extract was partitioned with isohexane, so the methanol phase was analysed

[b] Sum of isomers

Table 56 Extractability of egg and liver with the residue analytical method vs. hen metabolism study (Seiferlein, 2003a)

		Methanol (Metabolism Study) mg/kg (% TRR)			ACN/Hexane (Residue Method) mg/kg (% TRR)		
Matrix	TRR, mg/kg	Total extract	cycloxydim-5-OH-TSO	cycloxydim-5-OH-TS	Total extract	cycloxydim-5-OH-TSO	cycloxydim-5-OH-TS

		Methanol (Metabolism Study) mg/kg (% TRR)			ACN/Hexane (Residue Method) mg/kg (% TRR)		
Matrix	TRR, mg/kg	Total extract	cycloxydim-5-OH-TSO	cycloxydim-5-OH-TS	Total extract	cycloxydim-5-OH-TSO	cycloxydim-5-OH-TS
Egg	0.066	0.059 (88.8)	0.010 (14.7)	0.034 (50.7)	0.050 (75.2)	0.009 (13.2)	0.032 (48.5)
Liver	0.110	0.071 (64.6)	0.027 (24.4)	0.021 (19.4)	0.048 (43.7)	0.013 (11.4)	0.025 (22.5)

The efficiency of the acetonitrile/hexane (4:3) extraction of cycloxydim and its metabolites used in Method No. 513/0 was also investigated within goat metabolism studies. Samples were extracted and the radioactivity measured by LSC and HPLC. Milk ACN extract was subjected to a partitioning step followed acetone precipitation before HPLC (Leibold and Hoffman, 2001a). Overall, the extractabilities using the residue analytical method showed that the relevant metabolite cycloxydim-TSO was efficiently detected (Table 57). Tilting (2003b) demonstrated that although a higher amount of the total radioactivity could be extracted by methanol (78% vs. 61% in liver and 98% vs. 76% in kidney), acetonitrile was similarly efficient in extracting the target metabolites cycloxydim-5-OH-TSO and cycloxydim-5-OH-TSO by Method No. 513 (Table 58).

Table 57 Extractability and cycloxydim-TSO of milk and liver in the goat metabolism study versus the residue analytical method (Leibold and Hoffman, 2001a)

				Residue Analysis, mg/kg (% TRR)			
	Metabolism Study, mg/kg (% TRR)			Total	Acetone (milk) or acetonitrile (liver)		Hexane
Matrix	Total	Methanol extract	cycloxydim-TSO		Total	cycloxydim-TSO	Total
Milk	0.023 (100)	0.022 (96.5)	< 0.004 (14.8)	0.23 (100)	0.018 (81.0)	< 0.005 (14.6)	0.001 (3.7)
Liver	0.076 (100)	0.023 (30.5)	0.006 (8.1)	0.076 (100)	0.016 (21.2)	< 0.010 (13.3)	0.004 (5.5)

Table 58 Comparison of the quantities of cycloxydim-5-OH-TSO and cycloxydim-5-OH-TS determined in the goat metabolism study vs. residue analytical method

	Metabolism Study, mg/kg (% TRR)			Residue Method, mg/kg (% TRR)		
Matrix	Methanol extract	cycloxydim-5-OH-TSO	cycloxydim-5-OH-TS	ACN extract	cycloxydim-5-OH-TSO	cycloxydim-5-OH-TS
Liver	0.159 (78.4)	0.022 (10.62)	0.035 (17.44)	0.124 (61.0)	0.038 (18.46)	0.029 (14.35)
Kidney	0.253 (97.7)	0.098 (37.80)	0.065 (25.28)	0.198 (76.4)	0.079 (30.28)	0.052 (20.25)

Method L0105/01 follows the common moiety concept which determines cycloxydim and its metabolites as cycloxydim-TGSO2 and/or cycloxydim-5-OH-TGSO2, calculated in total as cycloxydim. Cycloxydim and cycloxydim-5-OH-TSO2 were chosen as representative analytes for fortification. The residues of cycloxydim and its metabolites were extracted from animal matrices using an isopropanol/distilled water (2 + 1), the acids removed precipitation using $Ca(OH)_2$ and residues oxidized using hydrogen peroxide/KOH to the common moieties cycloxydim-TGSO2 and cycloxydim-5-OH-TGSO2. The analytes were cleaned by SAX SPE and quantified by HPLC-MS/MS. Validation data are shown in Tables 59–60. The method was independently validated by Zhang (2009b).

Table 59 Validation data for analytical method L0105/01 for the determination of cycloxydim residues in food of animal origin. In parenthesis, the m/z transition in the LC-MS/MS; N = 5 (Grosshans & Mackenroth, 2009b)

	Level, mg/kg	cycloxydim (263/219)		cycloxydim (263/155)		cycloxydim-5-OH-TSO2 (279/217)		cycloxydim-5-OH-TSO2 (279/175)	
Crop		Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
Muscle	0.01	84	6.4	100	7.3	100	10	97	4.6
	0.1	95	3.8	93	6.1	97	14	97	8.5
Fat	0.01	84	4.7	85	3.6	89	7.9	89	3.2

Crop	Level, mg/kg	cycloxydim (263/219)		cycloxydim (263/155)		cycloxydim-5-OH-TSO2 (279/217)		cycloxydim-5-OH-TSO2 (279/175)	
		Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
	0.1	99	2.1	93	5.0	105	2.2	94	5.5
Kidney	0.01	95	18	101	8.3	101	9.9	86	6.0
	0.1	101	4.9	106	5.4	96	5.1	99	6.4
Liver	0.01	96	2.1	89	6.0	103	1.3	94	6.4
	0.1	98	0.8	97	4.7	96	2.3	96	5.8
Milk	0.01	92	7.5	91	4.8	92	8.9	95	3.5
	0.1	85	3.3	87	3.1	94	3.9	96	3.2
cream	0.01	90	9.3	85	8.4	91	19	85	11
	0.1	96	6.2	95	6.3	98	5.6	92	8.6
Skim milk	0.01	95	8.9	92	5.1	102	14	92	7.1
	0.1	90	4.9	93	4.4	95	5.4	91	7.0
Egg	0.01	77	5.1	77	6.0	94	5.1	92	5.8
	0.1	93	4.2	93	4.3	107	4.4	107	1.9

Table 60 Validation data for analytical method L0105/01 for the determination of cycloxydim residues in food of animal origin. In parenthesis, the m/z transition in the LC-MS/MS; N=5 (Richter, 2011b)

Substance	Level, mg/kg	Muscle		Liver		Fat		milk		Egg	
		Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD	Mean	RSD
cycloxydim-TSO (263/155) [a]	0.01	73.7	5.6	91.8	6.3	121	6.3	125	6.6	114	2.3
	0.10	98.5	3.4	107	6.3	117	4.8	111	2.5	105	4.0
cycloxydim-TSO (263/219)	0.01	80.6	3.7	98.4	4.2	106	5.8	118	4.3	105	4.2
	0.10	97.5	1.9	99.2	7.0	124	2.6	105	3.5	107	1.2
cycloxydim-T1SO (263/155) [a]	0.01	78.5	6.1	123	13	93.1	2.5	102	2.2	113	0.9
	0.10	95.9	4.8	96.5	4.3	104	2.7	97.2	2.6	102	3.1
cycloxydim-T1SO (263/219)	0.01	89.3	8.4	116	3.1	102	2.9	113	1.8	112	3.8
	0.10	95.1	2.7	99.6	2.0	110	2.6	95.9	5.4	105	4.8
cycloxydim-TSO2 (263/155 [a]	0.01	88	9.0	105	3.9	89.5	6.8	99.1	4.0	112	3.9
	0.10	106	6.1	105	4.7	103	6.6	98.6	3.3	108	4.7
cycloxydim-TSO2 (263/219)	0.01	92.4	6.4	118	3.2	84.0	6.7	103	5.0	121	2.7
	0.10	100	3.2	100	5.3	105	3.6	100	3.8	110	2.9
cycloxydim -T2SO (263/155) [a]	0.01	74.5	6.6	105	4.3	88.0	1.8	84.7	2.2	88.8	3.1
	0.10	76.1	5.2	92.2	2.3	77.9	11	80.5	6.1	87.0	3.0
cycloxydim-T2SO (263/219)	0.01	77.6	3.5	99.2	2.4	86.0	4.0	103	1.1	101	3.2
	0.10	77.1	1.8	94.0	2.4	82.8	12	84.6	1.4	86.5	3.9
cycloxydim-T1SO2 (263/155 [a]	0.01	97.7	5.4	97.2	6.1	101	6.0	105	11	94.8	2.3
	0.10	106	4.0	101	4.4	109	2.4	106	3.8	99.6	5.1
cycloxydim-T1SO2 (263/219)	0.01	92.2	2.5	102	4.1	106	4.0	84.0	17	96.4	2.2
	0.10	98.2	2.6	98.4	3.8	103	4.5	100	4.3	102	2.0
cycloxydim -5-OH-TSO (279/175) [a]	0.01	117	3.0	98.0	8.9	83.0	4.2	100	6.7	91.1	3.5
	0.10	101	2.1	88.2	2.1	96.0	3.8	98.5	4.0	90.0	2.6
cycloxydim-5-OH-TSO (279/217)	0.01	111	5.1	92.0	4.9	79.7	13	102	2.3	88.0	4.9
	0.10	97.7	4.7	80.2	2.3	100	3.4	92.7	2.9	89.9	3.1
cycloxydim-5-OH-TS (279/175) [a]	0.01	105	4.7	91.9	3.4	91.0	5.6	75.7	2.3	98.8	3.4
	0.10	88.8	5.3	79.2	4.0	89.2	2.0	78.4	5.4	101	1.1
cycloxydim-5-OH-TS (279/217)	0.01	101	2.1	89.2	5.5	98.2	3.1	77.2	7.8	95.9	2.7
	0.10	93.4	7.0	79.9	6.7	89.5	2.3	78.2	5.1	99.3	3.1

[a] Used for quantification

Stability of residues in stored analytical samples

For investigating the storage stability of cycloxydim residues in plant matrices (sugar beet, pineapple, oilseed rape seed), the sample material was fortified with a mixture of cycloxydim-TSO + cycloxydim-T2SO2 and cycloxydim-5-OH-TSO2 (Tilting, 1992a). The samples were stored at -20 °C over a period of 2 years. Samples were taken and analysed immediately after spiking, 30, 90, 180, 360 and 720 days thereafter using Method 263. The results are shown in Table 61.

Table 61 Storage stability investigations, in % remaining [a]

Crop	Day	Cycloxydim-TSO + cycloxydim-T2SO2 (0.5 mg/kg)	Cycloxydim-5-OH-TSO2 (0.435 mg/kg)
Sugar beet	0	103.0	90.8
	32	81.3	72.8
	89	91.9	94.7
	179	124.0	121.6
	357	125.0	119.1
	718	90.2	90.8
Rape seed	0	101.1	88.7
	32	95.6	99.3
	90	103.5	103.9
	181	120.5	119.1
	358	113.0	110.8
	719	92.4	86.2
Pineapple	0	88.0	84.7
	29	104.0	104.7
	90	88.6	84.4
	180	94.4	87.1
	361	121.0	119.5
	720	92.6	80.2

[a] from duplicate analysis

The deep freeze stability of cycloxydim and cycloxydim-5-OH-TSO2 in different plant matrices was investigated over a period of about two years at -20 °C (Lehmann & Mackenroth, 2003a).The samples were spiked at a concentration level of 0.45–0.5 mg/kg and analysed after different storing intervals with Method No. 407/1. The residue data are shown in Tables 62 and 63.

Table 62 Storage stability investigations in maize matrices, in % remaining

	Forage		Seed		Straw	
Days	Cycloxydim	Cycloxydim-5-OH-TSO2	Cycloxydim	Cycloxydim-5-OH-TSO2	Cycloxydim	Cycloxydim-5-OH-TSO2
0	100	100	100	100	100	100
32	90	110	113	134	102	116
82	73	95	89	119	96	119
186	84	90	94	112	92	106
747	98	80	82	79	78	96

Table 63 Storage stability investigations in plant matrices, in % remaining

	Oilseed Rape		Pea seed		Strawberry	
Days	Cycloxydim	Cycloxydim-5-OH-TSO2	Cycloxydim	Cycloxydim-5-OH-TSO2	Cycloxydim	Cycloxydim-5-OH-TSO2
0	100	100	100	100	100	100
32	111	87	96	110	97	92
82	116	95	88	95	99	92
186	86	83	81	90	97	96
747	86	72	91	80	101	76

Selected samples from animal metabolism studies with [^{14}C] cycloxydim or its metabolite [^{14}C] cycloxydim-5-OH-TSO were re-extracted with methanol or extracted with the solvent mixture used in the common moiety method in order to investigate their stability in animal matrices (Grosshans & Kloeppner, 2009a). The% remaining rates of cycloxydim and its metabolites over 78–89 months of storage at -20 °C are shown in Table 64.

Table 64 Stability of [^{14}C] cycloxydim and its metabolites in samples of goat liver and milk re-extracted after 78–89 months of storage determined by HPLC-radio detector

	Hafernann & Knoell, 2003		78–89 months		
	mg/kg	% TRR	mg/kg	% TRR	% Remaining [a]
Liver (methanol extract)					
cycloxydim-T1SO, sum of diastereomers	0.001	1.8	0.004	5.4	300
cycloxydim-TSO, sum of diastereomers	0.006	8.1	0.007	9.1	112
cycloxydim-T2SO2	0.001	2.0	0.001	1.5	75
cycloxydim	0.008	10.8	0.004	5.3	49
Milk (SPE eluate of methanol extract)					
cycloxydim-T1SO, sum of diastereomers	0.004	16.5	0.003	14.9	90
cycloxydim-TSO, sum of diastereomers	0.003	14.9	0.004	18.0	121
	Tilting, 2003		78–89 months		
Liver (methanol extract)	mg/kg	% TRR	mg/kg	% TRR	% Remaining
cycloxydim-5-OH-TSO	0.022	10.6	0.031	14.9	141
cycloxydim-5-OH-T2SO	0.004	2.1	0.014	6.9	330
cycloxydim-5-OH-TS	0.035	17.4	0.021	10.1	58
Milk (acetonitrile phase of methanol extract)					
cycloxydim-5-OH-TSO	0.007	33.9	0.004	19.5	58
cycloxydim-5-OH-T1SO	0.002	8.4	0.002	9.7	116
cycloxydim-5-OH-T2SO	0.001	6.1	0.004	20.4	400

[a] % TRR current study / % TRR metabolism study;

USE PATTERN

Cycloxydim is only registered as solo formulation to control annual and perennial grass weeds on a variety of crops. It is used as a post-emergence herbicide before the crop canopy prevents adequate spray penetration. The product is registered to be used in various countries in Europe, Africa and the Middle East. Table 65 shows the registered used of cycloxydim in countries and crops that are relevant to this evaluation.

Table 65 Registered use(s) of cycloxydim

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Apple	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Apple	Portugal	1			0.2–0.4	28
Apricot	Italy	1	0.125–0.3	200–400	0.5–0.6	30
Beans, except broad bean and soya bean	Albania	1	–	–	0.5	–
Beans, except broad bean and soya bean	Czech Republic	–	0.133–0.2	200–300	0.4	–
Beans, except broad bean and soya bean	Greece	1	0.1–0.2	200–400	0.4	28
Beans, except broad bean and soya bean	Slovakia	–	0.133–0.4	100–300	0.4	–
Beans, except broad bean and soya bean	Slovenia	1	0.075–0.2	200–400	0.3–0.4	28
Beans, except broad bean and soya bean	Sweden	1	0.2–0.6	100–200	0.4–0.6	60
Bean, dwarf Bean, dwarf (dry)	United Kingdom	1	0.136–0.225	200–330	0.45	35
Bean, scarlet runner (pods & seeds)	Austria	1	0.167–025	200–300	0.5	100
Beans, shelled	Albania	1	–	–	0.5	–
Beans, shelled	Czech Republic	–	0.133–0.2	200–300	0.4	–

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Beans, shelled	Norway	2	3.3–6	20–30	0.5–0.6	–
Beans, shelled	Slovakia	–	0.133–0.4	100–300	0.4	–
Beans, shelled	Slovenia	1	0.075–0.2	200–400	0.3–0.4	28
Beans, shelled	Sweden	1	0.2–0.6	100–200	0.4–0.6	60
Beans (dry)	Albania	1	–	–	0.5	–
Beans (dry)	Czech Republic	–	0.133–0.2	200–300	0.4	–
Beans (dry)	France	1	0.133–0.4	100–300	0.4	[b]
Beans (dry)	Greece	1	0.1–0.2	200–400	0.4	56
Beans (dry)	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Beans (dry)	Slovakia	–	0.133–0.4	100–300	0.4	–
Beans (dry)	Slovenia	1	0.075–0.2	200–400	0.3–0.4	[b]
Brassica vegetables, head cabbages, flowerhead brassicas	Czech Republic	–	0.133–0.2 (aerial appl.: 0.5–1.0)	200–300 aerial: 40–80	0.4	–
Brassica vegetables, head cabbages, flowerhead brassicas	Slovakia	–	0.133–0.2	100–300	0.4	–
Brassica vegetables, head cabbages, flowerhead brassicas	Spain	1	–	–	0.3–0.4	[a]
Brassica vegetables, head cabbages, flowerhead brassicas	Switzerland	1	–	–	0.5–0.6	28
Brassica leafy vegetables	Switzerland	1	–	–	0.5–0.6	28
Broad bean Broad bean (dry)	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Broad bean Broad bean (dry)	Ireland	1	0.133–0.4	100–300	0.4	56
Broad bean	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	28
Broad bean Broad bean (dry)	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	28
Broad bean Broad bean (dry)	Spain	1	–	–	0.3–0.4	[a]
Broad bean, shelled	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Brussels sprouts	Ireland	1	0.133–0.4	100–300	0.4	56
Brussels sprouts	Norway	2	3.3–6	20–30	0.5–0.6	–
Brussels sprouts	United Kingdom	1	0.136–0.225	200–330	0.45	56
Brussels sprouts	Portugal	1			0.2–0.4	56
Cabbages, head	Austria	1	0.167–025	200–300	0.5	28
Cabbages, head	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Cabbages, head	France	1	0.133–0.4	100–300	0.4	60
Cabbages, head	Ireland	1	0.133–0.4	100–300	0.4	28
Cabbages, head	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Cabbages, head	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	28
Cabbages, head	Norway	2	3.3–6	20–30	0.5–0.6	–
Cabbages, head	Portugal	1	0.067–0.2	200–600	0.4	28
Cabbages, head	Slovenia	1	0.075–0.2	200–400	0.3–0.4	28
Cabbages, head	Sweden	1	0.2–0.6	100–200	0.4–0.6	60
Cabbages, head	United Kingdom	1	0.136–0.225	200–330	0.45	28

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Carrot	Albania	1	–	–	0–5	–
Carrot	Austria	1	0.167–025	200–300	0.5	28
Carrot	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Carrot	France	1	0.133–0.4	100–300	0.4	42
Carrot	Ireland	1	0.133–0.4	100–300	0.4	[a]
Carrot	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Carrot	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	28
Carrot	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	42
Carrot	Norway	2	2.6–5	20–30	0.4–0.5	–
Carrot	Portugal	1	0.067–0.2	200–600	0.4	49
Carrot	Slovenia	1	0.075–0.2	200–400	0.3–0.4	28
Carrot	Sweden	1	0.2–0.6	100–200	0.4–0.6	60
Carrot	Switzerland	1	–	–	0.5–0.6	28
Carrot	United Kingdom	1	0.136–0.225	200–330	0.45	42
Cauliflower	Ireland	1	0.133–0.4	100–300	0.4	28
Cauliflower	Portugal	1			0.2–0.4	56
Cauliflower	Slovenia	1	0.075–0.2	200–400	0.3–0.4	28
Cauliflower	Sweden	1	0.2–0.6	100–200	0.4–0.6	60
Cauliflower	United Kingdom	1	0.136–0.225	200–330	0.45	28
Celeriac	France	1	0.133–0.4	100–300	0.4	48
Celeriac	Norway	2	3.3–6	20–30	0.5–0.6	–
Celeriac	Sweden	1	0.2–0.6	100–200	0.4–0.6	60
Celeriac	Switzerland	1	–	–	0.5–0.6	56
Field bean Field bean (dry)	Austria	1	0.167–025	200–300	0.5	56
Field bean Field bean (dry)	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Field bean	France	1	0.133–0.4	100–300	0.4	56
Field bean	Germany	1	0.25	200	0.5	56
Field bean Field bean (dry)	Ireland	1	0.133–0.4	100–300	0.4	56
Field bean	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	28
Field bean Field bean (dry)	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	28
Field bean Field bean (dry)	Portugal	1	0.067–0.2	200–600	0.4	49
Field bean Field bean (dry)	Romania	–	0.15–0.8	50–200	0.3–0.4	–
Field bean Field bean (dry)	Spain	1	–	–	0.3–0.4	[a]
Field bean Field bean (dry)	Switzerland	1	–	–	0.5–0.6	21
Field bean Field bean (dry)	United Kingdom	1	0.136–0.225	200–330	0.45	56
Field pea (dry)	Belgium	1	0.13–0.4	150–300	0.4–0.6	[b]
Field pea (dry)	Germany	1	0.167–0.333	150–300	0.5	[b]
Field pea (dry)	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	[b]
Field pea (dry)	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	[b]

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Fodder beet	Albania	1	–	–	0.5	–
Fodder beet	Austria	1	0.167–025	200–300	0.5	56
Fodder beet	Belgium	1	0.13–0.4	150–300	0.4–0.6	90
Fodder beet	Czech Republic	–	0.133–0.2	200–300	0.4	–
Fodder beet	Germany	1	0.167–0.25	200–300	0.5	[a]
Fodder beet	Ireland	1	0.133–0.4	100–300	0.4	56
Fodder beet	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	90
Fodder beet	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	21
Fodder beet	Slovakia	–	0.133–0.4	100–300	0.4	–
Fodder beet	Slovenia	1	0.15–0.222	180–200	0.3–0.4	56
Fodder beet	Switzerland	1	–	–	0.5–0.6	56
Fodder beet	United Kingdom	1	0.136–0.225	200–330	0.45	56
Garden pea	Belgium	1	0.13–0.4	150–300	0.4–0.6	56
Garden pea, shelled	Belgium	1	0.13–0.4	150–300	0.4–0.6	56
Garden peas, shelled	Germany	1	0.167–0.333	150–300	0.5	35
Garden peas, shelled	Hungary	1	0.15–0.4	100–200	0.3–0.4	60
Garden peas, shelled	Ireland	1	0.133–0.4	100–300	0.4	35
Garden peas, shelled	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Garden peas, shelled	Spain	1	–	–	0.3–0.4	[a]
Garden peas, shelled	United Kingdom	1	0.136–0.225	200–330	0.45	35
Grapes	Croatia	1	0.175–0.4	100–200	0.35–0.4	42
Grapes	France	1	0.133–0.4	100–300	0.4	90
Grapes	Greece	1	0.1–0.2	200–400	0.4	42
Grapes	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Grapes	Serbia	1	0.075–0.2	200–400	0.3–0.4	–
Grapes	Spain	1	–	–	0.3–0.4	[a]
Grapes	Switzerland	1	–	–	0.5–0.6	[a]
Green bean	Austria	1	0.167–025	200–300	0.5	28
Green bean	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Green bean	Italy	1	0.125–0.3	200–400	0.5–0.6	30
Green bean	Portugal	1	0.067–0.2	200–600	0.4	49
Green bean	Spain	1	–	–	0.3–0.4	[a]
Leek	Austria	1	0.167–025	200–300	0.5	28
Leek	France	1	0.133–0.4	100–300	0.4	42
Leek	Ireland	1	0.133–0.4	100–300	0.4	[a]
Leek	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Leek	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	42
Leek	Norway	2	2.7–6	20–30	0.4–0.6	–
Leek	Portugal	1	0.067–0.2	200–600	0.4	42
Leek	Slovenia	1	0.075–0.2	200–400	0.3–0.4	28
Leek	Switzerland	1	–	–	0.5–0.6	56
Leek	United Kingdom	1	0.136–0.225	200–330	0.45	56
Linseed	Czech Republic	–	0.133–0.2	200–300	0.4	–
Linseed	France	1	0.133–0.4	100–300	0.4	90

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Linseed	Ireland	1	0.133–0.4	100–300	0.4	[a]
Linseed	Latvia	1–2	0.075–0.2	100–200	0.15–0.2	40
Linseed	Lithuania	1	0.075–0.2	100–200	0.15–0.2	–
Linseed	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	[a]
Linseed	Norway	2	3.3–6	20–30	0.5–0.6	–
Linseed	Slovakia	–	0.133–0.4	100–300	0.4	–
Linseed	Sweden	1	0.2–0.6	100–200	0.4–0.6	[a]
Linseed	United Kingdom	1	0.136–0.225	200–330	0.45	84
Lettuce, leaf	France	1	0.133–0.4	100–300	0.4	21
Lettuce, leaf	Greece	1	0.1–0.2	200–400	0.4	28
Lettuce, leaf	Italy	1	0.125–0.3	200–400	0.5–0.6	30
Lettuce, leaf	Portugal	1	0.067–0.2	200–600	0.4	42
Lettuce, leaf	Slovenia	1	0.075–0.2	200–400	0.3–0.4	14
Maize	Belgium	1	0.26–0.4	150–300	0.4	[a]
Maize	Czech Republic	1	0.133	300	0.4	–
Maize	France	1	0.133–0.4	100–300	0.4	70 grain: 90
Maize	Germany	1	0.16–0.2	200–250	0.4	[a]
Maize	Hungary	1	0.1–0.16	250–300	0.3–0.4	–
Maize	Luxembourg	1	0.133–0.267	150–300	0.4	[a]
Maize	Poland	1	0.033–0.2	200–300	0.1–0.4	14
Maize	Switzerland	1	–	–	0.2–0.4	[a]
Onion	Albania	1	–	–	0.5	–
Onion	Austria	1	0.167–025	200–300	0.5	56
Onion	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Onion	Ireland	1	0.133–0.4	100–300	0.4	[a]
Onion	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Onion	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	28
Onion	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	21
Onion	Norway	2	2.7–6	20–30	0.4–0.6	–
Onion	Slovenia	1	0.075–0.2	200–400	0.3–0.4	21
Onion	Spain	1	–	–	0.3–0.4	[a]
Onion	Sweden	1	0.2–0.6	100–200	0.4–0.6	40
Onion	Switzerland	1	–	–	0.5–0–6	56
Onion	United Kingdom	1	0.136–0.225	200–330	0.45	42
Peach	Italy	1	0.125–0.3	200–400	0.5–0.6	30
Peach	Portugal	1	0.067–0.2	200–600	0.4	28
Peach	Slovenia	1	–	–	0.3–0.4	100
Peas	Albania	1	–	–	0.5	–
Peas	Austria	1	0.167–025	200–300	0.5	56
Peas	Czech Republic	–	0.133–0.2	200–300	0.4	–
Peas	France	1	0.133–0.4	100–300	0.4	42
Peas	Portugal	1	0.067–0.2	200–600	0.4	49

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Peas	Slovakia	–	0.133–0.4	100–300	0.4	–
Peas	Slovenia	1	0.075–0.2	200–400	0.3–0.4	35
Peas	Sweden	1	0.2–0.6	100–200	0.4–0.6	[a]
Peas	Switzerland	1	–	–	0.5–0.6	28
Peas (dry)	Albania	1	–	–	0.5	–
Peas (dry)	France	1	0.133–0.4	100–300	0.4	56
Peas (dry)	Hungary	1	0.15–0.4	100–200	0.3–0.4	[b]
Peas (dry)	Ireland	1	0.133–0.4	100–300	0.4	[b]
Peas (dry)	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Peas (dry)	Norway	2	3.3–6	20–30	0.5–0.6	
Peas (dry)	Slovakia	–	0.133–0.4	100–300	0.4	–
Peas (dry)	Slovenia	1	0.075–0.2	200–400	0.3–0.4	[b]
Peas (dry)	Spain	1	–	–	0.3–0.4	[a]
Peas (dry)	Sweden	1	0.2–0.6	100–200	0.4–0.6	[a]
Peas (dry)	Switzerland	1	–	–	0.5–0.6	[b]
Peas (dry)	United Kingdom	1	0.136–0.225	200–330	0.45	[b]
Peas, shelled	Albania	1	–	–	0.5	–
Peas, shelled	Austria	1	0.167–025	200–300	0.5	56
Peas, shelled	Czech Republic	–	0.133–0.2	200–300	0.4	–
Peas, shelled	France	1	0.133–0.4	100–300	0.4	42
Peas, shelled	Portugal	1	0.067–0.2	200–600	0.4	49
Peas, shelled	Slovakia	–	0.133–0.4	100–300	0.4	–
Peas, shelled	Slovenia	1	0.075–0.2	200–400	0.3–0.4	35
Peas, shelled	Sweden	1	0.2–0.6	100–200	0.4–0.6	[a]
Pear	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Pear	Portugal	1	0.067–0.2	200–600	0.4	28
Pear	Slovenia	1	–	–	0.3–0.4	100
Peppers, Chilli, sweet	Italy	1	0.125–0.3	200–400	0.5–0.6	20
Pome fruits	Croatia	1	0.175–0.4	100–200	0.35–0.4	42
Pome fruits	Switzerland	1	–	–	0.5–0.6	[a]
Oil poppy	France	1	0.133–0.4	100–300	0.4	90
Poppy seed	Slovakia	–	0.133–0.4	100–300	0.4	–
Potato	Belgium	1	0.13–0.4	150–300	0.4–0.6	56
Potato	Croatia	1	0.15–0.4	100–200	0.3–0.4	56
Potato	Czech Republic	–	0.133–0.2	200–300	0.4	–
Potato	Estonia	1	0.2–0.6	100–200	0.4–0.6	–
Potato	Finland	1	0.2–0.4	150–200	0.4–0.6	55
Potato	France	1	0.133–0.4	100–300	0.4	70
Potato	Greece	1	0.1–0.2	200–400	0.4	–
Potato	Hungary	1	0.15–0.4	100–200	0.3–0.4	45
Potato	Ireland	1	0.133–0.4	100–300	0.4	56
Potato	Italy	1	0.125–0.3	200–400	0.5–0.6	100

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Potato	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	56
Potato	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	56
Potato	Norway	2	3.3–6	20–30	0.5–0.6	–
Potato	Portugal	1	0.067–0.2	200–600	0.4	84
Potato	Serbia	1	0.075–0.2	200–400	0.3–0.4	–
Potato	Slovakia	–	0.133–0.4	100–300	0.4	–
Potato	Slovenia	1	0.15–0.222	180–200	0.3–0.4	77
Potato	Spain	1	–	–	0.3–0.4	[a]
Potato	Sweden	1	0.2–0.6	100–200	0.4–0.6	[a]
Potato	Switzerland	1	–	–	0.5–0.6	56
Potato	United Kingdom	1	0.136–0.225	200–330	0.45	56
Rape seed	Austria	1	0.167–025	200–300	0.5	84
Rape seed	Belgium	1	0.13–0.4	150–300	0.4–0.6	28
Rape seed	Croatia	1	0.15–0.4	100–200	0.3–0.4	–
Rape seed	Czech Republic	–	0.133–0.2	200–300	0.4	–
Rape seed	Estonia	1	0.2–0.6	100–200	0.4–0.6	–
Rape seed	France	1	0.133–0.4	100–300	0.4	90
Rape seed	Germany	1	0.167–0.333	150–300	0.5	[a]
Rape seed	Hungary	1	0.15–0.4	100–200	0.3–0.4	120
Rape seed	Ireland	1	0.133–0.4	100–300	0.4	84
Rape seed	Italy	1	0.125–0.3	200–400	0.5–0.6	100
Rape seed	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	28
Rape seed	Norway	2	3.3–6	20–30	0.5–0.6	–
Rape seed	Portugal	1	0.067–0.2	200–600	0.4	[a]
Rape seed	Slovakia	–	0.133–0.4	100–300	0.4	–
Rape seed	Slovenia	1	0.15–0.222	180–200	0.3–0.4	100
Rape seed	Sweden	1	0.2–0.6	100–200	0.4–0.6	[a]
Rape seed	Switzerland	1	–	–	0.5–0.6	84
Rape seed	United Kingdom	1	0.136–0.225	200–330	0.45	84
Red beet	Belgium	1	0.13–0.4	150–300	0.4–0.6	90
Red beet	Norway	2	2.6–5	20–30	0.4–0.5	–
Red beet	Spain	1	–	–	0.3–0.4	[a]
Red beet	Switzerland	1	–	–	0.5–0.6	56
Rice	Italy	1	0.1–0.2	200–400	0.4	[a]
Soya bean	Albania	1	–	–	0.5	–
Soya bean	Austria	1	0.167–025	200–300	0.5	56
Soya bean	Croatia	1	0.15–0.4	100–200	0.3–0.4	–
Soya bean	France	1	0.133–0.4	100–300	0.4	56
Soya bean	Germany	1	0.167–0.25	200–300	0.5	[a]
Soya bean	Hungary	1	0.15–0.4	100–200	0.3–0.4	90
Soya bean	Romania	–	0.15–0.8	50–200	0.3–0.4	–
Soya bean	Serbia	1	0.075–0.2	200–400	0.3–0.4	–
Soya bean	Slovakia	–	0.133–0.4	100–300	0.4	–
Soya bean	Slovenia	1	0.075–0.2	200–400	0.3–0.4	35

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Soya bean	Spain	1	–	–	0.3–0.4	[a]
Soya bean	Switzerland	1	–	–	0.5–0.6	56
Spinach	Cyprus	1	0.0875–0.225	200–400	0.35–0.45	28
Spinach	Cyprus	1	0.0875–0.225	200–400	0.35–0.45	28
Spinach	France	1	0.133–0.4	100–300	0.4	42
Spinach	Italy	1	0.125–0.3	200–400	0.5–0.6	30
Spinach	Slovenia	1	0.075–0.2	200–400	0.3–0.4	28
Stone fruits	Croatia	1	0.175–0.4	100–200	0.35–0.4	42
Stone fruits	Switzerland	1	–	–	0.5–0.6	[a]
Strawberry	Czech Republic	–	0.133–0.2	200–300	0.4	–
Strawberry	Norway	2	4–6	20–30	0.6	–
Strawberry	Romania	–	0.15–0.8	50–200	0.3–0.4	–
Strawberry	Slovakia	–	0.133–0.4	100–300	0.4	–
Strawberry	Slovenia	1	0.15–0.222	180–200	0.3–0.4	100
Strawberry	Switzerland	1	–	–	0.5–0.6	42
Strawberry	United Kingdom	1	0.136–0.225	200–330	0.45	42
Sugar beet	Austria	1	0.167–025	200–300	0.5	56
Sugar beet	Belgium	1	0.13–0.4	150–300	0.4–0.6	90
Sugar beet	Croatia	1	0.15–0.4	100–200	0.3–0.4	77
Sugar beet	Czech Republic	–	0.133–0.2	200–300	0.4	–
Sugar beet	Estonia	1	0.2–0.6	100–200	0.4–0.6	–
Sugar beet	France	1	0.133–0.4	100–300	0.4	70
Sugar beet	Germany	1	0.167–0.25	200–300	0.5	[a]
Sugar beet	Greece	1	0.1–0.2	200–400	0.4	–
Sugar beet	Hungary	1	0.15–0.4	100–200	0.3–0.4	60
Sugar beet	Ireland	1	0.133–0.4	100–300	0.4	56
Sugar beet	Italy	1	0.125–0.3	200–400	0.5–0.6	100
Sugar beet	Luxembourg	1	0.133–0.4	150–300	0.4–0.6	90
Sugar beet	Netherlands	1–2	0.1–0.3	200–400	0.4–0.6	[a]
Sugar beet	Portugal	1	0.067–0.2	200–600	0.4	84
Sugar beet	Romania	–	0.2–0.9	50–200	0.4–0.45	–
Sugar beet	Serbia	1	0.075–0.2	200–400	0.3–0.4	–
Sugar beet	Slovakia	–	0.133–0.4	100–300	0.4	–
Sugar beet	Slovenia	1	0.15–0.222	180–200	0.3–0.4	77
Sugar beet	Spain	1	–	–	0.3–0.4	[a]
Sugar beet	Switzerland	1	–	–	0.5–0.6	56
Sugar beet	United Kingdom	1	0.136–0.225	200–330	0.45	56
Sunflower seed	Albania	1	–	–	0.5	–
Sunflower seed	Austria	1	0.167–025	200–300	0.5	56
Sunflower seed	Croatia	1	0.15–0.4	100–200	0.3–0.4	–
Sunflower seed	Czech Republic	–	0.133–0.2	200–300	0.4	–
Sunflower seed	France	1	0.133–0.4	100–300	0.4	[a]
Sunflower seed	Germany	1	0.167–0.333	150–300	0.5	100
Sunflower seed	Hungary	1	0.15–0.4	100–200	0.3–0.4	90
Sunflower seed	Italy	1	0.125–0.3	200–400	0.5–0.6	80
Sunflower seed	Portugal	1	0.067–0.2	200–600	0.4	98

Crop	Country	No. per crop season	kg ai/hL	Water L/ha	kg ai/ha	PHI [days]
Sunflower seed	Romania	–	0.15–0.8	50–200	0.3–0.4	–
Sunflower seed	Serbia	1	0.075–0.2	200–400	0.3–0.4	–
Sunflower seed	Slovakia	–	0.133–0.4	100–300	0.4	–
Sunflower seed	Slovenia	1	0.15–0.222	180–200	0.3–0.4	100
Sunflower seed	Switzerland	1	–	–	0.5–0.6	56
Swedes	United Kingdom	1	0.136–0.225	200–330	0.45	56
Tomato	Albania	1	–	–	0.5	–
Tomato	France	1	0.133–0.4	100–300	0.4	60
Tomato	Greece	1	0.1–0.2	200–400	0.4	35
Tomato	Italy	1	0.125–0.3	200–400	0.5–0.6	60
Tomato	Serbia	1	0.075–0.2	200–400	0.3–0.4	–
Tomato	Switzerland	1	–	–	0.5–0.6	56
Turnip	Slovenia	1	0.075–0.2	200–400	0.3–0.4	56

[a] Application at the growing stage of 2–5 weed leaf for annual weeds or when the perennial weed are 20–30 cm high

[b] Beans or peas could be harvested at the stage of mature seeds, but the worst case scenario is covered by the harvest of immature seeds.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Four hundred and eighty one (481) supervised residue trials conducted with cycloxydim on a variety of crops in Europe were submitted to the Meeting. Table 66 summarizes the data. Studies were conducted according to GLP, with the exception of those conducted in the late 80s and early 90s. Unless specified concurrent determination of residues in untreated crops gave residues < LOQ. All trials were conducted in Europe using EC formulation as a soil or broadcast application in early post-emergence.

In all trials, samples were analysed using common moiety methods. In the LC methods, residues of cycloxydim-TGSO2 and cycloxydim-5-OH-TGSO2 were calculated as cycloxydim (factor of 1.23) and cycloxydim-5-OH-TSO2 (factor of 1.33), respectively. In the GC methods, residues of cycloxydim-TDME and cycloxydim-5-OH-TDME were calculated as cycloxydim (factor of 1.11) and cycloxydim-5-OH-TSO2 (factor of 1.21), respectively.

The results of the supervised trials in the tables are expressed as Cy1 to indicate the residues of cycloxydim and all metabolites that can be oxidized cycloxydim-TGSO2, calculated as cycloxydim, and as Cy2 to indicate the residues of cycloxydim-5-OH-TSO2 and all metabolites that can be oxidized to cycloxydim-5-OH-TGSO2, calculated as cycloxydim-5-OH-TSO2. Total residues arriving from the use of cycloxydim are the sum of Cy1 and Cy2 (Total cycloxydim), calculated as cycloxydim (factor of 0.87). In some samples, residues of cycloxydim-TSO (Cy-TSO) were also estimated using a specific method, and residues expressed as cycloxydim (factor of 0.95).

Residues of cycloxydim arising from use patterns where rate or PHI or ± 25% of GAP are underlined and considered for estimation of maximum residue levels, STMRs and HRs. When residues in samples harvested at a later stage were higher than those found at the critical PHI, they were used for the estimations. When a GAP with no specified PHI was used to support a trial, the highest residue found in the trials, except that of 0 day PHI, was selected for the estimation. In the tables, DAT means days after treatment.

Table 66 Summary or the residue trials conducted with cycloxydim in Europe

Table number	Crop	Number of trials	Period
67	Apple/pears	4	2006
68	Apricot/peach	5	1990–1991
69	Grape	16	2005–2006
70	Strawberry	16	1989–2008

Table number	Crop	Number of trials	Period
71	Onion	19	1995–2005
72	Leek	14	1989–2007
73	Brussels sprouts	12	2005–2007
74	Cabbage	14	2001–2006
75	Cauliflower	14	2001–2006
76	Pepper	8	1991–2006
77	Tomato	16	2005–2006
78	Kale, curly	22	2005
79	Lettuce	8	2001–2008
80	Spinach	8	2005
81	Green beans	15	1988–2008
82	Green peas	14	2006
83	Dry beans	21	2001–2006
84	Dry peas	14	1987–2002
85	Soya beans	13	2005–2007
86	Carrots	15	2001–2007
87	Celeriac	8	2006–2007
88	Potato	18	1986–2002
89	Turnips	6	1987–1989
90	Sugar beet	18	1988–2001
91	Maize	14	1995–1996
92	Rice	11	1993–1996
93	Rape seed	15	1987–2008
94	Sunflower	19	1993–2008
Feed commodities			
95	Beans	33	2001–2008
96	Peas	25	2005–2006
97	Sugar beet tops	16	1986–2001
98	Maize straw	19	1995–1996
99	Rice straw	8	1995–1996
100	Rape seed forage	3	1989

Pome fruits

Four residue trials were conducted in apples and pears in Spain and Italy in 2006. Samples were analysed according to method 407/1. The results are shown in Table 67.

Table 67 Results of residue trials conducted with cycloxydim on apples and pears

Country, year	Crop Variety	Application			DAT, days	Residues, mg/kg			Study Trial No
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	
Italy, 2006	Apple Granny Smith	1	0.500	0.125	0 22 28 35	< 0.05 < 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 <u>< 0.09</u> < 0.09	2007/ 1020716 06IT/006R
Spain, 2006	Apple Golden	1	0.500	0.125	0 22 28 35	< 0.05 < 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 <u>< 0.09</u> < 0.09	2007/ 1020716 06ES/005R
Italy, 2006	Pear William	1	0.500	0.125	0 22 28 35	< 0.05 < 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 <u>< 0.09</u> < 0.09	2007/ 1020716 06IT/006R
Spain, 2006	Pear Tosca	1	0.500	0.125	0 22 28 35	< 0.05 < 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 <u>< 0.09</u> < 0.09	2007/ 1020716 06ES/005R

Stone fruits

Five residue trials were conducted in apricots and peaches in Italy in 1990–1991. Residues were determined using Method 263. The results are shown in Table 68.

Table 68 Results of residue trials conducted with cycloxydim on apricots and peaches

Country, year	Crop Variety	Application			DAT, days	Residues, mg/kg			Study Trial No
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	
Italy, 1991	Apricot Precoce di Colomer	1	0.600	0.200	25 50 75	0.050 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04	≤ 0.09 < 0.09 < 0.09	1991/ 11589 IT5/037
Italy, 1991	Apricot Reale d'Imola	1	0.600	0.200	25 50 75	< 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04	≤ 0.09 < 0.09 < 0.09	1991/ 11589 IT5/038
Italy, 1990	Peach Fayette	1	0.600	0.200	25 50 73	< 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04	≤ 0.09 < 0.09 < 0.09	1991/ 11588 IT5/083
Italy, 1990	Peach Baby	1	0.600	0.200	24 49 74	< 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04	≤ 0.09 < 0.09 < 0.09	1991/ 11588 IT5/086
Italy, 1991	Peach Fayette	1	0.600	0.200	24 49 72	< 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04	≤ 0.09 < 0.09 < 0.09	1991/ 11588 IT5/062

Grapes

Sixteen residue trials were conducted in grapes in Europe in 2005–2006. Residues were determined using Method 407/1. The results are shown in Table 69.

Table 69 Results of residue trials conducted with cycloxydim on grapes

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
France, 2005 Centre	Grape Chenin	1	0.500	0.167	0 35 42 49	< 0.05 < 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 < 0.09 < 0.09	– – – –	2006/ 1030352 05 H VI FR P16
France, 2005 North	Grape Pinot Meunier	1	0.500	0.167	0 34 41 49	< 0.05 < 0.05 0.14 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 0.18 < 0.09	– – – –	2006/ 1030352 05 H VI FR P17
France, 2005 South	Grape Merlot	1	0.500	0.167	0 35 42	< 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04	< 0.09 < 0.09 < 0.09	– – –	2006/ 1030352 05 H VI FR P15
France, 2006 North	Grape Chardonnay	1	0.500	0.167	0 35 42	0.07 < 0.05 0.09	< 0.04 < 0.04 < 0.04	0.11 < 0.09 0.13	– – < 0.05	2007/ 1020717 06FR/022R
France, 2006 North	Grape Chenin	1	0.500	0.167	0 35 42 49	< 0.05 0.09 0.08 0.08	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 0.13 0.12 0.12	– – < 0.05 < 0.05	2007/ 1020717 06FR/023R
France, 2006 South	Grape Carignan	1	0.500	0.167	0 35 42 49	< 0.05 < 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 < 0.09 < 0.09	– – – –	2007/ 1020717 06FR/020R
Greece, 2005	Grape Muscat	1	0.500	0.167	0 35 42 49	< 0.05 < 0.05 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	< 0.09 < 0.09 < 0.09 < 0.09	– – – –	2006/ 1030352 05RF036
Greece, 2006	Grape Muschat	1	0.500	0.167	0 35	< 0.05 < 0.05	< 0.04 < 0.04	< 0.09 < 0.09	– –	2007/ 1020717 06GR/021R

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
Germany, 2006	Grape Kerner	1	0.500	0.167	0	< 0.05	< 0.04	< 0.09	–	2007/ 1020717 06GE/024R
					35	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
Germany, 2006	Grape Riesling	1	0.500	0.167	0	< 0.05	< 0.04	< 0.09	–	2007/ 1020717 06GE/025R
					35	0.07	< 0.04	0.11	–	
					42	< 0.05	< 0.04	< 0.09	–	
					49	0.09	0.09	0.18	< 0.05	
Germany, 2005	Grape Silvaner	1	0.500	0.167	0	< 0.05	< 0.04	< 0.09	–	2006/ 1030352 AT-07/004-1
					36	< 0.05	< 0.04	< 0.09	–	
					44	< 0.05	< 0.04	< 0.09	–	
					50	< 0.05	< 0.04	< 0.09	–	
Germany, 2005	Grape Silvaner	1	0.500	0.167	0	< 0.05	< 0.04	< 0.09	–	2006/ 1030352 AT-07/004-2
					35	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					48	< 0.05	< 0.04	< 0.09	–	
Italy, 2005	Grape Trebbiano Romagnolo	1	0.500	0.167	0	< 0.05	< 0.04	< 0.09	–	2006/ 1030352 0542R
					35	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
Italy, 2006	Grape Trebbiano	1	0.500	0.167	0	< 0.05	< 0.04	< 0.09	–	2007/ 1020717 06IT/019R
					36	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
Spain, 2006	Grape Merlot	1	0.500	0.167	0	0.10	< 0.04	0.14	–	2007/ 1020717 06ES/017R
					35	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
Spain, 2006	Grape Airen	1	0.500	0.167	0	< 0.05	< 0.04	< 0.09	–	2007/ 1020717 06ES/018R
					35	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	

Strawberries

Sixteen residue trials were conducted in strawberries in Europe from 1989 to 2008. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 70.

Table 70 Results of residue trials conducted with cycloxydim on strawberries

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
France, 2005 South	Aromas	1	0.500	0.167	35	0.45	0.12	0.57	–	2006/ 1026946 05FR/035R
					42	0.21	0.05	0.26	–	
					49	0.16	0.04	0.20	–	
France, 2006 South	Naiad	1	0.500	0.167	35	0.21	0.14	0.35	–	2007/ 1020730 06FR/092R
					41	0.14	0.12	0.26	< 0.05	
					49	0.13	0.15	0.28	< 0.05	
Germany, 2008	Elsanta	1	0.500	0.333	34	0.08	0.08	0.16	–	2009/ 1069375 L080155
					42	0.08	0.11	0.19	0.08	
					49	0.12	0.22	0.34	< 0.05	
Greece, 2005	Toulda	1	0.500	0.167	35	0.32	0.17	0.49	–	2006/ 1026946 05GR/038R
					42	0.26	0.16	0.42	–	
					49	0.21	0.12	0.33	–	
Greece, 2006	Aroma	1	0.500	0.167	35	1.1	0.30	1.4	–	2007/ 1020730 06GR/093R
					42	0.45	0.27	0.72	< 0.05	
					48	0.31	0.12	0.43	< 0.05	
Italy, 2005	Tetis	1	0.500	0.167	35	0.54	0.20	0.74	–	2006/ 1026946

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
					42 49	0.29 0.10	0.07 < 0.04	0.36 0.14	– –	05IT/036R
Italy, 2006	Tetis	1	0.500	0.167	0 36 43 50	14.7 0.62 0.45 0.81	0.04 0.32 0.16 0.43	15 0.94 0.61 1. 4	– – 0.09 0.07	2007/ 1020730 06IT/091R
Netherlands, 2008	Elsanta	1	0.500	0.333	35 42 49	0.10 0.06 < 0.05	0.12 0.11 0.07	0.22 0.17 0.12	– < 0.05 –	2009/ 1069375 L080156
Spain, 2005	Camarosa	1	0.500	0.167	36 43 49	0.19 0.14 0.13	0.10 0.07 0.06	0.29 0.21 0.19	– – –	2006/ 1026946 05ES/037R
Spain, 2005/2006	Camarosa	1	0.500	0.167	0 35 42 48	0.55 0.21 0.11 0.11	< 0.04 0.12 0.06 0.06	0.59 0.33 0.17 0.17	– – < 0.05 < 0.05	2007/ 1020730 06ES/090R
Sweden, 1989	Senga Sengang	1	0.600	0.120	39	0.35	0.28	0.63	–	1992/ 12145 89/18E
United Kingdom, 1995	Elsanta	1	0.500	0.215	42	0.23	0.24	0.47	–	1997/ 10554 OAT/32/95
United Kingdom, 1995	Elsanta	1	0.500	0.215	41	0.15	0.18	0.33	–	1997/ 10554 OAT/33/95
United Kingdom, 1995	Elsanta	1	0.500	0.215	42	0.14	0.15	0.29	–	1997/ 10554 OAT/34/95
United Kingdom, 1995	Pegasus	1	0.500	0.215	42	0.12	0.07	0.19	–	1997/ 10554 OAT/35/95
United Kingdom, 2008	Florence	1	0.500	0.333	35 43 50	0.31 0.25 0.17	0.30 0.27 0.21	0.61 0.52 0.38	– 0.17 0.06	2009/ 1069375 L080154

Onions

Twenty three residue trials were conducted on onions in Europe from 1995 to 2005. Residues were determined with Methods 407/1 or 493/0. The results are shown in Table 71.

Table 71 Results of residue trials conducted with cycloxydim on onions

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
France, 2005 North	Onion Hyfield	1	0.500	0.333	plant [a] bulb bulb bulb	0 21 28 35	2.2 0.27 0.21 0.18	< 0.04 < 0.04 < 0.04 < 0.04	2.2 0.31 0.25 0.22	– – – –	2006/ 1029595 05 H CL FR P07
France, 2005 South	Onion Elody	1	0.500	0.333	plant [a] bulb bulb bulb	0 20 27 34	4.2 0.24 0.23 0.28	< 0.04 < 0.04 < 0.04 < 0.04	4.2 0.28 0.27 0.32	– – – –	2006/ 1029595 05 H CL FR P08
Greece, 2001	Onion Regina	1	0.611	0.195	wh. plant shallots bulb bulb bulb	0 42 28 42 55	2.25 0.74 0.24 0.24 0.2	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04	2.3 0.78 0.28 0.28 0.24	2.36 0.06 0.07 < 0.05 < 0.05	2003/ 1001289 01RF020
Greece, 2005	Onion Bermuda	1	0.500	0.333	plant [a] bulb bulb bulb	0 20 28 35	3.9 1.3 0.92 0.94	< 0.04 < 0.04 < 0.04 < 0.04	3.9 1.3 0.96 0.98	– – – –	2006/ 1029595 05RF038

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Italy, 2001	Onion Globix	1	0.587	0.200	wh. plant	0	3.13	< 0.04	3.2	2.5	2003/
					shallots	28	0.51	< 0.04	0.55	0.07	1001289
					bulb	28	0.27	< 0.04	0.31	0.13	0138R
					bulb	42	0.24	< 0.04	0.28	< 0.05	
					bulb	56	0.15	< 0.04	0.19	< 0.05	
Italy, 2005	Onion Alix	1	0.500	0.333	plant [a]	0	3.3	< 0.04	3.3	–	2006/
					bulb	21	0.21	< 0.04	0.25	–	1029595
					bulb	28	0.18	< 0.04	0.22	–	0544R
					bulb	35	0.13	< 0.04	0.17	–	
Netherlands, 2005	Onion Stuttgarter Riesen	1	0.500	0.333	plant [a]	0	3.6	< 0.04	3.6	–	2006/
					bulb	21	0.17	< 0.04	0.21	–	1029595
					bulb	29	0.17	< 0.04	0.21	–	AGR/50/05
					bulb	35	0.16	< 0.04	0.2	–	
Spain, 2001	Onion Grano	1	0.600	0.200	wh. plant	0	5.97	< 0.04	6.0	–	2005/
					leaves	30	5.04	0.12	5.16	–	1007586
					bulbs	30	0.21	< 0.04	0.25	–	01/V01/HR/
					bulbs	42	0.17	< 0.04	0.21	–	001
					skinned b.	63	0.12	< 0.04	0.16	–	
					dried b.	63	0.14	< 0.04	0.18	–	
Spain, 2001	Onion Grano	1	0.600	0.200	wh. plant	0	6.75	< 0.04	6.79	–	2005/
					leaves	29	0.52	< 0.04	0.56	–	1007586
					leaves	41	0.07	< 0.04	0.11	–	01/V02/HR/
					bulbs	29	0.08	< 0.04	0.12	–	001
					bulbs	41	< 0.05	< 0.04	< 0.09	–	
					skinned b.	62	0.07	< 0.04	0.11	–	
					dried b.	62	< 0.05	< 0.04	< 0.09	–	
Spain, 2001	Onion Grano	1	0.600	0.200	wh. plant	0	6.48	< 0.04	6.5	–	2005/
					leaves	28	0.75	< 0.04	0.79	–	1007586
					bulbs	28	0.46	< 0.04	0.50	–	01/V03/HR/
					leaves	43	0.44	< 0.04	0.48	–	001
					bulbs	43	0.11	< 0.04	0.15	–	
					peeled b.	64	0.14	< 0.04	0.18	–	
					dried b.	64	0.19	< 0.04	0.23	–	
Spain, 2002	Onion Reka	1	0.600	0.200	plant [a]	0	3.08	< 0.04	3.1	–	2003/
					plant [a]	28	0.11	< 0.04	0.15	–	1001250
					plant [a]	42	0.05	< 0.04	0.09	–	ALO/06/02
					bulb	28	< 0.05	< 0.04	< 0.09	–	
					bulb	42	< 0.05	< 0.04	< 0.09	–	
					bulb	56	< 0.05	< 0.04	< 0.09	–	
					dried o.	63	< 0.05	< 0.04	< 0.09	–	
					peeled o.	63	< 0.05	< 0.04	< 0.09	–	
Spain, 2002	Onion Vaquero	1	0.600	0.200	plant [a]	0	7.87	< 0.04	7.9	–	2003/
					plant [a]	28	0.28	< 0.04	0.32	–	1001250
					plant [a]	42	0.12	< 0.04	0.16	–	AYE/05/02
					bulb	28	0.06	< 0.04	0.10	–	
					bulb	42	< 0.05	< 0.04	< 0.09	–	
					bulb	56	< 0.05	< 0.04	< 0.09	–	
					dried o.	63	< 0.05	< 0.04	< 0.09	–	
					peeled o.	63	< 0.05	< 0.04	< 0.09	–	
Spain, 2005	Onion Elody	1	0.500	0.333	plant [a]	0	2.1	< 0.04	2.1	–	2006/
					bulb	21	0.13	< 0.04	0.17	–	1029595
					bulb	28	0.12	< 0.04	0.16	–	05ES/089R
					bulb	36	0.09	< 0.04	0.13	–	
Sweden, 2005	Onion Hytech	1	0.500	0.333	plant [a]	0	2.5	< 0.04	2.5	–	2006/
					bulb	21	0.29	< 0.04	0.33	–	1029595
					bulb	28	0.27	< 0.04	0.31	–	HUS/19502-
					bulb	35	0.24	< 0.04	0.28	–	01
United Kingdom, 2005	Onion Sturon	1	0.500	0.333	plant [a]	0	3.8	< 0.04	3.8	–	2006/
					bulb	21	0.53	< 0.04	0.57	–	1029595
					bulb	28	0.35	< 0.04	0.39	–	774/ONI/1
					bulb	35	0.37	< 0.04	0.41	–	
United Kingdom, 1995	Onion White	2	0.505 0.223	0.215 0.096	wh. plant bulb	0 26	10.5 < 0.05	0.08 < 0.04	11 < 0.09	– –	1997/ 10561 OAT/37/95

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
	Lisbon				stem	26	0.05	< 0.04	0.09	–	
		1	0.476	0.214	wh. plant	0	29.9	0.09	30	–	
					bulb	26	< 0.05	< 0.04	< 0.09	–	
					stem	26	< 0.05	< 0.04	< 0.09	–	
United Kingdom, 1995	Onion Guardsman	2	0.480 0.228	0.215 0.095	wh. plant bulb	0 28	5 < 0.05	0.11 < 0.04	5.1 < 0.09	– –	1997/ 10561 OAT/38/95
					stem	28	0.05	< 0.04	0.09	–	
		1	0.437	0.215	wh. plant	0	11.5	0.12	12	–	
					bulb	28	< 0.05	< 0.04	< 0.09	–	
					stem	28	0.06	< 0.04	0.10	–	
United Kingdom, 1995	Onion Guardsman	2	0.471 0.204	0.215 0.095	wh. plant bulb	0 29	11.4 < 0.05	0.08 < 0.04	12 < 0.09	– –	1997/ 10561 OAT/39/95
					stem	29	0.07	< 0.04	0.11	–	
		1	0.500	0.216	wh. plant	0	3.4	< 0.04	3.4	–	
					bulb	29	< 0.05	< 0.04	< 0.09	–	
					stem	29	0.09	< 0.04	0.15	–	
United Kingdom, 1995	Onion Hikasi	2	0.514 0.226	0.215 0.095	wh. plant bulb	0 26	3.9 < 0.05	0.09 < 0.04	4.0 < 0.09	– –	1997/ 10561 OAT/44/95
					stem	26	< 0.05	< 0.04	< 0.09	–	
		1	0.501	0.215	wh. plant	0	1.9	< 0.04	1.9	–	
					bulb	26	< 0.05	< 0.04	< 0.09	–	
					stem	26	0.07	< 0.04	0.11	–	

[a] Without root

Leeks

Fourteen residue trials were conducted on leeks in Europe from 1989 to 2007. Residues were determined with Methods 407/1 or 493/0. The results are shown in Table 72.

Table 72 Results of residue trials conducted with cycloxydim on leeks (plants without roots)

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy– TSO	Study Trial No.
Belgium, 2007	Harston	1	0.500	0.170	0	3.9	< 0.04	4.0	N/A	2009/ 1075171
					34	0.24	< 0.04	0.28	N/A	L070355
					41	0.22	< 0.04	0.26	0.07	
					49	0.13	< 0.04	0.17	0.06	
Denmark, 2007	Pandora	1	0.500	0.170	0	8.1	< 0.04	8.1	N/A	2009/ 1075171
					35	0.29	< 0.04	0.33	N/A	L070353
					42	0.13	< 0.04	0.17	< 0.05	
					49	0.08	< 0.04	0.12	< 0.05	
France, 2006 South	Azur	1	0.500	0.170	0	1.4	< 0.04	1.4	–	2007/ 1020726
					35	0.08	< 0.04	0.12	–	06FR/039R
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
France, 2007 North	Fahrenheit	1	0.500	0.170	0	4.8	< 0.04	4.9	N/A	2009/ 1075171
					35	0.34	< 0.04	0.38	N/A	L070354
					42	0.35	< 0.04	0.39	0.07	
					49	0.25	< 0.04	0.29	0.07	
Germany, 2007	Puristo	1	0.500	0.170	0	8.8	< 0.04	8.8	N/A	2009/ 1075171
					35	2.2	0.06	2.3	N/A	L070350
					42	2.1	0.05	2.2	0.79	
					49	0.99	< 0.04	1.0	0.56	
Greece, 2006	Demi	1	0.500	0.170	0	5.1	< 0.04	5.2	–	2007/ 1020726
					35	0.06	< 0.04	0.10	–	06GR/040R
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
Italy, 2006	Lungo Riviera	1	0.500	0.170	0	5.6	< 0.04	5.7	–	2007/ 1020726
					35	0.06	0.09	0.15	–	06IT/038R

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy– TSO	Study Trial No.
					42 49	< 0.05 < 0.05	< 0.04 < 0.04	< 0.09 < 0.09	– –	
Netherlands, 1989	Arkona	1	0.600	0.106	42	0.07	< 0.04	0.11	–	1992/12153; NL4/025 517 01H 89/76 E
Netherlands, 1989	Portant	1	0.600	0.106	42	0.20	< 0.04	0.24	–	1992/ 12153 NL4/024 517 01H 89/75 E
Netherlands, 1989	Portant	1	0.600	0.106	42	0.166	< 0.04	0.21	–	1992/ 12146 NL4/024 517 22H 89/30 E
Netherlands, 1989	Arkona	1	0.600	0.106	42	0.066	0.053	0.12	–	1992/ 12146; NL4/025 517 22H 89/31 E
Netherlands, 2007	Shelton	1	0.500	0.170	0 35 42 49	1.2 0.58 0.48 0.34	< 0.04 < 0.04 < 0.04 < 0.04	1.2 0.62 0.52 0.38	N/A N/A 0.20 0.17	2009/ 1075171 L070351
Spain, 2006	Arial	1	0.500	0.170	0 36 41 48	4.8 < 0.05 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	4.9 < 0.09 0.09 < 0.09	– – – –	2007/ 1020726 06ES/037R
United Kingdom, 2007	Roxton	1	0.500	0.170	0 35 42 49	5.8 0.09 0.09 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	5.8 0.13 0.13 < 0.09	N/A N/A < 0.05 N/A	2009/ 1075171 L070352

Brussels sprouts

Twelve residue trials were conducted on Brussels sprouts in Europe from 2005 to 2007. Residues were determined with Methods 407/1 or 493/0. The results are shown in Table 73.

Table 73 Results of residue trials conducted with cycloxydim on Brussels sprouts

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Belgium, 2005/2006	Brussels sprouts Maximus	1	0.500	0.333	plant [a] sprouts sprouts sprouts	0 35 42 49	4.2 1.5 1.4 1.1	0.10 0.26 0.18 0.19	4.3 1.8 1.6 1.3	– – – –	2006/ 1034211 G021-05 H
France, 2006 South	Brussels sprouts Ambitus F1	1	0.500	0.333	plant [a] sprouts sprouts sprouts	0 35 42 51	8.2 1.4 1.8 0.28	0.05 0.12 0.11 < 0.04	8.2 1.5 1.9 0.32	– – 1.06 < 0.05	2007/ 1020722 06FR/058R
France, 2005/2006 North	Brussels sprouts Cumulus	1	0.500	0.333	plant [a] sprouts sprouts sprouts	0 35 42 49	3.2 0.84 0.97 0.97	0.04 0.05 0.05 0.06	3.2 0.89 1.0 1.0	– – – –	2006/ 1034211 05 H CL FR P06
France, 2006 North	Brussels sprouts Cumulus	1	0.500	0.333	plant [a] sprouts sprouts sprouts	0 35 42 50	2.9 3.08 2.66 3.25	0.08 0.35 0.28 0.32	3.0 3.4 2.9 3.6	– – 1.94 2.40	2007/ 1020722 06FR/059R
Germany, 2006	Brussels sprouts Cyrus	1	0.500	0.333	plant [a] sprouts sprouts sprouts	0 35 42 49	7.7 0.84 0.86 0.51	0.14 0.17 0.21 0.18	7.8 1.0 1.1 0.69	– – 0.25 0.22	2007/ 1020722 06GE/060R
Greece, 2005	Brussels sprouts Ikaros	1	0.500	0.333	plant [a] sprouts sprouts sprouts	0 36 42 49	5.4 2.5 3.1 2.6	0.16 0.30 0.44 0.36	5.6 2.8 3.5 3.0	– – – –	2006/ 1034211 05RF037

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Italy, 2005/2006	Brussels sprouts Mezzo Nano	1	0.500	0.333	plant [a]	0	8.6	0.44	9.0	–	2006/ 1034211 0541R
					sprouts	35	2.2	0.41	2.6	–	
					sprouts	41	2.1	0.33	2.4	–	
					sprouts	49	1.7	0.22	1.9	–	
Netherlands, 2006	Brussels sprouts Lunet	1	0.500	0.333	plant [a]	0	6.2	< 0.04	6.3	–	2007/ 1020722 06NL/061R
					sprouts	35	1.6	0.37	2.0	–	
					sprouts	41	1.2	0.29	1.5	0.52	
					sprouts	50	0.82	0.26	1.1	0.47	
Sweden, 2005	Brussels sprouts Rudius	1	0.500	0.333	plant [a]	0	9.1	0.16	9.3	–	2006/ 1034211 HUS/190503-01
					sprouts	36	1.0	0.14	1.1	–	
					sprouts	42	0.52	0.07	0.59	–	
					sprouts	50	0.75	0.10	0.85	–	
Spain, 2006/2007	Brussels sprouts Sanda	1	0.500	0.333	plant [a]	0	20	0.31	20	–	2007/ 1020722 06ES/057R
					sprouts	35	5.7	0.37	6.0	–	
					sprouts	42	2.3	0.33	2.6	1.15	
					sprouts	50	1.7	0.24	1.9	1.21	
United Kingdom, 2005	Brussels sprouts Abacus	1	0.500	0.333	plant [a]	0	8.1	0.32	8.4	–	2006/ 1034211 754/SPR/1
					sprouts	35	2.4	0.46	2.9	–	
					sprouts	42	1.8	0.53	2.3	–	
					sprouts	49	2.0	0.61	2.6	–	
United Kingdom, 2006	Brussels sprouts Maximus	1	0.500	0.333	plant [a]	0	7.4	0.30	7.7	–	2007/ 1020722 06UK/062R
					sprouts	36	1.5	0.38	1.8	–	
					sprouts	42	1.0	0.34	1.3	0.80	
					sprouts	48	1.3	0.37	1.7	0.74	

[a] Without root

Cabbage

Eighteen residue trials were conducted on cabbages in Europe in 2001 and 2006. Residues were determined with Methods 407/1 or 493/0.The results are shown in Table 74.

Table 74 Results of residue trials conducted with cycloxydim on cabbage head

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Belgium, 2001	Lion F1	1	0.600	0.200	0	2.5	0.16	2.7	–	2002/ 1004111 AGR/21/01
					28	1.1	0.17	1.3	–	
					42	0.75	0.14	0.89	–	
					57	1.14	0.10	1.2	–	
Belgium, 2006	Destinny	1	0.500	0.170	0	0.12	< 0.04	0.16	–	2007/ 1020721 06BE/065R
					20	0.38	0.10	0.48	–	
					28	0.26	0.05	0.31	0.23	
					34	0.40	0.10	0.50	0.26	
France, 2001 (south)	Calidor	1	0.600	0.200	28	0.94	0.33	1.2	–	2002/ 1007718 FTL/28/01
					43	0.55	0.14	0.67	–	
					56	0.38	0.11	0.48	–	
France, 2001 (north)	Atria	1	0.600	0.200	0	< 0.05	< 0.04	< 0.09	–	2002/ 1004111 FBM/11/01
					28	0.46	< 0.04	0.50	–	
					42	0.33	< 0.04	0.37	–	
					56	0.29	< 0.04	0.33	–	
France, 2006 (north)	Atria	1	0.500	0.170	0	0.14	< 0.04	0.18	–	2007/ 1020721 06FR/063R
					21	0.65	0.07	0.72	–	
					28	< 0.05	< 0.04	< 0.09	–	
					35	< 0.05	< 0.04	< 0.09	–	
Germany, 2006	Kalorama	1	0.500	0.170	0	0.17	< 0.04	0.21	–	2007/ 1020721 06GE/064R
					21	1.0	0.07	1.1	–	
					27	0.69	0.05	0.74	0.35	
					35	0.55	0.08	0.63	0.19	
Greece, 2001	Grass Plant	1	0.600	0.200	28	0.99	0.10	1.0	–	2002/ 1007718 HEL/11/01
					42	0.92	0.12	1.0	–	

Country, year	Variety	Application			DAT, days	Residues, mg/kg				Study Trial No.
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	Cy- TSO	
					56	1.25	0.17	1.4	–	
Italy, 2001	Concerto	1	0.600	0.200	27	1.1	0.11	1.2	–	2002/ 1007718
					41	1.6	0.17	1.7	–	ITA/39/01
					55	0.86	0.11	0.96	–	
Spain, 2001	Castello	1	0.600	0.200	28	0.75	0.15	0.88	–	2002/ 1007718
					42	0.52	0.14	0.64	–	ALO/34/01
					55	0.35	0.10	0.44	–	
Spain, 2001	Bronco	1	0.600	0.200	28	0.93	0.14	1.05	–	2002/ 1007718
					42	0.30	0.07	0.36	–	ALO/35/01
					56	0.27	0.07	0.33	–	
Sweden, 2001	Cabbage Carlton	1	0.600	0.200	0	0.36	< 0.04	0.40	–	2002/ 1004111
					28	0.43	0.05	0.48	–	HUS/04/01
					42	0.56	0.07	0.63	–	
					57	0.48	0.07	0.55	–	
United Kingdom, 2001	Cabbage Colt	1	0.600	0.200	0	32	0.46	33	–	2002/ 1004111
					28	0.24	0.16	0.40	–	OAT/05/01
					42	0.10	0.06	0.16	–	
					56	0.12	0.05	0.17	–	
United Kingdom, 2001	Cabbage Savoy	1	0.600	0.200	0	15	0.32	15	–	2002/ 1004111
					29	2.9	0.16	3.0	–	OAT/06/01
					43	2.4	0.10	2.5	–	
					55	2.2	0.10	2.3	–	
United Kingdom, 2006	Cabbage Stonehead	1	0.500	0.170	0	2.7	0.04	2.7	–	2007/ 1020721
					22	3.9	0.07	4.0	–	06UK/066R
					29	0.93	0.11	1.0	0.91	
					35	0.80	0.14	0.94	0.74	

Cauliflower

Fourteen residue trials were conducted on cauliflower in Europe in 2001 and 2006. The results are shown in Table 75.

Table 75 Results of residue trials conducted with cycloxydim on cauliflower

Country, year	Variety	Application			DAT, days	Residues, mg/kg				Study Trial No.
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	Cy-TSO	
Belgium, 2001	Cauliflower Siria	1	0.600	0.200	0	2.4	0.29	2.7	–	2002/ 1004111
					29	1.7	0.15	1.8	–	AGR/20/01
					42	1.7	0.14	1.9	–	
					57	1.5	0.11	1.6	–	
France, 2001 South	Cauliflower Aviso	1	0.600	0.200	0	7.1	0.24	7.3	–	2002/ 1007718
					29	0.86	0.13	0.97	–	FTL/26/01
					42	0.6	0.11	0.7	–	
					57	0.34	0.08	0.41	–	
France, 2001 North	Cauliflower Space Star	1	0.600	0.200	0	4.2	0.23	4.4	–	2002/ 1004111
					28	1.4	0.08	1.5	–	FBM/04/01
					42	0.69	0.05	0.74	–	
					56	0.45	0.06	0.51	–	
France, 2006 North	Cauliflower Space-star	1	0.500	0.170	0*	12	0.41	12	–	2007/ 1020720
					21	3.0	0.23	3.2	–	06FR/067R
					29	1.7	0.15	1.6	1.37	
					35	1.56	0.13	1.7	1.17	
Germany, 2006	Cauliflower Delfino	1	0.500	0.170	0*	3.5	0.04	3.5	–	2007/ 1020720
					21	1.6	0.18	1.8	–	06GE/068R
					28	1.4	0.16	1.5	1.26	
					34	1.2	0.14	1.3	1.09	
Greece, 2001	Cauliflower Siria	1	0.600	0.200	0	3.7	0.45	4.1	–	2002/ 1007718
					28	2.9	0.15	3.0	–	HEL/07/01
					42	2.7	0.37	3.0	–	
					56	2.9	0.22	3.1	–	

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Italy, 2001	Cauliflower Frimon	1	0.600	0.200	0 30 42 56	12 1.75 0.72 0.46	0.13 0.17 0.08 0.06	12 1.9 0.79 0.51	– – – –	2002/ 1007718 ITA/35/01
Sweden, 2006	Cauliflower Fremont	1	0.500	0.170	0* 22 29 36	15 1.3 0.09 0.17	1.0 0.34 < 0.04 0.1	16 1.6 0.13 0.27	– – 0.07 < 0.05	2007/ 1020720 06SE/069R
Spain, 2001	Cauliflower Fremu	1	0.600	0.200	0 28 42 55	5.4 0.6 0.47 0.39	0.09 0.07 0.12 0.1	5.5 0.66 0.57 0.48	– – – –	2002/ 1007718 ALO/47/01
Sweden, 2011	Cauliflower Aviso	1	0.600	0.200	0 29 41	1.8 0.65 2.19	0.15 < 0.04 0.13	2.0 0.69 2.3	– – –	2002/ 1004111 HUS/03/01
United Kingdom, 2001	Cauliflower Thalassa	1	0.600	0.200	0 29 41 55	6.9 0.48 0.31 0.18	0.58 0.11 0.1 0.05	7.4 0.59 0.41 0.23	– – – –	2002/ 1004111 OAT/17/01
United Kingdom, 2006	Cauliflower Freemont	1	0.500	0.170	0* 21 27 35	10 3.4 2.0 1.04	0.19 0.24 0.13 0.10	10 3.6 2.1 1.1	– – 1.5 0.78	2007/ 1020720 06UK/070R

[a] Whole plant

Peppers

Eight residue trials were conducted on peppers in Europe in 1991 and 2005/2006. Residues were determined with Methods 263, 407/1 or 493/0. The results are shown in Table 76.

Table 76 Results of residue trials conducted with cycloxydim on peppers

		Application			DAT,	Residues, mg/kg				Study
Country, year	Variety	No	kg ai/ha	kg ai/hL	days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Trial No.
France, 2006 South	Mariner F1	1	0.600	0.200	0 14 21 28	6.5 1.6 1.6 2.8	< 0.04 0.12 0.14 0.29	6.5 1.8 1.8 3.1	– – 0.62 0.76	2007/ 1020719 06FR/028R
Greece, 2005	Pepper Raiko	1	0.500	0.167	0 14 21 28	18 1.8 1.4 1.2	< 0.04 0.10 0.10 0.07	18 1.9 1.5 1.3	– – – –	2006/ 1025871 05GR/045R
Italy, 1991	Eldo	1	0.600	0.200	0 7 15 30	0.33 0.83 0.63 0.13	< 0.04 < 0.04 0.05 < 0.04	0.37 0.87 0.68 0.17	– – – –	1993/11315 IT6/024/91
Italy, 1991	Quadrato d'Asti	1	0.600	0.200	0 7 15 30	0.26 0.26 3.0 0.66	< 0.04 0.10 0.07 0.09	0.30 0.36 3.0 0.75	– – – –	1993/11315 IT6/025/91
Italy, 2005	Quadrato	1	0.500	0.167	0 14 21 28	8.6 2.1 1.4 1.5	0.04 0.13 0.12 0.15	8.6 2.2 1.5 1.6	– – – –	2006/ 1025871 05IT/044R
Italy, 2006	Corno di toro	1	0.600	0.200	0 14 21 28	47 4.5 4.9 4.3	0.13 0.60 0.46 0.47	47 5.1 5.3 4.7	– – 1.78 1.10	2007/ 1020719 06IT/027R
Spain, 2005	Piquillo del negrillo	1	0.500	0.167	0 14	14 1.4	< 0.04 0.07	14 1.5	– –	2006/ 1025871 05ES/043R

Country, year	Variety	Application No	kg ai/ha	kg ai/hL	DAT, days	Residues, mg/kg Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
					21	1.1	0.10	1.2	–	
					28	0.76	0.10	0.86	–	
Spain, 2006	Tilon	1	0.600	0.200	0	28	< 0.04	28	–	2007/ 1020719
					14	1.4	0.13	1.5	–	06ES/026R
					22	0.69	0.09	0.78	0.50	
					27	0.47	0.07	0.54	0.24	

Tomatoes

Sixteen residue trials were conducted on tomatoes in Europe in 2005/2006. Residues were determined with Methods 407/1 or 493/0. The results are shown in Table 77.

Table 77 Results of residue trials conducted with cycloxydim on tomatoes

Country, year	Variety	Application No	kg ai/ha	kg ai/hL	DAT, days	Residues, mg/kg Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
France, 2005	Felicia	1	0.500	0.167	0	8.5	0.10	8.6	–	2006/ 1024331
(north)					28	0.42	0.10	0.52	–	05FR/062R
					35	0.45	0.14	0.59	–	
					42	0.36	0.10	0.46	–	
France, 2005	Pyros	1	0.500	0.167	0	6.30	0.07	6.37	–	2006/ 1024331
(north)					28	0.36	0.11	0.47	–	05FR/063R
					35	0.40	0.12	0.52	–	
					42	0.34	0.10	0.44	–	
France, 2005	Promo	1	0.500	0.167	35	0.21	0.05	0.26	–	2006/ 1024331
(south)					42	0.16	0.04	0.20	–	05FR/066R
France, 2006	Promo	1	0.500	0.167	0	2.0	< 0.04	2.0	–	2007/ 1020718
(south)					28	0.08	< 0.04	0.12	–	06FR/011R
					35	0.06	< 0.04	0.10	< 0.05	
					42	0.13	< 0.04	0.17	< 0.05	
France, 2006	Pyros	1	0.500	0.167	0	9.9	0.16	10	–	2007/ 1020718
(north)					28	0.94	0.19	1.13	–	06FR/013R
					35	0.65	0.18	0.83	0.19	
					42	0.65	0.19	0.84	0.16	
Germany,	Viper	1	0.500	0.167	0	5.0	< 0.04	5.0	–	2006/ 1024331
2005					28	0.54	0.07	0.61	–	05GE/064R
					35	0.43	0.09	0.52	–	
					42	0.41	0.09	0.50	–	
Germany,	Viper	1	0.500	0.167	0	4.8	< 0.04	4.8	–	2007/ 1020718
2006					28	0.62	0.10	0.72	–	06GE/014R
					35	0.35	0.09	0.44	0.06	
					42	0.36	0.09	0.45	0.09	
Germany,	Viper	1	0.500	0.167	0	12	0.06	12	–	2007/ 1020718
2006					28	0.22	0.04	0.26	–	06GE/015R
					35	0.31	0.08	0.39	0.06	
					42	0.31	0.08	0.39	0.06	
Greece, 2005	Super Gali	1	0.500	0.167	0	8.4	0.31	8.7	–	2006/ 1024331
					28	0.26	0.10	0.36	–	05GR/069R
					35	0.21	0.10	0.31	–	
					43	0.14	0.07	0.21	–	
Greece, 2006	Belladonna	1	0.500	0.167	0	22	0.14	22	–	2007/1020718
					28	0.15	< 0.04	0.19	–	6GR/012R
					36	0.08	< 0.04	0.12	< 0.05	
					43	0.07	< 0.04	0.11	< 0.05	
Italy, 2005	Podium	1	0.500	0.167	0	11	0.07	11	–	2006/ 1024331
					28	0.27	0.05	0.32	–	05IT/068R
					35	0.21	0.04	0.25	–	
					41	0.15	0.04	0.19	–	
Italy, 2006	Galeon	1	0.500	0.167	0	4.6	< 0.04	4.6	–	2007/ 1020718
					28	0.35	0.08	0.43	–	06IT/010R

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
					35	0.35	0.08	0.43	0.11	
					42	0.18	< 0.04	0.22	< 0.05	
Netherlands, 2006	Viper	1	0.500	0.167	0	4.2	0.10	4.3	–	2007/ 1020718 06NL/016R
					28	0.27	0.07	0.34	–	
					35	0.28	0.09	0.37	0.12	
					42	0.29	0.10	0.39	0.10	
Netherlands, 2005	Viper	1	0.500	0.167	0	5.70	< 0.04	5.74	–	2006/ 1024331 05NL/065R
					28	0.22	< 0.04	0.26	–	
					35	0.13	< 0.04	0.17	–	
					42	0.17	< 0.04	0.21	–	
Spain, 2006	Juncal	1	0.500	0.167	0	14	0.13	14	–	2007/ 1020718 06ES/009R
					29	0.47	< 0.04	0.51	–	
					36	0.51	< 0.04	0.55	0.08	
					41	0.30	< 0.04	0.34	< 0.05	
Spain, 2005	Don Benito	1	0.500	0.167	0	22	0.18	23	–	2006/ 1024331 05ES/067R
					27	0.34	0.05	0.39	–	
					36	0.31	0.07	0.38	–	
					41	0.32	0.07	0.39	–	

Kale, curly/ Chinese cabbage

Eight residue trials were conducted on Chinese cabbage in Europe in 2005. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 78.

Table 78 Results of residue trials conducted with cycloxydim on curly/Chinese cabbage and kale

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Greece, 2007	Chinese cabbage Yuki	1	0.500	0.170	0	15.60	1.61	17.21	–	2009/ 1075174 L070373
					35	0.10	< 0.04	0.14	–	
					42	0.19	0.04	0.23	–	
					50	0.08	< 0.04	0.12	–	
Italy, 2007	Chinese cabbage Capoko	1	0.500	0.170	0	14	0.40	15	–	2009/ 1075174 L070372
					35	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					49	< 0.05	< 0.04	< 0.09	–	
France, 2006 (north)	Curly kale Coleor	1	0.500	0.170	0	5.9	0.13	6.1	–	2007/ 1020723 06FR/055R
					35	0.70	0.20	0.90	–	
					42	0.42	0.19	0.61	0.19	
					49	0.42	0.16	0.58	0.20	
France, 2006 (south)	Curly kale Proteor	1	0.500	0.170	0	11	0.10	11	–	2007/ 1020723 06FR/054R
					36	0.73	0.25	0.98	–	
					43	0.68	0.26	0.94	–	
					50	0.21	0.06	0.27	–	
Germany, 2007	Curly kale Winnetou	1	0.500	0.170	0	2.8	< 0.04	2.8	–	2009/ 1075174 L070370
					35	0.64	0.13	0.77	–	
					42	0.73	0.11	0.84	0.60	
					49	0.67	0.10	0.97	0.50	
Netherlands, 2006	Curly kale Reflex	1	0.500	0.170	0	20	0.28	21	–	2007/ 1020723 06NL/056R
					35	1.3	0.35	1.7	–	
					41	1.4	0.41	1.8	0.85	
					49	0.87	0.20	1.1	0.72	
Spain, 2006	Curly kale Veronsa	1	0.500	0.170	0	18	0.18	19	–	2007/ 1020723 06ES/053R
					34	1.0	0.07	1.1	–	
					42	0.79	0.10	0.89	0.35	
					49	0.68	0.09	0.77	0.25	
United Kingdom, 2007	Curly kale Winnetou	1	0.500	0.170	0	0.42	< 0.04	0.46	–	2009/ 1075174 L070371
					36	< 0.05	< 0.04	< 0.09	–	
					42	< 0.05	< 0.04	< 0.09	–	
					50	< 0.05	< 0.04	< 0.09	–	

Lettuce

Twenty two residue trials were conducted on lettuce in Europe from 2001–2008. Residues were determined with Methods 407/1 or 493/0. The results are shown in Table 79.

Table 79 Results of residue trials conducted with cycloxydim on lettuce

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Denmark, 2001	Roxette RZ	1	0.500	0.167	0 6 22	2.1 0.30 0.08	< 0.04 < 0.04 < 0.04	2.1 0.34 0.12	– – –	2002/ 1008792 ALB/05/01
Denmark, 2001	Elinas	1	0.500	0.167	0 7 20	4.8 0.330 0.070	< 0.04 < 0.04 < 0.04	4.8 0.37 0.11	– – –	2002/ 1008792 ALB/06/01
Denmark, 2003	Stallion	1	0.500	0.167	0 7 14 21	9.7 1.0 0.61 0.42	< 0.04 < 0.04 < 0.04 < 0.04	9.8 1.1 0.65 0.46	– – – –	2004/ 1015935 ALB/10/03
France, 2001 (north)	Pantheon	1	0.500	0.167	0 7 21	9.4 1.1 0.30	< 0.04 < 0.04 < 0.04	9.5 1.2 0.34	– – –	2002/ 1008792 FAN/04/01
France, 2001 (north)	Nadine	1	0.500	0.167	0 7 21	4.74 1.34 0.170	< 0.04 < 0.04 < 0.04	4.8 1.4 0.21	– – –	2002/ 1008792 FBM/05/01
France, 2003 (north)	Triathion	1	0.500	0.167	0 6 14 21	10 0.95 0.24 0.13	< 0.04 < 0.04 < 0.04 < 0.04	10 0.99 0.28 0.17	– – – –	2004/ 1015935 FAN/14/03
France, 2003 (north)	Daguan	1	0.500	0.167	0 7 14 20	7.6 1.6 0.65 0.31	< 0.04 < 0.04 < 0.04 < 0.04	7.6 1.6 0.69 0.35	– – – –	2004/ 1015935 FBM/07/03
France, 2005 (centre)	Campionas	1	0.500	0.333	0 14 21	13 0.24 0.22	< 0.04 < 0.04 < 0.04	13 0.28 0.26	– – –	2006/ 1029325 05FR/046R
France, 2005 (north)	Nobellan	1	0.500	0.333	0 14 21 28	12 0.67 0.22 0.08	< 0.04 < 0.04 < 0.04 < 0.04	12 0.71 0.26 0.12	– – – –	2006/ 1029325 05FR/047R
France, 2005 (south)	Lettuce Jordane	1	0.500	0.333	0 14 21 28	14 0.27 0.170 0.09	< 0.04 < 0.04 < 0.04 < 0.04	14 0.31 0.21 0.13	– – – –	2006/ 1029325 05FR/050R
France, 2006 (south)	Lettuce Querido	1	0.500	0.333	0 14 21 28	27 0.20 < 0.05 < 0.05	0.080 < 0.04 < 0.04 < 0.04	27 0.24 < 0.09 < 0.09	– – – –	2007/ 1020724 06FR/051R
Germany, 2005	Ponchito	1	0.500	0.333	0 15 20 27	13 0.07 0.30 0.08	< 0.04 < 0.04 < 0.04 < 0.04	13 0.11 0.34 0.12	– – – –	2006/ 1029325 05GE/048R
Germany, 2005	Estelle	1	0.500	0.333	0 15 20 27	9.3 0.44 0.17 0.08	< 0.04 < 0.04 < 0.04 < 0.04	9.3 0.48 0.21 0.12	– – – –	2006/ 1029325 05GE/049R
Greece, 2005	Lettuce Samson	1	0.500	0.333	0 14 22 28	25 0.96 0.69 0.43	< 0.04 < 0.04 < 0.04 < 0.04	25 1.0 0.73 0.47	– – – –	2006/ 1029325 05GR/052R
Greece, 2006	Lettuce Paris Island	1	0.500	0.333	0 14	15 0.37	< 0.04 < 0.04	15 0.41	– –	2007/ 1020724 06GR/052R

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
	Cos				21 28	0.06 < 0.05	< 0.04 < 0.04	0.10 < 0.09	< 0.05 –	
Italy, 2005	Lettuce Romana	1	0.500	0.333	0 14 21 28	7.9 0.34 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	7.9 0.38 < 0.09 < 0.09	– – – –	2006/ 1029325 05IT/051R
Italy, 2006	Lettuce Gentile	1	0.500	0.333	0 15 21 28	18 0.27 0.08 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	18 0.31 0.12 < 0.09	– – < 0.05 –	2007/ 1020724 06IT/050R
Italy, 2008	Justine open leaf	1	0.500	0.333	0 14 21 28	39 0.05 < 0.05 < 0.05	0.060 < 0.04 < 0.04 < 0.04	39 0.09 < 0.09 < 0.09	– – – –	2009/ 1069374 L080152
Spain, 2008	Francesca open leaf	1	0.500	0.333	0 15 21 28	30 0.07 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	30 0.11 < 0.09 < 0.09	– – – –	2009/ 1069374 L080153
Spain, 2006	Filipus	1	0.500	0.333	0 15 20 28	18 0.32 0.19 0.07	< 0.04 < 0.04 < 0.04 < 0.04	18 0.36 0.23 0.11	– – < 0.05 < 0.05	2007/ 1020724 06ES/049R
Spain, 2005	Filipus	1	0.500	0.333	0 15 22 28	160 0.37 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	16 0.41 < 0.09 < 0.09	– – – –	2006/ 1029325 05ES/053R
Sweden, 2003	Maximus	1	0.500	0.167	0 7 14 21	5.9 0.46 0.14 < 0.05	< 0.04 < 0.04 < 0.04 < 0.04	5.9 0.50 0.18 < 0.09	– – – –	2004/ 1015935 HUS/06/03

Spinach

Eight residue trials were conducted on spinach in Europe in 2005. Residues were determined using Method 407/1. The results are shown in Table 80.

Table 80 Results of residue trials conducted with cycloxydim on spinach

Country, year	Crop Variety	Application			DAT, days	Residues, mg/kg			Study Trial No
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	
France, 2005 (north)	Boing	1	0.500	0.333	0 21 28 35	69 0.07 < 0.05 < 0.05	0.10 0.05 < 0.04 < 0.04	69 0.12 < 0.09 < 0.09	2006/ 1029326 05FR/054R
France, 2005 (north)	Correnta	1	0.500	0.333	0 21 28 35	45.2 0.20 0.08 0.06	0.30 0.10 0.04 < 0.04	46 0.30 0.12 0.10	2006/ 1029326 05FR/055R
France, 2005 (south)	Correnta	1	0.500	0.333	0 21 28 35	70 1.8 2.2 0.87	0.06 0.17 0.23 0.11	70 2.0 2.4 0.98	2006/ 1029326 05FR/058R
Germany, 2005	Cheethu	1	0.500	0.333	0 20 27 34	52 < 0.05 < 0.05 < 0.05	0.17 < 0.04 < 0.04 < 0.04	52 < 0.09 < 0.09 < 0.09	2006/ 1029326 05GE/056R
Germany, 2005	Ventuf	1	0.500	0.333	0 20 27 34	53 0.08 < 0.05 < 0.05	0.12 < 0.04 < 0.04 < 0.04	53 0.12 < 0.09 < 0.09	2006/ 1029326 05GE/057R

Country, year	Crop Variety	Application			DAT, days	Residues, mg/kg			Study Trial No
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	
Greece, 2005	Ikos	1	0.500	0.333	0 20 28 35	27.80 0.15 0.10 0.07	0.15 0.14 0.09 0.06	28 0.29 <u>0.19</u> 0.13	2006/ 1029326 05GR/061R
Italy, 2005	Chica	1	0.500	0.333	0 21 28 35	27 0.08 < 0.05 < 0.05	0.14 < 0.04 < 0.04 < 0.04	27 0.12 <u>< 0.09</u> < 0.09	2006/ 1029326 05IT/060R
Spain, 2005	EPI-9703F1	1	0.500	0.333	0 21 28 34	32 0.27 0.14 < 0.05	0.30 0.10 0.06 < 0.04	32 0.37 <u>0.20</u> < 0.09	2006/ 1029326 05ES/059R

Green beans

Fifteen residue trials were conducted on <u>green beans</u> in Europe from 1988 to 2008. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 81.

Table 81 Results of residue trials conducted with cycloxydim on green beans with pods

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Belgium, 2005	Proton	1	0.500	0.330	21 28 35	0.82 0.30 0.15	0.10 0.10 0.13	0.92 <u>0.40</u> 0.28	– – –	2006/ 1031719 G020-05 H
Belgium, 2008	Cadillac	1	0.520	0.330	29 35	0.18 0.08	< 0.04 0.04	<u>0.22</u> 0.12	0.086 –	2008/ 1067444 L080147
France, 2008 North	Cantare	1	0.500	0.330	21 28 35	0.14 0.18 0.07	0.06 0.12 0.09	0.20 <u>0.30</u> 0.16	– 0.08 –	2008/ 1067444 L080150
France, 2005 North	Albany	1	0.500	0.330	28 36	< 0.05 < 0.05	< 0.04 < 0.04	<u>< 0.09</u> < 0.09	– –	2006/ 1031719 05 H CL FR P11
France, 2005 South	Montayo	1	0.500	0.330	22 28 36	0.52 0.31 0.12	0.04 0.04 0.06	<u>0.56</u> 0.37 0.18	– – –	2006/ 1031719 05 H CL FR P12
France, 2001 South	Big Borlotto	1	0.500	0.167	28 42 57	0.06 0.23 2.8	0.34 0.07 1.6	0.40 0.30 <u>4.4</u>	0.08 0.46 0.38	2003/ 1001265 X0106203
Greece, 1989	–	1	0.500	0.167	30 41 56	0.25 < 0.05 < 0.05	< 0.04 < 0.04 < 0.04	<u>0.29</u> < 0.09 < 0.09	< 0.05 < 0.05 < 0.05	2000/ 1013496 51701H89/ 23E
Greece , 2005	Zargana	1	0.500	0.330	21 28 35	4.3 2.2 0.82	7.1 3.0 0.96	<u>11</u> 5.2 1.8	– – –	2006/ 1031719 05 RF041
Italy, 1988	–	1	0.500	0.125	26	0.17	0.07	<u>0.24</u>	–	2000/ 1013496 51701H88/ 71E
Italy, 1989	–	1	0.500	0.167	40	–	–	<u>< 0.05</u>	–	2000/ 1013496 51701H89/ 23E
Italy, 1990	–	1	0.500	0.167	11 18 24 31	0.30 0.14 0.10 0.10	0.05 < 0.04 < 0.04 0.05	<u>0.35</u> 0.14 0.10 0.15	– – – –	2000/ 1013496 51701H90/ 35A
Italy, 2001	Festival	1	0.500	0.167	27 42 55	0.13 0.05 0.08	0.08 0.05 0.06	<u>0.20</u> 0.10 0.14	< 0.05 < 0.05 < 0.05	2003/ 1001265 0135R
Italy, 2005	Festina	1	0.500	0.330	21 27 35	0.47 0.10 0.05	0.05 0.04 0.05	<u>0.52</u> 0.14 0.10	– – –	2006/ 1031719 0539R
Netherlands, 1990	–	1	0.600	0.180	27	0.60	0.64	<u>1.2</u> [a]	–	2000/ 1013496 51722H90/3E

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Netherlands, 1990	–	1	0.600	0.180	23	0.40	0.34	0.73 [a]	–	2000/ 1013496 51722H90/4E
Netherlands, 2008	Speedy	1	0.500	0.330	21 29 35	0.24 0.18 0.09	0.07 0.08 0.06	0.31 0.26 0.15	– 0.08 –	2008/ 1067444 L080151
Sweeden, 2005	Montayo	1	0.500	0.330	21 28 35	0.93 0.42 0.44	0.84 0.71 0.90	1.8 1.1 1.3	– – –	2006/ 1031719 HUS/190504-1
Spain, 2005	Cilena	1	0.500	0.330	28 36	0.24 0.17	0.17 < 0.04	0.41 0.21	– –	2006/ 1031719 05ES/080R
Spain, 2001	Festival	1	0.500	0.167	26 40 54	0.13 0.06 < 0.05	0.08 < 0.04 < 0.04	0.20 0.10 < 0.09	< 0.05 < 0.05 < 0.05	2003/ 1001265 01S025R
United Kingdom, 2005	Nerina	1	0.500	0.330	21 27 34	0.70 0.42 0.29	0.24 0.22 0.18	0.94 0.64 0.47	– – –	2006/ 1031719 776/GBE/1
United Kingdom, 2008	Torpedo	1	0.530	0.330	29 36	0.16 0.09	0.05 0.05	0.21 0.14	– –	2008/ 1067444 L080149

[a] Mean of four samples

Green peas

Fourteen residue trials were conducted on green peas in Europe in 2006. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 82.

Table 82 Results of residue trials conducted with cycloxydim on green pea seeds

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
France, 2006 North	Cepia	1	0.500	0.250	28 35 41	2.1 1.1 2.6	2.4 1.4 3.4	4.5 2.5 5.9	– 0.71 1.3	2007/ 1020725 06FR/045R
France, 2005 North	Barley	1	0.500	0.250	27 34 42	1.5 1.4 1.7	1.1 1.7 2.7	2.6 3.1 4.4	– – –	2006/ 1034132 05 H CL FR P14
France, 2006 South	Frediro	1	0.500	0.250	28 35 42	2.43 1.23 0.64	0.7 0.7 0.44	3.1 1.9 1.1	– 1.05 0.47	2007/ 1020725 06FR/043R
France, 2005 South	Milan	1	0.500	0.250	29 35 41	1.10 0.61 0.31	1.20 0.78 0.66	2.3 1.4 0.97	– – –	2006/ 1034132 05 H CL FR P13
Germany, 2006	Riger	1	0.500	0.250	28 35 41	2.9 1.1 1.1	0.96 0.51 0.68	3.8 1.6 1.8	– 0.66 0.65	2007/ 1020725 06GE/046R
Greece, 2005	Ambassadeur	1	0.500	0.250	28 35 42	2.2 1.4 1.3	3.7 2.5 2.1	5.9 3.9 3.4	– – –	2006/ 1034132 05RF042
Greece, 2006	Lotus	1	0.500	0.250	28 35 42	0.47 0.43 0.25	0.37 0.37 0.23	0.84 0.80 0.48	– 0.37 0.11	2007/ 1020725 06GR/044R
Italy, 2005	Atlas	1	0.500	0.250	42 49	0.21 0.28	0.14 0.17	0.35 0.45	– –	2006/ 1034132 0546R
Italy, 2006	Budget	1	0.500	0.250	28 35 42	1.41 0.48 0.22	0.69 0.37 0.26	2.1 0.85 0.48	– 0.4 0.19	2007/ 1020725 06IT/042R
Netherlands, 1988	–	1	0.600	0.083	21	2.2	3.7	5.9	–	1988/10697 51701H88/33
Netherlands, 1988	–	1	0.600	0.083	21	1.6	2.7	4.3	–	1988/10697 51701H88/34

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Netherlands, 1988	–	1	0.600	0.083	21	1.9	3.1	5.0	–	1988/10697 51701H88/35
Netherlands, 1988	–	1	0.600	0.083	21	2.1	3.6	5.6	–	1988/10697 51701H88/36
Netherlands, 1988	–	1	0.600	0.083	21	2.0	3.6	5.6	–	1988/10698 51701H88/37
Netherlands, 1988	–	1	0.600	0.083	21	2.3	3.7	6.0	–	1988/10698 51701H88/38
Netherlands, 1988	–	1	0.600	0.083	21	2.0	3.5	5.5	–	1988/10698 51701H88/39
Netherlands, 1988	–	1	0.600	0.083	21	2.2	3.7	6.0	–	1988/10698 51701H88/40
Netherlands, 2005	Arabelle	1	0.500	0.250	28 35 42	0.36 0.21 0.06	0.60 0.59 0.14	0.96 0.80 0.20	– – –	2006/ 1034132 AGR/51/05
Spain, 2005	Jumbo	1	0.500	0.250	34 42	2.3 2.7	4.9 5.8	7.2 8.5	– –	2006/ 1034132 05ES/081 R
Spain, 2006	Lincoln	1	0.500	0.250	28 34 42	2.1 1.4 2.3	2.3 2.1 3.0	4.3 3.5 5.3	– 1.1 1.9	2007/ 1020725 06ES/041R
Sweden, 2005	S7	1	0.500	0.250	28 35 42	2.4 1.5 1.3	4.1 3.2 3.4	6.5 4.7 4.7	– – –	2006/ 1034132 05HUS 1190505-01
United Kingdom, 2005	Onward	1	0.500	0.250	27 34 41	1.9 0.60 0.32	2.0 0.65 0.60	3.9 1.2 0.92	– – –	2006/ 1034132 777/PEA1
UK, 2006	Legacy	1	0.500	0.250	28 35 42	2.1 1.8 0.96	1.0 1.4 0.7	3.2 3.2 1.7	– 1.6 0.81	2007/ 1020725 06UK/048R

Dry beans

Twenty one residue trials were conducted on dry beans in Europe from 2001 to 2006. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 83.

Table 83 Results of residue trials conducted with cycloxydim on dry bean seeds

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Denmark, 2005	Dry beans Vroma	1	0.500	0.330	49 56 63	2.6 3.0 2.7	1.4 1.4 1.2	4.0 4.4 3.9	– – –	2006/ 1024330 ALB/190501-01
France, 2005 North	Dry beans Lingots du Nord	1	0.500	0.330	49 56 63	0.34 0.3 0.14	0.17 0.19 0.06	0.51 0.49 0.2	– – –	2006/ 1024330 05 H CL FR P 10
France, 2005 South	Dry beans Linix	1	0.500	0.330	49 56 63	0.30 0.13 0.39	0.17 0.08 0.31	0.47 0.21 0.70	– – –	2006/ 1024330 05 H CL FR P09
France, 2006 North	Dry beans Irena	1	0.500	0.330	49 57 62	3.4 3.6 3.3	0.81 0.75 0.58	4.2 4.4 3.9	– 1.15 1.98	2007/ 1020731 06FR/086R
France, 2006 South	Dry beans Diva	1	0.500	0.330	49 56 63	3.3 3.1 0.72	0.85 0.9 0.1	4.1 4.0 0.82	– 0.96 0.71	2007/ 1020731 06FR084R
Germany, 2006	Dry beans Danko	1	0.500	0.330	49 56 63	11 9.3 3.2	3.3 3.0 2.7	15 12 6.0	– 6.6 4.2	2007/ 1020731 06GE/087R
Germany, 2006	Dry beans Danko	1	0.500	0.330	49 56 63	7.1 7.0 7.3	2.57 2.54 2.54	9.7 9.5 9.8	– 3.5 2.8	2007/ 1020731 06GE/088R

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Greece, 2002	Dry beans Express	1	0.500	0.330	54 68	0.16 0.14	< 0.04 < 0.04	0.20 0.18	0.12 0.13	2003/ 1001266 02RF003
Greece, 2005	Dry beans Express	1	0.500	0.330	49 56 63	0.28 0.40 0.51	0.04 0.05 0.06	0.32 0.45 0.57	– – –	2006/ 1024330 05/RF040
Greece, 2006	Dry beans super Aguadulce	1	0.500	0.330	48 55 62	1.4 1.5 1.6	0.45 0.42 0.44	1.9 1.9 2.0	– 0.61 1.5	2007/ 1020731 06GR085R
Italy, 2006	Dry beans Listra	1	0.500	0.330	48 56 63	0.73 2.8 2.0	0.41 0.7 0.4	1.1 3.6 2.4	– 1.31 1.5	2007/ 1020731 06IT/083R
Italy, 2001	Dry beans Siconia	1	0.500	0.330	55	0.78	0.30	1.1	0.36	2003/ 1001266 0130R
Italy, 2001	Dry beans Siviglia	1	0.500	0.330	55	4.9	1.14	6.1	1.7	2003/ 1001266 0131R
Italy, 2005	Dry beans Vesuvio	1	0.500	0.330	49 57 63	1.0 1.9 1.9	0.26 0.35 0.44	1.3 2.2 2.4	– – –	2006/ 1024330 0540R
Netherlands, 2006	Dry beans Danko	1	0.500	0.330	49 56 63	12 6.1 4.5	0.34 1.8 1.1	12 7.9 5.6	– 2.9 2.8	2007/ 1020731 06NL/089R
Spain, 2006	Dry beans Aguadulce	1	0.500	0.330	48 56 62	3.5 2.7 2.8	0.91 0.82 0.7	4.4 3.5 3.5	– 0.86 0.76	2007/ 1020731 06ES/082R
Sweden, 2005	Dry beans Vroma	1	0.500	0.330	50 57 64	1.1 1.0 0.38	0.43 0.43 0.17	1.5 1.5 0.55	– – –	2006/ 1024330 HUS/190501-02
Spain, 2001	Dry beans Alameda	1	0.500	0.330	56	2.0	0.95	3.0	1.3 1.1	2003/ 1001266 01S023R
Spain, 2001	Dry beans Corsario	1	0.500	0.330	56	3.0	1.4	4.5	1.4	2003/ 1001266 01S024R
Spain, 2005	Dry beans Luz de Olono	1	0.500	0.330	49 55 62	0.23 0.22 0.09	0.09 0.09 < 0.04	0.32 0.31 0.13	– – –	2006/ 1024330 05ES/079R
United Kingdom, 2005	Dry beans Compass	1	0.500	0.330	49 56 63	2.0 0.97 2.02	0.84 0.38 0.79	2.8 1.4 2.8	– – -	2006/ 1024330 775/DBE/1

Dry Peas

Fourteen residue trials were conducted on dry peas in Europe from 1987 to 2002. Residues were determined using Methods 263, 407/1 or 493/0. The results are shown in Table 84.

Table 84 Results of residue trials conducted with cycloxydim on dry pea seeds

	Application				Residues, mg/kg				
Country, year	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
France, 2001, South	1	0.600	0.300	56	0.45	1.16	1.6	0.3	2003/ 1001251; FBD/17/01
France, 2001, south	1	0.600	0.300	57	0.55	1.11	1.6	–	2003/ 1001252; FBD/13/02
Greece, 2001	1	0.600	0.300	57	0.10	0.20	0.30	–	2003/ 1001251; HEL/10/01
Greece, 2002	1	0.600	0.200	57	2.97	0.28	3.2	–	2003/ 1001263; 02RF025/1
Greece, 2002	1	0.600	0.200	56	2.9	0.28	3.2	0.49	2003/ 1001263; 02RF025/2
Italy, 2001	1	0.600	0.300	56	0.22	0.47	0.69	–	2003/ 1001251; ITA/36/01
Italy, 2002	1	0.600	0.300	56	0.29	0.55	0.84	–	2003/ 1001252; ITA/20/02

	Application				Residues, mg/kg				
Country, year	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Netherlands 1987	1	0.600	0.200	55	0.28	1.04	1.2 [b]	–	2006/ 1009633; 51701H87/66E
Netherlands, 1987	1	0.600	0.200	56	0.8	3.36	3.4 [b]	–	2006/ 1009633; 51701H87/71E
Spain, 2001	1	0.600	0.300	56	2.35	3.58	5.9	1.43	2003/ 1001251; ALO/31/01
Spain, 2002	1	0.600	0.300	56	1.23	2.35	3.6	–	2003/ 1001252; ALO/20/02
Spain, 2002	1	0.600	0.300	55	2.19	2.28	5.5	–	2003/ 1001252; AYE/15/02
United Kingdom, 1989	1	0.45	0.180	56	2.43	5.49	7.9 [a]	–	2006/ 1009633; R517/14/89
United Kingdom, 1989	1	0.450	0.180	56	4.31	7.69	12	–	2006/ 1009633; R517/14/89

[a] Mean of 3 values

[b] Mean of 4 values

Soya beans

Thirteen residue trials were conducted on soya beans in Europe from 2005 to 2007. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 85.

Table 85 Results of residue trials conducted with cycloxydim in soya bean seeds

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
France, 2005 Center	OAC Erin	1	0.50	0.33	49 56 63	19.4 18.4 16.8	10.1 8.96 8.11	30 27 25	– – –	2006/ 1030315 05 H SO FR P05
France, 2005 South	Dekabig	1	0.50	0.33	49 56 64	23.8 18 13.8	15.9 12.4 9.66	40 30 23	– – –	2006/ 1030315 05 H SO FR P04
France, 2006 South	Lanka	1	0.50	0.33	49 56 63	9.98 8.68 8.07	2.78 3.16 2.5	13 12 11	– 3.53 4.44	2007/ 1020732 06FR/079R
Germany, 2005	Dolores	1	0.50	0.33	48 54 62	14.6 11.2 15.9	11.4 10.9 16.8	26 22 33	– – –	2006/ 1030315 AC/05/084
Germany, 2006	Merlin	1	0.50	0.33	49 56 63	0.49 0.47 0.58	0.34 0.36 0.23	0.83 0.83 0.81	– 0.29 0.26	2007/ 1020732 06GE/080R
Greece, 2007	PR92B63-1026	1	0.50	0.33	49 57 64	1.4 1.89 1.62	0.5 0.9 1.03	1.9 2.8 2.6	– 0.66 0.6	2009/ 1075175 L070368
Italy, 2006	Cresir	1	0.50	0.33	49 56 64	26.6 14.78 21.07	3.25 4.55 4.61	30 19 26	– 2.58 1.47	2007/ 1020732 06IT/078R
Italy, 2005	Dekabig	1	0.50	0.33	49 56 63	1.44 2.81 4.72	0.81 2.71 4.49	2.2 5.5 9.2	– – –	2006/ 1030315 0543R
Italy, 2007	Pedro	1	0.50	0.33	49 56 63	7.03 7.56 8.78	3.25 4.96 5.44	10 12 14	– 3.47 3.71	2009/ 1075175 L070367
Netherlands, 2006	Merlin	1	0.50	0.33	49 56 63	0.7 0.55 0.76	0.3 0.46 0.48	1.0 1.0 1.2	– 0.29 0.46	2007/ 1020732 06NL/081R
Spain, 2005	Osumi	1	0.50	0.33	49 56 63	18.9 19.1 17.2	6.46 6.87 8.52	25 26 26	– – –	2006/ 1030315 05ES/076R

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Spain 2006	Osaka	1	0.50	0.33	50 56 63	0.1 0.09 0.14	0.05 0.05 0.09	0.15 0.14 0.23	– 0.07 0.04	2007/ 1020732 06ES/077R
Spain, 2007	Safrane	1	0.50	0.33	48 56 63	2.23 2.39 2.62	0.24 0.44 0.75	2.5 2.8 3.4	– 1.27 1.08	2009/ 1075175 L070369

Carrots

Fifteen residue trials were conducted on carrots in Europe from 2001 to 2007. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 86.

Table 86 Results of residue trials conducted with cycloxydim in carrot roots

		Application				Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Belgium, 2007	Carrots Nerac	1	0.500	0.300	28 35 42	0.39 0.34 0.27	0.05 0.04 0.04	0.44 0.38 0.31	– 0.15 0.15	2009/ 1075173 L070360
France, 2005 South	Carrots Biotek	1	0.500	0.330	28 35 42	0.97 0.76 0.59	0.10 0.09 0.07	1.1 0.85 0.66	– – –	2006/ 1025865 05FR/040R
France, 2007 North	Carrots Maestro	1	0.500	0.300	28 35 42	0.38 0.31 0.29	< 0.04 < 0.04 < 0.04	0.42 0.35 0.33	– 0.11 0.15	2009/ 1075173 L070361
Germany, 2007	Carrots Nebula	1	0.500	0.300	28 38 42	0.60 0.49 0.42	< 0.04 < 0.04 < 0.04	0.64 0.53 0.46	– 0.27 0.23	2009/ 1075173 L070362
Greece, 2005	Carrots Heltas	1	0.500	0.330	28 35 42	2.90 1.10 1.20	0.09 0.08 0.10	3.0 1.2 1.3	– – –	2006/ 1025865 05GR/042R
Greece, 2007	Carrots Tempo F1	1	0.500	0.300	28 35 42	0.24 0.21 0.17	0.05 0.06 0.05	0.29 0.27 0.22	0.07 0.05 –	2009/ 1075173 L070366
Italy, 2005	Carrots Agroblu SRL	1	0.500	0.330	28 35 42	0.36 0.22 0.20	0.11 0.09 0.08	0.47 0.31 0.28	– – –	2006/ 1025865 05IT/041R
Italy, 2007	Carrots Calibra	1	0.500	0.300	28 35 42	0.14 0.10 0.07	< 0.04 < 0.04 < 0.04	0.18 0.14 0.11	– < 0.05 < 0.05	2009/ 1075173 L070364
Netherlands, 2007	Carrots Nerac	1	0.500	0.300	28 35 42	0.28 0.18 0.16	< 0.04 < 0.04 < 0.04	0.32 0.22 0.20	– 0.10 0.08	2009/ 1075173 L070363
Spain, 2005	Carrots Agrologia	1	0.500	0.330	29 36 43	0.26 0.11 0.13	0.07 < 0.04 0.04	0.33 0.15 0.17	– – –	2006/ 1025865 05ES/039R
Spain, 2007	Carrots Nevis F 1	1	0.500	0.300	28 35 42	0.38 0.22 0.24	0.06 < 0.04 0.04	0.44 0.26 0.28	– 0.10 0.10	2009/ 1075173 L070365

Celeriac

Eight residue trials were conducted on celeriac in Europe in 2006 and 2007. Residues were determined using Methods 407/1 or 493/0. The results are shown in Table 87.

Table 87 Results of residue trials conducted with cycloxydim on celeriac

		Application			Residues, mg/kg	

Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
France, 2006 North	Celeriac Prinz	1	0.600	0.200	plants [a]	0	7.10	< 0.04	7.1	–	2007/
					tubers	49	0.10	< 0.04	0.14	–	1020727
					tubers	56	0.09	< 0.04	0.13	0.05	06FR/035R
					tubers	63	0.06	< 0.04	0.10	< 0.05	
					leaves	49	0.11	< 0.04	0.15	–	
					leaves	56	0.08	< 0.04	0.12	< 0.05	
					leaves	63	0.060	< 0.04	0.10	< 0.05	
France, 2007 South	Celeriac Rowena	1	0.600	0.200	plants [a]	0	16.39	< 0.04	16.4	–	2009/
					tubers	49	0.08	0.04	0.12	–	1075172
					tubers	56	0.06	0.07	0.13	< 0.05	L070359
					tubers	63	0.06	0.08	0.14	< 0.05	
					leaves	49	0.21	< 0.04	0.25	–	
					leaves	56	0.18	< 0.04	0.22	< 0.05	
					leaves	63	0.13	< 0.04	0.17	< 0.05	
Germany, 2006	Celeriac Prinz	1	0.600	0.200	plants [a]	0	14.30	< 0.04	14	–	2007/
					tubers	49	0.09	< 0.04	0.13	–	1020727
					tubers	55	0.09	< 0.04	0.13	< 0.05	06GE/036R
					tubers	63	0.05	0.04	0.09	< 0.05	
					leaves	49	0.13	< 0.04	0.17	–	
					leaves	55	0.05	0.08	0.13	< 0.05	
					leaves	63	0.050	0.050	0.10	< 0.05	
Greece, 2007	Celeriac Magnisias	1	0.600	0.200	plants [a]	0	23.38	0.04	23	–	2009/
					tubers	50	0.05	< 0.04	0.09	< 0.05	1075172
					tubers	57	0.05	< 0.04	0.09	< 0.05	L070358
					tubers	64	0.08	0.05	0.13	< 0.05	
					leaves	50	0.05	< 0.04	0.09	< 0.05	
					leaves	57	< 0.05	< 0.04	< 0.09	–	
					leaves	64	0.06	< 0.04	0.09	–	
Italy, 2006	Celeriac Cisko	1	0.600	0.200	plants [a]	0	16.60	< 0.04	17	–	2007/
					tubers	49	0.10	0.09	0.19	–	1020727
					tubers	56	0.07	0.04	0.11	< 0.05	06IT/034R
					tubers	64	0.05	< 0.04	0.09	< 0.05	
					leaves	49	0.15	< 0.04	0.19	–	
					leaves	56	0.06	0.04	0.1	< 0.05	
					leaves	64	0.05	< 0.04	0.09	< 0.05	
Netherlands, 2007	Celeriac Briljanft	1	0.600	0.200	plants [a]	0	14.10	< 0.04	14	–	2009/
					tubers	50	0.24	0.20	0.44	–	1075172
					tubers	56	0.32	0.32	0.64	0.10	L070357
					tubers	64	0.23	0.20	0.43	0.08	
					leaves	50	0.18	0.09	0.27	–	
					leaves	56	0.19	0.13	0.32	0.07	
					leaves	64	0.11	0.10	0.21	0.06	
Spain, 2006	Celeriac Cisko	1	0.600	0.200	plants [a]	0	29.00	0.05	29	–	2007/
					tubers	50	0.05	0.07	0.12	–	1020727
					tubers	55	< 0.05	0.05	0.10	–	06ES/033R
					tubers	63	< 0.05	0.04	0.09	–	
					leaves	50	0.07	< 0.04	0.11	–	
					leaves	55	< 0.05	< 0.04	< 0.09	–	
					leaves	63	0.050	0.040	0.090	< 0.05	
United Kingdom, 2007	Celeriac Kojak	1	0.600	0.200	plants [a]	0	11.60	< 0.04	12	–	2009/
					tubers	50	0.10	0.07	0.17	–	1075172
					tubers	56	0.06	0.04	0.10	< 0.05	L070356
					tubers	63	0.06	0.04	0.10	< 0.05	
					leaves	50	< 0.05	< 0.04	< 0.09	–	
					leaves	56	< 0.05	< 0.04	< 0.09	–	
					leaves	63	< 0.05	< 0.04	< 0.09	–	

[a] Whole plant without roots

Potatoes

Eighteen residue trials were conducted on potatoes in Europe from 1986 and 2007. Residues were determined using Methods 263, 407/1 or 493/0. The results are shown in Table 88.

Table 88 Results of residue trials conducted with cycloxydim on potatoes

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Belgium, 2007	Potato Victoria	1	0.600	0.200	plants [a]	0	11.3	0.09	11	–	2008/ 1090754 AF/11680/BA/3
					tubers	50	0.85	0.39	1.2	–	
					tubers	56	0.75	0.37	1.1	0.44	
					tubers	62	0.76	0.42	1.2	0.51	
Denmark, 2007	Potato Sava	1	0.600	0.200	plants [a]	0	24.8	0.10	25	–	2008/ 1090754 AF/11680/BA/1
					tubers	49	0.22	0.05	0.27	–	
					tubers	56	0.30	0.08	0.38	0.22	
					tubers	63	0.32	0.09	0.41	0.18	
France, 2001 South	Potato Sprinta	1	0.600	0.200	plants [a]	0	31.8	0.71	32	–	2002/ 1005446 FBD/18/01
					tubers	56	< 0.05	< 0.04	< 0.09	–	
					tubers	98	< 0.05	< 0.04	< 0.09	–	
France, 2001 South	Potato Monalisa	1	0.600	0.200	plants [a]	0	11.5	0.07	12	–	2002/ 1005446 FTL/27/01
					tubers	57	0.48	0.15	0.63	–	
					tubers	99	0.36	0.10	0.46	0.22	
France, 2006 North	Potato Mona Lisa	1	0.574	0.200	plants [a]	0	19.0	0.16	19	–	2007/ 1020729 06FR/029R
					tubers	48	0.58	0.07	0.65	–	
					tubers	56	0.62	0.10	0.72	0.47	
					tubers	63	0.19	< 0.04	0.23	0.10	
Germany, 2006	Potato Marabel	1	0.592	0.200	plants [a]	0	19.20	0.16	19	–	2007/ 1020729 06GE/030R
					tubers	49	0.48	0.21	0.69	–	
					tubers	56	0.54	0.11	0.65	0.30	
					tubers	63	0.44	0.08	0.52	0.33	
Germany, 2007	Potato Cilena	1	0.600	0.200	plants [a]	0	24.8	0.10	25	–	2008/ 1090754 AF/11680/BA/2
					tubers	49	1.78	0.40	2.2	–	
					tubers	56	1.21	0.34	1.6	0.51	
					tubers	62	1.12	0.32	1.4	0.38	
Italy, 2007	Potato Romanze	1	0.600	0.200	plants [a]	0	14.8	0.12	15	–	2008/ 1090754 AF/11680/BA/5
					tubers	49	0.19	0.23	0.42	–	
					tubers	56	0.12	0.15	0.27	0.05	
					tubers	63	0.19	0.23	0.42	0.07	
Netherlands, 1986	Potato	1	0.600	0.200	tubers	51	0.30	0.71	1.0 [b]	–	2000/ 1013495 51701H86/65E-68E
Netherlands, 2006	Potato Cilena	1	0.578	0.194	plants [a]	0	16.1	< 0.04	16	–	2007/ 1020729 06NL/031R
					tubers	49	0.88	0.10	0.98	–	
					tubers	55	0.57	0.08	0.65	0.46	
					tubers	63	0.69	0.10	0.79	0.52	
Netherlands, 1987	Potato	1	0.600	0.200	tubers	56	0.21	0.24	0.75 [b]	–	2000/ 1013495 51701H87/74E-77E
Spain, 2001	Potato Spunta	1	0.600	0.200	plants [a]	0	22.5	0.39	23	–	2002/ 1005446 ALO/32/01
					tubers	56	0.06	0.08	0.15	–	
					tubers	84	0.10	0.17	0.27	–	
Spain, 2001	Potato Berbe	1	0.600	0.200	plants [a]	0	35.1	0.53	36	–	2002/ 1005446 ALO/33/01
					tubers	56	0.14	0.26	0.40	–	
					tubers	84	< 0.05	0.05	0.10	–	
Spain, 2007	Potato Romana	1	0.600	0.200	plants [a]	0	15.4	0.17	15	–	2008/ 1090754 AF/11680/BA/6
					tubers	49	0.29	0.23	0.52	–	
					tubers	56	< 0.05	< 0.04	< 0.09	< 0.05	
					tubers	63	0.30	0.33	0.63	0.14	
					tubers	83	0.20	0.24	0.44	0.07	
United Kingdom, 2007	Potato Maris Piper	1	0.600	0.200	plants [a]	0	19.8	0.23	20	–	2008/ 1090754 AF/11680/BA/4
					tubers	49	0.19	0.16	0.35	–	
					tubers	55	0.16	0.15	0.31	0.44	
					tubers	63	0.13	0.07	0.20	0.51	
United Kingdom, 2006	Potato Marquis	1	0.575	0.200	plants [a]	0	7.58	< 0.04	7.6	–	2007/ 1020729 06UK/032R
					tubers	49	0.34	0.06	0.40	–	
					tubers	56	0.31	0.08	0.39	0.16	
					tubers	62	0.48	0.07	0.55	0.10	
Greece, 2001	Potato	1	0.600	0.200	plants [a]	0	21.4	0.27	22	–	2002/ 1005446

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
	Spuda				tubers	56	0.12	0.15	0.27	–	HEL/10/01
					tubers	96	0.11	0.10	0.21	< 0.05	
Italy, 2001	Potato Monalisa	1	0.600	0.200	plants [a]	0	22.1	0.16	22	–	2002/ 1005446
					tubers	57	0.06	< 0.04	0.10	–	ITA/38/01
					tubers	98	< 0.05	< 0.04	< 0.09	–	

[a] Without roots

[b] Mean of four values

Turnips

Six residue trials were conducted on turnips in Europe in 1987 and 1989. Residues were determined using Method 263. The results are shown in Table 89.

Table 89 Results of residue trials conducted with cycloxydim on turnips

Country, year	Variety	Application			Portion	DAT, days	Residues, mg/kg			Study Trial No
		No	kg ai/ha	kg ai/hL			Cy1	Cy2	Total cycloxydim	
Norway, 1987	Bangholm	1	0.600	0.120	roots	91	0.09	< 0.04	0.13	1987/10712 Norway 05.11.010
Norway, 1987	Gry	1	0.600	0.240	roots	103	0.06	< 0.04	0.10	1987/10702 Norway 04.91.013
Norway, 1989	Barke	1	0.600	0.240	roots	77	< 0.05	< 0.04	< 0.09	1993/11354 NO-H-XXA-89
Norway, 1989	Barke	1	0.600	0.240	leaves	77	n.r.	n.r.	0.12 [a]	1993/11354 NO-H-XXC-89
Norway, 1989	Barke	1	0.600	0.240	roots	77	< 0.05	< 0.04	< 0.09	1993/11355 NO-H-XXA-89
Norway, 1989	Barke	1	0.600	0.240	leaves	77	n.r.	n.r.	0.10	1993/11355 NO-H-XXC-89

[a] Residue in untreated control 0.12 mg/kg

Sugar beet

Eighteen residue trials were conducted on sugar beet in Europe in 1988 and 2001. Residues were determined using Methods 263, 407/1 or 493/0. The results are shown in Table 90.

Table 90 Results of residue trials conducted with cycloxydim on sugar beet root

	Application				Residues, mg/kg				
Country, year	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Germany, 1986	1	0.500	0.125	25	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				55	< 0.05	< 0.04	< 0.09	–	51701H86/ 26A
				87	< 0.05	< 0.04	< 0.09	–	
				126	< 0.05	< 0.04	< 0.09	–	
Germany, 1986	1	0.500	0.200	36	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				64	< 0.05	< 0.04	< 0.09	–	51701H86/ 27A
				107	< 0.05	< 0.04	< 0.09	–	
				132	< 0.05	< 0.04	< 0.09	–	
Germany, 1986	1	0.500	0.152	24	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				51	< 0.05	< 0.04	< 0.09	–	51701H86/ 28A
				84	< 0.05	< 0.04	< 0.09	–	
				132	< 0.05	< 0.04	< 0.09	–	
Germany, 1986	1	0.500	0.250	30	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				61	< 0.05	< 0.04	< 0.09	–	51701H86/ 29A
				90	< 0.05	< 0.04	< 0.09	–	
				118	< 0.05	< 0.04	< 0.09	–	

	Application				Residues, mg/kg				
Country, year	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Cy- TSO	Study Trial No.
Germany, 1986	1	0.500	0.125	35	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				64	< 0.05	< 0.04	< 0.09	–	51701H86/ 30A
				107	< 0.05	< 0.04	< 0.09	–	
				129	< 0.05	< 0.04	< 0.09	–	
Germany, 1987	1	0.500	0.250	52	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				71	< 0.05	< 0.04	< 0.09	–	51701H87/ 8A
				100	< 0.05	< 0.04	< 0.09	–	
				140	< 0.05	< 0.04	< 0.09	–	
Germany, 1987	1	0.500	0.125	42	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				72	< 0.05	< 0.04	< 0.09	–	51701H87/ 10A
				112	< 0.05	< 0.04	< 0.09	–	
				142	< 0.05	< 0.04	< 0.09	–	
Germany, 1987	1	0.500	0.250	35	< 0.05	< 0.04	< 0.09	–	2000/ 1013494
				64	< 0.05	< 0.04	< 0.09	–	51701H87/ 11A
				95	< 0.05	< 0.04	< 0.09	–	
				127	< 0.05	< 0.04	< 0.09	–	
Greece, 1993	1	0.500	0.143	76	< 0.05	< 0.04	< 0.09	–	2000/ 1013494; 51701H93/ 1E
Greece, 2001	1	0.593	0.200	55	< 0.05	< 0.04	< 0.09	< 0.05	2003/ 1001264
				97	< 0.05	< 0.04	< 0.09	< 0.05	01RF024/1
Greece, 2001	1	0.629	0.200	56	< 0.05	< 0.04	< 0.09	< 0.05	2003/ 1001264
				99	< 0.05	< 0.04	< 0.09	< 0.05	01RF024/2
Italy, 1987	1	0.600	0.120	100	< 0.05	< 0.04	< 0.09	–	2000/ 1013494; 51701H87/ 116E
Italy, 1987	1	0.600	0.150	84	< 0.05	< 0.04	< 0.09	–	2000/ 1013494; 51701H87/ 118E
Italy, 1988	1	0.500	0.125	105	< 0.05	< 0.04	< 0.09	–	2000/ 1013494; 51701H88/ 68E
Italy, 2001	1	0.563	0.200	56	< 0.05	< 0.04	< 0.09	< 0.05	2003/ 1001264
				96	< 0.05	< 0.04	< 0.09	< 0.05	132R
Italy, 2001	1	0.582	0.200	56	< 0.05	< 0.04	< 0.09	< 0.05	2003/ 1001264
				96	< 0.05	< 0.04	< 0.09	< 0.05	133R
Netherlands, 1987	1	0.600	0.126	58	0.065	< 0.04	0.10 [a]	–	2000/ 1013494 51701H87/33-36E
Netherlands, 1987	1	0.600	0.126	58	< 0.05	< 0.04	< 0.09 [a]	–	2000/ 1013494 51701H87/37-40E

[a] Mean of 4 values

Maize

Eight residue trials were conducted on maize grain in Europe in 1997. Residues were determined using Method 263/1. The results are shown in Table 91.

Table 91 Results of residue trials conducted with cycloxydim on maize grain

Country, year	Application			DAT, mg/kg	Residues, mg/kg			Study Trial number
	No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	
France, North 1995	1	0.400	0.133	132	< 0.05	< 0.04	< 0.09	1997/10456; FR8/07/95
France, North 1995	1	0.400	0.133	100	< 0.05	< 0.04	< 0.09	1997/10456; FR4/05/95
France, North 1996	1	0.400	0.133	122	< 0.05	< 0.04	< 0.09	1997/10518; FR4/02/96
Germany, 1995	1	0.400	0.133	132	< 0.05	< 0.04	< 0.09	1997/10456; D07/03/95
Germany, 1995	1	0.400	0.133	91	< 0.08	< 0.04	0.12	1997/10456; DU2/07/95
Germany, 1995	1	0.400	0.133	135	< 0.05	< 0.04	< 0.09	1997/10456; D07/03/96
Italy, 1995	1	0.400	0.133	100	< 0.05	< 0.04	< 0.09	1997/10417; IT10-95-H368
Italy, 1995	1	0.400	0.133	112	< 0.05	< 0.04	< 0.09	1997/10417; IT10-95-H369
Italy, 1996	1	0.400	0.133	121	< 0.05	< 0.04	< 0.09	1997/10403; IT10-96-R366
Italy, 1996	1	0.400	0.133	125	< 0.05	< 0.04	< 0.09	1997/10403; IT10-96-R367
Spain, 1995	1	0.400	0.133	85	< 0.05	< 0.04	< 0.09	1997/10456; AC/10/95
Spain, 1995	1	0.400	0.133	95	< 0.05	< 0.04	< 0.09	1997/10456; AC/11/95

Spain, 1996	1	0.400	0.133	98	< 0.05	< 0.04	< 0.09	1997/10518;AC/08/96
Spain, 1996	1	0.400	0.133	96	< 0.05	< 0.04	< 0.09	1997/10518; AC/09/93

Rice

Eleven residue trials were conducted on rice in Europe in 1993 to 1996. Residues were determined using Methods 263. The results are shown in Table 92.

Table 92 Results of residue trials conducted with cycloxydim on rice in Italy

		Application				Residues, mg/kg			
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cycloxydim	Cy2	Total cycloxydim	Study Trial No.
Italy, 1996	Loto	1	0.400	0.100	137	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H362
Italy, 1996	Loto	1	0.400	0.100	133	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H363
Italy, 1996	Selenio	1	0.400	0.100	147	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H364
Italy, 1996	Loto	1	0.400	0.100	145	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H365
Italy, 1995	Loto	1	0.300	0.075	136	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H364
Italy, 1995	Koral	1	0.300	0.075	147	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H365
Italy, 1995	Baldo	1	0.300	0.075	142	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H366
Italy, 1995	Cigalon	1	0.300	0.075	143	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H367
Italy, 1993	Senia	1	0.400	0.100	162	< 0.05	< 0.04	< 0.09	1993/11603; ES4-60
Italy, 1993	Tebre	1	0.400	0.100	162	< 0.05	< 0.04	< 0.09	1993/11604; ES4-61
Italy, 1993	Bahia	1	0.400	0.100	162	< 0.05	< 0.04	< 0.09	1993/11605; ES4-62

Rape seed

Eighteen residue trials were conducted on rape seed in Europe in 1987–2008. Residues were determined using Methods 263, 407/1 or 493/0. The results are shown in Table 93.

Table 93 Results of residue trials conducted with cycloxydim on rape seed

Country, year	Variety	Application			DAT, days	Residues, mg/kg				Study Trial No.
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	Cy- TSO	
Belgium, 2008	Monalisa	1	0.600	0.400	90 104	4.63 3.72	0.65 0.74	5.3 4.5	– 1.01	2008/ 1067445 L080119
Germany, 2008	Billy	1	0.600	0.400	93 99	1.75 0.76	0.16 0.13	1.9 0.89	– 0.20	2008/ 1067445 L080115
France, 2001 South		1	0.600	0.200	98	2.35	0.5	2.8	– 1.0	2003/ 1001267 X 01 062 01
France, 2001 South		1	0.600	0.200	98	3.32	0.66	4.0	0.94	2003/ 1001267 X 01 062 02
France, 2002 North		1	0.600	0.200	98	0.59	0.18	0.77	0.48	2003/ 1001253 FTL/05/02
France, 2008 North	Astrid	1	0.600	0.400	100 110	2.14 1.28	0.39 0.15	2.5 1.4	– 0.19	2008/ 1067445 L080117
Italy, 2001		1	0.600	0.200	96	0.386	0.153	0.54	0.071	2003/ 1001267 0134R
Netherlands, 2008	Standard	1	0.600	0.400	85 92	0.89 0.87	0.13 0.11	1.0 0.98	– 0.16	2008/ 1067445 L080116
Spain, 2001		1	0.600	0.200	79	2.48	0.609	3.1	0.629	2003/ 1001267 01S021R
Spain, 2001		1	0.600	0.200	79	2.17	0.599	2.8	0.68	2003/ 1001267 01S022R
Spain, 2002		1	0.600	0.200	98	1.33	0.28	1.6	1.1	2003/ 1001253 ALO/07/02
United Kingdom, 1987		1	0.500	0.250	93	1.76	< 0.04	1.8	– –	2000/ 1013493 51701H87/32A
United Kingdom, 1987		1	0.500	0.250	93	1.51	< 0.04	1.6	– –	2000/ 1013493 51701H87/34A
United		1	0.500	0.250	93	2.15	< 0.04	2.2	–	2000/ 1013493

Country, year	Variety	Application			DAT, days	Residues, mg/kg				Study Trial No.
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	Cy- TSO	
Kingdom, 1987					94 95	1.46 1.90	< 0.04 < 0.04	1.5 1.9	– –	51701H87/36A
United Kingdom, 2008	Castille	1	0.600	0.400	91 110	1.38 1.26	0.11 0.13	1.5 1.4	– 0.16	2008/ 1067445 L080118

Sunflower

Fifteen residue trials were conducted on sunflower seeds in Europe in 2006–2008. Residues were determined using Method 407/1 or 493/0. The results are shown in Table 94.

Table 94 Results of residue trials conducted with cycloxydim on sunflower

Country, year	Variety	Application			Portion analysed	DAT, days	Residues, mg/kg				Study Trial No.
		No	kg ai/ha	kg ai/hL			Cy1	Cy2	Total cycloxydim	Cy-TSO	
Belgium, 2008	Sunflower Marquis	1	0.500	0.333	plant [a] seeds seeds seeds seeds	0 90 101 110 119	37.40 < 0.05 < 0.05 < 0.05 < 0.05	0.14 < 0.04 < 0.04 < 0.04 < 0.04	38 < 0.09 < 0.09 < 0.09 < 0.09	– – – – –	2009/ 1069373 L080746
France, 2002 South	Sunflower PR64A70	1	0.600	0.200	plant [a] plant [a] seeds seeds	0 56 98 105	45.81 0.142 < 0.05 < 0.05	0.211 < 0.04 0.068 0.060	46 0.18 0.12 0.11	– – < 0.05 < 0.05	2004/ 1015930 X 02 062 01
France, 2005 South	Sunflower Caramba	1	0.600	0.400	plant [a] seeds seeds seeds	0 89 101 110	45.40 < 0.05 < 0.05 < 0.05	0.52 < 0.04 < 0.04 < 0.04	46 < 0.09 < 0.09 < 0.09	– – – –	2006/ 1034026 05 H TO FR P02
France, 2006 South	Sunflower Melodie	1	0.600	0.400	plant [a] seeds seeds seeds	0 90 100 110	21.3 0.09 0.09 0.09	0.13 0.23 0.24 0.29	21 0.32 0.33 0.38	– – < 0.05 < 0.05	2006/1020733 06FR/073R
Germany, 2008	Sunflower Kronosol	1	0.500	0.333	plant [a] seeds seeds seeds	0 91 100 109	9.82 0.84 0.81 0.91	< 0.04 1.71 1.55 1.85	9.9 2.6 2.4 2.8	– – 0.13 0.13	2009/ 1069373 L080745
Greece, 2001	Sunflower Turquoise	1	0.600	0.200	plant [a] plant [a] seeds	0 55 98	22.34 2.755 0.704	0.128 0.496 1.048	22 3.2 1.8	– – 0.095	2004/ 1015931 01RF021
Greece, 2002	Sunflower Turquoise	1	0.600	0.200	plant [a] plant [a] seeds	0 54 98	27.54 0.313 < 0.05	0.142 0.056 0.095	28 0.37 0.14	– – < 0.05	2004/ 1015930 02RF028
Greece, 2005	Sunflower Turquoiz	1	0.600	0.400	plant [a] seeds seeds seeds	0 90 100 110	30.00 0.25 0.47 0.42	0.24 0.27 0.47 0.45	30 0.52 0.94 0.87	– – – –	2006/ 1034026 05RF039
Greece, 2006	Sunflower Alhaja	1	0.600	0.400	plant [a] seeds seeds seeds	0 90 99 109	21.8 0.11 0.14 0.14	0.17 0.17 < 0.04 0.23	22 0.28 0.18 0.37	– – < 0.05 < 0.05	2006/ 1020733 06GR/074R
Italy, 1993	Sunflower Isoleic	1	0.600	0.150	plant [a] seeds	0 84	17.70 < 0.05	< 0.04 0.04	18 0.09	– –	1995/10368 IT 10-93-H 364
Italy, 1993	Sunflower Trisun 849	1	0.600	0.150	plant [a] seeds	0 99	26.90 < 0.05	< 0.04 < 0.04	27 < 0.09	– –	1995/10368 IT10-93-H 363
Italy, 2001	Sunflower Moreno	1	0.600	0.200	plant [a] plant [a] seeds	0 56 99	22.86 0.056 < 0.05	0.051 < 0.04 < 0.04	23 0.10 < 0.09	– – < 0.05	2004/ 1015931 0136R
Italy, 2001	Sunflower Floralie	1	0.600	0.200	plant [a] plant [a] seeds	0 56 98	24.98 0.223 0.108	< 0.04 0.076 0.144	25 0.30 0.25	– – < 0.05	2004/ 1015931 0137R
Italy, 2005	Sunflower	1	0.600	0.400	plant [a]	0	23.30	0.09	24	–	2006/ 1034026

Country, year	Variety	Application			Portion analysed	DAT, days	Residues, mg/kg				Study Trial No.
		No	kg ai/ha	kg ai/hL			Cy1	Cy2	Total cycloxydim	Cy-TSO	
	Proleic				seeds	89	0.13	0.37	0.50	–	0537R
					seeds	99	0.10	0.28	0.38	–	
					seeds	109	0.14	0.31	0.45	–	
Italy, 2006	Sunflower Panther	1	0.600	0.400	plant [a]	0	22.0	0.17	22	–	2007/ 1020733
					seeds	91	0.11	0.19	0.30	–	06IT/072R
					seeds	100	0.14	0.24	0.38	0.10	
					seeds	110	0.15	0.24	0.39	0.08	
Netherlands, 2006	Sunflower Sanluca	1	0.600	0.400	plant [a]	0	21.7	0.15	22	–	2007/ 1020733
					seeds	90	0.11	0.17	0.28	–	06NL/075R
					seeds	100	0.13	< 0.04	0.17	0.08	
					seeds	111	0.14	0.23	0.37	< 0.05	
Spain, 2005	Sunflower Latino	1	0.600	0.400	plant [a]	0	34.80	0.17	35	–	2006/ 1034026
					seeds	90	< 0.05	< 0.04	< 0.09	–	05ES/078R
					seeds	98	< 0.05	< 0.04	< 0.09	–	
					seeds	109	< 0.05	< 0.04	< 0.09	–	
Spain, 2006	Sunflower Latino	1	0.600	0.400	plant [a]	0	19.9	0.22	20	–	2007/ 1020733
					seeds	89	0.09	0.17	0.26	–	06ES/071R
					seeds	101	0.11	0.17	0.28	< 0.05	
					seeds	111	0.15	0.20	0.35	< 0.05	
United Kingdom, 2006	Sunflower Blizar	1	0.600	0.400	plant [a]	0	21.3	0.17	22	–	2007/ 1020733
					seeds	91	0.12	0.18	0.30	–	06UK/076R
					seeds	99	0.13	0.23	0.36	< 0.05	
					seeds	108	0.15	0.23	0.38	< 0.05	

[a] Without roots

Animal feed commodities

In some trials reported in the previous tables, commodities for animal consumption were analysed for cycloxydim residues and its metabolites. The results are shown in Tables 95–100.

Table 95 Results of residue trials conducted with cycloxydim on bean vines

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Belgium, 2005	Green bean Proton	1	0.500	0.330	wh. plant	0	20.60	< 0.04	21	–	2006/
					rest plant	21	0.88	0.06	0.94	–	1031719
					rest plant	28	0.93	0.06	0.99	–	G020–05 H
					rest plant	35	0.67	0.05	0.72	–	
Belgium, 2008	Green bean Cadillac	1	0.520	0.330	wh. plant	0	41.5	< 0.04	42	–	2008/
					wh. plant	21	1.99	0.06	2.0	–	1067444
					rest plant	29	0.85	0.05	0.9	–	L080147
					rest plant	35	0.61	0.04	0.65	–	
Denmark, 2005	Dry beans Vroma	1	0.500	0.330	plant	0	12.1	< 0.04	12	–	2006/
					straw	49	2.82	0.4	3.2	–	1024330
					straw	56	1.53	0.29	1.8	–	ALB/190501-
					straw	63	1.24	0.24	1.5	–	01
France, 2001 South	Green bean Big Borlotto	1	0.500	0.167	wh. plant	0	15.50	< 0.04	16	–	2003/
					rest plant	28	1.17	0.05	1.2	–	1001265
					rest plant	42	0.70	0.05	0.75	–	X0106203
					rest plant	57	0.32	0.06	0.39	–	
France, 2005 North	Green bean Albany	1	0.500	0.330	wh. plant	0	42.90	< 0.04	43	–	2006/
					wh. plant	21	0.26	< 0.04	0.30	–	1031719
					rest plant	28	0.23	< 0.04	0.27	–	05 H CL FR
					rest plant	36	0.06	< 0.04	0.10	–	P11
France, 2005 North	Dry beans Lingots du Nord	1	0.500	0.330	plant	0	11	< 0.04	11.0	–	2006/
					straw	49	1.5	1.47	3.0	–	1024330
					straw	56	1.31	2.01	3.3	–	05 H CL FR P
					straw	63	1.26	1.51	2.8	–	10
France, 2005	Dry beans Linix	1	0.500	0.330	plant	0	11.3	< 0.04	11	–	2006/
					straw	49	1.42	2.47	3.9	–	1024330

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
South					straw	56	1.3	2.63	3.9	–	05 H CL FR
					straw	63	0.67	1.47	2.1	–	P09
France, 2006 North	Dry beans Irena	1	0.500	0.330	plant	0	10.02	< 0.04	10	–	2007/
					straw	49	1.36	0.33	1.7	–	1020731
					straw	57	0.35	1.57	3.0	–	06FR/086R
					straw	62		0.1	0.45	–	
France, 2006 South	Dry beans Diva	1	0.500	0.330	plant	0	4.03	< 0.04	4.1	–	2007/
					straw	49	0.76	0.1	0.86	–	1020731
					straw	56	0.79	0.07	0.86	–	06FR084R
					straw	63	0.47	0.05	0.52	–	
France, 2008 North	Green bean Cantare	1	0.500	0.330	wh. plant	0	43.59	< 0.04	44	–	2008/
					wh. plant	21	1.42	0.06	1.5	–	1067444
					rest plant	28	0.93	0.04	0.97	–	L080150
					rest plant	35	0.6	< 0.04	0.64	–	
Greece, 2005	Dry beans Express	1	0.500	0.330	plant	0	24.5	< 0.04	25	–	2006/
					straw	49	0.27	< 0.04	0.31	–	1024330
					straw	56	0.2	< 0.04	0.24	–	05/RF040
					straw	63	1.13	< 0.04	1.2	–	
Greece, 2006	Dry beans super Aguadulce	1	0.500	0.330	plant	0	28.2	< 0.04	28	–	2007/
					straw	48	0.29	< 0.04	0.33	–	1020731
					straw	55	0.29	< 0.04	0.33	–	06GR085R
					straw	62	0.35	< 0.04	0.39	–	
Germany, 2006	Dry beans Danko	1	0.500	0.330	plant	0	20.3	< 0.04	20	–	2007/
					straw	49	3.25	0.41	3.7	–	1020731
					straw	56	3.66	0.79	4.4	–	06GE/087R
					straw	63	4.91	1.1	6.0	–	
Germany, 2006	Dry beans Danko	1	0.500	0.330	plant	0	15.59	< 0.04	16	–	2007/
					straw	49	3.25	1.19	4.4	–	1020731
					straw	56	3.58	1.16	4.7	–	06GE/088R
					straw	63	2.53	0.51	3.0	–	
Greece, 2002	Dry beans Express	1	0.500	0.330	plant	0	17.9	< 0.04	18	–	2003/
					pods w/ seed	28	0.694	< 0.04	0.73	0.784	1001266
					pods w/o seed	54	< 0.05	< 0.04	< 0.09	–	02RF003
					pods w/o seed	68	1.6	0.44	2.0	–	
					rest plant	28	1.18	< 0.04	1.2	–	
					rest plant	54	0.121	< 0.04	0.16	–	
					rest plant	68	0.069	< 0.04	0.11	–	
Italy, 2001	Green bean Festival	1	0.500	0.167	wh. plant	0	19.50	< 0.04	20	–	2003/
					rest plant	27	0.35	< 0.04	0.39	–	1001265
					rest plant	42	0.08	< 0.04	0.12	–	0135R
					rest plant	55	0.19	< 0.04	0.12	–	
Italy, 2005	Green bean Festina	1	0.500	0.330	wh. plant[#]	0	30.90	< 0.04	31	–	2006/
					rest plant[#]	21	0.71	0.06	0.77	–	1031719
					rest plant[#]	27	0.37	< 0.04	0.41	–	0539R
					rest plant[#]	35	0.37	0.04	0.41	–	
Italy, 2001	Dry beans Siconia	1	0.500	0.330	plant	0	92.5	< .04	92	–	2003/
					pods w/ seed	29	0.843	0.077	0.92	0.815	1001266
					pods w/o seed	55	0.676	0.125	0.80	–	0130R
					rest plant	29	0.629	< .04	0.67	–	
					rest plant	55	0.19	< .04	0.23	–	
Italy, 2001	Dry beans Siviglia	1	0.500	0.330	plant	0	237.4	< 0.04	240	–	2003/
					pods w/ seed	28	0.871	0.189	1.1	0.952	1001266
					pods w/o seed	55	4.12	0.448	4.6	–	0131R
					rest plant	28	0.548	0.049	0.60	–	
					rest plant	55	0.62	0.093	0.71	–	
Italy, 2005	Dry beans Vesuvio	1	0.500	0.330	plant	0	11.7	< 0.04	12	–	2006/
					straw	49	0.18	0.05	0.23	–	1024330
					straw	57	0.39	0.09	0.48	–	0540R
					straw	63	0.23	0.05	0.28		
Italy, 2006	Dry beans Listra	1	0.500	0.330	plant	0	9.12	< 0.04	9.2	–	2007/
					straw	48	2.79	0.58	3.4	–	1020731
					straw	56	3.52	0.61	4.1	–	06IT/083R

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
					straw	63	2.49	0.26	2.8	–	
Netherlands, 2006	Dry beans Danko	1	0.500	0.330	plant	0	15.68	< 0.04	16	–	2007/
					straw	49	8.94	0.69	9.6	–	1020731
					straw	56	7.64	0.23	7.8	–	06NL/089R
					straw	63	4.58	1.53	6.1	–	
Netherlands, 2008	Green bean Speedy	1	0.500	0.330	wh. plant	0	19.95	0.05	20	–	2008/
					wh. plant	21	0.75	0.07	0.82	–	1067444
					rest plant	29	0.75	0.09	0.84	–	L080151
					rest plant	35	0.46	0.05	0.51	–	
Spain, 2001	Green bean Festival	1	0.500	0.167	wh. plant	0	34.90	< 0.04	35	–	2003/
					rest plant	26	0.39	< 0.04	0.43	–	1001265
					rest plant	40	0.18	< 0.04	0.22	–	01S025R
					rest plant	54	0.14	< 0.04	0.18	–	
Spain, 2001	Dry beans Alameda	1	0.500	0.330	plant	0	5.73	< 0.04	5.8	–	2003/
					pods w/ seed	28	1.16	0.315	1.5	1.32	1001266
					pods w/o seed	56	1.59	0.552	2.1	–	01S023R
					rest plant	28	1.27	0.065	1.3	–	
					rest plant	56	0.633	< 0.04	0.67	–	
Spain, 2001	Dry beans Corsario	1	0.500	0.330	plant	0	127.4	< 0.04	130	–	2003/
					pods w/ seed	28	0.856	0.238	1.1	1.01	1001266
					pods w/o seed	56	2.07	0.702	2.8	–	01S024R
					rest plant	28	1.97	0.059	2.0	–	
					rest plant	56	0.883	0.187	1.1	–	
Spain, 2005	Dry beans Luz de Olono	1	0.500	0.330	plant	0	20.6	< 0.04	21	–	2006/
					straw	49	0.11	< 0.04	0.15		1024330
					straw	55	0.06	< 0.04	0.10	–	05ES/079R
					straw	62	0.06	< 0.04	0.10	–	
Spain, 2006	Dry beans Aguadulce	1	0.500	0.330	plant	0	15.34	< 0.04	15	–	2007/
					straw	48	2.58	0.35	3.0	–	1020731
					straw	56	0.95	0.14	1.1	–	06ES/082R
					straw	62	1.28	0.1	1.4	–	
Sweden, 2005	Dry beans Vroma	1	0.500	0.330	plant	0	6.29	< 0.04	6.3	–	2006/
					straw	50	0.17	< 0.04	0.21	–	1024330
					straw	57	0.24	< 0.04	0.28	–	HUS/190501-
					straw	64	0.17	< 0.04	0.21	–	02
United Kingdom, 2005	Green bean Nerina	1	0.500	0.330	wh. plant	0	29.70	0.08	30	–	2006/
					rest plant	21	0.99	0.20	1.2	–	1031719
					rest plant	27	0.60	0.15	0.75	–	776/GBE/1
					rest plant	34	0.58	0.14	0.72	–	
United Kingdom, 2005	Dry beans Compass	1	0.500	0.330	plant	0	9.96	< 0.04	10	–	2006/
					straw	49	0.45	0.1	0.55	–	1024330
					straw	56	1.16	0.12	1.3	–	775/DBE/1
					straw	63	0.31	0.06	0.37	–	
United Kingdom, 2008	Green bean Torpedo	1	0.530	0.330	wh. plant	0	29.92	< 0.04	30	–	2008/
					wh. plant	22	1.33	< 0.04	1.4	–	1067444
					rest plant	29	0.6	< 0.04	0.64	0.66	L080149
					rest plant	36	0.37	< 0.04	0.41	0.65	

Table 96 Results of residue trials conducted with cycloxydim on peas

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
France, 2001 South		1	0.600	0.300	plant	0	15.33	< 0.04	15	–	2003/
					plant w/o pod	35	0.79	0.08	0.87	–	1001251
					[a]	35	0.69	0.75	1.4	–	FBD/17/01
					pods w. seed	56	0.21	0.05	0.26	–	
					pods w/o seed	56	1.49	0.3	1.8		
					rest of plant						
France, 2001 North		1	0.600	0.300	plant	0	14.16	< 0.04	14	–	2003/
					plant w/o pod	35	3.02	0.07	3.1	–	1001252

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
					pods w. seed	35	0.53	1.2	1.7	–	FBD/13/02
					pods w/o seed	57	0.55	0.39	0.94	–	
					rest of plant	57	2.87	0.65	3.5	–	
France, 2005 South	Green pea Milan	1	0.500	0.250	plant [b]	0	15.60	< 0.04	16	–	2006/
					rest plant [b]	29	1.20	1.10	2.3	–	1034132
					rest plant [b]	35	0.58	0.85	1.4	–	05 H CL FR
					rest plant [b]	41	0.31	0.69	1.0	–	P13
France, 2005 North	Green pea Barley	1	0.500	0.250	plant [b]	0	13.60	< 0.04	14	–	2006/
					rest plant [b]	27	2.3	0.16	2.5	–	1034132
					rest plant [b]	34	1.5	0.10	1.6	–	05 H CL FR
					rest plant [b]	42	0.99	0.36	1.4	–	P14
France, 2006 North	Green pea Cepia	1	0.500	0.250	plant [b]	0	11.68	< 0.04	12	–	2007/
					rest plant [b]	28	0.9	0.17	1.1	–	1020725
					rest plant [b]	35	0.9	0.32	1.2	–	06FR/045R
					rest plant [b]	41	2.14	0.47	2.6	–	
Germany, 2006	Green pea Riger	1	0.500	0.250	plant [b]	0	15.81	< 0.04	16	–	2007/
					rest plant [b]	28	0.71	0.09	0.80	–	1020725
					rest plant [b]	35	0.56	0.07	0.63	–	06GE/046R
					rest plant [b]	41	0.53	0.1	0.63	–	
France, 2006 South	Green pea Frediro	1	0.500	0.250	plant [b]	0	11.07	< 0.04	11	–	2007/
					rest plant [b]	28	1.11	< 0.04	1.1	–	1020725
					rest plant [b]	35	0.93	< 0.04	0.97	–	06FR/043R
					rest plant [b]	42	0.44	< 0.04	0.48	–	
Greece, 2002		1	0.600	0.200	plant	0	14.28	< 0.04	14	–	2003/
					plant w/o pod	34	0.46	< 0.04	0.50	–	1001263
					pods w. seed	34	0.33	0.48	0.81	0.55	02RF025/1
					pods w/o seed	57	0.87	0.67	1.5	–	
					rest of plant	57	0.2	< 0.04	0.24	–	
Greece, 2002		1	0.600	0.200	plant	0	6.58	< 0.04	6.6	–	2003/
					plant w/o pod	36	1.34	0.1	1.4	–	1001263
					pods w. seed	36	0.34	0.49	0.82	–	02RF025/2
					pods w/o seed	56	1.11	0.87	2.0	–	
					rest of plant	56	0.17	< 0.04	0.21	–	
Greece, 2005	Green pea Ambassadeur	1	0.500	0.250	plant [b]	0	17.10	< 0.04	17	–	2006/
					rest plant [b]	28	2.20	3.70	5.9	–	1034132
					rest plant [b]	35	1.40	2.60	4.0	–	05RF042
					rest plant [b]	42	1.30	2.20	3.5	–	
Greece, 2006	Green pea Lotus	1	0.500	0.250	plant [b]	0	24.37	< 0.04	24	–	2007/
					rest plant [b]	28	0.05	< 0.04	0.09	–	1020725
					rest plant [b]	35	0.09	< 0.04	0.13	–	06GR/044R
					rest plant [b]	42	0.1	< 0.04	0.14	–	
Greece, 2001		1	0.600	0.300	plant	0	17.44	< 0.04	17	–	2003/
					plant w/o pod	35	0.47	< 0.04	0.51	–	1001251
					pods w. seed	35	0.44	< 0.04	0.48	–	HEL/10/01
					pods w/o seed	57	1.05	0.96	2.0	–	
					rest of plant	57	0.12	< 0.04	0.16	–	
Italy, 2001		1	0.600	0.300	plant	0	14.21	< 0.04	14	–	2003/
					plant w/o pod	34	0.35	< 0.04	0.39	–	1001251
					pods w. seed	34	0.39	0.23	0.62	–	ITA/36/01
					pods w/o seed	56	2.08	1.41	3.5	–	
					rest of plant	56	2.06	0.12	2.2	–	
Italy, 2002		1	0.600	0.300	plant	0	14.77	< 0.04	15	–	2003/
					plant w/o pod	35	0.47	< 0.04	0.51	–	1001252
					pods w. seed	35	0.3	0.45	0.75	–	ITA/20/02
					pods w/o seed	56	0.47	0.25	0.72	–	
					rest of plant	56	2.03	0.06	2.1	–	
Italy, 2005	Green pea Atlas	1	0.500	0.250	plant [b]	0	17.30	< 0.04	17	–	2006/
					plant [b]	28	0.14	< 0.04	0.18	–	1034132
					pods	35	0.23	< 0.04	0.27	–	0546R
					rest plant [b]	35	0.22	< 0.04	0.26	–	
					rest plant [b]	42	0.20	0.14	0.34	–	
					rest plant [b]	49	0.27	0.18	0.45	–	

		Application					Residues, mg/kg				
Country, year	Variety	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Cy-TSO	Study Trial No.
Italy, 2006	Green pea Budget	1	0.500	0.250	plant [b]	0	9.43	< 0.04	9.5	–	2007/
					rest plant [b]	28	0.23	< 0.04	0.27	–	1020725
					rest plant [b]	35	0.18	0.04	0.22	–	06IT/042R
					rest plant [b]	42	0.08	< 0.04	0.12	–	
Netherlands, 2005	Green pea Arabelle	1	0.50	0.250	plant [b]	0	27.50	< 0.04	28	–	2006/
					rest plant [b]	28	0.35	0.57	0.92	–	1034132
					rest plant [b]	35	0.15	0.38	0.53	–	AGR/51/05
					rest plant [b]	42	0.06	0.14	0.20	–	
Spain, 2006	Green pea Lincoln	1	0.50	0.250	plant [b]	0	20.47	< 0.04	20	–	2007/
					rest plant [b]	28	1.32	0.29	1.6	–	1020725
					rest plant [b]	34	3.66	0.34	4.0	–	06ES/041R
					rest plant [b]	42	3.91	1.55	5.5	–	
Spain, 2005	Green pea Jumbo	1	0.50	0.250	plant [b]	0	10.40	< 0.04	10	–	2006/
					pod w. seed	28	2.60	0.82	3.4	–	1034132
					rest plant [b]	28	2.70	0.79	3.5	–	05ES/081 R
					rest plant [b]	34	2.30	5.00	7.3	–	
					rest plant [b]	42	2.60	5.80	8.4	–	
Spain, 2001		1	0.60	0.300	plant	0	8.91	< 0.04	9.0	–	2003/
					plant w/o pod	35	3.55	0.44	4.0	–	1001251
					pods w. seed	35	2.82	4.83	7.6	–	ALO/31/01
					pods w/o seed	56	1.28	0.92	2.2	–	
					rest of plant	56	1.5	0.28	1.8	–	
Spain, 2002		1	0.60	0.300	plant	0	16.88	< 0.04	17	–	2003/
					plant w/o pod	36	1.17	0.08	1.3	–	1001252
					pods w. seed	36	1.77	1.97	3.7	–	ALO/20/02
					pods w/o seed	56	1.89	1.59	3.5	–	
					rest of plant	56	5.14	0.92	6.1	–	
Spain, 2002		1	0.60	0.300	plant	0	11.38	< 0.04	11	–	2003/
					plant w/o pod	34	6.63	0.69	7.3	–	1001252
					pods w. seed	34	1.63	4.04	5.7	–	AYE/15/02
					pods w/o seed	55	3.44	0.62	4.1	–	
					rest of plant	55	8.18	0.78	9.0	–	
Sweden, 2005	Green pea S7	1	0.50	0.250	plant [b]	0	16.10	< 0.04	16	–	2006/
					rest plant [b]	28	3.40	0.21	3.6	–	1034132
					rest plant [b]	35	2.30	0.10	2.4	–	05HUS
					rest plant [b]	42	1.80	0.08	1.9	–	1190505-01
United Kingdom, 2005	Green pea Onward	1	0.50	0.250	plant [b]	0	18.00	< 0.04	18	–	2006/
					rest plant [b]	27	1.90	2.00	3.9	–	1034132
					rest plant [b]	34	0.54	0.68	1.2	–	777/PEA1
					rest plant [b]	41	0.32	0.58	0.90	–	
United Kingdom, 2006	Green pea Legacy	1	0.50	0.250	plant [b]	0	12.05	< 0.04	12	–	2007/
					rest plant [b]	28	1.5	0.09	1.6	–	1020725
					rest plant [b]	35	2.27	0.15	2.4	–	06UK/048R
					rest plant [b]	42	0.29	0.09	0.38	–	

[a] Without pods

[b] Without roots

Table 97 Results of residue trials conducted with cycloxydim on sugar beet tops

	Application					Residues, mg/kg			
Country, year	No	kg ai/ha	kg ai/hL	Portion analysed	DAT, days	Cy1	Cy2	Total cycloxydim	Study Trial No.
Germany, 1986	1	0.500	0.125	Plants w/o root	0	17.8	< 0.04	18	2000/ 1013494
				leaf	25	< 0.05	< 0.04	< 0.09	51701H86/ 26A
				leaf	55	< 0.05	< 0.04	< 0.09	
				leaf	87	< 0.05	< 0.04	< 0.09	
				leaf	126	< 0.05	< 0.04	< 0.09	
Germany, 1986	1	0.500	0.200	Plants w/o root	0	18	< 0.04	18.	2000/ 1013494
				leaf	36	< 0.05	< 0.04	< 0.09	51701H86/ 27A
				leaf	64	< 0.05	< 0.04	< 0.09	

Country, year	Application			Portion analysed	DAT, days	Residues, mg/kg			Study Trial No.
	No	kg ai/ha	kg ai/hL			Cy1	Cy2	Total cycloxydim	
				leaf	107	< 0.05	< 0.04	< 0.09	
				leaf	132	< 0.05	< 0.04	< 0.09	
Germany, 1986	1	0.500	0.152	Plants w/o root	1	7.46	< 0.04	7.5	2000/ 1013494
				leaf	24	0.12	< 0.04	0.16	51701H86/ 28A
				leaf	51	< 0.05	< 0.04	< 0.09	
				leaf	84	< 0.05	< 0.04	< 0.09	
				leaf	132	< 0.05	< 0.04	< 0.09	
Germany, 1986	1	0.500	0.250	Plants w/o root	0	11.1	< 0.04	11	2000/ 1013494
				leaf	30	0.05	< 0.04	0.09	51701H86/ 29A
				leaf	61	< 0.05	< 0.04	< 0.09	
				leaf	90	< 0.05	< 0.04	< 0.09	
				leaf	118	< 0.05	< 0.04	< 0.09	
Germany, 1986	1	0.500	0.125	Plants w/o root	0	13.6	< 0.04	14	2000/ 1013494
				leaf	35	< 0.05	< 0.04	< 0.09	51701H86/ 30A
				leaf	64	< 0.05	< 0.04	< 0.09	
				leaf	107	< 0.05	< 0.04	< 0.09	
				leaf	129	< 0.05	< 0.04	< 0.09	
Germany, 1987	1	0.500	0.250	Plants w/o root	0	21.6	< 0.04	22	2000/ 1013494
				leaf	52	< 0.05	< 0.04	< 0.09	51701H87/ 8A
				leaf	71	< 0.05	< 0.04	< 0.09	
				leaf	100	< 0.05	< 0.04	< 0.09	
				leaf	140	< 0.05	< 0.04	< 0.09	
Germany, 1987	1	0.500	0.125	Plants w/o root	0	14.8	< 0.04	15	2000/ 1013494
				leaf	42	< 0.05	< 0.04	< 0.09	51701H87/ 10A
				leaf	72	< 0.05	< 0.04	< 0.09	
				leaf	112	< 0.05	< 0.04	< 0.09	
				leaf	142	< 0.05	< 0.04	< 0.09	
Germany, 1987	1	0.500	0.250	Plants w/o root	0	8.99	< 0.04	9.0	2000/ 1013494
				leaf	35	< 0.05	< 0.04	< 0.09	51701H87/ 11A
				leaf	64	< 0.05	< 0.04	< 0.09	
				leaf	95	< 0.05	< 0.04	< 0.09	
				leaf	127	< 0.05	< 0.04	< 0.09	
Greece, 2001	1	0.593	0.200	Plants w/o root	0	7.53	< 0.04	7.6	2003/ 1001264
				leaf w. top	55	:< 0.05	< 0.04	< 0.09	01RF024/1
				leaf w. top	97	< 0.05	< 0.04	< 0.09	
Greece, 2001	1	0.629	0.200	Plants w/o root	0	5.91	< 0.04	6.0	2003/ 1001264
				leaf w. top	56	0.057	< 0.04	0.10	01RF024/2
				leaf w. top	99	< 0.05	< 0.04	< 0.09	
Italy, 1987	1	0.600	0.120	leaf	100	< 0.05	< 0.04	< 0.09	2000/ 1013494 51701H87/ 116E
Italy, 1987	1	0.600	0.150	leaf	84	< 0.05	< 0.04	< 0.09	2000/ 1013494 51701H87/ 118E
Italy, 2001	1	0.563	0.200	Plants w/o root	0	10.3	< 0.04	10	2003/ 1001264
				leaf w. top	56	0.133	0.049	0.18	132R
				leaf w. top	96	< 0.05	< 0.04	< 0.09	
Italy, 2001	1	0.582	0.200	Plants w/o root	0	11.7	< 0.04	12	2003/ 1001264
				leaf w. top	56	0.155	0.053	0.21	133R
				leaf w. top	96	< 0.05	< 0.04	< 0.09	
Netherlands, 1987	1	0.600	0.126	leaf	58	n.r.	n.r.	0.50	2000/ 1013494 51701H86/ 26A
Netherlands, 1987	1	0.600	0.126	leaf	58	n.r.	n.r.	0.33	2000/ 1013494 51701H86/ 26A

Table 98 Results of residue trials conducted with cycloxydim on maize fodder

Country, year	Application			Portion analysed	DAT, mg/kg	Residues, mg/kg			Study Trial number
	No	kg ai/ha	kg ai/hL			Cy1	Cy2	Total cycloxydim	
Germany, 1995	1	0.400	0.133	Plants w/o roots	0	13.6	n.e.	14	1997/10456
				Plants w/o roots	35	< 0.05	< 0.04	< 0.09	D07/03/95
				cobs [a]	119	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	119	< 0.05	< 0.04	< 0.09	
				straw	132	< 0.05	< 0.04	< 0.09	
Germany, 1995	1	0.400	0.133	Plants w/o roots	0	8.74	n.e.	8.7	1997/10456
				Plants w/o roots	22	0.20	< 0.04	0.24	D08/04/95
				cobs w. husk	83	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	83	0.074	< 0.04	0.11	
Germany, 1995	1	0.400	0.133	Plants w/o roots	0	3.53	0.74	4.3	1997/10456
				Plants w/o roots	10	0.77	< 0.04	0.81	DU2/07/95
				cobs w. husk	63	0.06	< 0.04	0.10	
				rest pl. w/o roots	63	0.32	0.09	0.41	
				cob wo/ husk	91	0.08	< 0.04	0.12	
				straw	91	0.22	< 0.04	0.26	
France, 1995 North	1	0.400	0.133	Plants w/o roots	0	16.9	0.05	17	1997/10456
				Plants w/o roots	26	0.152	< 0.04	0.19	FR2/05/95
				cobs w.husk	84	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	84	< 0.05	< 0.04	< 0.09	
France, 1995 North	1	0.400	0.133	Plants w/o roots	0	12.7	< 0.04	13	1997/10456
				Plants w/o roots	14	0.37	< 0.04	0.41	FR4/05/95
				cobs w.husk	62	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	62	0.26	< 0.04	0.30	
				straw	100	0.23	< 0.04	0.27	
Germany, 1996	1	0.400	0.133	Plants w/o roots	0	12.0	< 0.04	12	1997/10518
				Plants w/o roots	48	< 0.05	< 0.04	< 0.09	D07/03/96
				cobs w.husk	104	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	104	< 0.05	< 0.04	< 0.09	
				cob w/o husk	135	< 0.05	< 0.04	< 0.09	
				straw	135	< 0.05	< 0.04	< 0.09	
Germany, 1996	1	0.400	0.133	Plants w/o roots	0	20.2	< 0.04	20	1997/10518
				Plants w/o roots	43	0.13	< 0.04	0.17	D08/02/96
				cobs	128	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	128	< 0.05	< 0.04	< 0.09	
France, 1996 North	1	0.400	0.133	Plants w/o roots	0	13.9	< 0.04	14	1997/10518
				Plants w/o roots	31	< 0.05	< 0.04	< 0.09	FR2/02/96
				cobs w. husk	106	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	106	< 0.05	< 0.04	< 0.09	
France, 1996 North	1	0.400	0.133	Plants w/o roots	0	19.0	< 0.04	19	1997/10518
				Plants w/o roots	21	0.26	< 0.04	0.30	FR4/02/96
				cobs w/husk	98	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	98	< 0.05	< 0.04	< 0.09	
				cob wo/ husk	122	< 0.05	< 0.04	< 0.09	
				straw	122	< 0.05	< 0.04	< 0.09	
Spain, 1995	1	0.400	0.133	Plants w/o roots	0	7.10	< 0.04	7.1	1997/10456
				Plants w/o roots	24	0.153	< 0.04	0.19	AC/10/95
				cobs w. husk	51	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	51	0.085	< 0.04	0.13	
				straw	85	1.05	0.06	1.1	
Spain, 1995	1	0.400	0.133	Plants w/o roots	0	14.0	< 0.04	14	1997/10456
				Plants w/o roots	34	< 0.05	< 0.04	< 0.09	AC/11/95
				cobs w. husk	55	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	55	< 0.05	< 0.04	< 0.09	
				straw	95	0.06	< 0.04	0.10	
France, 1995 North	1	0.400	0.133	Plants w/o roots	0	11.5	< 0.04	12	1997/10456
				Plants w/o roots	29	0.053	< 0.04	0.093	FR8/07/95
				cobs w/husk	83	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	83	< 0.05	< 0.04	< 0.09	
				cob w/o husk	132	< 0.05	< 0.04	< 0.09	
				straw	132	< 0.05	< 0.04	< 0.09	
Italy, 1995	1	0.400	0.133	Plants w/o roots	0	16.0	0.06	16	1997/10417

Country, year	Application			Portion analysed	DAT, mg/kg	Residues, mg/kg			Study Trial number
	No	kg ai/ha	kg ai/hL			Cy1	Cy2	Total cycloxydim	
				Plants w/o roots	71	< 0.05	< 0.04	< 0.09	IT10-95-H368
Italy, 1995	1	0.400	0.133	Plants w/o roots	0	18.6	< 0.04	19	1997/10417
				Plants w/o roots	70	< 0.05	< 0.04	< 0.09	IT10-95-H369
Spain, 1996	1	0.400	0.133	Plants w/o roots	0	16.2	< 0.04	16	1997/10518
				Plants w/o roots	22	0.057	< 0.04	0.06	AC/08/96
				cobs w/husk	50	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	50	0.085	< 0.04	0.12	
				cob w/o husk	98	< 0.05	< 0.04	< 0.09	
				straw	98	0.22	0.072	0.29	
Spain, 1996	1	0.400	0.133	Plants w/o roots	0	16.9	< 0.04	17	1997/10518
				Plants w/o roots	21	0.72	< 0.04	0.76	AC/09/96
				cobs w/husk	48	< 0.05	< 0.04	< 0.09	
				rest pl. w/o roots	48	0.089	< 0.04	0.13	
				cob w/o husk	96	< 0.05	< 0.04	< 0.09	
				straw	96	0.074	< 0.04	0.11	
Italy, 1996	1	0.400	0.133	Plants w/o roots	0	18.9	0.10	19	1997/10403
				Plants w/o roots	85	< 0.05	< 0.04	< 0.09	IT10-96-R366
Italy, 1996	1	0.400	0.133	Plants w/o roots	0	10.6	< 0.04	11	1997/10403
				Plants w/o roots	91	< 0.05	< 0.04	< 0.09	IT10-96-R367

Table 99 Results of residue trials conducted with cycloxydim in rice straw

		Application				Residues, mg/kg			
Country, year	Variety	No	kg ai/ha	kg ai/hL	DAT, days	Cy1	Cy2	Total cycloxydim	Study Trial No.
Italy, 1996	Loto	1	0.400	0.100	137	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H362
Italy, 1996	Loto	1	0.400	0.100	133	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H363
Italy, 1996	Selenio	1	0.400	0.100	147	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H364
Italy, 1996	Loto	1	0.400	0.100	145	< 0.05	< 0.04	< 0.09	1997/10302; IT10-96-H365
Italy, 1995	Loto	1	0.300	0.075	136	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H364
Italy, 1995	Koral	1	0.300	0.075	147	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H365
Italy, 1995	Baldo	1	0.300	0.075	142	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H366
Italy, 1995	Cigalon	1	0.300	0.075	143	< 0.05	< 0.04	< 0.09	1997/10290; IT10-95-H367

Table 100 Results of residue trials conducted with cycloxydim in rape seed forage (Study 2000/ 1013493)

Country, year	Variety	Application			DAT, days	Residues, mg/kg				Study Trial No.
		No	kg ai/ha	kg ai/hL		Cy1	Cy2	Total cycloxydim	Cy-TSO	
Norway, 1989	Emerald	1	0.600	0.250	76	n.r.	n.r.	0.25	–	51701H89/39E
Norway, 1989	Emerald	1	0.600	0.250	76	n.r.	n.r.	0.24	–	51722H89/1E
Norway, 1989	Emerald	1	0.500	0.250	55	n.r.	n.r.	0.26	–	51722H89/2E

Fates of residue in storage and processing

Hydrolysis

Cyclohexene-4,(6)-^{14}C-cycloxydim was dissolved in aqueous buffer solutions of different pH-values (Goetz, 2000a). To simulate pasteurisation, the test solution at pH 4 was heated for 20 min at 90 °C in

a round-bottom flask under reflux. To simulate baking, brewing and boiling, the test solution at pH 5 was treated in a round-bottom flask under reflux at 100 °C for 60 min. Sterilisation was performed at pH 6 at about 120 °C in autoclave for 20 minutes. Aliquots were taken right before starting and at the end of a test and measured by LSC and HPLC. The overall radioactivity before and after each test performed is given in Table 101. Cycloxydim degraded mainly to cycloxydim-T2S (oxazol).

Table 101 Material balance before and after processing simulation tests with ^{14}C-BAS 517 H, in % total applied radioactivity (TAR)

	pH 4, 90 °C		pH 5, 100 °C		pH 6, 120 °C	
Compound	before	after	before	after	before	after
cycloxydim	92.0	0.0	93.5	3.4	92.4	12.9
T2S	0.00	93.5	2.4	86.8	3.0	75.0
TSO	8.0	0.0	4.0	0.0	4.6	0.0
T2SO	0.0	6.4	0.0	4.7	0.0	11.0
Total recovery	99.9		94.9		99.0	

Strawberries

Residue levels of cycloxydim were determined in strawberries after processing from field trials at 0.650 kg ai/ha in Germany (Harant, 2009a). Samples were collected 42 ± 1 days after application and processed into washed fruits, jam and canned fruit. Samples were analysed with Method No. 407/1, in which cycloxydim and all its metabolites are oxidised to and measured as cycloxydim-TGSO2 and/or cycloxydim-OH-TGSO2 and residues expressed as cycloxydim. The results are shown in Table 102.

Table 102 Cycloxydim total residues on strawberry process fractions and processing factors

Portion Analysed	Cycloxydim total, (mg/kg)				Processing factor				
Trial	1	2	3	4	1	2	3	4	Mean
Strawberry fruit (RAC)	0.21	1.06	0.90	0.20	1	1	1	1	1
washed fruits	0.67	0.75	0.78	0.44	3.19	0.71	0.87	2.20	1.74
jam before cooking	0.50	0.46	0.55	0.16	2.38	0.43	0.61	0.80	1.06
jam after cooking	0.22	0.35	0.23	0.12	1.05	0.33	0.25	0.56	0.55
canned fruits	0.40	0.36	0.39	0.20	1.90	0.34	0.43	0.92	0.90
vegetable stock	0.20	0.22	0.40	0.11	0.95	0.21	0.44	0.54	0.54

Onion

Two field trials were conducted on onions in Italy and Greece at 0.6 L/ha and bulb samples taken after 56 days of application (Schulz, 2003a). After drying in the sun for 8 days, about 2.5 kg of the dried onions were peeled. Samples were analysed using Method 407/1. Residues in onion were 0.19 and 0.24 mg/kg, in dried onion were 0.22 and 0.33 mg/kg. Residues in peeled onions were 0.17 and 0.31 mg/kg, with processing factors of 0.88 and 1.32 (mean of 1.1).

Cabbage

Four field trials were conducted with cabbage in Germany and France when cycloxydim was applied once at a rate equivalent to 1.2 kg ai/ha and cabbage heads were collected 26 to 28 days thereafter (Reichert, 2006a). The samples were processed and the analysis for cycloxydim and its metabolites was carried out according to Method No. 407/1. The results are shown in Table 103.

Table 103 Cycloxydim total residues on raw and processed cabbage and its processing factors

Portion Analysed		Cycloxydim total, (mg/kg)		Processing factor				
Trial		Total	BH 517-TSO	1	2	3	4	Mean
white cabbage head		0.87–2.63	0.95–2.93	1	1	1	1	1
outer leaves		0.71–2.22	not analysed	0.70	0.82	0.75	1.41	0.92
outer stalks		1.11–1.64	not analysed	1.43	1.28	0.49	1.04	1.06
inner leaves		0.60–2.71	not analysed	1.26	0.75	0.23	1.72	0.99

Portion Analysed	Cycloxydim total, (mg/kg)		Processing factor				
Trial	Total	BH 517-TSO	1	2	3	4	Mean
inner stalks	0.72–2.72	not analysed	0.76	0.83	0.46	1.72	0.94
cooked white cabbage head	0.37–1.33	not analysed	0.70	0.43	0.27	0.84	0.56
non-past. juice of sauerkraut	0.88–2.02	not analysed	1.02	1.01	0.36	1.28	0.92
pasteurised sauerkraut	0.70–1.51	not analysed	1.05	0.80	0.30	0.96	0.78
pasteurised juice of sauerkraut	0.78–1.59	not analysed	0.99	0.99	0.30	1.01	0.82

Tomatoes

Cycloxydim was foliar-applied once to tomato plants in Germany at 0.65 kg ai/ha (Harant, 2009b). Tomatoes were harvested on 35 ± 1 days after application, processed and the tomato specimens and their processed products analysed using Method No. 407/1. The results are shown in Table 104.

Table 104 Cycloxydim total residues on raw and processed tomato and processing factors

Portion Analysed	Cycloxydim total, (mg/kg)				Processing factor				
Trial	1	2	3	4	1	2	3	4	Mean
fruit (RAC)	0.58	0.60	0.55	1.17	1	1	1	1	1
washed tomatoes	0.50	0.76	0.81	1.63	0.86	1.27	1.47	1.39	1.25
blanched tomatoes	0.53	0.59	0.93	0.96	0.91	0.98	1.69	0.82	1.10
peel	0.79	1.38	1.18	1.38	1.36	2.30	2.15	1.18	1.75
peeled tomatoes	0.74	0.69	0.88	1.11	1.28	1.15	1.60	0.95	1.25
canned tomatoes	0.17	0.40	0.40	0.69	0.29	0.67	0.73	0.59	0.57
raw mash	0.53	0.73	0.85	1.44	0.91	1.22	1.55	1.23	1.23
heated mash	0.34	0.50	0.41	0.77	0.59	0.83	0.75	0.66	0.71
wet pomace	0.70	1.14	0.70	1.34	1.21	1.90	1.27	1.15	1.38
raw juice	0.48	0.77	0.71	1.19	0.83	1.28	1.29	1.02	1.11
pasteurised juice	0.44	0.83	0.61	1.04	0.76	1.38	1.11	0.89	1.04
raw ketchup after concentration	0.65	0.96	1.06	2.12	1.12	1.60	1.93	1.81	1.62
raw ketchup after seasoning	0.58	1.10	1.46	2.00	1.00	1.83	2.65	1.71	1.80
ketchup after pasteurisation	0.62	0.65	1.53	1.59	1.07	1.08	2.78	1.36	1.57
puree after concentration	2.11	2.01	1.97	2.99	3.64	3.35	3.58	2.56	3.28
puree after pasteurisation	2.02	2.27	2.38	3.82	3.48	3.78	4.33	3.26	3.71

Lettuce

In four field trials conducted with lettuce in Denmark and Sweden at 1× 0.5 kg ai/ha, samples harvested at 14 days after treatment were processed and analysed by Method No. 407/1 (Schulz, 2005a). The samples were analysed for cycloxydim and cycloxydim-OH-TSO2 with. The results are shown in Table 105.

Table 105 Cycloxydim total residues on raw and processed lettuce and its processing factors

Portion Analysed	Cycloxydim total, (mg/kg)				Processing factor				
Trial	1	2	3	4	1	2	3	4	Mean
heads	0.28	0.69	0.65	0.18	1	1	1	1	1
outer leaves	0.56	1.35	1.61	0.40	2.0	1.9	2.5	2.2	2.2
inner leaves	0.28	0.30	0.44	0.24	1.0	0.4	0.7	1.3	0.9
washed outer leaves	0.36	1.33	1.13	0.30	1.3	1.9	1.7	1.6	1.6
washed inner leaves	0.15	0.30	0.36	0.20	0.5	0.4	0.6	1.1	0.7

Peas

Four field trials were conducted in Great Britain with cycloxydim on peas at 0.450 kg ai/ha (Beck *et al*., 2000a). The mature seed samples were harvested approximately 56 days after treatment and further processed. All samples were analysed with Method No. 263. Four trials were conducted in Germany and Southern France at 1.2 kg ai/ha and green seeds without pods were collected 34 to 36 days thereafter (Reichert, 2003a). For the processing, green peas were first cleaned and sorted out and

subsequently washed leading to the fractions washed peas and washing water. After addition of a salt-solution, sterilisation yielded canned peas and vegetable stock. The analysis for cycloxydim and its metabolites were with Method No. 407/1 (Reichert, 2003a). The results of all trials are shown in Table 106.

Table 106 Cycloxydim total residues on raw and processed peas and its processing factors

Portion Analysed	Cycloxydim total, (mg/kg)				Processing factor				
Trial	1	2	3	4	1	2	3	4	Mean
Peas (Beck *et al.,* 2000a)	2.90	2.62	1.28	1.22	–	–	–	–	–
washed pea	3.64	3.31	1.05	1.34	1.26	1.26	0.82	0.55	0.97
blanched pea	1.59	1.43	0.67	0.65	0.55	0.55	0.52	0.53	0.54
Peas (Reichert, 2003a)	3.75	14.36	7.60	6.51	–	–	–	–	–
peas/retain	4.19	13.39	6.31	5.83	1	1	1	1	1
washed peas	3.50	14.57	5.57	5.08	0.84	1	0.88	0.87	0.9
cooked peas	1.50	12.28	4.51	4.55	0.36	0.92	0.71	0.78	0.7
canned peas	0.58	2.96	1.01	1.21	0.14	0.22	0.16	0.21	0.2
vegetable stock	0.82	0.97	0.77	1.11	0.20	0.07	0.12	0.19	0.1

Carrots

Four field trials on carrots were conducted in Germany and Italy. The carrots were treated once with cycloxydim at 1.2 L/ha and samples taken 28 days thereafter at growth stage BBCH 48-49 (Reichert, 2005a). The samples were processed and analysed by methods 407/1 and 493/0. The results are shown in Table 107.

Table 107 Cycloxydim total residues on carrot process fractions and processing factors

Portion Analysed	Cycloxydim total, (mg/kg)				Processing factor				
Trial	1	2	3	4	1	2	3	4	Mean
carrot roots (RAC)	0.79	0.44	0.80	0.88	1	1	1	1	1
washed carrots	1.10	0.34	0.74	1.16	1.39	0.77	0.93	1.32	1.10
topped + peeled carrots	0.55	0.41	0.70	0.78	0.70	0.93	0.88	0.89	0.85
peel	0.51	0.37	0.43	0.59	0.65	0.84	0.54	0.67	0.68
cooked carrots	0.73	0.34	0.58	0.57	0.92	0.77	0.73	0.65	0.77
juice	0.42	0.24	0.34	0.42	0.53	0.55	0.43	0.48	0.50
pomace	0.48	0.25	0.41	0.60	0.61	0.57	0.51	0.68	0.59
canned carrots	0.32	0.18	0.23	0.28	0.41	0.41	0.29	0.32	0.36
vegetable stock	0.36	0.18	0.23	0.32	0.46	0.41	0.29	0.36	0.38

Potatoes

Potatoes treated once with cycloxydim at 30 L/ha in Germany and Italy were harvested 54–57 days after application and samples analysed with Method No. 407/1 (Schulz, 2003f). The results are shown in Table 108.

Table 108 Cycloxydim total residues on potato process fractions and processing factors

Portion Analysed	Cycloxydim total, (mg/kg)				Processing factor				
Trial	1	2	3	4	1	2	3	4	Mean
Potato tubers (RAC)	1.43	3.23	0.87	0.49	1	1	1	1	1
tubers / retain sample	1.27	2.60	0.38	0.75	0.9	0.8	0.4	1.5	0.9
washed potatoes	1.31	1.78	0.65	0.88	0.9	0.6	0.7	1.8	1.0
peels	1.76	2.41	0.46	0.97	1.2	0.7	0.5	2.0	1.1
peeled potatoes	2.04	1.98	0.54	1.28	1.4	0.6	0.6	2.6	1.3
boiled potatoes	2.10	3.58	0.67	1.17	1.5	1.1	0.8	2.4	1.5
steamed potatoes	1.54	2.37	0.54	0.82	1.1	0.7	0.6	1.7	1.0
peel/scrap	1.05	1.89	0.32	0.27	0.7	0.6	0.4	0.6	0.6
French fries	2.07	3.95	0.61	0.98	1.4	1.2	0.7	2.0	1.3

Rape seed

Rape seed was treated at rates of 0.450 to 0.90 kg ai/ha at four different locations in the UK and seed samples taken 89–98 days after application were further processed into oil and meal (press cake) simulating commercial processing (Steggles, 1992a). All samples were analysed with Method No. 263. In four trials conducted in Germany and Spain, cycloxydim was applied once with a 1.2 kg ai/ha, and seeds collected 79 to 116 days thereafter were processed into oil and meal. The analysis for cycloxydim and its metabolites was carried out according to Method No. 407/1 (Schulz, 2003g). The results of all trials are shown in Table 109.

Table 109 Cycloxydim total residues on raw and processed oilseed rape and its processing factors

Portion Analysed		Cycloxydim total, (mg/kg)				Processing factor				
Trial		1	2	3	4	1	2	3	4	Mean
whole plant w/o root (Steggles, 1992a)		2.13	3.37							
oil		0.54	0.33			0.25	0.10			0.18
meal		3.67	3.51			1.72	1.04			1.38
rape seed (RAC) (Schulz, 2003g)		1.74	1.05	2.91	3.87					
rape seed/retain sample		1.35	1.02	1.89	3.84	0.8	1.0	0.6	1.0	0.9
press cake		1.99	1.30	2.08	5.80	1.1	1.2	0.7	1.5	1.1
oil meal		2.07	2.18	4.63	7.53	1.2	2.1	1.6	1.9	1.7
crude oil		0.25	0.17	0.28	0.41	0.1	0.2	0.1	0.1	0.1
filter cake		0.09	< 0.09	< 0.09	0.12	0.05	< 0.09	< 0.03	0.03	0.05
fatty acids		< 0.09	< 0.09	< 0.09	< 0.09	< 0.05	< 0.09	< 0.03	< 0.02	< 0.05
refined rape oil		< 0.09	< 0.09	< 0.09	< 0.09	< 0.05	< 0.09	< 0.03	< 0.02	< 0.05

Sunflower

Two trials were carried out with cycloxydim on sunflowers in Italy at 0.30 or 0.6 kg ai/ha, and seed samples taken at 99 or 84 DALA, respectively, were processed to oil (Schulz, 1995a). All samples were analysed with Method No. 263. The results are shown in Table 110.

Table 110 Cycloxydim total residues in raw and processed oilseed rape and its processing factors

Portion Analysed		Cycloxydim total, (mg/kg)		Processing factor		
Trial [a]		1	2	1	2	Mean
seed	300 g ai/ha	< 0.9	< 0.9	n.a.	1	1
seed	600 g ai/ha	< 0.9	0.9	n.a.	1	1
oil	300 g ai/ha	< 0.09	0.09	n.a.	0.1	0.1
oil	600 g ai/ha	< 0.09	< 0.09	n.a.	< 0.1	< 0.1
expeller cake	300 g ai/ha	0.12 [b]–	0.67	n.a.	0.74	0.74
expeller cake	600 g ai/ha	0.11 [b]–	1.26	n.a.	1.4	1.4

[a] Trial 1: IT 10-93-H 363, Trial 2: IT 10-93-H 364

[b] Not exactly evaluable because of a disturbing peak. The chromatograms of the fortified samples showed that the residue concentrations must be close to LOQ.

Livestock feeding studies

Poultry

In a laying hen feeding study, cycloxydim and cycloxydim-OH-TSO at a ratio of 1:1 was administered orally to groups of three cages of four Leghorn hens by gelatine capsule for 28 days (Hopf *et al.,* 2011a). The nominal daily doses per animal were 0.15 mg, 0.45 mg, and 15 mg, corresponding to 2.29 mg/kg, 6.71 mg/kg and 23.2 mg/kg feed for the 1×, 3× and 10× dose level, respectively. Eggs were collected twice daily and pooled on a daily basis per cage for 28 days. Hens were scarified 23 hours after the last dose, liver, kidney, muscle and fat collected and samples of all animals from one cage were pooled. Samples were analysed using method L0105/01 in which non-hydroxylated metabolites and hydroxylated metabolites were oxidized to cycloxydim-TGSO2 and cycloxydim-5-OH-TGSO2, with H_2O_2 under alkaline conditions in animal matrices. The final

determinations of the two acids were performed by HPLC-MS/MS. Mean results of each treatment group are summarized in Table 111 for eggs.

Table 111 Group mean residues in eggs (sum of non-hydroxylated and hydroxylated metabolites; expressed as parent equivalents)

	Group Mean (and Maximum Individual), mg/kg			
Day of Study	Group 1 (Control)	Group 2 (1×)	Group 3 (3×)	Group 4 (10×)
0	n.a.	n.a.	< 0.02	0.023 (0.035)
2	n.a.	n.a.	< 0.02 (0.021)	0.077 (0.102)
4	n.a.	n.a.	0.021 (0.024)	0.076 (0.100)
6	n.a.	< 0.02	0.021 (0.024)	0.069 (0.093)
9	n.a.	n.a.	< 0.02	0.071 (0.081)
13	< 0.02	< 0.02	0.021 (0.022)	0.069 (0.082)
20	n.a.	n.a.	0.024 (0.032)	0.063 (0.098)
23	n.a.	n.a.	0.033 (0.058)	0.061 (0.068)
27	< 0.02	< 0.02	0.021 (0.023)	0.076 (0.090)
28 (1 DOW)	–	–	–	0.031 [a] (0.041)
29 (2 DOW)	–	–	–	< 0.02 [a]
30 (3 DOW)	–	–	–	< 0.02 [b]
31 (4 DOW)	–	–	–	< 0.02 [b]
33 (5 DOW)	–	–	–	< 0.02 [b]

[a] Results for cages 13 and 14

[b] Individual results cage 14; DOW: day of withdrawal (depuration)

Residues were not detected in muscle and fat at any dose group. In liver, residues were detected only at 10× dose group (Table 112).

Table 112 Group mean residues in tissues (sum of non-hydroxylated and hydroxylated metabolites; expressed as parent equivalents)

	Group Mean (and Maximum Individual) [mg/kg]		
Treatment Group	Muscle	Fat	Liver
1 (Control)	< 0.02	< 0.02	< 0.02
2 (1×)	< 0.02	< 0.02	< 0.02
3 (3×)	< 0.02	< 0.02	< 0.02
4 (10×)	< 0.02	< 0.02	0.022 (0.030)
10×, 3 days of withdrawal	< 0.02	< 0.02	< 0.02
10×, 7 days of withdrawal)	< 0.02	< 0.02	< 0.02

In order to determine the magnitude of the residues in chicken tissues and eggs following dietary exposure to cycloxydim and cycloxydim-5-OH-TSO (1:1 ratio), a feeding study was conducted on 56 laying hens in 14 cages with four animals (Grosshans & Kampke-Thiel, 2003a). For 28 days, laying hens were administered the mixture at a target dose level of 2.5, 7.5 and 25 ppm. Eggs from each cage were collected twice daily and combined as one pooled sample. Animals were sacrificed within 23 hours after the final morning dose and tissue samples were taken, except for hens in two cages of the 10× group, which were sacrificed three and seven days after the final morning dose to determine residue decline. Analysis of the samples was carried out according to Method No. 513/0. The results are shown in Table 113.

Table 113 Summary of residues in eggs and tissues

Treatment Group	Group Mean (Maximum Individual), sum of cycloxydim-TSO, cycloxydim-5-OH-TS and cycloxydim-5-OH-TSO			
	Muscle	Liver	Fat	Eggs (days 2 to 27)
Control	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)
1×	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)
3×	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (0.041)
10×	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)	0.046 (0.069)
10×, 3 days withdrawal	< 0.03	< 0.03	< 0.03	< 0.03
10×, 7 days withdrawal	< 0.03	< 0.03	< 0.03	< 0.03

Cattle

In a dairy cattle feeding study, cycloxydim and cycloxydim-5-OH-TSO at a ratio of 2:1 was administered orally to groups of three Holstein cattle by gelatine capsule (with one control group of three, fed capsules only) for 28 days (Grosshans, 2009a). The actual dose levels were 5.1 mg/kg, 15.2 mg/kg and 50.2 mg/kg feed for the 1×, 3× and 10× dose level, respectively. Milk was collected in the morning and afternoon and pooled on a daily basis for 28 days. Animals were sacrificed 22–24 hours after the last dose. In milk, a plateau was rapidly reached and residues in milk and tissues were eliminated from the animals in less than one week (Table 114). Residues were detected in muscle only at the 3× dose level (Table 115).

Table 114 Group mean residues in milk generated (sum of non-hydroxylated and hydroxylated metabolites; expressed as parent equivalents)

	Group Mean (and Maximum Individual), mg/kg			
Day of Study	Group A (control)	Group B 1×	Group C 3×	Group D 10×
1	< 0.019	< 0.019	< 0.019	0.035 (0.054)
5	n.a.	n.a.	< 0.019	0.042 (0.064)
10	n.a.	n.a.	< 0.019	0.042 (0.064)
14	< 0.019	< 0.019	n.a.	n.a.
17	n.a.	n.a.	n.a.	0.044 (0.061)
21	n.a.	n.a.	< 0.019	0.054 (0.080)
24	n.a.	n.a.	n.a.	0.046 (0.068)
28	< 0.019	< 0.019	< 0.019	0.038 (0.057)
2 DOW	–	–	–	< 0.019 [a]
4 DOW	–	–	–	< 0.019 [b]
6 DOW	–	–	–	< 0.019 [b]
7 DOW	–	–	–	< 0.019 [b]
Skim Milk (21 d)	< 0.019	< 0.019	< 0.019	0.044 (0.057)
Cream (21 d)	< 0.019	< 0.019	< 0.019	0.033 (0.043)

n.a. = Not analysed

[a] Results cow 13

[b] results cow 14

DOW: Day of withdrawal (depuration)

Table 115 Group mean residues in tissues (Sum of non-hydroxylated and hydroxylated metabolites; expressed as parent equivalents)

Treatment Group	Group Mean (and Maximum Individual), mg/kg			
	Muscle	Liver	Kidney	Fat
A (control)	< 0.019 (< 0.019)	< 0.019 (< 0.019)	< 0.019 (< 0.019)	< 0.019 (< 0.019)
B (1× dose level)	< 0.019 (< 0.019)	0.043 (0.045)	0.068 (0.073)	< 0.019 (< 0.019)
C (3× dose level)	0.023 (0.026)	0.128 (0.151)	0.202 (0.239)	0.025 (0.030)
D (10× dose level)	0.073 (0.088)	0.336 (0.381)	0.593 (0.727)	0.119 (0.138)
D 2 days withdrawal	< 0.019	0.079	0.057	0.020
D 7 days withdrawal	< 0.019	< 0.019	< 0.019	< 0.019

In another study, 14 lactating dairy cows were administered a 2:1 ratio of cycloxydim and cycloxydim-5-OH-TSO for 28 days at a target dose level of 5.0 mg/kg (1×), 15.0 mg/kg (3×) and 50 mg/kg (10×) (Seiferlein & Kampke-Thiel, 2003a). Milk samples from each animal were collected twice daily for 28 days and combined as one pooled sample. On Day 21, milk was also separated into cream and skim milk. Animals were sacrificed within 23 hours after the final morning dose and tissue samples were taken, except for two cows of the 10× group, which were sacrificed two and seven days after the final morning dose to determine residue decline. Analysis of the samples was carried out according to Method No. 513/0. The results are shown in Table 116.

Table 116 Summary of residues in milk and tissues

Treatment Group	Muscle	Liver	Kidney	Fat	Milk (days 3 to 28)	Skim Milk	Cream
1 (control)	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.03 (< 0.03)	< 0.009	< 0.009	< 0.009
2 (1×)	< 0.03 (< 0.03)	0.03 (0.04)	0.05 (0.06)	< 0.03 (< 0.03)	< 0.009	< 0.009	< 0.009
3 (3×)	< 0.03 (< 0.03)	0.12 (0.15)	0.14 (0.18)	< 0.03 (< 0.03)	< 0.009	< 0.009	< 0.009
4 (10×)	0.06 (0.07)	0.29 (0.31)	0.44 (0.51)	0.10 (0.12)	0.020	0.020	0.016
4 (10×, 2 days withdrawal)	< 0.03	0.06	0.05	< 0.03	< 0.009	–	–
4 (10×, 7 days withdrawal)	< 0.03	< 0.03	< 0.03	< 0.03	< 0.009	–	–

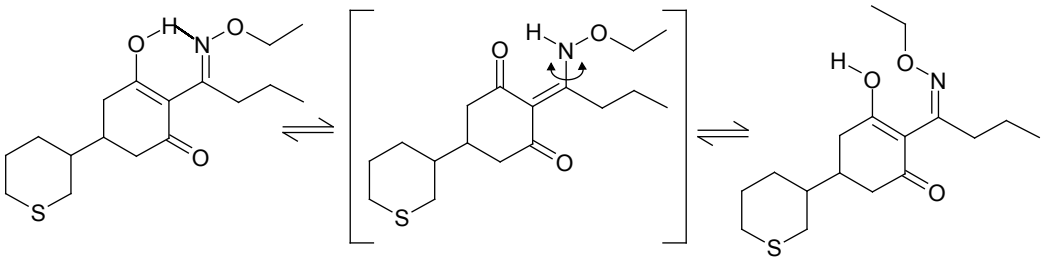
APPRAISAL

Cycloxydim is a post-emergence cyclohexene oxime herbicide that inhibits the acetylcoenzyme A carboxylase (ACCase) in chloroplasts of sensitive weeds. Cycloxydim was firstly evaluated by JMPR in 1992 (T, R). In 2009, an ADI of 0–0.07 mg/kg bw and an ARfD of 2 mg/kg bw for women of childbearing age were established; an ARfD was unnecessary for the general population. Cycloxydim was scheduled at the 43rd session of the CCPR (2011) for the periodic re-evaluation of residues by the 2012 JMPR.

Data on physical and chemical properties, metabolism in plants and livestock animals, environmental fate and analytical methods, animal feeding studies and processing studies were submitted. Residue supervised trials were submitted on pome fruits, stone fruits, grapes, strawberries, potatoes, carrots, celeriac, onions, tomatoes, peppers, cauliflower, Brussels sprouts, head cabbage, curly kale, lettuce, spinach, green beans and peas, leek, sugar beet roots, sugar beet tops, dry beans and peas, oilseed rape, sunflower, soya bean, rice and maize.

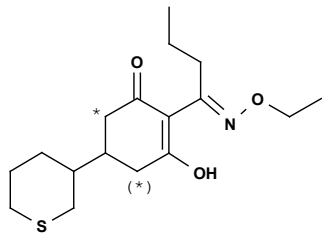
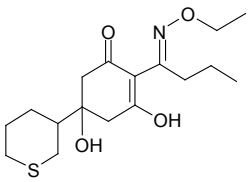
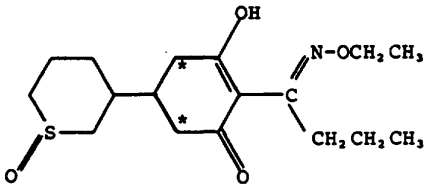
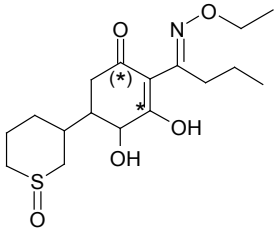
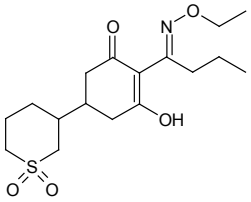
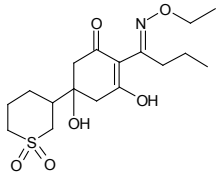
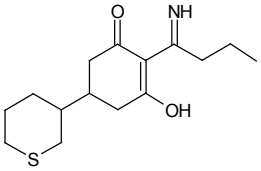
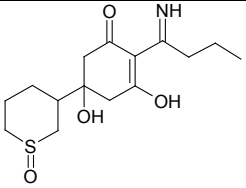
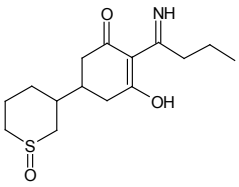
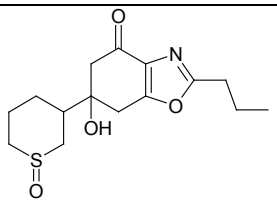
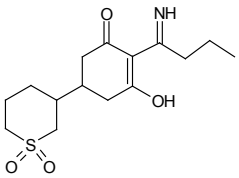
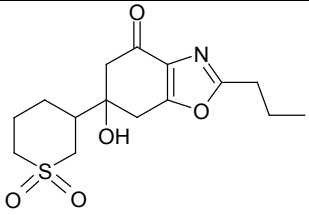
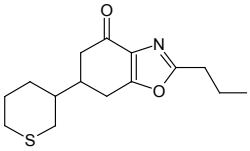
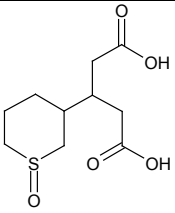
Metabolism studies

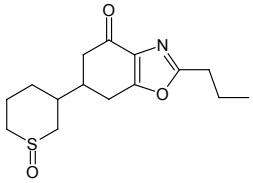
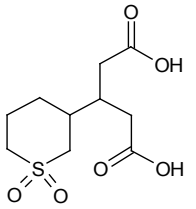
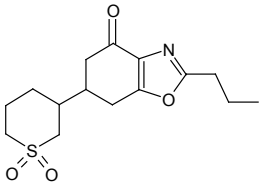
Cycloxydim is a racemic mixture. In the [^{14}C]labelled cycloxydim stored in toluene, the compound is almost exclusively present as E-isomer, but E/Z isomerisation in the ethoxyimino group may occurs on in plants and in solution depending on solvent polarity, temperature and pH. In all metabolism studies, residues were reported as the sum of the two isomers.



E-Isomer Hypothetical transition state Z-Isomer

The positions of the radiolabel compounds used in the metabolism studies and the structures of the main metabolites found in animals and plants are shown next.

Name	Structure	Name	Structure
^{14}C-Cycloxydim		Cycloxydim-5-OH-TS	
^{14}C-Cycloxydim-TSO		^{14}C-Cycloxydim-5-OH-TSO	
Cycloxydim-TSO2		Cycloxydim-5-OH-TSO2	
Cycloxydim-T1S		Cycloxydim-5-OH-T1SO	
Cycloxydim-T1SO		Cycloxydim-6-OH-T2SO	
Cycloxydim-T1SO2		Cycloxydim-6-OH-T2SO2	
Cycloxydim-T2S		Cycloxydim-TGSO	

Name	Structure	Name	Structure
Cycloxydim-T2SO		Cycloxydim-TGSO2	
Cycloxydim-T2SO2			

Animal metabolism

Rats

Metabolism studies conducted in rats were evaluated by the JMPR 2009. The studies were conducted after oral administration of the vinylogus acid [^{14}C]cycloxydim-TS and its sodium salt and after intravenous administration of the sodium salt, at a nominal dose of 10 mg/kg bw (no-effect level) or 300 mg/kg bw (toxic effect level). The major metabolite found in urine was cycloxydim-TSO, followed by cycloxydim-T1SO and cycloxydim-TSO. In addition to unchanged parent, other metabolites were cycloxydim-T1SO2, cycloxydim-T2SO, and the metabolite hydroxylated at the 5-position of the cyclohexene ring of the parent. Patterns of metabolites in the bile were similar after the administration of either the free acid or the sodium salt of cycloxydim.

Lactating goats

[^{14}C]cycloxydim was administrated for 7 days to goats at 15 ppm feed. About 85% of the dose was recovered at the end of the experiment, mainly in urine (72%) and faeces (12%), and 0.09% in milk. Radioactivity in milk was constant during the 7 days dosing period (mean of 0.023 mg/kg). Total radioactive residue (TRR) was 0.005–0.006 mg/kg eq. in fat and muscle, and fat to 0.062–0.076 mg/kg eq. in liver and kidney, respectively. Non-extracted residues accounted for up to 64.1%TRR in liver, most of it solubilised by pronase. The major metabolite identified in milk was cycloxydim-T1SO (16%TRR). Cycloxydim-TSO represented 15%TRR in milk, 8%TRR in liver and 12%TRR in kidney. Parent compound was only detected in liver (10.8%TRR, 0.008 mg/kg). Minor metabolites found are cycloxydim-T1S, cycloxydim-TSO2 in liver (up to 2%TRR, 0.001 mg/kg) and cycloxydim-T2SO2 in milk (up to 2%TRR, < 0.001 to < 0.001 mg/kg).

A lactating goat was dosed with [^{14}C]cycloxydim-TSO at 100 ppm feed for five days. At sacrifice, 89.1% of the administered dose was recovered, mostly in urine (78%) and faeces (10%). TRR in milk accounted for 0.11% of the dose (0.09–0.12 mg/kg). Liver and kidney had the highest residues, 0.46, 0.52 and, 0.52 mg/kg eq., respectively. Concentrations in fat and muscle were 0.04 mg/kg, and were not further identified. Cycloxydim-TSO and cyclodixim-T1SO were the major components in milk (about 23%TRR, 0.06 mg/kg and the only residues identified in liver (22%TRR, 0.10 mg/kg; and 10%TRR, 0.05 mg/kg, respectively). Cycloxydim-T2SO cycloxydim-T1SO2 (5%TRR, 0.01 mg/kg) and cycloxydim-TSO2 (2.7%TRR, < 0.01 mg/kg) were also found in milk. Residues in kidney were not characterized.

The [^{14}C]cycloxydim-5-OH-TSO was administrated to goats at a dose level of 12 ppm feed on nine consecutive days. In average, 97% of the administered dose was recovered in the experiment, of which 75% was in urine and 15.7% in faeces. TRR was about 0.02 mg/kg eq. in milk, 0.024 in fat and 0.025 in muscle, 0.203 in liver and 0.26 mg/kg eq. in kidney. Non-extracted residues accounted for up

to 10.2% TRR in muscle. Cycloxydim-5-OH-TSO was the main residue in milk, muscle, kidney and fat (31 to 38%TRR; 0.007 to 0.10 mg/kg). In liver, the main residue was cycloxydim-5-OH-TS (17.4%TRR, 0.03 mg/kg), followed by cycloxydim-5-OH-TSO (11%TRR, 0.02 mg/kg). Cycloxydim-5-OH-TS was also a significant residue in kidney (25%TRR, 0.06 mg/kg) and fat (11%TRR, 0.003 mg/kg). Cycloxydim-6-OH-T2SO residues reached 13%TRR in muscle and cycloxydim-5-OH-T1SO represented < 10%TRR in all matrices.

Laying hens

The metabolism and distribution of [^{14}C]cycloxydim was investigated in laying hens following oral administration at 12 ppm feed for 10 days. About 80% of the administered dose was recovered at the end of the experiment, mostly in the excreta (78.0%), 0.33% in eggs. 0.11 % in muscle, 0.07 % in liver and 0.02% in fat. TRR in eggs ranged from 0.05 mg/kg eq. at day 1 to 0.14 mg/kg at day 10 (mean of 0.12 mg/kg eq.), starting to plateau at day 8. In tissues, TRR was 0.051–0.053 mg/kg eq. in fat and muscle and 0.28 mg/kg eq. in liver. Unextracted residues in muscle accounted for 4.5% TRR. Residues in eggs were mostly cycloxydim-TSO (0.04 mg/kg eq., 30.9%TRR), followed by cycloxydim-TSO2 (0.008 mg/kg eq., 6.4%TRR) and the parent compound (0.004 mg/kg eq., 3.4%TRR). In muscle, only cycloxydim-TSO2 was detected (0.001 mg/kg eq., 0.9%TRR) and in fat only cycloxydim-TSO (0.008 mg/kg, 18%TRR). In liver, the main residue detected was also cycloxydim-TSO (0.02 mg/kg eq., 7.4%TRR), followed by cycloxydim (0.005 mg/kg eq., 1.7%TRR) and cycloxydim-TSO2 (0.002 mg/kg eq., 0.6%TRR).

The [^{14}C]cycloxydim-TSO was administered to laying hens at a dose level of 50 ppm feed for 7 days. Treated animals were sacrificed 6 (group 2), 24 (group 3) and 48 hours (group 4) after the last dose. About 94% of the administered dose was recovered in group 4, mostly (92.3%) in the excreta, and 0.08% in eggs. In this group, TRR in eggs increased rapidly to an apparent plateau of 0.08–0.10 mg/kg after day 2. TRR from group 2 ranged from 0.10 mg/kg eq. in muscle to 0.99 mg/kg eq. in kidney, and were < 0.1 mg/kg in all tissues from group 4. Residues were only identified in eggs and liver. In eggs, cycloxydim-TSO (0.12 mg/kg, 41.4%TRR) was the major residue, followed by cycloxydim-TSO2 (0.03 mg/kg, 8.8 %TRR) and cycloxydim-T2SO (0.02 mg/kg, 5.5%TRR). In liver, cycloxydim-TSO was the major residue (0.19 mg/kg, 33%TRR), followed by cycloxydim-T2SO (0.14 mg/kg, 24%TRR) and cycloxydim-T1SO (0.10 mg/kg, 17%TRR).

The [^{14}C]cycloxydim-5-OH-TSO was administered to hens at a dose of 12 ppm feed for 11 days. The radioactivity was rapidly excreted within 24 hours after the last dose, with 93% of the applied dose recovered, mainly on excreta (89.7%). In eggs, mean residues were 0.066 mg/kg eq. (0.21% of the applied dose). In tissues, the highest radioactivity was found in liver (0.11 mg/kg eq.), followed by muscle (0.028 mg/kg eq.), and fat (0.02 mg/kg eq.). Non-extracted residues ranged from 24.1%TRR in fat to 38% TRR in muscle, with over 80% being released by protease. Cycloxydim-5-OH-TSO accounted for 15% TRR in eggs (0.01 mg/kg) to 29%TRR in fat (0.005 mg/kg). Cycloxydim-5-OH-TS accounted for about 19–22%TRR in muscle, fat and liver (0.004–0.021 mg/kg) and 50.7%TRR in eggs (0.034 mg/kg).

In summary, studies conducted with cycloxydim or its metabolites cycloxydim-TSO and cycloxydim-5-OH-TSO showed that the primary metabolic pathway of cycloxydim in animals involves two main routes: 1) oxidation to cycloxydim-TSO and subsequently to cycloxydim-TSO2, and 2) Beckmann re-arrangement with subsequent ring closure to form the oxazol cycloxydim-T2S, which can be oxidized further to cycloxydim-T2SO and cycloxydim-T2SO2. Cycloxydim was a minor component in eggs and tissues from dosed hens and was only detected in goat liver (11%TRR).

Plant metabolism

Soya beans, cotton and sugar beet

The metabolism of [^{14}C]cycloxydim was studied in soya beans, cotton and sugar beet using two different treatments. To evaluate root uptake, the plants were cultivated in a nutrient solution containing 5 mg/L [^{14}C]cycloxydim. To evaluate the uptake by the leaf, 10 µg [^{14}C]cycloxydim was

applied to the upper leaf surface (soya bean, sugar beet) or one cotyledon (cotton). Samples were taken 3 and 7 days (soya bean and cotton) or 4 and 8 days (sugar beets) after the application, respectively. [^{14}C] cycloxydim was taken up by the roots and transported acropetally to the cotyledons, stem and the remaining leaves. The highest radioactivity was detected in the primary leaves and roots (25–26 µg [^{14}C]cycloxydim/[^{14}C]equiv at 7 DAT in soya beans). After leaf application, about 30% of the total radioactivity is translocated to the untreated soya bean plant parts at 7 days DAT, 8.3% to untreated cotton parts and 11.8% to untreated sugar beet parts.

In another study conducted with sugar beet at the 3-leaves stage, the seedlings were treated with [^{14}C]cycloxydim at 0.2 kg ai/ha. Immediately after application, TRR was 8.48 mg/kg eq.in the tops. At harvest (119 DAT), TRR was 0.13 mg/kg eq. in the tops and 0.015 mg/kg eq. in roots. At 22 DAT, cycloxydim-TSO2 and cycloxydim-T2SO were found in the tops at 0.18 mg/kg eq. (13.9% TRR) and 0.14 mg/kg eq. (11% TRR), respectively. At 46 DAT, various metabolites were found in the tops at low levels (0.0026 to 0.023 mg/kg eq., up to 4%TRR). No hydroxylated metabolites where detected in any sample.

In a third study on sugar beet, [^{14}C]cycloxydim was applied at 0.65 kg ai/ha 2 months after sowing and samples taken 1 day after treatment and at crop maturity (94 DAT). At 1 DAT, TRR were 23 and 4 mg/kg eq. in leaves and roots, respectively, mostly extracted in methanol and dichloromethane. At harvest, residues were 2.2 and 0.16 mg/kg eq. in tops and roots, respectively. Cycloxydim was not detected in any of the top and root samples taken. At 1 DAT, residues mainly of cycloxydim-TSO, with 7.6 mg/kg(31.6%) in the tops and 2.4 mg/kg (60.1%) in roots. Cycloxydim-TSO2 accounted for 16–18%TRR. At harvest, both metabolites were still present (< 10%TRR), but cycloxydim-T1SO was the predominant metabolite (0.42–0.02 mg/kg, 19–14%TRR in tops and roots, respectively). Only cycloxydim-T2S was present in amounts greater than 10%TRR (13.7%). No hydroxylated metabolite was observed.

[^{14}C]cycloxydim was applied to soya beans at 0.2 kg ai/ha two to eight weeks after sowing (three trials, group 1) or at 1 kg ai/ha two months after sowing (one trial, group 2). TRR were 20, 2.3 and 0.46 mg/kg eq. at 45, 71 and 82 DAT in seeds from the group 1 and 12.5, 0.76 and 0.31 mg/kg eq. in straw and stalk. TRR in straw and seed from group 2 were 91 and 38.4 mg/kg eq., respectively. In most plant samples, more than 85% of the TRR were extracted with aqueous methanol and at later sampling intervals, considerable amounts of radioactivity were detected in the aqueous phase. The parent molecule was detected only at the day of application in all trials. The major metabolites found in seed samples were cycloxydim-TSO (0.078 to 0.42 mg/kg, 11.9 to 18.3%TRR), cycloxydim-T2SO (0.11 to 3.7 mg/kg, 4.8 to 18%TRR), cycloxydim-5-OH-TSO (0.2 to 1.3 mg/kg, 6.4 to 8.7% TRR) and cycloxydim-5-OH-TSO2 (0.06 to 0.90 mg/kg, 4.5 to 12%TRR). In straw the hydroxylated metabolites were detected at low levels (up to 3.4%TRR).

Maize

In two studies conducted in tolerant maize, [^{14}C]cycloxydim was applied at 0.4 kg ai/ha (normal rate) at BBCH growth stage 22–23 or at 0.8 kg ai/ha during flowering (BBCH 61-67). In the normal use rate, TRR at harvest (96 DAT) were 0.123 mg/kg eq. in grain, 0.06 mg/kg eq.in cobs, 0.118 mg/kg eq.in husks and 0.17 mg/kg in straw; residues in forage at 72 DAT were 31 mg/kg eq. In the exaggerated rate samples, the TRRs ranged from 4.9 mg/kg eq. in grain to 13 mg/kg in straw eq.. Cycloxydim was not detected in any sample from any trial. In the normal rate trials, metabolite levels accounted for up to 1%TRR in grain, up to 8.7%TRR in straw (0.015 mg/kg cycloxydim-TGS + cycloxydim-TGSO2) and up to 11%TRR in forage (3.6 mg/kg cycloxydim-T1SO). In grain from the exaggerated rate trial, cycloxydim-TSO (0.53 mg/kg, 10.6%TRR), cycloxydim-T1S0 + cycloxydim-T2SO2 and cycloxydim-T2S02 + cycloxydim-T1SO2 (0.71 mg/kg, 14%TRR each) were the major metabolites. Only cycloxydim-T2SO accounted for higher than 10%TRR in straw, husks and cobs. In all normal use rate samples except forage, the most prominent peak was very polar and eluted with the void volume from the HPLC column. The metabolites formed were further degraded and radioactivity incorporated into the carbohydrate pool.

In summary, cycloxydim is metabolized in plants mainly by four steps: 1) oxidation at the sulphur of the thiopyrane ring to the sulfoxide and to the sulphone, 2) cleavage of the oxime ether group (loss of the alkyl side chain), 3) hydroxylation at the 5-position of the cyclohexenone ring system and 4) oxidative cleavage of the cyclohexenone ring resulting in substituted glutaric acid derivatives. Cycloxydim was not present in any of the samples from treated crops at harvest, with the main metabolites being cycloxydim-TSO, cycloxydim-T2SO, cycloxydim-T1SO (sugar beet root) and cycloxydim-5-OH-TSO (soya bean).

Environmental fate in soil

The aerobic degradation and metabolism of [^{14}C]cycloxydim was studied in a loamy sand soil treated with 10 mg/kg dry soil, equivalent to a maximum single application rate of about 7.5 kg ai/ha, and to a multiple dose of 0.6 kg ai/ha. The soil was incubated at 22 ± 2 °C for 90 day. About 14% TRR was detected as cycloxydim at day 0, decreasing to 0.9%TRR after 14 days. Cycloxydim-TSO/cycloxydim-T1SO/cycloxydim-T2SO represented 16% TRR at 56 DAT and cycloxydim-T1S/cycloxydim-T2S/cycloxydim-TSO2 represented 2.7% TRR at 28 DAT. After three months, bound residues accounted for about 40% TRR and CO_2 to 38% TRR. In another study conducted under the same conditions with loamy sand and a loam soils, cycloxydim accounted for over 90% TRR at day 0, decreasing to 4.6 and 0%TRR at day 21, respectively. Cycloxydim-TSO was the major metabolite found in soils (up to 39.5%TRR at 21 DAT and to 11% TRR at 90 DAT). In another study conducted with seven batches of sandy loam and loam sandy soils treated at 0.8 mg/kg, the soils were incubated for 119 days in the dark at 20 °C and 40% maximum water holding capacity. The degradation of cycloxydim was very fast in the soils (DT_{50} < 9 hours), with up to 18% of the applied radioactivity (TAR) found at 0 DAT. Cycloxydim-TSO (5–6%TAR), cycloxydim-TSO2 (1.5–2.8%TAR) and cycloxydim-T2SO (about 1%TAR) were the major metabolites at 60 DAT, with DT_{50} of 9.3–1.6 days, 8.8–13 days and 19–291 days, respectively.

The photolytic degradation of cycloxydim was studied on loamy sand soil treated with 10 mg/kg dry and incubated at 30 ± 5 °C for 8 hours. Bound residues were similar with and without irradiation (maximum about 8–15% TAR). Polar degradation products did not accumulate in the course of the study. Cycloxydim residues dropped to 2% TAR at 8 hours. Cycloxydim-TSO/cycloxydim-T2SO accounted to 77–81% TAR after 3 to 8 hours of incubation.

Confined rotational studies were conducted with [^{14}C]cycloxydim applied to soils at 0.65 kg ai/ha. The treated soils were aged for 30 days (radish and lettuce), 80 days (wheat), 120 days and 365 days. Residues in soil were 4.84 mg/kg eq. at day 0 and 0.034 to 0.136 mg/kg eq. after aging and harvesting in all cases. At 30 DAT, TRRs in lettuce (67 days after planting, DAP) and radish root and top (86 DAP) ranged from 0.032 to 0.051 mg/kg eq. At 120 and 365 DAT they ranged from 0.003 to 0.011 mg/kg. At 80, 120 and 365 DAT (118 to 169 DAP) residues ranged from 0.014 to 0.098 mg/kg eq. in wheat grain, from 0.07 to 0.14 mg/kg in straw and from 0.05 to 0.14 mg/kg in chaff. In wheat forage (57 to 70 DAP), they ranged from 0.008 to 0.031 mg/kg. At 30 DAT, cycloxydim-TSO and cycloxydim-TSO2 were the major metabolites in lettuce (0.008 mg/kg, 16%TRR), radish root (0.002 mg/kg, 6.3% TRR) and top (0.006 mg/kg, 12% TRR). At 120 DAT, only cycloxydim-TSO2 could be detected as a single compound (in radish, 10%TRR, 0.001 mg/kg). At 80 DAT, cycloxydim-TGSO2 was the major metabolite in wheat forage (0.008 mg/kg, 26% TRR), and cycloxydim-T2SO (0.03 mg/kg, 20% TRR) and cycloxydim-T1SO (0.02 mg/kg, 16% TRR) in straw. No single residues > 10%TRR were found in wheat commodities at 120 and 365 DAT.

In summary, cycloxydim is extensively and rapidly degraded in soil (DT_{50} < 9 hours), mainly to cycloxydim-TSO, cycloxydim-T1SO and cycloxydim-T2SO. Cycloxydim-TSO and cycloxydim-TSO2 were found in lettuce and radish planted on aged treated soil. Cycloxydim-TGSO2 (cycloxydim-1-dicarboxylic acid) was the main residue in succeeding wheat forage, and cycloxydim-T1S0 and cycloxydim-T2SO in wheat straw (80 DAT). No single residues were detected in wheat grain planted in aged treated soil.

Methods of analysis

Two common moiety analytical methods were developed, allowing the determination of cycloxydim, cycloxydim-5-OH-TSO2 and all metabolites that can be oxidized to cycloxydim-TGSO2 or cycloxydim-5-OH-TGSO2 with H_2O_2 under alkaline conditions in various plant matrices. In the LC-MS/MS method, the residues are extracted with isopropanol/water and hydrogen peroxide to form the corresponding pentane acids, the acids removed by precipitation with $Ca(OH)_2$, the excess of oxidizing agent eliminated using a C_{18}-column and, after a NH_2-column clean-up, cycloxydim-TGSO2 and cycloxydim-5-OH-TGSO2 are analysed. In the GC method, the acids are converted into the dimethyl esters cycloxydim-TDME and cycloxydim-OH-TDME, cleaned up in silica gel and analysed by GC/FPD or GC/MS. [^{14}C]cycloxydim studies showed that extraction with isopropanol/water released from 66 to 94%TRR. Both GC and LC methods were satisfactorily validated at 0.05 mg/kg (LOQ) or higher levels (up to 5 mg/kg), using cycloxydim and cycloxydim-OH-SO2 as representative analytes for the non-hydroxylated and hydroxylated compounds, respectively, or with different metabolites.

Cycloxydim-TSO can be selectively analysed in plant matrices after extraction with methanol/water buffer, saturation with NaCl-solution and partitioned with dichloromethane. The aqueous phase is acidified, residues extracted with isooctane/dichloromethane, the extract washed with saturated NaCl-solution, re-extracted with NaCl-solution, the aqueous phase purified by C_{18}SPE and residues determined by HPLC/UV. LOQ was 0.05 mg/kg.

Common moiety methods were also validated in matrices of animal origin, with an LOQ of 0.05 mg/kg for tissues and eggs and 0.01 mg/kg for milk (GC method) or 0.01 mg/kg (tissues and eggs) and 0.003 mg/kg (milk) in the LC methods. Efficiency of the acetonitrile/hexane extraction was comparable with the results from hen metabolism studies (methanol extraction) for eggs and muscle, but not for liver, where acetonitrile/hexane only extracts about 50–70% of the radioactivity. The efficiency of the acetonitrile/hexane extraction of liver, kidney and milk was also comparable with the results obtained in the goat metabolism studies.

Stability of residues in stored analytical samples

The stability of cycloxydim, cycloxydim-TSO + cycloxydim-T2SO2 and/or cycloxydim-5-OH-TSO2 residues was investigated in various plant matrices fortified at 0.4 to 0.5 mg/kg levels stored at -20 °C over a period of up to 2 years. Samples were analysed immediately after spiking and after different storing intervals using the common moiety methods. The results showed that the residues were stable (70–110% remaining) for at least 2 years of storage in most cases.

Liver and milk samples from animal metabolism studies with [^{14}C]cycloxydim or [^{14}C]cycloxydim-5-OH-TSO were re-extracted with methanol or acetonitrile/hexane (4:3) in order to investigate their stability in animal matrices stored for a period of 78–89 month at -20 ºC. The results showed that cycloxydim levels decreased over time while its metabolites increased (cycloxydim-T1SO and cycloxydim-5-OH-T2SO). This degradation is not relevant when the common moiety method is used to analyse the samples in the trials.

Residue definition

Metabolism studies conducted in hens and goats with cycloxydim showed that the parent compound was only detected in liver (0.008 mg/kg, 10.8%TRR). The main metabolites found were cycloxydim-TSO, cycloxydim-TSO2 (hens and goats) and cycloxydim-T1SO (up to 14.8%TRR in milk). Residues were present in fat and muscle at about the same level.

Metabolism studies conducted in soya bean, cotton, sugar beet and maize with cycloxydim have showed that the parent compound was not present in any of the samples at harvest, with the main metabolites being cycloxydim-TSO, cycloxydim-T2SO, cycloxydim-TSO2, cycloxydim-T1SO and cycloxydim-5-OH-TSO.

In all the supervised trials and animal feeding studies submitted to the Meeting, residues were measured using one of the common moiety methods, where residues present in the samples are

oxidized to cycloxydim-TGSO2 (including cycloxydim-TSO, cycloxydim-T2SO, cycloxydim-TSO2, cycloxydim-T1SO) and/or cycloxydim-5-OH-TGSO2 (including cycloxydim-5-OH-TSO) and analysed by LC-MS/MS or further methylated to cycloxydim-TMDE and/or cycloxydim-5-OH-TMDE for analysis by GC/FPD or GC/MS.

The Meeting agreed that the common moiety analytical methods analyse all the relevant residues formed when cycloxydim is applied on the field or when residues present in feed are ingested by farm animals.

The residue definition for animal and plant commodities for enforcement and risk assessment purposes is: *Cycloxydim, metabolites and degradation products which can be oxidized to 3-(3-thianyl) glutaric acid S-dioxide and 3-hydroxy-3-(3-thianyl) glutaric acid S-dioxide, expressed as cycloxydim*.

The residue is not fat-soluble

Results of supervised residue trials on crops

In some countries, cycloxydim label does not specify the PHI, indicating that the product should be applied when the weeds are actively growing. When a GAP with no specified PHI was used to support the trials, the highest residue found in the trials at any DAT, except the 0 day, was selected for the estimation.

The OECD MRL calculator was used to assist in the estimation of maximum residue levels from the selected residue data set obtained from the supervised residue trials. The Meeting reviewed the trial conditions and other relevant factors related to each dataset to arrive at a best estimate of the maximum residue level using expert judgment. When the OECD calculator suggested a different value, an explanation on the discrepancy was included in the text.

Pome fruits and stone fruits

Cycloxydim is registered to be used by direct spraying to control weeds in apple and pear orchards in Portugal using one application at 0.4 kg ai/ha and 28 days PHI. Two trials were conducted with apples and two with pears in Italy and Spain, matching the GAP of Portugal, gave residues of cycloxydim < 0.09 mg/kg (4).

Cycloxydim is registered to be used by direct spraying to control weeds in Italy in apricot and peach orchards up to 0.6 kg ai/ha with a 30 day PHI. In three trials in peaches and two in apricots conducted in Italy, at GAP, residues were < 0.09 mg/kg (5).

The Meeting concluded that on the basis of the data from these nine trials that the application of cycloxydim to the orchard floor, matching GAP, does not result in residues in the fruit.

The Meeting estimated a maximum residue level of 0.09* mg/kg, a HR of 0.09 mg/kg and a STMR of 0.09 mg/kg for cycloxydim in pome fruits and stone fruits

Grapes

The critical application rate of cycloxydim used for directed spraying to control weeds in grapes is a single application at 0.4 kg ai/ha in Spain and 0.6 kg ai/ha in Switzerland, with no specified PHI.

Eight trials were conducted in northern/central France and Germany, matching the GAP of Switzerland, gave residues of (n = 8) < 0.09 (4), 0.13 (2) and 0.18 (2) mg/kg.

In eight trials conducted in Italy, Greece and Spain according to Spanish GAP, residues were < 0.09 mg/kg (8).

Based on the residue data from trials conducted in northern Europe, the Meeting estimated a maximum residue level of 0.3 mg/kg, a HR of 0.18 mg/kg and a STMR of 0.11 mg/kg for cycloxydim in grape.

The Meeting withdrew its previous recommendation of 0.5 mg/kg for cycloxydim in grapes.

Strawberry

Cycloxydim is registered for use in strawberries in Switzerland at 1 × 0.6 kg ai/ha with a 42 day PHI and in Slovakia at 1 × 0.4 kg ai/ha and no PHI specified. The product is also registered in Romania at 1 × 0.4 kg ai/ha with no PHI specified.

Seven trials conducted according to Slovakian GAP in France (north), the Netherlands, Germany and the UK, gave total cycloxydim residues of 0.19, 0.22, 0.29, 0.33, 0.34 0.47 and 0.61 mg/kg. One trial conducted in Sweden according to the GAP of Switzerland gave residues of 0.63 mg/kg.

Eight trials were conducted in southern France, Greece, Italy and Spain matching Romanian GAP, gave residues of 0.29, 0.33, 0.35, 0.49, 0.57, 0.74, 1.2 1.4 and 1.4 mg/kg

Based on the residue data from trials conducted in southern Europe, the Meeting estimated a maximum residue level of 3 mg/kg, a HR of 1.4 mg/kg and a STMR of 0.53 mg/kg for cycloxydim in strawberries.

The Meeting withdrew its previous recommendation of 0.5 mg/kg for cycloxydim in strawberries.

Onion, Bulb

Cycloxydim is registered in the Netherlands at up to 2 × 0.6 kg ai/ha with a 21 day PHI. In Belgium, the GAP is for 1 × 0.6 kg ai/ha and a 28 day PHI. In Italy, the GAP is for 1 × 0.6 kg ai/ha and a 60 day PHI. In Spain, it is 1 × 0.4 kg ai/ha with no PHI specified.

In four trials conducted in the UK matching the GAP of the Netherlands, residues were < 0.09 (4).

In seven trials conducted in France, the Netherlands, Sweden and the UK, complying with Belgian GAP, residues were: < 0.09 (3), 0.21, 0.25, 0.31 and 0.39 mg/kg.

In four trials conducted in Greece, Spain and Italy matching Italian GAP, residues were < 0.09 (2), 0.19 and 0.24 mg/kg.

In four trials conducted in France (south), Italy, Greece and Spain according to Spanish GAP residues were: 0.17, 0.25, 0.32 0.98, and 1.3 mg/kg

Based on the residue trials conducted according to GAP in Spain, and with the results from the other trials conducted in Europe used as supporting data, the Meeting estimated a maximum residue level of 3 mg/kg, a HR 1.3 mg/kg and a STMR of 0.285 mg/kg for cycloxydim in bulb onions.

Leek

Cycloxydim is registered for use in leeks in Portugal with a GAP of 1 × 0.4 kg ai/ha and a 42 day PHI. In Ireland, the rate is the same but no PHI is specified. In Switzerland, GAP consists of 1 × 0.6 kg ai/ha and a 56 day PHI

In six trials conducted in Belgium, Denmark, Germany and the UK matching Irish GAP, residues were 0.13, 0.28, 0.33, 0.39, 0.62 and 2.3 mg/kg.

In four trials conducted in the Netherlands according to the GAP of Switzerland, residues were 0.11, 0.12, 0.21 and 0.24 mg/kg.

In three trials conducted in southern France, Italy and Spain according to Portuguese GAP, residues were < 0.09 (2) and 0.09 mg/kg.

Based on the residue trials conducted according to the GAP in Ireland, the Meeting estimated a maximum residue level of 4 mg/kg, a HR of 2.3 mg/kg and a STMR of 0.36 mg/kg for cycloxydim in leek.

Brassica vegetables

Cycloxydim is registered in Brassica vegetables in Switzerland at 1 × 0.6 kg ai/ha and a 28 day PHI, and in Spain at 1 × 0.4 kg ai/ha with no specified PHI.

Twelve trials were conducted in Brussels sprouts in Europe at 0.5 kg ai/ha. Residues from seven trials conducted in Belgium, France (north), Germany, the Netherlands and the UK according to the GAP in Switzerland were: 1.0, 1.1, 1.8 (2), 2.0, 2.9 and 3.6 mg/kg.

Four trials conducted in France (south), Italy, Spain and Greece according to Spanish GAP gave residues of 1.9, 2.6, 3.5 and 6.0 mg/kg.

Residues in Brussels sprouts from trials matching comparable GAP in northern and southern Europe were considered similar and could be combined, were (n = 12): 1.0 (2), 1.1, 1.8 (2), 1.9, 2.0, 2.6, 2.9, 3.5, 3.6 and 6.0 mg/kg.

Fourteen trials were conducted in cabbages at 0.5–0.6 kg ai/ha. In nine trials conducted in France (north), Germany, Sweden, Belgium and the UK according to the GAP of Switzerland, residues were: < 0.09, 0.40, 0.50 (2), 0.63, 0.74, 1.0, 1.3 and 3.0 mg/kg. In five trials conducted in Spain, France (south), Greece and Italy according to Spanish GAP (sample taken at 28 days after application), residues were: 0.88, 1.0, 1.2, 1.4 and 1.7 mg/kg.

Twelve trials were conducted in Europe in cauliflower at 0.5–0.6 kg ai/ha. In eight trials conducted in Belgium, France (north), Sweden and the UK according to the GAP of Switzerland, residues were: 0.27, 0.59, 1.5 (2), 1.7, 1.9, 2.1 and 2.3 mg/kg. Four trials conducted in southern Europe did not match the GAP.

Based on the residue data for Brussels sprouts the Meeting estimated a maximum residue level of 9 mg/kg, a HR of 6 mg/kg and a STMR of 5 mg/kg for cycloxydim in Brassica (Cole or Cabbage) Vegetables, Head Cabbage and Flowerhead Brassicas

The Meeting withdrew its previous recommendations.

Peppers

Cycloxydim is registered in peppers (chili and sweet) in Italy at 1 × 0.60 kg ai/ha and a 20 day PHI. In eight trials conducted peppers in southern Europe according to this GAP, residues were: 0.68, 0.78, 1.2, 1.5, 1.6, 3.0, 3.1, and 5.3 mg/kg.

The Meeting estimated a maximum residue level of 9 mg/kg, a HR of 5.3 mg/kg and a STMR of 1.55 mg/kg for cycloxydim in peppers.

The Meeting also estimated a maximum residue level of 90 mg/kg, a HR-P of 53 mg/kg and a STMR-P of 15.5 mg/kg for cycloxydim in dried chili peppers, by applying a factor of 10 to the MRL, HR and STMR values estimated for peppers.

Tomatoes

Cycloxydim is registered in tomatoes at 1 × 0.4 kg ai/ha and a 35 day PHI in Greece, and at 0.6 kg ai/ha and a 56 day PHI in Switzerland.

In eight trials conducted in southern Europe according to Greek GAP, residues were: 0.12, 0.17, 0.25, 0.26, 0.31, 0.39, 0.43 and 0.55 mg/kg.

In eight trials conducted in northern Europe according to the GAP of Switzerland, residues were: 0.21, 0.39 (2), 0.44, 0.45, 0.46, 0.50 and 0.84 mg/kg.

Based on the residue trial population in North of Europe, the Meeting estimated a maximum residue level of 1.5 mg/kg, a HR of 0.84 mg/kg and a STMR of 0.445 mg/kg for cycloxydim in tomatoes.

Chinese cabbage

Cycloxydim is registered for brassica vegetables in Spain at 1 × 0.4 kg ai/ha with no specified PHI.

In two trials conducted in Greece and Italy according to this GAP, residues were < 0.09 and 0.23 mg/kg

The Meeting agreed that there were insufficient data complying with GAP with which to estimate a maximum residue level for cycloxydim in Chinese cabbage.

Kale

Cycloxydim is registered for brassica vegetables in Switzerland at 1 × 0.6 and a 28 day PHI and in Spain at 1 × 0.4 kg ai/ha with no specified PHI.

In four trials conducted in kale, curly in France (north), Germany and the Netherlands according to the GAP of Switzerland, residues were: 0.77, 0.90 and 1.8 mg/kg.

In four trials conducted in France (south), Greece, Italy and Spain according to Spanish GAP, residues were: < 0.09, 0.23, 0.98 and 1.1 mg/kg.

Based on the residue data from trials conducted in south of Europe, the Meeting estimated a maximum residue level of 3 mg/kg, a HR of 1.1 mg/kg and a STMR of 0.65 mg/kg for cycloxydim in kale.

Lettuce

Cycloxydim is registered in lettuce (leaf and head) in Austria at 1 × 0.5 kg ai/ha and a 14 day PHI, in France at 0.4 kg ai/ha and a 21 day PHI and in Slovenia at 0.4 kg ai/ha with a 14 day PHI. Twenty two trials were conducted in lettuce in Europe at 0.5 kg ai/ha, matching GAP rate in northern and southern Europe.

Six trials were conducted in northern Europe according to Austrian GAP, giving residues of 0.18, 0.28, 0.48, 0.65, 0.69 and 0.71 mg/kg.

Six trials were conducted in northern Europe according to French GAP, giving residues of 0.11, 0.12, 0.21, 0.28 and 0.34 (2) mg/kg.

In ten trials conducted in southern Europe according to Slovenian GAP, residues were: 0.09, 0.11, 0.24, 0.31 (2), 0.36, 0.38, 0.41 (2) and 1.0 mg/kg

Based on the residue trial population in southern Europe, the Meeting estimated a maximum residue level of 1.5 mg/kg, a HR of 1 mg/kg and a STMR of 0.335 mg/kg for cycloxydim in lettuce, head and lettuce, leaf.

The Meeting withdrew its previous recommendation of 0.2 mg/kg for cycloxydim in lettuce, head and lettuce, leaf.

Spinach

Cycloxydim is registered in spinach at 1 × 0.4 kg ai/ha in Slovenia and in France, with PHIs of 28 days and 42 days, respectively. Eight trials were conducted in Europe at 0.5 kg ai/ha.

In four trials conducted in north of Europe according to French GAP, residues were: < 0.09 (3) and 0.10 mg/kg

In four trials conducted in south of Europe according to Slovenian GAP, residues were: < 0.09, 0.19, 0.20 and 2.4 mg/kg.

The Meeting agreed that there were insufficient trials according to GAP to estimate a maximum residue level of cycloxydim in spinach.

Green beans with pods

Cycloxydim is registered in green beans at 1 × 0.6 kg ai/ha in Belgium with a 28 day PHI. In Spain, the rate is 0.4 kg ai/ha with no PHI specified.

In ten trials conducted in northern Europe according to Belgian GAP, residues were: < 0.09, 0.21, 0.22, 0.26, 0.30, 0.40, 0.64, 0.73, 1.2 and 1.3 mg/kg

In eleven trials conducted in southern Europe according to Spanish GAP, residues were: < 0.05, 0.20 (2), 0.24, 0.29, 0.35, 0.41, 0.52, 0.56, 4.4 and 11 mg/kg.

Based on the residue trial population from southern Europe (statistically higher), the Meeting estimated a maximum residue level of 15 mg/kg, a HR of 11 mg/kg and a STMR of 0.35 mg/kg for cycloxydim in beans except broad bean & soya bean (green pods & immature seeds).

The Meeting withdrew its previous recommendation of 1 mg/kg for cycloxydim in common bean (pods and/or immature seeds).

Peas, Shelled (succulent seeds)

Cycloxydim is registered in green peas at 1 × 0.5 kg ai/ha in Germany with a 35 day PHI. In Spain, the rate is up to 0.4 kg ai/ha and no PHI is specified.

In seven trials conducted in northern Europe according to German GAP, residues in peas (seeds) were: 0.80, 1.2, 1.8, 2.5, 3.2, 4.4 and 4.7 mg/kg.

In eight trials conducted in south of Europe according to Spanish GAP rate (PHI from 28 to 49 days) were: 0.45, 0.84, 2.1, 2.3, 3.1, 5.3, 5.9 and 8.5 mg/kg.

Based on the residue trials in southern Europe, the Meeting estimated a maximum residue level of 15 mg/kg, and a STMR of 2.7 mg/kg for cycloxydim in peas, shelled (succulent seeds). The Meeting withdrew its previous recommendation of 2 mg/kg for cycloxydim in peas, shelled (succulent seeds) and of 1 mg/kg in peas (pods and succulent = immature seeds).

Dry beans

Cycloxydim is registered in dry beans at 1 × 0.45 kg ai/ha in France, with no PHI specified. Twenty one trials were conducted in Europe matching this GAP.

In eight trials conducted in northern Europe, residues were: 0.51, 1.5, 2.8, 4.4 (2), 7.9, 9.8 and 15 mg/kg.

In thirteen trials conducted in southern Europe, residues were: 0.20, 0.31, 0.57, 0.70, 1.1, 2.0, 2.4, 3.0, 3.5, 3.6, 4.0, 4.5 and 6.1 mg/kg

Based on the trials conducted in northern Europe the Meeting estimated a maximum residue level of 30 mg/kg, and a STMR of 4.4 mg/kg for cycloxydim in beans, dry.

The Meeting withdrew its previous recommendation of 2 mg/kg for cycloxydim in beans (dry).

Dry peas

Cycloxydim is registered in peas at 1 × 0.60 kg ai/ha in Sweden with no PHI specified. In Italy, the rate is the same with a 60 day PHI.

In four trials conducted in the Netherlands and the UK matching the GAP rate in Sweden, residues at 55 days PHI were: 1.2, 3.4, 5.9 and 12 mg/kg.

In ten trials conducted in south of Europe according to Italian GAP, residues were: 0.30, 0.69, 0.84, 1.6 (2), 3.2 (2), 3.6, 5.5 and 5.9 mg/kg.

The Meeting estimated a maximum residue level of 30 mg/kg, and a STMR of 5.6 mg/kg for cycloxydim in peas, dry.

Soya beans

Cycloxydim is registered in soya beans at 1 × 0.40 kg ai/ha in France with a 56 day PHI. In Spain, the rate is the same, with no PHI specified,

In thirteen trials conducted in Europe at 0.5 kg ai/ha and PHI of 49–63, matching GAP in France and Spain, residues were: 0.23, 0.83, 1.2, 2.8, 3.4, 9.2, 13, 14, 26, 30 (2), 33 and 40 mg/kg.

The Meeting estimated a maximum residue level of 80 mg/kg, and a STMR of 13 mg/kg for cycloxydim in soya bean, dry.

The Meeting withdrew its previous recommendation of 2 mg/kg for cycloxydim in soya beans.

Carrots

Cycloxydim is registered in carrots in Belgium at a single application up to 0.60 kg ai/ha and in Portugal at 0.40 kg ai/ha, with a PHI of 28 days. Eleven trials were conducted in Europe at 0.50 kg ai/ha, matching both the GAP rates of Belgium and Portugal.

Residues, at the 28 day PHI, from northern European trials according to Belgian GAP were: 0.32, 0.42, 0.44 and 0.64 mg/kg.

Trials from southern Europe, according to Portuguese GAP at the 28 day PHI, gave residues of: 0.18, 0.29, 0.33, 0.44, 0.47, 1.1 and 3.0 mg/kg.

Based on the residue trials from southern Europe, the Meeting estimated a maximum residue level of 5 mg/kg, a HR of 3 mg/kg and a STMR of 0.44 mg/kg for cycloxydim in carrots.

The Meeting withdrew its previous recommendation of 0.5 mg/kg for cycloxydim in carrots.

Celeriac

Cycloxydim is registered in celeriac in France at 1× 0.60 kg ai/ha and a 48 day PHI.

In eight trials conducted in Europe according to this GAP, residues were: 0.10, 0.12, 0.13 (3), 0.14, 0.19 and 0.64 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, a HR of 0.64 mg/kg and a STMR of 0.13 mg/kg for cycloxydim in celeriac.

Potatoes

Cycloxydim is registered in potatoes at 1 × 0.60 kg ai/ha in Belgium and the Netherlands with a 56 day PHI. In Italy, the rate is the same with a 100 day PHI. In ten trials conducted in northern Europe according to Belgian GAP, residues were: 0.31, 0.41, 0.55, 0.65, 0.72, 0.75, 0.79, 1.0, 1.2 and 1.6 mg/kg.

Seven trials conducted in southern Europe, according to Italian GAP, residues were: < 0.09 (2), 0.10, 0.21, 0.27, 0.44 and 0.46 mg/kg.

Based on the residue trials in north of Europe, the Meeting estimated a maximum residue level of 3 mg/kg, a HR of 1.6 mg/kg and a STMR of 0.735 mg/kg for cycloxydim in potatoes.

The Meeting withdrew its previous recommendation of 2 mg/kg for cycloxydim in potatoes.

Turnips

From six trials on turnips conducted in Norway at a rate of 0.6 kg ai/ha residues in turnip roots 77 to 103 days post application were: < 0.09 to 0.13 mg/kg. The GAP rate in Europe is up to 0.4 kg ai/ha.

As no trials were conducted according to GAP, the Meeting did not estimate a maximum residue level for cycloxydim in turnips.

Sugar beet

Cycloxydim is registered in sugar beet at 1 × 0.50 and 0.60 kg ai/ha in Germany and the Netherlands, respectively, no PHI specified. In Italy, the rate is 0.60 kg ai/ha and 100 days PHI. GAP for swede in THE UK is 0.45 kg ai/ha with 56 days PHI and for beetroot in Switzerland is 0.60 kg ai/ha with 56 days PHI.

In ten trials conducted in north of Europe according to German GAP, residues were < 0.09 (9) and 0.10 mg/kg.

In eight trials conducted in south of Europe according to Italian GAP, residues were < 0.09 (8) mg/kg.

Based on the residue trials in north of Europe, the Meeting estimated a maximum residue level of 0.2 mg/kg, a HR of 0.1 mg/kg and a STMR of 0.09 mg/kg for cycloxydim in sugar beet.

The Meeting agreed to extrapolate these estimations to beetroot and swede.

The Meeting confirms its previous recommendation of 0.2 mg/kg for cycloxydim in sugar beet.

Maize

Cycloxydim is registered in Germany for use in maize at 1 × 0.40 kg ai/ha (no PHI specified) and France (90 days PHI).

In six trials conducted in northern Europe, matching German GAP, residues were: < 0.09 (5) and 0.12 mg/kg.

In eight trials conducted in southern Europe, according to French GAP, residues were: < 0.09 (8) mg/kg.

Based on trials conducted in northern Europe, the Meeting estimated a maximum residue level of 0.2 mg/kg, and a STMR of 0.09 mg/kg for cycloxydim in maize grain.

Rice

Cycloxydim is registered in rice in Italy at 1 × 0.40 two days before sowing, with no PHI specified. In 11 trials conducted in the country according to GAP, residues found in grain, 133 to 162 days after treatment, were: < 0.09 (11) mg/kg.

The Meeting estimated a maximum residue level of 0.09* mg/kg, and a STMR of 0.09 mg/kg for cycloxydim in rice.

Rape seed

Cycloxydim is registered in rape seed at 1 × 0.60 kg ai/ha in Italy, with a PHI of 100 days. In Germany the rate is 0.50 kg ai/ha with no PHI specified.

In nine trials conducted in northern Europe, according to German GAP, residues at PHIs from 85 to 100 days PHI were: 0.77, 1.0, 1.5, 1.6, 1.8, 1.9, 2.2, 2.5 and 5.3 mg/kg

In six trials conducted in southern Europe, according to Italian GAP residues were: 0.54, 1.6, 1.8, 2.8, 3.1 and 4.0 mg/kg

The fifteen trials conducted according to the same GAP in the south and north of Europe belonged to the same residue population and were combined: 0.54, 0.77, 1.0, 1.5, 1.6 (2), 1.8, 1.9, 2.2, 2.5, 2.8 (2), 3.1, 4.0 and 5.3 mg/kg.

The Meeting estimated a maximum residue level of 7 mg/kg, and a STMR of 1.9 mg/kg for cycloxydim in rape seed.

The GAP for linseed in Sweden is 0.6 kg ai/ha with no PHI specified. The Meeting agreed to extrapolate the rape seed estimates to linseed.

The Meeting withdrew its previous recommendation of 2 mg/kg for cycloxydim in rape seed.

Sunflower

Cycloxydim is registered in sunflower at 1 × 0.60 kg ai/ha in Italy, with a PHI of 80 days. In Germany the rate is 0.50 kg ai/ha with a 100 day PHI.

In four trials conducted in northern Europe according to German GAP, residues were: < 0.09, 0.37, 0.38 and 2.8 mg/kg.

In 15 trials conducted in southern Europe, according to Italian GAP, residues were: < 0.09 (4), 0.09, 0.12, 0.14, 0.25, 0.28, 0.37, 0.38, 0.39, 0.50, 0.94 and 1.8 mg/kg.

Based on the data coming from northern Europe, and with the support of the other trials conducted in Europe, the Meeting estimated a maximum residue level of 6 mg/kg, and a STMR of 0.375 mg/kg for cycloxydim in sunflower seed.

Feed commodities

Maximum residue levels will not be estimated for forage commodities as it is understood that the international trade of such commodities is unlikely. Highest residue and/or STMR will be estimated for commodities listed in the OECD feeding table for animal dietary burden calculation purposes.

Bean vines

Residues of cycloxydim in bean vine (whole plant or rest of the plant) from trials conducted in northern Europe, according to GAP (0.4–0.45 kg ai/ha, no PHI specified), were (n = 7): 0.3, 0.84, 0.9, 0.99, 1.2, 1.4 and 1.5 mg/kg.

Residues of cycloxydim in bean vine from trials conducted in southern Europe, according to GAP, were (n = 9): 0.34, 0.43, 0.67, 0.71, 0.77, 1.2 (2), 1.3 and 2.0 mg/kg.

Based on the southern European trials, which gave the highest residues, the Meeting estimated a highest residue of 2 mg/kg and a STMR of 0.77 mg/kg for cycloxydim in bean vines.

Pea vines

Residues of cycloxydim in pea vine (whole plant or without the seed, or rest of the plant) from trials conducted in northern Europe, according to GAP (0.6 kg ai/ha, no PHI specified) were (n = 8): 0.8, 0.93, 2.4, 2.5, 2.6, 3.5, 3.6 and 3.9 mg/kg.

Residues of cycloxydim in pea vine from trials conducted in southern Europe, according to GAP (0.6 kg ai/ha, 60 day PHI or 0.4 kg ai/ha no PHI specified), were (n = 17): 0.14, 0.16, 0.21, 0.24, 0.27, 0.45, 1.1, 1.8 (2), 2.1, 2.2, 2.3, 5.5, 5.9, 6.1, 8.4 and 9.0 mg/kg.

Based on the southern European trials, which gave the highest residues, the Meeting estimated a highest residue of 9 mg/kg and a STMR of 1.48 mg/kg for cycloxydim in pea vines.

The Meeting also recommends a maximum residue level of 60 mg/kg for pea vines (dry) (25% DM).

Sugar beet leaves or tops

In ten trials conducted in northern Europe, according to GAP (0.5 kg ai/ha, no PHI specified), residues in the leaves (tops) were: < 0.09 (6), 0.09, 0.16, 0.33 and 0.50 mg/kg.

In six trials conducted in southern Europe according to GAP (0.6 kg ai/ha, 100 day PHI), residues were: < 0.09 (6) mg/kg.

Based on the northern European trials, the Meeting estimated a highest residue of 0.50 mg/kg and a STMR of 0.09 mg/kg for cycloxydim in sugar beet leaves or tops.

Maize fodder

In ten trials conducted with cycloxydim in northern Europe according to GAP (0.4 kg ai/ha, no PHI specified), residues in fodder were: < 0.09 (7), 0.11, 0.3 and 0.41 mg/kg.

In four trials conducted in south of Europe according to GAP (0.4 kg a.i./ha, 90 days PHI), residues were 0.10, 0.11, 0.29 and 1.1 mg/kg

Based on the trials conducted in southern Europe and with the support of the trials conducted in northern Europe the Meeting estimated a highest residue of 1.1 mg/kg and a STMR of 0.247 mg/kg for cycloxydim in maize fodder.

The Meeting also estimated a maximum residue level of 2 mg/kg in maize fodder, dry (85% DM).

Rice straw and fodder

In eight trials conducted with cycloxydim in Italy, according to GAP, residues in rice straw were: < 0.09 (8) mg/kg.

The Meeting estimated a maximum residue level of 0.09* mg/kg, a HR of 0.09 mg/kg and a STMR of 0.09 mg/kg for cycloxydim rice straw (DM=90%).

Rape forage

In three trials conducted in Norway according to GAP, residues in rape seed forage were 0.24, 0.25 and 0.26 mg/kg.

The Meeting agreed that three trials according to GAP was sufficient to estimate a highest residue for cycloxydim in rape forage.

Fate of residues in processing

The [^{14}C]-cycloxydim was dissolved in aqueous buffer solution at pH 4 and heated for 20 minutes at 90 °C to simulate pasteurization, at pH 5 and refluxed at 100 °C for 60 minutes to simulate baking, brewing and boiling, and at pH 6 at about 120 °C in an autoclave for 20 minutes to simulate pasteurization. Cycloxydim degraded mainly to cycloxydim-T2S, which accounted for 93.5, 86.8 and 75% of the total applied radioactivity (TAR), respectively. T2SO accounted for up to 11% TAR (at pH 6).

A variety of processing studies were conducted with crops treated with cycloxydim. Processing factors (PF) in commodities with relevance for dietary exposure assessment and for animal dietary burden calculation are shown in the Table below. The estimated PFs were multiplied by the estimated HR and STMR of the raw commodity to estimate the HR-P and STMR-P for the processed commodity.

Processing factor (PF) and estimations for processed commodities

Commodity	Best estimate PF (n)*	STMR-P, mg/kg	HR-P, mg/kg
Strawberry, STMR= 0.53 mg/kg, HR=1.4 mg/kg			
Strawberry jam	0.55 (4)	0.291	
Strawberry canned	0.90 (4)	0.447	1.26
Onion, STMR=0.285 mg/kg, HR= 1.3 mg/kg			
Onion, peeled	1.1 (2)	0.31	1.43
Cabbage, STMR=1.95 mg/k, HR= 9.0 mg/kg			
Cabbage, cooked	0.56 (4)	1.09	5.04
Pasteurized sauerkraut	0.78 (4)	1.17	
Tomato, STMR=0.445mg/kg , HR= 0.89 mg/kg			
Tomato, canned	0.57 (4)	0.254	0.51
Tomato juice	1.1 (4)	0.49	

Commodity	Best estimate PF (n)*	STMR-P, mg/kg	HR-P, mg/kg
Ketchup	1.8 (4)	0.801	
Tomato pure, pasteurized	3.7 (4)	1.65	
Pea, STMR=5.6 mg/kg			
Pea, cooked	0.7 (4)	3.92	
Pea, canned	0.2 (4)	1.12	
Carrot, STMR= 0.44 mg/k, HR= 3.0 mg/kg			
Carrot, cooked	0.77 (4)	0.339	2.31
Carrot, juice	0.50 (4)	0.22	
Carrot, canned	0.36 (4)	0.158	1.08
Potato, STMR= 0.735 mg/kg, HR= 1.6 mg/kg			
Potato, peeled	1.3 (4)	0.960	2.08
Potato, boiled	1.5 (4)	1.10	2.4
Potato, steamed	1 (4)	0.735	1.6
French fries	1.3 (4)	0.956	2.08
Rape seed, STMR= 1.9 mg/kg			
Rape oil, refined	< 0.05 (4)	0.095	
Rape oil meal	1.5 (6)	2.85	
Sunflower, STMR=0.05 mg/kg			
Sunflower oil	0.1 (2)	0.00	

[a] number of processing studies

Residues in animal commodities

Farm animal dietary burden

The Meeting estimated the dietary burden of cycloxydim in farm animals on the basis of the diets listed in Annex 6 of the 2006 JMPR Report (OECD Feedstuffs Derived from Field Crops), the STMR, STMR-Ps or highest residue levels estimated at the present Meeting (see Table below). Dietary burden calculations are provided in Annex 6 of the 2012 JMPR Report.

Livestock dietary burden for cycloxydim, ppm of dry matter diet

	US-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	3.54	2.09	22.4	7.26	26.8[a]	8.5 [c]	2.28	2.28
Dairy cattle	8.1	2.96	22.6[b]	7.55[d]	20.4	7.0	1.55	1.55
Poultry - broiler	3.47	3.47	6.06	3.92	5.1	5.1	0.07	0.07
Poultry - layer	3.47	3.47	10.89 [e]	4.32	5.1[f]	5.1	0.08	0.08

[a] Highest maximum beef or dairy cattle dietary burden suitable for maximum residue level estimated for mammalian tissues

[b] Highest maximum dairy cattle dietary burden suitable for maximum residue level estimated for mammalian milk

[c] Highest mean beef or dairy cattle dietary burden suitable for STMR estimated for mammalian tissues.

[d] Highest mean dairy cattle dietary burden suitable for STMR estimated for milk.

[e] Highest maximum poultry dietary burden suitable for maximum residue level estimated for poultry tissues and eggs.

[f] Highest mean poultry dietary burden suitable for STMR estimated for poultry tissues and eggs.

Animal feeding studies

Cattle

A mixture of cycloxydim and cycloxydim-5-OH-TSO (2:1) was administered orally to cattle for 28 days at 5.1, 15.2 and 50.2 ppm feed levels. Residues (sum of non-hydroxylated and hydroxylated metabolites; expressed as parent equivalents) in milk was only detected at the highest dose, with a

mean of 0.044 mg/kg. Mean residues in skim milk and cream (from day 21) were similar (0.044 and 0.033 mg/kg, respectively).

In muscle, residues were not detected at the lowest dose. Mean and highest residues were 15.2 ppm of 0.023 and 0.026 mg/kg, respectively. Mean and highest residues at 50.2 ppm were 0.073 and 0.088 mg/kg, respectively

In liver, mean residues were 0.043, 0.128 and 0.336 mg/kg were at feeding levels of 5.1, 15.2 and 50.2 ppm, respectively, with the highest residues of 0.045, 0.151 and 0.381 mg/kg, respectively. Residue 2 days after the dose withdrawal was 0.079 mg/kg.

In kidney, mean residues were 0.068, 0.202 and 0.593 mg/kg at feeding levels of 5.1, 15.2 and 50.2 ppm, respectively, with the highest residues of 0.073, 0.239 and 0.727 mg/kg, respectively. The residue 2 days after dose withdrawal was 0.057 mg/kg.

In fat, mean residues were < 0.019, 0.025 and 0.119 mg/kg at feeding levels of 5.1, 15.2 and 50.2 ppm, respectively, with the highest residues of < 0.019, 0.030 and 0.138 mg/kg, respectively. The residue 2 days after dose withdrawal was 0.020 mg/kg.

In another study conducted at the same dose levels, residues in milk (total, skin and cream), muscle and fat were only detected at the highest dose: mean of 0.020 mg/kg in milk and skim milk and 0.016 mg/kg in cream; mean and highest in muscle of 0.06 and 0.07 mg/kg, respectively and in fat of 0.10 and 0.12 mg/kg.

In liver, mean residues were 0.03, 0.12 and 0.29 mg/kg at 5, 15 and 50 mg/kg dose levels, respectively, with the highest residues of 0.04, 0.15 and 0.31 kg/kg, respectively. The residue 2 days after dose withdrawal was 0.06 mg/kg.

In kidney, mean residues were 0.05, 0.14 and 0.44 mg/kg at 5, 15 and 50 mg/kg dose levels, respectively, with the highest residues of 0.06, 0.18 and 0.51 kg/kg, respectively. The residue 2 days after dose withdrawal was 0.05 mg/kg.

Poultry

A mixture of cycloxydim and cycloxydim-OH-TSO (1:1) was administered orally to groups of hens for 28 days at doses of 2.29, 6.71 and 23.2 ppm in the feed. Mean and highest residues in eggs detected at 6.71 ppm dose were 0.022 and 0.058 mg/kg, respectively (n = 9). At the highest dose, mean and highest values were 0.065 and 0.102 mg/kg, respectively (n = 9). Residues were < 0.02 mg/kg during the depuration phase (between 29 and 33 days). Residues were not detected above the LOQ in muscle and fat in any dose group. In liver, residues were detected only at the highest dose group (mean of 0.022 mg/kg and highest of 0.03 mg/kg).

In another study, cycloxydim and cycloxydim-OH-TSO (1:1) was administered to laying hens at a target dose level of 2.5, 7.5 and 25 ppm. Residues were not detected in muscle, liver and fat at any dose level. In eggs, residues were detected at the 7.5 ppm (mean of < 0.03 mg/kg, highest of 0.041 mg/kg) and at the 25 mg/kg dose (mean of 0.046 mg/kg, highest of 0.069 mg/kg). Residues during the depuration phase (3–7 days) were < 0.03 mg/kg.

Animal commodity maximum residue levels

The residues expected in animal commodities based on the calculated animal burden and the feeding studies are shown in Table 3. The levels which the estimations were based are in bold.

Residues in kidney and liver at the expected dietary burden are outlined below.

	Feed level, ppm, for		Residue, mg/kg					
	Milk residues	Tissues and eggs residues	Milk	Muscle	Liver	Kidney	Fat	Eggs
Highest residue level, cattle								
Feeding study	50	15 50	0.032	0.026 0.088	0.151 0.381	0.239 0.727	0.030 0.138	
Burden and	22.6	26.8	0.014	0.047	0.228	0.40	0.066	

	Feed level, ppm, for		Residue, mg/kg					
	Milk residues	Tissues and eggs residues	Milk	Muscle	Liver	Kidney	Fat	Eggs
residue								
STMR, cattle								
Feeding study	50	5 15	0.032	< 0.019 0.026	0.036 0.124	0.059 0.171	< 0.019 0.027	
Burden and residue	7.55	8.5	0.0054	0.021	0.067	0.0984	0.022	
Highest residue level, hens								
Feeding study		6.7/7.5 23.2		< 0.02	0.03		< 0.02/< 0.03	0.058/0.041
Burden and residue		10.8		< 0.03	0.014		< 0.03	0.092/0.023
STMR, hens								
Feeding study		6.7/7.5 23.5		< 0.02	0.022		< 0.03	0.022/0.03
Burden and residue		5.1		0	0.0054		0	0.02/0.017

Based on the results obtained for cattle, the Meeting estimated for cycloxydim a maximum residue level of 0.02 mg/kg and a STMR of 0.005 mg/kg in milks; a maximum residue level of 0.06 mg/kg, a HR of 0.047 mg/kg and a STMR of 0.021 mg/kg in meat (from mammalian other than marine mammals); a maximum residue of 0.1 mg/kg, a HR of 0.066 mg/kg and a STMR of 0.021 mg/kg for mammalian fats (except milk fats); and a maximum residue level of 0.5 mg/kg, a HR of 0.403 mg/kg and a STMR of 0.098 mg/kg in edible offal (mammalian).

Based on the results obtained for hens, the Meeting estimated for cycloxydim a maximum residue level of 0.15 mg/kg, a HR of 0.092 mg/kg, and a STMR of 0.018 mg/kg in eggs; a maximum residue level of 0.03* mg/kg, a HR of 0.03 mg/kg and a STMR of 0 mg/kg in poultry meat and poultry fats; and a maximum residue level of 0.02 mg/kg, a HR of 0.014 mg/kg and a STMR of 0.005 mg/kg for cycloxydim in poultry edible offal.

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 compliance with maximum residue levels and estimation of dietary intake in plant and animal commodities: *Cycloxydim, metabolites and degradation products which can be oxidized to 3-(3-thianyl) glutaric acid S-dioxide and 3-hydroxy-3-(3-thianyl) glutaric acid S-dioxide, expressed as cycloxydim*.

The residue is not fat-soluble

CCN	Commodity name	Maximum residue level (mg/kg)		STMR (P) mg/kg	HR (P) mg/kg
		New	Previous		
VD 0071	Beans, dry	30	2	4.4	
VP 0061	Beans except broad bean & soya bean (green pods & immature seeds)	15		0.35	11
VR 0574	Beetroot	0.2		0.09	0.10
VB 0040	Brassica (Cole or Cabbage) Vegetables, Head Cabbage, Flowerhead Brassicas	9	2	1.95	6
VR 0577	Carrot	5	0.5	0.44	3
VR 0578	Celeriac	1		0.13	0.64
MO 0105	Edible offal (Mammalian)	0.5		0.098	0.403
PE 0112	Eggs	0.15		0.018	0.092
FB 0269	Grapes	0.3	0.5	0.11	0.18

CCN	Commodity name	Maximum residue level (mg/kg)		STMR (P) mg/kg	HR (P) mg/kg
		New	Previous		
VL 0480	Kale	3		0.65	1.1
VA 0384	Leek	4		0.36	2.3
VL 0482	Lettuce, head	1.5	0.2	0.335	1
VL 0483	Lettuce, leaf	1.5	0.2	0.335	1
SO 0693	Linseed	7		1.9	
MF 0100	Mammalian fats (except milk fats)	0.1		0.021	0.066
GC 0645	Maize	0.2		0.09	
AS 0645	Maize fodder	3		0.247	1.1
MM 0095	Meat (from mammals other than marine mammals)	0.06		0.021	0.047
ML 0106	Milks	0.02		0.005	
VA 0385	Onion, bulb	3		0.31	1.43
VP 0063	Peas (pods and succulent=immature seeds)	W	1		
VP 0072	Pea, dry	30		5.6	
VP 0064	Peas, Shelled (succulent seeds)	15	2	2.7	
VO 0051	Peppers	9		1.55	5.3
	Peppers, chilli dried	90		15.5	53
FP 009	Pome fruits	0.09*		0.09	0.09
VR 0589	Potato	3	2	0.735	1.6
PM 0110	Poultry meat	0.03*		0	0.03
PF 0111	Poultry fats	0.03*		0	0.03
PO 0111	Poultry, edible offal of	0.02		0.005	0.014
SO 0495	Rape seed	7	2	1.9	
GC 0649	Rice	0.09*		0.09	
AS 0649	Rice straw or fodder	0.09		0.09	0.09
VD 4521	Soya bean (dry)	80	2	13	
FS0012	Stone fruits	0.09*		0.09	0.09
FB 0275	Strawberry	3	0.5	0.53	1.4
VR 0596	Sugar beet	0.2	0.2	0.09	0.10
SO 0702	Sunflower seed	6		0.375	
VR 0497	Swede	0.2		0.09	0.10
VO 0448	Tomato	1.5		0.445	0.84

Commodities for which no maximum residue levels were recommended

Commodity name	STMR, mg/kg	Highest residue, mg/kg
Bean vines	0.77	2
Pea vines	1.8	9.0
Rape seed meal	2.85	
Sugar beet leaves	0.09	0.50

DIETARY RISK ASSESSMENT

Long-term intake

The ADI for cycloxydim is 0–0.07 mg/kg bw. The International Estimated Daily Intakes (IEDI) for cycloxydim was estimated for the 13 GEMS/Food cluster diets using the STMR or STMR-P values estimated by the current JMPR. The results are shown in Annex 3 of the 2012 JMPR Report. The IEDI ranged from 7–50% of the maximum ADI. The Meeting concluded that the long-term intake of residues of cycloxydim from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

An ARfD for cycloxydim for women of childbearing age is 2 mg/kg bw; ARfD was unnecessary for the general population. The International Estimated Short-Term Intake (IESTI) for cycloxydim was calculated for the plant commodities for which STMRs, HRs and maximum residue levels were

estimated by the current Meeting and for which consumption data were available. The results are shown in Annex 4 of the 2012 JMPR Report. The IESTI represented a maximum of 10% of the ARfD for peppers, chili dried. The Meeting concluded that the short-term intake of cycloxydim residues from uses considered by the current Meeting was unlikely to present a public health concern.

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1009861	Kroehl, T	2008a	UV/VIS spectrum in basic medium of Cycloxydim PAI (Reg. No. 172 999, BAS 517 H) BASF SE; Limburgerhof; Germany. GLP, unpublished
11669	Pawliczek, JB	1988a	Determination of the solubility of Cycloxydim in water at various pH values. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1021518	Class, T	2008a	Cycloxydim (BAS 517 H): Determination of solubility in water at pH 4, pH 7 and pH 9. PTRL Europe GmbH; Ulm; Germany. GLP, unpublished
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1002594	Anonymous	1997a	MT 181 solubility in organic solvents. CIPAC—Collaborative International Pesticides Analytical Council; Harpenden Hertfordshire AL5 2HG; United Kingdom. No GLP, unpublished
10545	Redeker, J	1988a	Partition coefficient of Cycloxydim in the system n-octanol/water BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1090871	Hassink, J	2009a	Aqueous hydrolysis of BAS 517 H. BASF SE; Limburgerhof; Germany. GLP, unpublished
1000143	von Goetz, N	2000a	Aqueous photolysis of Cycloxydim (BAS 517 H). BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
10557	Redeker, J	1988b	Determination of the dissociation constant of Cycloxydim in water, BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
10319	Sarafin, R	1991a	Photochemical oxidative degradation of Cycloxydim (Atkinson) BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
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1020044	Leibold & Hofmann	2001a	^{14}C-BAS 517 H—Absorption, distribution and excretion after repeated oral administration in lactating goats. BASF AG; Ludwigshafen/Rhein; Germany. GLP, unpublished
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1013374	Tilting	2003b	Report amendment No. 01 to final report: The metabolism of ^{14}C-BH 517-5-OH-TSO (metabolite of BAS 517 H) in lactating goats. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1004486	Tilting	2003a	The metabolism of ^{14}C-BH 517-5-OH-TSO (metabolite of BAS 517 H) in lactating goats. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
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1004487	Fabian & Knoell	2003a	The metabolism of ^{14}C-BAS 517 H (Cycloxydim) in laying hens. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
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1004488	Seiferlein	2003a	The metabolism of ^{14}C-BH 517-5-OH-TSO (Reg. No. 217 383), a metabolite of BAS 517 H, in laying hens. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
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0445	Huber & Schepers	1986a	Investigations of the metabolism of BAS 517 H in sugar beets. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. No GLP, unpublished
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10990	Beutel	1987b	BAS 517 H—Accountability in canola. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
10989	Beutel	1987c	BAS 517 H—Accountability in soy beans. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
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10446	Beutel	1988a	Cycloxydim—Total method GC final determination. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
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1007914	Tilting	2002a	Validation of method 263/3: GC method for the determination of Cycloxydim (BAS 517 H) and its metabolites in various plant matrices. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
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1012387	Bross & Lehmann	2000a	Independent lab validation of method 263 for the determination of Cycloxydim (BAS 517 H) and its metabolites in plant matrices. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. unpublished
10774	Tillkes	1993a	Validation of BASF-method 263 resp 263/1 for the residue determination of Cycloxydim and its metabolite 5-OH-TSO2 in scorzonera and chicory. Dr. Specht & Partner Chemische Laboratorien GmbH; Hamburg; Germany. GLP, unpublished
10068	Schulz	1995a	Determination of residues of Cycloxydim in plant material and oil—Validation of the method. Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
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1030173	Grosshans	2009a	BASF method 263 for the determination of Cycloxydim (BAS 517 H) and its metabolites in various plant matrices: Summary of recovery data generated during analysis of sample material from supervised residue trials BASF SE; Limburgerhof; Germany. No GLP, unpublished
10830	Sasturain	1997b	Validation of BASF method 407/0: Determination of Cycloxydim (Reg. No. 172 999) in onion matrices using HPLC-MS/MS determination. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1000953	Lehmann & Mackenroth	2003b	Validation of the analytical method 407/1: Method for the determination of BAS 517 H and its metabolite BH 517-5-OH-TSO2 in plant matrices. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1035849	Richter	2011a	Validation of BH 517-TSO, BH 517-T1SO, BH 517-T2SO and BH 517-5-OH-TSO with BASF Method No. L0018/01 in plant matrices. BASF SE; Limburgerhof; Germany. GLP, unpublished
1019855	Jones	2006b	Validation study of the SOP-PA.0271 for determination of Cycloxydim and its metabolite residues in tomato, pepper, bean, grape, sunflower seed, green peas, soya bean, kale, onion, carrot, strawberry, lettuce and spinach. BASF SA; Resende; Brazil. GLP, unpublished
1000949	Bross & Mackenroth	2003b	Validation of the analytical method 493/0: Determination of BH 517-TSO (Lab 211 725) in plant matrices by LC/LC/UV. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1001416	Schylz	2003b	Determination of BH 517-TSO (LAB 211725) in plant matrices by LC/LC/UV—Validation of the method no. 493/0. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
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1004482	Grosshans	2003b	Validation of BASF method 513/0: The determination of BH 517-TSO, BH 517-5-OH-TSO and BH 517-5OH-TS (metabolites of BAS 517 H) in animal matrices. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1004483	Class	2002b	Independent laboratory validation (ILV) of BASF method No. 513/0 for the determination of BH 517-TSO, BH 517-5-OH-TSO and BH 517-5-OH-TS (metabolites of BAS 517 H) in animal matrices. PTRL Europe GmbH; Ulm; Germany. GLP, unpublished

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1025505	Richter	2011b	Validation of BH 517-TSO, BH 517-T1SO, BH 517-TSO2, BH 517-T2SO, BH 517-T1SO2, BH 517-5-OH-TSO and BH 517-5-OH-TS with BASF Method No. L0105/01 in animal matrices. BASF SE; Limburgerhof; Germany.. GLP, unpublished
7019237	Zhang	2009b	Independent laboratory validation of BASF method number L0105/01: Method for the determination of Cycloxydim (BAS 517 H, Reg. No. 172999) in animal matrices. Alliance Pharma Inc.; Malvern PA; United States of America. GLP, unpublished
11454	Tilting	1992a	Two years storage stability study of Cycloxydim metabolites. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1004113	Lehmann & Mackenroth	2003a	Investigation of the storage stability of BAS 517 H (Cycloxydim) residues in plant matrices under usual storage conditions. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1090653	Grosshans & Kloeppner	2009a	Stability of BAS 517 H and its metabolites in representative samples generated during animal metabolism studies. BASF SE; Limburgerhof; Germany. GLP, unpublished
1020716		2007a	Study on the residue behavior of Cycloxydim in apple and pear after soil application of BAS 517 24 H under field conditions in Italy and Spain, 2006 Agrologia SL; Palomares; Spain. GLP, unpublished
11589	Specht, W	1993b	Residues of Cycloxydim in apricots. Dr. Specht & Partner Chemische Laboratorien GmbH; Hamburg; Germany. GLP, unpublished
1991/11588	Specht W.	1993a	Residues of Cycloxydim in peaches. Dr. Specht & Partner Chemische Laboratorien GmbH; Hamburg; Germany. GLP, unpublished
1020717	Schroth, E & Martin, T	2007b	Study on the residue behavior of Cycloxydim in grape after soil application of BAS 517 24 H under field conditions in France (North and South), Germany, Greece, Italy, and Spain, 2006. Agrologia SL; Palomares; Spain. GLP unpublished
1030352	Schulz, H	2006a	Study on the residue behaviour of Cycloxydim and its metabolites in grapes after soil treatment with BAS 517 24 H under field conditions in France (N & S), Germany, Italy and Greece, 2005. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1069375	Schaeufele, M	2009a	Residue study (decline) with BAS 517 24 H applied to strawberries (field) in the UK, Germany and the Netherlands in 2008. Huntingdon Life Sciences Ltd.; Huntingdon Cambridgeshire PE28 4HS; United Kingdom. GLP, unpublished
1020730	Schroth, E & Martin, T	2007c	Study on the residue behavior of Cycloxydim in strawberry after the application of BAS 517 24 H under field conditions in France (South), Greece, Italy and Spain, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1102127	Schroth, E	2009c	Amendment No. 1: Study on the residue behavior of Cycloxydim in strawberry after the application of BAS 517 24 H under field conditions in France (South), Greece, Italy and Spain, 2006. Agrologia SL; Palomares; Spain. GLP unpublished
1026946	Schroth, E	2006a	Study on the residue behavior of Cycloxydim (BAS 517 H) in strawberries after the application of BAS 517 24 H under field conditions in France, Greece, Italy and Spain, 2005. Agrologia SL; Palomares; Spain. GLP, unpublished
10554	Beck, J & Sasturain, J	1997a	Study on the residue behaviour of Cycloxydim in strawberries after treatment with Laser (BAS 517 01 H) under field conditions in Great Britain, 1995 BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP. unpublished
12145	Regenstein, H	1992a	Residues of Cycloxydim in strawberries. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
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1007586	Perez, J & Lehmann, A	2004a	Study on the residue of Cycloxydim after post-emergency treatment with BAS 517 24 H in onions under field test conditions in Spain, 2001. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1001289	Schulz, H	2003a	Determination of the residues of Cycloxydim and its metabolite in onions and processed products following treatment with BAS 517 24 H under field conditions in Italy and Greece 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished

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10561	Beck, J & Sasturain, J	1997b	Study on the residue behaviour of BAS 517 H (Cycloxydim) in salad-onions after treatment with Laser (BAS 517 01 H) under field conditions in Great Britain, 1995. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1075171	Oxspring, S	2009e	Study on the residue behaviour of BAS 517 H in leek after treatment with BAS 517 24 H under field conditions in Northern Europe during 2007. Agrisearch UK Ltd.; Melbourne Derbyshire DE73 8AG; United Kingdom. GLP, unpublished
1020726	Schroth, E & Martin, T	2007m	Study on the residue behavior of Cycloxydim in leek after the application of BAS 517 24 H under field conditions in France (South), Greece, Italy and Spain, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
12153	Regenstein, H	1992a	Residues of Cycloxydim in leeks—BAS 517 01 H. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
12146	Regenstein, H	1992b	Residues of Cycloxydim in leeks. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1020721	Schroth, E & Martin, T	2007j	Study on the residue behavior of Cycloxydim in head cabbage after the application of BAS 517 24 H under field conditions in Belgium, France (North), Germany and United Kingdom, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1007718	Jones, S	2002a	Study on the residue behaviour of BAS 517 H in cauliflower and cabbages after application of BAS 517 24 H under field conditions in Spain, France (S), Italy, Greece, 2001.BASF plc; Gosport Hampshire PO13 0AU; United Kingdom. GLP, unpublished
1004111	Trewhitt, J *et al.*	2002a	Study on the residue behaviour of Cycloxydim in white cabbage and cauliflower after application of BAS 517 24 H under field conditions in Belgium, France (N), United Kingdom and Sweden, 2001. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1012095	Trewhitt, J *et al.*	2002b	Report amendment No. 1 to final report: Study on the residue behaviour of Cycloxydim in white cabbage and cauliflower after application of BAS 517 24 H under field conditions in Belgium, France (N), United Kingdom and Sweden, 2001. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1020722	Schroth, E & Martin, T	2007i	Study on the residue behavior of Cycloxydim in Brussels sprouts after the application of BAS 517 24 H under field conditions in France (North and South), Germany, Netherlands, Spain and United Kingdom, 2006 Agrologia SL; Palomares; Spain. GLP, unpublished
1034211	Schulz, H	2007a	Study on the residue behaviour of Cycloxydim and its metabolite in Brussels sprouts (field) after treatment with BAS 517 24 H under field conditions in France (N), England, Sweden, Belgium, Italy and Greece, 2005. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1020720	Schroth, E & Martin, T	2007h	Study on the residue behavior of Cycloxydim in cauliflower after the application of BAS 517 24 H under field conditions in France (North), Germany, Sweden and United Kingdom, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1075174	Oxspring, S	2009d	Study on the residue behaviour of BAS 517 H in curly kale and Chinese cabbage after treatment with BAS 517 24 H under field conditions in Northern and Southern Europe during 2007. Agrisearch UK Ltd.; Melbourne Derbyshire DE73 8AG; United Kingdom. GLP, unpublished
1020723	Schroth, E & Martin, T	2007k	Study on the residue behavior of Cycloxydim in curly kale after the application of BAS 517 24 H under field conditions in France (North and South), Netherlands and Spain 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1020719	Schroth, E & Martin, T	2007g	Study on the residue behavior of Cycloxydim in pepper after the application of BAS 517 24 H under field conditions in France (South), Italy and Spain 2006 Agrologia SL; Palomares; Spain. GLP, unpublished
1025871	Schroth, E	2006c	Study on the residue behavior of Cycloxydim (BAS 517 H) in peppers after the application of BAS 517 24 H under field conditions in Greece, Italy and Spain, 2005. Agrologia SL; Palomares; Spain. GLP, unpublished
11315	Regenstein, H	1993c	Residues of Cycloxydim in peppers. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished

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1020718	Schroth, E & Martin, T	2007f	Study on the residue behavior of Cycloxydim in tomato after the application of BAS 517 24 H under field conditions in France (North and South), Germany, Greece, Italy, Netherlands and Spain 2006. Agrologia SL; Palomares; Spain GLP, unpublished
1024331	Schroth, E	2006d	Study on the residue behavior of Cycloxydim (BAS 517 H) in tomato after the application of BAS 517 24 H under field conditions in France, Germany, Greece, Italy, Netherlands and Spain, 2005. Agrologia SL; Palomares; Spain GLP, unpublished
1069374	Schaeufele, M	2009b	Residue study (decline) with BAS 517 24 H applied to lettuces in Italy and Spain in 2008. Huntingdon Life Sciences Ltd.; Huntingdon Cambridgeshire PE28 4HS; United Kingdom. GLP, unpublished
1102131	Schroth, E	2009d	Amendment No. 1: Study on the residue behavior of Cycloxydim in head lettuce after the application of BAS 517 24 H under field conditions in France (South), Greece, Italy and Spain, 2006. Agrologia SL; Palomares; Spain GLP, unpublished
1020724	Schroth, E & Martin, T	2007l	Study on the residue behavior of Cycloxydim in head lettuce after the application of BAS 517 24 H under field conditions in France (South), Greece, Italy and Spain, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1029325	Schroth, E	2007a	Study on the residue behavior of Cycloxydim (BAS 517 H) in head lettuce after the application of BAS 517 24 H under field conditions in France, Germany, Greece, Italy and Spain, 2005. Agrologia SL; Palomares; Spain GLP, unpublished
1015935	Schulz, H	2005a	Processing and field residue study on the residue behaviour of Cycloxydim in lettuce after application of BAS 517 24 H under field conditions in North France, Denmark and Sweden, 2003. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1008792	Trewhitt, J *et al.*	2002c	Study on the residue behaviour of Cycloxydim in head lettuce after application of BAS 517 24 H under field conditions in Denmark and France (N), 2001 BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1029326	Schroth, E	2007b	Study on the residue behavior of Cycloxydim (BAS 517 H) in spinach after the application of BAS 517 24 H under field conditions in France, Germany, Greece, Italy and Spain, 2005. Agrologia SL; Palomares; Spain. GLP, unpublished
1067444	Schaeufel, M	2009c	Residue study (decline) with BAS 517 24 H applied to fresh beans (dwarf french) in Belgium, Germany, The UK, Northern France and The Netherlands in 2008. Huntingdon Life Sciences Ltd.; Huntingdon Cambridgeshire PE28 4HS; United Kingdom. GLP, unpublished
1001265	Schulz, H	2003b	Determination of the residues of Cycloxydim and its metabolite in beans following treatment with BAS 517 24 H under field conditions in Italy, Spain, Southern France and Greece 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1031719	Schulz, H	2006c	Study on the residue behaviour of Cycloxydim and its metabolite in green beans (Phaseolus, field) after treatment with BAS 517 24 H under field conditions in France (N & S), England, Sweden, Belgium, Italy, Spain and Greece, 2005. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1013496	Mayer, F	2000b	Summary of residue data: Cycloxydim in beans. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
1102116	Schroth, E	2009e	Amendment No. 1: Study on the residue behavior of Cycloxydim in green peas after the application of BAS 517 24 H under field condtions in France (North and South), Greece, Germany, Italy, Netherlands, Spain and United Kingdom, 2006. Agrologia SL; Palomares; Spain.GLP, unpublished
1020725	Schroth, E &, Martin, T	2007q	Study on the residue behavior of Cycloxydim in green peas after the application of BAS 517 24 H under field conditions in France (North and South), Greece, Germany, Italy, Netherlands, Spain and United Kingdom, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1034132	Schulz, H	2007b	Study on the residue behaviour of Cycloxydim and its metabolite in green peas (field) after treatment with BAS 517 24 H under field conditions in France (N & S), England, Sweden, The Netherlands, Italy, Spain and Greece, 2005 SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, npublished
10698	Anonymous	1989d	Pflanzenschutzmittel-Rueckstaende–Erbsen. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. No GLP, unpublished
10697	Anonymous	1989e	Pflanzenschutzmittel-Rueckstaende–Erbsen. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. No GLP, unpublished

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1102124	Schroth, E	2009f	Amendment No. 2: Study on the residue behavior of Cycloxydim in dry beans (Vicia faba) after the application of BAS 517 24 H under field conditions in France (North and South), Germany, Greece, Italy, Netherlands and Spain, 2006. Agrologia SL; Palomares; Spain., GLP, unpublished
1019231	Schroth, E	2009a	Amendment No. 1: Study on the residue behavior of Cycloxydim in dry beans (Vica faba) after the application of BAS 517 24 H under field conditions in France (North and South), Germany, Greece, Italy, Netherlands and Spain, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1020731	Schroth, E & Martin, T	2007n	Study on the residue behavior of Cycloxydim in dry beans (Vicia faba) after the application of BAS 517 24 H under field conditions in France (North and South), Germany, Greece, Italy, Netherlands and Spain, 2006. Grologia SL; Palomares; Spain. GLP, unpublished
1024330	Schulz, H	2006d	Study on the residue behaviour of Cycloxydim and its metabolite in dry beans (Vicia faba) after treatment with BAS 517 24 H under field conditions in France (N & S), England, Sweden, Denmark, Italy, Spain and Greece, 2005 SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1001266	Schulz, H	2003c	Determination of the residues of Cycloxydim and its metabolite in dry beans (Vicia faba) following treatment with BAS 517 24 H under field conditions in Italy and Spain 2001 and in Greece 2002. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1013496	Mayer, F	2000b	Summary of residue data: Cycloxydim in beans. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. No GLP, unpublished
1009633	Grosshans, F	2006b	Summary of residue data: Cycloxidim in peas. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
1001263	Schulz, H	2004a	Determination of the residues of Cycloxydim and its metabolites in dry peas following treatment with BAS 517 24 H under field conditions in Greece 2002 Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1001252	Smalley, R	2003c	Study on the residue behaviour of BAS 517 H in dry peas after application of BAS 517 24 H under field conditions in Italy, France South and Spain, 2002 BASF plc; Gosport Hampshire PO13 0AU; United Kingdom. GLP, unpublished
1001251	Smalley, R	2003d	Study on the residue behaviour of Cycloxidim in peas after application of BAS 517 24 H under field conditions in France (S), Spain, Italy and Greece, 2001 BASF plc; Gosport Hampshire PO13 0AU; United Kingdom. GLP unpublished
1075175	Oxspring, S	2009f	Study on the residue behaviour of BAS 517 H in soya bean after treatment with BAS 517 24 H under field conditions in Southern Europe during 2007 Agrisearch UK Ltd.; Melbourne Derbyshire DE73 8AG; United Kingdom. GLP unpublished
1020732	Schroth, E & Martin, T	2007p	Study on the residue behavior of Cycloxydim in soya beans after the application of BAS 517 24 H under field conditions in France (South), Germany, Italy, Netherlands and Spain, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1030315	Schulz, H	2006f	Study on the residue behaviour of Cycloxydim and its metabolite in soya bean after treatment with BAS 517 24 H under field conditions in France (N & S), Germany, Italy and Spain, 2005. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1075173	Oxspring, S	2009b	Study on the residue behaviour of BAS 517 H in carrot after treatment with BAS 517 24 H under field conditions in Northern and Southern Europe during 2007. Agrisearch UK Ltd.; Melbourne Derbyshire DE73 8AG; United Kingdom. GLP, unpublished
1025865	Schroth, E	2006b	Study on the residue behavior of Cycloxydim (BAS 517 H) in carrots after the application of BAS 517 24 H under field conditions in France, Greece, Italy and Spain, 2005. Agrologia SL; Palomares; Spain. GLP, unpublished
1004555	Jones, S	2003a	Study on the residue behaviour of BAS 517 H in carrots after application of BAS 517 24 H under field conditions in France (S), Italy, Greece, 2001 BASF plc; Gosport Hampshire PO13 0AU; United Kingdom. GLP, unpublished
1075172	Oxspring, S	2009c	Study on the residue behaviour of BAS 517 H in celeriac after treatment with BAS 517 24 H under field conditions in Northern and Southern Europe during 2007. Agrisearch UK Ltd.; Melbourne Derbyshire DE73 8AG; United Kingdom. GLP, unpublished
1020727	Schroth, E & Martin, T	2007e	Study on the residue behavior of Cycloxydim in celeriac after the application of BAS 517 24 H under field conditions in France (North), Germany, Italy and Spain, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished

Code	Author(s)	Year	Title, Institute, Report reference
1020729	Schroth, E & Martin, T	2007d	Study on the residue behavior of Cycloxydim in potato after the application of BAS 517 24 H under field conditions in France (North), Germany, Netherlands and United Kingdom, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1090754	Oxspring, S	2009a	Study on the residue behaviour of BAS 517 H in potato after treatment with BAS 517 24 H under field conditions in Northern and Southern Europe during 2007. Agrisearch UK Ltd.; Melbourne Derbyshire DE73 8AG; United Kingdom. GLP, unpublished
1013495	Mayer, F	2000a	Summary of residue data: Cycloxydim in potatoes. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
1005446	Smalley, R	2003a	Study on the residue behaviour of Cycloxydim in potatoes after application of BAS 517 24 H under field conditions in France (S), Spain, Italy and Greece, 2001. BASF plc; Gosport Hampshire PO13 0AU; United Kingdom. GLP, unpublished
11354	Regenstein, H	1993a	Residues of Cycloxydim in turnips. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
11355	Regenstein, H	1993b	Residues of Cycloxydim in turnips. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
10712	Anonymous	1988e	Pesticide residue analysis—Swedish turnip. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
10702	Anonymous	1988f	Pesticide residue analysis—Swedish turnip. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
1001264	Schulz, H	2003e	Determination of the residues of Cycloxydim and its metabolite in sugar-beets following treatment with BAS 517 24 H under field conditions in Italy and Greece 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1013494	Mayer, F	2000d	Summary of residue data: Cycloxydim in sugar beet. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
10456	Beck, J *et al*.	1997a	Study on the residue behaviour of BAS 517 H (Cycloxydim) in Cycloxydim tolerant maize after one application with BAS 517 22 H under field conditions in France, Germany and Spain, 1995. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
10417	Schulz, H .	1997a	Determination of the residues of BAS 517 H in Cycloxydim-tolerant maize following treatment with BAS 517 22 H under field conditions in Italy 1995 Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
10518	Beck, J & Schulz, H	1997a	Study on the residue behaviour of BAS 517 H (Cycloxydim) in Cycloxydim-tolerant maize after one application with BAS 517 22 H under field conditions in France, Germany and Spain, 1996. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
10403	Schulz, H	1997b	Determination of the residues of BAS 517 H in Cycloxydim-tolerant maize following treatment with BAS 517 22 H under field conditions in Italy 1996 Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
10302	Schulz, H	1997c	Determination of the residues of Cycloxydim in rice following a pre-sowing treatment of the test field with BAS 517 22 H under field conditions in Italy 1996. Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
10290	Schulz, H	1997d	Determination of the residues of Cycloxydim in rice following a pre-sowing treatment of the test field with BAS 517 22 H under field conditions in Italy 1995. Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
11605	Anonymous	1994e	Pesticide residue analysis—Rice. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. No GLP, unpublished
11604	Anonymous	1994f	Pesticide residue analysis—Rice. BASF AG Agrarzentrum Limburgerhof; Germany. No GLP, unpublished
11603	Anonymous	1994g	Pesticide residue analysis—Rice. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. No GLP, unpublished
1067445	Schaeufele, M	2009e	Residue study (decline) with BAS 517 24 H applied to winter oilseed rape in the UK, Northern France, Belgium, The Netherlands and Germany in 2008 Huntingdon Life Sciences Ltd.; Huntingdon Cambridgeshire PE28 4HS; United Kingdom. GLP, unpublished
1001253	Smalley, R	2003e	Study on the residue behaviour of BAS 517 H in winter rape after application of BAS 517 24 H under field conditions in Italy, France South and Spain, 2002 BASF plc; Gosport Hampshire PO13 0AU; United Kingdom. GLP, unpublished

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1001267	Schulz, H	2003d	Determination of the residues of Cycloxydim and its metabolite in winter rape following treatment with BAS 517 24 H under field conditions in Italy, Spain and Southern France 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1013493	Mayer, F	2000c	Summary of residue data: Cycloxydim in oilseed rape. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. No GLP, unpublished
1069373	Schaeufele, M	2009d	Residue study (decline) with BAS 517 24 H applied to sunflowers in Belgium and Germany in 2008. Huntingdon Life Sciences Ltd.; Huntingdon. Bridgeshire PE28 4HS; United Kingdom. GLP, unpublished
1020733	Schroth, E & Martin, T	2007o	Study on the residue behavior of Cycloxydim in sunflower after the application of BAS 517 24 H under field conditions in France (South), Greece, Italy, Netherlands, Spain and United Kingdom, 2006. Agrologia SL; Palomares; Spain. GLP, unpublished
1034026	Schulz, H	2006e	Study on the residue behaviour of Cycloxydim and its metabolite in sunflower after treatment with BAS 517 24 H under field conditions in France (N & S), Italy, Spain and Greece, 2005. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1015931	Schulz, H	2004b	Determination of the residues of Cycloxydim and its metabolites in sunflower following treatment with BAS 517 24 H under field conditions in Italy, Southern France and Greece 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1015930	Schulz, H	2004c	Determination of the residues of Cycloxydim and its metabolites in sunflower following treatment with BAS 517 24 H under field conditions in Southern France and Greece 2002. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
10368	Schulz, H	1995a	Determination of the residues of Cycloxydim in sunflowers following treatment with BAS 517 01 H under field conditions in Italy 1993 Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
10419	Schulz, H	1995b	Addendum No. 1 to the report: Determination of the residues of Cycloxydim in sunflowers following treatment with BAS 517 01 H under field conditions in Italy 1993. Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1000144	von Goetz, N	2000a	Hydrolysis of BAS 517 H (Cycloxydim) at 90 °C, 100 °C, and 120 °C. BASF AG Agrarzentrum Limburgerhof; Limburgerhof; Germany. GLP, unpublished
1065669	Harant, H	2009a	Determination of residues of BAS 517 H in strawberries and its processed products after one application of BAS 517 24 H in Germany. BioChem agrar Labor fuer biologische und chemische Analytik GmbH; Gerichshain; Germany. GLP unpublished
1001268	Schulz, H	2003f	Determination of the residues of Cycloxydim and its metabolite in potatoes and processed products following treatment with BAS 517 24 H under field conditions in Germany and Italy 2001 Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1026923	Reichert, N	2005a	Determination of the residues of Cycloxydim and its metabolites in carrots and processed products following treatment with BAS 517 24 H under field conditions in Germany and Italy 2001. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1001289	Schulz, H	2003a	Determination of the residues of Cycloxydim and its metabolite in onions and processed products following treatment with BAS 517 24 H under field conditions in Italy and Greece 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
1065670	Harant, H	2009b	Determination of residues of BAS 517 H in tomato and its processed products after one application of BAS 517 24 H in Germany. BioChem agrar Labor fuer biologische und chemische Analytik GmbH; Gerichshain; Germany. GLP, unpublished
1015935	Schulz, H	2005a	Processing and field residue study on the residue behaviour of Cycloxydim in lettuce after application of BAS 517 24 H under field conditions in North France, Denmark and Sweden, 2003. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1000247	Beck, J *et al.*	2000a	Determination of residues of Cycloxydim in raw and processed commodities of combining peas following treatment with BAS 517 01 H (= Laser) under field conditions in Great Britain, 1994. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished

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1001269	Reichert, N	2003a	Determination of the residues of Cycloxydim and its metabolite in peas and processed products following treatment with BAS 517 24 H under field conditions in Germany and Southern France 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
10658	Steggles, HA	1992a	Residues of Cycloxydim in oilseed rape seed, oil, meal and whole plant following the application of two different formulations of BAS 517 H in the UK in 1990. BASF UK; Hadleigh Suffolk IP7 6BQ; United Kingdom. No GLP, unpublished
1001270	Schulz, H	2003g	Determination of the residues of Cycloxydim and its metabolite in rape and processed products following treatment with BAS 517 24 H under field conditions in Germany and Spain 2001. Institut Fresenius Chemische und Biologische Laboratorien AG; Taunusstein; Germany. GLP, unpublished
10368	Schulz, H	1995a	Determination of the residues of Cycloxydim in sunflowers following treatment with BAS 517 01 H under field conditions in Italy 1993. Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
10419	Schulz, H	1995b	Addendum No. 1 to the report: Determination of the residues of Cycloxydim in sunflowers following treatment with BAS 517 01 H under field conditions in Italy 1993. Institut Fresenius Chemische und Biologische Laboratorien GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1008084	Reichert, N	2006a	Determination of the residues of Cycloxydim and its metabolite in white cabbage and processed products following treatment with BAS 517 24 H under field conditions in Germany and Southern France 2001. SGS Institut Fresenius GmbH; Taunusstein; Germany Fed. Rep. GLP, unpublished
1177209	Hopf, B *et al.*	2011a	A meat and egg magnitude of the residue study with BAS 517 H (Cycloxydim) and its metabolite BH 517-5-OH-TSO in laying hens; reanalysis of specimen using the common moiety method. BASF SE; Limburgerhof; Germany. GLP, unpublished
1004490	Grosshans, F & Kampke-Thiel, K	2003a	A meat and egg magnitude of the residue study with BAS 517 H (Cycloxydim) and BH 517-5-OH-TSO (Reg. No. 217 383, metabolite of BAS 517 H) in laying hens. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished
1090651	Grosshans, F	2009a	A meat and milk magnitude of the residue study with BAS 517 H. Cycloxydim) and its metabolite BH 517-5-OH-TSO in lactating dairy cows—Reanalysis of specimen using the common moiety method. BASF SE; Limburgerhof; Germany. GLP, unpublished
1004489	Seiferlein, M & Kampke-Thiel, K	2003a	A meat and milk magnitude of the residue study with BAS 517 H (Cycloxydim) and BH 517-5-OH-TSO (Reg. No. 217 383, a metabolite of BAS 517 H) in lactating dairy cows. BASF AG Agrarzentrum Limburgerhof; Germany. GLP, unpublished