GLUFOSINATE AMMONIUM (175)

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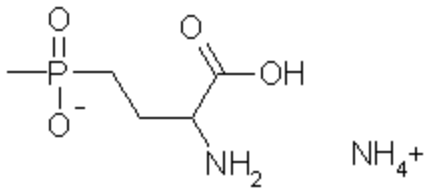
Glufosinate ammonium is an herbicide used in a variety of crops and was first reviewed by the 1991 JMPR. Glufosinate ammonium was scheduled at the Forty-third Session of the CCPR (2011) for periodic re-evaluation of toxicology and residues by the 2012 JMPR.

IDENTITY

Common name	Glufosinate ammonium
Chemical name	
IUPAC:	ammonium (3-amino-3-carboxypropyl)methyl phosphinate
Previous name:	ammonium-DL-homoalanin-4-yl(methyl)phosphinate
CAS:	Butanoic acid, 2-amino-4-(hydroxymethylphosphinyl)-, monoammonium salt (9CI)
Manufacturer's	
code numbers:	AE F039866
CAS number:	77182-82-2
CIPAC Code:	437.007
Molecular formula:	$C_5H_{15}N_2O_4P$
Molecular mass:	198.2 g/mol
Structural formula:	



Formulations	Active ingredient content
SL 150	150 g/L glufosinate-ammonium
SL 200	200 g/L glufosinate-ammonium
SL 280	280 g/L glufosinate-ammonium

Specifications

Specifications for glufosinate ammonium have not been developed by FAO.

PHYSICAL AND CHEMICAL PROPERTIES

Property	Results (method)	Reference
Appearance	Pure active substance: white crystalline powder (visual) [d]	Kocur 2001a C011318
	Technical grade active substance: white to light brown crystalline powder (visual) [e]	Heinrich & Rexer 1985
	TK50 b: colourless clear liquid (visual)	Rexer 2010 M-364528-01-2

Property	Results (method)	Reference
Odour	Pure active substance: weakly pungent [d]	Kocur 2001b C011319
	Technical grade active substance: odourless to mildly pungent [e]	Heinrich &Rexer 1985
	TK50 b: odourless	Rexer 2010 M-364528-01-2
Melting point	215–218 °C with decomposition (92/69/EEC A.1) [a]	Hoffmann 2000a C008925
Boiling point	Not measurable due to thermal decomposition in the range 245–305 °C (92/69/EEC EC A.2) [a]	Hoffmann 2000a C008925
Relative density	1.32 g/mL at 23 °C (92/69/EEC EC A.3) [a]	Hoffmann 2000b C008926
	1.204 g/mL at 20 °C (92/69/EEC EC A.3) [b]	Rexer 2010 M-364528-01-2
Vapour pressure	Estimated to be < 3.1 × 10-5 Pa at 50 °C (OECD 104 EC A.4) [c]	Cicotti 1992 A47879
Henry's Law constant	KH = 4.48 × 10-9 (Pa m3/mole) (calculated)	Schollmeier 1992 A48583
Solubility in water including effect of pH	water at pH 5.4, 7, 8.9: > 500 g/L at 20 °C (OECD 105, EC A.6, shake flask method) [f]	Goerlitz 1990 A43735
	≈1370 g/L at pH 5, 22 °C (in-house method) [g]	Goerlitz 1983 A25476
Solubility in organic solvents (at 20 °C) d	Acetone < 0.25 mg/L	Muehlberger 2001a C011179
	Acetonitrile < 0.25 mg/L	
	1,2-dichloro-ethane < 0.25 mg/L	
	DMSO 48.9 mg/L	
	ethyl acetate < 0.25 mg/L	
	n-heptane < 0.25 mg/L	
	methanol 5.73 g/L	
	p-xylene < 0.25 mg/L	
Partition coefficient n-octanol/water	log Pow at pH 5, 7, 9: –3.77, –4.01, –4.07 respectively (OECD 107)	Schneider 1996 A56104
Hydrolysis	Half life at 25 °C and pH 5, 7 and 9: > 300 days (EPA 161-1) [h]	Goerlitz 1985 A32265
		Goerlitz 1986 A32265
Photolysis	stable under abiotic conditions, at pH5, 7, 9; no volatile or non-volatile break-down products observed (EPA 161-2)	Sarafin 1989 A40989
UV absorption	Peak maxima molar ext. coef. [L/mol·cm]	Bogdoll 2001 PA00/028
	193 nm 382	
Quantum yield	Quantum yield is zero: the active substance is not phototransformed	
Dissociation constant	pKa = 9.15 + 0.07 (potentiometric titration method on ammonium salt at 23 °C, OECD 112) [d]	Muehlberger 2001b C011178
Surface tension	72.1 mN/m at 20 °C [i]	Hoffmann 2000c C009005
	65.4 mN/m at 40 °C [j]	Bittner 2000 C009917
	62.5 mN/m (undiluted at 25 °C), 71.5 mN/m (1 g/L at 20 °C) [b]	Rexer 2010 M-364528-01-2

[a] Glufosinate ammonium pure (batch AE F039866 00 1B99 0005, purity > 99.5%)

[b] Glufosinate ammonium TK50 (batch EFKJ000429, purity 50.6%)

[c] Glufosinate ammonium pure (batch AE F039866 00 1B99 0002, purity 99.5%)

[d] Glufosinate ammonium pure (batch AE F039866 1B99 0006, purity 99.2%)

[e] Glufosinate ammonium technical (batch AE F039866 0H ZD95 0001, purity ≥ 95%)

[f] Glufosinate ammonium pure (batch AE F039866 1B99 0003,purity > 99.5%)

[g] Glufosinate ammonium pure (batch AE F039866 0H ZB99 0001, purity > 99%)

[h] Glufosinate ammonium pure (batch Hoe 039866 OH ZB99 0002, purity 99.5%)

[i] Glufosinate ammonium technical (batch AE F039866 00 1D96 0001, purity 96.5%)

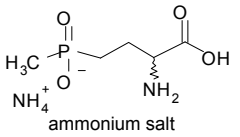
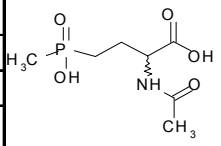
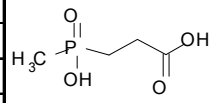
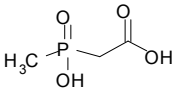
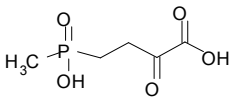
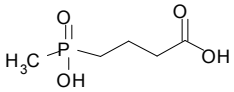
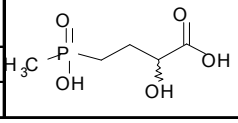
[j] Glufosinate ammonium TK50 (batch AE F039866 00 TK50 A100, purity 50.2%)

METABOLISM AND ENVIRONMENTAL FATE

Glufosinate ammonium is a racemic mixture of the D- and L-isomers of the ammonium salt of ammonium-DL-homoalanin-4-yl(methyl)phosphinate or ammonium-(3-amino-3-carboxypropyl) methyl phosphinate.

Glufosinate ammonium and its metabolites are available in different forms such as the racemate, the D- and L-enantiomers as well as free acids and their sodium salts. Metabolites are given various abbreviations and code numbers in the studies. Structures and abbreviations and codes are shown below.

Glufosinate ammonium - list of metabolites including company codes

Substance	Racemate	L-Enantiomer	D-Enantiomer	Structural formula of the parent acid
glufosinate ammonium (abbreviated GA)				 ammonium salt
free acid	AE F035956	AE F057740	AE F090532	
ammonium salt	AE F039866	AE F058192	AE F093854	
hydrochloride salt	AE F035125	AE F057742	AE F057741	
N-acetyl-glufosinate (abbreviated NAG)				
free acid	AE F085355	AE F099729	AE F124451	
disodium salt	AE F098412	AE F099730	AE F124450	
3-methyl-phosphinico-propionic acid (abbreviated MPP)				
free acid	AE F061517	n/a	n/a	
disodium salt	AE C527855	n/a	n/a	
2-methyl-phosphinico-acetic acid (abbreviated MPA)				
free acid	AE F064619	n/a	n/a	
disodium salt	AE F159481	n/a	n/a	
4-methyl-phosphinico-2-oxo-butanoic acid (abbreviated PPO)				
free acid	AE F065594	n/a	n/a	
4-methyl-phosphinico-butanoic acid (abbreviated MPB)				
free acid	AE F039046	n/a	n/a	
4-methyl-phosphinico-hydroxy-butanoic acid (abbreviated MHB)				
free acid	AE F053705	n/a	n/a	
disodium salt	AE F042231	n/a	n/a	

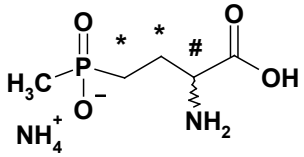
n/a: not applicable or code number not assigned

The identification of residue components in the animal and plant metabolism studies was achieved using multiple chromatographic systems combined with authentic standards of the compounds involved.

Animal metabolism

The Meeting received studies on the metabolism of glufosinate in rats, lactating goats and laying hens. The metabolism of glufosinate in plants and animals was investigated using [2-^{14}C]- and [3,4-^{14}C]-glufosinate ammonium. The structural formula and the positions of the ^{14}C label are shown below. The studies on rats were evaluated by the WHO Core Assessment Group.

Label positions of glufosinate ammonium: [2-^{14}C] marked as # and [3,4-^{14}C] marked as *



Livestock metabolism studies in lactating goats and laying hens were conducted with the radio-labelled glufosinate ammonium (glufosinate) and with the main metabolite detected in genetically modified glufosinate-tolerant crops, N-acetyl-glufosinate (NAG).

Lactating goat

Huang (1995a A54158) studied the metabolism of glufosinate in a lactating goat (Toggenberg breed, 3–4 years old and 60 kg). Alfalfa grass hay and tap water were provided *ad libitum* with an additional 1 kg grain-based feed provided daily. The goat was dosed twice a day (after morning and afternoon milking) with capsules via a balling gun for a period of four consecutive days and at 3.0 mg glufosinate ammonium/kg bw/day equivalent to 101 ppm in the feed. During the study average milk yield was 2.0 kg/day while feed consumption was 1.6 kg/day. Approximately 15 hours after the last dose the animal was sacrificed. Samples were stored at -15 °C or less prior to analysis. Extracts of kidney, liver, and milk were profiled within 5 months of sacrifice.

The majority of the administered dose was recovered in the faeces (68.8%) with less than 3% of the found in urine and 0.02% in milk. TRR in milk appeared to reach a plateau by the second day of dosing. The amount of administered radioactivity found in tissues was 0.1% while the gastrointestinal tract contained 12.2% giving a total recovery of administered radioactivity of 84.2%.

TRR levels were highest in kidneys (0.609 mg eq/kg) followed by the liver (0.401 mg eq/kg), muscle (0.007 mg eq/kg) and fat (0.004 mg eq/kg). TRR in milk between ranged between 0.003 (morning milk of the first day of dosing) and 0.022 (morning milk of day three) mg eq/kg (Table 1).

Ethanol and water extraction of tissue and milk samples resulted in extraction efficiencies of 89% (milk), 94.5% (kidney) and 65% (liver) TRR (Table 2). Mild acid and alkaline hydrolysis released an additional 31.5% of liver TRR. Muscle and fat were not subject to extraction and further analysis as the TRR levels were insignificant (< 0.01 mg eq/kg).

In kidneys, the main ^{14}C residue components were parent glufosinate (49% TRR) and its metabolite MPP (29% TRR). NAG and MPA were detected as minor metabolites, < 5% of TRR. No other single metabolite comprised more than 3% of TRR.

Extraction of liver with ethanol and water released MPP (36% TRR) and parent glufosinate (25% TRR). MPA was observed as a very minor metabolite (0.4% TRR). Hydrolysis of the extracted liver under mild acid and alkaline conditions released further glufosinate (27.7% TRR) and MPP (0.9% TRR). The total identified residues in the liver accounted for 90% of TRR and consisted of glufosinate (53%), MPP (36.5%) and MPA (0.4%). NAG was not detected in the liver. No other single metabolite comprised more than 3% of TRR.

Residues in milk (sample from second day) were extracted by dialysis. Extracted residues were low (0.020 mg eq/kg, 63% TRR). Unchanged glufosinate was the main component (49% TRR) with low levels of MPP (6% TRR), MPA (5% TRR) and NAG (2% TRR) also detected. No other single metabolite comprised more than 2% of TRR.

Table 1 Total radioactive residues (TRR) in milk of a goat following daily dosing of [^{14}C] glufosinate ammonium (Huang 1995a A54158)

Day of collection	TRR, morning milking	TRR, afternoon milking	Daily averaged TRR residues
	(mg eq/kg)		
1	0.003	0.009	0.006
2	0.016	0.020	0.018
3	0.022	0.020	0.021
4	0.020	0.014	0.017

Table 2 Total radioactive residues (TRR) and extractability in tissues and milk of a goat dosed with [^{14}C] glufosinate ammonium (Huang 1995a A54158)

	Milk [a]	Fat	Muscle	Liver	Kidney
TRR (mg/kg as glufosinate ammonium)	0.020	0.004 [c]	0.007 [c]	0.401	0.609
		%TRR			
Extracted with EtOH and water	88.8			65.2	94.5
Glufosinate	48.9	–	–	24.6	49.0
NAG	2.2	–	–	ND	4.2
MPP	6.3	–	–	35.6	29.4
MPA	5.3	–	–	0.4	1.2
Liberated on hydrolysis of PES [b] Glufosinate MPP	–	–	–	31.8 28.1 0.9	–
Unextracted	11.2		74	5.3	5.5

[a] Milk from day 2.

[b] Following extraction of the liver additional 31.5% of TRR (0.126 mg eq/kg) was released by acid and alkaline hydrolysis

[c] Residues below 0.01 mg eq/kg were not extracted

In summary, glufosinate was rapidly eliminated from a lactating goat via the excreta, with only low levels detected in the milk and tissues. In milk, a residue plateau was reached after two days of dosing. Glufosinate was the major component of the ^{14}C residue in milk, tissues and excreta. Glufosinate was metabolized primarily to MPP with lesser amounts of NAG and MPA. The metabolite profile in the goat was similar to that found in other animal studies (faeces: glufosinate 75.9% TRR, NAG 8.3%, MPP 12%, MPA 2%; urine: glufosinate 80.9% TRR, NAG 2.4% TRR, MPP 13.7% TRR, MPA 0.7% TRR).

L-NAG

In a separate study Huang (1995b A54155) investigated the metabolism of [3,4-^{14}C]-L-N-acetyl glufosinate (L-NAG) in a lactating goat (cross bred Alpine, 3 years old and 36 kg). The goat was fed a combination of hay and a high protein dairy goat feed and provided water *ad libitum*. The dose was orally administered in gelatin capsules to the test animal twice a day (after morning and afternoon milking) for three days via a balling gun. The dose level was 3.0 mg/kg bw/day, equivalent to 84.3 ppm in the feed. During the study the average milk yield was 1.3 L/day while feed consumption was 1.3 kg/day. Approximately 16 hours after the last dose, the animal was sacrificed. Samples of tissues and milk were stored at -15 °C or less prior to analysis.

The majority of the administered ^{14}C was recovered with the faeces (68.1%) and urine (7.3%) with 18.8% of the dose found in the gastrointestinal tract. The accountability of the ^{14}C in tissues, blood and excreta was 94.4% of the total administered dose. TRR in milk appeared to reach a plateau by the second day of dosing.

TRR levels were highest in kidneys (0.93 mg eq/kg) followed by the liver (0.29 mg eq/kg), muscle (0.007 mg eq/kg) and fat (< 0.01 mg eq/kg). TRR in milk ranged between 0.005 (morning milk of the first day of dosing) and 0.023 (morning milk of day three) mg eq/kg (Table 3).

Ethanol and water extraction of tissue and milk samples resulted in extraction efficiencies of 70% (milk), 99% (kidney) and 61% (liver) TRR (Table 4). Mild acid, alkaline and enzymatic hydrolysis released an additional 39% of the initial liver radioactivity. No attempt was made to identify residue components in muscle and fat as the TRR levels were too low (< 0.01 mg eq/kg). Residue levels in the cream and skim milk showed a similar metabolite profile to that of the whole milk.

In kidneys, the main ^{14}C residues were glufosinate (40% TRR), unchanged NAG (32% TRR) and MPP (20% TRR). MPA was detected as a minor metabolite, < 5% of TRR. In liver (ethanol/water extract) glufosinate (18% TRR), unchanged NAG (18% TRR) and MPP (17% TRR) were the major components of ^{14}C residues. MPA was a minor metabolite (1.3% TRR). Hydrolytic digestion of liver

under mild acid, alkaline and enzymatic conditions released additional ^{14}C as glufosinate (15% TRR), NAG (1% TRR), MPP (4% TRR) and MPA (1% TRR).

Milk (day 3; 0.023 mg eq/kg) was extracted by dialysis (48 hours). More than 70% of TRR in milk was present as water soluble residues while the remainder was comprised mostly of proteinaceous solid but only representing 0.007 mg eq/kg. The ^{14}C in the dialysis extract consisted of glufosinate (40% TRR), unchanged NAG (9% TRR), MPP (14% TRR) and MPA (5% TRR). Samples of whole milk were separated into skim milk and cream. Radioactive residues in whole milk were 0.005–0.023 mg eq/kg, skim milk 0.007–0.023 mg eq/kg and cream 0.022–0.046 mg eq/kg with the profile of components similar in all three products.

Table 3 Total radioactive residues (TRR) in milk of a goat following daily dosing of [3,4-^{14}C]-NAG at a level equivalent to 84 ppm in the feed for 3 consecutive days (Huang 1995b A54155)

Day of collection	TRR, morning milking (mg eq/kg)	TRR, afternoon milking	Daily averaged TRR residues
1	0.005	0.012	0.008
2	0.018	0.020	0.019
3	0.023	0.022	0.022

Table 4 Total radioactive residues (TRR) and extractability in tissues and milk of a goat dosed with [3,4-^{14}C]-NAG at a level equivalent to 84 ppm in the feed for 3 consecutive days (Huang 1995b A54155)

	Milk [a]	Fat	Muscle	Liver	Kidney
TRR (mg/kg as NAG)	0.023	< 0.01 [c]	0.007 [c]	0.29	0.93
		%TRR			
Extracted with EtOH and water	70.5			61.2	99.4
Glufosinate	40.0	–	–	18.1	40.3
NAG, L-isomer	9.2	–	–	17.8	32.0
MPP	14.3	–	–	16.7	20.0
MPA	4.8	–	–	1.3	1.6
Liberated on hydrolysis of PES [b] Glufosinate NAG MPP MPA	– 	– 	–	38.6 14.6 0.9 3.9 0.7	–
Unextracted	29.5	–	–	0.2	0.6

[a] day 3 milk

[b] Following extraction of the liver additional 38.6% of TRR was released by acid (8.0%), alkaline (21.0%) and enzymatic (9.6%) hydrolysis

[c] Residues below 0.01 mg eq/kg were not extracted

In summary, NAG comprised a major portion of the ^{14}C residues in milk, faeces, urine and tissues and was also readily deacetylated by the goat to form glufosinate. Glufosinate was a significant, and in the case of the milk and tissues, the principal metabolite. MPP and lesser amounts of MPA comprised the remainder of the ^{14}C residues.

Laying hen

The metabolism of glufosinate in laying hens was studied by Huang (1995c A54159). Twelve laying hens (White Leghorn, 25–35 weeks old and 1.3–1.6 kg), were dosed via gelatin capsules administered orally to the test animals twice a day (early morning and late afternoon) for a period of 14 consecutive days. The dose level was 2.0 mg/kg bw/day, equivalent to 24.5 ppm glufosinate ammonium in the feed (although feed not specified whether feed consumption was on a dry or fresh weight basis, the difference between the two is minor for a grain based diet). During the study average laying efficiency was 92% while feed consumption was 0.122 kg/day. Approximately 15–16 hours after the last dose, the birds were sacrificed. Samples of tissues and eggs were stored frozen at -20 °C prior to analysis.

The majority of the administered ^{14}C was recovered from the excreta (91.9%) with 1.3% found in the gastrointestinal tract. Overall, the accountability of ^{14}C was 93.3% of the total administered dose.

The maximum ^{14}C level in egg yolk was 0.024 mg eq/kg and occurred on the last day of dosing (Table 5). The maximum ^{14}C level in egg white was 0.067 mg eq/kg at days 12–14. Cumulative radioactivity recovered in the egg white and yolk accounted for less than 0.07% of the total administered dose.

Ethanol and water extraction of tissue and egg samples resulted in extraction efficiencies of 98% for egg white, 73% for egg yolk and 96% for liver (Table 6). The TRR in muscle was below the limit of detection of 0.004 mg eq/kg while TRR in fat was 0.003 mg eq/kg. As ^{14}C levels were very low in muscle and fat no attempt was made to identify individual components in these tissues.

The major ^{14}C components in liver were MPP (44% TRR) and glufosinate (31% TRR). NAG and MPA were observed as minor metabolites (< 5% of TRR). No other single metabolite comprised more than 1.3% of TRR.

Residues plateaued in egg by day 12 of dosing. The major components in egg yolk (day 13; 0.022 mg eq/kg) were unchanged glufosinate (53% TRR) together with minor amounts of MPP (4% TRR), MPA (3% TRR) and NAG (2.4% TRR). No other single metabolite comprised more than 3% of TRR.

In egg white (day 14; 0.067 mg eq/kg), ^{14}C residue components consisted of unchanged glufosinate (78% TRR) and MPP (1.3% TRR) as a very minor metabolite. MPA and NAG were not detected in egg white.

The principal component found in excreta was the parent compound glufosinate (81% TRR), with minor amounts of NAG (7% TRR), MPP (6% TRR) and MPA (3% TRR) also detected.

Table 5 Total radioactive residues (TRR) in eggs of laying hens continuously dosed with [^{14}C] glufosinate ammonium at a level equivalent to 25 ppm feed for 14 consecutive days (Huang 1995c A54159).

Day of collection	Mean TRR in egg white [a] (mg eq/kg)	Mean TRR in egg yolk [a] (mg eq/kg)
1	< 0.003	< 0.003
2	0.004	< 0.003
3	0.034	0.005
4	0.053	0.009
5	0.049	0.012
6	0.057	0.015
7	0.056	0.016
8	0.056	0.017
9	0.058	0.019
10	0.059	0.021
11	0.058	0.021
12	0.067	0.021
13	0.065	0.022
14	0.067	0.024

[a] Mean of each six birds per day

Table 6 Distribution of total radioactive residue (mg/kg glufosinate ammonium equivalents) and identification of metabolites in liver, muscle and skin with fat after dosing laying hens with [^{14}C] glufosinate ammonium (Huang 1995c A54159).

	Egg white	Egg yolk	Liver	Fat	Muscle
TRR (mg/kg as Glufosinate ammonium)	0.067	0.022	0.114	0.003*	< 0.004
			%TRR		
Total solvent extracted	97.9	73.5	96.2	-	-
Glufosinate	77.8	53.1	31.1		
NAG	ND	2.4	4.9		

	Egg white	Egg yolk	Liver	Fat	Muscle
MPP	1.3	4.1	44.1		
MPA	ND	3.1	3.5		
PES	2.1	26.5	3.8	–	–

ND = not detected

In summary, glufosinate was the major component of ^{14}C residues in eggs and excreta. Glufosinate was metabolized by the hens to MPP (major metabolite in liver) with lower amounts of NAG and MPA. The metabolite profile in the hen was similar to that found in other animal studies.

L-NAG

The metabolism of NAG was also studied in laying hens (Huang 1995d A54157). Six laying hens (DeKalb Delta, 37 weeks old and weighing 1.2–1.6 kg) were dosed with gelatine capsules containing NAG orally twice a day, following the morning and the afternoon egg collections and for a period of 14 consecutive days. The dose level was 2.2 mg/kg bw/day, equivalent to 27.3 ppm NAG in the diet (although feed not specified whether feed consumption was on a dry or fresh weight basis, the difference between the two is minor for a grain based diet). During the study average laying efficiency was 100% while respective feed consumption was 0.116 kg/day. Approximately 15 hours after the last dose, the birds were sacrificed. Samples of tissues and eggs were stored at ≤ -15 °C.

The majority of the administered ^{14}C was recovered from the excreta (85.8%) with a further 1.0% found in the gastrointestinal tract at sacrifice. Overall, the accountability of ^{14}C was 86.8% of the total administered dose.

Radioactive residues found in the egg yolk appeared to achieve a plateau by day 12, with a maximum ^{14}C level of 0.056 mg eq/kg (Table 7). The residues in the egg white appeared to attain a plateau by Day 9, with a maximum ^{14}C level of 0.015 mg eq/kg.

As ^{14}C levels were very low in muscle and fat no attempt was made to identify individual components in these tissues.

Extraction of liver with ethanol and water released the test substance NAG (27% TRR), MPP (17% TRR) and glufosinate (15% TRR) as major residue components (Table 8). MPA was not observed in the liver. No other single extractable metabolite comprised more than 2% of TRR.

The egg yolk (day 14; 0.056 mg eq/kg) was extracted by dialysis with 28% TRR recovered. Residues consisted of the test substance NAG (13% TRR) together with glufosinate (3% TRR), MPP (2% TRR) and MPA (1% TRR). The largest unknown was 0.5% of TRR. No attempt was made to further characterise ^{14}C residues remaining in the post-extraction solids.

The egg white of day 13 (0.015 mg eq/kg) was also extracted by dialysis. Almost 29% of TRR was released on dialysis. These residues consisted of glufosinate (14% TRR), the unchanged NAG (5% TRR), MPP (2% TRR) and MPA (1% TRR). The largest extracted unknown comprised 5% of TRR or 0.001 mg eq/kg. No attempt was made to further characterise ^{14}C residues remaining in the post-extraction solids.

Table 7 Total radioactive residues (TRR) in eggs of laying hens continuously dosed with [^{14}C] NAG at a level equivalent to 27. 3 ppm feed for 14 consecutive days (Huang 1995d A54157)

Day of collection	Mean TRR in egg white [a] (mg eq/kg)	Mean TRR in egg yolk [a] (mg eq/kg)
1	< 0.009	< 0.002
2	< 0.009	< 0.002
3	< 0.009	0.012
4	0.010	0.020
5	0.010	0.027
6	0.011	0.035
7	< 0.009	0.037
8	0.012	0.040
9	0.014	0.042
10	0.012	0.045

Day of collection	Mean TRR in egg white [a] (mg eq/kg)	Mean TRR in egg yolk [a] (mg eq/kg)
11	0.012	0.046
12	0.013	0.050
13	0.015	0.049
14	0.014	0.052
necroscopy	< 0.009	0.056

[a] Mean of each six birds per day

LOQ for egg white: 0.009 mg eq/kg and for egg yolk 0.002 mg eq/kg

Table 8 Distribution of total radioactive residue and identification of metabolites in liver, muscle and skin with fat after dosing laying hens with ^{14}C-NAG (Huang 1995d A54157)

	Egg white	Egg yolk	Liver	Fat	Muscle
TRR (mg/kg as NAG)	0.014	0.056	0.076	0.011	0.013
		%TRR			
Total solvent extracted	28.8	28.0	64.3	62.1	45.1
Glufosinate	14.0	2.8	14.9		
NAG	5.1	12.8	26.7		
MPP	2.0	2.2	16.6		
MPA	1.1	0.6	ND		
PES	71.2	72.0	35.7	37.9	54.9

ND = not detected

The metabolism in hens was similar to that found in goats with NAG deacetylated to form glufosinate which undergoes further metabolism to MPP and MPA. Residues remaining in tissues and eggs after solvent extraction were higher for the birds dosed with NAG than for those dosed with glufosinate.

In summary, for goats and laying hens dosed with [^{14}C] glufosinate, the major residue component in milk, eggs, tissues and excreta was parent glufosinate. Glufosinate was metabolized primarily to MPP with lesser amounts of NAG and MPA. The proposed metabolic pathway of glufosinate in the goat and hens is shown in Figure 1.

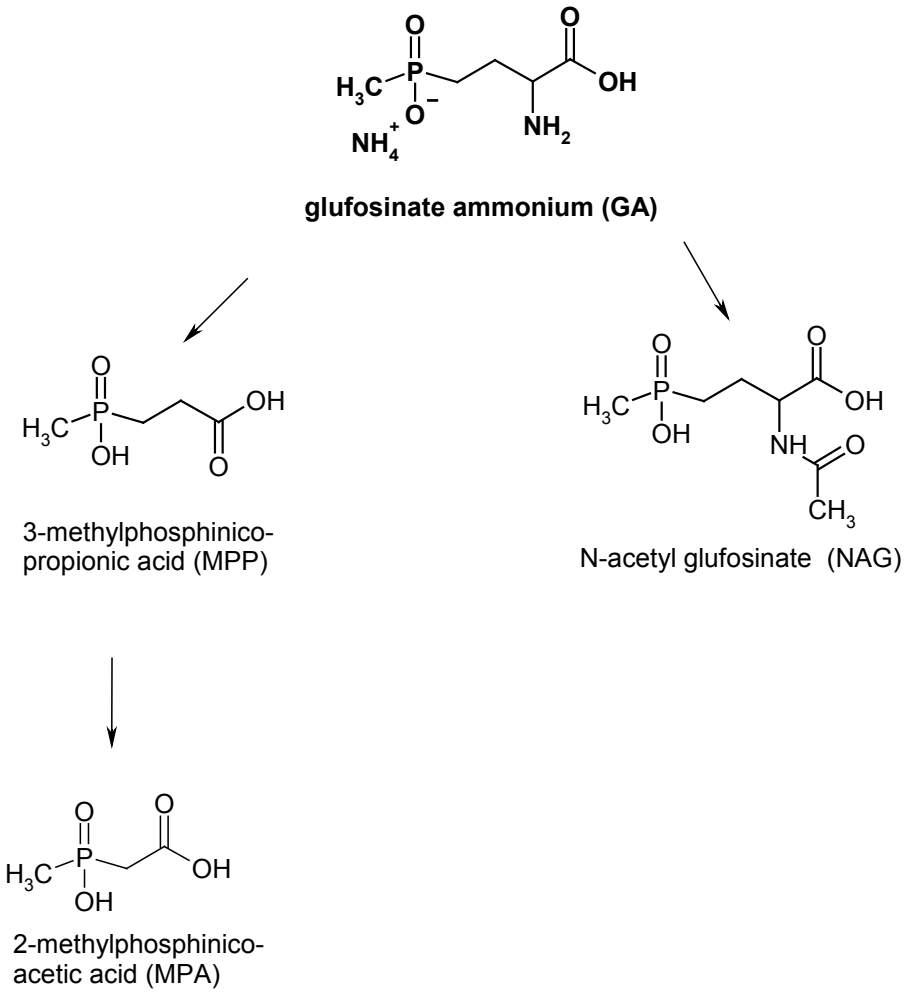


Figure 1 Proposed metabolic pathway of glufosinate in goat and hen

When a goat was dosed with NAG, a major portion of the ^{14}C residues in milk, faeces, urine and tissues was NAG. Additionally NAG deacetylated to form glufosinate which formed a significant component of the ^{14}C residue, and in the case of the milk and tissues, the principal metabolite. MPP and lesser amounts of MPA comprised the remainder of the ^{14}C residues. The metabolism in hens was similar to that found in goats with NAG deacetylated to form glufosinate which undergoes further metabolism to MPP and MPA. Figure 2 shows the proposed pathway for the metabolism of NAG in goats and hens.

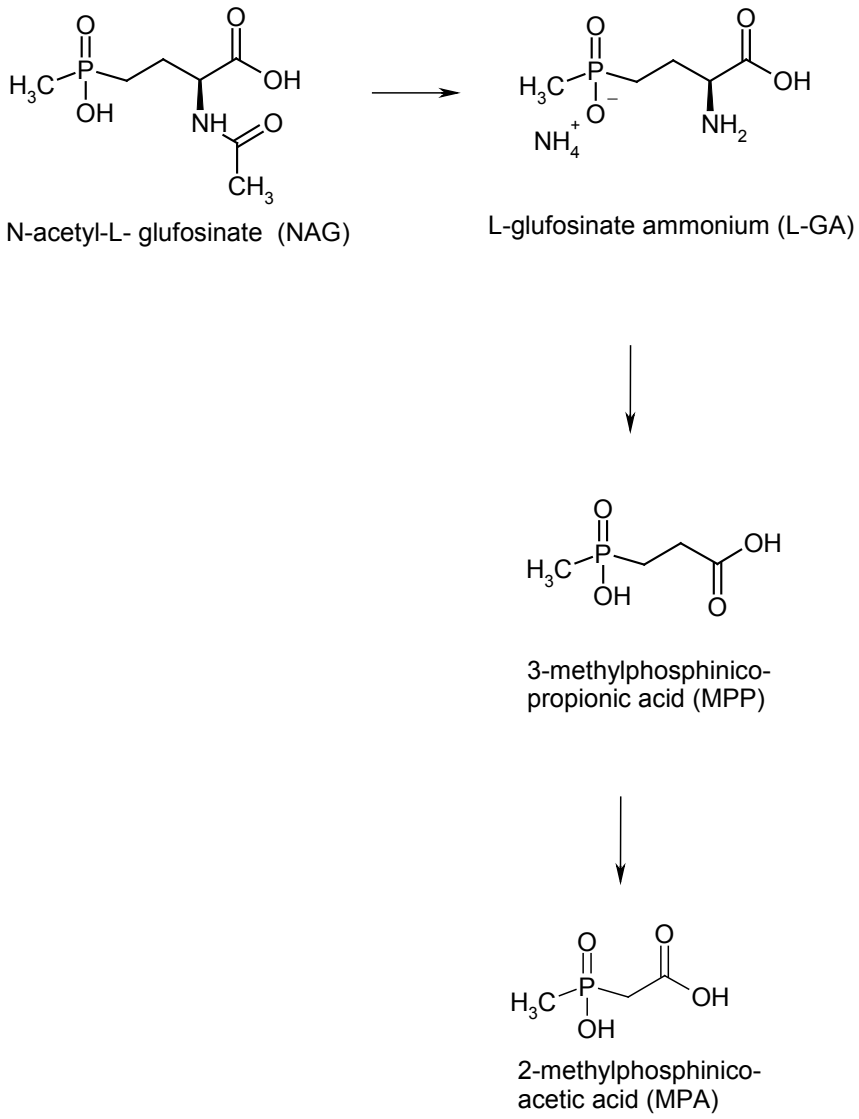


Figure 2 Proposed metabolic pathway of L-NAG in goat and hen

Plant metabolism

Glufosinate ammonium is typically used for three different situations:

- Directed sprays for weed control (crop not intentionally treated)
- Use as a crop desiccant to facilitate the harvest (crop treated)
- Selective use in genetically modified glufosinate-tolerant crops (crop treated)

For weed control where the crop is not treated, metabolism studies were conducted in corn/maize, spring wheat following pre-emergent application and in potato, apple, grape and lettuce with uptake via the roots. The use as a desiccant was investigated in potato, oilseed, rape and beans following foliar spray application. In genetically modified plants resistant genes (PAT or BAR) have been incorporated coding for the enzyme phosphinotricine acetyltransferase that detoxifies the herbicidally acting L-enantiomer by acetylation resulting in tolerance to glufosinate. The use as a

selective herbicide on tolerant plants was investigated in the genetically modified, glufosinate-tolerant crops corn/maize, oilseed rape, soya bean, rice, tomato and cotton after foliar application.

As glufosinate is a non-selective contact herbicide, direct application to conventional (susceptible) plants damages them. Investigation on metabolism in conventional plants has therefore in part been conducted using artificial systems such as excised roots and leaves and cell cultures of a variety of weed and crop species. In these studies the unstable intermediate PPO is formed via deamination and undergoes rapid decarboxylation to form MPP. PPO can also be reduced to form MHB.

The physicochemical properties of glufosinate and NAG suggest these compounds should be translocated in plants. The translocation of glufosinate and metabolites has been studied. After application of L-glufosinate, NAG and further metabolites on distinct leaves there is preferential transport into the upper leaves with only a low level of translocation into the lower plant parts.

In metabolism studies relevant to directed use for weed control in conventional crops, ^{14}C-labelled glufosinate ammonium was sprayed to the ground below the foliar canopy of the crops. In the soil, glufosinate ammonium is rapidly degraded with only low uptake of degradation products by crops. The major residue component observed in these crops is MPP. MPA, a minor soil metabolite that could be taken up by plants, is not found in plants under these conditions. Some ^{14}C from labelled-glufosinate ammonium is incorporated into natural plant constituents. This is most likely from decarboxylation and mineralization of the ^{14}C-labelled part of glufosinate in soil followed by incorporation of ^{14}C via root uptake and/or absorption of $^{14}CO_2$ and incorporation as part of normal photosynthetic processes. Additional studies with absorption by the roots or the shoots in lettuce, grape vine as well as a number of confined crop rotation studies performed on wheat, lettuce and radish confirmed the metabolic pathway.

When applied to conventional crops for desiccation, the residues consist primarily of parent glufosinate and its metabolite MPP. This is to be expected as the plants are senescent at the time of application, or if still growing die quickly after application.

In genetically modified, glufosinate-tolerant crops, an exclusive plant metabolite is formed, i.e., NAG. Following foliar application of glufosinate ammonium to glufosinate-tolerant crops, both glufosinate enantiomers are absorbed but only the herbicidally active L- glufosinate is acetylated to NAG and thus detoxified. NAG levels increase as L- glufosinate decreases. D-glufosinate is slowly metabolized to form MPP together with lower levels of MPB and MPA. D-glufosinate may be converted to L-glufosinate, presumably by microbial action.

The metabolism of glufosinate ammonium by plants is well understood for the three different uses. The metabolism of glufosinate ammonium by conventional crops (glufosinate susceptible) is in principle the same for the five standard crop: fruit crops, cereal/grass crops, leafy crops, root crops and pulses and oilseeds. For these crop groups major residue components consist of parent glufosinate and the metabolite MPP. In case of glufosinate-tolerant crops the major metabolite is NAG. Figure 3 summarises the metabolism of glufosinate in conventional (susceptible) and tolerant plants.

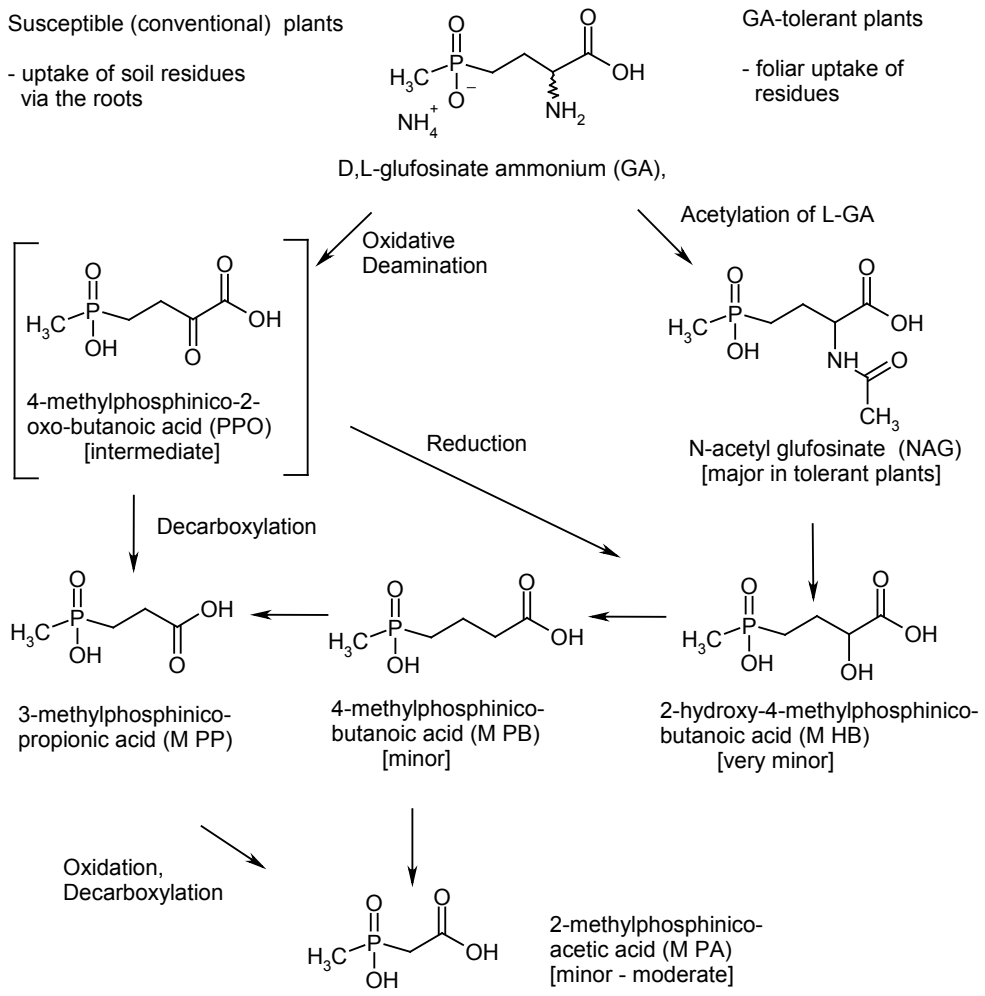


Figure 3 Metabolism of glufosinate in conventional (susceptible) and tolerant plants

Metabolism studies with excised shoots and leaves of conventional plants

Jansen *et al.* (2000) studied the metabolism of [^{14}C] glufosinate in excised shoots and leaves of 20 weed and non-weed species. Glufosinate was fed through the xylem and after 24 or 48 h of incubation, the plant material was examined for phytotoxic symptoms, analysed by autoradiography, extracted and analysed by high-performance liquid chromatography. [^{14}C] Glufosinate was rapidly absorbed with most of the radioactivity able to be extracted (91.3 to 99.7%). The main metabolite observed with all species was MPP with lower amounts of MHB also found in 14 of the 20 species studied. The metabolites, MPB, MPP and MHB were identified by Dröge-Laser *et al.* (1994) in a study of the metabolism of L-glufosinate in tobacco, alfalfa and carrot plants.

Weed control under an apple tree

The metabolism of [2-^{14}C]-glufosinate ammonium was investigated in an apple tree (*cv.* Cox Orange Rennet) following application to soil (Wieneke 1981 A35594, 1982 A28151, Künzler 1983 A28152). The tree was planted into sandy loam soil in its fourth cropping year and was located in the outdoor area. Application of glufosinate was to the soil surface at a rate equivalent to 1.5 kg ai/ha. During this application the tree stem was protected with plastic foil. At the time of application the tree had 24 fruits. Samples of leaves and fruit were stored frozen until analysis.

TRR in leaves increased from approximately 0.02 mg eq/kg one week after application to 0.41–0.46 mg eq/kg at 9 and 14 weeks after application (Table 9). The respective TRR level in apple fruits increased from 0.03 to approx. 0.10 mg eq/kg in mature fruits, 14 weeks after application. In the second year, TRR levels in leaves and fruits dropped to approximately one tenth of those in the first year.

Soil radioactivity showed a decrease of TRR in the upper 0–5 cm soil horizon from 1.10 mg eq/kg three weeks after application to 0.41 mg eq/kg 14 weeks after application. TRR in the 5–10 cm layer amounted to approximately ⅓ to ½ of the upper layer accompanied with even lower TRR levels in the 15–20 cm soil layer indicating no significant leaching.

Extractability of residues from mature apples using hot water was 89% of the TRR (0.10 mg eq/kg). The ^{14}C residues in the extracts consisted entirely of MPP.

Table 9 Total radioactive residues (TRR) in apple leaves, fruits and wood following application of [^{14}C] glufosinate ammonium to the soil below an apple tree (Wieneke 1981 A35594, 1982 A28151)

	TRR (mg /kg as glufosinate)	
Time after application	Leaves (from extension growth)	Fruit
First year of trial		
1 wk	0.019	0.033
3 wk	0.117	–
6 wk	0.352	–
9 wk	0.458	0.083
14 wk	0.405	0.104
spur shoots	0.811	
old wood	0.385	
Second year of trial		
15 months	0.051	0.012

Vegetation control below grapevines

Dorn (1987 A35543) studied the transfer of glufosinate related residues from soil to grapes. In a two-year field trial the soil (texture: sandy soil) beneath two grapevine plants (*cv.* Müller-Thurgau, non-grafted) was treated with [^{14}C] glufosinate ammonium in order to determine the uptake of radioactive residues into berries and leaves. The application mixture was evenly applied to the soil beneath the vine plants. In the first year of the trial, one application was made to the soil beneath both vine plants. In the second year, two applications (interval 57 days) were made to the soil of one of the two plants. The application rate was generally 1.5 kg ai/ha per treatment. The first application was made using [2-^{14}C]-, the second and the third applications with [3,4-^{14}C]-glufosinate. Grapes and leaves were sampled 61 days after application in the first year and 57 days after the first as well as 70 days after the second application in the second year. In addition, soil core samples (0–20 cm) were taken 61 days after the first and 70 days after the last application.

TRR in grapes were 0.007 mg eq/kg at 61 days after the first application, 0.008 mg eq/kg at 57 days after the first application in the second year and < 0.005 mg eq/kg in both vine plants 70 days after the last application in the second year. TRR in the leaves were 0.007 mg eq/kg at 61 days after the first application, 0.01 mg eq/kg at 57 days after the first application in the second year and < 0.005 mg eq/kg 70 days after the last application.

There was limited leaching of ^{14}C in soil with the majority of ^{14}C retained in the upper soil layers.

Uptake by lettuce simulated in a hydroponic test

Dorn (1981 A21859) studied the metabolism of [3-^{14}C]-glufosinate ammonium in lettuce (*cv.* Selma) using a hydroponic test approach. The lettuce was sown in soil and grown until the 10-leaf growth stage. The plants were then transferred into glass beakers containing tap water. The roots of the plants were dipped into the water by way of the narrow end of a funnel so that the leaves would not contact

the water. After an acclimatization period of 3 days to allow healing of potentially damaged roots, [^{14}C] glufosinate ammonium was added to give a glufosinate ammonium concentration of 3 mg/L in the hydroponic water. The roots were not sterilised to eliminate attached microbes. The test plants were cultivated for 10 days in a greenhouse.

After 10 days of hydroponic incubation, ^{14}C in leaves were 0.85 mg eq/kg and 8.8 mg eq/kg in the roots. About 90% of TRR in leaves was extracted with water and was identified as MPP.

Pre-emergence (soil) application to potatoes

The metabolism of [3,4-^{14}C]-glufosinate was investigated in potatoes (cv. Berolina) grown outdoors by Stumpf (1994a A52894). Potato tubers were pre-sprouted in plant pots filled with sandy loam soil (OC 0.95%, pH 5.5). The soil application rate was equivalent to 1.0 kg ai/ha. The application amount was evenly mixed with a batch of the test soil resulting in a treated soil concentration of 3.33 mg ai/kg wet weight. When the potato shoots appeared at the soil surface of the plant pot, the treated soil was applied as a top layer to the plant pots to simulate a pre-emergence application.

The plants were harvested 83 days after soil treatment and separated into tubers, leaves and stems. In addition, soil samples were taken at the day of treatment and the day of harvest. A part of the tubers was radio-assayed and the rest was stored in a cellar room at 18 °C for approximately 3–4 months. During this period young sprouts developed which were separately sampled and radio-assayed. A part of the plant samples was stored frozen at –20 °C for a total period of three years. During this period the samples were repeatedly extracted and analysed. The re-analysis showed that the residues were stable during storage.

TRR levels ranged from 0.28 to 0.34 mg eq/kg in tubers, their peels and pulp and were 0.15–0.89 mg eq/kg in the foliage (leaves and stems) 83 days after application (Table 10). Radioactivity was 0.42 mg eq/kg in sprouts freshly developed during subsequent 4-months storage.

Extractability of ^{14}C using water/methanol was > 90% of TRR from the tubers and the newly developed sprouts and > 80% of TRR from leaves and stems. MPP was the only residue component detected and was present at 0.25–0.33 mg eq/kg in tubers and 0.13–0.75 mg eq/kg in sprouts and foliage.

The TRR in soil decreased from 3.2 mg eq/kg dry soil at day 0 to 0.35 mg eq/kg at day 83 after application. At day 83, approximately 50% of TRR present was extracted comprising glufosinate (13.5% TRR), MPP (16% TRR), NAG (12% TRR) and MPA (3% TRR).

Table 10 Residues in potatoes 83 days following pre-emergence application to soil of [^{14}C] glufosinate ammonium at an application rate of 1.0 kg ai/ha (Stumpf 1994a A52894).

		Extracted				Unextracted	
Sample	TRR	Total		MPP			
	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)	%TRR	(mg eq/kg)
Leaves	0.886	84.2	0.746	84.2	0.746	3.9	0.034
Stems	0.153	83.7	0.128	83.7	0.128	20.2	0.031
Tubers (Whole)	2.280	90.5	0.253	90.5	0.253	5.6	0.016
Peel	0.343	96.6	0.331	96.6	0.331	9.9	0.034
Pulp	0.285	93.5	0.266	93.5	0.266	8.1	0.023
New sprouts	0.418	97.8	0.409	97.8	0.409	7.8	0.033

Application to barley

Mersey *et al.* (1990) studied the absorption, translocation and metabolism of glufosinate in barley, a relatively glufosinate tolerant conventional crop. Limited metabolism occurred following foliar application with glufosinate accounting for 88% TRR in shoots and 94.8% TRR in roots at up to 96 hours after treatment. Low levels of MPP were detected in shoots (4.1% TRR) but not in roots.

Pre-emergent application to corn/maize

Stumpf (1989 A41451) studied the metabolism of [3,4-^{14}C]-glufosinate in corn (*cv.* Funk's hybrid G-4733) that was sown in an outdoor plot three days before spray application. The application rate was equivalent to 1.9 kg ai/ha with the spray mixture evenly sprayed to the soil (silt loam). Plants were harvested at the forage stage (80 days after application) and finally at maturity (164 days after application). All samples were stored at -20 °C until analysis.

Generally low TRR levels were detected in the corn samples, indicating limited uptake of soil residues of [^{14}C] glufosinate. At 80 days after application ^{14}C residues in forage were 0.068 mg eq/kg while at harvest ^{14}C residues were 0.115 mg eq/kg in fodder and 0.035 mg eq/kg in grain. The ^{14}C residues in leaves and inner parts of the cobs at harvest were similar (0.079 mg eq/kg and 0.051–0.081 mg eq/kg, respectively).

Characterisation of ^{14}C was only attempted for fodder harvested 164 day after treatment. Approximately 60% of TRR was extracted with water and was due to MPP. No parent glufosinate or other metabolites were detected in any of the samples. The ^{14}C residues in the PES of fodder (approximately 43% TRR) were further characterized by chemical and enzymatic methods and could be attributed to incorporation of ^{14}C into natural components; hemicellulose fraction (23.9% TRR), lignin fraction (2.0% TRR), cellulose fraction (3.8% TRR) as well as proteins and starch. The incorporation of ^{14}C into different plant constituents suggested the assimilation of soil formed $^{14}CO_2$ during photosynthesis.

The initial post-application concentration in soil was approximately 2.0 mg/kg soil. At harvest, 164 days after application, the concentration was 0.22 mg eq/kg in the upper 0–5 cm and 0.028 mg eq/kg in the 5–10 cm soil layers.

Pre-sowing (soil) application to corn/maize

In another study the metabolism of [3,4-^{14}C]-glufosinate ammonium was investigated in corn (*cv.* Ricca) sown following application of glufosinate to a cover crop (Schwalbe-Fehl 1987 A36441). The container was maintained under outdoor conditions (Seville, Spain). An application equivalent to 1.0 kg ai/ha was made to a mustard crop that provided about 30% coverage of the soil surface. Six days after application when the mustard was already damaged, 40 corn grains were sown. The test plants were cultivated under natural meteorological conditions, but protected from rain. Plants were harvested 37, 62, 94, and finally 125 days after application. All samples collected were stored frozen at -20 °C until analysis.

Generally only low TRR levels were detected in the corn samples indicating limited uptake of radioactivity from the soil (Table 11). The highest levels of ^{14}C residue were in dry leaves sampled 94 days after application (0.26 mg eq/kg) and in the male blooms at harvest (0.20 mg eq/kg). However, as the dry leaves were in direct contact with the soil they may have become contaminated and may not be representative of ^{14}C levels resulting from uptake via the roots. Grain radioactivity was 0.014 mg eq/kg being only slightly higher than the LOQ (0.01 mg eq/kg).

Only the dry leaves with soil contact taken 94 days after application, and the dry leaves and cob leaves removed at harvest contained sufficient ^{14}C to attempt further characterisation. The only glufosinate related compound detected was MPP. No parent glufosinate was detected in any of the samples.

In soil, the majority of the applied radioactivity remained in the upper 10 cm layer of soil. Parent glufosinate and MPP were the only relevant compounds identified in the soil. Glufosinate declined from a maximum of 0.79 mg/kg (85% of TRR) to 0.01 mg/kg (14% of TRR) at day 125. MPP reached a maximum absolute concentration of 0.31 mg eq/kg by day six followed by a decline to approximately 0.03 mg eq/kg by day 125. MPA was only detected at trace levels ≤ 0.03 mg eq/kg).

Table 11 Total radioactive residues (TRR) in corn following pre-sowing application of [^{14}C] glufosinate ammonium at an application rate of 1.0 kg ai/ha (Schwalbe-Fehl 1987 A36441).

Days after pre-sowing application	Plant sample	TRR (mg eq/kg)
37	whole plant	0.07
62	whole plant	0.03
94	green leaves	0.04
	dry leaves [a]	0.26
	immature cobs	0.01
	cob leaves	0.01
	stem	0.01
	male bloom	0.06
125	dry leaves	0.08
	stem	0.03
	cob leaves	0.06
	grain	0.01
	cob	0.01
	male bloom	0.20

[a] Leaves were contaminated by contact with the soil

Pre-emergence (soil) application to spring wheat

Dorn (1986 A34005) studied the metabolism of [2-^{14}C]-glufosinate ammonium, in spring wheat (*cv.* Kolibri) sown in a plant container that was filled with sandy loam soil (OC 1.46%, pH 7.4). The application rate was equivalent to 1.25 kg ai/ha. The spray mixture was evenly sprayed to the surface of the plant container two hours after the wheat seeds were sown. The test plants were cultivated under natural meteorological conditions with additional watering as required.

Levels of ^{14}C in plant parts were low at 0.20 mg eq/kg in forage at day 60, 0.49 in straw, 0.50 in husks and 0.22 mg eq/kg in grain at harvest 119 days after pre-emergence application (Table 12).

Extractability of ^{14}C with water at room temperature was 33% from grain, 46% from straw and 70% from the husks. MPP was the main residue component in all samples (32–35% TRR in straw, 29–38% TRR in grain or approx. 0.06 mg eq/kg and 73–96% TRR in husks). Glufosinate was not unambiguously identified in any matrix. Results from anion exchange chromatography suggested glufosinate accounted for ≤ 5% of TRR (≤ 0.02 mg/kg).

Table 12 Residues in wheat following pre-emergence application of [^{14}C] glufosinate ammonium at an application rate of 1.25 kg ai/ha (Dorn 1986 A34005)

Days after application	Plant sample	TRR (mg eq/kg)	%TRR
60 (forage)	leaves and stem	0.20–0.21	–
119 (mature plants)	straw	0.49	100
	extracted with water	approx. 0.23	46
	MPP [a]	approx. 0.16	32–35
	glufosinate [b]	< 0.02	≤ 2
	husks	0.50	100
	extracted with water	approx. 0.35	70
	MPP [a]	approx. 0.37	73–96
	glufosinate [b]	< 0.02	≤ 5
	grain	0.22	100
	extracted with water	approx. 0.07	33
	MPP [a]	approx. 0.06	29–38
	glufosinate [b]	< 0.02	≤ 5

[a] estimated by anion exchange chromatography or determined by GLC/FPD and GLC/MS

[b] estimated by cation exchange chromatography

Plant metabolism studies with glufosinate ammonium used as crop desiccant

Potato pre-harvest desiccation

Schwalbe-Fehl (1988 A39955) studied the metabolism of [3,4-^{14}C]-glufosinate ammonium in potatoes (*cv.* Berolina) after its use as a desiccant to facilitate harvest. The potatoes were cultivated in plant pots filled with sandy loam soil (OM 1.57%, pH 5.7) and maintained under outdoor conditions until the first signs of maturity became visible. Ten days before the final harvest, the plants were homogenously sprayed at an application rate equivalent to 1.0 kg ai/ha.

High levels of ^{14}C (200–250 mg eq/kg) were detected in dried leaves a few days after treatment but only low levels of the ^{14}C translocated into the tubers (Table 13). Mean TRRs in large tubers were 0.014 mg eq/kg ten days after desiccant application with slightly higher levels in the peel.

Parent glufosinate was the major residue component accounting for 72–85% of the TRR in the dried leaves and in the peels of large tubers. MPP contributed to 5–8% of TRR in the leaves. The soil metabolite MPA was not detected in potatoes.

Table 13 Radioactive residues in potatoes following foliar spraying of [^{14}C] glufosinate at a use rate of 1.0 kg ai/ha for desiccant application (Schwalbe-Fehl 1988 A39955)

Days after		TRR	Extracted (%TRR)				Unextracted
application	Sample	(mg eq/kg)	Total	Glufosinate	MPP	MPA	(%TRR)
3	Dry leaves	247.6	80.2	72	8	< 0.05	19.8
7	Dry leaves	188.0	90.6	85	5	< 0.05	9.4
10	Dry leaves	236.3	89.5	83	6	< 0.05	10.5
	Green leaves	10.0	88.7	82	7	< 0.05	11.3
	Tuber whole	0.014 [a]	–	–	–	–	–
	Peel	0.039	80.8	81	< 0.05	< 0.05	19.2
	Pulp	0.029	–	–	–	–	–

[a] Different samples were analysed for whole tubers and in the preparation of pulp and peel.

In an additional study the translocation behaviour of [3,4-^{14}C]-glufosinate ammonium was investigated by Köcher (1994 A56990) in potatoes (*cv.* Grata) after application to fully developed leaves. The potatoes were pre-cultivated in plant pots filled with sandy soil with a high organic matter content under outdoor conditions. The plants were treated at two growth stages, prior to and after occurrence of foliar senescence. A solution (300 µL) containing 0.2% glufosinate ammonium was applied to three leaves in the form of micro-droplets using a syringe. The droplets were evenly spread over the surface of the leaves.

Seven days after treatment, the plants were harvested and separated into treated leaves, shoots above and below the treated leaves, as well as into peeled tubers and tuber peelings. The translocation of ^{14}C in potato plants was low. Seven days after treatment, approximately 0.1–1.1% of the applied ^{14}C was detected in the shoots above the treated leaves and 0.05–0.07% in the shoots below the treated leaves. Approximately 0.1–0.2% of the applied ^{14}C was transported basipetally into the tubers with 0.01–0.05% located in the peel, independent of the growth stage of the leaves at application.

Beans pre-harvest desiccation

The metabolism of [3,4-^{14}C]-glufosinate ammonium was investigated in beans (*cv.* Vollenda) after use as a desiccant to facilitate harvest (Stumpf 1995a A55061). The bean plants were pre-cultivated in pots (sandy loam soil OC: 1.13%, pH 5.8). When the first signs of maturity of the bean plants was apparent (hulls parchment-like) they were homogenously sprayed at an application rate equivalent to 0.6 kg ai/ha. The treated plants were cultivated in a greenhouse until harvest. Plants were harvested 0, 4, 7 and 14 days after treatment and divided into pods and leaves. The pods were separated into hulls and beans.

Radioactivity was 100–400 mg eq/kg in the leaves sampled 0, 4, 7 and 14 days after treatment (Table 14). Levels in hulls of the beans were 25–40 mg eq/kg with significantly lower levels detected in the beans (0.05–0.30 mg eq/kg). A significant amount of TRR could be rinsed off with water from

leaves and pod hulls. Analyses of the surface wash showed that glufosinate was the major residue component (89–100%).

Unusually 12% of TRR was removed by washing from the beans collected at final harvest. As seeds are protected from spray this surface residue is assumed to result from contamination during the separation of the seeds from the hulls.

At final harvest, 14 days after treatment, more than 99% of the radioactive residues remaining after the surface washing could be extracted from the homogenized beans, hulls and foliage samples with water/methanol. The majority of the residues at harvest (> 78% TRR) was identified as glufosinate; 0.3 mg/kg in beans, 29.6 mg/kg in hulls and to 91 mg/kg in foliage. The metabolite MPP was detected in the leaves and accounted for no more than 12.1% TRR. Two to three additional components in the range of 0–5% TRR were detected in several samples and corresponded to minor impurities of the test substance.

Table 14 Radioactive residues in beans following foliar spraying of [^{14}C] glufosinate ammonium for crop desiccation (Stumpf 1995a A55061).

Days after application	sample	TRR (mg eq/kg)	Surface rinse (%TRR)	Extract (%TRR)	Surface rinse/extract glufosinate (%TRR)	(mg eq/kg)	MPP (%TRR)	(mg eq/kg)
0	Beans	0.08	–	–	–	–	–	–
	Hulls	25.6	75	26	75	19.2	–	–
	Leaves	133	24.3	28[a]	22.4	29.8	0.3	0.4
4	Beans	0.09	–	–	–	–	–	–
	Hulls	40.5	56.4	12.1 [a]	65.1	26.4	–	–
	Leaves	351	41.0	30.3 [a]	65.9	231.5	1.0	3.7
7	Beans	0.05	–	–	–	–	–	–
	Hulls	42.2	79.3	18.5	92.9	39.2	1.0	0.4
	Leaves	401	60.5	29.4	83.1	333.3	1.9	7.5
14	Beans	0.3	11.5	96.5	108	0.3		–
	Hulls	31.1	66.5	23.3	95.2	29.6	1.3	0.4
	Leaves & stems	116	8.1	91.8	78.5	91.0	12.1	14.0

[a] Non-exhaustive extraction as only one extraction step was employed

Rape seed – pre-harvest desiccation

Stumpf (1991 A46783) studied the metabolism of [3,4-^{14}C]-glufosinate ammonium in rape seed (*cv.* Noko) after use as a desiccant to facilitate harvest. The rape seeds were pre-cultivated and transplanted into pots (sandy loam soil; OC: 1.13%, pH 5.8). When the first signs of maturity became apparent (hulls parchment-like in colour and consistency) the rape seed plants were homogenously sprayed at an application rate equivalent to 0.6 kg ai/ha. The treated plants were cultivated in a greenhouse until harvest. Plants were harvested 0 and 7 days after treatment and divided into seeds, hulls and leaves plus stems. Rape oil was prepared from ground seeds by extraction with hot hexane after washing with water. The filter cake was washed with hexane and the combined hexane extract was washed with water and dried with anhydrous sodium sulphate. The hexane was evaporated and the remaining oil was dissolved in ethyl acetate.

Highest ^{14}C levels were detected in the dry hulls, 0 and 7 days after treatment (60–100 mg eq/kg) followed by the foliage (leaves and stems, 20–50 mg eq/kg) (Table 15). Significantly lower ^{14}C residues were detected in the seeds (1.76 mg eq/kg 7 days after treatment). More than 96% of TRR was extracted from all plant matrices with water and methanol.

The majority of the^{14}C in all seed, hull and leaf samples consisted of the unchanged glufosinate. A minor metabolite MPP only accounted for 2–4% of TRR.

Table 15 Radioactive residues in oil seed rape following foliar spraying of [^{14}C] glufosinate ammonium at a use rate of 0.6 kg ai/ha for desiccant application (Stumpf 1991 A46783)

			Extracted				
Days after	Sample	TRR	Total	Glufosinate		MPP	
application		(mg eq/kg)	(%TRR)	(%TRR)	(mg eq/kg)	(%TRR)	(mg eq/kg)
0	Seeds	3.77					
	Dry hulls	65.4					
	Green hulls	19.8					
	Leaves & stems	22.7					
7	Seeds	1.8	104.9	94.5	1.4	3.9	0.06
	Hulls	104.7	96.6	89.3	96.6	2.0	2.2
	Leaves & stems	45.8	102.9	95.3	42.5	2.8	1.2

A sample of seeds was processed into oil. Seeds were washed prior to extraction with the majority of ^{14}C in seeds (1.76 mg eq/kg) recovered in the wash water (90% TRR) and comprising mainly of glufosinate. Purified oil prepared from washed seeds (0.19 mg eq/kg) contained extremely low levels of ^{14}C at 0.004 mg eq/kg oil. The ^{14}C residues in the filter cake of the seeds following hexane extraction were 0.26 mg eq/kg.

Plant metabolism studies with glufosinate ammonium for selective use in genetically modified, glufosinate-tolerant crops

Glufosinate-tolerant tomato

Stumpf (1995b A53473) studied the metabolism and enantiomeric composition of [3,4-^{14}C]-glufosinate ammonium in tomato plants (*cv.* TOMAC 15-3, genetically modified for glufosinate tolerance). Tomato shoots were transplanted at the 4-leaf stage into pots (sandy loam soil; OC: 1.13%, pH 5.8) and cultivated in a greenhouse. When at the 7–8-leaf stage, plants were sprayed at an application rate equivalent to 0.80 kg ai/ha.

Following foliar application of racemic [^{14}C] glufosinate ammonium to glufosinate-tolerant tomato plants the radioactivity in foliage decreased from approximately 130 mg eq/kg at 0–1 days after treatment to 9 mg eq/kg at maturity, 74 days after treatment (Table 16). Leaves newly grown 12–27 days after treatment and therefore not directly treated, also showed significant residues (2.5–11.5 mg eq/kg) but at concentrations lower than the old leaves taken at similar sampling intervals. TRR in mature tomato fruits harvested 60–74 days after treatment were 0.118–0.297 mg eq/kg.

More than 50% of TRR could be rinsed off (surface residues) from treated leaves and stems sampled up to 32 days after spraying. At the final harvest the washable portion was 19–42% of TRR. Extraction four to five times with water/methanol (9/1, v/v) or pure water recovered more than 95% of TRR.

All surface rinses contained unchanged glufosinate as the main ^{14}C residue component (> 90%). After rinsing, the remaining ^{14}C residues extracted from sprayed foliage samples were composed of NAG as a major metabolite and unchanged glufosinate at a ratio of approximately 50:50. MPP was present as a minor metabolite (< 10% TRR). In fruit that had developed after spraying, ^{14}C residues consisted almost entirely of NAG with no glufosinate detected.

The enantiomeric composition of glufosinate in leaves at 74 days after application was investigated. Glufosinate residues in the surface rinse were composed of D- and L-glufosinate in the original ratio of approximately 50:50. In contrast, glufosinate in extracts of rinsed leaves was composed of the D- and L-enantiomers in a ratio of approximately 90:10 or higher.

Additionally a comparison of residues was conducted with conventional (wild-type) tomato plants and genetically modified tomato plants. The plants were cut and placed in nutrient solutions with racemic [^{14}C] glufosinate ammonium. Shoots from tolerant plants took up about twice as much ^{14}C from solution compared to shoots from conventional plants. The major residues in tolerant plants were glufosinate (45.7% TRR leaves, 21.2% stems TRR, 100% D-enantiomer) and NAG (51.3% TRR

leaves, 74.6% TRR stems) whereas in conventional plants they were glufosinate (90.8% TRR leaves, 100% TRR stems, 54% D-enantiomer) with small amounts of MPP also present (4.4% TRR in leaves). No NAG was detected in the conventional tomatoes.

Table 16 Nature of residues in selected samples of glufosinate-tolerant tomatoes following foliar spray treatment of [^{14}C] glufosinate ammonium at a 0.8 kg ai/ha (Stumpf. 1995b A53473)

		TRR	Rinse	%rinse TRR				Extract	%extract TRR			
DAT	Sample	(mg eq/kg)	%TRR	glufosinate	NAG	MPP	UI	%TRR	glufosinate	NAG	MPP	UI
1	leaves	133	68.8	94.6	-	1.5	3.9	31.2	63.6	32.6	–	3.9
32	leaves	18.14	56.6	92.9	1.2	1.4	4.5	43.4	57.0	36.9	3.5	2.6
12	new leaves	11.48							30.0	65.2	1.6	3.2
14	new leaves	5.31							16.9	83.1	–	–
27	new leaves	2.47							–	100	–	–
74	leaves	9.09	42.6	86.2	3.3	4.1	6.4	57.4	43.9	50.5	5.6	–
74	leaves	8.66							50.8	41.5	2.5	5.2
60	red fruit	0.297							–	100	–	–
74	red fruit	0.203							–	100	–	–
74	red fruit	0.145	1.5	–	–	6.2	–	98.5	–	93.8	–	–

UI = unidentified

The unextracted residues amounted to 3–5% of TRR in all matrices of mature plants (74 days after treatment)

Glufosinate-tolerant soya beans

The metabolism of [3,4-^{14}C]-glufosinate ammonium was investigated in soya beans (*cv.* Ignite), genetically modified by incorporation of the "*pat*" gene to be tolerant of glufosinate (Rupprecht 1994 A53607, Rupprecht 1996 A55809). The soya beans were grown in a sandy loam soil (OM: 1.9%) under a polycarbonate roof outdoors. The soya bean plants were sprayed at the third trifoliate leaf stage (V3) and one month later at full bloom stage (R2) at application rates equivalent to 0.50 kg ai/ha.

TRR in foliage decreased from approximately 28–71 mg eq/kg at the days of the first and the second treatment to 3.2, 4.7 and 1.4 mg eq/kg in mature straw, pods and beans respectively, at 85 days after the second treatment (Table 17). Most of the residues were able to be rinsed off with water with the remaining able to be extracted with water after homogenisation. However, in beans more than 20% of TRR remained un-extracted. This portion was completely released by acid and base hydrolysis of the bean post-extraction solids.

The foliage water rinse from the day of application (43–47% TRR) exclusively contained the parent glufosinate. The ^{14}C extracted from foliage after rinsing contained additional glufosinate (26–31% TRR), however, even at day zero 11–12% of ^{14}C residue was NAG and approximately 1% MPP as well as minor amounts of MPA (0.3%).

NAG was the main residue component in forage collected one day before the second spray (60% TRR) followed by glufosinate (23% TRR) with MPP and MPA observed as minor metabolites. The profile of residues at harvest was similar for the major components in straw (NAG 53% TRR, glufosinate 18.5% TRR) however the minor metabolites MPP and MPA were present as larger proportions of the TRR. Similar results were found for pods.

In beans, NAG was the main residue component of the extracts (50% TRR) with small amounts of MPP (13% TRR) and MPA (5% TRR) also detected.

Table 17 Metabolite profile of radioactive residues extracted from glufosinate-tolerant soya beans following two foliar spray treatments of [^{14}C] glufosinate ammonium at single use rates of 0.5 kg ai/ha

Matrix [a]	glufosinate		NAG		MPP		MPA		Total identified	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Forage	23.2	0.448	60.2	1.157	6.5	0.126	0.7	0.014	90.6	1.745
Straw	18.5	0.575	53.2	1.655	13.6	0.423	5.7	0.176	91.0	2.829
Pods	5.8	0.287	62.6	3.092	22.3	1.098	2.9	0.142	93.6	4.619
Beans [b]	6.2	0.091	60.8	0.891	16.0	0.233	7.1	0.103	90.1	1.318

[a] Forage collected one day before 2nd treatment; straw, pod and bean samples were obtained from mature plants 85 days after the last treatment.

[b] Including release after base and acid hydrolysis of the beans (releasing a significant portion of NAG)

Obrist (1998 C000897) also studied the metabolism of [3,4-^{14}C]-glufosinate ammonium in transgenic soya beans, genetically modified by incorporation of the "*pat*" gene. Soya bean seeds were planted in an outdoor plot covered by a plastic house to protect from the environment. The soil used was a sandy loam (OC: 0.6%, pH 6.9). The soya bean plants were sprayed at the 6–7 trifoliate leaf stage and one month later when the plants reached a height of 65–70 cm and the pods were 2–6 cm long. The treatments were later than the intended commercial use-pattern in order to generate high residues for easier analysis. The application rates were equivalent to approximately 0.53–0.55 kg ai/ha.

TRR in foliage decreased from about 28–51 mg eq/kg on the days of the first and the second treatment to 21.9, 18.0 and 5.0 mg eq/kg in mature straw, husks and beans respectively at 84 days after the second application (Table 18). The majority of ^{14}C residues (> 80% TRR) were extracted with water with approximately 60–70% of TRR recovered in the first extraction.

As in the earlier study, glufosinate was the main ^{14}C residue component in foliage extracts on the day of application (55% TRR) with significant metabolism already occurring as NAG accounted for 25% of the TRR (Table 19). Forage harvested 17 days after the first treatment contained NAG as the main ^{14}C residue component (30% TRR) together with glufosinate (18.5% TRR). MPP and MPA were minor metabolites in the range of 1–6% of TRR.

The profile of ^{14}C components in aqueous extracts of straw, husks and beans at final harvest 84 days after the last application was similar. NAG was the main ^{14}C residue component amounting to 28–47% of TRR followed by glufosinate (11–14% TRR) and MPP (9.6–13% TRR).

The metabolic pathway of glufosinate in glufosinate-tolerant soya beans expressing the "*pat*" gene is proposed in Figure 4.

Table 18 Total radioactive residues (TRR) in glufosinate-tolerant soya beans following two foliar spray treatments of [^{14}C] glufosinate ammonium (Obrist 1998)

Day after application	Sample	TRR	Extraction with water		Acid /base/ enzymatic hydrolysis	Unextracted
[a]		(mg eq/kg)	1st extraction (%TRR)	2nd extraction (%TRR)	(%TRR)	(%TRR)
0/1 or 2	Whole plants	28–51	81–86	6–10	–	0.7–1.5
5/1 or 2	Whole plants	24–28	78–83	11–12	–	3.3–3.9
17/1	Forage	10.2	66.0	20.8	–	7.8
84/2	Straw	21.9	72.4	13.2	8.5 [b]	2.6
	Husks	18.0	59.0	21.7	–	7.6
	Beans	5.01	72.5	10.4	–	3.0

[a] *x/y* means *x* days after the *y*th treatment

[b] Hydrolysis of the extracted soya bean straw released 2.4% of TRR with 0.1 N NH_4OH, 2.2% with 0.1 N HCl, 1.5% with pronase E, 0.8% with cellulase, and 1.6% of TRR with 3 N HCl.

Table 19 Metabolite profile of the radioactive residues in the first aqueous extract from glufosinate-tolerant soya beans following two foliar sprays with [^{14}C] glufosinate (Obrist 1998)

Day after application [a]	glufosinate		NAG		MPP		MPA		unknowns	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Whole plant										
0/1	55.4	28.5	24.7	12.7	0.9	0.45	0.5	0.27	3.5	1.80
5/1	43.2	12.2	25.2	7.1	4.2	1.2	0.7	0.20	9.4	2.61
Forage										
17/1	18.5	1.9	29.4	3.0	6.3	0.64	1.1	0.12	9.5	0.97
Straw										
84	14.3	3.1	35.0	7.7	9.6	2.1	1.8	0.39	8.1	1.75
Husks										
84	11.7	2.1	27.7	5.0	13.0	2.3	1.3	0.23	11.6	2.10
Beans										
84	10.6	0.53	46.7	2.3	11.6	0.58	1.7	0.08	18.3	0.92

[a] *x/y* means *x* days after the *y*th treatment

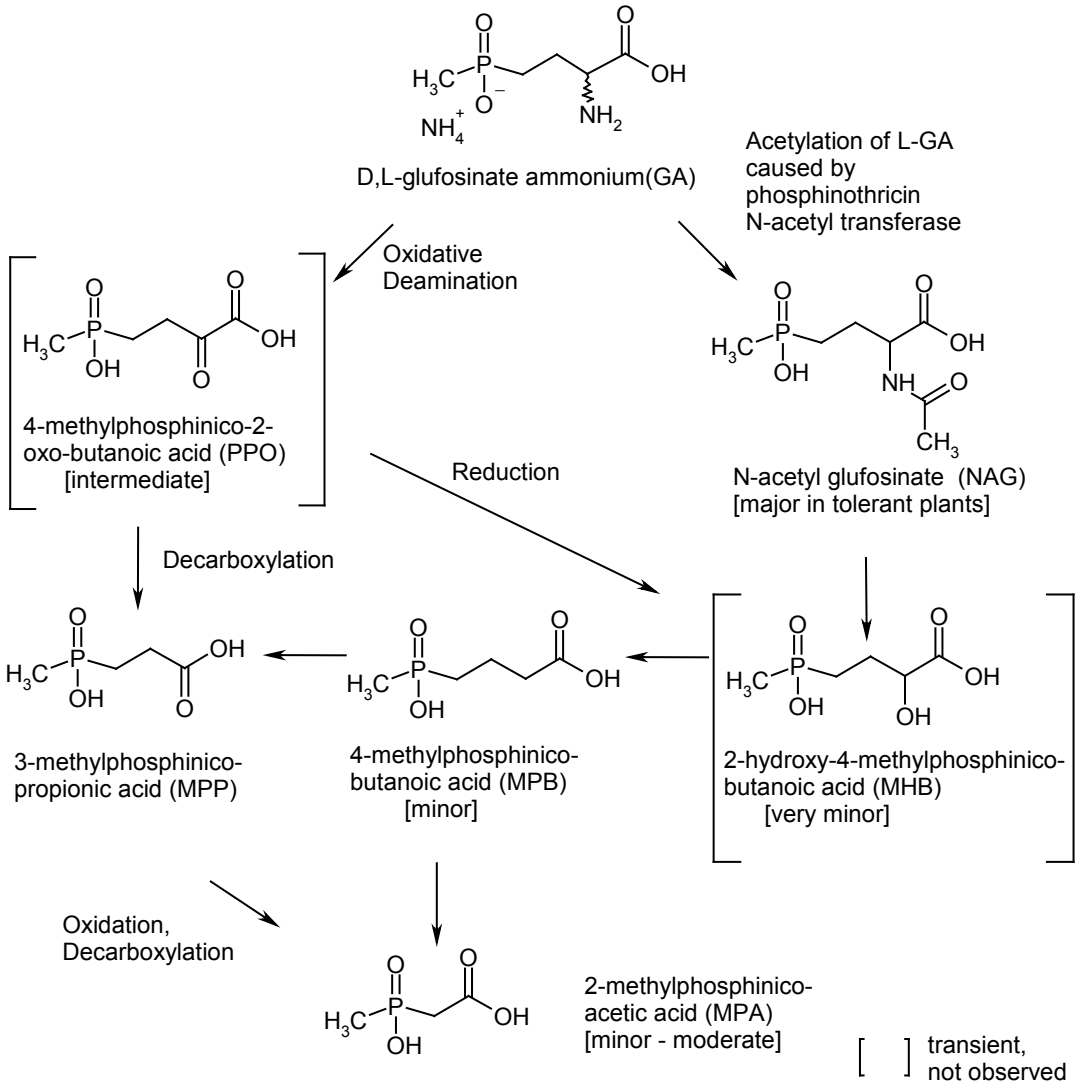


Figure 4 Proposed metabolic pathway of glufosinate in glufosinate-tolerant soya beans

Glufosinate-tolerant sugar beet

The metabolism of [3,4-^{14}C]-glufosinate was investigated in sugar beets (*cv.* Hybrid 1-Transgenic), genetically modified to be tolerant of glufosinate (Allan 1996 A58109). Seeds were sown into plant containers filled with sandy loam soil (OC: 0.91%, pH 6.25). The plants were cultivated in a greenhouse. Sugar beets were sprayed twice with [^{14}C] glufosinate ammonium at 0.6 kg ai/ha, the first spray 5 weeks after sowing and the second 22-days later.

The enantiomeric composition of the residues on/in glufosinate-tolerant sugar beet leaves was followed for 15 days after the first treatment (Table 20). The ratio of D:L-isomers remained unchanged for the surface (rinse) residues. In contrast, for absorbed ^{14}C residues (extracts of rinsed plants) the L-glufosinate component was converted to NAG leading to a progressive increase in the D:L-isomer ratio.

The extent of absorption of ^{14}C by leaves increased with time after the second application (Table 21). The unabsorbed portion recovered in the water rinses of leaves decreased from 60% of TRR on the day of the second application to 3% of TRR 146 days later, while the amount absorbed (extracts of rinsed leaves + unextracted) increased from 42 to 95% of TRR. Almost all of the ^{14}C on the surface was represented by parent glufosinate. Absorbed glufosinate accounted for 24–28% TRR while the proportion of NAG increased with time from 13 to 67% TRR. MPP was present as a minor component of ^{14}C at 0.4 to 2.7% of TRR.

The extractability from beet roots was 96–97% of TRR at all sampling intervals. The ^{14}C consisted of NAG (63–68% TRR) with lower amounts of glufosinate (19–33% TRR). MPP was present as a minor component (2–6% TRR).

Only L-glufosinate is metabolised to NAG. As a result, glufosinate (as D-glufosinate) remains a significant component of the ^{14}C residue in leaves (24% TRR) and roots (19% TRR) at harvest, 149 days after the second application.

Table 20 Composition of the residues in/on the leaves of glufosinate-tolerant sugar beets following one foliar spray treatment of [^{14}C] glufosinate ammonium (Allan 1996 A58109)

Composition of glufosinate residues	3 hours	8 days	15 days
	(%TRR)		
Rinse (water)	40.5	19.3	14.2
glufosinate (D:L glufosinate ratio)	40.5 (0.94)	18.8 (1.0)	13.8 (0.97)
Extracted (after rinsing)	57.8	74.5	80.0
glufosinate (D:L glufosinate ratio)	45.1 (1.2)	35.6 (5.2)	29.3 (7.6)
NAG	9.0	38.9	48.6
Unextracted	1.8	6.2	5.7
Mass balance (sum of glufosinate, NAG and unextracted)	96.4	97.8	96.6

Table 21 Composition of the residues in/on the leaves and beets of glufosinate-tolerant sugar beets following two foliar spray treatments of [^{14}C] glufosinate ammonium (Allan 1996 A58109)

	0 days	21 days	146 days
Leaves TRR (mg eq/kg)	20.08	12.26	2.05
	(%TRR)		
Rinse (water)	59.5	13.7	3.0
glufosinate	54.9	13.7	2.3
MPP	–	–	0.3
NAG	–	–	0.2
Extracted (after rinsing)	40.5	86.3	94.5
glufosinate	25.2	28.1	24.0
MPP	0.4	1.1	2.7
NAG	13.4	55.2	66.9
Unextracted	1.0	1.3	2.5
Beets TRR (mg eq/kg)	2.01	6.75	0.93
	(% of TRR)		
Extracted	97.4	96.4	96.3

	0 days	21 days	146 days
glufosinate	30.9	30.6	19.1
MPP	2.2	2.0	6.0
NAG	64.3	63.3	67.9
Unextracted	2.6	3.1	3.8

Müller *et al.* (2001) studied the metabolism of the glufosinate in heterotrophic cell suspension and callus cultures of tolerant ("*bar*"-gene) and conventional sugar beet using ^{14}C-labelled (racemic) glufosinate, L-glufosinate, and D-glufosinate, as well as the metabolites L-NAG and MPP. Cellular absorption was generally low, but depended noticeably on plant, substance and enantiomer. Portions of unextracted residues ranged from 0.1% to 1.2% of applied ^{14}C. Soluble metabolites resulting from glufosinate or L-glufosinate were between 0.0% and 26.7% of absorbed ^{14}C in cultures from the conventional crop and 28.2–59.9% in tolerant sugar beet. D-Glufosinate, MPP and L-NAG proved to be stable. The main metabolite of glufosinate in tolerant sugar beet cultures was NAG, besides traces of MPP and MPB. In conventional sugar beet cultures, glufosinate was transformed to a limited extent to MPP and trace amounts of MPB.

Glufosinate-tolerant corn/maize

Stumpf (1994 A53536) studied the metabolism of [3,4-^{14}C]-glufosinate ammonium in transgenic corn/maize, genetically modified by incorporation of the "*pat*" gene. Corn seeds were planted in an outdoor plot (sandy loam, OC: 0.7%, pH 6.0). The corn plants were sprayed with [^{14}C] glufosinate when plants were 40 cm high and 10 days later when 60 cm high at application rates equivalent to 0.53 kg ai/ha.

The majority of the ^{14}C residues (≥ 90% TRR) in forage (PHI 28 days), silage (PHI 55 days), fodder, husk and cobs (PHI 102 days) that were extracted with water with lower levels extracted from corn grain (77.6% TRR) (Table 22).

As with other tolerant crops, significant metabolism occurred within the first 5 days of application as evidenced by the majority of ^{14}C in aqueous extracts of whole plants attributed to NAG (43–49% TRR) and only 32% TRR to glufosinate (Table 23).

At harvest, glufosinate was a minor component of the ^{14}C residue in fodder, cobs, husks and grain (1.5–9.9% TRR) with NAG the major component in fodder (54% TRR) and MPP the major component in cobs (44% TRR), husks (41% TRR) and grain (33% TRR). MPA represented 2.9 to 12% TRR in the various commodities at harvest.

Table 22 Total radioactive residues (TRR) in glufosinate-tolerant corn following two foliar spray treatments of [^{14}C] glufosinate

Matrix	TRR	Extract		Unextracted
	(mg eq/kg)	Extraction with water (%TRR)	Acid/base/ enzymatic hydrolysis (%TRR)	(%TRR)
Whole plants at day zero	14.5–23	104.8–110.9	–	1.5–1.6
Whole plants at day 5	5.8–9.9	91.7–101.0	–	2.9–3.2
Forage, 28 days after 2nd treatment	2.64	96.0	–	3.4
Silage, 55 days after 2nd treatment	1.82	92.2	7.4 [a]	1.2
Mature plant material 102 days after the last treatment				
Fodder	2.01	89.8	6.4 [b]	2.9
Husks	0.872	91.1	–	8.0
Cob	0.251	97.9	–	11.1
Corn grain	0.130	76.7	13.8 [c]	6.1

[a] Hydrolysis of the extracted silage released 3.5% of TRR with 0.1 N NH_4OH and 3.9% with 0.1 N HCl

[b] Hydrolysis of the extracted fodder released 2.3% of TRR with 0.1 N NH_4OH and 4.1% with 0.1 N HCl

[c] Hydrolysis of the extracted grain released 2.8% of TRR with 0.1 N NH_4OH, 10.3% with 0.1 N HCl, 0.7% with alpha-amylase

Table 23 Metabolite profile of the radioactive residues in extracts of glufosinate-tolerant corn following two foliar spray treatments of [^{14}C] glufosinate ammonium

Matrix	glufosinate		NAG		MPP		MPA		unknowns	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Forage, 28 days	13.3	0353	52.1	1.384	12.0	0.319	4.6	0.122	11.0	0.293
Silage, 55 days	11.3	0.203	54.8	0.980	11.6	0.208	3.9	0.070	14.9 [a]	0.271
Mature plant material 102 days after the last treatment										
Fodder	9.9	0.200	54.4	1.102	10.9	0.220	2.9	0.058	14.2 [a]	0.287
Cobs	2.6	0.006	20.1	0.046	43.9	0.101	12.2	0.028	13.8	0.032
Husks	2.1	0.018	18.9	0.166	41.1	0.361	11.0	0.097	13.4	0.119
Grain [b]	1.5	0.002	9.1	0.012	32.7	0.043	4.4	0.006	42.5 [a]	0.035

[a] The unknowns were characterized by acid and basic hydrolysis of the plant matrix

[b] The residues in corn grain additionally contain 9.8% of TRR (0.013 mg eq/kg) of MPB.

Zumdick (1998a A67070,1998b A67122) conducted studies comparing the metabolism of [3,4-^{14}C]-glufosinate ammonium in two transgenic varieties of corn/maize tolerant of glufosinate. The genetically modified corn varieties were generated by incorporation of the "*pat*" gene (event T25, *cv.* KX6107 LL) and the "*bar*" gene (event CBH351). Corn was sown into plant pots (silt loam soil, OC: 1.18%, pH 7.3) and cultivated in an open vegetation hall with a glass roof. Three weeks after sowing, when the emerged plants had reached the 5–6 leaf growth stage and a height of 40–60cm, they were treated with ^{14}C-glufosinate. Prior to application, the bare soil between the plants was covered with a water-absorbing fleece to avoid direct soil contamination and absorption by the plants of the metabolites formed in soil. After application this fleece was removed. Application rates were 0.86 kg ai/ha for the "*pat*"-corn and 0.80 kg ai/ha for the "*bar*"-corn. The "*bar*"-maize variety (event: CBH351) being in an early breeding stage was characterized by a hemizygotus seed-lot and only results for tolerant plants are reported here.

More than 97% of the TRR in the above ground (aerial) plants was extracted with solvent. NAG was the main ^{14}C residue component in both varieties of the genetically modified maize plants at 14 and 28 days after application amounting to 47–62% of TRR (Table 24). Glufosinate was the other major component and was present at 26–45% of TRR. Minor metabolites i.e. MPP, MPB and MHB did not exceed 5% of TRR.

Table 24 Comparison of the ^{14}C residues in the aerial parts of glufosinate-tolerant maize containing the "*pat*" or the "*bar*" gene following foliar application of ^{14}C-glufosinate ammonium (Zumdick 1998ab)

	0 days		14 days		28 days	
	pat	bar	pat	bar	pat	bar
TRR (mg eq/kg)	42.45	57.30	7.59	8.38	2.48	1.05
Rinse (surface ^{14}C) (%TRR)	57.4	72.0	–	–	–	–
Extract (absorbed ^{14}C) (%TRR)	42.5	28.0	98.6	98.4	98.07	97.0
glufosinate			45.27	40.59	27.92	25.64
MPB			1.42	0.22	2.73	1.36
MPP + MHB			2.14	2.33	4.30	4.13
NAG			47.53	54.05	59.78	62.40
MPA			0.78	0.92	1.49	1.45
Identified			97.14	98.11	96.21	94.99

Only the results for those "*bar*"-plants that were tolerant are reported

Everman *et al.* (2009a) studied the metabolism of [^{14}C] glufosinate in tolerant corn (*cv.* Pioneer 34A55 LL). Corn plants were grown in pots and treated at the 4-leaf growth stage with a commercial formulation of glufosinate that did not include radio-labelled material. Immediately after spraying, [^{14}C] glufosinate in an aqueous solution was spotted onto the first fully expanded leaf. Absorption of [^{14}C] glufosinate was low reaching 16% of the applied ^{14}C by 24 hours after application. Translocation of absorbed ^{14}C was observed as evidenced by ^{14}C accumulation above the

treated leaf and in the roots. Accumulation of ^{14}C-radioactivity above the treated leaf ranged from 25 to 41% of the absorbed ^{14}C and from 22 to 27% in the roots at 24 to 72 hours after treatment. Metabolism was relatively rapid with 83% of ^{14}C present as glufosinate at one hour after application declining to 52% by 72 hours after application. Metabolite 1, not identified in the study but assumed here to mostly be NAG based on chromatographic properties, increased from 0% of TRR to 42% by 72 hours after application.

A study by Ruhland *et al*. (2002) compared the metabolism of [^{14}C] glufosinate as well as L- and D-glufosinate in cell cultures of conventional and tolerant rape and corn. Transformation of glufosinate in both conventional and tolerant rape cells remained at a low rate of about 3–10% in contrast to corn cells, where 20% was transformed in conventional and 43% in tolerant cells after 14 days of incubation, the rest remaining as unchanged glufosinate. In cells from conventional rape and corn the main metabolite was PPO (7.3% TRR rape, 16.4% TRR corn) together with low amounts of MPP, MHB, MPB and MPA. In cells from tolerant crops NAG was also formed (3.2% TRR in rape, 16.1% TRR in corn at 14 days). L-glufosinate was transformed into the same metabolites as the glufosinate racemate while D-glufosinate was not metabolized in cell cultures obtained from conventional or tolerant rape and corn.

Ruhland *et al.* (2004) investigated the metabolism of glufosinate in tolerant rape and maize plants treated separately with L-glufosinate, D-glufosinate or the racemic mixture and grown under outdoor conditions. Plants were treated at the 4–6 leaf stage with ^{14}C-labelled test substance applied to all the leaves of the plants at a rate equivalent to 0.075–0.65 kg ai/ha. Results for maize are reported here with those for oilseed rape reported later. The study was conducted over a period of three years with seeds sown in pots. Glufosinate was studied in the first, L-glufosinate in the second and D-glufosinate in the third year. Most of the applied radioactivity was lost from the plants in the period between application and harvest (84–90%), mainly due to rain washing off surface residues. ^{14}C levels were found to increase in the soil. In maize, the majority of ^{14}C was extracted with water (> 95% TRR) with less than 4% TRR remaining unextracted by water and organic solvent. The metabolites identified in glufosinate, L-glufosinate and D-glufosinate treated plants were the same and consisted mostly of NAG and the methylphosphinyl fatty acid metabolites MPP, MPA, MPB and MHB (Table 25). In contrast to sterile plant cell cultures, D-glufosinate is metabolised to some extent in transgenic plants grown under outdoor conditions. However, as D-glufosinate is not a substrate for PAT, it was suggested the formation of NAG may have been due to conversion of D-glufosinate to the L-enantiomer by micro-organisms, which then provides a substrate for further metabolism by micro-organisms and plants. The low proportion of applied ^{14}C recovered at harvest of D-glufosinate treated maize plants could indicate lower absorption of the D-enantiomer compared to the L-enantiomer.

Table 25 Nature of ^{14}C in tolerant maize plants after application of glufosinate (racemate), L-glufosinate or D-glufosinate (%TRR)

	glufosinate [a]			L-glufosinate [b]			D-glufosinate [c]		
Fraction	glufosinate	NAG	ΣMPFs	L-glufosinate	NAG	ΣMPFs	D-glufosinate	NAG	ΣMPFs
Leaves treated	11.5	68.1	18.3	3.8	78.0	22.3	39.2	36.0	32.1
Leaves untreated	9.1	41.1	27.4	2.2	29.7	29.7	16.1	57.5	17.8
Stems	6.7	66.7	18.7	9.6	30.0	50.4	12.3	41.9	40.9
Ears	3.7	80.0	10.7	13.2	19.8	26.4	4.6	36.8	50.6
Spathaceous bracts	9.6	73.6	9.6	13.2	2.0	39.6	5.5	36.8	36.8
Grain	4.6	22.9	68.6	8.8	8.8	70.4	2.8	46.0	36.8
Whole plant	10.8	66.9	18.5	4.4	61.6	27.4	24.0	47.0	26.6

ΣMPFs = sum of methylphosphinyl fatty acid metabolites MPP, MPA, MPB and MHB

[a] At harvest 16% of the applied radioactivity was located in the above ground plant with 96% identified

[b] At harvest 13.2% of the applied radioactivity was located in the above ground plant with 93% identified

[c] At harvest 9.2% of the applied radioactivity was located in the above ground plant with 98% identified

The main transformation pathway of glufosinate is the inactivation of L-glufosinate by acetylation to form NAG accounting for 62% of the L-glufosinate taken up by the plants. Glufosinate

(D- and L-glufosinate) may also undergo deamination and subsequent decarboxylation to form MPP and other methylphosphinyl fatty acids.

Glufosinate-tolerant rice

Rupprecht (2000 B002617) investigated the metabolism of [3,4-^{14}C]-glufosinate ammonium rice plants (*cv.* Taipei) genetically modified to be tolerant to glufosinate. Seeds were sown into two plant tanks filled with loamy sand soil. The plants were cultivated in a greenhouse. Plants were treated twice at 0.5 kg ai/ha; the first application was at the 2–4 leaf stage, the second at the 2-4 tiller stage with a 19-day spray interval. Two different flooding regimes were used to allow for differing agricultural water management practices. One tank was flooded 2 days prior to the first treatment (Tank A). The second tank was flooded approximately one day after the second treatment (Tank B). Watering of the tanks was stopped 183 days after the first treatment (20 days before the harvest of mature plants) to allow drying out of the paddy soil.

Glufosinate was rapidly absorbed by rice plants as only 26–28% of the TRR was recovered in forage surface rinses (water) on the day of treatment decreasing to 2–6% of TRR in forage by 18 days after a single treatment (Table 26). At harvest, NAG was the major component of the ^{14}C residue in straw and stubble (50–60% TRR) with glufosinate (10–18% TRR) and MPP (10–25% TRR) being the other significant components (Table 27). The major component in grain was MPP (68–72% TRR) with smaller amounts of NAG (11% TRR) and glufosinate (5–6% TRR).

There were no significant quantitative or qualitative differences in the residues or the metabolite profiles of the rice samples grown and subjected to different water management practices (Tanks A and B).

Residue levels in the paddy water of Tank A (flooded before the first treatment) decreased rapidly from 0.63 mg eq/kg at day 7 to 0.03 mg eq/kg by day 18 and further to 0.01 mg eq/kg at day 102 (the final sampling point at which water was still present). Little residue was associated with the soil at the earlier time points (0.07–0.08 mg eq/kg), although there was a slight increase to 0.16 mg eq/kg by day 202, when the soil had dried out.

Significant amounts of the parent glufosinate remained in Tank A water and soil at day 7 (36.8 and 27.8% of TRR, respectively), but the major residue component was MPP (47.6 and 39.1% of TRR, respectively). NAG (4.3–7.1% of TRR), MP-acrylic acid (5.3–7.1% of TRR), MPF (2.2–2.4% of TRR) and MPA (1.0% of TRR or less) were minor components of the residue.

MPP was also the major metabolite (17.8–57.5% of TRR) in all water and sediment samples analysed at intermediate time points (days 7–102) and in these samples little parent glufosinate remained (3.2–8.2% of TRR). At one day prior to final harvest (day 202), parent glufosinate was the major residue in the soil of both Tanks A and B (25.2–34.5% of TRR), the majority of which was recovered from soil hydrolysates. The remainder comprised MPP (16.2–19.7% of TRR) and traces (2.9% of TRR or less) of other metabolites.

Table 26 Total radioactive residues (TRR) and extractability in glufosinate-tolerant rice plants following one and two foliar spray treatments of [^{14}C] glufosinate ammonium

			TRR		%TRR	
Time after spray	Sample	Tank [a]	(mg eq/kg)	Surface Rinse	Extracted [b]	Unextracted [b]
1st spray, 0 days	Forage	A	93.68	28.2	69.2	2.6
		B	80.12	26.0	73.0	1.0
1st spray 18 days	Forage	A	2.05	5.8	76.1	18.0
		B	2.64	1.8	86.1	12.0
2nd spray, 184 days	Grain	A	1.36	–	93.0	6.9
(final harvest)		B	1.12	–	86.6	13.5
	Straw	A	9.50	–	94.2	5.8
		B	13.08	–	94.6	5.4
	Stubble	A	9.59	–	92.4	7.6
		B	8.85	–	91.4	8.6

[a] Different water regimes in Tank A and B: Tank A was flooded 2 days prior to the first application. Tank B was flooded one day after the second application. The paddy soil of both tanks was allowed to dry 20 days before the final harvest of rice.

[b] The extracted and unextracted amount is from samples after a surface rinse (if conducted)

Table 27 Nature of glufosinate residues in glufosinate-tolerant rice plants following one and two foliar spray treatments of [^{14}C] glufosinate ammonium

					%TRR			
Time after spray	Sample	Tank [a]	glufosinate	MPP	NAG	MPA	MPF	% identified
1st spray, 0 days	Forage	A	65.4	1.0	25.6	ND	0.8	92.8
		B	59.2	0.8	34.0	ND	ND	94.0
1st spray 18 days	Forage	A	8.1	7.8	54.2	0.2	0.3	71.3
		B	8.1	9.0	63.6	ND	0.8	82.6
2nd spray, 184 days	Grain	A	5.0	72.3	11.2	0.6	ND	89.0
(final harvest)		B	6.0	68.5	11.4	0.8	ND	86.6
	Straw	A	17.7	10.0	59.6	2.6	ND	89.8
		B	17.5	13.4	59.7	2.4	ND	92.8
	Stubble	A	10.4	16.6	60.1	1.6	ND	88.6
		B	9.8	25.4	50.0	1.6	ND	86.7

[a] Different water regimes in Tank A and B: Tank A was flooded 2 days prior to the first application. Tank B was flooded one day after the second application. The paddy soil of both tanks was allowed to dry 20 days before the final harvest of rice.

ND = not detected

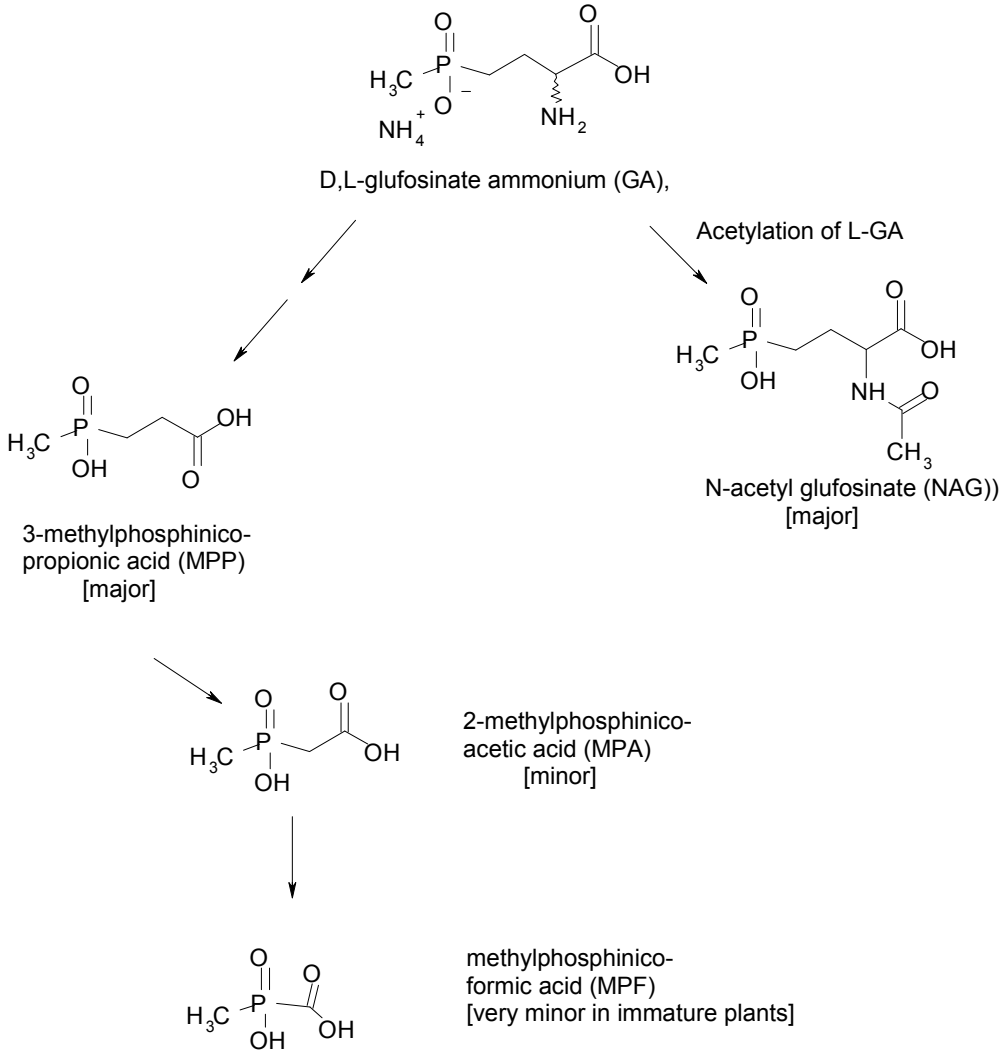


Figure 5 Metabolic pathway of glufosinate in glufosinate-tolerant rice

Glufosinate tolerant cotton

Everman *et al.* (2009b) studied the metabolism of [^{14}C] glufosinate in conventional (*cv.* Fibermax 958) and tolerant cotton (*cv.* Fibermax 958 LL). Cotton plants were grown in pots and treated at the 10 cm (4-leaf) growth stage with a commercial formulation of glufosinate at the equivalent of 0.47 kg ai/ha. Immediately after spraying, ^{14}C-glufosinate in an aqueous solution was spotted onto the adaxial surface of the first fully expanded leaf. Absorption of [^{14}C] glufosinate was low for conventional and tolerant cotton reaching 11–13% of the applied ^{14}C by 48 hours after application. Translocation in tolerant and conventional cotton reached 10 and 15%, respectively, at 72 hours after treatment and is lower than observed for tolerant corn (Everman *et al.* 2009a). Tolerant cotton showed high levels of [^{14}C] glufosinate metabolism with metabolite 1 (not identified in the study, assumed here to be mostly NAG) accounting for 72% of TRR at 72 hours after treatment whereas in conventional cotton glufosinate accounted for 73% of TRR and Metabolite 1 only 16% of TRR at 72 hours after application. No information was available on residues in cotton seed.

Glufosinate-tolerant oil seed rape

The metabolism of [3,4-^{14}C]-glufosinate ammonium was investigated in canola (*cv.* 19-2XACS-N3, genetically modified for glufosinate tolerance) (Tshabalala 1993 A51529, Thalacker 1994 A53141). The canola seeds were cultivated in plant pots (sandy loam soil; OM: 1.4%, pH 7.6). The canola plants were sprayed at the 3–5 leaf stage at an application rate equivalent to 0.75 kg ai/ha. Monitoring

indicated some phyto-toxicity at one day after application however most of the plants overcame these symptoms by three days post-application. The treated plants were cultivated in a greenhouse (Porterville, California, USA).

Levels of ^{14}C directly after the treatment were up to 145 mg eq/kg and declined to be approximately 4.3–4.5 mg eq/kg in foliage and roots at 21 days after treatment (Table 28). At full maturity 120 days after treatment, ^{14}C levels were generally lower than 0.3 mg eq/kg in foliage, roots, hulls and seeds. TRR in seeds were 0.045–0.109 mg eq/kg. Most of the ^{14}C in seeds was extracted with the solvents used, with an overall extraction efficiency of approximately 90% (4–7% TRR extracted with hexane, 32–52% TRR extracted with water/methanol). Sequential treatment of the extracted seeds with phosphate buffer, methanol/chloroform and acid and base hydrolysis released a further 4%TRR with about 6% of the ^{14}C residues remaining unextracted.

Glufosinate was only a minor component of the ^{14}C residue in seeds. The major component was MPP with small amounts of NAG and two minor unknown metabolites. A large amount of the ^{14}C residue (35–50% of TRR) was incorporated into the components of the plant cell wall and was released as a water-soluble polysaccharide/protein fraction, a lipid fraction, and a conjugated fraction.

Residues in hulls comprised parent glufosinate, small amounts of NAG and the major metabolite, MPP.

For foliage, approximately 99% of ^{14}C of the one-hour and the 21-day samples could be extracted with water. One hour after spray treatment, already 18% of TRR was present as NAG while the remaining approximately 73% of TRR was present as unchanged glufosinate. MPP was formed as a minor metabolite amounting to approximately 7% of TRR.

Table 28 Radioactive residues in glufosinate-tolerant canola following foliar spraying of [^{14}C] glufosinate at a use rate of 0.75 kg ai/ha

Days after application	Matrix	TRR (mg eq/kg)	Glufosinate (%TRR)	MPP (%TRR)	NAG (%TRR)	Unknowns (%TRR)
0 (1 h)	Whole plant	145	72.9	–	18.2	
21	Foliage	4.28	20.7	6.7	60.2	
	Roots	4.5				
120	Foliage	0.076–0.263				
	Roots	0.124–0.220				
	Hulls	0.076–0.263				
	Seeds	0.045–0.109	0–14	3–45	≤ 2	2–23

Buerkle (2001 C009418) studied the metabolism of [3,4-^{14}C]-glufosinate ammonium in genetically modified glufosinate-tolerant winter rape (*cv.* Liberty Link Falcon 6Ac). The rape seeds were sown and cultivated in plant containers (sandy loam soil; OC: 0.97%, pH 5.9) and maintained most of the time in an open vegetation hall with a glass roof. Plants were moved to the indoors for short periods to avoid frost damage. The plants received two spray applications. The first application was when the plants were at the 5–6-leaf growth stage (BBCH 15-16) and approximately 40–50 cm high. The second application was at the forage growth stage (BBCH 49-51). The application rates were equivalent to 0.76 and 0.77 kg ai/ha.

Highest ^{14}C levels were detected in leaves directly after the treatments (43–72 mg eq/kg) (Table 29). Approximately 30–40% of ^{14}C was washed off leaves with water with most of the remaining ^{14}C extracted with water/methanol (55–60% TRR). The TRR levels decreased as the plants grew and matured. At harvest 102 days after the second application, ^{14}C residues were 12.7 mg eq/kg in straw, 7.1 mg eq/kg in the hulls, 5.7 mg eq/kg in the roots and to 0.53 mg eq/kg in the seeds.

Extraction efficiencies (sum of surface rinses + water/methanol extracts) for leaves, forage, straw, hulls and roots were high with > 95% of TRR recovered and a little lower for seeds at 75% TRR. Most of the ^{14}C remaining after initial extraction was released by enzymatic digestion and chemical hydrolysis. The released ^{14}C residues were very polar (water-soluble) in nature.

NAG comprised 16–34% of TRR in leaves at the day of the first and the second application. The proportion of NAG increased to 71% of TRR in forage by day 154 after the first application. At harvest NAG was the major ^{14}C residue component representing 57% of TRR in straw, 77% in hulls, 65% in roots and 32% in seeds. Other metabolites were formed in minor proportions: MPP at 4–10% of TRR in mature rape plants and MPB and MPA at 1–2% of TRR each. Glufosinate was present at maturity at 31% of TRR in straw, 21% in roots, 14% in hulls, and 6% of TRR in seeds.

Table 29 Radioactive residues in glufosinate-tolerant winter rape following one or two foliar sprays of [^{14}C] glufosinate at a use rate of 0.8 kg ai/ha

Days after application [a]	Matrix	TRR (mg eq/kg)	Extracted (%TRR) Total	Glufosinate	NAG	MPP	MPB	MPA
0/1st	Leaves	54–72	[b]	36–44	16–36	–	–	–
0/2nd	Leaves	43	[c]	39.5	18.9	–	–	–
21/2nd	Leaves	40	[d]	26.2	37.3	1.4	2.0	
154/1st	Forage	0.82	95.3	7.9	70.8	4.3	1.6	–
102/2nd	Straw	12.7	96.7	30.7	56.9	4.3	0.8	–
	Hulls	7.09	98.0	14.1	77.4	5.0	–	–
	Roots	5.66	97.3	20.9	65.3	6.1	2.0	1.1
	Seeds	0.53	75.2	6.2	32.2	9.7	1.6	1.9

[a] *x/y* means *x* days after the *y*th treatment

[b] 0/1: 31–36% of TRR washed off with water, 55–61% of TRR extracted after washing

[c] 0/2: 40% of TRR washed off with water, 60% of TRR extracted after washing

[c] 21/2: 31.5% of TRR washed off with water, 67.5% of TRR extracted after washing

Following application of glufosinate to glufosinate-tolerant rape the major residue components consisted of the parent glufosinate and the metabolites NAG and MPP.

Becker (2000 A67440) compared the metabolism of [3,4-^{14}C]-glufosinate ammonium in two genetically modified varieties of rape; variety 1: Liberty Link; Falcon 6Ac with the "*pat*" gene and variety 2: MS1/MF1; PGSW1 with the "*bar*" gene. Both varieties were (at least partially) tolerant of glufosinate ammonium. The rape seeds of the two varieties were sown separately into plant pots (sandy loam soil; OC: 1.18%, pH 7.3). The plants were cultivated under outdoor conditions but protected from rain. Three weeks after sowing, when the plants had reached the 4–5 leaf stage, they were treated with [^{14}C] glufosinate ammonium by foliar spraying at 0.77 kg ai/ha for the *bar* variety and 0.80 kg ai/ha for the *pat* variety.

Approximately one week after treatment the majority of the *pat*-rape plants showed chlorosis with partial necrosis and brown coloured spots on most of the treated plant leaves. Leaves formed after the herbicide application did not show any phytotoxicity symptoms. The *bar*-rape also showed sporadically brown coloured spots. The expression of the "*pat*" and "*bar*" genes was insufficient to confer adequate tolerance.

Directly after spraying in the 4–5 leaf stage, TRR was 45 mg eq/kg in *pat* rape and to 60 mg eq/kg in *bar* rape declining to 3.5 and 2.3 mg eq/kg respectively by 28 days after application. The extraction efficiency for all samples using water:methanol (9:1) was greater than 93% (Table 30).

NAG was the main residue component for both "*pat*" and "*bar*" crops at 60–69% of TRR. Unchanged glufosinate was also a significant component of the ^{14}C residue and was present 24–34% of TRR at 14 and 28 days after application. MPP, MPB and MHB did not exceed 3% of TRR.

Table 30 Comparison of the radioactive residues in glufosinate-tolerant rape shoots containing the "*pat*" and the "*bar*" gene following foliar application of [^{14}C] glufosinate ammonium

Trait	pat	bar	pat	bar
Days after application	14	14	28	28
TRR (mg eq/kg)	13.7	7.66	3.49	2.31
Extracted (%TRR)	93.9	95.9	97.7	97.4
Glufosinate	34.56	32.64	30.53	23.87

Trait	pat	bar	pat	bar
MPB	1.25	1.00	1.24	1.00
MPP and MHB	2.68	2.09	2.32	2.39
NAG	54.72	59.61	63.20	69.37
MPA	ND	ND	ND	ND

Even though the expression of *pat* was not sufficient to confer adequate glufosinate tolerance the experiment demonstrates that the metabolites formed in oilseed rape containing the *pat* and *bar* genes are the same.

Beriault *et al.* (1999) studied the phloem transport of glufosinate and L-NAG in both conventional (*cv.* Excel) and tolerant rape (HCN27). Based on its physicochemical properties (pKa < 2, 2.9 and .9.8; log K_{ow} –3.9) it would be expected that glufosinate would translocated in plants. However, localised phyto-toxicity caused by the active L-isomer of glufosinate leads to little translocation being observed in conventional plants. When [^{14}C] glufosinate (racemate) was applied to an expanded leaf, 25% of the ^{14}C was translocated in tolerant rape able to convert L-glufosinate to the herbicidally inactive NAG compared to only 6.3% in conventional rape. Studies with [^{14}C] glufosinate (racemate) and [^{14}C-D] glufosinate showed that when absorbed, the inactive D-isomer is much more mobile than the racemic mixture. [^{14}C] NAG was extensively translocated in both conventional and tolerant plants.

As reported earlier, Ruhland *et al.* (2004) also investigated the metabolism of glufosinate in tolerant rape plants treated separately with glufosinate or L-glufosinate and grown under outdoor conditions. Plants were treated with at the 4–6 leaf stage with ^{14}C-labelled test substance applied to all the leaves of the plants at a rate equivalent to 0.075–0.65 kg ai/ha. The study was conducted over a period of two years with seeds sown in pots. For oilseed rape, glufosinate (racemate) was studied in the first and L-glufosinate in the second year.

Most of the applied radioactivity was lost from the plants in the period between application and harvest (53–65%), mainly due to rain washing off surface residues as ^{14}C levels were found to increase in the soil but potentially also through microbial degradation at the plant surface. The majority of ^{14}C was extracted with water (> 95% TRR) with less than 4% TRR remaining unextracted. The metabolites identified in glufosinate and L-glufosinate treated plants were the same and consisted mostly of NAG and smaller amounts of the methylphosphinyl fatty acid metabolites MPP, MPA, MPB and MHB (Table 31).

Table 31 Nature of ^{14}C in tolerant oilseed rape plants after application of glufosinate (racemate) or L-glufosinate (%TRR)

	glufosinate [a]			L-glufosinate [b]		
Fraction	glufosinate	NAG	ΣMPFs	L-glufosinate	NAG	ΣMPFs
Leaves treated	20.7	74.2	3.8	4.5	86.8	6.4
Leaves untreated	19.3	74.4	4.5	3.7	90.7	6.6
Stems	15.6	68.1	10.2	1.2	93.0	4.4
Pods	29.8	27.5	44.2	0.7	95.3	5.6
Grain	7.9	31.5	15.8	0.9	34.6	8.6
Whole plant	19.9	65.8	11.3	2.4	90.5	5.5

ΣMPFs = sum of methylphosphinyl fatty acid metabolites MPP, MPA, MPB and MHB

[a] At harvest 47.3% of the radioactivity applied was located in the above ground plant parts with 97% identified

[b] At harvest 34.7% of the radioactivity applied was located in the above ground plant parts with 98% identified

Plant growth stage did not make a significant difference. Uptake of L-glufosinate when applied to oilseed rape plants at the 16 leaf growth stage was also fast and nearly complete after 24 hours. The acetylation of L-glufosinate was fast with 60% of the residues extracted from rinsed leaves attributed to NAG by one hour after application.

Confined rotational crops

Studies with confined rotational crops have been included in the glufosinate plant metabolism section as they are relevant for understanding uptake from soil for this herbicide which uses include bare soil treatments as well as pre-sowing, pre-planting and pre-emergence applications. Residues at harvest from uptake of ^{14}C from soil applied glufosinate were generally low and mostly comprised of MPP and natural products, the exception being foliage (wheat forage and radish tops) in which glufosinate was a significant part of the ^{14}C residues at the shortest plant-back interval (28 days).

In a confined crop rotation study, the metabolism of [3,4-^{14}C]-glufosinate ammonium was investigated in rotational crops that were sown into pre-treated soil (sandy loam soil, OM: 1.81%) (Meyer 1995 A54272, Meyer 2000 A55794). The application rate to soil was 1.0 kg ai/ha. Treated soil was aged for plant back intervals (PBIs) of 28 days, 119 and 300 days to simulate crop failure re-planting, immediate re-cropping and re-cropping in the following year. The soil in the plots was cultivated prior to planting to provide a good seed bed. Three major crop groups, leafy vegetables, root crops and small grain cereals represented by lettuce (*cv.* Black-Seeded Simpson), radish (*cv.* Cherry Belle) and spring wheat (*cv.* Butte 86) were sown into the treated soil after the three plant-back intervals (PBIs). The crops were cultivated in a greenhouse and irrigated manually on an "as needed" basis. Lettuce and radish were also grown in separate untreated pots in close proximity to the treated plots to monitor for foliar uptake of $^{14}CO_2$ or other volatiles. In addition, wheat was also sown onto the soil of the first container 364 days after soil treatment, following harvest of the crops of the first rotation.

TRRs ranged between approximately 0.1 (radish root) and 1.0 (lettuce) mg eq/kg in the crops of the first rotation at harvest (PBI 28 days). TRR decreased to 0.01 (radish root)–0.15 (wheat straw) mg eq/kg in the second rotation (PBI 119 days) and further to 0.005 (radish root and top)–0.064 (wheat straw) mg eq/kg in the third rotation (PBI 300 days) (Table 32). Wheat sown 364 days after soil treatment showed TRR levels of 0.02 (forage), 0.03 (grain) and 0.069 (straw) mg eq/kg.

The extractability (acetonitrile/water 1/1) of ^{14}C in crops was moderate in the first rotation, 43–68% of TRR except radish roots for which 82% was extracted. Alkaline and acid hydrolysis of the PES released a further 20–48% of TRR. The amount of ^{14}C recovered in the solvent extraction decreased with rotation while the amount released on hydrolysis of PES increased.

MPP was the main ^{14}C residue component in all succeeding crops of the first rotation (25% TRR for lettuce–46% TRR for wheat forage) (Table 33). Hydrolysis of post-extraction solids released additional MPP. MPA was a minor component in all succeeding crops (3.5% TRR in wheat grain – 8% TRR in wheat straw). An unknown polar component ("Metabolite A") was detected in radish root (20% TRR), lettuce (15.5% TRR), wheat straw (25% TRR), wheat forage (9.2% TRR) and wheat grain (10.6% TRR).

In wheat straw and grain of the second rotation, MPP and "Metabolite A" were the main residue components (MPP: 21.5% TRR in straw; 12% TRR in grain; "Metabolite A": 26% TRR in wheat straw, 14% TRR in grain).

Low levels of radioactivity were detected in plants used to monitor uptake of $^{14}CO_2$ and volatiles that may have been released by soil degradation of [^{14}C] glufosinate. The highest ^{14}C levels for monitoring plants were detected in wheat straw (0.008 mg eq/kg in the first rotation and 0.025 mg eq/kg in the second rotation) and in wheat grain (0.017 mg eq/kg in the first and 0.029 mg eq/kg in the second rotation).

Soil ^{14}C residues declined with time from 0.36 mg eq/kg at day zero to 0.05–0.06 mg eq/kg 364–447 days after soil treatment. The major metabolite 119 days after treatment was MPP (30% TRR), with lesser amounts of MPA (15% TRR) and "Metabolite A" (8% TRR). The occurrence of metabolites in rotational crops could be due in part to uptake from the soil.

Table 32 Total radioactive residues (TRR) and the extractability of residues in crops rotated 29, 119 and 300 days after application of [^{14}C] glufosinate to bare soil

Crop	Matrix	TRR	Extracted	Hydrolysed [a]	Unextracted
		(mg eq/kg)	(%TRR)	(%TRR)	(%TRR)
Plant back interval: 28 days (first rotation)					
Radish	Top	0.119	57.4	28.8	14.2
	Root	0.099	82.3	–	17.7
Lettuce	Whole plant	0.986	58.3	–	41.7
Wheat	Forage	0.327	68.5	19.7	11.8
	Straw	0.851	57.0	33.4	9.6
	Grain	0.360	43.1	48.5	8.4
Plant back interval: 119 days (second rotation)					
Radish	Top	0.014	33.1	–	66.9
	Root	0.010	66.8	–	33.2
Lettuce	Whole plant	0.014	38.8	–	61.2
Wheat	Forage	0.051	28.0	–	72.0
	Straw	0.148	28.1	43.5	28.4
	Grain	0.129	10.1	40.6	49.3
Plant back interval: 300 days (third rotation)					
Radish	Top	0.005	–	–	–
	Root	0.005	–	–	–
Lettuce	Whole plant	0.004	–	–	–
Wheat	Forage	0.031	–	–	–
	Straw	0.064	50.9	–	49.1
	Grain	0.043	–	–	–
Plant back interval : 364 days; wheat sown into the soil of the 28–day tank following harvest of the crops of the first rotation					
Wheat	Forage	0.020	–	–	–
	Straw	0.069	14.5	–	85.6
	Grain	0.030	–	–	–

[a] hydrolysis of PES by 1M ethanolic KOH at 50 °C for 16–24 hours followed by 10% HCl/dioxane at 50 °C.

Table 33 Nature of the residues in crops rotated in soil that has been treated with [^{14}C] glufosinate at a rate of 1 kg ai/ha

Crop Matrix	Released by	MPP		MPA		"Metabolite A" [c]		Total [a]
		% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 28 days (first rotation)								
Radish Tops	CH_3CN/H_2O	29.5	0.039	5.5	0.007	8.6	0.012	65.8
	Hydrolysis [b]	14.1	0.019	–	–	8.1	0.011	
Radish Roots	CH_3CN/H_2O	39.2	0.051	7.2	0.009	20.0	0.026	66.4
Lettuce	CH_3CN/H_2O	24.8	0.022	7.4	0.007	15.5	0.014	47.7
Wheat Forage	CH_3CN/H_2O	46.5	0.152	5.6	0.018	5.6	0.018	73.0
	hydrolysis [b]	10.7	0.035	1.0	0.003	3.6	0.012	
Wheat Straw	CH_3CN/H_2O	39.0	0.332	7.8	0.066	6.7	0.057	79.5
	hydrolysis [b]	6.3	0.053	1.7	0.014	18.0	0.152	
Wheat Grain	CH_3CN/H_2O	33.3	0.120	3.5	0.013	3.9	0.014	53.0
	hydrolysis [b]	–	–	6.8	0.025	6.7	0.024	
Plant back interval: 119 days (second rotation)								
Wheat Straw	CH_3CN/H_2O	8.5	0.013	2.4	0.004	13.0	0.019	53.0
	hydrolysis [b]	13.0	0.019	3.2	0.005	12.9	0.019	
Wheat Grain	CH_3CN/H_2O	2.0	0.003	0.3	< 0.001	6.1	0.008	26.0
	hydrolysis [b]	9.9	0.013	–	–	7.7	0.010	

[a] No other single unidentified component exceeded 0.018 mg eq/kg (2.5% of TRR in 28 day wheat straw) in any extract of fraction

[b] hydrolysis of PES by 1M ethanolic KOH at 50 °C for 16–24 hours followed by 10% HCl/dioxane at 50 °C.

[c] The ^{14}C assigned to metabolite A (single chromatographic peak) was subsequently resolved and identified as natural products such as sugars together with additional polar species that are also likely natural products

An additional confined crop rotation study was conducted to characterize and identify the radioactivity previously assigned to "Metabolite A" (Sur 2007 MEF 05/508). The metabolism of [3,4-^{14}C]-glufosinate ammonium, was investigated in rotational crops that were sown to two soils (sandy loam soil "Langenfeld", OM 1.59%, pH 5.4 or loamy sand soil "Pikeville", OM 2.03% pH 5.3) pre-treated at 1.0–1.1 kg ai/ha after a plant back interval of 28 days after application. Three rotational crops were sown/ planted after the aging period into the "Langenfeld" soil (lettuce seedlings *cv.* Fulmaria, radish *cv.* Cyros and spring wheat *cv.* Thasos) while only wheat was sown into the "Pikeville" soil.

Radish was harvested 61 days after soil application or 33 days after sowing (BBCH 47-48). Lettuce (BBCH 46-47) and wheat forage (BBCH 28-29) were harvested 74 days after soil application or 46 days after planting/sowing. Wheat hay was sampled 116 days after soil application or 88 days after sowing (BBCH 73-75). Wheat straw and grain were harvested 154 days after soil application or 126 days after sowing (BBCH 93).

The ^{14}C residues in the rotational crops ranged between approximately 0.05 (wheat forage) and 0.8 (wheat straw) mg eq/kg for the sandy loam ("Langenfeld") and were 0.9 and 0.2 mg eq/kg in wheat straw and grain respectively for the loamy sand ("Pikeville") (Table 34).

The shortest interval between soil application and harvest was for radish (61 days after soil application and 33 days after sowing). The main component of the ^{14}C residues in radish was due to glufosinate with significant amounts of MPP and NAG also detected (Table 35). For crops harvested at later times (lettuce and wheat forage at 74 days after soil application) and wheat straw and grain at 116 days after soil application) MPP was the major component of the ^{14}C residue (Table 36). A chromatographic peak ("A05") due to very polar material reached 11% of TRR in wheat forage, 3% in wheat grain and up to 26% of TRR (0.024 mg eq/kg) in lettuce. Based on chromatographic properties, "A05" is assumed to be the same as "Metabolite A" observed in the previous rotational crop study.

The polar A05 fraction extracted from lettuce was able to be separated into sub-fractions A05a and A05b with fraction A05a further resolved into [^{14}C] glucose and [^{14}C] fructose fractions. Fraction A05b was separated into at least six smaller components. The identification of glucose and fructose was performed by TLC co-chromatography of A05a with radiolabelled reference sugars and confirmed after acetylation of A05a and TLC co-chromatography with acetylated reference items of glucose and fructose.

In contrast, the polar fraction A05 isolated from wheat grain and straw of crops rotated in loamy sand "Pikeville" was resolved into many components by radio-TLC on amino-coated Silicagel plates (Table 37). These components showed a similar chromatographic profile as an extract of the Pikeville soil. In contrast to lettuce, the polar fraction A05 in wheat mostly comprised metabolites that have been formed in soil and absorbed by the roots of wheat.

Degradation products of [^{14}C] glufosinate in soil were mainly MPP, NAG and MPA. The degradation was more rapid in the loamy sand "Pikeville" than the sandy loam "Langenfeld".

The common metabolic steps consist of an oxidative deamination of glufosinate followed by a rapid decarboxylation to form MPP, a stepwise oxidative decarboxylation of MPP to form MPA and MP formic acid, the alpha-oxidation of MPP followed by dehydration to form MP acrylic acid, the N-acetylation to form NAG (in soil) and finally the photosynthetic incorporation of carbon moieties from completely degraded glufosinate in soil.

Table 34 Total radioactive residues (TRR) and the extractability of residues in crops rotated 28 days after application of [^{14}C] glufosinate to bare soil at a rate of 1.0 kg ai/ha

Crop	Commodity	TRR	Extracted (soluble CH_2Cl_2)	Extracted (water soluble)	PES
		(mg eq/kg)	(%TRR)	(%TRR)	(%TRR)
sandy loam ("Langenfeld")					
Radish	Top	0.175	1.7	82.7	15.5

Crop	Commodity	TRR	Extracted (soluble CH_2Cl_2)	Extracted (water soluble)	PES
		(mg eq/kg)	(%TRR)	(%TRR)	(%TRR)
	Root	0.424	0.4	97.0	2.6
Lettuce	Whole plant	0.094	4.7	55.5	39.8
Wheat	Forage	0.054	1.4	68.7	29.9
	Hay	0.313	2.3	80.6	17.1
	Straw	0.758	0.2	86.0	13.8
	Grain	0.308	0.1	71.8	28.1
loamy sand ("Pikeville")					
Wheat	Straw	0.944	0.5	82.0 + 7.8 [a]	9.7
	Grain	0.242	0.1	67.1	32.8

[a] Aqueous extract and hydrolysate after KOH digestion of PES

Table 35 Nature of residues in lettuce and radish rotated 28 days after application of [^{14}C] glufosinate to bare soil at a rate of 1.0 kg ai/ha (sandy loam "Langenfeld")

	Lettuce		Radish top		Radish roots	
	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR (mg eq/kg)		0.092		0.175		0.424
glufosinate	4.2	0.004	70.1	0.122	41.0	0.174
MPP	18.5	0.017	5.0	0.009	30.0	0.127
MPA	ND	ND	ND	ND	ND	ND
NAG	nd	nd	7.6	0.013	22.6	0.096
Peak A05	(25.8)	(0.024)	ND	ND	3.4	0.015
Fructose	9.8	0.009	ND	ND	–	–
Glucose	7.5	0.007	ND	ND	–	–
peak A05b [a]	8.5	0.008	ND	ND	–	–
Unknown (A03)	6.9	0.006	ND	ND	ND	ND
Total identified	40.0	0.037	82.7	0.144	93.6	0.397
Total characterized [b]	20.1	0.018	1.7	0.003	3.8	0.017
PES	39.8	0.037	15.5	0.027	2.6	0.011

ND not detected,

– = Not analysed

[a] Peak A05b consisted of at least six components

[b] Characterization due to polar extraction, partitioning against CH2Cl2 and chromatographic profiling

Table 36 Nature of residues in wheat rotated 28 days after application of [^{14}C] glufosinate to bare soil at a rate of 1.0 kg ai/ha (sandy loam "Langenfeld")

RAC	Wheat forage		Wheat hay		Wheat straw		Wheat grain	
TRR (mg eq/kg)	0.054		0.313		0.758		0.309	
Residue components	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg
glufosinate	19.7	0.011	ND	ND	8.6	0.065	4.6	0.014
MPP	16.7	0.009	66.0	0.207	58.0	0.439	60.3	0.186
MPA	10.7	0.006	14.6	0.046	14.2	0.107	5.8	0.018
NAG	ND	ND	ND	ND	ND	ND	ND	ND
peak A05	11.3	0.006	ND	ND	ND	ND	1.2	0.004
peak A03	10.2	0.006	ND	ND	5.2	0.040	ND	ND
Total identified	47.1	0.026	80.6	0.252	80.8	0.612	70.7	0.218
Total characterized but not identified [a]	22.9	0.013	2.3	0.007	5.3	0.041	10.3	0.004
Total bound (PES)	29.9	0.016	17.1	0.053	13.8	0.125	28.1	0.087

ND not detected

[a] Characterization due to polar extraction, partitioning against CH_2Cl_2 and chromatographic profiling

Table 37 Nature of residues in wheat rotated 28 days after application of [^{14}C] glufosinate to bare soil at a rate of 1.0 kg ai/ha (loamy sand "Pikeville")

	Wheat straw		Wheat grain	
	% TRR	mg eq/kg	% TRR	mg eq/kg
TRR (mg eq/kg)		0.944		0.242
glufosinate	1.6	0.015	ND	ND
MPP	59.0	0.556	56.1	0.136
MPA	10.2	0.096	8.1	0.020
MP formic acid	6.0	0.056	ND	ND
MP acrylic acid	1.7	0.016	ND	ND
A05	1.7	0.016	2.9	0.007
Total identified	78.5	0.741	67.1	0.163
Total characterized but not identified [a]	11.9	0.112	67.2	0.163
PES	9.7	0.092	32.8	0.079

ND not detected

[a] Characterization due to polar extraction, partitioning against CH_2Cl_2 and chromatographic profiling

Summary of plant metabolism following three different uses of glufosinate ammonium (GA)

Crop	Rate (kg ai/ha)	PHI (days)	TRR (mg eq/kg)	Sample	Residue Components [a]			
					GA	MPP	MPA	NAG
Non-selective weed control								
Corn	1.9	80	0.068	Forage	–	M	–	–
		164	0.115	Fodder	–	M	–	–
			0.035	Grain	–	M	–	–
Corn	1.0	125	0.077	Dry leaves	–	M	–	–
			0.011	Cob	–	M	–	–
			0.014	Grain	–	M	–	–
Wheat	1.25	119	0.22	Grain	–	M	–	–
			0.50	Husk	–	M	–	–
			0.49	Straw	–	M	–	–
Potato	1.0	83	0.886	Leaves	–	M	–	–
			0.153	Stems	–	M	–	–
			0.280	Whole Tubers	–	M	–	–
Apple	1.5	~98	0.104	Fruit, Picked	–	M	–	–
Lettuce	3 mg/L	Hydroponic model			–	M	–	–
Grape	1.5	61	≤ 0.008	Grape	nd	nd	nd	nd
Use as a desiccant								
Potatoes	1.0	10	236.3	Dry Leaves	M	m	–	–
			10.0	Green Leaves	M	m	–	–
			0.014	Whole tuber	nd	nd	nd	nd
			0.039	Potato Peel	M	–	–	–
Oilseed rape	0.6	7	1.76	Seeds	M	vm	–	–
			104.68	Hulls	M	vm	–	–
			45.81	Stems + Leaves	M	vm	–	–
Bean	0.6	14	0.30	Beans	M	–	–	–
			31.15	Hulls	M	–	–	–
			116.0	Stems + Leaves	M	M	–	–
Selective use in genetically modified, glufosinate-tolerant crops								
Canola	0.75	21	5.3	Top growth	M	m	–	M
	0.75	120	0.045–0.109	Seeds	m–M	m–M	–	vm
Oilseed rape	2 × 0.8	102	12.71	Straw	M	vm	–	M
			7.09	Hulls	M	m	–	M

Crop	Rate (kg ai/ha)	PHI (days)	TRR (mg eq/kg)	Sample	Residue Components [a]			
					GA	MPP	MPA	NAG
			0.53	Seeds	m	m	vm	M
Tomato	0.8	74	8.66	Leaves	M	vm	–	M
			9.09	Leaves Rinse Extract	 M M	 vm m	 – –	 vm M
			0.203	Fruit	–	–	–	M
Soya beans	2 × 0.5	85	3.2	Straw	M	M	m	M
			4.7	Pods	m	M	vm	M
			1.4	Beans	m	M	m	M
Soya beans	2 × 0.54	84	21.9	Straw	M	m	vm	M
			18.0	Husks	M	M	vm	M
			5.01	Beans	M	M	vm	M
Corn	2 × 0.53	102	2.01	Fodder	m	M	vm	M
			0.251	Cobs	vm	M	M	M
			0.872	Husks	vm	M	M	M
			0.130	Grain	vm	M	vm	m
Sugar beet	2 × 0.6	146	2.05	Leaves Rinse Extract	 vm M	 vm vm	 – –	 vm M
			0.93	Beets	M	m	–	M
Paddy rice	2 × 0.5	184	1.12–1.36	Grain	m	M	vm	M
			9.50–13.08	Straw	M	M	vm	M
			2.0–94	Forage	M	vm	vm	M
Confined crop rotation								
Wheat	1 (bare soil)	plant back intervals: 28 and 119 days		Forage Straw Grain	M m vm	M M M	M m–M m	– – –
Radish				Root	M	m–M	–	M
Lettuce				Leaves	vm	M	m	–

[a] Indication of the level of glufosinate (GA) and metabolites at the indicated sampling time

M = major > 10% of TRR

m = minor 5–10% of TRR

vm = very minor < 5% of TRR,–= not found in sample

nd = not determined.

Environmental fate in soil

The 2009 FAO Manual for the "Submission and evaluation of pesticide residues data for the estimation of maximum residue levels in food and feed" (FAO Plant Production and Protection Paper 197, 2009 FAO Rome. p 21 Table 3.2: Requirements for submission of data on environmental fate for the JMPR) explained the data requirements for studies of environmental fate. The focus should be on those aspects that are most relevant to MRL setting. For glufosinate ammonium, supervised residue trials data are available for root and tuber crops as well as for paddy rice, which means that aerobic degradation in soil is relevant, as well as the normal requirements for hydrolysis and rotational crop studies.

The route and rate of degradation of glufosinate ammonium in aerobic soil was investigated using [^{14}C] glufosinate ammonium labelled in the C-4 alkyl chain (1- and 3,4-position) and in the methyl group attached to the phosphorous atom. Investigations of the degradation behaviour NAG were performed using [3,4-^{14}C]-NAG.

Glufosinate ammonium is readily degraded in aerobic soil to form $^{14}CO_2$, observed as the primary product of microbial conversion. The conversion includes de-amination to PPO followed by stepwise oxidation to the major and transient metabolites MPP and MPA. The proposed degradation pathway is summarized in Figure 6.

The rapid conversion in aerobic soil is documented by DT_{50} values ranging from about 4 to 10 days for glufosinate. The DT_{50} values estimated in soil aerobic degradation studies in the laboratory are summarized for glufosinate in Table 38 and for the metabolites MPP, MPA and NAG in Table 39.

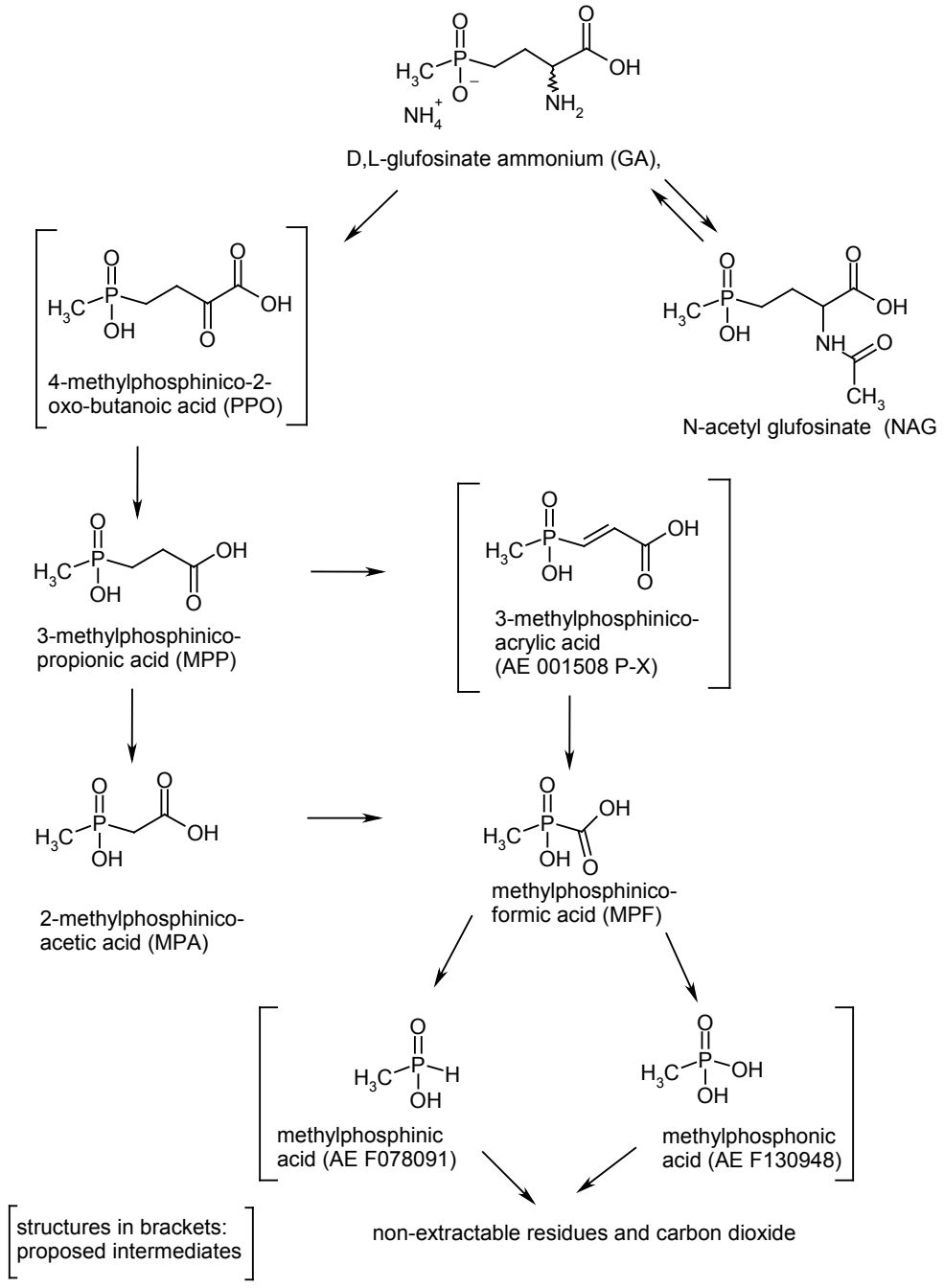


Figure 6 Proposed route of degradation of glufosinate ammonium in aerobic soil

Table 38 Degradation DT_{50} values for glufosinate ammonium in aerobic soil under laboratory conditions

Soil	MWHC (%)	Initial Conc (mg/kg)	Aerobic bacteria (cells/g)	Microbial biomass	DT_{50} (days)	Reference
sandy loam SLV	40	2.3	1.9×10^6	–	6	A36942

Soil	MWHC (%)	Initial Conc (mg/kg)	Aerobic bacteria (cells/g)	Microbial biomass	DT_{50} (days)	Reference
sandy loam SLV	60	2.3	1.9×10^6	–	5	A36942
sandy loam SLV	40	7.7	1.9×10^6	–	10	A36942
silty loam SL2	40	2.3	6.1×10^6	–	8	A36942
loamy sand LS 2.2, fresh	40	2.3	1.9×10^6	34.2	5	A36943
loamy sand LS 2.2, stored	Air dry	2.3	4.2×10^6	18.0	13	A36943
sandy loam SLV Tor Süd	40	1.6	6.0×10^6	24.8	3.7	A41299
silty loam SL2 Leland	40	1.6	6.1×10^6	18.6	8.3	A41299
loamy sand LS 2.2 Speyer	40	1.6	0.4×10^6	28.6	6.4	A41299
Marshy soil CHA Chatteris	40	1.6	3.7×10^6	48.7	6.6	A41299
Peaty soil SOU Southery	40	1.6	5.5×10^6	66.8	10 [a]	A41299
sandy loam SLV Tor Süd	40	2.0	–	–	5.9	Allan 1995
L-glufosinate						
Loamy sand LS 2.2	40	2.1	5.9×10^6	65.6	2.4	Zumdick 1995a
sandy loam SLV Tor Süd	40	2.1	1.4×10^6	31.9	3.1	Zumdick 1995a
Sandy loam SL 2.3	75	1.0	2.8×10^6	32.5	3.6	Zumdick 1995b
Silt loam SLNE	75	1.0	7.7×10^6	47.3	3.4	Zumdick 1995b

[a] Estimated, includes metabolite NAG

Table 39 Summary of DT_{50} values of metabolites of glufosinate in aerobic soil

Soil	MWHC (%)	Initial Conc (mg/kg)	Aerobic bacteria (cells/g)	Microbial biomass	DT50 (days)	Reference
MPP						
sandy loam SLV Tor Süd	40	1.6	6.0×10^6	24.8	13.4	A41299
silty loam SL2 Leland	40	1.6	6.1×10^6	18.6	20.1	A41299
loamy sand LS 2.2 Speyer	40	1.6	0.4×10^6	28.6	16.0	A41299
Marshy soil CHA Chatteris	40	1.6	3.7×10^6	48.7	22.0	A41299
MPA						
sandy loam SLV Tor Süd	40	1.6	6.0×10^6	24.8	18.2	A41299
NAG						
Loamy sand LS 2.2	40	2.1	5.9×10^6	65.6	1.0	Zumdick 1995a
sandy loam SLV Tor Süd	40	2.1	1.4×10^6	31.9	< 1	Zumdick 1995a
Sandy loam SL 2.3	75	1.0	2.8×10^6	32.5	0.9	Zumdick 1995b
Silt loam SLNE	75	1.0	7.7×10^6	47.3	0.9	Zumdick 1995b

Glufosinate aerobic soil metabolism

Stumpf (1987a A36942) studied the aerobic soil degradation of [^{14}C] glufosinate ammonium in the dark at 22 ± 2 °C in two soils, a sandy loam "SLV" (sand 59%, silt 34%, clay 6.9%, %OM 1.3%; cation exchange capacity 5.6 meq/100 g; pH 5.6; 36% of maximum water holding capacity = MWHC) and silt loam "SL2" (sand 11%, silt 70%, clay 19%, %OM 1.2%; cation exchange capacity 18.4 meq/100 g; pH 5.2; 46% of maximum water holding capacity). [1-^{14}C]-Labelled glufosinate ammonium was applied at a concentration of 2.3 mg as/kg soil (dry weight), equivalent to a field rate of 2.0 kg ai/ha, to both soils and also at 7.7 mg as/kg soil (dry weight) equivalent to a field rate of 6.7 kg ai/ha for the SLV soil. The soils were microbially viable throughout the study.

Glufosinate ammonium was extensively metabolised (mineralised) to form CO_2 as the predominant product; 59.2 to 80.9% of the applied radioactivity (AR) after 28 days in soil SLV and 50.5% after 28 days in soil SL2. PPO was observed as a minor component of the ^{14}C. The distribution of radioactive residues is summarized in Tables 40 to 42 for soil SLV and Table 43 for soil SL2.

Table 40 Distribution of radioactive residues after incubation of [1-^{14}C] glufosinate ammonium in soil SLV at 40% MWHC, rate of 2.3 mg/kg soil (% AR)

Day	Extracted			Unextracted	$^{14}CO_2$	Total
	Total	glufosinate	PPO			
0	84.5	84.5	< 1.0	3.6	-/-	88.1
1	72.0	72.0	< 1.0	8.4	5.9	86.3
4	49.4	46.8	2.6	11.5	18.6	79.5
7	32.9	10.8	3.2	13.0	27.2	73.1
14	17.5	29.7	< 1.0	9.6	44.4	71.5
21	9.7	9.7	< 1.0	8.3	57.1	75.1
28	3.0	3.0	< 1.0	6.3	61.7	71.0

Table 41 Distribution of radioactive residues after incubation of [1-^{14}C] glufosinate ammonium in soil SLV at 60% MWHC, rate of 2.3 mg/kg soil (% AR)

	Extracted			Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	PPO			
0	84.5	84.5	< 1.0	3.6	–/–	88.1
1	–/–	–/–	–/–	–/–	13.3	–/–
4	38.8	34.1	4.3	9.7	37.2	85.7
7	30.9	28.6	2.3	4.9	51.4	87.2
14	12.3	12.3	< 1.0	7.9	68.9	89.1
21	–/–	–/–	–/–	–/–	78.1	–/–
28	1.0	1.0	< 1.0	5.0	80.9	86.9

Table 42 Distribution of radioactive residues after incubation of [1-^{14}C] glufosinate ammonium in soil SLV at 40% MWHC, exaggerated rate of 7.7 mg/kg soil (% AR)

	Extracted			Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	PPO			
0	80.0	80.0	< 1.0	2.9	–/–	82.9
1	72.6	70.7	1.9	10.3	5.0	87.9
4	59.4	58.4	1.1	9.1	16.9	85.4
7	44.8	43.3	1.5	13.6	26.2	84.6
14	27.3	27.3	< 1.0	12.8	41.4	81.5
21	15.7	15.7	< 1.0	9.5	52.4	77.6
28	11.5	11.5	< 1.0	6.1	59.2	76.8

Table 43 Distribution of radioactive residues after incubation of [1-^{14}C] glufosinate ammonium in soil SL2 at 40% MWHC, rate of 2.3 mg/kg soil (% AR)

	Extracted			Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	PPO			
0	66.2	66.2	< 1.0	14.1	–/–	80.3
1	52.1	45.9	6.2	21.9	4.5	78.5
4	35.3	30.2	5.1	26.8	13.5	75.6
7	25.6	25.6	< 1.0	20.7	20.9	67.2
14	18.4	18.4	< 1.0	18.0	33.9	70.3
21	11.2	9.6	1.7	11.2	43.9	66.3
28	4.6	4.6	< 1.0	9.7	50.5	64.8

Degradation of [1-^{14}C] glufosinate ammonium was faster for higher moisture content soils (SLV, 60% MWHC vs. 40% MWHC) and slower for soils treated at higher doses (SLV, 7.7 mg/kg vs. 2.3 mg/kg). The DT_{50} for glufosinate ammonium under aerobic conditions was 5 to 8 days.

In a separate study Stumpf (1987b A36943) compared the aerobic degradation in field fresh soil to dry stored soil. [1-^{14}C] glufosinate ammonium was applied at 2.3 mg ai/kg soil (dry weight), equivalent to a field rate of 1.7 kg ai/ha to freshly collected soil (LS 2.2 'field fresh' sand 92%, silt

2.3%, clay 5.2%, %OM 5.4; cation exchange capacity 8.7 meq/100 g; pH 5.4; 41% of maximum water holding capacity) and samples of the same soil that had been stored air-dry under outdoor conditions and protected from rainfall for about 1 year prior to application.

Glufosinate ammonium was mineralised to form carbon dioxide (66.8% AR for field fresh samples and 17.0 % AR for stored soil at 28 days) (Table 44, 45). Degradation was slower in soil stored under dry under outdoor conditions compared to "field fresh" soil.

Table 44 Distribution of radioactive residues after incubation of [1-^{14}C] glufosinate ammonium in freshly collected soil LS 2.2 at 40% MWHC, rate of 2.3 mg/kg soil (% AR)

	Extracted			Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	PPO			
0	83.0	80.6	2.4	6.7	–/–	89.7
1	73.4	67.8	5.6	12.6	8.2	94.2
4	48.7	45.2	3.5	18.0	24.2	90.9
7	38.8	37.4	1.9	19.9	33.7	92.4
14	11.1	9.5	1.6	13.6	50.8	75.5
21	6.2	6.2	< 1.0	14.0	60.7	80.9
28	2.3	2.3	< 1.0	9.6	66.8	78.7

Table 45 Distribution of radioactive residues after incubation of [1-^{14}C] glufosinate ammonium in stored soil LS 2.2 at 40% MWHC, rate of 2.3 mg/kg soil (% AR)

	Extracted			Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	PPO			
0	83.6	83.6	< 1.0	6.9	–/–	90.5
1	79.9	79.9	< 1.0	10.3	0.4	90.6
4	63.8	41.4	22.5	21.6	1.4	86.8
7	58.5	53.7	4.8	23.9	2.7	85.1
14	53.0	43.5	9.5	26.9	7.0	86.9
21	24.9	21.1	3.8	35.0	12.5	72.4
28	24.1	16.3	5.2	29.3	17.0	70.4

The DT_{50}/DT_{90} values soil were 5/18 days for freshly collected soil while they were 13/42 days for soil that had been stored dry outdoor for 1 year prior to incubation.

Stumpf (1989b A41299) studied the aerobic and anaerobic degradation of [^{14}C] glufosinate ammonium in the dark in five soils: Tor Süd sandy loam, Leland silty loam, Speyer loamy sand, Chatteris marshy soil and Southern peaty soil. Soil samples were each treated with [3,4-^{14}C]-labelled glufosinate ammonium at a concentration of 1.6 mg as/kg soil (dry weight), equivalent to a field rate of 1.2 kg ai/ha.

In all soils glufosinate ammonium was mineralised to form carbon dioxide. Amounts of $^{14}CO_2$ as a proportion of the AR recovered by 120 days incubation were 60.3% for sandy loam, 24.6% for silty loam, 19.7% for loamy sand, 41.1% for marshy soil and 25.8% for peaty soil. Unextracted ^{14}C was up to 43.7% AR. MPP and MPA were major though transient metabolites. The distribution of radioactive residues is summarized in Table 46 (soil SLV), Table 47 (soil SL2), Table 48 (soil LS2.2), Table 49 (soil CHA) and Table 50 (soil SOU).

Table 46 Distribution of radioactive residues after aerobic incubation of [3,4-^{14}C] glufosinate ammonium in Tor Süd sandy loam at 40% MWHC, rate of 1.6 mg/kg soil (% AR)

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
0	97.4	90.3	7.1	< 0.5	1.8	–	99.2
1	85.5	57.1	20.1	8.2	7.0	0.3	92.8
4	87.8	40.5	38.6	8.7	7.2	1.6	96.6
7	85.0	22.3	46.9	15.8	13.2	3.5	101.7
14	74.1	3.4	44.3	26.4	19.1	10.0	103.2
20	60.5	2.6	36.5	21.3	21.0	16.4	97.9

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
29	43.9	1.2	21.9	20.7	24.3	25.0	93.2
60	9.0	0.7	3.5	4.7	28.5	46.3	83.8
90	7.5	< 0.5	3.0	4.5	22.2	55.5	85.2
120	4.1	< 0.5	2.0	2.1	20.5	60.3	84.9

Table 47 Distribution of radioactive residues after aerobic incubation of [3,4-^{14}C] glufosinate ammonium in Leland silty loam at 40% MWHC, rate of 1.6 mg/kg soil (% AR)

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
0	81.0	64.8	11.7	< 0.5	10.9	–	91.9
1	54.3	32.3	18.9	3.0	29.2	0.3	83.8
4	58.9	41.2	17.7	< 0.5	20.9	1.1	80.9
7	54.7	31.7	23.0	< 0.5	35.6	2.2	92.5
14	42.0	16.9	25.1	< 0.5	39.1	4.7	85.8
20	34.0	10.6	21.8	1.6	40.5	6.7	81.2
29	23.0	4.3	17.8	0.9	43.7	9.2	75.9
60	7.4	0.2	5.3	1.9	40.1	15.3	62.8
90	2.9	< 0.5	1.9	1.0	29.2	20.5	52.6
120	1.9	< 0.5	1.0	0.9	29.5	24.6	56.0

Table 48 Distribution of radioactive residues after aerobic incubation of [3,4-^{14}C] glufosinate ammonium in soil Speyer loamy sand at 40% MWHC, rate of 1.6 mg/kg soil (% AR)

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
0	92.8	83.5	9.3	< 0.5	3.7	–	96.5
1	75.1	56.9	18.2	< 0.5	17.9	0.1	93.1
4	76.7	49.8	26.9	< 0.5	19.5	0.6	96.8
7	67.1	24.8	36.7	5.6	28.2	1.3	96.6
14	54.5	10.7	35.4	8.4	35.5	3.2	93.2
20	41.9	5.9	26.1	9.9	43.3	5.0	90.2
29	32.7	3.9	19.5	9.3	41.6	7.1	81.4
60	8.6	0.8	4.1	3.7	42.8	13.4	64.8
90	4.5	< 0.5	2.5	2.0	31.8	17.0	53.3
120	3.2	< 0.5	2.0	1.2	27.6	19.7	50.5

Table 49 Distribution of radioactive residues after aerobic incubation of [3,4-^{14}C] glufosinate ammonium in Chatteris marshy soil at 40% MWHC, rate of 1.6 mg/kg soil (% AR)

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
0	93.7	79.0	14.7	< 0.5	5.6	–	99.3
1	63.3	38.9	24.4	< 0.5	29.2	0.2	92.7
4	62.3	30.7	29.8	0.8	29.8	1.3	93.4
7	63.0	20.6	40.5	1.9	30.0	2.5	95.5
14	60.2	14.4	39.8	5.9	32.7	6.3	99.2
20	37.6	2.2	29.2	6.2	37.2	9.2	84.0
29	43.1	4.3	32.2	6.7	33.5	13.8	90.4
60	11.7	0.9	8.0	2.7	35.0	26.7	73.4
90	4.7	< 0.5	3.0	1.7	28.9	35.4	69.0
120	3.0	< 0.5	2.0	1.0	29.2	41.1	73.3

Table 50 Distribution of radioactive residues after aerobic incubation of [3,4-^{14}C] glufosinate ammonium in Southery peaty soil at 40% MWHC, rate of 1.6 mg/kg soil (% AR)

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
0	92.4	62.1	10.5	< 0.5	5.7	–	98.1
29	57.2	9.1	30.6	7.9	30.4	7.4	95.0
60	38.0	1.4	32.8	3.7	33.9	13.1	85.0
120	11.5	< 0.5	8.0	3.5	37.6	25.8	74.9

DT_{50} values for glufosinate ammonium in five aerobic soils ranged from 3.7 to 10 days thus confirming the potential for rapid degradation in soil. DT_{50} values were also calculated for the metabolites assuming simple first-order kinetics. For MPP half-lives ranged from 13 to 22 days while for MPA a half-life of 18 days was calculated for the sandy loam soil data, the only soil with suitable data for analysis.

After 7 days of aerobic incubation, a sample of Tor Süd sandy loam was flooded to induce anaerobic conditions. Under anaerobic conditions the mineralisation to $^{14}CO_2$ was interrupted as only 3.4% AR was recovered as $^{14}CO_2$ during 71 days of essentially anaerobic incubation (Table 51).

Table 51 Distribution of radioactive residues after anaerobic incubation of [3,4-^{14}C] glufosinate ammonium in Tor Süd sandy loam, rate of 1.6 mg/kg soil (% AR)

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
–7	97.4	90.3	7.1	< 0.5	1.8	–	99.2
–6	85.5	57.1	20.1	8.2	7.0	0.3	92.8
–3	87.8	40.5	38.6	8.7	7.2	1.8	96.8
0	85	22.3	46.9	15.8	13.2	3.8	102
11	81.9	8.9	50.2	22.5	8.2	4.2	94.3
26	83.9	< 0.5	64.2	15.8	6.6	4.3	94.8
46	82	< 0.5	62.9	18.3	5.8	5.1	92.9
71	75.1	< 0.5	63.5	21.7	7.5	7.2	89.8

In a separate study Allan (1995 A55393) compared the aerobic soil metabolism of glufosinate following incorporation of bush bean leaves containing glufosinate residues into soil with that when glufosinate was directly incorporated into soil. A sandy loam soil (Tor Süd) was directly treated with [3,4-^{14}C]-labelled glufosinate ammonium at a concentration of 2.0 mg ai/kg soil (dry weight), equivalent to a field rate of 1.5 kg ai/ha or at the same rate but via incorporation of bush bean foliage. The samples were incubated under aerobic conditions in the dark at 20 ± 1 °C and soil moisture of 40% of MWHC for up to 120 days.

Glufosinate ammonium was found to be extensively degraded to form carbon dioxide as the predominant product. Unextracted ^{14}C reached a maximum of 24.9% by day 90 followed by a decrease to 21.3% AR after 120 days. Further analysis of ^{14}C that was not solvent extracted showed it was incorporated into fulvic acid, humin and humic acid.

The distribution of radioactive residues is summarized in Table 52.

Table 52 Distribution of radioactive residues after incubation of [3,4-^{14}C] glufosinate ammonium in directly treated soil SLV at 40% MWHC, rate of 2.0 mg/kg soil (% AR)

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
0	105.6	104.3	0.0	0.0	1.8	–/–	107.4
1	89.8	76.5	11.0	0.0	8.3	0.6	98.7
3	82.3	56.0	19.4	2.1	11.4	1.5	95.2
7	77.0	32.0	30.8	6.4	14.5	5.6	97.1
14	70.3	17.5	37.2	10.5	20.9	11.1	102.3
21	61.2	7.8	33.4	14.8	21.0	17.4	99.6
28	59.6	3.3	33.7	19.4	19.5	22.5	101.6

	Extracted				Unextracted	$^{14}CO_2$	Total
Day	Total	glufosinate	MPP	MPA			
41	45.8	0.9	21.5	20.1	19.4	25.9	91.1
59	32.4	0.5	12.7	17.3	24.3	43.2	99.9
90	8.7	0.1	1.4	6.8	24.9	57.4	91.0
120	5.6	0.5	1.6	1.6	21.3	62.3	89.2

The degradation pattern was similar for direct application of glufosinate and when incorporated via bush bean foliage (data not shown). However, the DT_{50} value for foliage inclusion was shorter than for direct addition, presumably as a result of the increased microbial activity in soil following foliage inclusion.

The DT_{50} value for glufosinate ammonium in aerobic Tor Süd sandy loam was 5.9 days following direct application and 2.9 days when added by incorporation of bush bean foliage.

In a study by Stumpf (1995 A53581) the route of aerobic degradation of [^{14}C-methyl] glufosinate ammonium in a sandy loam soil was studied. Soil was treated at a concentration of 2.0 mg ai/kg soil (dry weight), equivalent to a field rate of 1.5 kg ai/ha. The samples were incubated under aerobic conditions in the dark at 20 ± 1 °C and soil moisture of 40% of MWHC for 160 days in maximum.

Glufosinate ammonium was extensively degraded. The proportion of radioactivity recovered as volatiles and $^{14}CO_2$ was low < 1.8%. Residues remaining after solvent extraction represented 20.6% AR at day 160. The distribution of radioactive residues is summarized in Table 53. The main metabolite detected was methylphosphonic acid (maximum 58% AR). Other significant, but transient, metabolites were MPP and MPA. The low recovery of radioactivity as $^{14}CO_2$ is attributed to the position of the ^{14}C label.

Table 53 Distribution of radioactive residues after incubation of [^{14}C-methyl] glufosinate ammonium in soil SLV at 40% MWHC, rate of 2.0 mg/kg soil (% AR)

Day	Extracted					Unextracted	$^{14}CO_2$	Total
	Total	glufosinate	MPP	MPA	methylphosphonic acid			
42	69.0	0.0	22.9	2.0	44.0	22.7	–	91.7
100	65.0	0.0	24.7	0.0	40.4	19.4	–	84.4
160	56.8	0.0	0.0	0.0	58.3	21.0	–	77.8
160 [a]	48.7	–	–	–	–	20.6	1.8	71.1

[a] Closed system used to detect evolution of $^{14}CO_2$

The degradation of [^{14}C-methyl]-labelled glufosinate ammonium in aerobic soil proceeded *via* the same transformation steps as observed for other positions of radiolabel. Microbial transformation results in formation of methylphosphonic acid as an intermediate soil residue prior to its incorporation into the soil matrix.

Studies carried out in Canada support the above reports. Smith (1988, 1989) reported the transformation of [^{14}C] glufosinate on three prairie soils: a clay, clay loam and sandy loam when present initially at 2 mg/kg, 20–40% maximum water holding capacity and at 10 or 20 °C. The half-lives at 20 °C were 3–7 days while at 10 °C they were 8–11 days. Over the 90 day incubation period at 20 °C between 28 and 55% AR was mineralised to $^{14}CO_2$. MPP was a significant but transient metabolite. Between 2.4 and 9.5% of the initial ^{14}C was incorporated into soil microbial biomass and 7–13% into fulvic acid, humic acid and humin soil fractions. Gallina and Stephenson (1992) studied the degradation of [^{14}C] glufosinate in two Ontario soils; a sandy loam and a loam. Glufosinate was rapidly converted to MPP and MPA and eventually mineralised to CO_2. The half-life for glufosinate was between 3 and 7 days.

Tebbe and Reber (1991) using [1-^{14}C]- and [3,4-^{14}C]-labelled glufosinate noted that decarboxylation of glufosinate precedes oxidation and that degradation was principally due to

microbial activity as sterilisation of soil by gamma-irradiation decreased the degradation rate by over 95%.

NAG aerobic soil metabolism

NAG is a metabolite formed from glufosinate in glufosinate-tolerant plants. Residues in plants may therefore reach the soil. Consequently, the degradation behaviour of NAG has been investigated in soil under aerobic conditions. Degradation of NAG was extensive *via* spontaneous de-acetylation to form glufosinate. The further steps of metabolism followed the same route as determined from direct application of glufosinate ammonium to aerobic soil: CO_2 was formed as the predominant product besides unextracted residues. The degradation was accompanied by the formation of metabolites MPP and MPA as major and transient products. As with glufosinate, their successive occurrence at maximum levels suggests that the transformation proceeds in a stepwise fashion with de-amination and the oxidative removal of one carbon atom unit in each step ending in mineralisation to CO_2.

Zumdick (1995a A54356) studied the aerobic degradation of NAG in a sandy loam soil (Speyer) and a loamy sand (Tor Süd) soils. In addition the degradation of NAG when incorporated via residues in tolerant tomato foliage was studied using the loamy sand soil. Soils were treated with [3,4-^{14}C]-NAG at 2.1 mg ai/kg soil (dry weight) equivalent to a field rate of 2.8 kg ai/ha and incubated under aerobic conditions in the dark at 20 ± 2 °C and soil moisture of 40% of MWHC for up to 120 days. The rate of incorporation when applied as residues present in tomato foliage was 1 mg glufosinate eq/kg soil.

Degradation of [3,4-^{14}C]- NAG was via de-acetylation to form L-glufosinate with further steps the same as following direct application of glufosinate ammonium to aerobic soil: CO_2 was the predominant degradation product accompanied by the formation of metabolites glufosinate, MPP and MPA as major but transient transformation products. The distribution of radioactive residues is summarized in Tables 54 and Table 55.

Table 54 Distribution of radioactive residues after incubation of [3,4-^{14}C] NAG in directly treated soil LS2.2 at 40% MWHC, rate of 2.1 mg/kg soil (% AR)

Day	Extracted					Unextracted	$^{14}CO_2$	Total
	Total	L-NAG	L-glufosinate	MPP	MPA			
0	85.6	69.8	19.9	8.4	0.4	2.4	–	88.0
0.25	81.4	14.2	58.4	12.9	1.2	3.8	0.3	85.5
1	85.8	15.1	23.4	34.5	4.7	10.0	1.1	96.9
2	83.2	6.2	22.5	38.1	5.9	10.3	3.6	97.1
3	81.2	5.1	16.0	41.9	7.4	8.4	2.8	92.4
7	75.9	4.1	6.3	35.6	9.6	13.2	11.4	100.5
14	58.7	2.8	5.2	26.8	7.6	13.8	20.7	93.2
28	33.3	1.3	2.2	5.2	0.8	14.1	38.6	86.0
62	10.7	0.5	1.3	1.9	0	16.3	49.4	76.4
90	4.2	0.5	1.1	0.7	0	16.2	51.5	71.9
120	3.2	0.3	0.4	0.1	0	18.2	71.9	93.3

Table 55 Distribution of radioactive residues after incubation of [3,4-^{14}C] NAG in directly treated soil SLV at 40% MWHC, rate of 2.1 mg/kg soil (% AR)

Day	Extracted					Unextracted	$^{14}CO_2$	Total
	Total	L-NAG	L-glufosinate	MPP	MPA			
0	96.4	61.2	29.9	4.7	0	2.6	–	99.0
0.25	96.1	28.8	57.5	8.1	0.7	2.2	0.2	98.5
1	85.6	9.2	49.2	21.9	2.2	8.9	0.9	95.4
2	85.2	4.3	41.1	28.8	3.4	8.1	2.3	95.6
3	78.4	3.1	23.5	37.2	5.9	11.4	4.9	94.7
7	87.0	1.8	37.6	33.9	8.1	10.7	1.1	98.8
14	69.2	1.0	2.6	38.8	19.4	11.8	10.5	91.5
28	53.4	0.4	1.0	28.2	18.0	14.9	22.7	91.0
62	6.7	0.2	1.0	2.1	1.2	23.4	55.6	85.7

Day	Extracted					Unextracted	$^{14}CO_2$	Total
	Total	L-NAG	L-glufosinate	MPP	MPA			
90	3.8	0.4	1.0	0.7	0.3	21.5	67.0	92.3
120	2.2	0.2	0.4	0.5	0.1	19.6	66.4	88.2

Half-life for disodium N-acetyl-L-glufosinate was 1.0 day in both soils and for both direct application and application as residues in tomato foliage. The half-life for degradation of the formed L-glufosinate was also rapid (< 3.1 days). Compared to direct addition, incorporation of tomato foliage led to increased rates of degradation with half-lives for NAG and L-glufosinate of < 1 and 3.1 days for direct addition and 1.0 and < 1 days following incorporation via tomato foliage. This may be due to increased microbial biomass following incorporation of plant derived organic matter.

In a separate study Zumdick (1995b A54528) investigated the aerobic degradation of ^{14}C-NAG in a German sandy loam soil (SL 2.3) and a US silt loam (SLNE). The soils were treated with [3,4-^{14}C]-NAG at a concentration of 1.0 mg ai/kg soil (dry weight), equivalent to a field rate of 1.3 kg ai/ha and incubated under aerobic conditions in the dark at 20 ± 2 °C and a soil moisture of 75% of MWHC at 0.33 bar.

Degradation of [3,4-^{14}C]-NAG was extensive via spontaneous de-acetylation to form glufosinate. The further steps of metabolism were found to be the same as determined from direct application of glufosinate ammonium to aerobic soil: carbon dioxide was formed as the predominant product at levels of 77.3% applied ^{14}C (soil SLV) and 66.7% (soil SLNE) after 120 days of incubation. Unextracted residues were low amounting to 21.3% applied ^{14}C (soil SLV) and 21.8% (soil SLNE) after 120 days. The distribution of radioactive residues is summarized in Tables 56 and Table 57.

L-glufosinate, MPP and MPA were formed as major and transient products of transformation. As in the other studies, the successive occurrence of their maximum levels suggests that the transformation proceeds stepwise following de-amination and the oxidative removal of one carbon atom unit each by microbial processes to end up in mineralisation (L-NAG→L-glufosinate→MPP→MPA).

Table 56 Distribution of radioactive residues after incubation of [3,4-^{14}C] NAG in directly treated soil SL2.3 at 75% MWHC at 0.33 bar, rate of 1.0 mg/kg soil (% of applied ^{14}C)

Day	Extracted					Unextracted	$^{14}CO_2$	Total
	Total	L-NAG	L-glufosinate	MPP	MPA			
0.1	102.3	91.4	9.7	0.8	0.1	0.3	–	102.6
0.25	90.5	15.6	65.5	7.3	0.6	7.0	0.2	97.7
1	84.8	7.5	47.6	20.9	1.8	11.4	0.8	97.0
3	81.8	4.7	25.6	35.1	5.2	6.9	7.0	95.7
7	66.1	3.2	10.1	32.2	9.1	10.1	16.1	92.3
14	48.2	1.9	4.3	19.5	11.9	13.8	29.4	91.4
21	37.7	1.2	6.1	11.5	8.8	13.0	40.8	91.5
30	24.0	0.7	4.2	5.8	5.3	15.5	47.9	87.4
59	7.7	1.3	1.5	1.5	0.2	17.6	63.7	89.0
90	4.0	0.3	0.6	0.3	nd	21.2	68.3	93.5
120	2.4	–	–	–	–	21.3	77.3	101

Table 57 Distribution of radioactive residues after incubation of [3,4-^{14}C]-NAG in directly treated soil SLNE at 75% MWHC at 0.33 bar, rate of 1.0 mg/kg soil (% of applied ^{14}C)

Day	Extracted					Unextracted	$^{14}CO_2$	Total
	Total	L-NAG	L-glufosinate	MPP	MPA			
0.1	100.8	85.4	13.1	1.7	0.1	1.8	–	102.6
0.25	75.4	26.4	35.6	11.4	0.3	16.9	0.3	92.6
1	91.3	10.5	45.7	23.4	3.2	6.1	1.7	99.1
3	77.1	3.5	25.5	25.2	11.2	11.3	6.3	94.7
7	66.8	2.4	10.8	18.3	20.6	11.7	15.2	93.7
14	36.2	0.7	2.6	2.8	17.9	20.0	35.8	92.0

Day	Extracted					Unextracted	$^{14}CO_2$	Total
	Total	L-NAG	L-glufosinate	MPP	MPA			
21	28.8	0.3	2.8	1.9	15.2	17.2	43.5	89.5
30	11.3	0.3	1.5	0.4	4.4	19.3	59.4	90.0
59	4.7	nd	0.8	nd	nd	22.0	62.9	89.6
90	1.4	–	–	–	–	25.2	65.3	92.0
120	2.4	–	–	–	–	21.8	66.7	90.9

The half-life for NAG was below 1 day (0.9 days) for the two soils SL 2.3 and SLNE thus indicating very rapid degradation in soil maintained under aerobic conditions.

Glufosinate and its metabolites are not persistent in soil and are not expected to accumulate with successive applications.

Photolysis on soil surfaces

Stumpf (1989c A40990) studied the photolysis of glufosinate on soil surfaces. The test was conducted in a sterilised (chloroform) sandy loam soil (SLV) treated with [3,4-^{14}C]- glufosinate ammonium at a concentration of 25 mg as/kg soil (dry weight). The samples were continuously irradiated (xenon lamp) at 25 ± 5 °C for 120 hours in maximum. [3,4-^{14}C]-labelled glufosinate ammonium was found to be stable on sterilised soil surfaces after continuous irradiation for 120 hours, essentially no degradation occurred.

Photolysis on soil surfaces is not a significant route of degradation for glufosinate.

Environmental fate in water

Aqueous hydrolysis

Goerlitz (1985 A32265) and Goerlitz (1986 A33845) studied the hydrolysis of glufosinate in sterile aqueous solution at pH 5, 7 and 9 for 300 days at 25 ºC. The concentration of glufosinate remained constant throughout the study period and no breakdown products were detected. Glufosinate is stable in aqueous solution.

Aqueous hydrolysis is not a significant route of degradation for glufosinate.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Methods are available that adequately quantify residues in crops and animal commodities. Most of the analytical methods for the determination of glufosinate-derived residues that were developed prior to 2006 follow the same general principle. The relevant residues are usually extracted in water. Thereafter, the aqueous extract is concentrated to dryness and reacted with trimethyl orthoacetate in the presence of acetic acid. After silica gel purification the obtained derivatives are measured by GC/FPD or more recently by LC-MS/MS.

Depending on the substrate to be analysed, and the variant of the method, a more or less extensive purification of the aqueous extract is performed prior to derivatisation. The most common purification techniques used include precipitation of high molecular weight carbohydrates and proteins with acetone, liquid/liquid partitioning with dichloromethane, cation exchange and anion exchange clean-up.

When reacted with trimethyl orthoacetate in the presence of acetic acid, glufosinate and NAG yield the same derivative. Therefore, glufosinate and NAG are usually determined together as a sum. However, if the two compounds are separated before derivatisation by means of cation exchange clean-up it is possible to determine the residues of glufosinate and NAG separately.

With the availability of new liquid chromatography-columns specially designed for highly polar character and comparatively low molecular weight compounds such as glufosinate and its

metabolites it is possible to quantify glufosinate, MPP and NAG using LC/MS/MS without prior derivatisation. In another method derivatisation of glufosinate was carried out with a mixture of o-phthalic dialdehyde and mercapto propionic acid in the presence of sodium borate. The thus formed derivative of glufosinate is quantified by LC-MS/MS while the metabolites MPP and NAG are measured by LC-MS/MS without prior derivatisation as in the previous approach.

A brief description of the methods is given in Table 58.

Table 58 Summary of major analytical methods used for the determination of glufosinate and metabolites in various matrices

Method/reference	Matrix	Extraction	Clean-up	Detection, LOQ
01188 MR-10/173	Plant commodities	H_2O	Glufosinate: Centrifugation/ ultrafiltration MPP, NAG: Centrifugation, passed over cationic resin, concentrated to dryness, taken up in CH_3CN:ammonium acetate buffer	Glufosinate: Derivatisation (o-phthalic dialdehyde + mercapto propionic acid in presence of boric acid). C18 column LC/MS/MS m/z 386→252 quantification, 386→172, 280 confirmation MPP, NAG: HILIC column, LC/MS/MS MPP: m/z 153→135 quantification, 153→97, 79 confirmation NAG: m/z 224→118 quantification, 224→164, 136 confirmation LOQ 0.01 mg/kg all analytes
AL43/82 A24283 AL65/82 A24976	Plant commodities	H_2O (Soxhlet 8 h reflux)	Glufosinate: add ethanol, filter, cation exchange column, rinse EtOH/H_2O, elute H_2O, evaporate to dryness MPP: Centrifugation, passed over anion exchange column, elute 10% acetic acid, evaporate to dryness	Glufosinate: derivatise (acetic acid, trimethyl orthoacetate, 2 h reflux) 1.5% OV 275 on Chromosorb W-DMCS column, GC/FPD MPP: derivatise (acetic acid, trimethyl orthoacetate, 2 h reflux), clean-up gel permeation chromatography. 1.5% OV 275 on Chromosorb W-DMCS column, GC/FPD LOQ 0.02 mg/kg for each analyte.
AL16/83 A28287 AL17/83 A30727	Plant commodities	H_2O (dialysis 24 h)	Glufosinate: concentrate dialysate to $1/5^{th}$ original volume, add ethanol, filter, cation exchange column, rinse EtOH/H_2O, elute H_2O, evaporate to dryness MPP: Centrifugation, passed over anion exchange column, elute 10% acetic acid, evaporate to dryness	Glufosinate: derivatise (acetic acid, trimethyl orthoacetate, 2 h reflux) 1.5% OV 275 on Chromosorb W-DMCS column, GC/FPD MPP: derivatise (acetic acid, trimethyl orthoacetate, 2 h reflux), clean-up gel permeation chromatography. 1.5% OV 275 on Chromosorb W-DMCS column, GC/FPD LOQ 0.02 mg/kg for each analyte.
AL38/85 A32892, A33483	Plant commodities	H_2O (dialysis 24 h)	concentrate dialysate to dryness, dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on silica gel. OV 170 column, GC/FPD LOQ 0.05 mg/kg for each analyte.
AL11/87	Plant commodities	H_2O (stirring 30 min)	Centrifuge, concentrate to dryness, dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on silica gel. Carbowax or DB17 megabore column, GC/FPD Instant coffee: LOQ 0.01 mg/kg Other matrices: LOQ 0.05 mg/kg for each analyte.
AL9/87	Plant commodities	H_2O (stirring 30 min)	Centrifuge, add equal volume acetone to an aliquot, centrifuge, concentrate to dryness dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on silica gel. Carbowax or DB17 megabore column,

Method/reference	Matrix	Extraction	Clean-up	Detection, LOQ
				GC/FPD LOQ 0.05 mg/kg for each analyte.
AL24/87	Plant commodities	H_2O (stirring 30 min)	Centrifuge, reflux with conc. HCl 7 h, filter, concentrate to dryness, dilute with water, concentrate to dryness, dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on silica gel. Carbowax or DB17 megabore column, GC/FPD LOQ 0.05 mg/kg for each analyte.
AL37/87	Plant commodities (high oil content)	H_2O (stirring 30 min)	Centrifuge, add equal volume acetone to an aliquot, centrifuge, partition with dichloromethane, concentrate aqueous phase to dryness, dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on silica gel. Carbowax or DB17 megabore column, GC/FPD LOQ 0.05 mg/kg for each analyte.
AL40/87	Fats and oils	Dissolve in dichloromethane, extract by partitioning with H_2O	Centrifuge, clean-up aqueous phase on RP18 cartridge, concentrate to dryness, dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on silica gel. Carbowax megabore column, GC/FPD Beef fat: LOQ glufosinate 0.05 mg/kg, MPP 0.1 mg/kg. Other matrices: LOQ 0.05 mg/kg for each analyte but poor recoveries for oenothera oil.
HRAV-5A A40790	Plant commodities	H_2O (stirring 30 min)	Filter, pass over anion exchange column, wash water, elute dilute formic acid, concentrate to dryness, dilute with acetic acid.	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4-5 h reflux). Clean-up on silica gel. DB-Wax megabore column, GC/FPD LOQ 0.05 mg/kg for each analyte.
HRAV-5A A40790	Plant commodities	Maize, soya oils: 5% acetic acid in denatured ethanol/water, 1:1 Other: H_2O (stirring 30 min)	Maize, soya oils: centrifuge, partition dichloromethane, aqueous fractions evaporated to dryness, taken up in denatured ethanol/H_2O, continue as for other samples below. Other samples: Centrifuge, pass over anion exchange column, elute dilute formic acid, concentrate to dryness, dilute with acetic acid. The anion exchange step can be omitted for maize forage/fodder	Glufosinate (Fraction B): derivatise (acetic acid, trimethyl orthoacetate, 4 h reflux). Clean-up on SPE silica gel cartridge. DB-Wax megabore column, GC/FPD LOQ 0.05 mg/kg. MPP, NAG (Fraction A): derivatise (acetic acid, trimethyl orthoacetate, 4 h reflux). Clean-up on SPE silica gel cartridge. DB-Wax megabore column, GC/FPD LOQ 0.05 mg/kg for each analyte.
HRAV-24 A52270 AE-24 A54051	Plant commodities (glufosinate tolerant crops)	H_2O (stirring 30 min)	Maize, soya oils: centrifuge, partition dichloromethane, aqueous fractions evaporated to dryness, taken up in denatured ethanol/H_2O, continue as for other samples below. Other samples: Filter, pass over anion exchange column, wash water, elute dilute formic acid, concentrate to dryness, dilute with ethanol/water (50:50), load on cation exchange column. Elute NAG/MPP with ethanol water (Fraction A). Elute glufosinate with ethanolic ammonia Fraction B). Each fraction is evaporated to dryness. The anion exchange step can be omitted for maize forage/fodder	Glufosinate (Fraction B): derivatise (acetic acid, trimethyl orthoacetate, 4 h reflux). Clean-up on SPE silica gel cartridge. DB-Wax megabore column, GC/FPD LOQ 0.05 mg/kg. MPP, NAG (Fraction A): derivatise (acetic acid, trimethyl orthoacetate, 4 h reflux). Clean-up on SPE silica gel cartridge. DB-Wax megabore column, GC/FPD LOQ 0.05 mg/kg for each analyte.

Method/reference	Matrix	Extraction	Clean-up	Detection, LOQ
AE-24A	Plant commodities	H_2O (stirring 30 min)	soya seeds, meal, hulls: centrifuge then proceed as for other samples. other samples: filter, pass over anion exchange column, wash with water, elute dilute formic acid, concentrate to dryness, dilute with acetic acid. For oil, the extraction and anion exchange step can be omitted	Glufosinate/NAG, MPP: derivatise (trimethyl orthoacetate, 4-5 h reflux). Clean-up on silica gel. DB-Wax megabore column, GC/FPD LOQ 0.05 mg/kg for each analyte. Note: method does not distinguish between glufosinate and NAG
01109 (GL-001-P07-01)		H_2O (stirring 30 min)	Add acetone, centrifuge, concentrate to dryness, dilute in water/methanol (95/5 v/v), clean-up SAX cartridge, elute using water/methanol (1/1, v/v), concentrate to dryness, dilute in CH_3CN:ammonium acetate buffer	HILIC column, LC/MS/MS: Glufosinate: m/z 182→136 MPP: m/z 153→135 (or 153→79) NAG: m/z 224→118 Potato: LOQ 0.02 mg/kg Other matrices: LOQ 0.01 mg/kg all analytes
AL2/87 A35957	Milk	H_2O (dialysis 24 h)	concentrate dialysate to dryness, dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on RP 18 and silica gel cartridges in series. Carbowax column, GC/FPD LOQ 0.02 mg/kg for each analyte.
AL32/88	Eggs	Add ethanol to denature proteins, extract with H_2O (stirring 30 min)	Centrifuge, partition first with dichloromethane, then n-hexane. Also counter-extract the hexane phase with water. Concentrate pooled aqueous phases to dryness, dilute with acetic acid.	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on one or two silica gel cartridges. Carbowax column, GC/FPD LOQ 0.05 mg/kg for each analyte.
AL35/88	Bovine tissues	H_2O (stirring 30 min)	Add acetone, centrifuge, concentrate, clean-up on an anion exchange column, elute using formic acid, concentrate to dryness, dilute with acetic acid	Glufosinate, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on one or two silica gel cartridges. Carbowax or RTX-2330 column, GC/FPD LOQ 0.05 mg/kg muscle (raw/cooked) 0.1 mg/kg for diaphragm, kidney, liver for each analyte.
BK/03/95	Animal tissues, milk, eggs	Milk and fat: n-isopropanol/H_2O 1/1 v/v (stirring 30 min) Eggs, muscle, liver, kidney: H_2O (stirring 30 min)	Centrifuge, add acetone, centrifuge, concentrate to an aqueous residue, clean-up on an anion exchange column, wash with water, elute with formic acid, concentrate to dryness, dilute in acetic acid	Glufosinate/NAG, MPP: derivatise (trimethyl orthoacetate, 4 h reflux). Clean-up on SPE silica gel cartridge or packed silica gel columns. DB-Wax column, GC/FPD LOQ 0.02 mg/kg milk LOQ 0.05 mg/kg eggs, muscle, fat 0.1 mg/kg liver, kidney, all expressed as glufosinate. Note: method does not distinguish between glufosinate and NAG

Plant materials

A method suitable for enforcement purposes was developed by Rosati (2011a MR-10-173) and subjected to independent laboratory evaluation (Stuke 2011a MR 11/078). Method 01188 allows separate determination of glufosinate, MPP and NAG in plant matrices but requires two different procedures in which glufosinate is determined after derivatisation while MPP and NAG are determined directly. The residues of glufosinate, MPP and NAG are extracted by stirring the homogenised sample with distilled water for about 30 minutes at room temperature. A first aliquot of

the centrifuged extract is used for the determination of glufosinate. After addition of a stable isotope internal standard the extract is purified by ultrafiltration and reacted with a mixture of o-phthalic dialdehyde and mercapto propionic acid in the presence of sodium borate. The obtained derivative is measured by LC-MS/MS on a C18 column using the ion transition m/z 386 → 252 for quantification and the ion transitions m/z 386 → 172 or m/z 386 → 280 for confirmation. A second aliquot of the centrifuged extract is used for the determination of MPP and NAG. After addition of stable isotope internal standards the extract is shaken for 15 minutes with a cationic resin, centrifuged, concentrated to dryness and reconstituted in a mixture of acetonitrile and ammonium acetate buffer before analysis by means of LC-MS/MS using a hydrophilic interaction liquid chromatography (HILIC) column. MPP is determined using the ion transition m/z 153 → 135 for quantification and the ion transitions m/z 153 → 79 or m/z 153 → 97 for confirmation. NAG is determined using the ion transition m/z 224 → 118 for quantification and the ion transitions m/z 224 → 136 or m/z 224 → 164 for confirmation.

Quantification is conducted using stable isotope internal standards. The LOQ for each glufosinate, MPP and NAG was 0.01 mg/kg in lettuce (matrix with water content), orange (acidic matrix), potato (matrix with high starch content), dry bean (matrix with high protein content) and sunflower seed (oily matrix). Recovery data are summarised in Table 59.

Table 59 Enforcement method for the determination of glufosinate-derived residues in plant matrices – overview of recovery data

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg)	n	range	mean	RSD	Reference
Lettuce	glufosinate	0.01	5	75–92	81	8.4	Rosati 2011a
		0.10	5	93–99	96	2.5	MR-10-173
	MPP	0.01	5	91–100	96	3.5	
		0.10	5	83–90	88	3.3	
	NAG	0.01	5	102–113	108	38	
		0.10	5	96–99	97	1.3	
Orange	glufosinate	0.01	5	90–99	94	3.6	
		0.10	5	91–95	93	2.0	
	MPP	0.01	5	72–94	83	12.1	
		0.10	5	78–86	82	4.5	
	NAG	0.01	5	97–107	102	3.9	
		0.10	5	95–101	98	2.7	
Potato	glufosinate	0.01	5	88–98	94	4.6	
		0.10	5	93–96	94	1.4	
	MPP	0.01	5	80–88	84	4.1	
		0.10	5	78–92	87	6.8	
	NAG	0.01	5	90–108	99	6.8	
		0.10	5	92–99	94	2.9	
Kidney bean	glufosinate	0.01	5	87–98	91	5.0	
(dry)		0.10	5	96–104	100	3.0	
	MPP	0.01	5	85–100	91	6.7	
		0.10	5	88–98	93	3.9	
	NAG	0.01	5	92–108	100	6.0	
		0.10	5	97–100	99	1.1	
Sunflower	glufosinate	0.01	5	104–117	110	5.3	
seed		0.10	5	99–106	103	2.9	
	MPP	0.01	5	87–117	98	12.2	
		0.10	5	92–95	93	1.8	
	NAG	0.01	5	100–104	102	1.5	
		0.10	5	97–101	99	1.5	
Lettuce	glufosinate	0.01	5	78–84	81	2.8	Method
		0.10	5	73–74	73	0.7	01188 (ILV)
	MPP	0.01	5	97–105	100	3.4	Stuke, 2011a
		0.10	5	95–99	97	1.6	MR-11/078
	NAG	0.01	5	97–109	103	5.1	
		0.10	5	97–99	98	1.0	
Potato	glufosinate	0.01	5	89–101	95	5.2	
		0.10	5	87–98	91	4.5	

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg)	n	range	mean	RSD	Reference
	MPP	0.01	5	92–101	97	3.3	
		0.10	5	96–104	100	3.1	
	NAG	0.01	5	104–107	106	1.2	
		0.10	5	92–101	96	4.0	
Sunflower	glufosinate	0.01	5	98–110	105	4.3	
seed		0.10	5	96–102	99	2.9	
	MPP	0.01	5	95–99	97	2.1	
		0.10	5	96–101	99	2.1	
	NAG	0.01	5	84–97	92	5.7	
		0.10	5	93–100	97	3.1	

In Rosati (2011) the fortification levels are expressed as glufosinate equivalents. For the ILV they expressed fortification levels in terms of glufosinate, MPP and NAG. The ILV fortification levels expressed as glufosinate were 0.009 and 0.09 mg/kg for glufosinate, 0.012 and 0.12 mg/kg for MPP and 0.007 mg/kg and 0.07 mg/kg for NAG.

The above recovery-rates were obtained with the quantification ion transitions.

Animal matrices

Rosati (2011b MR 11/071) also developed a method suitable for enforcement, method 01199 which also underwent independent laboratory validation (Stuke 2011b MR 11/089). The method allows separate determination of glufosinate, MPP and NAG in animal matrices. It is based on the same principle as the method 01188 for plant matrices. The residues of glufosinate, MPP and NAG are extracted from muscle, offal, eggs and milk by stirring the homogenised sample with distilled water for about 30 minutes at room temperature. In the case of fat, samples are dissolved in ethyl acetate followed by liquid/liquid partitioning in water. In all cases a first aliquot of the centrifuged aqueous extract is used for the determination of glufosinate. After addition of a stable isotope internal standard the extract is filtered and reacted with a mixture of o-phthalic dialdehyde and mercapto propionic acid in the presence of sodium borate. The obtained derivative is measured by LC-MS/MS on a C18 column using the ion transition m/z 386 → 252 for quantification and the ion transitions m/z 386 → 172 or m/z 386 → 280 for confirmation. A second aliquot of the centrifuged aqueous extract is used for the determination of MPP and NAG. After addition of stable isotope internal standards the extract is shaken for 15 minutes with a cationic resin, centrifuged, concentrated to dryness and reconstituted in a mixture of acetonitrile and ammonium acetate buffer before analysis by means of LC-MS/MS using a hydrophilic interaction liquid chromatography (HILIC) column. MPP is determined using the ion transition m/z 153 → 135 for quantification and the ion transitions m/z 153 → 79 or m/z 153 → 97 for confirmation. NAG is determined using the ion transition m/z 224 → 136 for quantification and the ion transitions m/z 224 → 118 or m/z 224 → 164 for confirmation.

Quantification is conducted using stable isotope internal standards. The LOQ for glufosinate, MPP and NAG was established at 0.01 mg/kg for each analyte in beef muscle, beef liver, beef fat, whole milk and eggs. Recovery data are summarised in Table 60.

Table 60 Enforcement method for the determination of glufosinate-derived residues in animal matrices-overview of recovery data

		Fortification		Recovery (%) [b]			
Matrix	Analyte	level (mg/kg)	n	range	mean	RSD	Reference
Beef muscle	Glufosinate	0.01	5	109–114	112	1.8	Method
		0.10	5	107–118	112	4.3	01199
	MPP	0.01	5	105–115	109	4.0	Rosati, 2011
		0.10	5	94–98	96	1.5	MR-11/071
	NAG	0.01	5	87–104	98	6.7	
		0.10	5	99–102	101	1.3	
Beef liver	Glufosinate	0.01	5	99–102	100	1.3	
		0.10	5	95–100	98	2.0	
	MPP	0.01	5	83–96	88	5.7	
		0.10	5	86–88	87	0.8	

		Fortification		Recovery (%) [b]			
Matrix	Analyte	level (mg/kg)	n	range	mean	RSD	Reference
	NAG	0.01	5	86–96	91	4.1	
		0.10	5	92–98	94	3.1	
Beef fat	Glufosinate	0.01	5	84–94	90	5.4	
		0.10	5	88–94	91	3.0	
	MPP	0.01	5	85–99	92	6.2	
		0.10	5	88–99	93	4.5	
	NAG	0.01	5	88–96	93	3.6	
		0.10	5	89–95	92	2.5	
Whole milk	Glufosinate	0.01	5	87–105	97	8.8	
		0.10	5	98–113	106	5.6	
	MPP	0.01	5	81–93	85	5.9	
		0.10	5	83–92	89	4.0	
	NAG	0.01	5	86–101	94	6.6	
		0.10	5	91–96	94	1.9	
Whole egg	Glufosinate	0.01	5	89–98	93	3.6	
		0.10	5	88–95	91	2.8	
	MPP	0.01	5	94–115	101	8.2	
		0.10	5	93–98	96	2.2	
	NAG	0.01	5	85–113	99	11.4	
		0.10	5	91–98	94	3.3	
Beef muscle	Glufosinate	0.01	5	91–96	93	2.3	Method
		0.10	5	93–97	94	2.0	01199 (ILV)
	MPP	0.01	5	92–96	94	1.8	Stuke 2011b
		0.10	5	94–97	95	1.3	MR-11/089
	NAG	0.01	5	84–90	87	2.6	[a]
		0.10	5	92–98	96	3.0	
Beef fat	Glufosinate	0.01	5	91–95	92	2.4	
		0.10	5	89–94	90	2.8	
	MPP	0.01	5	86–94	90	4.5	
		0.10	5	88–93	89	2.9	
	NAG	0.01	5	84–89	86	2.4	
		0.10	5	83–89	88	4.2	
Whole milk	Glufosinate	0.01	5	92–104	98	4.6	
		0.10	5	83–90	88	3.5	
	MPP	0.01	5	96–102	98	2.8	
		0.10	5	95–97	96	1.5	
	NAG	0.01	5	89–113	98	11.4	
		0.10	5	93–97	95	1.7	

[a] In Rosati (2011b) the fortification levels are expressed as glufosinate equivalents. For the ILV fortification levels were expressed in terms of glufosinate, MPP and NAG. The actual fortification levels expressed as glufosinate were 0.009 and 0.09 mg/kg, 0.012 and 0.12 mg/kg for MPP and 0.007 and 0.07 mg/kg for NAG.

[b] The above recovery-rates were obtained with the quantification ion transitions.

Specialised methods

A variety of methods were used for determination of glufosinate and related compounds in residue trials. Methods were modified and adapted as technology and experience allowed.

Plants

Kuenzler (1982a A24283, 1982b A24976) developed methods for measuring glufosinate and MPP residues in plant commodities (Method AL43/82 and AL65/82). The residues of glufosinate and MPP are extracted by reflux in water for 8 hours using a Soxhlet apparatus (Extract A). For glufosinate, an aliquot of extract A is mixed with an equal volume of ethanol and precipitates removed by filtration. The extract is passed through a cation exchange column, the column rinsed with a mixture of water and ethanol (50/50, v/v) and residues of glufosinate eluted with water. The eluate is concentrated to dryness, diluted with acetic acid and reacted for two hours under reflux with trimethyl orthoacetate.

The derivative AE F064706 is determined by GC/FPD using a packed column (1.5% OV 275 on Chromosorb W-DMCS).

For MPP, an aliquot of extract A is passed over an anion exchange column and residues of MPP eluted with 10% acetic acid. The eluate is concentrated to dryness, diluted with acetic acid and reacted for two hours under reflux with trimethyl orthoacetate. After clean-up by gel permeation chromatography, the derivative AE F070951 is determined by GC/FPD using a packed column (1.5% OV 275 on Chromosorb W-DMCS). Quantification for glufosinate and MPP is performed against matrix-matched standards. The limit of quantification is 0.02 mg/kg for each compound.

In a minor modification the reflux extraction was replaced by dialysis (Methods 16/83 and 17/83, Kuenzler 1984a A28287, 1984b A28283). Subsequently, methods AL16/83 and AL17/83 were simplified by replacing the ion exchange purification steps before derivatisation by a silica gel clean-up after derivatisation (Method AL38/85 Kuenzler 1985 A32892, Specht 1986 A33483). This allowed a common sample preparation for determination of both glufosinate and MPP.

Further simplification was achieved by replacing the dialysis extraction by stirring with water for 30 minutes (method AL11/87, Idstein 1987a A35793, 1987b A35795, Sochor 1987a A37220, Specht 1991a A46285, 1991b A46332). The procedures for methods AL11/87, AL9/87 have been published as part of the method DFG 651. Method AL9/87 is identical to the method AL11/87 except for an additional clean-up step of the aqueous extract with acetone (Idstein 1987c A35794). Method AL24/87 is a further variant the method AL11/87 and includes an additional hydrolysis step of the aqueous extract with concentrated hydrochloric acid (Idstein 1987d A36894).

A separate method for determination of glufosinate and MPP residues in plant matrices with high oil content was developed by Sochor (1987b A44130) and modifies method AL11/87 by inclusion of a de-fatting step with dichloromethane. A more general method for fats and oils was developed by Sochor (1987c A38447) in which the sample is dissolved in dichloromethane and the residues of glufosinate and MPP are extracted in water by liquid/liquid partitioning in water (method AL40/87). The aqueous phase is isolated by centrifugation and an aliquot thereof is cleaned up on an RP18 cartridge.

The various analytical methods developed in the late 1980s for the determination of glufosinate-derived residues in plant and livestock matrices were published in 1991 as the method DFG 651 (Sochor 1991 A48915). This method DFG 651 includes procedures for different types of plant matrices:

- The procedure for plant material with high water content corresponds to the method AL11/87
- The procedure for plant material with high content of water soluble carbohydrates or proteins corresponds to the method AL 9/87
- The procedure for fatty plant material corresponds to the method AL37/87
- The procedure for fats and oils corresponds to the method AL40/87.

Radiovalidation of the residue analytical method AL11/87 was carried out as part of a corn metabolism study by Stumpf (1989a A41451). Radioassay determined an MPP concentration of 0.069 mg eq/kg in corn fodder. Using the residue method AL11/87, residues of MPP in corn fodder were 0.060 mg/kg or 0.065 mg eq/kg when expressed in equivalents of glufosinate ammonium, in good agreement with value determined using radioassay.

The method HRAV-5A was developed in the USA for the determination of glufosinate and MPP residues in plant matrices (Czarnecki 1989 A40790, Meikle 1991a A54163, 1991b A54164). The method is similar to AL 11/87 but includes an additional purification step on an anion exchange column prior to derivatisation.

Allan (1996 A58109) validated the primary extraction step (extraction with water) of method HRAV-5A by comparison of the quantity of extracted residues with those exhaustively extracted with water/methanol (4 ×) within a metabolism trial. The residue components extracted from leaves and beets according to HRAV-5A with water represented 98% of the TRR. The sum of glufosinate, MPP

and NAG was almost identical with the TRR. It is concluded that the extraction used in method HRAV-5A recovers almost the complete incurred glufosinate residues from glufosinate-tolerant sugar beets.

As glufosinate-tolerant crops are able to inactivate glufosinate by acetylating it to NAG which is usually the major residue component, methods were developed to also determine NAG, either measured together with glufosinate as a common derivative or separately. Method AE-24 determines glufosinate, NAG and MPP compounds individually (Czarnecki 1993 A52270, Czarnecki 1994 A54051, Norby 1994 A53397, Bertrand 1994 A53623, Castro 1994 A53696, Holzwarth 1996a A56405, 1996b A56405). For most matrices the residues of glufosinate, MPP and NAG are extracted by stirring the homogenised sample with distilled water for about 30 minutes at room temperature. A filtered aliquot of the aqueous extract is purified by means of an anion exchange column. Once the column has been washed with water, the compounds of interest are eluted with formic acid. The eluate is evaporated to dryness, reconstituted in ethanol / water (50/50, v/v) and loaded on a cation exchange column. The two metabolites NAG and MPP are eluted with an ethanol / water mixture (fraction A), whereas glufosinate is eluted using an ethanolic ammonia solution (fraction B). Each of the two fractions is concentrated to dryness, re-dissolved in acetic acid and reacted with trimethyl orthoacetate. Using this procedure glufosinate and NAG are converted to AE F064706 while MPP is converted to AE F070951. Thereafter, the two fractions are cleaned up on an SPE silica gel cartridge and analysed by GC/FPD. Although glufosinate and NAG are converted to the same derivative AE F064706, they can be determined separately since they are eluted in different fractions on the cation exchange column.

A study was conducted to investigate possible interference from other pesticides with the main pesticides registered for use in maize or soya bean considered. Based on their molecular structure, 69 compounds were not thought to be likely to present an interference problem. The study focussed on the remaining 16 compounds for which it was deemed necessary to generate additional experimental data. Untreated maize forage was fortified with these 16 compounds, either singly or in combination with glufosinate, MPP and NAG. The fortified samples were analysed according to the method AE-24. No peak interference was found at or near the retention times of the derivatives AE F064706 and AE F070951. Moreover the presence of other pesticides did not impact the detector response for the derivatives AE F064706 and AE F070951. Therefore, none of the 16 pesticides tested was found to interfere in any way with the determination of glufosinate-derived residues according to the method AE-24.

As it is sometimes acceptable to determine glufosinate and NAG together their separation is not always required and a modification of AE-24 was developed (Czarnecki 1995a A54323, 1995b C005657, 1995c A56922, Niedzwiadek 1995 A55259, Snowdon 1995 A55211, Werner 1998 C000939). In the modification (AE-24A also BK/04/95 and BK/05/95) the cation exchange step is omitted and glufosinate and NAG are quantified as a sum of the two compounds making the procedure more straightforward and less time consuming. This simplified version of the method AE-24 is very similar to the method HRAV-5A for the determination of glufosinate and MPP in conventional crops.

In a soya bean metabolism study the results from radio-assay were compared with those using method AE-24 (Czarnecki 1994 A54051). Residue levels of glufosinate, MPP and NAG were determined using method AE-24 (GLC) with results calculated on the basis of glufosinate equivalents to enable direct comparison to residue levels determined by radio-HPLC. The sum of glufosinate, MPP and NAG in the aqueous extracts as determined by method AE-24 were in good agreement (81.1–103.2%) with those quantified by radio-HPLC (Table 61).

Table 61 Accountability of the aqueous extractable glufosinate residues by the analytical method AE-24 (Czarnecki 1994 A54051)

Matrix	Residue (mg eq/kg)		Accountability of AE-24 to
	Radio-HPLC Method [a]	Method AE-24 [b]	Radio-HPLC (%)
Forage	1.427	1.414	99.1
Straw	2.523	2.436	95.6

Matrix	Residue (mg eq/kg)		Accountability of AE-24 to
	Radio-HPLC Method [a]	Method AE-24 [b]	Radio-HPLC (%)
Pods	4.148	4.280	103.2
Beans	0.947	0.768	81.1

[a] By radioactivity counting, the radio-HPLC fractions corresponding to glufosinate, NAG and MPP were added.

[b] By standard residue analytical method AE-24, the sum of GC recoveries corresponding to glufosinate, NAG and MPP were added (Czarnecki and Bertrand, 1994, M-134651-01-1).

In the case of straw the residue levels for MPP determined by AE-24 were only in moderate agreement with those determined by radio-HPLC determination (Table 62). However, the total residues (sum of glufosinate, NAG and MPP) were in good agreement (95.6%).

Table 62 Accountability of the aqueous glufosinate residues extractable from straw by the standard residue analytical method AE-24 (Czarnecki 1994 A54051)

Residue component	Residue (mg eq/kg)		Accountability of AE-24 to
	Radio-HPLC Method	Radio-HPLC(%)	Radio-HPLC (%)
glufosinate	3.01	2.73	90.7
MPP	2.03	1.34	66.0
NAG	7.35	7.78	105.9
Total	12.39	11.85	95.6

Laporte (2001 C013029) reported an additional radio-validation study using straw from glufosinate-tolerant rape harvested from a crop treated with [3,4-^{14}C] glufosinate-ammonium. The purpose was to determine the extraction efficiency of the methods AE-24 and BK/05/95. Three straw samples were analysed according to the method AE-24 and three according to the method BK/05/95. In both methods, the residues are extracted by stirring the homogenised sample with distilled water and determined by GC/FPD after derivatisation with trimethyl orthoacetate. The aqueous extract is purified by means of an anion exchange column and the derivatised extract by means of a silica gel SPE cartridge. The method AE-24 also includes a separation step on a cation exchange column prior to derivatisation. After the extraction step with distilled water the radioactivity was determined in the aqueous extract and in the filter cake. The combined radioactivity in the aqueous extract and in the filter cake was defined as the TRR. The extraction efficiency was defined as the ratio between the radioactivity in the aqueous extract and the TRR. Similarly, the overall efficiency of the method was defined as the ratio between the radioactivity in the final extract and the TRR. Besides the radioactivity measurements, the final extracts were also analysed by means of the classical GC/FPD technique. The extraction efficiency was found to be 94.5 % for the method AE-24 and 96.1% for the method BK/05/95 (means of three repeats). The extraction efficiency of water compares very well with the efficiency of 96.7% determined in the metabolism study where the rape straw had been extracted with water/methanol (9:1, v/v) (Buerkle and Slezer 2001). The overall efficiency was 75.1 % for the method AE-24 and 80.9% for the method BK/05/95 (means of three repeats).

Sochor (1998 C002130) described some modifications to method DFG 651 to demonstrate the method is capable of determining the metabolite NAG in addition to glufosinate and MPP. The revised version of the method DFG 651 was validated for the determination of glufosinate-derived residues in tolerant crops, meat, milk, and eggs. The procedure for plant commodities with high water content (e.g. maize forage), high content of carbohydrate (e.g. maize grain) and oily plant commodities (e.g. rape seed) is the same as in the original method DFG 651. The new procedure was introduced for commodities with high sugar content. It includes an anion exchange purification step as in method AE-24A or BK/05/95. Calibration and quantification are performed using standard solutions prepared in the solvent (methyl acetate). During the initial method validation the LOQ (expressed as glufosinate) was established at 0.05 mg/kg for the combined residues of glufosinate and NAG, and at 0.05 mg/kg for MPP in all the tested matrices. An independent laboratory validation (ILV) was conducted but it was only partly successful (Leeson 2001 C012971). For some analytes and matrices up to three batches were necessary to obtain satisfactory results while for some analytes and matrices even the third batch did not fulfil the guideline validation criteria. It may be concluded

that the (modified) method DFG 651 requires experience since small details of the procedure may have important effects on the results.

To improve the robustness and sensitivity of the method DFG 651, GC/FPD was replaced with LC-MS/MS (Anspach 2003 C035882, 2006 00915/M001, 2009 C036792) and designated method 00852 when used to analyse plant matrices with high water content and method 00915 or 00915/M001 when used for the analysis of potato tubers and processed potato commodities. The derivatives AE F064706 and AE F070951 are determined by LC-MS/MS using a C18 column. The derivative AE F064706 is quantified using the ion transition m/z 252 → 150 while AE F070951 is quantified using the ion transition m/z 181 → 93. The transitions m/z 252 → 210 and m/z 181 → 149, respectively, can be used for confirmatory analysis. An additional anion exchange purification step prior to derivatisation is used if necessary to improve the sensitivity of the determination (e.g. onion, potato crisps or flakes).

For analysis of oil, the oil sample is dissolved in dichloromethane. Thereafter the residues of glufosinate and MPP are extracted in water by liquid/liquid partitioning in water. The aqueous phase is isolated by centrifugation and an aliquot is cleaned up on an RP18 cartridge, concentrated to dryness, before derivatisation, silica gel clean-up and LC-MS/MS quantification.

Quantification is performed using standard solutions prepared in water/methanol (9/1, v/v). When the procedure was first validated the LOQ (expressed as glufosinate) was established at 0.05 mg/kg for each of glufosinate and MPP in all of the investigated matrices. Later on, when the anion exchange purification step was systematically applied to all matrices, this LOQ was lowered to 0.01 mg/kg.

The method GL-001-P07-01 (also 01109) allows determination of glufosinate, MPP and NAG in plant matrices without derivatisation (Murphy 2007a GL 001 P07 01, Rosati 2011c 01109). The residues are extracted by stirring the homogenised sample in water for 30 minutes. After addition of stable isotope internal standards, an aliquot of the extract is mixed with acetone, centrifuged to remove precipitates, concentrated to dryness, reconstituted in water/methanol (95/5, v/v) and purified by means of a SAX cartridge using water/methanol (1/1, v/v) as the eluant. The eluate is concentrated to dryness and reconstituted in a mixture of acetonitrile and ammonium acetate buffer for quantification by means of LC-MS/MS using a hydrophilic interaction liquid chromatography (HILIC) column. Glufosinate, MPP and NAG are quantified using the ion transitions m/z 182 → 136, m/z 153 → 135 (or m/z 153 → 79) and m/z 224 → 118, respectively. For each analyte at least one additional ion transition may be used for confirmatory analysis. Quantification is conducted using stable isotope internal standards. LOQs for glufosinate, MPP and NAG were 0.01 mg/kg in lettuce, black currant, kidney bean and sunflower seed and 0.02 mg/kg in potato tuber.

Table 63 Method used in supervised trials for the determination of glufosinate-derived residues in plant matrices—overview of recovery data

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg) [a]	n	range	mean	RSD	reference
Soya bean	glufosinate	0.02	1		56		Method AL16/83
seed		0.05	1		65		Thier, 1985 A30727
		0.10	1		71		
	MPP	0.02	1		65		
		0.05	1		91		
		0.10	1		85		
Potato	glufosinate	0.5	3	81–88	84	4	Method AL38/85
	MPP	0.6	3	73–81	76	6	Specht, 1986 A33483
French bean	glufosinate	0.5	3	72–75	73	2	
(plant)	MPP	0.6	3	75–83	80	5	
French bean	glufosinate	0.5	3	68–82	74	10	
(bean)	MPP	0.6	3	71–80	75	6	
French bean	glufosinate	0.5	3	80–87	83	4	
(straw)	MPP	0.6	3	78–90	83	8	
French bean	glufosinate	0.5	3	71–87	80	10	
(husks)	MPP	0.6	3	76–93	83	10	

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg) [a]	n	range	mean	RSD	reference
French bean	glufosinate	0.08	3	73–88	84	6	
(seed)		0.5	3	81–90	81	9	
	MPP	0.09	3	74–89	79	6	
		0.6	3	74–82	84	10	
Pea (husks)	glufosinate	0.5	3	68–76	71	6	
		2.5	3	80–87	82	5	
	MPP	0.6	3	90–92	91	1	
		3.0	3	81–92	86	6	
Pea (seed)	glufosinate	0.5	3	76–80	78	3	
	MPP	0.6	3	74–81	78	5	
Corn (plant)	glufosinate	0.5	3	70–81	77	8	
	MPP	0.6	3	76–82	79	4	
Corn (cob)	glufosinate	0.5	3	67–75	73	5	
	MPP	0.6	3	75–79	77	3	
Corn (grain)	glufosinate	0.5	3	77–79	78	1	
	MPP	0.6	3	74–78	76	3	
Broad bean	glufosinate	0.5	3	79–87	82	5	
(bean)		1.5	3	82–88	85	4	
	MPP	0.6	3	69–81	76	8	
		1.8	3	79–82	80	2	
Broad bean	glufosinate	1.0	3	76–80	78	3	
(husk)	MPP	1.5	3	79–84	82	3	
Broad bean	glufosinate	0.5	3	72–83	79	7	
(seed)	MPP	0.6	3	78–86	81	5	
Sunflower	glufosinate	0.5	3	82–90	86	5	
(plant)	MPP	0.6	3	85–87	86	1	
Sunflower	glufosinate	0.5	3	70–83	78	9	
(seed)	MPP	0.6	3	76–82	78	4	
Coffee beans	glufosinate	0.05	2	99–101	100		Method AL11/87
		0.10	1	105			Specht, 1991c A46284
		0.5	1	75			
	MPP	0.05	2	102–103	103		
		0.10	1	110			
		0.5	1	93			
Coffee	glufosinate	0.05	2	94–107	101		
(roasted)		0.10	1	94			
		0.5	1	76			
	MPP	0.05	2	92–116	104		
		0.10	1	88			
		0.5	1	88			
Coffee	glufosinate	0.05	2	110–110	110		
(instant)		0.10	2	77–108	93		
		0.5	2	74–78	76		
	MPP	0.05	2	93–95	94		
		0.10	2	73–111	92		
		0.5	2	73–77	75		
Potato tubers	glufosinate	0.05	2	85–96	90		Method AL11/87
		0.5	2	112–113	113		Specht, 1991a A46285
		5	2	91–92	91		
	MPP	0.05	2	87–110	99		
		0.5	2	94–101	97		
		5	2	80–96	88		
Brown rice	glufosinate	0.05	2	70–72	71		Method AL11/87
		0.5	2	99–110	105		Specht, 1991b A46332
		0.05	2	77–88	79		
		0.5	2	95–98	96		
Polished rice	glufosinate	0.05	2	82–114	98		
		0.2	2	94–99	96		
	MPP	0.05	2	88–106	97		
		0.2	2	107–119	113		

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg) [a]	n	range	mean	RSD	reference
Rice husk	glufosinate	0.05	2	112–124	118		
		0.2	2	83–90	86		
		0.5	2	96–116	106		
	MPP	0.05	2	104–128	116		
		0.2	2	84–86	85		
		0.5	2	101–103	102		
Rice bran	glufosinate	0.05	2	78–80	79		
		0.2	2	86–104	95		
		1	2	17–119	103		
	MPP	0.05	2	80–96	88		
		0.2	2	93–97	95		
		1	2	91–103	97		
Rapeseed	glufosinate	0.05	2	98–103	101		Method AL37/87
		0.1	2	94–114	104		Sochor 1987b A44130
		0.2	2	95–101	98		
	MPP	0.05	2	101–121	113		
		0.1	2	117–136	127		
		0.2	2	112–118	115		
Oenothera	glufosinate	0.05	1	84			Method AL40/87
oil		0.1	2	52–103	77		Sochor 1987c A38447
	MPP	0.05	1	70			
		0.1	2	63–69	61		
Apple	glufosinate	0.05	5	80–107	97	11	Method DFG 651 (similar to
		0.25	3	79–98	88	11	method AL11/87)
	MPP	0.05	5	82–112	97	11	Tillkes 1997 A58624
		0.25	3	83–127	99	25	
Peach	glufosinate	0.05	2	69–75	72		
		0.25	1	77			
	MPP	0.05	2	73–82	77		
		0.25	1	86			
Cherry	glufosinate	0.05	4	69–89	77	12	
		0.25	2	78–97	87		
	MPP	0.05	4	80–93	88	7	
		0.25	2	85–92	88		
Plum	glufosinate	0.05	1	78			
	MPP	0.05	1	110			
Grape	glufosinate	0.05	7	71–110	87	16	
(berries)		0.25	5	74–107	90	16	
	MPP	0.05	7	72–92	84	9	
		0.25	5	74–103	86	13	
Grape must	glufosinate	0.05	2	71–96	84		
	MPP	0.05	2	67–90	79		
Grape	glufosinate	0.05	2	70–98	84		
pomace	MPP	0.05	2	76–82	79		
Grape wine	glufosinate	0.05	2	83–91	87		
	MPP	0.05	2	74–77	76		
Apple	glufosinate	0.05–0.2	5		93	10	Method HRAV-5A
	MPP	0.05–0.2	5		84	6	Czarnecki 1989 A40790
Grapes	glufosinate	0.05–0.2	12		90	9	
	MPP	0.05–0.2	12		78	5	
Soya bean	glufosinate	0.05–0.2	12		97	7	
seed	MPP	0.05–0.2	12		97	7	
Maize grain	glufosinate	0.05–0.2	6		98	6	
	MPP	0.05–0.2	6		94	6	
Maize fodder	glufosinate	0.05–0.2	3		82	5	
	MPP	0.05–0.2	3		98	4	
Maize forage	glufosinate	0.05–0.2	4		85	2	
	MPP	0.05–0.2	4		85	4	
Almond	glufosinate	0.05–0.2	9		81	9	
nutmeat	MPP	0.05–0.2	9		88	10	

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg) [a]	n	range	mean	RSD	reference
Walnut meat	glufosinate	0.05–0.2	7		91	10	
	MPP	0.05–0.2	7		90	8	
Pecan	glufosinate	0.05–0.2	18		94	6	
nutmeat	MPP	0.05–0.2	18		98	10	
Citrus fruit	glufosinate	0.05	3	83–92	87	5	Method HRAV-5A
		0.1	3	78–90	86	8	Meikle 1991a A54163
		0.2	3	77–91	84	8	
	MPP	0.05	3	72–79	75	5	
		0.1	3	78–80	79	1	
		0.2	3	70–79	75	6	
Molasses	glufosinate	0.05	2	85–89	87		
		0.1	2	91–92	91		
		0.2	2	89–90	89		
	MPP	0.05	2	88–88	88		
		0.1	2	77–80	78		
		0.2	2	73–82	77		
Citrus oil	glufosinate	0.05	2	76–82	79		
		0.1	2	71–90	80		
		0.2	2	66–81	73		
	MPP	0.05	2	89–101	95		
		0.1	2	87–93	90		
		0.2	2	77–85	81		
Kiwifruit	glufosinate	0.05	3	88–102	97	8	Method HRAV-5A
		0.1	3	89–94	91	3	Meikle 1991b A54164
		0.2	3	95–100	97	3	
	MPP	0.05	3	82–97	88	9	
		0.1	3	83–92	88	5	
		0.2	3	77–96	84	12	

[a] The fortification levels are expressed as glufosinate.

Animal matrices

The method AL2/87 was developed for the determination of glufosinate and MPP residues in milk (Idstein 1987e A35957) and was used to analyse samples from a cow feeding study. Residues of glufosinate and MPP are extracted from milk by dialysis against water for 24 h and an aliquot of the extract concentrated to dryness, diluted with acetic acid and reacted for 4 hours under reflux with trimethyl orthoacetate. The reaction mixture is cleaned up using an RP 18 cartridge followed by a silica gel cartridge, with the resulting derivatives AE F064706 and AE F070951 eluting in separate fractions. Each fraction is analysed by GC/FPD on a Carbowax packed column using different GC parameters.

Calibration and quantification are performed using standard solutions prepared in the solvent (methyl acetate). The LOQ is 0.02 mg/kg for glufosinate and MPP (expressed as glufosinate).

In a method developed for analysis of eggs (AL32/88, Schuld 1988 A39229), samples of homogenised egg are first mixed with ethanol to denature proteins. Residues of glufosinate and MPP are extracted in water by stirring for 30 minutes. After centrifugation, an aliquot of the supernatant aqueous phase is cleaned up with dichloromethane, followed by n-hexane. The n-hexane phase is subsequently counter-extracted with water. The combined aqueous phases are concentrated to dryness, diluted with acetic acid and reacted for 4 hours under reflux with trimethyl orthoacetate. After one or two clean-up steps on a small silica gel column, the derivatives AE F064706 and AE F070951 are determined by GC/FPD on a Carbowax megabore capillary column. Calibration and quantification is performed using either standard solutions prepared in the solvent (methyl acetate) or matrix-matched standard solutions. The LOQ is 0.05 mg/kg for each glufosinate and MPP (expressed as glufosinate).

Sochor (1988 A39949) developed method AL35/88 for the determination of glufosinate and MPP residues in muscle and offal. Residues of glufosinate and MPP are extracted in water by stirring or shaking for 30 minutes at room temperature (except for blood samples, which are extracted at 60 °C). After centrifugation, an aliquot of the supernatant is mixed with acetone to precipitate proteins, centrifuged and an aliquot of the supernatant concentrated to an aqueous residue and cleaned-up on an anion exchange column. Residues were eluted with formic acid, concentrated to dryness, diluted with acetic acid and reacted for 4 hours under reflux with trimethyl orthoacetate. After one or two clean-up steps on a small silica gel column, the derivatives AE F064706 and AE F070951 are determined by GC/FPD on a Carbowax or RTX-2330 megabore capillary column. Calibration and quantification is performed using either standard solutions prepared in the solvent (methyl acetate) or matrix-matched standard solutions. The LOQ for glufosinate and its metabolite MPP (expressed as glufosinate) is 0.05 mg/kg in bovine muscle (raw or boiled), 0.06 mg/kg in bovine blood and 0.10 mg/kg in bovine diaphragm, kidney and liver.

Several analytical methods developed in the late 1980s for the determination of glufosinate-derived residues in plant and livestock matrices were published in 1991 as the method DFG 651. This method DFG 651 includes procedures for two types of animal matrices:

- The procedure for fats and oils corresponds to the method AL40/87.
- The procedure for meat and offal corresponds to the method AL35/88-0.

Czarnecki (1995d A54161, 1995e A53974) developed a method (BK/03/95) that determines glufosinate and its metabolites MPP and NAG in food commodities of animal origin. Initially, the methodology was issued under the method reference BK/01/95 and validated for glufosinate and MPP only but later was applied without modification for the determination of MPP and the combined residues of glufosinate and NAG. Calibration and quantification are performed using standard solutions prepared in the solvent (methyl acetate). The LOQ for the combined residues of glufosinate and NAG and for MPP was 0.02 mg/kg in milk, 0.05 mg/kg in eggs, muscle and fat, and 0.10 mg/kg in liver and kidney (expressed as glufosinate).

Sochor (1998 C002130) reported the extension and validation of the method DFG 651 revised to include determination of NAG while Leeson (2001 C012971) reported results of an independent laboratory validation study. Validation data were obtained for crops, meat, milk, and eggs. In general, calibration and quantification are performed using standard solutions prepared in the solvent (methyl acetate) but during the independent laboratory validation a matrix effect for AE F064706 was observed in milk. Thus, the determination of glufosinate and NAG residues in milk was performed using matrix-matched standards of AE F064706. During the initial method validation the LOQ (expressed as glufosinate) was established at 0.05 mg/kg for the combined residues of glufosinate and NAG, and at 0.05 mg/kg for MPP in all the tested matrices. Since the method was intended for enforcement purposes, an independent laboratory validation (ILV) was conducted but it was only partly successful. For some analytes and matrices up to three batches were necessary to obtain satisfactory results while for some analytes and matrices even the third batch did not fulfil the guideline validation criteria. It may be concluded that the (modified) method DFG 651 requires experience since small details of the procedure may have important effects on the results.

Table 64 Specialised methods for the determination of glufosinate-derived residues in animal matrices—overview of recovery data

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg) [a]	N	Range	Mean	RSD	reference
Beef fat	Glufosinate	0.05	8	71–98	86	12	Method AL40/87
		0.10	2	69–75	72		Sochor 1987c A38447
		0.20	1	–	71		
		0.25	2	73–80	76		
	MPP	0.10	2	59–98	78		
		0.20	1		74		
		0.25	2	85–94	89		
Eggs	Glufosinate	0.05	2	79–79	79		Method AL32/88

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg) [a]	N	Range	Mean	RSD	reference
		0.1	2	62–82	72		Schuld 1988 A39229
		0.2	2	80–89	84		
	MPP	0.05	2	93–98	95		
		0.1	2	84–103	93		
		0.2	2	79–100	89		
Bovine muscle	Glufosinate	0.05	2	62–88	75		Method AL35/88
(raw)		0.1	4	81–98	91	8	Sochor 1988 A39949
		0.2	2	85–98	97		
	MPP	0.05	2	83–90	86		
		0.1	4	63–116	92	26	
		0.2	2	107–120	114		
Bovine muscle	Glufosinate	0.1	4	95–119	109	10	
(boiled)		0.2	3	110–114	113	2	
	MPP	0.1	4	106–117	114	5	
		0.2	3	92–103	97	6	
Broth boiled	Glufosinate	0.05	3	92–108	102		
Bovine muscle		0.1	3	84–116	103	9	
		0.2	2	88–101	94		
	MPP	0.05	3	86–95	90	5	
		0.1	3	88–115	100	14	
		0.2	2	81–98	89		
Bovine blood	Glufosinate	0.06	1	95	95		
		0.13	1	117	117		
		0.25	1	113	113		
	MPP	0.06	1	91	91		
		0.13	1	114	114		
		0.25	1	104	104		
Bovine	Glufosinate	0.1	4	102–123	114	8	
diaphragm		0.2	4	89–111	97	10	
	MPP	0.1	4	86–115	99	12	
		0.2	4	91–124	109	13	
Bovine kidney	Glufosinate	0.1	3	76–92	82	10	
		0.2	2	90–112	101		
		1	1	111	111		
		5	1	95	95		
	MPP	0.1	3	93–120	103	15	
		0.2	2	88–98	93		
		1	1	96	96		
		5	1	60	60		
Milk	Glufosinate	0.02	3	94–104	98	5	Method BK/03/95
		0.1	3	78–90	83	8	Czarneki 1995e A53974
	MPP	0.02	3	78–95	86	10	
		0.1	3	84–86	85	1	
Eggs	Glufosinate/NAG	0.05	3	105–115	111	5	
		0.25	2	100–119	110		
	MPP	0.05	3	77–91	83	9	
		0.25	2	79–89	84		
Bovine muscle	Glufosinate/NAG	0.05	3	82–86	85		
		0.25	2	80–98	89		
	MPP	0.05	3	70–73	71		
		0.25	2	84–96	90		
Bovine fat	Glufosinate	0.05	3	87–99	94		
		0.25	3	84–93	88		
	MPP	0.05	3	74–77	75		
		0.25	3	73–75	74		

		Fortification		Recovery (%)			
Matrix	Analyte	level (mg/kg) [a]	N	Range	Mean	RSD	reference
Bovine liver	Glufosinate/NAG	0.1	3	97–103	100		
		0.5	2	99–100	99		
	MPP	0.1	3	93–101	97		
		0.5	2	88–89	88		
Bovine kidney	Glufosinate	0.1	2	105–113	109		
		0.5	4	93–107	98	6	
	MPP	0.1	2	110–110	110		
		0.5	4	95–108	101	5	
Poultry muscle	Glufosinate/NAG	0.05	3	95–103	99	4	
		0.25	2	111–113	112		
	MPP	0.05	3	88–95	92	4	
		0.25	2	99–101	100		
Poultry fat	Glufosinate/NAG	0.05	3	102–117	108	7	
		0.25	2	98–100	99		
	MPP	0.05	3	85–87	86	1	
		0.25	2	84–89	87		
Poultry liver	Glufosinate/NAG	0.1	3	83–92	87	5	
		0.5	3	90–94	92	2	
	MPP	0.1	3	86–110	96	13	
		0.5	3	99–103	101	2	
Poultry kidney	Glufosinate/NAG	0.05	3	81–89	86	5	
		0.25	3	82–88	85	4	
	MPP	0.05	3	80–99	90	11	
		0.25	3	95–101	98	3	
Eggs	Glufosinate	0.05	3	105–120	111	7	Method DFG 651
		0.5	3	98–106	101	4	Sochor 1998 C002130
	MPP	0.05	6	66–90	82	11	
		0.5	6	80–94	89	6	
	NAG	0.05	3	106–108	107	1	
		0.5	3	104–110	107	3	
Milk	Glufosinate	0.05	3	97–118	105	11	
		0.5	3	119–127	122	4	
	MPP	0.05	6	87–107	94	8	
		0.5	6	87–94	91	4	
	NAG	0.05	3	96–123	114	14	
		0.5	3	114–121	117	3	
Bovine meat	Glufosinate	0.05	5	43–59	55	12	Method DFG 651 (ILV)
		0.5	5	40–60	54	15	Leeson 2001 C012971
	MPP	0.05	5	76–81	79	3	
		0.5	5	72–80	77	4	
	NAG	0.05	5	72–85	80	5	
		0.5	5	52–74	67	14	
Milk	Glufosinate	0.05	5	60–74	69	8	
		0.5	5	39–58	53	15	
	MPP	0.05	5	49–70	62	14	
		0.5	5	33–62	53	22	
	NAG	0.05	5	65–83	75	9	
		0.5	5	57–76	71	11	
Eggs	Glufosinate	0.05	5	74–90	82	9	
		0.5	5	73–82	78	4	
	MPP	0.05	5	74–88	80	7	
		0.5	5	83–89	86	3	
	NAG	0.05	5	88–106	95	7	
		0.5	5	69–84	79	8	

[a] The fortification levels are expressed as glufosinate.

Stability of residues in stored analytical samples

The freezer storage stability of glufosinate, NAG and MPP in fortified plant, animal tissues, milk and eggs samples was studied. Residues were generally stable for the duration of the studies (Table 65).

Stability of residues in plant products

Apples, maize and soya beans

Idstein (1987 A36196, 1988 A39283) studied the storage stability of glufosinate ammonium and MPP in apple, maize grain and soya bean seed under frozen conditions. Homogenised samples of these commodities were fortified with glufosinate ammonium and MPP at three different fortification levels (0.05, 0.10 and 0.20 mg/kg, expressed as glufosinate) and analysed after about 0, 6/8, 8/8.5, 18 and 24 months of storage at about -20 °C. The analyses were conducted using method AL38/85 (0 month storage) or AL11/87 (6, 8, 18 and 24 month storage) with quantification by GC/FPD. Glufosinate and MPP are stable for at least 24 months in apple, maize grain, and soya bean seed.

Almonds and oranges

Sochor (1990a A43485) investigated the storage stability of glufosinate ammonium and MPP in almonds and oranges under frozen conditions. Homogenised samples of these commodities were fortified with ^{14}C-labelled glufosinate ammonium and MPP at 0.16 and 0.24 mg/kg, respectively (expressed as glufosinate), and analysed after about 0, 6, 12 and 24 months of storage at about -20 °C. The analyses used methods AL11/87 (for the orange samples) and AL9/87 Ba (for the almond samples) with quantification by GC/FPD. Glufosinate and MPP are stable for at least 12 months in almond and at least 24 months in orange.

Kiwifruit

A study by Meikle (1991c A46904) investigated the storage stability of glufosinate ammonium and MPP in kiwifruit under frozen conditions. Homogenised samples of kiwifruit were fortified with 0.2 mg/kg of glufosinate ammonium and MPP (expressed as glufosinate) and analysed after about 0, 3 and 6 months of storage at between -23 and -26 °C using method HRAV-5A with quantification by GC/FPD. Glufosinate ammonium and MPP are stable for at least 6 months in kiwifruit.

Blueberries

The storage stability of glufosinate ammonium and MPP in blueberries under frozen conditions was investigated during a residue study (Salzman 2002 05291). Homogenised samples of blueberry were fortified with 0.5 mg/kg of glufosinate ammonium and MPP (expressed as glufosinate) and analysed after about 20 months of storage at < -20 °C. The analyses were conducted according to the method HRAV-5A with quantification by GC/FPD. Glufosinate and MPP are stable for at least 20 months in blueberries.

Peaches

Samoil (2009 08720) studied the storage stability of glufosinate ammonium, NAG and MPP in peaches under frozen conditions as part of a residue study. Homogenised samples of peach were fortified with 0.5 mg/kg of glufosinate ammonium, NAG and MPP (expressed as glufosinate) and analysed after 24–26 months of storage at about -20 °C. The analyses utilised method BK/01/99 (specific module) with quantification by GC/FPD. Glufosinate, NAG and MPP are stable for at least 24 months in peaches.

Tolerant sugar beet root

A study by Werner (1997 A59404) investigated the storage stability of glufosinate ammonium, NAG and MPP in transgenic glufosinate-tolerant sugar beet root under frozen conditions. Homogenised samples of transgenic glufosinate-tolerant sugar beet root were fortified with 0.5 mg/kg of glufosinate ammonium and MPP or with 0.5 mg/kg of NAG and MPP (expressed as glufosinate). The fortified

samples were stored deep frozen and analysed after about 0, 1, 3, 6, 12, 18 and 24 months of storage. The analyses were by method AE-24A with quantification by GC/FPD. Glufosinate, NAG and MPP are stable for at least 24 months in glufosinate tolerant sugar beet root under frozen conditions. Since the analytical method used in the study does not differentiate between glufosinate and NAG it would not have been possible to detect a possible interconversion between glufosinate and NAG.

Tolerant sugar beet tops

Bertrand (1998 A67531) investigated the storage stability of glufosinate ammonium, NAG and MPP in glufosinate tolerant sugar beet tops and roots under frozen conditions. Homogenised samples of these commodities were fortified with 0.25 mg/kg of glufosinate ammonium and MPP or with 0.25 mg/kg of NAG and MPP (expressed as glufosinate). The fortified samples were stored at approximately -20 °C and analysed after 0, 3, 6, 12 and 24 months of storage. The analyses were by method BK/04/95 with quantification by GC/FPD. Glufosinate, NAG and MPP are stable for at least 24 months in glufosinate-tolerant sugar beet root under frozen conditions. Some of the stored samples were analysed according to the method AE-24 which allows separate determination of glufosinate and NAG. No interconversion between glufosinate and NAG was observed.

Tolerant sugar beet processed fractions

Wyatt (2002 B003971) studied the storage stability of glufosinate ammonium, NAG and MPP in glufosinate tolerant sugar beet pulp and molasses and in refined sugar under frozen conditions. Homogenised samples of these commodities were fortified with 0.25 mg/kg of glufosinate ammonium and MPP or with 0.25 mg/kg of NAG (expressed as glufosinate). The fortified samples were stored at about -20 °C and analysed after about 0, (1), 3, 6 and 9 months of storage. The analyses used method BK/01/99 (combined variant) with quantification by GC/FPD. Glufosinate, NAG and MPP are stable for at least 9 months in glufosinate-tolerant sugar beet pulp and molasses and in refined sugar under frozen conditions. Since the analytical method used in the study does not differentiate between glufosinate and NAG it would not have been possible to detect a possible interconversion between glufosinate and NAG.

Soya bean seed and hay

The frozen storage stability of glufosinate ammonium, NAG and MPP in glufosinate-tolerant soya bean seed and hay was investigated as part of a residue study (Czarnecki 1996a A53624). Homogenised samples of these commodities were fortified separately with 0.5 mg/kg of glufosinate ammonium, NAG or MPP (expressed as glufosinate). The fortified samples were stored frozen and analysed after about 0, 3, 6, 12 and 24 months of storage. The analyses were by method AE-24 (0, 3 and 6 month storage intervals) or according to the method BK/05/95 (12 and 24 month storage intervals) with quantification by GC/FPD. Method AE-24 allows separate determination of glufosinate, NAG and MPP while the method BK/05/95 yields the combined level of glufosinate and NAG residues with MPP being quantified separately.

In the soya bean seed samples analysed with the method AE-24, about 50% of glufosinate was found to be converted into NAG due to residual enzymatic activity. As both components are included in the residue definition the recovery for glufosinate in the soya bean seed samples was calculated as the sum of glufosinate and NAG quantified in the stored samples divided with the fortification level of glufosinate. This approach is consistent with the use of the method BK/05/95 (which determines glufosinate and NAG as a sum) for the analysis of the 12 and 24 month samples. No acetylation of glufosinate to NAG was observed in the soya bean hay samples. Glufosinate, NAG and MPP are stable under frozen conditions for at least 24 months in glufosinate-tolerant soya bean seed and for at least 12 months in glufosinate tolerant soya bean hay. An apparent degradation of glufosinate and NAG was observed in hay after 24 months of storage but may have been related to analytical difficulties.

Soya bean processed fractions

Czarnecki (1995 ER 93 USA 02) studied the frozen storage stability of glufosinate ammonium, NAG and MPP in glufosinate-tolerant soya bean meal, hulls and oil. Homogenised samples of these commodities were fortified separately with 0.5 mg/kg of glufosinate ammonium, NAG or MPP (expressed as glufosinate). The fortified samples were stored frozen and analysed after about 0, 3, 6 and 12 months of storage using method AE-24 (0, 3 and 6 month storage intervals) or according to the method BK/05/95 (12 month storage interval) with quantification by GC/FPD. Method AE-24 allows separate determination of glufosinate, NAG and MPP while the method BK/05/95 yields the combined level of glufosinate and NAG residues with MPP quantified separately. In the soya bean meal and hull samples analysed with method AE-24 up to 30% of glufosinate was converted to NAG. The recovery for glufosinate in the soya bean meal and hull samples as the sum of glufosinate and NAG in the stored samples divided by the glufosinate ammonium fortification level. No acetylation of glufosinate to NAG was observed in the soya bean oil samples. Glufosinate, NAG (or the sum of glufosinate and NAG) and MPP are stable under frozen conditions for at least 12 months in glufosinate-tolerant soya bean meal, hulls and oil.

Tolerant rape seed

Werner (1997b A59116) studied the frozen storage stability of glufosinate ammonium, NAG and MPP in transgenic glufosinate-tolerant rape seed. Homogenised samples of transgenic glufosinate-tolerant rape seed were fortified with 0.5 mg/kg of glufosinate, NAG and MPP (expressed as glufosinate). The fortified samples were stored deep frozen and analysed after about 0, 2, 4, 6, 12, 18 and 23 months of storage. Most of the analyses were conducted according to the method AE-24 with quantification by GC/FPD (separate determination of glufosinate, NAG and MPP) with the 6 month and some 23 month storage interval samples analysed according to the method AE-24A (combined glufosinate and NAG, MPP separate). Recoveries for glufosinate were calculated for the sum of glufosinate and NAG. Glufosinate, NAG and MPP are stable for at least 23 months in glufosinate-tolerant rape seed under frozen conditions. No interconversion between glufosinate and NAG was observed.

Tolerant field maize

The storage stability under frozen conditions of glufosinate ammonium, NAG and MPP in glufosinate-tolerant corn (maize) grain, fodder and forage was investigated (Czarnecki 1996 A53691). Homogenised samples of these commodities were fortified separately with 0.5 mg/kg of glufosinate, NAG or MPP (expressed as glufosinate).The fortified samples were stored frozen and analysed after about 0, 3, 6, 12 and 24 months of storage. The analyses used method AE-24 (0, 3 and 6 month storage intervals) or method BK/05/95 (12 and 24 month storage intervals) with quantification by GC/FPD. Glufosinate, NAG and MPP are stable for at least 24 months in glufosinate-tolerant corn grain, fodder and forage under frozen conditions with no interconversion between glufosinate and NAG observed.

Two studies investigated the freezer storage stability of glufosinate ammonium, NAG and MPP in transgenic glufosinate-tolerant maize green material and grain (Werner 1997c A58845, 1997d A59403). Homogenised samples of these commodities were fortified with 0.5 mg/kg of glufosinate ammonium, NAG and MPP (expressed as glufosinate). The fortified samples were stored deep frozen and analysed after about 0, 1–2, 3–4, 6, 12, 15, 18 and 24 months of storage. Most of the analyses utilised method AE-24 with quantification by GC/FPD (separate determination of glufosinate, NAG and MPP) with samples of the 6 and 12 month and some 24 month storage interval samples analysed using method AE-24A (glufosinate + NAG, MPP separate). Recoveries for glufosinate were calculated for the sum of glufosinate and NAG. Glufosinate, NAG and MPP are stable for at least 24 months in glufosinate-tolerant maize green material and grain under frozen conditions with no interconversion between glufosinate and NAG observed.

Brady (1995a A53690) studied the storage stability under frozen conditions of glufosinate, NAG and MPP in glufosinate-tolerant corn hulls, grits, flour and oil. Homogenised samples were

fortified separately with 0.5 mg/kg of glufosinate ammonium, NAG or MPP (expressed as glufosinate). The fortified samples were stored frozen and analysed after about 0, 3, 6 and 12 months of storage. Analyses were by method AE-24 (0, 3 and 6 month storage intervals) or method BK/05/95 (12 month storage interval) with quantification by GC/FPD. Glufosinate, NAG and MPP are stable under frozen conditions for at least 12 months in glufosinate-tolerant corn hulls, grits and flour. In corn oil low recoveries were observed for glufosinate while NAG and MPP were found to remain stable throughout the study. No interconversion between glufosinate and NAG was observed.

Sweet corn

In another study, Arsenovic (2005a 06515) studied freezer storage stability of glufosinate ammonium and MPP in glufosinate-tolerant sweet corn ear, forage and stover. Homogenised samples were fortified with 0.5 mg/kg of glufosinate ammonium and MPP (expressed as glufosinate) and analysed after about 26/27 months of frozen storage. Analyses were by method BK/05/95 with quantification by GC/FPD (glufosinate + NAG, with MPP quantified separately). Glufosinate (as glufosinate + NAG) and MPP are stable for at least 26/27 months in glufosinate-tolerant sweet corn forage and stover and for at least 30 months in glufosinate-tolerant sweet corn ear.

Tolerant rice

The storage stability under frozen conditions of glufosinate ammonium, NAG and MPP in glufosinate-tolerant rice grain and straw was investigated by Brady (1998 A57819). Homogenised samples of these commodities were fortified with 1 mg/kg of glufosinate ammonium and MPP or with 1 mg/kg of NAG (expressed as glufosinate). The fortified samples were stored frozen and analysed after about 0, 3, 6 and 12 months of storage according to the method BK/04/95 with quantification by GC/FPD. Glufosinate, NAG and MPP are stable for at least12 months in glufosinate-tolerant rice grain and straw under frozen conditions.

Table 65 Freezer storage stability of glufosinate-derived residues in plant matrices

Matrix	Storage period	% remaining			Mean procedural recovery (%)			Reference
	(months)	Glufosinate	NAG	MPP	Glufosinate	NAG	MPP	
Apple	0	65						Idstein 1987
	6	84						A36196
	8	88						Idstein 1988
	18	67						A36283
	24	91						
Maize grain	0	80						
	8	85						
	8.5	81						
	18	101						
	24	99						
Soya bean	0	78						
Seed	7.5	94						
	8	89						
	18	60						
	24	101						
Almond	0	93						Sochor 1990
	6	65						A43485
	12	59						
	24	72						
Orange	0	91						
	6	80						
	12	70						
	24	76						
Kiwifruit	0	93						Meikle 1991
	3	101						A46904
	6	99						
blueberry	20	95		72	90		78	Salzman 2002 IR-4 05291

Matrix	Storage period	% remaining			Mean procedural recovery (%)			Reference
	(months)	Glufosinate	NAG	MPP	Glufosinate	NAG	MPP	
Peach	24–26	91	78	74	100	79	76	Samoil 2009 IR-4 08720
Sugar beet	0	74	64	67	79	78	71	Werner 1997
Roots	1	74	72	71	67	74	66	A59404
	3	67	67	63	86	88	81	
	6	86	79	80	78	92	93	
	12	94	96	77	92	95	83	
	18	98	103	86	97	102	83	
	24	102	81	82	96	102	101	
Sugar beet	0	72	75	80	72	75	80	Bertrand 1998
Tops	3	68	77	91	61	86	97	A67531
	6	70	58	101	52	50	101	
	12	66	81	92	69	70	95	
	24	64	72	108	80	80	117	
Sugar beet	0	82	104	74	82	104	74	
Roots	3	79	77	94	87	87	97	
	6	85	106	112	86	100	94	
	12	100	101	99	108	99	91	
	24	79	85	118	64	82	111	
Sugar beet	0	80	87	83	80	87	83	Wyatt 2002
Pulp	1	90	93	91	84	91	78	B003971
	3	64	90	98	75	84	111	
	6	82	105	93	76	109	88	
	9	88	108	92	110	93	89	
Sugar beet	0	83	93	113	83	93	113	
Molasses	3	85	86	62	82	88	62	
	6	94	98	94	105	89	76	
	9	116	94	97	101	108	94	
Sugar beet	0	91	83	81	91	83	81	
Refined sugar	1	88	95	84	81	115	89	
	3	72	99	85	78	86	91	
	6	91	104	106	99	95	101	
	9	78	77	103	61	72	81	
Soya bean	0	88	94	84	88	94	84	Czarnecki 1996
Seed	3	90	99	91	93	100	104	A53624
	6	75	91	107				
	12	84	77	107				
	24	79	81	92	92	85	96	
Soya bean	0	93	97	101	93	97	101	
Fodder/hay	3	80	89	82	85	87	81	
	6	77	100	109				
	12	73	88	103				
	24	37	56	81	50	69	83	
Soya bean meal	0	73	82	108	73	82	108	Czarnecki 1995
	3	86	92	101	81	91	101	A53606/A55305
	6	93	100	90				
	12	83	78	93	81	77	96	
Soya bean hulls	0	87	89	92	87	89	92	
	3	86	82	96	91	84	89	
	6	96	104	93				
	12	94	96	95	96	103	88	
Soya bean oil	0	78	104	87	78	104	87	
	3	77	86	96	90	99	98	
	6	78	97	82				
	12	93	103	87	103	109	87	
Rapeseed	0	96	100	87	96	85	84	Werner 1997
	2	84	91	91	67	82	71	A59116

Matrix	Storage period	% remaining			Mean procedural recovery (%)			Reference
	(months)	Glufosinate	NAG	MPP	Glufosinate	NAG	MPP	
	4	85	97	90	84	96	94	
	12	103	88	81	100	93	84	
	18	92	91	86	88	85	83	
	23	101	97	90	92	80	88	
	6	85		71	90		88	
	23	104		106	104		106	
Corn grain	0	95	99	88	95	99	88	Czarnecki 1996
	3	89	93	86	87	97	86	A53691
	6	91	109	90	92	105	99	
	12	83	93	94				
	24	88	101	90	85	99	90	
Corn fodder	0	87	88	91	87	88	91	
	3	97	89	99	100	88	93	
	6	91	86	91	96	105	81	
	12	85	95	103				
	24	79	88	89	84	97	89	
Corn forage	0	94	96	98	94	96	98	
	3	91	90	97	86	82	75	
	6	83	100	93	80	105	77	
	12	76	88	105				
	24	76	78	86	80	90	89	
Maize green	0	76	75	65	88	103	95	Werner 1997
material	2	67	75	73	88	103	95	A58845
	4	75	60	51	85	63	52	
	15	87	68	57	84	68	58	
	18	92	77	77	100	84	77	
	24	83	88	72	99	86	73	
	6	106		108	105		73	
	12	100		96	91		97	
	24	91		86	83		75	
Maize grain	0	81	59	68	88	62	72	Werner 1997
	2	92	66	74	88	62	72	A59403
	4	80	83	61	46	83	73	
	15	100	58	84	106	68	91	
	18	106	89	68	104	105	81	
	24	113	97	86	104	93	76	
	6	68		78	63		78	
	12	63		73	84		82	
	24	91		88	87		84	
Corn hulls	0	88	80	82	88	80	82	Brady 1995
	3	81	91	92	92	91	96	A53690
	6	76	90	87				
	12	87	80	77	95	102	79	
Corn grits	0	92	101	93	92	101	93	
	3	84	89	81	87	110	104	
	6	73	93	86				
	12	88	93	81	89	98	82	
Corn flour	0	94	86	72	94	86	72	
	3	71	82	86	72	84	73	
	6	83	93	85				
	12	103	108	75	99	110	71	
Corn oil	0	83	102	89	83	102	89	
	3	67	94	84	86	90	94	
	6	68	85	79				
	12	58	83	76	89	87	79	
Sweet corn	26	79		72	75		73	Arsenovic 2005
	30	72		90	64		86	IR-4 06515
Sweet corn forage	26	77		70	87		72	

Matrix	Storage period	% remaining			Mean procedural recovery (%)			Reference
	(months)	Glufosinate	NAG	MPP	Glufosinate	NAG	MPP	
Sweet corn stover	27	91		80	94		77	
Rice grain	0	93	93	90	93	93	90	Brady 1998
	3	100	108	81	103	109	89	A57819
	6	77	73	85	73	84	80	
	12	87	83	83	84	90	83	
Rice straw	0	92	92	95	92	92	95	
	3	90	93	96	93	99	90	
	6	86	86	101	87	82	106	
	12	92	107	94	98	111	84	

Stability of residues in animal products

Krebs (1989 A40885) studied storage stability under frozen conditions of glufosinate ammonium and MPP in milk and cow tissues. Homogenised samples of milk were fortified with 0.1 mg/kg or 0.5 mg/kg of glufosinate ammonium and MPP (expressed as glufosinate) and analysed after 3.5–4.5 months of storage at about -20 °C. Homogenised samples of cow liver, kidney, diaphragm, muscle, cooked muscle, blood and fat were fortified with 0.5 mg/kg of glufosinate ammonium and MPP (expressed as glufosinate) and analysed after about 16 months of storage at about -20 °C. The analyses were conducted by GC/FPD using methods AL2/87 (milk), AL35/88 (muscle, diaphragm, liver, kidney and blood) and AL40/87 (fat). Glufosinate and MPP are stable for at least 4 months in milk and at least 16 months in cow tissues.

In a separate study Czarnecki (1996c A54503) studied the freezer storage stability of glufosinate ammonium, NAG and MPP in milk and cow tissues during a cow feeding study. Homogenised samples of cow milk, muscle, liver and kidney were fortified separately with 0.25 mg/kg of glufosinate ammonium, NAG and MPP (expressed as glufosinate). The fortified samples were stored deep frozen at between -12 and -27 °C and analysed after about 0, 1, 3 and 14–15 months of storage. The analyses were by GC/FPD using method BK/03/95. Glufosinate ammonium, NAG and MPP are overall stable for at least 14–15 months in cow milk, muscle, liver and kidney.

Sochor (1989a A40697) investigated the storage stability under frozen conditions of glufosinate ammonium and MPP in hen tissues. Homogenised samples of hen liver, kidney, muscle, cooked muscle, blood and fat were fortified with 1.0 mg/kg of glufosinate ammonium and MPP (expressed as glufosinate) and analysed after 2–6 weeks of storage at about -20 °C. The analyses were by GC/FPD using methods AL35/88 (muscle, liver, kidney and blood) and AL40/87 (fat). Glufosinate ammonium and MPP are stable for at least 2 weeks in hen muscle and blood, for at least 3 weeks in hen liver and kidney and for at least 6 weeks in hen fat.

Czarnecki (1997 A54485) also studied the freezer storage stability of glufosinate ammonium, NAG and MPP in eggs and hen tissues. Homogenised samples of hen eggs, muscle and liver were fortified separately with 0.25 mg/kg of glufosinate ammonium, NAG and MPP (expressed as glufosinate). The fortified samples were stored deep frozen at between -12 and -27 °C and analysed after about 0, 1, 3 and 15 months of storage using method BK/03/95 and GC/FPD. Glufosinate, NAG and MPP are overall stable for at least 15 months in hen eggs, muscle and liver.

Table 66 Storage stability of glufosinate-derived residues in animal matrices

Matrix	Storage period	%remaining			Mean procedural recovery (%)			Reference
	(months)	Glufosinate	NAG	MPP	Glufosinate	NAG	MPP	
Milk	3.5–4.4	100		127				Krebs 1989
Bovine liver	16	125		91				A40885
Bovine kidney	16	89		81				
Bovine diaphragm	16	86		85				

Matrix	Storage period	%remaining			Mean procedural recovery (%)			Reference
	(months)	Glufosinate	NAG	MPP	Glufosinate	NAG	MPP	
Bovine muscle	16	102		105				
Bovine muscle (cooked)	16	107		94				
Bovine blood	16	88		85				
Bovine fat	16	62		79				
Bovine kidney	0	122	104	91	122	104	91	Czarnecki 1996c
	1	86	90	91	102	88	90	A54503
	3	84	90	89	84	85	83	
	14	107	112	100	103	102	92	
Bovine muscle	0	93	86	73	93	86	73	
	1	88	85	79	88	88	79	
	3	68	75	71	79	80	73	
	15	76	93	81	87	89	79	
Bovine liver	0	89	94	85	89	94	85	
	1	98	89	90	117	109	85	
	3	81	82	85	86	82	81	
	15	97	107	92	97	85	93	
milk	0	110	112	73	110	112	73	
	1	95	104	83	112	102	83	
	3	97	90	71	106	103	80	
	14	86	83	82	91	88	82	
Poultry liver	0.75	84		83	84		83	Sochor 1989a
Poultry kidney	0.75	106		94	106		94	A40697
Poultry muscle	0.5	94		87	94		87	
Poultry muscle (cooked)	0.5	89		97	89		97	
Poultry blood	0.5	114		85	114		85	
Poultry fat	1.5	98		86	98		86	
Poultry muscle	0	102	103	96	102	103	96	Czarnecki 1997
	1	104	103	89	97	99	97	A54485
	3	79	76	86	84	85	85	
	15	102	108	90	106	89	93	
Poultry liver	0	106	111	88	106	111	88	
	1	102	102	92	90	106	98	
	3	81	91	87	93	87	86	
	15	105	108	84	113	99	88	
Eggs	0	105	102	85	105	102	85	
	1	109	101	82	116	103	78	
	3	83	81	79	92	87	86	
	15	100	86	87	101	93	84	

USE PATTERN

Glufosinate is a broad-spectrum non-selective herbicide that destroys green parts of treated weeds. A second use pattern is desiccation to facilitate harvest and a third is the use of glufosinate as foliar sprays on glufosinate-tolerant crops (Liberty Link crops or LLcrops).

The basic formulations have glufosinate ammonium contents of 150, 200 or 280 g/L.

Table 67 Use patterns of glufosinate ammonium

Crop	Country	Use	Application Rate kg ai/ha	Spray concentration kg ai/hL or spray volume L/ha	No.	PHI (days)
Almond	USA	Directed [i, j]	0.84–1.68 [k]	187–374 L/ha		14 [h]
Apple	Argentina	Directed	0.50–1.61	100–1000 L/ha	1	
Apple	Brazil [m]	Directed	0.40	300–600 L/ha	1	7
Apple	Canada	Directed	0.40–0.75 (Max 1 kg ai/ha/yr)	330–1100 L/ha	1	40
Apple	Mexico	Directed	0.45–0.75	200–400 L/ha		
Apple	USA	Directed [i, j]	0.84–1.68$	187–374 L/ha		14 [h]
Apricot	Japan	Directed	1.9	0.031	1–3	1
Asparagus	Canada	Broadcast after spears harvested	0.40–0.75	110–330 L/ha	1	
Asparagus	Canada	Pre-emergence	0.40–0.75	110–330 L/ha	1	
Asparagus	Germany	Directed	0.60	300–400 L/ha	1	
Avocado	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Banana	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Banana	Brazil [m]	Directed	0.40	300–600 L/ha	1	10
Banana	Colombia	Directed		0.113–0.15 kg ai/hL	1	
Banana	Colombia	Directed	0.30	100–400 L/ha	1	
Banana	Malaysia	Directed	0.50	450 L/ha		14
Banana	Mexico	Directed	0.15–0.45	200–400 L/ha	1	
Banana	Mexico	Directed	0.42–0.56	200–400 L/ha	1	
Banana	Philippines	Directed	0.25–2	(0.127–0.188 kg ai/hL)		
Banana	Portugal	Directed	0.45–1.50	400–1000 L/ha	1–2	
Banana	Taiwan	Directed	1.05	0.175 kg ai/hL	1	
Bare soil pre-cultivation	France	Broadcast	0.75			
Barley	Portugal	Pre-emergence	0.45–0.75	400–1000 L/ha	1	
Bean (Ph. v.)	Brazil	PH desiccation. Apply when ≈50% of dry pods. Apply higher dose only when 70% of the pods dried.	0.36–0.40	300–600 L/ha (for desiccation by air 30–40 L/ha)	1	5
Bean (Ph. v.)	Portugal	Pre-sowing, pre-planting, pre-emergence	0.45–0.75	400–1000 L/ha	1	
Bean (Ph. v.) Dry common bean	Canada	PH desiccation. Apply when approximately 50–75% of the bean pods have naturally changed colour from green to yellow or brown (pod turn).	0.375–0.45	Desiccant > 110 L/ha (dense canopy 170–220 L/ha)	1	9 [c]
Bean, bush	Germany	Directed [n]	1	300–400 L/ha	1	14
Beans, field dry	UK	PH desiccation. Apply when crop is mature (i.e. Stems brown and pods black), usually 10–14 days before harvesting.	0.45	200–400 L/ha	2	7 [e]
Blackberry	Australia	Directed		0.100 kg ai/hL	1	0 [b] (56G)
Blackberry	Netherlands	Directed [n]	0.75	300–500 L/ha		
Blackberry	Netherlands	Directed [n]	1	(0.2 kg ai/hL)		
Blueberry	USA	Directed [i]	0.84–1.68 [l]	187–374 L/ha		14
Boysenberry	Australia	Directed		0.100 kg ai/hL	1	0 [b] (56G)
Cabbage	Brazil [m]	Protect the plant with plastic cups	0.3–0.4	300–600 L/ha	1	7
Cabbage	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Carambola	Malaysia	Directed	0.50	450 L/ha		14
Carrot	Canada	Pre-emergence or pre-sowing	0.41–0.75	110–330 L/ha	1	
Carrot	Germany	Pre-emergence to BBCH 03	0.60	300–400 L/ha	1	
Cashew nut	Malaysia	Directed	0.50	450 L/ha		14
Chilli	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14

Crop	Country	Use	Application Rate kg ai/ha	Spray concentration kg ai/hL or spray volume L/ha	No.	PHI (days)
Chinese cabbage	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Chinese cabbage	Taiwan	Directed	0.45	0.075 kg ai/hL	1	
Citrus	Australia	Directed	0.20–1.0	–	1	0 [b] (56G)
Citrus	Brazil [m]	Directed	0.40	300–600 L/ha	1	40
Citrus	Colombia	Directed	0.15–0.22	100–400 L/ha	1	
Citrus	Colombia	Directed	0.30	100–400 L/ha	1	
Citrus	Japan	Directed	1.9	–	1–3	21
Citrus	Malaysia	Directed	0.50	450 L/ha		14
Citrus	Mexico	Directed	0.42–0.60	200–400 L/ha	1	
Citrus	Philippines	Directed	0.30–0.96	(0.127–0.15 kg ai/hL)		
Citrus	Portugal	Directed	0.45–1.5	400–1000 L/ha	1–2	
Cocoa	Malaysia	Directed	0.3–0.5	100–450 L/ha		14
Cocoa	Philippines	Directed	0.30–0.96	(0.127–0.15 kg ai/hL)		
Coffee	Brazil [m]	Directed	0.40–0.60	300–600 L/ha	1	20
Coffee	Colombia	Directed	0.15–0.22	100–400 L/ha	1	
Coffee	Colombia	Directed	0.30–0.60	100–400 L/ha	1	
Coffee	Malaysia	Directed	0.3–0.5	100–450 L/ha		20
Coffee	Philippines	Directed	0.30–0.96	(0.127–0.15 kg ai/hL)		
Corn	Brazil [m]	Directed	0.3–0.4	300–600 L/ha	1	nr
Corn, sweet	Germany	Pre-sowing, directed up to BBCH 16-19	1.0	300–400 L/ha	2	
Cotton	Brazil [m]	When crop 40 cm high	0.40	300–600 L/ha	1	28
Cotton	Colombia	When crop > 30 cm	0.225–0.27	100–400 L/ha	1	
Cotton	Colombia	When 50 cm high, using a screen or as a defoliant.	0.3 Defoliant: 0.15, aerial spraying when about 80% open capsules	100–400 L/ha	1	
Cotton	Mexico	Directed	0.30–0.50	200–400 L/ha		
Crops	France	Directed	0.6–0.75			
Crops	Netherlands	Pre-emergence, apply no later than 3 days before emergence or planting of crop	0.6	(0.2 kg ai/hL)		
Cucumber	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Cucumber	Taiwan	directed	0.45	0.075 kg ai/hL	1	
Currant	Netherlands	Directed [n]	0.75	300–500 L/ha		
Currant	Netherlands	Directed [n]	1	(0.2 kg ai/hL)		
Currant	USA	Directed [i]	0.84–1.68 [l]	187–374 L/ha		14
Durian	Malaysia	Directed	0.50	450 L/ha		14
Elderberry	USA	Directed [i, j]	0.84–1.68 [l]	187–374 L/ha		14
Filbert tree	USA	Directed [i,j]	0.84–1.68 [k]	187–374 L/ha		14 [h]
Flax	France	For retting	0.45–0.75			
French bean	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Fruit trees	Netherlands	Directed	0.75	300–500 L/ha		
Fruit trees	Netherlands	Directed	1	(0.2 kg ai/hL)		
Gooseberry	Germany	Directed [n]	1 spring/summer	300–600 L/ha	1	14
Gooseberry	USA	Directed [i]	0.84–1.68 [l]	187–374 L/ha		14
Grape	Argentina	Directed	0.50–1.61		1	
Grape	Australia	Directed	0.20–1.00		1	0 [b] (56G)
Grape	Brazil [m]	Directed	0.40	300–600 L/ha	1	7
Grape	Canada	Directed	0.40–0.75 (Max 1 kg ai/ha/yr)	330–1100 L/ha	1	40

Crop	Country	Use	Application Rate kg ai/ha	Spray concentration kg ai/hL or spray volume L/ha	No.	PHI (days)
Grape	Colombia	Directed	0.225–0.30	100–400 L/ha	1	
Grape	Germany	Directed	1.5 spring/ 1 summer	300–600 L/ha	2	14
Grape	Japan	Directed	1.9		1–3	1
Grape	Mexico	Directed	0.30–0.45	200–400 L/ha	1	
Grape	Taiwan	Directed	0.75	0.125 kg ai/hL	1	
Grape	USA	Directed [i, j]	0.84–1.40 [k]	187–374 L/ha		14
Grape suckers	France	Directed		0.188– 0.3 kg ai/hL	1–2	14
Grass pasture	Netherlands	Spot treatment of weeds	0.55–1	300–500 L/ha		(7G) [g]
Guava	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Guava	Malaysia	Directed	0.50	450 L/ha		14
Hickory n.	USA	Directed [i, j]	0.84–1.68 [k]	187–374 /ha		14 [h]
Huckleberry	USA	Directed [i]	0.84–1.68 [l]	187–374 L/ha		14
Indian jujube	Taiwan	Directed	0.45	0.075 kg ai/hL	1	
Jackfruit	Malaysia	Directed	0.50	450 L/ha		14
Juneberry	USA	Directed [i]	0.84–1.68 [l]	187–374 L/ha		14
Kiwifruit	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Kiwifruit	Japan	Directed	1.4	–	1–3	21
Leek	Germany	Pre-emergence to BBCH 03	0.60	300–400 L/ha	1	
Lettuce	Brazil+	Directed. Protect plants with plastic cups	0.30–0.40	300–600 L/ha	1	7
Lettuce	Canada	Pre-emergence or pre-sowing	0.41–0.75	110–330 L/ha	1	
Corn salad	Germany	Pre-emergence to BBCH 03	0.60	300–400 L/ha	1	
Lettuce	Japan	Directed	0.9		1–2	30
Lettuce	Portugal	Pre-sowing, pre-planting, pre-emergence	0.45–0.75	400–1000 L/ha	1	
Lingonberry	USA	Directed [i]	0.84–1.68 [l]	187–374 L/ha		14
Linseed	UK	PH desiccation. Apply when 95% of the bolls are brown usually at least 14 days before harvest.	0.45	400 L/ha	2	7 [e]
Litchi	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Loganberry	Australia	Directed		0.100	1	0 [a] (56G)
Long bean	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Macadamia	USA	Directed [i, j]	0.84–1.68 [k]	187–374 L/ha		14 [h]
Maize/Corn	Colombia	Directed	0.225–0.27	100–400 L/ha	1	
Maize/Corn	Mexico	Directed	0.15–0.42	200–400 L/ha	1	
Mango	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Mango	Colombia	Directed	0.225	100–400 L/ha	1	
Mango	Colombia	Directed	0.30	100–400 L/ha	1	
Mango	Malaysia	Directed	0.50	450 L/ha		14
Melon (musk)	Taiwan	directed	0.45	0.075 kg ai/hL	1	
Melon honeydew	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Nectarine	Brazil [m]	Directed	0.40	300–600 L/ha	1	7
Olive	Australia	Directed	0.20–1.00		1	0 [b] (56G)
Olive	Portugal	Directed	0.45–1.50	400–1000 L/ha	1–2	
Olive	Spain	Directed	0.45–1.50	0.300–0.375	1–2	(21G) [d]
Onion	Canada	Pre-emergence or pre-sowing	0.41–0.75	110–330 L/ha	1	
Onion	Germany	Pre-emergence to BBCH 03	0.60	300–400 L/ha	1	
Onion	Portugal	Pre-sowing, pre-planting, pre-emergence	0.45–0.75	400–1000 L/ha	1	

Crop	Country	Use	Application Rate kg ai/ha	Spray concentration kg ai/hL or spray volume L/ha	No.	PHI (days)
Other than woody plants	Spain	Directed, use screens to protect crop [n]	0.45–1.50		1–2	(21G)[d]
Palm oil	Colombia	Directed	0.375–0.45	100–400 L/ha		
Palm oil	Malaysia	Directed	0.3–0.5	100–450 L/ha		14
Palm oil	Philippines	Directed	0.30–0.96	(0.127–0.15 kg ai/hL)		
Papaya	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Papaya	Colombia	Directed	0.225–0.30	100–400 L/ha	1	
Papaya	Malaysia	Directed	0.50	450 L/ha		14
Papaya	Taiwan	Directed	0.75	0.125 kg ai/hL	1	
Passion fruit	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Passion fruit	Colombia	Directed	0.30	100–400 L/ha	1	
Passion fruit	Colombia	Directed	0.225	100–400 L/ha	1	
Peach	Argentina	Directed	0.50–1.61		1	
Peach	Brazil [m]	Directed	0.40	300–600 L/ha	1	7
Peach	Canada	Directed	0.40–0.75 (Max 1 kg ai/ha/yr)	330–1100 L/ha	1	40
Pear	Argentina	Directed	0.50–1.61		1	
Pear	Canada	Directed	0.40–0.75 (Max 1 kg ai/ha/yr)	330–1100 L/ha	1	40
Pear	Taiwan	Directed	0.45	0.075 kg ai/hL	1	
Peas	Portugal	Directed	0.45–0.75	400–1000 L/ha	1	
Peas dry	UK	PH desiccation. Apply when crop is mature (i.e. has an overall yellow appearance), usually 10-14 days before harvest. Bottom pods should be yellow/brown and seeds hard. Top pods should be fleshy, pitted and turning yellow. Peas should have a moisture content of 45% or less.	0.45	200–400 L/ha	2	7 (7G) [f]
Pecan	USA	Directed [i, j]	0.84–1.68 [k]	187–374 L/ha		14 [h]
Pepper, black	Malaysia	Directed	0.50	450 L/ha		14
Peppers (Bell)	Taiwan	directed	0.45	0.075 kg ai/hL	1	
Persimmon	Taiwan	Directed	0.75	0.125 kg ai/hL	1	
Pineapple	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Pistachio	USA	Directed [i, j]	0.84–1.68 [k]	187–374 L/ha		14 [h]
Plantain	Colombia	Directed		0.113–0.15 kg ai/hL	1	
Plum	Canada	Directed	0.40–0.75 (Max 1 kg ai/ha/yr)	330–1100 L/ha	1	40
Pome fruit	Australia	Directed	0.20–1.00	–	1	21 [b] (56G)
Pome fruit	Germany	Directed	1 spring/summer–1.5spring	300–600 L/ha	1–2	14
Pome fruit	Portugal	Directed	0.45–1.50	400–1000 L/ha	1–2	
Pome fruit	UK	Directed, apply between 1March and 30 September	0.45–0.75	200–400 L/ha	1–3	[e]
Potato	Brazil [m] 0.2	Pre-emergence or for PH desiccation	0.40	300–600 L/ha	1	10
Potato	Canada	Pre-emergence: no later than ground crack	0.4–0.75	110–330 L/ha	1	40
Potato	Colombia	Pre-emergence and directed	0.22–0.30	100–400 L/ha	1	
Potato	France	PH desiccation.	0.375		1–2 (5d)	14
Potato	Germany	Pre-emergence	0.60		1	

Crop	Country	Use	Application Rate kg ai/ha	Spray concentration kg ai/hL or spray volume L/ha	No.	PHI (days)
		PH desiccation. Apply at crop growth stage BBCH 90	0.50		1	14
Potato	Mexico	PH desiccation.	0.45–0.60	200–400 L/ha	1	
Potato	Netherlands	Pre-emergence and directed: Spraying should preferably be carried out before potato emergence or when the first potatoes are emerging. Treatment between the rows of already emerged potatoes is only possible when using special equipment with shields.	0.45–0.75	300–500 L/ha	1–2	
		PH desiccation: after natural senescence has started i.e. after the crop starts yellowing or the berries are ripe and start shrivelling (after crop stage BBCH 90). A crop that has not yet reached the natural senescence stage must be treated as a seed potato crop and haulm must be flailed beforehand.	0.45 (0.375 flailed crops)	200–400 L/ha (250L/ha flailed crops)	1–2	
Potato	Portugal	Pre-sowing or pre-planting	0.45–1.50	400–1000 L/ha		
		PH desiccation, March to September.	0.30–0.45	400–1000 L/ha	1–2	14
Potato	UK	Apply pre-emergence or during emergence but no later than 10% emergence for earlies or maincrop potatoes (10% emergence represents 1 plant emerged out of 10). Individual plants should be no more than 5 cm high and have no more than one shoot per tuber emerged.	0.45	200–400 L/ha	2	7 [e]
		PH desiccation.	0.45	200–400 L/ha	1–4	7 [e]
Potato	USA	PH desiccation [i]	0.43 –	76–374 L/ha (47–94 L/ha air)	1	9
Rambutan	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Rape	Germany	PH desiccation: 2/3 pods darkish yellow brown	0.5	300–400 L/ha	1	14
Rape, spring and winter.	UK	PH desiccation: Apply one treatment per crop when most of the pods in the centre third of the stem are yellow and the majority of seeds within are reddish to dark brown in colour, usually 14-21 days before harvest	0.45	300–400 L/ha	2	7 [e]
Raspberry	Australia	Directed		0.100	1	0 [a] (56G)
Raspberry	Germany	Directed [n]	1 spring/summer	300–600 L/ha	1	14
Raspberry	Netherlands	Directed [n]	0.75–1	300–500 L/ha (0.2 kg ai/hL)		
Raspberry.	Canada	Directed: for the control of primocanes in established crops. Apply when shoots are about 10–20 cm in height. DO NOT apply to immature or weak plantings	1.0	Use > 330 L/ha	1–2	
Rice	Colombia	Pre-planting: plant 10 days after application	0.30	100–400 L/ha	1	
Rice	Colombia	Pre-sowing	0.4	100–400 L/ha	1	
Salal	USA	Directed [i, j]	0.84–1.68[l]	187–374 L/ha		14
Sawi kalain	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Soft fruit = cane fruit, currants, small berries	UK	Directed: Apply between 1 March and 30 September	0.45–0.75	200–400 L/ha	1–3	[e]

Crop	Country	Use	Application Rate kg ai/ha	Spray concentration kg ai/hL or spray volume L/ha	No.	PHI (days)
Sorghum	Colombia	PH desiccation: As a desiccant when the grain is fully formed, with 25–29% moisture. For the case of varieties of sorghum hybrid D-61 and ST Guapo at 96 days after germination	0.075–0.113	100–400 L/ha	1	
Soya bean	Brazil+	Pre-sowing or PH desiccation	0.50–0.60	300–600 L/ha (for desiccation by air 30–40 L/ha)	1	10
Soya bean	Colombia	Directed, when crop is 30–40 cm use screen	0.22–0.30	100–400 L/ha	1	
Soya bean	Mexico	Directed	0.22–0.30	200–400 L/ha	1	
Stone fruit	Australia	Directed	0.20–1.00	–	1	21 [b] (56G)
Stone fruit	Portugal	Directed	0.45–1.50	400–1000 L/ha	1–2	
Stone fruit	UK	Directed: Apply between 1 March and 30 September	0.45–0.75	200–400 L/ha	1–3	[e]
Stone fruit (except peach)	Germany	Directed	1 spring/summer–1.5spring	300–600 L/ha	1–2	14
Stone fruit (root bolters)	Germany	Directed	1.5 1st spray–1 2nd spray	300–600 L/ha	2 (6–14d)	14
Strawberry	UK	Directed: Apply between 1 March and 30 September	0.45–0.75	200–400 L/ha	1–2	[e]
Strawberry	Australia	Directed	0.20–1.00		1	0 [b] (56G)
Strawberry	Germany	Directed, pre-flowering [n]	0.80	300–600 L/ha	1	42
Sugar beet	Portugal	Pre-emergence	0.45–0.75	400–1000 L/ha	1	
Sugar beet and vegetable crops	UK	Pre-emergence	0.45	200–400 L/ha	1	[e]
Sugar cane	Colombia	Directed	0.1–0.3	100–400 L/ha	1	
Sugar cane	Mexico	Directed	0.28–0.56	200–400 L/ha	1	
Sugar beet	Germany	Pre-sowing	1.00	300–400 L/ha	1	
Sunflower	Germany	PH desiccation: 2/3 seeds brown, moisture < 30%	0.5	300–400 L/ha	1	14
Sweet pea	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Tea	India	Directed	0.375–0.5			
Tea	Malaysia	Directed	0.3–0.5	100–450 L/ha		7
Tomato	Australia	Directed	0.20–1.00		1	0 [b] (56G)
Tomato	Colombia	Directed	0.225	100–400 L/ha	1	
Tomato	Colombia	Directed	0.30	100–400 L/ha	1	
Tomato	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Tree nuts	Australia	Directed	0.20–1.00	–	1	0 [b] (56G)
Tree nuts	UK	Directed: Apply between 1 March and 30 September	0.45–0.75	200–400 L/ha	1–3	[e]
Triticale	Portugal	Pre-emergence	0.45–0.75	400–1000 L/ha	1	
Vines forestry	UK	Directed: Apply between 1 March and 30 September	0.45–0.75	200–400 L/ha	1–3	[e]
Walnut	Mexico	Directed	0.45–0.75	200–400 L/ha	1	
Walnut	USA	Directed [i, j]	0.84–1.68 [k]	187–374 L/ha		14
Watermelon	Malaysia	Pre-emergence and directed	0.50	450 L/ha		14
Wax apple	Taiwan	Directed	0.75	0.125 kg ai/hL	1	
Welsh onion	Taiwan	directed	0.45	0.075 kg ai/hL	1	
Wheat	Brazil [m]	Pre-sowing	0.40	300–600 L/ha	1	nr
Wheat	Portugal	Pre-emergence	0.45–0.75	400–1000 L/ha	1	

Crop	Country	Use	Application Rate kg ai/ha	Spray concentration kg ai/hL or spray volume L/ha	No.	PHI (days)
Woody plants (shoots at base, suckers)	Spain	Directed	0.45–1.50	1–2		(21G)[d]
Woody plants (trees, shrubs)	Spain	Directed, use screens to protect crop	0.45–1.50		1–2	(21G)[d]

[a] Cane fruit: Apply as a directed spray to suckers and primocanes. Contact with flowers, developing fruit or desirable foliage will cause damage. Ensure complete coverage of primocanes / suckers by spraying to the point of runoff, preferably when they are less than 15 cm high. A non-ionic wetting agent may be added. Do not graze or cut treated areas for stock food for 8 weeks after application

[b] Other than cane fruit: Apply as a directed or shielded spray (to weeds). Do not graze or cut treated areas for stock food for 8 weeks after application

[c] Livestock Feeding: Grain and meal from treated crops can be fed to livestock. Do not graze the treated crops or cut for hay; sufficient data are not available to support such use.

[d] Grazing: Between treatment and livestock input: 21 days

[e] Keep livestock out of treated areas until foliage of any poisonous weeds, such as ragwort, have died and become unpalatable (expect takes 10-14 days).

[f] Treated haulm may be fed to livestock from 7 days after spraying.

[g] Apply when no livestock is present in the field. The fields may not be grazed within 1 week after application.

[h] Do not graze, harvest, and/or feed treated orchard cover crops to livestock.

[i] Do not apply this product through any type of irrigation system.

[j] Do not apply this product aerially to tree and vine crops.

[k] Maximum annual application is 5 kg ai/ha/year

[l] Maximum annual application is 3.36 kg ai/ha/year

[m] Add spreader/adhesive or crop oil adjuvants to spray mixture

[n] When the crop is present at the time of spraying weeds, use a spray shield to protect the crop from contamination

Table 68 Use patterns for glufosinate tolerant crops

			Application			
Crop	Country	Use	Rate kg ai/ha	Spray volume L/ha	No.	PHI
LLCanola	Australia	Post-emergence from 2 leaf until early bolting stage	0.30–0.4	50–100 L/ha	1 – 2	0 (56) [a]
LLCanola	Canada [i]	Post-emergence	0.20–0.60, if 2nd spray use 0.3–0.5	> 110 L/ha (> 55 L/ha post-emergence)	1–2	0 [c, d]
LLCanola	USA 1	Pre-planting or pre-emergence	0.59–0.74 (max 0.74 kg ai/ha/season)		1	
	USA 2	Post-emergence from the cotyledon stage up to the early bolting stage of the canola	0.47–0.50 (max 0.99 kg ai/season) [h]	140–187 L/ha (> 47 L/ha air)	1–2	65 [f]
LLCorn	Argentina	Post-emergence, 3–4 leaf stage only add adjuvant	0.3–0.7	100–150 L/ha	1	35
LLCorn	Canada [i]	Post-emergence, 1–8 leaf stage of the corn plant or 5–6 visible collars	0.30–0.40 (max 0.9 kg ai/ha/yr) [e]	> 110 L/ha	1–2	86 (20G)
LLCorn	USA 1	Pre-planting or pre-emergence	0.59–0.74 (max 0.74 kg ai/ha/season)		1	

			Application			
Crop	Country	Use	Rate kg ai/ha	Spray volume L/ha	No.	PHI
	USA 2	Post-emergence until corn is 61 cm tall or in the V-7 stage of growth, i.e., 7 developed collars, whichever comes first. For corn 61 to 91 cm tall, only apply using ground application and drop nozzles and avoid spraying into the whorl or leaf axils of the corn stalks	0.41–0.50 (max 0.91 kg ai/ha/season) [h]	–	1–2	60 forage 70 grain fodder
LLCorn	Brazil	Post-emergence, if 2 sprays first must be made when corn is with 3–4 leaves. The second must be made when corn is with 5–6 leaves	0.30–0.60	200–300 L/ha (for air 30–40 L/ha)	1–2	50
Corn/maize tolerant	Colombia	Post-emergence, targeted	0.225–0.27	100–400 L/ha	1	
LLCotton	Australia	Post-emergence, directed	0.75	> 100 L/ha	1–3	70 [b]
LLCotton	Brazil	Post-emergence	0.40-0.70	200–300 L/ha (for air 30–40 L/ha)	1	116
LL Cotton	USA 1	Pre-planting or pre-emergence	0.59		1	
		Post-emergence from emergence up to the early bloom stage	0.45–0.59 (max 1.8 kg ai/ha/yr		3	70
	USA 2	Pre-planting or pre-emergence	0.61–0.88		1	
		Post-emergence from emergence up to the early bloom stage	0.45–0.59 (max 1.5 kg ai/ha/yr		2	70
	USA 3	Post-emergence from emergence up to the early bloom stage	0.47–0.58 (max 1.2 kg ai/ha/yr	140–374 L/ha (> 94 L/ha air)	1–3	70
LLRice	USA 1	Pre-planting or pre-emergence	0.59–0.74 (max 0.74 kg ai/ha/season)		1	
	USA 2	Post-emergence from the 1-leaf stage through the mid-tillering stage of development	0.41–0.50 (max 0.99 kg ai/ha/season)	> 94–374 L/ha (> 94 L/ha air)	1–2	70
LLSoya bean	Canada [i]	Post-emergence cotyledon to the flowering stage	0.30–0.40 (Max 0.9 kg ai/ha/yr) [e]	> 110 L/ha	1–2	70 (20G)
LLSoya bean	USA	Pre-planting or pre-emergence	0.59-0.74 (max 0.74 kg ai/ha/season)		1	
		Post-emergence to the bloom growth stage	0.45–0.74 for 1st 0.45–0.59 for 2nd (max 1.3 kg ai/ha/season)	140–187 L/ha (> 47 L/ha air)	1–2	70 [g]
LLSugarbeet	USA	Pre-planting or pre-emergence	0.59–0.74 (max 0.74 kg ai/ha/season)		1	
		Post-emergence from the cotyledon stage up to the 10-leaf stage of the sugar beet.	0.29–0.61 (max 1.2 kg ai/season)	140–187 L/ha (> 47 L/ha air)	1-3	60[f]

*: LLsweet corn USA: petition under evaluation, approval pending

[a] Do not graze or cut for stock feed for 8 weeks after application.

[b] Do not graze or cut treated vegetation for stock feed. Do not feed cotton trash to livestock.

[c] If tank mixed with clethodim, observe a PHI of 60 days from the date of treatment (or last treatment when a second application has been made)

[d] Grain and meal from treated crop can be fed to livestock. Do not graze the treated crop or cut for hay; sufficient data are not available to support such use.

[e] If using a split spray program, the first application must be a minimum of 0.4 kg ai/ha and at the proper weed stage. For the second application, the rate of 0.25 kg ai/ha may be utilized.

Note in general a 2nd application may be made provided the 1st was at < = 0.5 kg ai/ha.

[f] Do not graze the treated crop or cut for hay. DO NOT add surfactants. Anti-foams or drift control agents may be added if needed.

[g] Do not harvest treated green soya bean plants for forage and hay feed for livestock.

[h] Glufosinate must be applied with ammonium sulphate (AMS).

[i] Eastern Canada and British Columbia

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Glufosinate is a phosphinic acid analogue of glutamic acid and is a herbicide that is highly active against growing weeds. The Meeting received information on supervised field trials for glufosinate on the following crops or crop groups:

Commodity	Table no.
Citrus fruit	Table 70
Pome fruit	Table 71, 72
Stone fruit	Table 73
Berries and small fruit	Tables 74–79
Tropical fruit, edible peel	Tables 80, 81
Tropical fruit, inedible peel	Tables 82–87
Bulb vegetables	Table 88
Fruiting vegetables, other than cucurbits	Table 89
Leafy vegetables (lambs lettuce, lettuce)	Tables 90, 91
Legume vegetables and pulses	Tables 92–94
Root and tuber vegetables	Tables 95–97
Stalk and stem vegetables	Table 98
Cereals	Tables 99–101
Tree nuts	Table 102
Oilseeds	Tables 103–106
Beverage seeds	Table 107
Animal feed	Tables 108–121

Trials were generally well documented with laboratory and field reports; trials from the 1980s followed the standards of those times. Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Control samples are indicated in the summary tables with a "c". Unless stated otherwise, residue data are recorded unadjusted for recovery.

Label directions for use of glufosinate in the USA often suggest the addition of ammonium sulphate (AMS) as an adjuvant to the spray and AMS was added in many of the supervised trials conducted in the USA. In the tables of residue data, the use of AMS is indicated in the reference column by "+ AMS". Some trials included a non-ionic antifoam agent and this is indicated as “+NIA” in the summary tables.

The application rate, kg ai/ha, is expressed in terms of glufosinate ammonium, i.e., the active ingredient is taken as glufosinate ammonium and not the free acid while residues of glufosinate, MPP and NAG are all reported as glufosinate free acid equivalents.

Residues and application rates have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. Those results included in the evaluation are underlined.

Growth stage of the crop at the time of application may be an important determinant of residues in the harvested commodity. Growth stage data for the time of the last application have been included in the trial data summaries where available. Because of the importance of growth stage at time of treatment and the nature of the field crops being tested, in some residue trials separate plots were treated at various growth stages and then all were harvested at crop maturity on the same day.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most trial designs used non-replicated plots. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Table 69 Summary of sprayers, plot sizes and field sample sizes in the supervised trials

Crop	Location	Year	Sprayer	Plot size	Sample size	Sample to analysis interval (days)
Orange	Brazil	2007	CO_2 backpack sprayer	60–96 m^2	?	231
Orange	Spain /Italy	1999	plot sprayer hand carried	50–60 m^2	≥ 12 fruit	76–133
Orange	Spain /Greece /Italy	2000	Plot Sprayer	54–90 m^2	≥ 12 fruit	101–132
Orange	USA	2008	CO_2 backpack sprayer, hand boom, Tractor mounted orchard sprayer, under canopy sprayer	67–245 m^2	≥ 24 fruit	336
Lemon	USA	2008	CO_2 backpack sprayer	67–245 m^2	≥ 24 fruit	336
Grapefruit	USA	2008	CO_2 backpack sprayer, hand boom, Tractor mounted boom, under canopy sprayer	111–245 m^2	≥ 24 fruit	336
Macadamia	Australia	1992	Small plot sprayer, Directed spray, hand held boom	21–30 m^2	≥ 1 kg	< 30
Hazelnut	Italy	1985	?	20 m^2	≥ 1 kg	216–224
Walnut	USA	1985	?	9 trees	≥ 0.5 kg	< 694
Almond	USA	1985	CO_2 backpack sprayer	22–23 m^2	1 kg	502–686
Pecan	USA	1985	CO_2 backpack sprayer, tractor mounted sprayer	1 tree—23–42 m^2	1 kg	405–464
Walnut	USA	1985	CO_2 backpack sprayer, hand held plot sprayer, backpack sprayer	14–28 m^2	1 kg	458–526
Apple	Brazil	2006	Backpack sprayer	58–75 m^2	?	< 161
Apple	Germany	1983	?	2–6 trees	30–40 units > 0.5 kg	< 224
Apple	Europe	1996	plot sprayer hand carried (air pressured)	18–36 m^2	≥ 1 kg	<151
Apple	Europe	1997	plot sprayer (single wheel—compressed air)	24–105 m^2	≥ 1 kg	111–205
Apple	France	1997	plot sprayer hand carried (air pressured)	15–18 m^2	≥ 1 kg	174–223
Apple	Europe	1999	plot sprayer hand carried (air pressured)	45 m2	≥ 12 fruit	55–125
Apple	Europe	2000	knapsack sprayer, wheel barrow sprayer, plot sprayer hand carried	12–37.5 m^2	≥ 12 fruit	27–85
Apple	USA	1984/85	Solo Mist Blower, CO_2 Hand-Held Boom, Ground Boom, Solo Sprayer, CO_2 Backpack Sprayer	17–194 m^2, 1–3 trees	≥ 24 fruit	124–655
Pear	USA	2008	Tractor-mounted Boom, CO_2 Backpack Sprayer, Rack Sprayer	76–201 m^2	≥ 2 kg	162–224
Apricot	Germany	1982	?	50–80 m^2	≥ 1 kg	< 219
Cherry, sour	Germany	1981	?	33 m^2	≥ 2 kg	< 733
Cherry, sour	Germany	1987	?	115 m^2	≥ 2 kg	< 168
Cherry, sour	Canada /USA	2008	Backpack sprayer, Hand-held CO_2 Spray Boom	84–223 m2	?	< 323
Cherry, sweet	USA	2008	CO_2 Backpack Sprayer	111–223 m^2	?	< 323
Peach /Nectarine	Brazil	2008	CO_2 Backpack Sprayer	75–98.1 m^2	?	< 442
Peach	Germany	1982	?	8 trees, 30 m^2	30–40 fruit	< 229
Peach	Europe	1999	boom sprayer with nozzles, motorized knapsack sprayer, plot sprayer hand carried	12.8–60 m^2	≥ 24 fruit	68–172
Peach	Europe	2000	plot sprayer hand carried, Knapsack Sprayer, motorized	36–56 m^2	≥ 24 fruit	193–249
Peach	USA	2007/08	CO_2 Backpack Sprayer	6–10 trees	≥ 24 fruit	< 791
Plum	USA	2008	CO_2 Backpack Boom Sprayer, Hand boom	148–201 m^2	?	< 323
Plum	Europe	2004	Knapsack Sprayer Spraying boom	11–135 m^2	≈2 kg	< 360
Plum	Europe	2007	Knapsack Sprayer Spraying boom	35–63 m^2	≥ 2 kg	287–360
Blueberry	USA	1997	Backpack sprayer CO_2	74–279 m^2	1.3–1.8 kg	< 703

Crop	Location	Year	Sprayer	Plot size	Sample size	Sample to analysis interval (days)
Currant, black	Germany	1982	?	50–80 m^2, 4–9 plants	≥ 1 kg	< 388
Currant, black	Europe	2005	Knapsack Sprayer	8–28 m^2	≥ 0.6 kg	319–392
Currant, black/red	France	2008	Knapsack Sprayer	10–12 m^2	≥ 0.6 kg	324–357
Gooseberry	Germany	1983	?	30–45 m^2, 30 plants	≈1 kg	< 401
Grapes	Brazil	2006	Backpack sprayer	29–90 m^2	?	< 143
Grape	Germany	1983	?	10 m^2, 40 plants	≥ 1 kg	< 134
Grape	Europe	1999	Plot sprayer, knapsack Sprayer, motorized	40–60 m^2	≥ 1 kg	61–306
Grape	Europe	2004	knapsack Sprayer	13–90 m^2	≥ 1 kg	< 210
Grape	Germany/ France	2004	knapsack Sprayer	90 m^2	≥ 1 kg	< 195
Grape	Germany France Italy	2004	knapsack Sprayer	13–150 m^2	≥ 1 kg	< 326
Grape	USA	1984/86	Backpack sprayer, tractor mounted sprayer, boom sprayer	13–270 m^2, 3 plants	≥ 0.5 kg	< 450
Raspberry	Germany	1983	Plot sprayer	10–50 m^2	≥ 1 kg	375
Raspberry	Europe	2008	Knapsack sprayer	8.4–14 m^2	≥ 0.6 kg	< 377
Strawberry	Finland	1986	?	?	0.5 kg	384
Strawberry	Germany	1987	Plot sprayer, spray shield	42–80 m^2	1 kg	< 141
Strawberry	France	2005	Knapsack sprayer	18–80 m^2	≥ 1 kg	< 408
Strawberry	France	2005/06	Knapsack sprayer	20–50 m^2	≥ 0.5 kg	< 297
Carambola	Malaysia	1995	Hand operated knapsack sprayer	600–630 m^2	≥ 2 kg	156
Olive	Europe	2005	Knapsack sprayer/Spraying boom	63–120 m^2	≥ 1 kg	220–278
Olive	USA	2008	Backpack sprayer	141–428 m^2	≥ 1 kg	259
Banana	Brazil	1989	Plot sprayer	180 m^2	≥ 24 fruit	20
Banana	Brazil	2001	Plot sprayer	18–36 m^2	?	< 355
Banana	Brazil	2005	CO_2 Backpack Sprayer, Backpack Sprayer	45–50 m^2	?	< 118
Banana	Columbia/ Mexico/ Costa Rico/ Ecuador	1989	Knapsack sprayer	800–2000 m^2	≥ 20 fruit	Few months
Banana	Philippines	1984	?	20–994 m^2	≥ 20 fruit ≥1 kg	< 269
Avocado	Australia	1991	Plot sprayer	4 trees	≈2 kg	< 237
Avocado	Australia	1995/96	Plot sprayer, reverse decline	4 trees	≈2 kg	< 125
Guava	Malaysia	1995	Hand operated knapsack sprayer	800–1050 m^2	≈2 kg	< 109
Kiwifruit	Italy	2005	Knapsack sprayer	40–48 m^2	≥ 2 kg	272
Kiwifruit	USA	1989	CO_2 backpack sprayer	4.2–8.9 m^2	≥ 24 fruit	< 177
Mango	Australia	1990	Plot sprayer	60 m^2	≈2 kg	< 190
Mango	Australia	1994/95	hand held boom	4 trees	≈5 kg	< 412
Papaya	Australia	1996	hand held boom	4 trees	≈5 kg	< 22
Carrot	Germany	1984/85	?	11.5–48 m^2	≈1 kg	< 285
Carrot	Europe	1999	Plot sprayer	20–48 m^2	20–30 units, ≈1 kg	54–256
Carrot	Europe	2001	Plot sprayer	20–22.5 m^2	> 24 units	83–218
Potato	Brazil	2000	Backpack sprayer	34–36 m^2	?	150–209
Potato	Brazil	2006	Backpack sprayer	10 m^2	?	117–127
Potato	France	2002	Boom sprayer	15–90 m^2	> 24 units, ≥ 2 kg	239–304
Potato	Europe	2003	Boom sprayer	23–120 m^2	≥ 2 kg	< 339
Potato	Europe	2003	Boom sprayer	34–90 m^2	≥ 2 kg	< 371
Potato	Europe	2009	Boom sprayer	18–100 m^2	≥ 2 kg	118–312
Potato	USA	1997	Knapsack sprayer, Ground rig, boom sprayer	4 rows × 12 m—10 rows × 12 m	> 24 units	< 189
Sugar beet,	USA	1995	Ground rig, Backpack sprayer,	4 rows ×	≥ 1.4 kg	< 370

Crop	Location	Year	Sprayer	Plot size	Sample size	Sample to analysis interval (days)
transgenic			Bicycle sprayer	9.1 m—8 rows × 24 m		
Sugar beet, transgenic	USA	1996	Ground rig, Bicycle sprayer	4 rows × 9.1 m –8 rows × 18 m	≥ 1.4 kg	< 175
Onion, bulb	Germany	1984	?	10–48 m^2	≥ 1kg	< 575
Onion, bulb	Europe	2001	boom sprayer, plot sprayer	27.5–45 m^2	≥ 2 kg	293–322
Onion, bulb	Europe	2002	boom sprayer, plot sprayer	23–40 m^2	≥ 2 kg	314–378
Onion, bulb	Europe	2004	Knapsack sprayer	34–75 m^2	≥ 2 kg	< 365
Corn sweet tolerant	USA	1997/98	Backpack sprayer, Tractor mounted sprayer	28–260 m^2	≥ 12 ears	< 950
Corn sweet tolerant	USA	1999	Tractor mounted sprayer, ATV mounted sprayer, Backpack sprayer	45–186 m^2	≥ 12 ears	< 245
Lettuce, lambs	Germany	1987	?	30–100 m^2	≥ 0.5 kg	< 295
Lettuce	Brazil	2006	Backpack sprayer	12–15 m^2	?	< 132
Lettuce	Europe	1999	Plot sprayer, Motorised knapsack sprayer	22–40 m^2	≥ 12 heads	34–275
Lettuce	Europe	2001	Plot sprayer	22–56.3 m^2	≥ 12 heads, ≥ 1 kg	48–211
Lettuce	Europe	2008	Knapsack sprayer	30–120 m^2	Mature plants > 1 kg	164–378
Lettuce	Europe	2009	Knapsack sprayer	36–120 m^2	> 1 kg	166–310
Bean, kidney	Germany	1985/86	?	30–90 m^2	≥ 2 kg	< 630
Bean, kidney	Europe	2008/09	Knapsack sprayer	51–100 m^2	≥ 1 kg	262–413
Asparagus	Germany	1987	?	10–220 m^2	≥ 1 kg	132–149
Asparagus	Europe	2008	Knapsack sprayer	75–120 m^2	≥ 1 kg	353–433
Bean, dry	Brazil	2000	Backpack sprayer	60–100 m^2	?	165–231
Bean, dry	Brazil	2005	Backpack sprayer	15–30 m^2	?	294–323
Soya bean, dry	USA	1994	Backpack sprayer, Tractor mounted sprayer	4 rows × 10 m—8 rows × 12 m	≥ 1.8 kg	< 237
Soya bean, dry	USA	1995	backpack sprayer, plot sprayer, CO_2 boom	8 rows × 15 m—20 rows × 15 m	≥ 1.8 kg	< 210
Soya bean, dry	USA	2009	Tractor mounted sprayer, backpack sprayer, Kubola mounted sprayer,	67–294 m^2	≥ 1 kg	< 297
Rape	Germany	1985–1988	Plot sprayer	25–102 m^2	≥ 0.5 kg	< 608
Rape (canola) tolerant	USA	1999	Talley built sprayer, Compressor sprayer, Tractor mounted sprayer, Backpack sprayer	30 rows × 17m–40 rows × 30 m	≥ 1.8 kg	< 219
Cotton, tolerant	USA		Research sprayer, tractor sprayer, Weasley walker sprayer, Wylie boom sprayer, Backpack sprayer	93–1962 m^2	≥ 1.5 kg	< 156
Sunflower, tolerant	Germany	1985, 1987	?	30–200 m^2	≥ 1 kg	< 175
Sunflower, tolerant	Hungary	2006	Atomizer sprayer	200 m^2	≥ 1 kg	< 112
Maize, tolerant	USA	1993	Research sprayer, tractor mounted sprayer, backpack sprayer	28–186 m^2	≥ 0.9 kg	< 365
Maize, tolerant	USA	1994	Tractor mounted sprayer, Backpack sprayer, ground rig, Hip pack sprayer	6 rows × 6m—10 rows × 7.6 m	≥ 1.4 kg	< 292
Maize, tolerant	USA	1995	Tractor mounted sprayer, Backpack sprayer, ground rig	8 rows × 18 m—32 rows × 30 m	≥ 1.4 kg	< 210
Maize, tolerant	USA	1996/7	Tractor mounted, sprayer, plot sprayer, ground rig, backpack	8 rows × 9 m—4 rows ×	≥ 1.4 kg	< 430

Crop	Location	Year	Sprayer	Plot size	Sample size	Sample to analysis interval (days)
			sprayer	30 m		
Maize, tolerant	USA	2000	Tractor mounted sprayer, boom sprayer, plot sprayer	46–70 m^2	?	< 176
Rice, tolerant	USA	1996–1999	Backpack sprayer, plot sprayer, boom sprayer, tractor mounted sprayer	Row 12–21 m, 60–320 m^2	≥ 1.4 kg	< 306
Coffee	Brazil	2007	?	30–75 m^2	1 kg	< 218

Where duplicate field samples from an unreplicated plot were taken at each sampling time and were analysed separately, the mean of the two analytical results was taken as the best estimate of the residues in the plot and only the means are recorded in the tables. Similarly where samples were collected from replicate plots the mean result is reported (see general consideration JMPR 2010).

The method for calculating total residues for conventional and glufosinate-tolerant crops and is illustrated below:

Glufosinate	MPP	NAG	Total
< 0.05	< 0.05	< 0.05	< 0.05 or where LOQs differ, the highest LOQ
< 0.05	< 0.05	0.06	0.06
0.05	< 0.05	0.09	0.14
< 0.05	0.06	< 0.05	0.06

Table 70 Residues of glufosinate in citrus fruit (directed sprays for weed control)

CITRUS		Application						Residue (mg/kg)				
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Orange												
Altinopolis SP Brazil 2007 (Pera Rio)	200SL	1	0.40	300	80	fruit	40	< 0.03	< 0.04	[a]	< 0.04	RA-1262/07
Paulinia SP Brazil 2007 (Pera Rio)	200SL	1	0.40	300	79	Fruit	10 20 30 40 50	0.53 0.21 0.08 < 0.03 < 0.03	0.18 0.10 < 0.04 < 0.04 < 0.04	[a]	0.71 0.31 0.08 < 0.04 < 0.04	RA-1263/07
Genoves, Comunidad Valenciana Spain 1999 Salustiana	150SL	2 (29) 1.12 0.75		300 300	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS570
Moncada, Comunidad Valenciana, Spain, 1999 Navel	150SL	2 (28) 1.12 0.75		300 300	85	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS570
Castellaneta, Puglia Italy, 1999 Navelina	150SL	2 (29) 1.12 0.75		400 400	83	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS570
Marconia, Basilicata, Italy, 1999 Navelina	150SL	2 (29) 1.12 0.75		400 400	83	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS570
Scanzano Ionico, Basilicata, Italy, 1999 Washington	150SL	2 (29) 1.12 0.75		400 400	81	Fruit	0 7 14	< 0.05 < 0.05 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 0.05	ER99ECS570

CITRUS		Application						Residue (mg/kg)				
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Navel												
Moncada, Comunidad Valenciana Spain, 2000 Navel	150SL	2 (30)	1.12 0.75	300 300	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS570
Ireo – Argolida, Pelepones, Greece, 2000 San Loutsiana	150SL	2 (30)	1.12 0.75	300 300	81	Fruit	0 20	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS570
Marconia, Basilicata, Italy, 2000	150SL	2 (30)	1.12 0.75	300 300	83	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS570
Navelina	200SL	2 (30)	1.50 1.00	300 300	83	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS570
Tursi, Puglia, Italy 2000	150SL	2 (30)	1.13 0.75	300 300	83	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS570
Navelina	200SL	2 (30)	1.50 1.00	300 300	83	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS570
Oviedo FL USA 2008 Navel	200SL	3 (13 14)	1.69 1.67 1.65	282 278 276	81	Fruit	7 11 14 17 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	RAGLP024 +AMS/NIA
Oviedo FL USA 2008 Hamlin		3 (13 14)	1.71 1.67 1.68	284 278 280	81	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	
Groveland FL USA 2008 Hamlin	200SL	3 (14 14)	1.70 1.69 1.68	174 175 176	83	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Haines City FL USA 2008 Hamlin	200SL	3 (14 14)	1.72 1.66 1.68	168 152 156	83	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Ft Pierce FL USA 2008 Hamlin	200SL	3 (14 15)	1.70 1.70 1.74	210 218 228	89	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Vero Beach FL USA 2008 Hamlin	200SL	3 (14 15)	1.60 1.60 1.70	198 205 223	89	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Stuart FL USA 2008 Hamlin	200SL	3 (14 14)	1.72 1.69 1.69	216 191 194	83	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS
Stuart FL USA 2009 Pineapple	200SL	3 (14 14)	1.71 1.73 1.68	217 192 191	83	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	
Alamo TX USA 2008 N-33	200SL	3 (14 14)	1.70 1.75 1.72	189 195 191	83	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Sanger CA USA 2008 Washington Navel	200SL	3 (14 14)	1.67 1.73 1.67	175 182 170	83	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Porterville CA USA 2008 Washington	200SL	3 (13 15)	1.69 1.68 1.69	190 191 194	83	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA

CITRUS		Application						Residue (mg/kg)				
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Navel												
Mandarin												
Orlando CA USA 2008 Satsuma	200SL	3 (14 14)	1.68 1.69 1.68	187 186 187	81	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Lemon												
Ft. Pierce FL USA 2008 Eureka	200SL	3 (14 14)	1.70 1.69 1.66	187 217 222	81	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS
Fresno CA USA 2008 Meyer	200SL	3 (14 14)	1.68 1.69 1.68	256 258 256	89	Fruit	7 10 14 17 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	RAGLP024 +AMS
Sanger CA USA 2008 Lizbon 8A	200SL	3 (14 14)	1.70 1.69 1.68	283 282 280	83	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Arroyo Grande CA USA 2008 Lisbon	200SL	3 (14 13)	1.68 1.69 1.70	229 188 190	83	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Neman CA USA 2008 Lemonaria 8A	200SL	3 (14 14)	1.68 1.68 1.68	141 142 142	81	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS
Grapefruit												
Oviedo FL USA 2008 Flame	200SL	3 (13 14)	1.72 1.67 1.67	286 279 279	81	Fruit	7 11 14 17 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	RAGLP024 +AMS/NIA
Haines City FL USA 2008 Ruby Red	200SL	3 (14 14)	1.68 1.66 1.67	164 153 155	83	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Vero Beach FL USA 2008 Duncan	200SL	3 (14 15)	1.68 1.68 1.70	208 216 223	89	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA
Alamo TX USA 2008 Rio Red	200SL	3 (14 14)	1.68 1.74 1.72	187 194 191	83	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS
Neman CA USA 2008 Star Ruby	200SL	3 (14 14)	1.68 1.68 1.68	141 142 142	81	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS
Sanger CA USA 2008 Rio Red	200SL	3 (14 14)	1.67 1.74 1.70	176 183 173	83	fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP024 +AMS/NIA

[a] Analytical method measures NAG together with glufosinate as a common derivative

int = interval between sprays

Pome fruit

Table 71 Residues of glufosinate in apple fruit (directed sprays for weed control)

APPLES	Application							Residue (mg/kg)			
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference

APPLES	Application							Residue (mg/kg)			
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Vacaria RS Brazil 2006 Fuji	200SL	1	0.40	600	81	Fruit	0 3 5 7 10	0.69 0.12 0.05 < 0.03 < 0.03	< 0.04 0.11 < 0.04 < 0.04 < 0.04	0.73 0.23 0.05 < 0.04 < 0.04	RA-1091/06 [a]
		1	0.80	600	81	Fruit	7	< 0.03	< 0.04	< 0.04	
Caxias do Sul—RS Brazil 2006 Fuji	200SL	1	0.40	600	81	Fruit	0 3 5 7 10	0.64 0.10 0.04 < 0.03 < 0.03	< 0.04 0.08 < 0.04 < 0.04 < 0.04	0.68 0.18 0.04 < 0.04 < 0.04	RA-1092/06 [a]
		1	0.80	600	81	Fruit	7	< 0.03	< 0.04	< 0.04	
Frei Rodrigo	200SL	1	0.40	600	81	Fruit	7	< 0.03	< 0.04	< 0.04	RA-1093/06
— SC Brazil 2006 Gala/M.9/Marubakaido		1	0.80	600	81	Fruit	7	< 0.03	< 0.04	< 0.04	[a]
Bom Jardim	200SL	1	0.40	600	81	Fruit	7	< 0.03	< 0.04	< 0.04	RA-1094/06
da Serra—SC Brazil 2006 Gala		1	0.80	600	81	Fruit	7	< 0.03	< 0.04	< 0.04	[a]
Heidesheim Germany 1983 Golden	200SL	2 (49)	1.50 1.00	300 300	n.a.	Fruit	5 10 14	< 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06	DEU83H55826
Delicious		3 (77 49)	1.50 1.00 1.00	300 300 300	n.a.	Fruit	5 10 14	< 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06	
Langenau-Albeck Germany 1983 Golden Delicious	200SL	2 (45)	1.50 1.00	300 300	n.a.	Fruit	5 10 14	< 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06	DEU83H55831
Alfter Bornheim Germany 1983 Cox Orange	200SL	3 (42 46)	1.50 1.00 1.00	600 600 600	n.a.	Fruit	5 10 14	< 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06	DEU83H55821
Alfamen Zaragoza, Spain 1996 Golden	200SC	2 (60)	1.18 1.18	300 300	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER96ECS401
Smootee		3 (70 60)	0.59 0.59 0.59	300 300 300	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
La Almunia De Dona Godina, Zaragoza, Spain	200SC	2 (56)	1.18 1.18	300 300	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER96ECS401
1996 Golden 972		3 (74 56)	0.59 0.59 0.59	300 300 300	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Saint Pardoux, Poitou-Charéntes, France	200SC	2 (68)	1.18 1.18	250 250	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER96ECS401
S 1996 Golden		3 (49 68)	0.59 0.59 0.59	250 250 250	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Bologna, Emilia Romagna, Italy 1996 Nero	200SC	2 (61)	1.18 1.18	400 400	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER96ECS401
Red-Rome		3 (57 61)	0.59 0.59 0.59	400 400 400	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Bologna Emilia Romagna, Italy 1996 Top Red	200SC	2 (54)	1.18 1.18	400 400	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
		3 (57 54)	0.59 0.59 0.59	400 400 400	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	

APPLES	Application							Residue (mg/kg)			
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Lamagistére Midi Pyrénées, France S 1997	300SC	2 (33)	1.12 1.12	250 250	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER97ECN401
Golden Delicious		3 (86 33)	0.56 0.56 0.56	250 250 250	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
St. Nicolas de la Balerme Aquitaine,	300SC	2 (70)	1.12 1.12	250 250	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER97ECN401
France S 1997 Granny Smith		3 (78 70)	0.56 0.56 0.56	250 250 250	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Saint Vincent de Paul Aquitaine, France S 1999 Delbar Estival	150SL	2 (31)	1.12 0.75	250 250	81	Fruit	0 7 13	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS571
Korifi – Imathia Macedonia, Greece 1999 Gran	150SL	2 (30)	1.12 0.75	300 300	85	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS571
Smith	200SL	2 (30)	1.50 1.00	300 300	85	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Goumenissa—Pella, Macedonia, Greece 1999	150SL	2 (30)	1.12 0.75	300 300	85	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS571
Golden	200SL	2 (30)	1.50 1.00	300 300	85	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Gallo Di Poggio Renatico	150SL	2 (30)	1.12 0.75	400 400	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS571
Emilia Romagna, Italy 1999 Red Chief	200SL	2 (30)	1.50 1.00	400 400	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Arelho-Óbidos, Ribatejo e Oeste, Portugal 1999 Starking	150SL	2 (29)	1.12 0.75	300 300	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS571
Geisenheim, Hessen Germany 2000 Jonagold	200SL	2 (29)	1.50 1.00	300 300	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DR00EUN571
Swisttal—Miel, Nordshein-Westfalen Germany 2000 Elstar	200SL	2 (27)	1.50 1.00	300 300	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DR00EUN571
Wurzen-Roitzsch, Sachsen, Germany 2000 Rubin	200SL	2 (31)	1.50 1.00	300 300	81	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DR00EUN571
Boissy–L'allerie / Osny Ile-de-	150SL	2 (35)	1.12 0.75	250 250	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DR00EUN571
France, France N 2000 Golden Delicious	200SL	2 (35)	1.50 1.00	250 250	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Ecquevilly Ile-de-France	150SL	2 (30)	1.12 0.75	250 250	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DR00EUN571
France N 2000 Gala	200SL	2 (30)	1.50 1.00	250 250	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Doylestown PA USA	200SL	1	1.12	480	n.a.	Fruit	173	< 0.05	< 0.05	< 0.05	A48447
1983		1	2.24	480	n.a.	Fruit	173	< 0.05	< 0.05	< 0.05	
Storrs CT USA	200SL	2 (27)	1.68 1.68	370 370	n.a.	Fruit	77	< 0.05	< 0.05	< 0.05	A48447
1983 Empire		1	2.24	370	n.a.	Fruit	104	< 0.05	< 0.05	< 0.05	
Sunnyside WA USA 1983 Bisbee	200SL	3 (89 60)	1.12 1.12 1.12	338 338 338	n.a.	Fruit	77	< 0.05	< 0.05	< 0.05	A48447

APPLES	Application							Residue (mg/kg)			
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
		3 (89 60)	1.68 1.12 1.12	338 338 338	n.a.	Fruit	77	< 0.05	< 0.05	< 0.05	
		2 (89)	1.12 1.12	338 338	n.a.	Fruit	137	< 0.05	< 0.05	< 0.05	
		2 (89)	1.12 2.24	338 338	n.a.	Fruit	137	< 0.05	< 0.05	< 0.05	
Berrian Co MI USA 1984 Golden delicious	200SL	2 (57)	1.68 1.12	374 374	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	A48447
Berrian Co MI USA 1984 Golden delicious		2 (57)	3.36 2.24	374 374	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	
Berrian Co MI USA 1984 Jonathan		2 (57)	1.68 1.12	374 374	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	
Fletcher NC USA 1984 Red delicious	200SL	3 (43 41)	1.68 1.12 1.12	234 234 234	n.a.	Fruit	15	< 0.05	< 0.05	< 0.05	A48447
		3 (43 41)	3.36 2.24 1.12	234 234 234	n.a.	Fruit	15	< 0.05	< 0.05	< 0.05	
		3 (43 41)	1.68 1.12 1.12	234 234 234	n.a.	Fruit	15	< 0.05	< 0.05	< 0.05	
		3 (43 41)	3.36 2.24 1.12	234 234 234	n.a.	Fruit	15	< 0.05	< 0.05	< 0.05	
Ithica NY USA 1984	200SL	2 (44)	2.24 2.24	374 374	n.a.	Fruit	75	< 0.05	< 0.05	< 0.05	A48447
Cortland		2 (44)	4.48 4.48	374 374	n.a.	Fruit	75	< 0.05	< 0.05	< 0.05	
Cardiff NY USA 1984 Early Tydeman	200SL	2 (41)	2.24 2.24	711 711	n.a.	Fruit	25	< 0.05	< 0.05	< 0.05	A48447
Doylestown PA USA	200SL	2 (94)	2.24 1.12	281 281	n.a.	Fruit Fruit [b]	25	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A48447
1984 Golden delicious		3 (94 11)	4.48 2.24 2.24	281 281 281	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	
Sunnyside WA USA 1984 Bisbee	200SL	3 (54, 77)	1.68 1.12 1.12	299 299 299	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	A48447
		3 (54, 77)	3.36 2.24 2.24	299 299 299	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	
Kearneysville WV USA 1985 Golden delicious	200SL	3 (32 92)	2.24 2.24 2.24	267 267 267	n.a.	Fruit	14 27	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A48447
		3 (32 92)	4.48 4.48 4.48	267 267 267	n.a.	Fruit	14 27	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Woodburn OR USA 1985 Jonathan	200SL	3 (69 43)	1.68 1.68 1.68	374 374 374	n.a.	Fruit	13	< 0.05	< 0.05	< 0.05	A48447
		3 (69 43)	3.36 3.36 3.36	374 374 374	n.a.	Fruit	13	< 0.05	< 0.05	< 0.05	
		3 (69 43)	4.48 4.48 4.48	374 374 374	n.a.	Fruit	13	< 0.05	< 0.05	< 0.05	
Fresno CA USA 1985 Jonathan	200SL	3 (88 25)	2.24 2.24 2.24	281 281 281	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	A48447

APPLES	Application							Residue (mg/kg)			
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
		3 (8825)	4.48 4.48 4.48	281 281 281	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	
Blacksburg VA USA 1985 Golden delicious	200SL	3 (4649)	2.24 2.24 2.24	287 287 287	n.a.	Fruit	7	< 0.05	< 0.05	< 0.05	A48447
		3 (4649)	4.48 4.48 4.48	287 287 287	n.a.	Fruit	7	< 0.05	< 0.05	< 0.05	
		5 (43177 28)	4.48 4.48 4.48 4.48 4.48	287 287 287 287 287	n.a.	Fruit	28	< 0.05	< 0.05	< 0.05	
		3 (5457)	2.24 2.24 2.24	287 287 287	n.a.	Fruit	7	< 0.05	< 0.05	< 0.05	
		3 (5457)	4.48 4.48 4.48	287 287 287	n.a.	Fruit	7	< 0.05	< 0.05	< 0.05	

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Dropped fruit = fruit harvested from the ground

Table 72 Residues of glufosinate in pear fruit (directed sprays for weed control)

PEARS	Application						Residue (mg/kg)				
Location, year, variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
North Rose NY USA 2008 Bosc	3 (1212)	1.67 1.66 1.67	186 185 186	78	fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP027 + AMS
Live Oak CA USA 2008 Bartlett	3 (1414)	1.68 1.68 1.68	140 141 143	85	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP027 + AMS/NIA
Madera CA USA 2008 Asian	3 (1414)	1.69 1.70 1.70	235 236 236	77	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP027 + AMS
Buhl ID USA 2008 Bartlett	3 (1314)	1.71 1.71 1.69	198 196 205	79	Fruit	7 9 14 16 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	RAGLP027 + AMS
Ephrata WA USA 2008 Concord	3 (1414)	1.70 1.68 1.70	190 188 195	85	Fruit	14	< 0.05	0.08	< 0.05	0.08	RAGLP027 + AMS/NIA
Hood River OR USA 2008 Red d' Anjou	3 (1414)	1.69 1.62 1.69	224 176 199	81	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP027 + AMS

All applications were made with a 200 SL formulation

Table 73 Residues of glufosinate in stone fruit (directed sprays for weed control)

STONE	Application							Residue (mg/kg)				
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Apricot												

STONE	Application							Residue (mg/kg)				
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Marxheim Germany 1982 Ungarische Beste	200SL	2 (32)	1.065 1.065	300 300	78	Flesh	10 18 26 33	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 0.05 < 0.02		< 0.02 < 0.02 0.05 < 0.02	DEU82H53626
Heidesheim Germany 1982 Mombache	200SL	2 (32)	1.065 1.065	300 300	78	Flesh	11 17 25	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02		< 0.02 < 0.02 < 0.02	DEU82H53641
Cherry sour												
Hofheim-Diedenbergen Germany 1981 Schattenmorelle	200SL	2 (54)	1.065 1.065	300 300	n.a	Flesh	10 17 24 31	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 0.02 < 0.02		< 0.02 < 0.02 0.02 < 0.02	DEU81H52241
Hattersheim Germany 1987 Schattenmorelle	200SL	2 (60)	1.50 1.00	400 400	87	Flesh	5 10 14 21	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05 < 0.05	DEU87H43641
Kempton USA 2008 Montmorency	200SL	2 (28)	1.76 1.70	293 282	85	Fruit	12	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS/NIA
Simcoe Canada 2008 Montmorency	200SL	2 (28)	1.73 1.76	206 209	85	Fruit	13	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS/NIA
Perry USA 2008 Montmorency	200SL	2 (25)	1.73 1.70	207 211	85	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS
Cherry sweet												
Conklin USA 2008 Napoleon	200SL	2 (28)	1.71 1.68	220 228	76	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS
Marysville USA 2008 Lapin	200SL	2 (28)	1.68 1.68	186 187	77	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS/NIA
Mosier USA 2008 Lapin	200SL	2 (26)	1.68 1.69	191 227	81	Fruit	8 11 14 17 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	RAGLP026 +AMS
Peach/ Nectarine												
Itupeva SP Brazil 2008 Aurojima (Necatrine)	200SL	1	0.4	350	81	Fruit	0 3 5 7 10	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	[a]	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	BPL-JM-039-002-08
Porto Amazonas Brazil 2008 Sunripe (Nectarine)	200SL	1	0.4	350	87	Fruit	7	< 0.04	< 0.05	[a]	< 0.05	BPL-JM-039-002-08
Porto Amazonas Brazil 2008 Dourado	200SL	1	0.4	350	87	Fruit	7	< 0.04	< 0.05	[a]	< 0.05	
Jundiai SP Brazil 2008 Ouro Mel	200SL	1	0.4	350	81	Fruit	0 3 5 7 10	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	[a]	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	BPL-JM-039-003-08
Alfter Germany 1982 Amsden	200SL	2 (48)	1.065 1.065	300 300	78	Flesh	15 28 50	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02		< 0.02 < 0.02 < 0.02	DEU82H53521
Hofheim Germany 1982 Fruehe rote Ingelheimer	200SL	2 (48)	1.065 1.065	300 300	78	Flesh	11 16 21	< 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02		< 0.02 < 0.02 < 0.02	DEU82H53541

STONE	Application							Residue (mg/kg)				
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Villaverde del Rio, Andalucia, Spain 1999 Springcrest	150SL	2 (30)	1.12 0.75	300 300	75	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS572
Marmande, Aquitaine, France S 1999 Starlite	150SL	2 (29)	1.49 0.99	250 250	77	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS572
Gallo di Poggio Renatico, Emilia Romagna, Italy 1999 Duchessa d'Este	150SL	2 (30)	1.12 0.75	400 400	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS572
Salvaterra de Magos, Ribatejo e Oeste, Portugal 1999 Early Red Havan	150SL	2 (32)	1.12 0.75	250 250	77	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS572
Arelho-Óbidos, Ribatejo e Oeste, Portugal 1999 Catarino	150SL	2 (29)	1.12 0.75	300 300	81	Fruit	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05		< 0.05 < 0.05 < 0.05	ER99ECS572
Villaverde del Rio, Andalucia, Spain 2000 Springcrest	150SL	2 (32)	1.12 0.75	30 300	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS572
Castelculier Aquitaine, France S 2000 Orelie	150SL	2 (31)	1.12 0.75	250 250	85	Fruit	0 14	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS572
Masi Torello Emilia-Romagna, Italy 2000	150SL	2 (30)	1.12 0.75	300 300	85	Fruit	0 13	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	DR00EUS572
Paola Cavicchi	200SL	2 (30)	1.50 1.00	300 300	85	Fruit	0 13	< 0.05 < 0.05	< 0.05 < 0.05		< 0.05 < 0.05	
Almeirim Ribatejo e Oeste, Portugal 2000	150SL	2 (30)	1.12 0.75	300 300	78	Fruit	0 14	< 0.05 < 0.05	0.08 0.07		0.08 0.07	DR00EUS572
Baby Gold 9	200SL	2 (30)	1.50 1.00	300 300	78	Fruit	0 14	< 0.05 < 0.05	< 0.05 0.08		< 0.05 0.08	
Parlier CA USA 2007	120SL [b]	2 (28)	1.70 1.69	365 383	75–79	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	08720
Flavor Crest	120SL [c]	2 (28)	1.64 1.65	374 374	75–79	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	
95616 Davis CA USA 2007 Cling	120SL	2 (26)	1.74 1.66	224 215	75–79	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	08720
Holt MI USA 2007 Coral Star	120SL	2 (14)	1.66 1.65	374 365	75–79	Fruit	16	< 0.05	< 0.05	< 0.05	< 0.05	08720
Jackson Springs NC USA 2008 Contender	120SL	2 (29)	1.64 1.66	290 290	75–79	Fruit	16	< 0.05	< 0.05	< 0.05	< 0.05	08720
Bridgeton NJ USA 2007 Blake	120SL	2 (28)	1.60 1.60	206 206	75–79	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	08720
Lansing NY USA 2007 Harrow Beauty	120SL	2 (26)	1.68 1.69	290 299	75–79	Fruit	7	< 0.05	< 0.05	< 0.05	< 0.05	08720

STONE	Application							Residue (mg/kg)				
Location, year, variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Troy TN USA 2007 Red Skin	120SL	2 (27)	1.79 1.68	206 206	75–79	Fruit	15	< 0.05	< 0.05	< 0.05	< 0.05	08720
Fredericksburg TX USA 2007 Red Globe	120SL	2 (28)	1.68 1.68	234 234	75–79	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	08720
Plum												
Pouligny Saint Pierre, Centre, France N 2004 Opale	150SL	2 (30)	1.12 0.75	300 300	77	Fruit	0 14	< 0.01 < 0.01	< 0.01 < 0.01		< 0.01 < 0.01	RA-2073/04
Pobla del Duc Comunidad Valanciana, Spain 2004 Royal Dayamont	150SL	2 (31)	1.12 0.75	300 300	77	Fruit	0 15	< 0.01 < 0.01	< 0.01 < 0.01		< 0.01 < 0.01	RA-2074/04
Bombarral, Ribatejo e Oeste, Portugal 2004 Angelene	150SL	2 (29)	1.23 0.75	336 300	81	Fruit	0 17	< 0.01 < 0.01	< 0.01 < 0.01		< 0.01 < 0.01	RA-2074/04
Cambridge, Cambridgeshire, UK 2004 Edwards	200SL	2 (30)	1.50 1.00	300 300	85	Fruit	0 16	< 0.01 < 0.01	< 0.01 < 0.01		< 0.01 < 0.01	RA-2076/04
Bornheim, Nordheim Westfalen, Germany 2004 Ortenauer	200SL	2 (31)	1.50 1.00	300 300	85	Fruit	0 15	< 0.01 < 0.01	< 0.01 < 0.01		< 0.01 < 0.01	RA-2076/04
Monheim, Nordheim Westfalen, Germany 2007 Ersinger Frühzwetsche	200SL	2 (28)	1.50 1.00	300 300	85	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01		< 0.01 < 0.01 < 0.01 0.01	RA-2608/07
Tettnang, Baden Württemburg, Germany 2007 Presenta	200SL	2 (28)	1.59 1.00	300 300	85	Fruit	0 7 15 21	< 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01		< 0.01 < 0.01 < 0.01 0.01	RA-2608/07
Oosterblokker, Noord-Holland, Netherlands 2007 Reine Victoria	200SL	2 (27)	1.50 1.00	300 300	85	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01		< 0.01 < 0.01 < 0.01 0.01	RA-2608/07
Conklin USA 2008 Stanley	200SL	2 (28)	1.67 1.68	218 223	81	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS
Live Oak USA 2008 French	200SL	2 (28)	1.69 1.69	141 142	85	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS/NIA
Orland USA 2008 French	200SL	2 (28)	1.68 1.68	187 186	85	Fruit	14	< 0.05	0.07	< 0.05	0.07	RAGLP026 +AMS
Hickman USA 2008 Grand Rosa	200SL	2 (26)	1.70 1.67	283 278	85	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS
Fresno USA 2008 Flavor Rich	200SL	2 (28)	1.64 1.63	281 281	81	Fruit	7 10 14 17 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 0.05 < 0.05 < 0.05 0.06	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 0.05 < 0.05 < 0.05 0.06	RAGLP026 +AMS
Dallas USA 2008 Moyer	200SL	2 (28)	1.69 1.72	279 286	85	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP026 +AMS/NIA

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Trees 5–9 years old, applications made on 8 May and 5 June, no rain in the interval between application and harvest

[c] Trees 19 years old, application made on 15 May and 12 June, no rain in the interval between application and harvest

Table 74 Residues of glufosinate in blueberry (directed sprays for weed control)

BLUEBRRY	Application						Residue (mg/kg)			
Location, year, variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Bridgeton NJ USA 1997 Blue Ray and Duke	2 (25)	1.69 1.67	282 287	81	Fruit	13	0.06	< 0.05	0.06	05291
Dunham NH USA 1997 Nelson and Blue Ray	2 (29)	1.59 1.65	286 294	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	05291
Castle Hayne NC USA 1997 Croatan	2 (29)	1.70 1.67	188 185	79	Fruit	15	< 0.05	< 0.05	< 0.05	05291
Castle Hayne NC USA 1997 Croatan	2 (26	1.71 1.66	191 185	71-79	Fruit	15	< 0.05 c0.07	< 0.05	< 0.05 c0.07	05291
East Lansing MI USA 1997 Jersey	2 (28)	1.68 1.70	187 189	79	Fruit	14	< 0.05	< 0.05	< 0.05	05291
Onondaga MI USA 1997 Blue Crop	2 (26)	1.71 1.71	191 188	81	Fruit	13	< 0.05	< 0.05	< 0.05	05291

All applications were made with a 120 SL formulation

Table 75 Residues of glufosinate in currants (directed sprays for weed control)

CURRANTS	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Currant black										
Moorende Germany 1982 Silvergieters	2 (38)	1.00 1.00	300 300	n.a	Fruit	21	< 0.02	< 0.02	< 0.02	DEU82H52411
Bornheim Germany 1982 Silvergieters	2 (48)	1.00 1.00	300 300	n.a	Fruit	3 8 15 28	0.03 < 0.02 0.03 < 0.02	0.07 0.12 0.12 0.48	0.10 0.12 0.15 0.48	DEU82H52421
Langenau-Albeck Germany 1982 Silvergieters	2 (28)	1.00 1.00	300 300	n.a	Fruit	7 11 18 28	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 0.12 0.05	< 0.02 < 0.02 0.12 0.05	DEU82H52431
Hattersheim Germany 1982 Silvergieters	2 (40)	1.00 1.00	300 300	n.a	Fruit	4 10 15 21	< 0.02 < 0.02 < 0.02 < 0.02	0.05 < 0.02 < 0.02 < 0.02	0.05 < 0.02 < 0.02 < 0.02	DEU82H52441
Burscheid, Nordrheim Westfalen Germany 2005 Ben Lemond	2 (31)	1.50 1.00	300 300	81	Fruit	0 7 14 21	0.06 < 0.01 0.02 < 0.01	0.02 0.01 0.02 0.03	0.08 0.01 0.03 0.03	RA-2080/05 [a]
Noordbroek, Groningen, Netherlands 2005 Ben Alder	2 (30)	1.50 1.00	300 300	85	Fruit	0 7 14 21	2.3 0.96 0.90 0.79	0.09 0.12 0.11 0.14	2.4 1.1 1.0 0.93	RA-2080/05 [a]
Monheim, Nordrheim Westfalen, Germany 2005 Titania	2 (28)	1.50 1.00	300 300	81	Fruit	0 7 14 21	0.40 0.28 0.19 0.01	0.03 0.04 0.06 0.04	0.43 0.32 0.25 0.05	RA-2080/05 [a]
Colchester United, Essex, Kingdom 2005 Ben Hope	2 (30)	1.59 1.00	318 300	85	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	RA-2080/05 [a]

CURRANTS	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Morancé, Rhone-Alpes, France S 2008 Noir de Bourggne	3 (28 28)	0.80 0.75 0.75	319 300 300	85	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	08-2125 [a]
Sablons, Rhone-Aples, France S 2008 Noir de Bourgogne	3 (28 28)	0.75 0.75 0.75	300 300 300	85	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 < 0.01	< 0.01 < 0.01 0.01 < 0.01	08-2125 [a]
Currant red										
Dardilly, Rhone-Aples, France S 2008 Jenifer	3 (28 23)	0.75 0.75 0.75	300 300 300	85	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 0.02 0.02	0.02 0.01 0.02 0.02	08-2125 [a]
Noziere, Rhone-Alpes, France S 2008 Rovada	3 (28 28)	0.75 0.75 0.75	300 300 300	81	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	08-2125 [a]

All applications were made with a 200 SL formulation

[a] Application to soil in-between rows, spray shield used to minimise contamination of crop

Table 76 Residues of glufosinate in gooseberries (directed sprays for weed control)

GOOSEBERRY	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Hoopte, Winsen-Luhe Germany 1983	2 (43)	1.50 1.50	300 300	71	Fruit	4 9 13 20	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	DEU83H52311
Alfter Germany 1983 Weisse Triumphbeere	2 (54)	1.50 1.50	300 300	71	Fruit	5 10 14 21	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	DEU83H52321
Nordenstadt Germany 1983 Rote Triumph	2 (11)	1.50 1.50	300 300	71	Fruit	5 10 14 21	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	DEU83H52341

All applications were made with a 200 SL formulation

Table 77 Residues of glufosinate in grapes (directed sprays for weed control)

GRAPES	Application							Residue (mg/kg)			
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Paulinia, Brazil 2006 Maria	200SL	1	0.40	600	79	Fruit	0 7 14	0.82 < 0.03 < 0.03	< 0.04 < 0.04 < 0.04	0.82 < 0.04 < 0.04	RA-1095/06 [a]
		1	0.80	600	79	Fruit	7	< 0.03	< 0.04	< 0.04	
Londrina, Brazil 2006 Benefuji	200SL	1	0.40	600	79	Fruit	0 7 14	0.74 < 0.03 < 0.03	< 0.04 < 0.04 < 0.04	0.74 < 0.04 < 0.04	RA-1096/06 [a]
		1	0.80	600	79	Fruit	7	< 0.03	< 0.04	< 0.04	
Vacaria,	200SL	1	0.40	600	79	Fruit	7	< 0.03	< 0.04	< 0.04	RA-1097/06
Brazil 2006 Benefuji		1	0.80	600	79	Fruit	7	< 0.03	< 0.04	< 0.04	[a]
Jundiai, Brazil	200SL	1	0.40	600	81	Fruit	14	< 0.03	< 0.04	< 0.04	RA-1098/06
2006 Niagara Rosada		1	0.80	600	81	Fruit	14	< 0.03	< 0.04	< 0.04	[a]

GRAPES	Application							Residue (mg/kg)			
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Marintal Germany 1983 Spaet-burgunder	200SL	3 (82 67)	1.06 1.06 1.06	600 600 600	–	Bunch	0 5 10 14	0.06 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.06 < 0.02 < 0.02 < 0.02	DEU83H54121
Winningen Germany 1983 Riesling	200SL	2 (35)	1.60 1.06	600 600	–	Bunch	0 5 10 14	0.03 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	0.03 < 0.02 < 0.02 < 0.02	DEU83H54122
Duttweiler Germany 1983 Mueller-Thurgau	200SL	2 (47)	1.60 1.06	1000 1000	–	Bunch	0 5 10 14	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 < 0.02	DEU83H54141
Dois Portos, Ribarejo e Oeste, Portugal 1999 Periquita	150SL	2 (30)	1.12 0.75	300 300	85	Bunch	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS573
Sasso Morelli – Imola, Emilia Romagna, Italy	150SL	2 (30)	1.12 0.75	400 400	85	Bunch	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS573
1999 Trebbiano	200SL	2 (30)	1.50 1.00	400 400	85	Bunch	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
N.Risio Macedonia, Greece 1999	150SL	2 (30)	1.12 0.75	300 300	85	Bunch	0 7 14	< 0.05 0.13 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 0.13 < 0.05	ER99ECS573
Rosaki	200SL	2 (30)	1.50 1.00	300 300	85	Bunch	0 7 14	< 0.05 0.08 0.12	< 0.05 < 0.05 < 0.05	< 0.05 0.08 0.12	
VillenaComunidad Valenciana, Spain 1999 Monastrell	150SL	2 (32)	1.12 0.75	300 300	85	Bunch	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	ER99ECS573
Goumenissa – Kilkis, Macedonia,	150SL	2 (30)	1.12 0.75	300 300	85	Bunch	0 7 14	0.13 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.13 < 0.05 < 0.05	ER99ECS573
Greece 1999 Xonomavro	200SL	2 (30)	1.50 1.00	300 300	85	Bunch	0 7 14	< 0.05 0.09 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 0.09 < 0.05	
Neustadt–Mußbach	200SL	2 (30)	1.5 1	300 300	85	Bunch Bunch	0 14	< 0.01 < 0.01	0.02 0.02	0.02 0.02	RA-2081/04
Germany 2004 Riesling						Berry	14	< 0.01	0.02	0.02	
Bléré France 2004	200SL	2 (30)	1.5 1	300 300	85	Bunch Bunch	0 14	< 0.01 < 0.01	0.05 0.08	0.05 0.08	RA-2081/04
Cabernet						Berry	14	< 0.01	0.06	0.06	
Bléré France 2004		2 (30)	1.5 1	300 300	85	Bunch Bunch	0 14	< 0.01 < 0.01	0.02 < 0.01	0.02 < 0.01	
Sauvignon						Berry	14	< 0.01	< 0.01	< 0.01	
Laudun, Languedoc Roussillon, France 2004	150SL	2 (30)	1.12 0.75	300 300	85	Bunch Bunch	0 14	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	RA-2082/04
Merlot; red variety						Berry	14	< 0.01	< 0.01	< 0.01	
Lozanne, Rhone-Alpes, France 2004	150SL	2 (29)	1.12 0.75	300 300	85	Bunch Bunch	0 14	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	RA-2082/04
Gamay; red variety						Berry	14	< 0.01	< 0.01	< 0.01	
San Prospero, Emilia Romagna, Italy 2004	150SL	2 (30)	1.12 0.75	300 300	85	Bunch Bunch	0 14	< 0.01 < 0.01	< 0.01 < 0.01	< 0.01 < 0.01	RA-2082/04

GRAPES	Application							Residue (mg/kg)			
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Lambrusco di Sorbara; red variety						Berry	14	< 0.01	< 0.01	< 0.01	
Bléré France 2005 Cabernet franc; red variety	200SL	2 (31)	1.5 1	300 300	85	Bunch	0 14 21	0.03 < 0.01 < 0.01	0.01 0.02 0.02	0.04 0.02 0.02	RA-2082/05
Berg / Kressbronn Germany 2005 Blauer Spätburgunder; red variety	200SL	2 (29)	1.5 1	300 300	85	Bunch	0 14 21	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01	RA-2082/05
Lodi USA 1984 Chemin	200SL	2 (70)	1.68 1.12	252 252	n.a.	Fruit	37	< 0.05	< 0.05	< 0.05	A48458
blanc		2 (70)	3.36 2.24	252 252	n.a.	Fruit	37	< 0.05	< 0.05	< 0.05	
Lockford USA 1984 Petite Sirah	200SL	2 (104)	1.68 1.12	252 252	89	Fruit	15	< 0.05	< 0.05	< 0.05	A48458
		2 (104)	3.36 2.24	252 252	89	Fruit	15	< 0.05	< 0.05	< 0.05	
Courtland USA 1984 Merlot	200SL	2 (128)	1.68 1.12	280 280	n.a.	Fruit	31	< 0.05	< 0.05	< 0.05	A48458
		2 (128)	3.36 2.24	280 280	n.a.	Fruit	31	< 0.05	< 0.05	< 0.05	
Berrien County USA	200SL	2 (57)	1.68 1.12	374 374	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	A48458
1984 Niagara		2 (57)	3.36 2.24	374 374	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	
Fredonia USA 1984 Concord	200SL	2 (37)	2.24 2.24	1871 1871	n.a.	Fruit	84	< 0.05	< 0.05	< 0.05	A48458
		2 (37)	4.48 2.24	1871 1871	n.a.	Fruit	84	< 0.05	< 0.05	< 0.05	
Doylestown USA 1984	200SL	2 (99)	2.24 1.12	281 281	89	Fruit	14	< 0.05	< 0.05	< 0.05	A48458
Concord		2 (99)	4.48 2.24	281 281	89	Fruit	14	< 0.05	< 0.05	< 0.05	
		3 (99 25)	2.24 1.12 1.12	281 281 281	89	Fruit	14	< 0.05	< 0.05	< 0.05	
North East USA 1984	200SL	2 (30)	2.24 2.24	374 374	n.a.	Fruit	87	< 0.05	< 0.05	< 0.05	A48458
Concord		2 (30)	4.48 4.48	374 374	n.a.	Fruit	87	< 0.05	< 0.05	< 0.05	
Sunnyside USA 1984 Concord	200SL	3 (54 77)	3.36 2.24 2.24	299 299 299	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	A48458
		3 (54 77)	1.68 1.12 1.12	299 299 299	n.a.	Fruit	14	< 0.05	< 0.05	< 0.05	
Courtland USA 1985 French Columbard	200SL	3 (98 102)	2.24 2.24 2.24	373 373 373	79	Fruit	14	< 0.05	< 0.05	< 0.05	A48458
		3 (98 102)	4.48 4.48 4.48	373 373 373	79	Fruit	14	< 0.05	< 0.05	< 0.05	
		3 (98 102)	1.68 1.68 1.68	373 373 373	79	Fruit	14	< 0.05	< 0.05	< 0.05	
Haslett USA 1985 Concord	200SL	3 (98 102)	2.24 2.24 2.24	334 334 334	83	Fruit	14	< 0.05	0.09	0.09	A48458

GRAPES	Application							Residue (mg/kg)			
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
		3 (98 102)	4.48 4.48 4.48	334 334 334	83	Fruit	14	< 0.05	0.06	0.06	
Fresno USA 1986 Thomson	200SL	5 (12 16 14 14)	4.48 4.48 4.48 4.48 4.48	373 373 373 373 373	n.a.	Fruit	28	< 0.05	< 0.05	< 0.05	A48458
		5 (12 16 14 14)	4.48 4.48 4.48 4.48 4.48	373 373 373 373 373	n.a.	Fruit	28	< 0.05	< 0.05	< 0.05	

All applications were made with a 150 SL formulation

[a] Analytical method measures NAG together with glufosinate as a common derivative

Table 78 Residues of glufosinate in raspberry (directed sprays for weed control)

RASPBERRY	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Kriftel Germany 1983 CV2	2 (45)	1.5 1.5	300 300	71	Fruit	4 9 14 18	< 0.02 < 0.02 < 0.02 0.03	< 0.02 < 0.02 < 0.02 < 0.02	< 0.02 < 0.02 < 0.02 0.03	A58097
Hoopte, Winsen-Luhe Germany 1983 Schoenemanns	2 (74)	1.5 1.5	300 300	71	Fruit	4 9 13 20	< 0.02 0.04 < 0.02 < 0.02	< 0.02 < 0.02 0.02 < 0.02	< 0.02 0.04 0.02 < 0.02	A58098
Saint Maurice l'Exil, Rhone-Alpes, France S 2008 Galion	3 (30 26)	0.79 0.75 0.75	321 300 300	87	Fruit	0 7 15 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	08-2124 [a]
Dardilly, Rhone-Aples France S 2008 Mecker	3 (29 27)	0.75 0.75 0.75	300 300 300	89	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.03 0.02	0.02 0.02 0.03 0.02	08-2124 [a]
Viarago Pergine Valsugana, Trentino-Alto Adige, Italy 2008 Tulameen	3 (28 28)	0.75 0.75 0.75	300 300 300	81	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	08-2124 [a]
Viarago Pergine Valsugana, Trentino-Alto Adige, Italy 2008 Eritas	3 (28 28)	0.75 0.75 0.75	300 300 300	81	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	0.03 0.02 0.01 0.01	0.03 0.02 0.01 0.01	

All applications were made with a 200 SL formulation

[a] Application to soil in-between rows, spray shield used to minimise contamination of crop

Table 79 Residues of glufosinate in strawberry (directed sprays for weed control)

STRAWBERRY	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	Ll/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Piikkioe Finland 1986 Halla	1	1.00	400	n.a.	Fruit	33	< 0.05	< 0.05	< 0.05	A36138

STRAWBERRY	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	Ll/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Elbstorf Germany 1987 Asrieta	1	0.80	300	61	Fruit	43 54 61	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A58099
Alfter Germany 1987 Tago	1	0.80	300	60	Fruit	55 62 69	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A58100
Aichach Germany 1987 Senga Sengana	1	0.80	300	61	Fruit	42 55 63	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A58101
Eddersheim Germany 1987 Korona	1	0.80	300	60	Fruit	40 47 54	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A58102
Courzieu,	1	0.75	300	89	Fruit	4	< 0.01	< 0.01	< 0.01	RA-2078/04
Rhone-Alpes, France S 2004	2 (101)	0.75 0.75	300 300	89	Fruit	4	0.03	< 0.01	0.03	
Mara des bois	3 (101 25)	0.75 0.75 0.75	300 300 300	89	Fruit	4	0.02	< 0.01	0.02	
	4 (101 25 22)	0.75 0.75 0.75 0.75	300 300 300 300	89	Fruit	4	0.15	< 0.01	0.15	
Limas,	1	0.75	300	89	Fruit	4	0.02	< 0.01	0.02	RA-2078/05
Rhone-Alpes, France S 2005	2 (52)	0.750.75	300 300	89	Fruit	4	0.01	< 0.01	0.01	RA-2078/05
Mara des bois	3 (52 53)	0.75 0.75 0.75	300 300 300	89	Fruit	4	0.01	< 0.01	0.01	RA-2078/05
	4 (52 53 21)	0.75 0.75 0.75 0.75	300 300 300 300	89	Fruit	4	0.01	< 0.01	0.01	RA-2078/05
Beauregard et	1	0.75	300	87	Fruit	4	< 0.01	0.02 c0.03	0.02 c0.03	RA-2078/05
Bassac, Aquitaine,	2 (46)	0.75 0.75	300 300	87	Fruit	4	< 0.01	< 0.01	< 0.01	
France S 2005 Charlotte Beaugier	3 (46 39)	0.75 0.75 0.75	300 300 300	87	Fruit	4	0.03	< 0.01	0.03	
	4 (46 39 21)	0.75 0.75 0.75 0.75	300 300 300 300	87	Fruit	4	< 0.01	< 0.01	< 0.01	
Saint Genis	1	0.75	300	89	Fruit	4	< 0.01	< 0.01	< 0.01	RA-2078/05
l'Argentière, Rhone-Alpes,	2 (49)	0.75 0.80	300 318	89	Fruit	4	0.02	< 0.01	0.02	RA-2078/05
France S 2005 Mara des bois	3 (49 45)	0.75 0.80 0.75	300 318 300	89	Fruit	4	< 0.01	< 0.01	< 0.01	RA-2078/05
	4 (49 45 21)	0.75 0.80 0.75 0.75	300 318 300 300	89	Fruit	4	0.01	< 0.01	0.01	RA-2078/05
St Romain de	1	0.75	300	87	Fruit	4	0.01	0.04 c0.04	0.05 c0.04	RA-2078/05
Monpazier Aquitaine,	2 (25)	0.75 0.75	300 300	87	Fruit	4	0.05	0.01	0.06	
France S 2005 Elsinore	3 (25 22)	0.75 0.75 0.75	300 300 300	87	Fruit	4	0.02	< 0.01	0.02	

STRAWBERRY	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	Ll/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	4 (25 22 20)	0.75 0.75 0.75 0.75	300 300 300 300	87	Fruit	4	< 0.01	< 0.01	< 0.01	
Reynies, Midi	1	0.75	300	89	Fruit	4	< 0.01	< 0.01	< 0.01	RA-2520/06
Pyrénées, France S 2006	2 (38)	0.75 0.75	300 300	89	Fruit	4	< 0.01	< 0.01	< 0.01	[a]
Mathys	4 (38 20 21)	0.75 0.75 0.75 0.75	300 300 300 300	89	Fruit	4	0.02	< 0.01	0.02	
Dommartin	1	0.75	300	89	Fruit	4	< 0.01	< 0.01	< 0.01	RA-2520/06
Rhone-Alpes, France S 2006	2 (70)	0.75 0.75	300 300	89	Fruit	4	< 0.01	< 0.01	< 0.01	[a]
Mara des bois	3 (70 21)	0.75 0.75 0.75	300 300 300	89	Fruit	4	< 0.01	< 0.01	< 0.01	
	4 (70 21 22)	0.75 0.75 0.75 0.75	300 300 300 300	89	Fruit	0 2 4 6	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	
Castelculier	1	0.75	300	87	Fruit	4	< 0.01	< 0.01	< 0.01	RA-2520/06
Aquitaine, France S 2006 Gariguette	2 (29)	0.75 0.75	300 300	87	Fruit	4	< 0.01	< 0.01	< 0.01	[a]

All applications were made with a 200 SL formulation for the Finland/Germany trials and a 150 SL formulation for the France trials

[a] Application to soil in-between rows, spray shield used to minimise contamination of crop

Table 80 Residues of glufosinate in carambola (directed sprays for weed control)

CARAMBOLA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Jenderam, Ulu Malaysia 1995 B10	3 (43 55)	0.50 0.50 0.50	450 450 450	n.a.	Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	A57295
	3 (43 55)	1 1 1	450 450 450	n.a.	Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	A57295
Bidor Malaysia 1995 B10	3 (41 42)	0.50 0.50 0.50	450 450 450	n.a.	Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	A57295
	3 (41 42)	1 1 1	450 450 450	n.a.	Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	A57295

All applications were made with a 150 SL formulation

Table 81 Residues of glufosinate in olives (directed sprays for weed control)

OLIVE	Application			Residue (mg/kg)	

Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Avlona, Central Greece, Greece 2005 Megaritiki	2 (35)	1.12 0.75	300 300	87	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01		< 0.01 < 0.01 < 0.01 < 0.01	RA-2133/05
Santarém, Ribatejo e Oeste, Portugal 2005 Galega	2 (30)	1.12 0.75	300 300	85	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	0.01 < 0.01 0.01 0.01		0.01 < 0.01 < 0.01 < 0.01	RA-2133/05
St. Llorenç d'Hortons, Cataluña, Spain 2005 Bacarut	2 (37)	1.12 0.75	300 300	81	Fruit	0 7 14 19	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01		< 0.01 < 0.01 < 0.01 < 0.01	RA-2133/05
Biancavilla, Sicilia, Italy 2005 Moresca	2 (29)	1.12 0.75	300 300	79	Fruit	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.01 0.02 0.03		0.02 0.01 0.02 0.03	RA-2133/05
Artios, CA USA 2008 Manzanillo	3 (14 14)	1.50 1.50 1.50	142 141 140	87	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP028 +AMS
Porterville, CA USA 2008 Manzanillo	3 (14 14)	1.53 1.50 1.49	202 205 199	87	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP028 +AMS/NIA
Cholame, CA USA 2008 Mission	3 (14 14)	1.50 1.50 1.51	185 181 161	89	Fruit	14	< 0.05	< 0.05	< 0.05	< 0.05	RAGLP028 +AMS/NIA

All applications were made with a 150 SL formulation for the Europe trials and a 200 SL formulation for the USA trials

Table 82 Residues of glufosinate in avocado (directed sprays for weed control)

AVOCADO	Application						Residue (mg/kg)			
country, year (variety)	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Rochedale	1	1.0	250	89	Fruit	21	< 0.09	< 0.12	< 0.12	A59027
Australia				89	Fruit	12	< 0.09	< 0.12	< 0.12	
1991 Sharwil				89	Fruit	7	< 0.09	< 0.12	< 0.12	
	1	2.0	250	89	Fruit	21	< 0.09	< 0.12	< 0.12	
				89	Fruit	12	< 0.09	< 0.12	< 0.12	
				89	Fruit	7	< 0.09	< 0.12	< 0.12	
Childers Australia 1995 Haas	2 (52)	1.2 1.2	173 200	89	Fruit	0 14 24 31	< 0.05 < 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06 < 0.06	A59025
	2 (52)	2.4 2.4	173 200	89	Fruit	0 14 24 31	< 0.05 < 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06 < 0.06	
Woombye Australia 1996 Fuerte	2 (37)	1.2 1.2	180 180	89	Fruit	0 14 24 28	< 0.05 < 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06 < 0.06	A59026
	2 (37)	2.4 2.4	180 180	89	Fruit	0 14 24 28	< 0.05 < 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06 < 0.06	

All applications were made with a 200 SL formulation

Table 83 Residues of glufosinate in banana (directed sprays for weed control)

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Eldorado Brazil 1989 Nanica	4 (34 28 33)	0.4 0.4 0.4	500 500 500	n.a	Pulp	11 21 31	< 0.05 < 0.05 < 0.05	< 0.05< 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A41975 [a]
		0.4	500		Peel	11 21 31	< 0.05 < 0.05 < 0.05	< 0.05< 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
	4 (34 28 33)	0.8 0.8 0.8	500 500 500	n.a	Pulp	11 21 31	< 0.05 < 0.05 < 0.05	< 0.05< 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A41976 [a]
		0.8	500		Peel	11 21 31	< 0.05 < 0.05 < 0.05	< 0.05< 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Caconde	1	0.3	300	79	Fruit	7	< 0.05	< 0.06	< 0.06	ER02BRAH13-1
Brazil 2001 Prata	1	0.6	300	79	Fruit	7	< 0.05	< 0.06	< 0.06	[a]
Paulinia Brazil	1	0.3	300	86	Fruit	7	< 0.05	< 0.06	< 0.06	ER02BRAH13-2
2001 Nanica	1	0.6	300	86	Fruit	7	< 0.05	< 0.06	< 0.06	[a]
Tapirai Brazil	1	0.3	300	86	Fruit	7	< 0.05	< 0.06	< 0.06	ER02BRAH13-3
2001 Nanica	1	0.6	300	86	Fruit	7	< 0.05	< 0.06	< 0.06	[a]
2005 Nanica	1	0.3	600	79	Fruit	10	< 0.03	< 0.04	< 0.04	RA-1040/06
MG Brazil 2005 Nanica	1	0.6	600	79	Fruit	10	< 0.03	< 0.04	< 0.04	[a]
Rio Verde GO	1	0.4	600	79	Fruit	10	< 0.03	< 0.04	< 0.04	RA-1041/06
Brazil 2005 Nanica	1	0.8	600	79	Fruit	10	< 0.03	< 0.04	< 0.04	[a]
Paulinia—SP Brazil 2005 Nanica	1	0.4	600	79	Fruit	0 3 7 10 15	0.49 0.06 0.03 < 0.03 < 0.03	< 0.04 0.05 < 0.04 < 0.04 < 0.04	0.49 0.11 0.03 < 0.04 < 0.04	RA-1042/06 [a]
	1	0.8	600	79	Fruit	10	< 0.03	< 0.04	< 0.04	
Jundiai—SP Brazil 2005 Nanica	1	0.4	600	79	Fruit	0 3 7 10 15	0.53 0.08 0.04 < 0.03 < 0.03	< 0.04 0.06 < 0.04 < 0.04 < 0.04	0.53 0.14 0.04 < 0.04 < 0.04	RA-1043/06 [a]
	1	0.8	600	79	Fruit	10	< 0.03	< 0.04	< 0.04	
Cienaga Colombia 1987 Valery	4 (60 63 62)	0.6 0.6 0.6	300 300 300	n.a.	Pulp	4 35 62	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A46001
		0.6	300		Peel	4 35 62	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
	6 (60 63 62	0.6 0.6 0.6	300 300 300	n.a.	Pulp	30 84 155	< 0.05 < 0.05 < 0.05	0.05 < 0.05 < 0.05	0.05 < 0.05 < 0.05	
	69 59)	0.6 0.6 0.6	300 300 300		peel	30 84 155	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
	4 (60 63 62)	1.2 1.2 1.2	300 300 300	n.a.	Pulp	4 35 62	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 0.08	< 0.05 < 0.05 0.08	A46001
		1.2	300		peel	4 35 62	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	6 (60 63 62	1.2 1.2 1.2	300 300 300	n.a.	Pulp	30 84 155	< 0.05 < 0.05 < 0.05	0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
	69 59)	1.2 1.2 1.2	300 300 300		Peel	30 84 155	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Predio "El Vivero" Mexico 1987	4 (59 62 60)	1.00 × 4	230 × 4	n.a.	Pulp	8 36 63	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A46002
Enano Gigante					peel	8 36 63	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
	6 (59 62 60	1.00 × 5 0.60	230 × 6	n.a.	Pulp	21 49 76	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
	65 61)	× 1			Peel	21 49 76	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
	7 (59 62 60 65	1.00 × 5 0.60 × 2	230 × 7	n.a.	Pulp	7 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	61 88)				peel	7 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	8 (59 62 60 65 61)	1.00 × 5 0.60 × 3	230 × 7 200 × 1	n.a.	Pulp	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	88 59				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	9 (59 62 60 65 61 88	1.00 × 5 0.60 × 4	230 × 7 200 × 2	n.a.	Pulp	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	59 63)				peel	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	10 (59 62 60 65 61 88 59	1.00 × 5 0.60 × 5	230 × 7 200 × 3	n.a.	Pulp	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	63 56)				peel	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	11 (59 62 60 65 61 88 59 63	1.00 × 5 0.60 × 6	230 × 7 200 × 4	n.a.	Pulp	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56 56)				peel	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	12 (59 62 60 65 61 88 59 63 56	1.00 × 5 0.60 × 7	230 × 7 200 × 5	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56 56)				peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	13 (59 62 60 65 61 88 59 63 56 56	1.00 × 5 0.60 × 8	230 × 7 200 × 6	n.a.	Pulp	8 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	56 56)				peel	8 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	4 (59 62 60)	2.00 × 4	230 × 4	n.a.	Pulp	8 36 63	< 0.05 < 0.05 < 0.05	< 0.05 0.08 < 0.05	< 0.05 0.08 < 0.05	A46002
					peel	8 36 63	< 0.05 < 0.05 < 0.05	< 0.05 0.06 < 0.05	< 0.05 0.06 < 0.05	
	6 (59 62 60	2.00 × 5 1.20	230 × 6	n.a.	Pulp	21 49 76	< 0.05 < 0.05 < 0.05	0.07 0.10 < 0.05	0.07 0.10 < 0.05	
	65 61)	× 1			peel	21 49 76	< 0.05 < 0.05 < 0.05	0.05 0.07 < 0.05	0.05 0.07 < 0.05	
	7 (59 62 60 65	2.00 × 5 1.20 × 2	230 × 7	n.a.	Pulp	7 58	< 0.05 < 0.05	< 0.05< 0.05	< 0.05 < 0.05	
	61 88)				peel	7 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	8 (59 62 60 65 61)	2.00 × 5 1.20 × 3	230 × 7 200 × 1	n.a.	Pulp	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	88 59				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	9 (59 62 60 65 61 88	2.00 × 5 1.20 × 4	230 × 7 200 × 2	n.a.	Pulp	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	59 63)				peel	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	10 (59 62 60 65 61 88 59	2.00 × 5 1.20 × 5	230 × 7 200 × 3	n.a.	Pulp	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	63 56)				peel	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	11 (59 62 60 65 61 88 59 63	2.00 × 5 1.20 × 6	230 × 7 200 × 4	n.a.	Pulp	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56 56)				peel	7 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	12 (59 62 60 65 61 88 59 63 56	2.00 × 5 1.20 × 7	230 × 7 200 × 5	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 0.07	< 0.05 0.07	
	56 56)				peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	13 (59 62 60 65 61 88 59 63 56 56	2.00 × 5 1.20 × 8	230 × 7 200 × 6	n.a.	Pulp	8 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56 56)				peel	8 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Sevilla Colombia	1	0.60	300	n.a.	Pulp	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
1989 Cavendish					peel	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
(Valery)	2 (68)	0.60 × 2	300 × 2	n.a.	Pulp	8 65	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	8 65	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (68 70)	0.60 × 3	300 × 3	n.a.	Pulp	8 57	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	8 57	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	4 (68 70	0.60 × 4	300 × 4	n.a.	Pulp	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	73)				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	5 (68 70	0.60 × 5	300 × 5	n.a.	Pulp	7 63	< 0.05 < 0.05	0.06 < 0.05	0.06 < 0.05	
	73 63)				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	6 (68 70 73 63	0.60 × 6	300 × 6	n.a.	Pulp	7 21 35 56[F]	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
	63)				peel	7 21 35 56[F]	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
	1	1.2	300	n.a.	Pulp	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
					peel	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2 (68)	1.2 × 2	300 × 2	n.a.	Pulp	8 65	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	8 65	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (68 70)	1.2 × 3	300 × 3	n.a.	Pulp	8 57	< 0.05 < 0.05	< 0.05 0.06	< 0.05 0.06	
					peel	8 57	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	4 (68 70	1.2 × 4	300 × 4	n.a.	Pulp	7 63	< 0.05 < 0.05	0.08 0.08	0.08 0.08	
	73)				peel	7 63	< 0.05 < 0.05	0.06 < 0.05	0.06 < 0.05	
	5 (68 70	1.2 × 5	300 × 5	n.a.	Pulp	7 63	< 0.05 < 0.05	0.08 0.08	0.08 0.08	
	73 63)				peel	7 63	< 0.05 < 0.05	< 0.05 0.07	< 0.05 0.07	
	6 (68 70 73 63	1.2 × 6	300 × 6	n.a.	Pulp	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 0.13	< 0.05 < 0.05 < 0.05 0.13	
	63)				Peel	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 0.06	< 0.05 < 0.05 < 0.05 0.06	
Cienaga Colombia 1989 Cavendish (Valery)	1	0.60	300	n.a.	Pulp	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
					peel	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2 (68)	0.60 × 2	300 × 2	n.a.	Pulp	8 65	< 0.05 < 0.05	0.07 0.05	0.07 0.05	

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
					peel	8 65	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (68 70)	0.60 × 3	300 × 3	n.a.	Pulp	8 57	< 0.05 < 0.05	< 0.05 0.11	< 0.05 0.11	
					peel	8 57	< 0.05 < 0.05	< 0.05 0.06	< 0.05 0.06	
	4 (68 70	0.60 × 4	300 × 4	n.a.	Pulp	7 63	< 0.05 < 0.05	< 0.05 0.06	< 0.05 0.06	
	63)				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	5 (68 70	0.60 × 5	300 × 5	n.a.	Pulp	7 63	< 0.05 < 0.05	< 0.05 0.06	< 0.05 0.06	
	63 73)				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	6 (68 70 63 73	0.60 × 6	300 × 6	n.a.	Pulp	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 0.06 0.05	< 0.05 < 0.05 0.06 0.05	
	63)				peel	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
	1	1.2	300	n.a.	Pulp	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
					peel	6 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2 (68)	1.2 × 2	300 × 2	n.a.	Pulp	8 65	< 0.05 < 0.05	0.07 0.05	0.07 0.05	
					peel	8 65	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (68 70)	1.2 × 3	300 × 3	n.a.	Pulp	8 57	< 0.05 < 0.05	< 0.05 0.11	< 0.05 0.11	
					peel	8 57	< 0.05 < 0.05	< 0.050.06	< 0.05 0.06	
	4 (68 70	1.2 × 4	300 × 4	n.a.	Pulp	7 63	< 0.05 < 0.05	< 0.05 0.06	< 0.05 0.06	
	63)				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	5 (68 70	1.2 × 5	300 × 5	n.a.	Pulp	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	63 73)				peel	7 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	6 (68 70 63 73	1.2 × 6	300 × 6	n.a.	Pulp	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 0.06 0.05	< 0.05 < 0.05 0.06 0.05	
	63)				peel	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
Guapiles, Pococi Costa	1	0.60	240	n.a.	Pulp	6 48	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
Rica 1989 Gran Enano					peel	6 48	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2 (48)	0.60 × 2	240 × 2	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (48 56)	0.60 × 3	240 × 3	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	4 (48 56	0.60 × 4	240 × 4	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56)				peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	5 (48 56	0.60 × 5	240 × 5	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56 56)				Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	6 (48 56 56 56	0.60 × 6	240 × 6	n.a.	Pulp	6 20 34 55	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
	56)				peel	6 20 34 55	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
28 Millas Costa Rica	1	0.60		n.a.	Pulp	7 49	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
1989 Cavendish					peel	7 49	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2 (49)	0.60 × 2		n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
					Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (49 56)	0.60 × 3		n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	4 (49 56	0.60 × 4		n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56)				Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	5 (49 56	0.60 × 5		n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56 56)				Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	6 (49 56 56 56	0.60 × 6		n.a.	Pulp	6 20 34 55	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
	56)				Peel	6 20 34 55	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
Lorenzo de Garaicoa	1	0.60	250	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
Ecuador 1989 Valery					Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2 (56)	0.60 × 2	250 × 2	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (56 56)	0.60 × 3	250 × 3	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	4 (56 56	0.60 × 4	250 × 4	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56)				Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	5 (56 56	0.60 × 5	250 × 5	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	56 56)				Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	6 (56 56 56 56	0.60 × 6	250 × 6	n.a.	Pulp	7 21 35 56 [F]	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
	56)				peel	7 21 35 56 [F]	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
San Juan de Pueblo Viejo	1	0.60	250	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A46037
Ecuador 1989 Cavendish					Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2 (56)	0.60 × 2	250 × 2	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	3 (56 56)	0.60 × 3	250 × 3	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	4 (56 56	0.60 × 4	250 × 4	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56)				Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	5 (56 56	0.60 × 5	250 × 5	n.a.	Pulp	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	56 56)				Peel	7 56	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	6 (56 56 56 56	0.60 × 6	250 × 6	n.a.	Pulp	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
	56)				peel	7 21 35 56	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
Sto Tomas	1	0.5	400	n.a.	Pulp	80	0.05	0.04	0.09	A30169
Davao Norte					peel	80	< 0.01	< 0.01	< 0.01	
Philippines	1	3.0	400	n.a.	Pulp	80	< 0.01	0.04	0.04	A30170
1984 Lacatan					peel	80	< 0.01	0.04	0.04	
	2 (137)	0.5 0.5	400 400	n.a.	Pulp	6	< 0.01	0.04	0.04	A30163
					peel	6	< 0.01	< 0.01	< 0.01	
	2 (46)	3.0 2.0	400 400	n.a.	Pulp	97	< 0.01	0.02	0.02	A30164
					peel	97	< 0.01	0.05	0.05	
Tagum Davao	1	0.3	200	n.a.	Pulp	65	< 0.01	< 0.01	< 0.01	A30167
Norte					peel	65	< 0.01	0.02	0.02	
Philippines	1	1.00	200	n.a.	Pulp	65	< 0.01	0.06	0.06	A30168
1984 Dwarf					peel	65	< 0.01	< 0.01	< 0.01	
Cavendish	3 (66 56)	0.3 0.3	200 200	n.a.	Pulp	7	< 0.01	0.04	0.04	A30166
		0.3	200		peel	7	< 0.01	0.05	0.05	
	2	1.0	200	n.a.	Pulp	63	< 0.01	0.02	0.02	A30165
	(66)	1.0	200		peel	63	< 0.01	0.02	0.02	
Delta Farms, Kapalong Philippines 1984	5 (38 23 65 38)	1.0 × 5	750 × 5	n.a.	Pulp	8	< 0.05	< 0.05	< 0.05	A32244

BANANA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
					peel	8	< 0.05	< 0.05	< 0.05	
Dwarf Cavendish	5 (38 23 65 38)	2.0 × 5	750 × 5	n.a.	Pulp	8	< 0.05	< 0.05	< 0.05	A32245
					peel	8	< 0.05	< 0.05	< 0.05	
La Libertad Philippines	1	0.3	200	n.a.	Pulp	9 30	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A35616
1986 Cavendish					peel	9 30	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
	2	0.3	200	n.a.	Pulp	23	< 0.05	< 0.05	< 0.05	A35617
	(38)	0.3	200		peel	23	< 0.05	< 0.05	< 0.05	
	3 (38 39)	0.3 0.3	200 200	n.a.	Pulp	31 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A35618
		0.3	200		peel	31 58	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	

All applications were made with the same formulation at each trial location, either a 200 SL or 220 SL formulation

[a] Analytical method measures NAG together with glufosinate as a common derivative

Table 84 Residues of glufosinate in guava (directed sprays for weed control)

GUAVA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Bidor Malaysia 1995 common guava, with seeds	4 (28 28 42)	0.5 0.5 0.5 0.5	450 450 450 450		Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	A57294
	4 (28 28 42)	1.0 1.0 1.0 1.0	450 450 450 450		Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	
Bidor Malaysia 1995 common guava, seedless	4 (28 28 42)	0.5 0.5 0.5 0.5	450 450 450 450		Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	A57294
	4 (28 28 42)	1.0 1.0 1.0 1.0	450 450 450 450		Fruit	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	

All applications were made with a 150 SL formulation

Table 85 Residues of glufosinate in kiwifruit (directed sprays for weed control)

KIWIFRUIT	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Bologna, Emilia-Romagna Italy 2005 Hayward	2 (30)	1.40 1.00	300 300	81	Fruit	14	< 0.01	< 0.01	< 0.01	RA-2081-05
San Martino , Emilia-Romagna Italy 2005 Hayward	2 (30)	1.40 1.00	300 300	85	Fruit	14	< 0.01	< 0.01	< 0.01	RA-2081-05

KIWIFRUIT	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Orosi CA USA 1989 Hayward	3 (93 127)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	< 0.05	< 0.05	A54166
	3 (93 127)	2.00 2.00 2.00	187 187 187		Fruit	14	< 0.05	< 0.05	< 0.05	
Dinuba CA USA 1989 Hayward	3 (79 94)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	0.37	0.37	A54166
Hanford CA USA 1989 Hayward	3 (84 105)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	0.07	0.07	A54166
	3 (84 105)	2.00 2.00 2.00	187 187 187		Fruit	14	< 0.05	< 0.05	< 0.05	
Kingsburg CA USA 1989 Hayward	3 (79 94)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	< 0.05	< 0.05	A54166
Orosi CA USA 1989 Hayward	3 (93 127)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	< 0.05	< 0.05	A54166
Kerman CA USA 1989 Hayward	3 (22 90)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	< 0.05	< 0.05	A54166
Trenton SC USA 1989 Hayward	3 (84 107)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	< 0.05	< 0.05	A54166
	3 (84 107)	1.00 1.00 1.00	187 187 187		Fruit	14	< 0.05	0.10	0.10	

All applications were made with a 200 SL formulation

Table 86Residues of glufosinate in mango (directed sprays for weed control)

MANGO	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Nambour, Qld 1990 Kensington	1	1.0	250		Fruit	7 14 20	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	A59030
Pride	1	2.0	250		Fruit	7 14 20	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1	
Childers Qld 1995 Kensington Pride	2 (41)	1.2 1.2	253 307		Fruit	0 14 21 28	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	A59028
	2 (41)	2.4 2.4	253 307		Fruit	0 14 21 28	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	
Cooroy Qld 1994 Fascell	2 (42)	1.2 1.2	290 290		Fruit	0 14 21 28	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	A59029
	2 (42)	2.4 2.4	290 290		Fruit	0 14 21 28	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	

All applications were made with a 200 SL formulation

Table 87 Residues of glufosinate in papaya (directed sprays for weed control)

PAPAYA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Glenwood, Queensland, Australia1996 Riktor Gold	2 (61)	1.2 1.2	233 233	71–79	Fruit	0 14 21 30	< 0.05 < 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06 < 0.06	YH 94 AA
	2 (61)	2.4 2.4	233 233	71–79	Fruit	0 14 21 30	< 0.05 < 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06 < 0.06	

All applications were made with a 200 SL formulation

Table 88 Residues of glufosinate in bulb onions (pre-emergent or pre-planting control of weeds)

ONION	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Bahlburg Germany	1	0.60	300	PRE	Whole	85 105	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H402 11
1984 Hyper					Leaf	134	< 0.05	< 0.05	< 0.05	
					Bulb	134	< 0.05	< 0.05	< 0.05	
					Bulb dry	144	< 0.05	< 0.05	< 0.05	
Elbstorf, Germany	1	0.60	300	PRE	Whole	66 86	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H402 12
1984					Leaf	115	< 0.05	< 0.05	< 0.05	
Stuttgarter					Bulb	115	< 0.05	< 0.05	< 0.05	
Riesen					Bulb dry	125	< 0.05	< 0.05	< 0.05	
Bornheim, Germany	1	0.60	300	PRE	Whole	80 98	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H402 21
1984 Zittauer-					Leaf	136	< 0.05	< 0.05	< 0.05	
Gelbe					Bulb	136	< 0.05	< 0.05	< 0.05	
					Bulb dry	146	< 0.05	< 0.05	< 0.05	
Gersthofen, Germany	1	0.60	300	PRE	Whole	68 82	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H402 31
1984					Leaf	106	< 0.05	< 0.05	< 0.05	
Stuttgarter					Bulb	106	< 0.05	< 0.05	< 0.05	
Riesen					Bulb dry	117	< 0.05	< 0.05	< 0.05	
Hattersheim, Germany	1	0.60	300	PRE	Whole	55 76	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H402 41
1984 Ontario					Leaf	96	< 0.05	< 0.05	< 0.05	
					Bulb	96	< 0.05	< 0.05	< 0.05	
					Bulb dry	118	< 0.05	< 0.05	< 0.05	
Montoison,	1	0.75	250	PP	Bulb	148	< 0.05	< 0.05	< 0.05	01R249
France 2001 Daytona					Bulb dry	155	< 0.05	< 0.05	< 0.05	
Bologna, Italy	1	0.75	300	PP	Bulb	127	< 0.05	< 0.05	< 0.05	01R249
2001 Dorata Di Parma					Bulb dry	134	< 0.05	< 0.05	< 0.05	
Alginet, Spain	1	0.75	300	PP	Bulb	125	< 0.05	< 0.05	< 0.05	01R249
2001 De Grano					Bulb dry	132	< 0.05	< 0.05	< 0.05	
Dilofo –	1	0.75	300	PP	Bulb	162	< 0.05	< 0.05	< 0.05	01R249
Lariss, Greece 2001 De Grano					Bulb dry	169	< 0.05	< 0.05	< 0.05	
Montoison,	1	0.75	250	PP	Bulb	156	< 0.05	< 0.05	< 0.05	02R249
France 2002 Ready					Bulb dry	163	< 0.05	< 0.05	< 0.05	

ONION	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Zapponeta,	1	0.75	350	PP	Bulb	117	< 0.05	< 0.05	< 0.05	02R249
Italy 2002 Giugnarola					Bulb dry	124	< 0.05	< 0.05	< 0.05	
Brenes, Spain	1	0.75	300	PP	Bulb	111	< 0.05	< 0.05	< 0.05	02R249
2002 Tardía Blanca Pais					Bulb dry	118	< 0.05	< 0.05	< 0.05	
Alginet, Spain	1	0.75	300	PP	Bulb	96	< 0.05	< 0.05	< 0.05	02R249
2002 Puma					Bulb dry	103	< 0.05	< 0.05	< 0.05	
Salvaterra de	1	0.75	300	PP	Bulb	104	< 0.05	< 0.05	< 0.05	01R249
Magos, Portugal 2001 Valenciana					Bulb dry	111	< 0.05	< 0.05	< 0.05	
Lodge Heath, UK 2004 Sherpa	1	0.75	300	PRE	Bulb	148	< 0.01	< 0.01	< 0.01	RA-2070/04
Luzille, France 2004 Spirit F1	1	0.75	300	PRE	Bulb	140	< 0.01	< 0.01	< 0.01	RA-2070/04
Lusia, Italy 2004 Pandero	1	0.75	300	PRE	Bulb	105	< 0.01	< 0.01	< 0.01	RA-2071/04
Lamontjoie, France 2004 Yellowstone	1	0.75	300	PRE	Bulb	231	< 0.01	< 0.01	< 0.01	RA-2071/04
Lodge Heath, UK 2004 Sherpa	1	0.75	300	PRE	Bulb	148	< 0.01	< 0.01	< 0.01	RA-2072/04
Hennef, Germany 2004 Stuttgarter Riesen	1	0.75	300	PRE	Bulb	148	< 0.01	< 0.01	< 0.01	RA-2072/04

PRE = application after sowing but before crop emergence = pre-emergence

PP = before planting of seedlings = pre-planting

All applications were made with 150 SL or 200 SL formulations. No trial site involved a side-by-side comparison of formulation

Table 89 Residues of glufosinate in sweet corn glufosinate tolerant sweet corn

SWEET CORN	Application						Residue (mg/kg)[a]			
Location, year variety	No. (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
East Lansing, WI USA 1997 GH-0937	2 (14)	0.41 0.41	185 185	V8–10	Ear no husk	46	< 0.05	0.06	0.06	06515 + AMS
Arlington, WI USA 1997 Rodgers 0937	2 (14)	0.41 0.41	191 188	V6–7	Ear no husk	50	< 0.05	< 0.05	< 0.05	06515 + AMS 2nd spray only
	2 (14)	0.41 0.41	191 188	V6–7	Ear no husk	50	< 0.05	< 0.05	< 0.05	
Fremont, OH USA 1998 GH 0937 Corn G	2 (14)	0.41 0.41	141 199	–	Ear no husk	47	< 0.05	< 0.05	< 0.05	06515 + AMS
Arlington, WI USA 1998 GH 0937	2 (15)	0.41 0.41	186 190	V6	Ear no husk	43	< 0.05	< 0.05	< 0.05	06515 + AMS

SWEET CORN	Application						Residue (mg/kg)[a]			
Location, year variety	No. (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Freeville, NY USA 1999 GH 0937	2 (16)	0.43 0.41	283 273	5 collars	Ear no husk	45	< 0.05	< 0.05	< 0.05	06953 Tractor oil leak, 10% necrosis
		0.44 0.42	289 277	5–6 collars	Ear no husk	45	< 0.05	< 0.05	< 0.05	06953
Gainesville, FL USA 1999 GSS-0966VP	2 (14)	0.42 0.45	379 284	–	Ear no husk	30	< 0.05	0.14	0.14	06953
	2 (14)	0.44 0.43	395 289	–	Ear no husk	38	< 0.05	< 0.05	< 0.05	06953
Prosser, WA USA 1999 Liberty Link	2 (14)	0.42 0.42	144 145	–	Ear no husk	44	< 0.05	< 0.05	< 0.05	06953
Holtville, CA USA 1999 Attribute	2 (14)	0.50 0.50	332 327	–	Ear no husk	45	< 0.05	< 0.05	< 0.05	06953
Kimberly, ID USA 1999	2 (14)	0.44 0.43	246 236	–	Ear no husk	46	0.06	< 0.05	0.06	06953

All applications were made with a 200 SL formulation.

[a] Analytical method measures NAG together with glufosinate as a common derivative

Table 90 Residues of glufosinate in lamb's lettuce (corn salad) (pre-emergent weed control)

LAMBS LETTUCE	Application						Residue (mg/kg)			
Location, year variety	No.	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Stelle, Germany	1	1.00	400	PRE	Whole	76 96	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU87H404 11 (slow crop growth)
1987 Dunkelgruener					Leaf	114 129	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Alfter, Germany	1	1.00	300	PRE	Whole	35 42	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU87H404 21
1987 Hollaender					Leaf	50 59	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Gersthofen, Germany	1	1.00	300	PRE	Whole	73 84	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU87H404 31
1987 Matador					Leaf	95 114	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Hochheim-Massenheim,	1	1.00	300	PRE	Whole	131 148	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU87H404 41
Germany 1987 Polar					Leaf	168 189	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	

PRE = application after sowing but before crop emergence = pre-emergence. Applications were between 3 and 11 days after sowing.

Whole = whole plant + roots

All applications were made with a 200 SL formulation.

Table 91 Residues of glufosinate in head lettuce (one directed spray or one pre-planting spray or the combination of one pre-planting spray and one directed spray)

LETTUCE	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Paulinia – SP Brazil 2006 Crespa	1	0.4	500	45	Head	0 3 5 7 10	0.85 0.14 0.06 < 0.03 < 0.03	< 0.04 0.14 0.04 < 0.04 < 0.04	0.85 0.28 0.10 < 0.04 < 0.04	RA-1099/06 [a]
	1	0.8	500	45	Head	7	< 0.03	< 0.04	< 0.04	
Ribeirao Preto – SP Brazil 2006 Crespa	1	0.4	500	45	Head	0 3 5 7 10	0.91 0.14 0.07 < 0.03 < 0.03	< 0.04 0.11 < 0.04 < 0.04 < 0.04	0.91 0.25 0.07 < 0.04 < 0.04	RA-1100/06 [a]
	1	0.8	500	45	Head	7	< 0.03	< 0.04	< 0.04	
Uberlandia	1	0.4	500	45	Head	7	< 0.03	< 0.04	< 0.04	RA-1101/06
MG Brazil 2006 Crespa	1	0.8	500	45	Head	7	< 0.03	< 0.04	< 0.04	[a]
Londrina PR	1	0.4	500	45	Head	7	< 0.03	< 0.04	< 0.04	RA-1102/06
Brazil 2006 Crespa	1	0.8	500	45	Head	7	< 0.03	< 0.04	< 0.04	[a]
Korifi, Macedonia Greece 1999 Paris	1	0.75	300	PP	Head	21 42	< 0.05 < 0.05	0.29 0.07	0.29 0.07	ER99ECS575
Ishland Kosh	1	0.75	300	47	Head	0 21	0.06 < 0.05	< 0.05 < 0.05	0.06 < 0.05	
Chalkidona Macedonia Greece 1999	1	0.75	300	PP	Head	39 60	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS575
Atraxion	1	0.75	300	47	Head	0 21	1.2 < 0.05	< 0.05 < 0.05	1.2 < 0.05	
Andria Puglia, Italy 1999 Musette	1	0.75	300	PP	Head	36 57	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS575
	1	0.75	500	41	Head	0 21	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Salvaterra de Magos, Ribatejo e Oeste,	1	0.75	300	PP	Head	45 67	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS575
Portugal 1999 Vanity	1	0.75	300	43	Head	0 22	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Caldas da Rainha, Ribatejo e Oeste,	1	0.75	300	PP	Head	34 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS575
Portugal 1999 Vanity	1	0.75	300	43	Head	0 21	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Boara, Emilia Romagna, Italy 2001 Gentile	1	0.75	350	PP	Head	25 46	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	01R243
Fanly	1 [a]	0.75	350	44	Head	0 21	0.12 < 0.05	< 0.05 < 0.05	0.12 < 0.05	
Andria, Puglia, Italy 2001	1	0.75	350	PP	Head	62 83	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	01R243
Nabucco	1 [a]	0.75	350	48	Head	0 21	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	

LETTUCE	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Nea Magnisia, Macedonia, Greece 2001	1	0.75	300	PP	Head	29 51	< 0.05 < 0.05	0.10 < 0.05	0.10 < 0.05	01R243
Paris Island	1[a]	0.75	300	19	Head	0 22	0.45 < 0.05	< 0.05 < 0.05	0.45 < 0.05	
Alginet, Valencia, Spain 2001 Splendor	1	0.75	300	PP	Head	42 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	01R243
	1[a]	0.75	300	48	Head	0 21	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Maucourt, Picardie, France N 2008 Access (loose-leaf variety)	2 (29)[b]	0.75 0.67	300 273	45	Head	0 7 14 21	1.2 0.04 0.01 0.01	0.04 0.02 < 0.01 < 0.01	1.2 0.06 0.01 0.01	08-2059
Zwaagdijk-Oost, Noord_Holland, Netherlands 2008 Lollo Rosso (loose-leaf variety)	2 (29)[b]	0.75 0.80	300 321	45	Head	0 7 14 21	0.30 0.01 < 0.01 < 0.01	0.01 0.01 < 0.01 < 0.01	0.31 0.02 < 0.01 < 0.01	08-2059
Little Shelford, Cambrigeshire, UK 2008 Lollo Rosso (loose-leaf variety)	2 (29)[b]	0.75 0.75	300 300	18	Head	0 7 14 21	1.2 0.05 0.02 0.01	0.04 0.05 0.03 0.01	1.24 0.10 0.05 0.02	08-2059
Meckenbeuren Germany 2008 Feska (loose-leaf variety)	2 (29)[b]	0.75 0.75	300 300	42	Head	0 6 13 20	0.15 0.01 < 0.01 < 0.01	0.02 0.02 0.02 < 0.01	0.17 0.03 0.02 < 0.01	08-2059
Chazay d'Azergues France S 2008 Murai (loose-leaf variety)	2 (28)[b]	0.75 0.75	300 300	19	Head	0 7 15 21	1.3 0.04 0.02 0.01	0.05 0.03 0.01 0.02	1.35 0.07 0.03 0.03	08-2131
Alginet, Valencia, Spain 2008 Jamai (loose-leaf variety)	2 (33)[b]	0.75 0.75	300 300	43	Head	0 7 14 21	0.43 0.04 0.01 0.01	0.01 < 0.01 < 0.01 < 0.01	0.44 0.04 0.01 0.01	08-2131
Andria, Puglia, Italy 2008 Anthony (loose-leaf variety)	2 (28)[b]	0.75 0.75	300 300	18	Head	0 7 14 21	0.59 0.04 0.04 < 0.01	0.09 0.05 0.03 0.01	0.68 0.09 0.07 0.01	08-2131

LETTUCE	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Agia Marina, Macedonia, Greece 2008 Forseca (loose-leaf variety)	2 (28)[b]	0.75 0.75	300 300	42	Head	0 7 13 20	0.26 0.04 < 0.01 < 0.01	0.12 0.02 0.01 < 0.01	0.38 0.06 0.01 < 0.01	08-2131
Fondettes France N 2009 Feska (open-leaf variety)	2 (28)[b]	0.75 0.75	300 300	39	Head	0 7 14 21	1.6 0.03 < 0.01 < 0.01	0.11 0.03 0.01 < 0.01	1.71 0.06 0.01 < 0.01	09-2139
Zwaagdijk Netherlands 2009 Lollo Rosso (open-leaf variety)	2 (28)[b]	0.75 0.75	300 300	18	Head	0 7 14 21	0.35 0.04 < 0.01 < 0.01	0.01 0.02 0.01 < 0.01	0.36 0.06 0.01 < 0.01	09-2139
Cergy France N 2009 Quenty (open-leaf variety)	2 (29)[b]	0.75 0.75	300 300	19	Head	0 7 14 21	0.73 0.02 < 0.01 < 0.01	0.05 0.03 < 0.01 0.01	0.78 0.05 < 0.01 0.01	09-2139
Villers-Perwin Belgium 2009 Funnas (open-leaf variety)	2 (28)[b]	0.75 0.75	300 300	42	Head	0 7 14 21	0.02 0.03 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.02 0.03 0.01 < 0.01	09-2139
Sautard France S 2009 9539 Bio (open-leaf variety)	2 (28)[b]	0.75 0.75	300 300	42	Head	0 7 14 21	0.10 0.01 < 0.01 < 0.01	0.03 0.01 0.01 0.03	0.13 0.02 0.01 0.03	09-2140
Alginet Spain 2009 Jamai (open-leaf variety)	2 (28)[b]	0.75 0.75	300 300	43	Head	0 7 14 21	1.0 0.02 < 0.01 < 0.01	0.01 < 0.01 < 0.01 < 0.01	1.01 0.02 < 0.01 < 0.01	09-2140
Polignano a Mare Italy 2009 Antony (open-leaf variety)	2 (30)[b]	0.75 0.75	300 300	16	Head	0 7 14 21	0.96 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.96 < 0.01 < 0.01 < 0.01	09-2140
Vasilika Greece 2009 Marina (open-leaf variety)	2 (28)[b]	0.75 0.75	300 300	18	Head	0 7 14 21	0.77 0.05 < 0.01 < 0.01	0.14 0.08 0.03 0.02	0.91 0.13 0.03 0.02	09-2140

PP = before transplanting of seedlings = pre-planting

In the trials conducted in 1999/2001 and 2008/2009 a spray shield was used during application or the crop was covered during application in order to limit crop contamination. In the 2008/2009 trials the first spray was 5 days prior to transplanting seedlings.

All applications were made with 150SL or 200 SL formulations. No trial site involved a side-by-side comparison of formulation

[a] Plants were covered with plastic bags or covers prior to application

[b] Spray shield used for second application

GS: BBCH growth stages for lettuce (Meier, 2001) :

- 12: 2nd true leaf unfolded

- 13: 3rd true leaf unfolded
- 13–18: Stages continuous till . . .
- 19: 9 or more true leaves unfolded
- 41: Heads begin to form: the two youngest leaves do not unfold
- 42: 20% of the expected head size reached
- 43: 30% of the expected head size reached
- 44: 40% of the expected head size reached
- 45: 50% of the expected head size reached
- 46: 60% of the expected head size reached
- 47: 70% of the expected head size reached
- 48: 80% of the expected head size reached
- 49: Typical size, form and firmness of heads reached

Table 92 Residues of glufosinate in common (kidney) bean (directed applications)

KIDNEY BEAN	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Stelle Germany 1985 Dufix	1	1.00	300	–	Bean + pod	0 7 14	0.3 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.3 < 0.05 < 0.05	DEU85H41 111
					Bean dry	35	< 0.05	< 0.05	< 0.05	
Bornheim Germany 1985 Marona	1	1.00	300	–	Bean + pod	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	DEU85H41 121
					Bean dry	42	< 0.05	< 0.05	< 0.05	
Elbstorf Germany 1986 Sperlings Dufrix	1	1.00	300	61	Bean + pod	7 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU86H40 811
					Bean dry	67	< 0.05	< 0.05	< 0.05	
Stelle Germany 1986 Hills Maja	1	1.00	300	61	Bean + pod	7 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU86H40 812
					Bean dry	57	< 0.05	< 0.05	< 0.05	
Bornheim Germany 1986 Hilds Marona	1	1.00	300	65	Bean + pod	7 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU86H40 821
					Bean dry	69	< 0.05	< 0.05	< 0.05	
Langenhain Germany	1	1.00	300	65	Bean + pod	20	< 0.05	< 0.05	< 0.05	DEU86H40 826
1986 Prelude					Bean dry	48	< 0.05	< 0.05	< 0.05	
Gersthofen Germany 1986 Prelude	1	1.00	300	63	Bean + pod	7 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU86H40 831
					Bean dry	46	< 0.05	< 0.05	< 0.05	
Hattersheim Germany 1986 Prelude	1	1.00	300	61	Bean + pod	7 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU86H40 841
					Bean dry	63	< 0.05	< 0.05	< 0.05	
Chilly France N 2008 Lingot	2 (27)	0.75 0.75	300 300	13	Bean + pod	49	< 0.01	< 0.01	< 0.01	08-2123
					Bean green	73	< 0.01	< 0.01	< 0.01	
					Bean dry	122	< 0.01	< 0.01	< 0.01	
Zwaagdijk-Oost	2 (27)	0.75 0.75	300 300	12	Bean + pod	43	< 0.01	< 0.01	< 0.01	08-2123
Netherlands					Bean green	78	< 0.01	< 0.01	< 0.01	
2008 Allure					Bean dry	95	< 0.01	< 0.01	< 0.01	

KIDNEY BEAN	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Toulouse France S 2008	2 (30)	0.75 0.75	300 300	15	Bean + pod	34	< 0.01	< 0.01	< 0.01	08-2134
Myka					Bean green	46	< 0.01	< 0.01	< 0.01	
					Bean dry	72	< 0.01	< 0.01	< 0.01	
Alginet Spain 2008 Cleo	2 (28)	0.75 0.75	300 300	14	Bean + pod	26	< 0.01	< 0.01	< 0.01	08-2134
					Bean green	48	< 0.01	< 0.01	< 0.01	
					Bean dry	68	< 0.01	< 0.01	< 0.01	
Fresnoy les Roye France	2 (28)	0.75 0.75	300 300	14	Bean + pod	36	< 0.01	0.01	0.01	09-2137
N 2009 Lingot					Bean green	50	< 0.01	0.01	0.01	
Suisse blanc					Bean dry	84	< 0.01	< 0.01	< 0.01	
Zwaagdijk Netherlands	2 (28)	0.75 0.75	300 300	19	Bean + pod	36	< 0.01	< 0.01	< 0.01	09-2137
2009 Scylla					Bean green	70	< 0.01	< 0.01	< 0.01	
					Bean dry	81	< 0.01	< 0.01	< 0.01	
Toulouse France S 2009	2 (29)	0.75 0.75	300 300	12	Bean + pod	32	< 0.01	< 0.01	< 0.01	09-2138
Myka					Bean green	48	< 0.01	< 0.01	< 0.01	
					Bean dry	75	< 0.01	< 0.01	< 0.01	
Ladispoli Italy 2009 Taylors	2 (28)	0.75 0.75	300 300	12	Bean + pod	38	< 0.01	< 0.01	< 0.01	09-2138
					Bean green	47	< 0.01	< 0.01	< 0.01	
					Bean dry	62	< 0.01	< 0.01	< 0.01	

PH = pre-harvest desiccation

1985 trials: Application was for pre-harvest desiccation

1986 trials: a directed application to weeds was made around flowering of the crop

2008/2009 trials: The first application was on the whole plot area about 5 days before sowing. The second application was between the plant rows using a spray shield at early post-emergence growth stages (BBCH 12-19).

All applications were made with a 200 SL formulation.

GS: BBCH growth stages for beans (Meier, 2001):

- 12: 2 full leaves (first leaf pair unfolded)
- 13: 3rd true leaf (first trifoliate leaf) unfolded
- 14–18: Stages continuous till . . .
- 19: 9 or more leaves (2 full leaves, 7 or more trifoliate) unfolded
- 14: Fourth true leaf (second trifoliate leaf) unfolded.
- 24: Fourth side shoot visible.
- 51: First flower buds visible.
- 55: First flower buds enlarged.
- 59: First petals visible, flowers still closed.
- 61: Beginning of flowering: 10% of flowers open
- 62: 20% of flowers open
- 63: 30% of flowers open
- 64: 40% of flowers open
- 65: Full flowering: 50% of flowers open

Table 93 Residues of glufosinate in beans, dry (pre-harvest desiccation)

BEANS DRY	Application						Residue (mg/kg)[a]			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Paulinia, Brazil 2000 Perola	1	0.24	200	89	bean, dry	5	< 0.05	< 0.05	< 0.05	QO-09/24-Aventis-2001
	1	0.48	200	89	bean, dry	5	< 0.05	< 0.05	< 0.05	
Itapetininga, Brazil 2000 Perola	1	0.24	250	89	bean, dry	5	< 0.05	< 0.05	< 0.05	QO-10/24-Aventis-2001
	1	0.48	250	89	bean, dry	5	< 0.05	< 0.05	< 0.05	
Londrina, Brazil 2000 Preto	1	0.24	200	89	bean, dry	5	< 0.05	< 0.05	< 0.05	QO-11/24-Aventis-2001
	1	0.48	200	89	bean, dry	5	< 0.05	< 0.05	< 0.05	
Paulinia, Brazil 2005 Carioca	1	0.4	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	RA-1032/06
	1	0.8	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	
Ribeirao Preto, Brazil 2005 Carioca	1	0.4	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	RA-1033/06
	1	0.8	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	
Londrina, Brazil 2005 Carioca	1	0.4	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	RA-1034/06
	1	0.8	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	
Uberlandia, Brazil 2005 Perola	1	0.4	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	RA-1035/06
	1	0.8	200	93	bean, dry	5	< 0.03	< 0.04	< 0.04	

All applications were made with 200SL or 220 SL formulations. No trial site involved a side-by-side comparison of formulation

[a] Analytical method measures NAG together with glufosinate as a common derivative

GS: BBCH growth stages for beans (Meier, 2001):

- 85: 50% of pods ripe (beans hard) Main period of ripening2
- 86: 60% of pods ripe (beans hard)
- 87: 70% of pods ripe (beans hard)
- 88: 80% of pods ripe (beans hard)
- 89: Fully ripe: pods ripe (beans hard)

Table 94 Residues of glufosinate in glufosinate tolerant soya bean

SOYA BEAN	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Proctor AR USA 1994 W62	150SL	2 (34)	0.39 0.50	102 100	R2	Seed	82	0.68	0.06	[a]	0.74	A54156
A5403	200SL	2 (34)	0.39 0.50	102 100	R2	Seed	82	0.83	0.06	[a]	0.89	
Molino FL USA 1994 W62	150SL	2 (20)	0.39 0.49	94 94	R2	Seed	93	0.58	0.08	[a]	0.64	A54156
A5403	200SL	2 (20)	0.39 0.50	94 94	R2	Seed	93	0.62	0.08	[a]	0.70	
Webster City IA USA 1994	150SL	2 (32)	0.38 0.52	93 97	R2	Seed	86	1.06	0.14	[a]	1.20	A54156

SOYA BEAN	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
W98 A3322	200SL	2 (32)	0.38 0.53	93 99	R2	Seed	86	1.04	0.10	[a]	1.14	
Richmond IL USA 1994	150SL	2 (33)	0.39 0.50	94 94	R2	Seed	69	0.64	0.07	[a]	0.71	A54156
Glufosinate tolerant	200SL	2 (33)	0.39 0.50	94 94	R2	Seed	69	0.52	0.06	[a]	0.58	
Noblesville IN USA 1994	150SL	2 (25)	0.38 0.50	94 94	R2	Seed	77	1.33	0.34	[a]	1.67	A54156
W98 A3322	200SL	2 (25)	0.40 0.52	94 94	R2	Seed	77	0.88	0.15	[a]	1.03	
Clarence MO USA 1994 W98	150SL	2 (20)	0.40 0.50	103 90	R2	Seed	77	0.66	0.18	[a]	0.84	A54156 +NIA
A3322	200SL	2 (20)	0.39 0.50	103 90	R2	Seed	77	0.41	0.17	[a]	0.58	+NIA
Pikeville NC USA 1994 W62	150SL	2 (34)	0.39 0.49	95 93	R2	Seed	102	0.32	< 0.05	[a]	0.32	A54156 +NIA
A5403	200SL	2 (34)	0.39 0.49	92 93	R2	Seed	102	0.43	< 0.05	[a]	0.43	+NIA
New Holland OH USA 1994	150SL	2 (20)	0.39 0.48	94 88	R2	Seed	76	0.80	0.22	[a]	1.02	A54156
W98 A3322	200SL	2 (20)	0.41 0.52	98 93	R2	Seed	76	0.70	0.18	[a]	0.88	
Germansville PA USA 1994	150SL	2 (19)	0.40 0.52	95 95	R2	Seed	91	0.73	0.08	[a]	0.81	A54156
W98 A3322	200SL	2 (19)	0.40 0.52	95 95	R2	Seed	91	0.54	0.06	[a]	0.60	
Emporia VA USA 1994 W62	150SL	2 (43)	0.39 0.50	94 93	R2	Seed	82	1.60	0.28	[a]	1.88	A54156
A5403	200SL	2 (43)	0.39 0.50	94 94	R2	Seed	82	1.26	0.36	[a]	1.62	
Noblesville IN USA 1995	150SL	2 (29)	0.39 0.50	100 98	R2.5–R3	Seed	64	1.14	0.16	[a]	1.30	A55780
MG III W98 A3322	200SL	2 (29)	0.39 0.50	100 98	R2.5–R3	Seed	64	0.96	0.19	[a]	1.15	
Martinsville USA 1995	150SL	2 (29)	0.39 0.50	101 98	R2	Seed	62	1.06	0.22	[a]	1.28	A55780
MG III W98 A3322	200SL	2 (29)	0.39 0.50	101 98	R2	Seed	62	0.90	0.24	[a]	1.14	
Danville USA 1995 MG III	150SL	2 (31)	0.39 0.50	94 95	R2	Seed	60	0.58	0.10	[a]	0.68	A55780
W98 A3322	200SL	2 (31)	0.39 0.50	94 94	R2	Seed	60	0.68	0.10	[a]	0.78	
Delavan USA 1995 MG III	150SL	2 (22)	0.38 0.49	90 91	R2.5	Seed	82	0.64	0.10	[a]	0.74	A55780
W98 A3322	200SL	2 (22)	0.38 0.52	90 95	R2.5	Seed	82	0.93	0.18	[a]	1.11	
Webster City IA USA 1995	150SL	2 (28)	0.39 0.50	93 95	R2	Seed	84	0.32	< 0.05	[a]	0.32	A55780
MG III W98 A3322	200SL	2 (28)	0.39 0.50	93 95	R2	Seed	84	0.28	< 0.05	[a]	0.28	
Leonard USA 1995 MG III W62 A5403	150SL	2 (21)	0.39 0.50	94 95	R2	Seed	71	0.62	0.11	[a]	0.73	A55780
	200SL	2 (21)	0.39 0.50	94 95	R2	Seed	71	0.83	0.13	[a]	0.96	
New Holland OH USA 1995	150SL	2 (22)	0.39 0.50	98 97	R2	Seed	86	0.32	0.07	[a]	0.39	A55780

SOYA BEAN	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
MG III W98 A3322	200SL	2 (22)	0.39 0.50	98 97	R2	Seed	86	0.18	0.06	[a]	0.24	
Conklin USA 1995 MG III	150SL	2 (28)	0.39 0.50	94 94	R2	Seed	81	0.36	0.15	[a]	0.51	A55780
W98 A3322	200SL	2 (28)	0.39 0.50	94 95	R2	Seed	81	0.33	0.10	[a]	0.43	
York USA 1995 MG III	150SL	2 (33)	0.39 0.50	94 94	R3	Seed	80	0.12	0.05	[a]	0.17	A55780
W98 A3322	200SL	2 (33)	0.39 0.50	94 94	R3	Seed	80	0.62	0.06	[a]	0.68	
Bendena USA 1995 MG III	150SL	2 (24)	0.39 0.52	94 100	R2	Seed	78	0.50	0.06	[a]	0.56	A55780
W98 A3322	200SL	2 (24)	0.38 0.50	93 97	R2	Seed	78	0.42	0.05	[a]	0.47	
Rosa USA 1995 MG III W62 A5403	150SL	2 (42)	0.40 0.54	95 96	R2	Seed	70	0.95	0.61	[a]	1.56	A55780
	200SL	2 (42)	0.38 0.53	92 94	R2	Seed	70	0.64	0.84	[a]	1.48	
Greenvillle USA 1995	150SL	2 (35)	0.39 0.50	94 95	R2	Seed	90	1.08	0.16	[a]	1.24	A55780
MG III W62 A5403	200SL	2 (35)	0.39 0.50	94 95	R2	Seed	90	0.92	0.15	[a]	1.07	
Chula USA 2009	280SL	2 (7)	0.73 0.59	160 160	14	Seed	123	< 0.01	0.01	0.03	0.04	RAGLP034 + AMS/NIA
S080120		2 (7)	0.73 0.44	160 160	14	Seed	123	< 0.01	< 0.01	0.02	0.02	
		2 (7)	0.45 0.45	160 170	14	Seed	123	< 0.01	< 0.01	0.02	0.02	
Blackville USA 2009	280SL	2 (6)	0.73 0.60	160 160	14	Seed	111	< 0.01	0.02	0.06	0.08	RAGLP034 + AMS
S080120		2 (6)	0.73 0.45	160 160	14	Seed	111	< 0.01	0.01	0.03	0.04	
		2 (6)	0.45 0.45	160 160	14	Seed	111	< 0.01	< 0.01	0.02	0.02	
Proctor AR USA 2009	280SL	2 (16)	0.73 0.60	140 150	15	Seed	79	0.03	0.03	0.04	0.13	RAGLP034 + AMS
SG4489NLL		2 (16)	0.73 0.45	140 150	15	Seed	79	0.02	0.02	0.23	0.27	
		2 (16)	0.45 0.45	140 140	15	Seed	79	0.04	0.02	0.33	0.39	
Greenville USA 2009	280SL	2 (9)	0.73 0.59	130 120	15	Seed	105	0.06	0.06	0.28	0.40	RAGLP034 + AMS/NIA
S080120		2 (9)	0.74 0.46	130 130	15	Seed	105	0.06	0.05	0.28	0.39	
		2 (9)	0.45 0.45	130 130	15	Seed	105	0.04	0.05	0.22	0.31	
Cheneyville USA 2009 S080120	280SL	2 (12)	0.75 0.60	150 150	16	Seed	85 100 110 120 125	0.04 0.04 0.04 0.06 0.06	0.02 0.02 0.03 0.03 0.03	0.45 0.31 0.36 0.32 0.34	0.51 0.37 0.43 0.41 0.43	RAGLP034 + AMS
		2 (12)	0.75 0.45	150 150	16	Seed	85 100 110 120 125	0.02 0.03 0.04 0.04 0.06	0.03 0.03 0.05 0.03 0.04	0.26 0.24 0.27 0.20 0.32	0.31 0.30 0.36 0.27 0.42	

SOYA BEAN	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
		2 (12)	0.45 0.44	150 150	16	Seed	85 100 110 120 125	0.02 0.01 0.02 0.03 0.04	0.02 0.02 0.02 0.03 0.04	0.17 0.13 0.12 0.14 0.19	0.21 0.16 0.16 0.20 0.27	
Springfield USA 2009 S070139	280SL	2 (5)	0.73 0.61	130 130	14	Seed	78 95 104 112 118	< 0.01 0.01 < 0.01 0.02 < 0.01	0.04 0.06 0.07 0.05 0.06	0.07 0.14 0.11 0.15 0.11	0.11 0.21 0.18 0.22 0.17	RAGLP034 + AMS
		2 (5)	0.74 0.46	130 130	14	Seed	78 95 104 112 118	< 0.01 0.01 0.01 < 0.01 < 0.01	0.04 0.04 0.07 0.03 0.04	0.07 0.10 0.12 0.09 0.10	0.11 0.15 0.20 0.12 0.15	
		2 (5)	0.45 0.45	130 130	14	Seed	78 95 104 112 118	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.02 0.02 0.02 0.02 0.02	0.05 0.09 0.09 0.06 0.08	0.07 0.11 0.11 0.08 0.10	
Bagley USA 2009	280SL	2 (14)	0.73 0.60	160 160	15	Seed	76	0.07	0.06	0.1	0.23	RAGLP034 + AMS/NIA
S080141		2 (14)	0.72 0.45	160 160	15	Seed	76	0.06	0.08	0.78	0.94	
		2 (14)	0.45 0.45	160 160	15	Seed	76	0.06	0.04	0.99	1.09	
Berkley USA 2009	280SL	2 (8)	0.74 0.58	180 160	14	Seed	85	0.01	0.06	0.42	0.49	RAGLP034 + AMS/NIA
S080141		2 (8)	0.74 0.46	180 160	14	Seed	85	0.02	0.03	0.37	0.42	
		2 (8)	0.44 0.45	170 160	14	Seed	85	0.02	0.04	0.44	0.50	
Geneva USA 2009	280SL	2 (10)	0.73 0.60	170 170	14	Seed	98	0.02	0.22	0.51	0.75	RAGLP034 + AMS/NIA
S080117		2 (10)	0.73 0.45	170 170	14	Seed	98	0.02	0.34	0.38	0.74	
		2 (10)	0.45 0.45	170 170	14	Seed	98	0.02	0.18	0.49	0.69	
Campbell USA 2009	280SL	2 (11)	0.73 0.60	190 190	14	Seed	107	0.02	0.02	0.28	0.32	RAGLP034 + AMS
S080119		2 (11)	0.73 0.45	190 190	14	Seed	107	0.01	0.01	0.20	0.22	
		2 (11)	0.45 0.45	190 190	14	Seed	107	< 0.01	0.01	0.19	0.20	
York USA 2009	280SL	2 (11)	0.73 0.59	140 140	14	Seed	93	0.04	0.07	0.54	0.65	RAGLP034 + AMS
S070139		2 (11)	0.73 0.45	140 140	14	Seed	93	0.01	0.02	0.21	0.24	
		2 (11)	0.45 0.46	140 140	14	Seed	93	< 0.01	< 0.01	0.17	0.17	
Gardner USA 2009	280SL	2 (6)	0.74 0.61	140 140	14	Seed	98	0.02	0.02	0.14	0.18	RAGLP034 + AMS/NIA
S070144		2 (6)	0.74 0.46	150 140	14	Seed	98	< 0.01	0.03	0.09	0.12	
		2 (6)	0.45 0.45	140 140	14	Seed	98	< 0.01	0.02	0.12	0.14	
Richland USA 2009	280SL	2 (15)	0.74 0.59	180 190	15	Seed	102	0.01	0.04	0.14	0.19	RAGLP034 + AMS

SOYA BEAN	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
S070144		2 (15)	0.74 0.45	180 190	15	Seed	102	0.01	0.02	0.14	0.19	
		2 (15)	0.46 0.45	180 190	15	Seed	102	0.02	0.02	0.18	0.22	
		2 (15)	0.74 0.59	180 180	15	Seed	102	0.02	0.02	0.20	0.24	
		2 (15)	0.74 0.45	180 180	15	Seed	102	0.02	0.02	0.22	0.24	
		2 (15)	0.46 0.45	180 190	15	Seed	102	0.01	0.02	0.16	0.29	
Richwood USA 2009	280SL	2 (8)	0.74 0.60	160 160	14	Seed	106	< 0.01	0.01	0.08	0.09	RAGLP034 + AMS/NIA
S0701344		2 (8)	0.74 0.45	160 160	14	Seed	106	< 0.01	0.02	0.08	0.10	
		2 (8)	0.45 0.45	160 160	14	Seed	106	< 0.01	0.01	0.06	0.07	
Marysville USA 2009	280SL	2 (8)	0.74 0.60	170 160	14	Seed	106	< 0.01	0.01	0.08	0.09	RAGLP034 + AMS
S0701344		2 (8)	0.74 0.45	170 160	14	Seed	106	< 0.01	0.02	0.09	0.11	
		2 (8)	0.45 0.45	170 160	14	Seed	106	0.01	0.05	0.26	0.32	
Clarence USA 2009	280SL	2 (7)	0.74 0.59	190 180	14	Seed	104	0.01	0.05	0.26	0.32	RAGLP034 + AMS
S070146		2 (7)	0.75 0.45	190 180	14	Seed	104	< 0.01	0.02	0.12	0.14	
		2 (7)	0.45 0.46	190 180	13	Seed	104	< 0.01	0.02	0.12	0.14	
Lime Springs USA 2009	280SL	2 (10)	0.77 0.59	200 180	14	Seed	127	0.03	0.06	0.36	0.42	RAGLP034 + AMS/NIA
S080119		2 (10)	0.77 0.45	200 180	14	Seed	127	0.02	0.04	0.28	0.34	
		2 (10)	0.48 0.44	200 180	14	Seed	127	0.02	0.04	0.26	0.32	
Cherry Grove USA 2009	280SL	2 (12)	0.76 0.60	200 190	14	Seed	117	0.02	0.03	0.30	0.35	RAGLP034 + AMS/NIA
S080119		2 (12)	0.75 0.46	190 190	14	Seed	117	0.04	0.04	0.46	0.54	
		2 (12)	0.46 0.45	190 190	14	Seed	117	0.03	0.02	0.36	0.41	
Seymour USA 2009	280SL	2 (6)	0.67 0.60	160 180	14	Seed	82	0.04	0.04	0.22	0.30	RAGLP034 + AMS
S070141		2 (6)	0.75 0.45	170 180	14	Seed	82	0.03	0.03	0.18	0.24	
		2 (6)	0.44 0.45	180 180	14	Seed	82	0.02	0.02	0.12	0.16	

R2 = bloom

[a] Analytical method measures NAG together with glufosinate as a common derivative

GS: , BBCH growth stages for soya beans(Meier, 2001):

- 11: First pair of true leaves unfolded (unifoliate leaves on the first node)
- 12: Trifoliate leaf on the second node unfolded.
- 13: Trifoliate leaf on the third node unfolded.
- 14: Trifoliate leaf on the fourth node unfolded.
- 19: Trifoliate leaf on the ninth node unfolded. No side shoots visible.

- 23: Third side shoot of first order visible.
- 55: First flower buds enlarged.
- 65: Full flowering: about 50% of flowers open.
- 67: Flowering declining.
- 69: End of flowering: first pods visible.
- 71: About 10% of pods have reached final length.

Table 95 Residues of glufosinate in carrot (pre-emergent sprays for weed control)

CARROT	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Ref
Stelle, Germany	1	0.60	300	PRE	Whole	66 80	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H40311
1984 Sperlings Zino					Leaf	93 103	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Root	93 103	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Bornheim, Germany	1	0.60	300	PRE	Whole	62 73	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H40321
1984 Nantaise					Leaf	88 103	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Root	88 103	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Hattersheim, Germany	1	0.60	300	PRE	Whole	55 76	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU84H40341
1984 Lange rote Stumpfe					Leaf	96 118	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Root	96 118	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Stelle, Germany	1	0.60	300	PRE	Whole	58 72	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU85H40311
1985 Sperlings Fruehbund					Leaf	86 100	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Root	86 100	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Stelle, Germany	1	0.60	300	PRE	Whole	53 67	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU85H40312
1985 Sperlings Fruehbund					Leaf	81 95	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Root	81 95	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Bornheim, Germany	1	0.60	300	PRE	Whole	60 73	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU85H40321
1985 Nantaise					Leaf	87 100	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Root	87 100	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Gersthofen, Germany	1	0.60	300	PRE	Whole	55 63	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU85H40331
1985 Nantaise					Leaf	80 98	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
					Root	80 98	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Hattersheim, Germany	1	0.60	300	PRE	Whole	42 55	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	DEU85H40341
1985 Nantaise					Leaf	79 105	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	

CARROT	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Ref
					Root	79 105	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Saint Sylvestre France 1999 Junior	1	0.75	300	PRE	Root	69 83	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS574
Salvaterra de Magos Portugal 1999 Nantes	1	0.75	300	PRE	Root	85 99	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS574
Zapponeta, Italy 1999 Aldino	1	0.75	300	PRE	Root	91 105	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS574
Alcacer Spain 1999 Mantesa	1	0.75	300	PRE	Root	93 107	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS574
Nea Chalkidona, Greece 1999 Bolero	1	0.75	300	PRE	Root	99 113	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	ER99ECS574
Lusia, Italy 2001 Bolero	1	0.75	350	PRE	Root	63	< 0.05	< 0.05	< 0.05	01R241
Zapponeta, Italy 2001 Bolero	1	0.75	300	PRE	Root	92	< 0.05	< 0.05	< 0.05	01R241
Alginet Spain 2001 Valdo	1	0.75	300	PRE	Root	97	< 0.05	< 0.05	< 0.05	01R241
Nea Chalkidona, Greece 2001 Maestro	1	0.75	300	PRE	Root	109	< 0.05	< 0.05	< 0.05	01R241

PRE = application made pre-emergence = after sowing but prior to crop emerging

All applications were made with a 200 SL formulation in the Germany 1984/85 trials and a 150 SL formulation in the other trials

Table 96 Residues of glufosinate in potato (crop desiccation use)

POTATO	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Paulinia Brazil 2000 Bintje	1	0.24	200	49	tuber	10	< 0.05	< 0.05	< 0.05	QO-12/24-Aventis-2001 [a]
	1	0.48	200	49	tuber	10	< 0.05	< 0.05	< 0.05	
Itapetininga Brazil 2000 Bintje	1	0.24	250	49	tuber	10	< 0.05	< 0.05	< 0.05	QO-13/24-Aventis-2001 [a]
	1	0.48	250	49	tuber	10	< 0.05	< 0.05	< 0.05	
Vargem Grande do Sul Brazil 2000 Monalisa	1	0.24	250	49	tuber	10	< 0.05	< 0.05	< 0.05	QO-14/24-Aventis-2001 [a]
	1	0.48	250	49	tuber	10	< 0.05	< 0.05	< 0.05	
Uberlandia Brazil 2006 Agata	1	0.40	600	49	tuber	7	< 0.03	< 0.04	< 0.04	RA-1088/06 [a]
	1	0.80	600	49	tuber	7	< 0.03	< 0.04	< 0.04	
Ponta Grossa Brazil 2006 Agata	1	0.40	600	49	tuber	7	< 0.03	< 0.04	< 0.04	RA-1089/06 [a]
	1	0.80	600	49	tuber	7	< 0.03	< 0.04	< 0.04	

POTATO	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Itaara Brazil 2006 Agata	1	0.40	600	49	tuber	7	< 0.03	< 0.04	< 0.04	RA-1090/06 [a]
	1	0.80	600	49	tuber	7	< 0.03	< 0.04	< 0.04	
Paulinia Brazil 2006 Agata	1	0.40	600	49	tuber	7	< 0.03	< 0.04	< 0.04	RA-1087/06 [a]
	1	0.80	600	49	tuber	7	< 0.03	< 0.04	< 0.04	
Chazay D'Azergues France 2002 Bintje	2 (5)	0.38 0.38	250 250	91	tuber	0 14	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	02R242
Cestas France 2002 Bintje	2 (5)	0.38 0.38	250 250	91	tuber	0 13	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	02R242
Le Bocage France 2002 Bintje	2 (4)	0.38 0.38	250 250	91	tuber	0 13	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	02R242
St Andre Farivillers	2 (5)	0.38 0.38	250 250	91	tuber	0 14	0.07 0.08	< 0.05 < 0.05	0.07 0.08	02R242
France 2002 Bintje	1	0.60	250	85	tuber	0 7 13	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Davenescourt France 2002	2 (5)	0.38 0.33	250 250	91	tuber	0 14	0.08 < 0.05	< 0.05 < 0.05	0.08 < 0.05	02R242
Bintje	1	0.6	250	85	tuber	0 7 13	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Fresnoy Les Roye France	2 (5)	0.33 0.33	250 250	91	tuber	0 14	0.11 0.09	< 0.05 < 0.05	0.11 0.09	02R242
2002 Russet Burbank	1	0.6	250	85	tuber	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	
Luzillé France 2003 Mona Lisa	2 (5)	0.38 0.38	300 300	91	tuber	0 7 14 21	0.17 0.17 0.14 0.16	< 0.05 < 0.05 < 0.05 < 0.05	0.17 0.17 0.14 0.16	RA-2668/03
Gersthofen Germany 2003 Bintje	2 (5)	0.38 0.38	300 300	49*	tuber	0 7 15	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	RA-2668/03
Hopton United Kingdom 2003 Stemster	2 (5)	0.38 0.38	300 300	91	tuber	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	RA-2668/03
Feuquerolles France 2003 Charlotte	2 (5)	0.38 0.38	300 300	91	tuber	0 7 14 21	0.06 0.06 0.07 0.11	< 0.05 < 0.05 < 0.05 < 0.05	0.06 0.06 0.07 0.11	RA-2668/03
Les Valayans Europe, South 2003 Adora	2 (5)	0.38 0.38	300 300	91	tuber	0 7 14 21	0.08 0.10 < 0.05 0.07	< 0.05 < 0.05 < 0.05 < 0.05	0.08 0.10 < 0.05 0.07	RA-2669/03
Mas Grenier France 2003 Lisetta	2 (5)	0.38 0.38	300 300	91	tuber	0 7 14	0.18 0.16 0.15	< 0.05 < 0.05 < 0.05	0.18 0.16 0.15	RA-2669/03
Les Valayans France 2009 Agata	2 (5)	0.38 0.38	300 300	48 tb	tuber	0 7 15 21	0.18 0.12 0.16 0.14	0.01 < 0.01 < 0.01 < 0.01	0.19 0.12 0.16 0.14	09-2146
Piacenza Italy 2009 Kennebek	2 (5)	0.38 0.38	300 300	93	tuber	0 7 14 21	0.03 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	0.03 < 0.01 < 0.01 < 0.01	09-2146

POTATO	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Llerona Spain 2009 Red Pontiac	2 (4)	0.4 0.4	320 320	48 tb	tuber	0 7 14 19	0.74 0.53 0.52 0.63	0.01 0.02 0.02 0.02	0.75 0.55 0.54 0.65	09-2146[b]
Aldeia Gavinha Portugal 2009 Agria	2 (5)	0.38 0.38	300 300	89	tuber	0 7 14 21	0.21 0.23 0.19 0.22	< 0.01 < 0.01 < 0.01 < 0.01	0.21 0.23 0.19 0.22	09-2146
Burscheid Germany 2003 Cilena	1	0.6	300	85	tuber	0 7 14	< 0.05 < 0.05 0.07	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 0.07	RA-2667/03 Flailed
	1	0.6	300	85	tuber	0 7 14	< 0.05 0.33 0.34	< 0.05 < 0.05 < 0.05	< 0.05 0.33 0.34	Not-flailed
Feuquerolles France 2003 Charlotte	1	0.6	300	91	tuber	0 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05	RA-2667/03 Flailed
	1	0.6	300	91	tuber	0 7 14 21	< 0.05 0.15 0.16 0.21	< 0.05 < 0.05 < 0.05 < 0.05	< 0.05 0.15 0.16 0.21	Not-flailed
Zwaagdijk Netherlands 2003 Agria	1	0.6	300	85	tuber	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	RA-2667/03 Flailed
	1	0.6	300	85	tuber	0 7 14	< 0.05 0.07 0.12	< 0.05 < 0.05 < 0.05	< 0.05 0.07 0.12	Not-flailed
Saint-Amand Belgium 2003 Bintje/Ukama	1	0.6	300	85	tuber	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	RA-2667/03 Flailed
	1	0.6	300	85	tuber	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	Not-flailed
Hopton United Kingdom	1	0.6	300	85	tuber	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	RA-2667/03 Flailed
2003 Stemster	1	0.6	300	85	tuber	0 7 14	< 0.05 0.30 0.27	< 0.05 < 0.05 < 0.05	< 0.05 0.30 0.27	Not-flailed
Bornheim Germany 2003 Seccura	1	0.6	300	85	tuber	0 7 14	< 0.05 < 0.05 0.06	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 0.06	RA-2667/03 Flailed
	1	0.6	300	85	tuber	0 7 14	< 0.05 < 0.05 0.06	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 0.06	Not-flailed
Villers-Perwin Belgium 2009 Bintje	1	0.6	300	85	tuber	0 7 14 21	< 0.01 < 0.01 0.01 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 0.01 0.01	09-2144
Nagele Flevoland Netherlands 2009 Agria	1	0.6	300	85	tuber	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	09-2144
Luzillé France 2009 Samba	1	0.6	300	85	tuber	0 7 14 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	09-2144
Stotzheim Bas-Rhin France 2009 Cicero	1	0.6	300	85	tuber	0 7 13 21	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01	09-2248

POTATO	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
North Rose NY USA 1997 Chieftain	1	0.44	94	91	tuber	10	< 0.05	< 0.05	< 0.05	A57885
Germansville PA USA 1997 Kathadin	1	0.46	103	91–93	tuber	10	0.28	< 0.05	0.28	A57885
Hamburg PA USA 1997 Kathadin	1	0.46	94	91–93	tuber	9	0.10	< 0.05	0.10	A57885
Cohansey NJ USA 1997 Superior	1	0.47	94	91–93	tuber	10	0.10	< 0.05	0.10	A57885
East Brunswick NJ USA 1997 Kathadin	1	0.44	84	91–93	tuber	9	0.14	< 0.05	0.14	A57885
Immokalee FL USA 1997 Red LaSoa	1	0.44	103	91	tuber	9	0.32	< 0.05	0.32	A57885
Lehigh Acres FL USA 1998 Red LaSoa	1	0.47	103	91	tuber	9	0.62	< 0.05	0.62	A57885
Carlyle IL USA 1997 Irish Cobbler	1	0.46	94	91–93	tuber	9	0.06	< 0.05	0.06	A57885
Maple Island MN USA 1997 Norland	1	0.45	94	91–93	tuber	10	0.38	< 0.05	0.38	A57885
Clear Lake IA USA 1997 Wisconsin Superior	1	0.45	94	91–93	tuber	9	0.18	< 0.05	0.18	A57885
Northwood ND USA 1997 Kenneher	1	0.45	94	91–93	tuber	10	< 0.05	< 0.05	< 0.05	A57885
Smithfield UT USA 1997 Ranger Russet	1	0.45	94	91	tuber	9	0.24	< 0.05	0.24	A57885
Bothwell UT USA 1997 Chipeta	1	0.44	94	91	tuber	9	< 0.05	< 0.05	< 0.05	A57885
Porterville CA USA 1997 White Rose	1	0.45	94	91–93	tuber	9	< 0.05	< 0.05	< 0.05	A57885
Dayton ID USA 1997 Ranger Russet	1	0.45	94	91	tuber	9	0.12	< 0.05	0.12	A57885
American Falls ID USA 1997 Russet Burbank	1	0.46	103	91–93	tuber	9	0.26	< 0.05	0.26	A57885
Aberdeen ID USA 1997 Russet Burbank	1	0.44	94	91–93	tuber	10	0.11	< 0.05	0.11	A57885
Declo ID USA 1997 Russet Burbank	1	0.45	94	91–93	tuber	9	0.12	< 0.05	0.12	A57885

POTATO	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Pocatello ID USA 1997 Russet Burbank	1	0.44	94	91–93	tuber	9	0.06	< 0.05	0.06	A57885
Idaho Falls ID USA 1997 Russet Burbank	1	0.46	94	91–93	tuber	9	0.16	< 0.05	0.16	A57885

All applications were made with 120 SL, 150 SL, 200 SL or 220 SL formulations. No trial site involved a side-by-side comparison of formulation

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Significant precipitation occurred prior to the second application. This trial should not be used for estimation of maximum residue levels.

GS:BBCH growth stages for potato (Meier, 2001):

- 48: Maximum of total tuber mass reached, tubers detach easily from stolons, skin set not yet complete (skin easily removable with thumb)
- 49: Skin set complete: (skin at apical end of tuber not removable with thumb) 95% of tubers in this stage
- 85: Berries in the first fructification ochre-coloured or brownish
- 89: Berries in the first fructification shrivelled, seed dark
- 91: Beginning of leaf yellowing
- 93: Most of the leaves yellowish
- 95: 50% of the leaves brownish
- 97: Leaves and stem dead, stems bleached and dry

Table 97 Residues of glufosinate in glufosinate tolerant sugar beet

SUGAR BEET	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Fresno CA USA 1995 Liberty Link	2 (7)	0.60 0.60	102 100	16	Root	139	< 0.05	0.29	0.29	A55839
	3 (8 7)	0.21 0.20 0.20	98 101 101		Root	139	< 0.05	0.14	0.14	
	3 (8 7)	0.42 0.40 0.40	97 101 100		Root	139	< 0.05	0.31	0.31	
Fisher MN USA 1995 Liberty Link	2 (7)	0.63 0.60	96 94		Root	95	0.11	< 0.05	0.11	A55839
	3 (10 8)	0.21 0.20 0.21	99 96 99		Root	95	< 0.05	< 0.05	< 0.05	
	3 (8 7)	0.41 0.43 0.40	97 99 95		Root	95	0.09	< 0.05	0.09	
Horace ND USA 1995 Liberty Link	2 (7)	0.60 0.62	94 95	19	Root	104	0.14	< 0.05	0.14	A55839
	3 (9 6)	0.40 0.41 0.41	93 94 97		Root	103	0.07	< 0.05	0.07	

SUGAR BEET	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	3 (4 10)	0.43 0.43 0.46	99 98 107		Root	104	0.14	< 0.05	0.14	
Jerome ID USA 1995 Liberty Link	2 (7)	0.86 0.64	100 99		Root	41	0.21	0.06	0.27	A55839
	3 (4 10)	0.21 0.21 0.22	99 97 103		Root	41	0.05	< 0.05	0.05	
	3 (4 10)	0.43 0.43 0.46	99 98 107		Root	41	0.16	< 0.05	0.16	
Conklin MI USA 1996	2 (4)	0.60 0.60	98 93		Root	109	0.12	0.06	0.18	A57730
	3 (10 7)	0.60 0.35 0.60	92 95 90		Root	106	0.22	0.05	0.27	
New Holland OH USA 1996 Liberty Link	2 (8)	0.60 0.60	94 89	18	Root	83	0.20	< 0.05	0.20	A57730
	3 (8 14)	0.60 0.35 0.60	97 94 90	18	Root	77	0.67	< 0.05	0.67	
Horace ND USA 1996 Liberty Link	2 (5)	0.60 0.60	94 94	18	Root	67	0.16	< 0.05	0.16	A57730
	3 (11 10)	0.60 0.35 0.60	94 94 94	18	Root	62	0.62	< 0.05	0.62	
Scottsbluff NB USA 1996 Liberty Link	2 (7)	0.60 0.60	94 94	18	Root	115	< 0.05	< 0.05	< 0.05	A57730
	3 (18 14)	0.60 0.35 0.60	96 94 90	18	Root	108	0.06	< 0.05	0.06	
Minot ND USA 1996 Liberty Link	2 (7)	0.60 0.60	99 96	18	Root	73	0.13	< 0.05	0.13	A57730
	3 (15 14)	0.60 0.35 0.60	94 94 95		Root	66	0.30	0.09	0.39	
Eaton CO USA 1996 Liberty Link	2 (14)	0.60 0.60	100 101	18	Root	80	< 0.05	< 0.05	< 0.05	A57730
	3 (15 26)	0.60 0.35 0.60	94 96 102	18	Root	68	0.54	< 0.05	0.54	
Campion CO USA 1996 Liberty Link	2 (14)	0.60 0.60	97 99	18	Root	86	0.11	< 0.05	0.11	A57730
	3 (15 26)	0.60 0.35 0.60	92 94 97	18	Root	81	0.28	< 0.05	0.28	
Fresno CA USA 1996 Liberty Link	2 (14)	0.60 0.60	92 95	18	Root	132	0.07	0.06	0.13	A57730
	3 (10 19)	0.60 0.35 0.60	96 92 94	18	Root	122	0.36	0.06	0.42	

SUGAR BEET	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Jerome ID USA 1996 Liberty Link	2 (4)	0.60 0.60	95 95	19	Root	128	0.06	< 0.05	0.06	A57730
	3 (7 14)	0.60 0.35 0.60	96 94 94		Root	121	0.20	< 0.05	0.20	

All applications were made with a 200 SL formulations.

[a] Analytical method measures NAG together with glufosinate as a common derivative

GS: BBCH growth stages for beet (Meier, 2001):

- 10: First leaf visible (pinhead-size): cotyledons horizontally unfolded
- 11: First pair of leaves visible, not yet unfolded (pea-size)
- 12: 2 leaves (first pair of leaves) unfolded
- 14: 4 leaves (2nd pair of leaves) unfolded
- 15: 5 leaves unfolded
- 16–18: Stages continuous till . . .
- 19: 9 and more leaves unfolded

Table 98 Residues of glufosinate in asparagus (pre-emergent)

ASPARAGUS	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Stelle Germany 1987 Huchels Auslese	1	0.60	300	PRE	Spear	0 3 7 14 22 28	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	DEU87H41211
Hoopte Germany 1987 Huchels Hochzucht	1	0.60	400	PRE	Spear	0 3 7 14 21	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05	DEU87H41212
Bornheim Germany 1987 Huchels Leistungsriesen	1	0.60	300	PRE	Spear	0 3 7 14 21 28 34	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	DEU87H41221
Mainz-Bretzenheim Germany 1987 Schwetzinger Meisterschuss	1	0.60	400	PRE	Spear	0 3 7 14 20 27	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	DEU87H41226
Brunnen Germany 1987 Huchel	1	0.60	300	PRE	Spear	0 3 7 14 21 28 35 39	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	DEU87H41231

ASPARAGUS	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Edelshausen Germany 1987 Schwetzinger	1	0.60	300	PRE	Spear	0 3 7 14 21 28 35 39	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05 < 0.05	DEU87H41232
Manfredonia Italy 2008 UC 157	1	0.90	300	49	Spear	0 3 7 14 21	0.16 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.16 < 0.01 < 0.01 < 0.01 < 0.01	08-2126
Talayuela Caceres Spain 2008 Groling	1	0.90	300	49	Spear	0 3 7 14 21	0.01 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.01 0.01 < 0.01 < 0.01 < 0.01	08-2126
Poggio Renatico Italy 2008 Eros	1	0.90	300	49[a]	Spear	0 3 7 14 21	1.42 1.03 0.11 0.01 < 0.01	< 0.01 0.01 < 0.01 < 0.01 < 0.01	1.42 1.04 0.11 0.01 < 0.01	08-2126 spears already emerged (3-5 cm) at application
Les Valayans France S 2008 Gamma	1	0.90	300	49	Spear	0 3 7 14 21	0.27 < 0.01 < 0.01 < 0.01 < 0.01	< 0.01 < 0.01 < 0.01 < 0.01 < 0.01	0.27 < 0.01 < 0.01 < 0.01 < 0.01	08-2126

[a] In this trial the application was conducted at a time when the asparagus spears had already emerged 3–5 cm above the ground. Therefore, the trial is not considered valid.

All applications were made with a 200 SL formulation.

Table 99 Residues of glufosinate in glufosinate tolerant maize

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Webster City, Hamilton Co IA USA 1993	150SL	1	0.36	96	V5	Grain	118	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.51	95	V5	Grain	118	< 0.05	< 0.05	< 0.05	< 0.05	
		1	0.50	103	V6	Grain	114	< 0.05	< 0.05	< 0.05	< 0.05	
York, York Co NE USA 1993	150SL	1	0.37	96	V6–7	Grain	107	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.51	95	V6–7	Grain	107	< 0.05	< 0.05	< 0.05	< 0.05	
		1	0.64	119	V9-10	Grain	95	< 0.05	< 0.05	< 0.05	< 0.05	
Bethany, Macon Co IL USA (1993) (LH59 × LH51) (LH119) (4) × (T14)	150SL	1	0.38	97	V6	Grain	100	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.53	94	V6	Grain	100	< 0.05	< 0.05	< 0.05	< 0.05	
		1	0.52	92	V8	Grain	95	< 0.05	< 0.05	< 0.05	< 0.05	
		2 (9)	0.36 0.53	93 93	V8, 61 cm	Grain	95	< 0.05	< 0.05	< 0.05	< 0.05	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Sheridan IN USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.35	93	V5	Grain	115	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.51	96	V5	Grain	115	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		2 (9)	0.38 0.55	100 96	V7, 107 cm	Grain	106	< 0.05	< 0.05	< 0.05	< 0.05	A53691
Horace ND USA (1993) (LH85 × LH160) (LH119) (4) × (T14)	150SL	1	0.35	92	V5–6	Grain	107	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.49	92	V5–6	Grain	107	< 0.05	< 0.05	< 0.05	< 0.05	
		2 (10)	0.35 0.49	92 91	V6, 48 cm	Grain	97 107	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Leonard MO USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.37	105	V3–4	Grain	95	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		2 (10)	0.34 0.50	98 94	V7, 61 cm	Grain	95	< 0.05	< 0.05	< 0.05	< 0.05	
Madera CA USA (1993) (LH59 × LH51) (LH119) (4) × (T14)	150SL	1	0.37	99	V4–5	Grain	119 129	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	A53691
		2 (13)	0.39 0.53	105 102	V5–8, 81 cm	Grain	106 116	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	
Renner Minnehaha Co SD USA (1993) (LH85 × LH160) (LH119) (4) × (T14)	150SL	1	0.38	50	V8	Grain	95	< 0.05	< 0.05	< 0.05	< 0.05	A53691 [b]
		1	0.53	50	V8	Grain	95	< 0.05	< 0.05	0.07	0.07	
		2 (13)	0.37 0.52	48 51	V10, 61 cm	Grain	95	< 0.05	< 0.05	0.07	0.07	
Halifax VA USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.37	49	V5	Grain	106	< 0.05	< 0.05	< 0.05	< 0.05	A53691 [c]
		1	0.53	49	V6-7	Grain	106	< 0.05	< 0.05	< 0.05	< 0.05	
		2 (9)	0.37 0.53	49 49	V6-7, 61 cm	Grain	97	< 0.05	< 0.05	0.07	0.07	
Molino USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2(13)	0.39 0.50	94 94	63 cm	Grain	69	0.05	< 0.05	[a]	0.05	A54160
	200SL	2 (13)	0.39 0.50	94 94	63 cm	Grain	69	< 0.05	< 0.05	[a]	< 0.05	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Webster City USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (8)	0.42 0.53	92 94	61 cm	Grain	122	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (8)	0.40 0.50	96 93	61 cm	Grain	122	< 0.05	< 0.05	[a]	< 0.05	
Richmond USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Grain	118	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Grain	118	< 0.05	< 0.05	[a]	< 0.05	
East Grand Forks USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (21)	0.38 0.48	91 90	71 cm	Grain	114	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (21)	0.39 0.50	92 94	71 cm	Grain	114	0.06	< 0.05	[a]	0.06	
Clarence USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (13)	0.40 0.53	92 107	66 cm	Grain	89	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (13)	0.39 0.56	93 112	66 cm	Grain	89	< 0.05	< 0.05	[a]	< 0.05	
Pikeville USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2 (7)	0.40 0.46	96 94	61 cm	Grain	92	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (7)	0.38 0.53	96 94	61 cm	Grain	92	< 0.05	< 0.05	[a]	< 0.05	
Phelps USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (10)	0.38 0.53	91 98	66 cm	Grain	88	0.07	< 0.05	[a]	0.07	A54160
	200SL	2 (10)	0.40 0.53	97 97	66 cm	Grain	88	0.05	< 0.05	[a]	0.05	
Levelland USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2 (9)	0.38 0.46	92 91	61 cm	Grain	97	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (9)	0.39 0.49	93 92	61 cm	Grain	97	< 0.05	< 0.05	[a]	< 0.05	
Ephrata USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (13)	0.39 0.52	94 94	61 cm	Grain	86	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (13)	0.40 0.50	94 92	61 cm	Grain	86	< 0.05	< 0.05	[a]	< 0.05	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Delevan USA (1994) (LH85 × LH160) (LH119) (4) × (T14)	150SL	2 (9)	0.38 0.55	93 97	61 cm	Grain	107	< 0.05	< 0.05	[a]	< 0.05	A54160
	200SL	2 (9)	0.38 0.50	93 97	61 cm	Grain	107	< 0.05	< 0.05	[a]	< 0.05	
Doniphan USA (1995) (LH51) (B73) (5) × (T25)	200SL	2 (9)	0.39 0.50	94 94	66 cm	Grain	95	< 0.05	< 0.05	[a]	< 0.05	A57728
Ottawa USA (1995) (LH168) (LH119) (9) × (T14)	200SL	2 (7)	0.39 0.50	94 93	71 cm	Grain	111	< 0.05	< 0.05	[a]	< 0.05	A57728
Yellow Medicine USA (1995) (LH202) (LH82) (4) × (T25)	200SL	2 (10)	0.39 0.49	94 92	V7-8	Grain	100	< 0.05	< 0.05	[a]	< 0.05	A57728
Shelby USA (1995) (LH51) (B73) (5) × (T25)	200SL	2 (10)	0.39 0.52	89 86	61 cm	Grain	90	< 0.05	< 0.05	[a]	< 0.05	A57728
York USA (1995) (LH51) (LH119) (9) × (T14)	200SL	2 (8)	0.39 0.52	94 94	61 cm	Grain	89	< 0.05	< 0.05	[a]	< 0.05	A57728
Fayette USA (1995) (LH51) (LH119) (9) × (T14)	200SL	2 (10)	0.39 0.52	90 94	61 cm	Grain	117	< 0.05	< 0.05	[a]	< 0.05	A57728
Uvalde USA (1995) (LH216) (B73) (5) × (T25)	200SL	2 (7)	0.40 0.50	96 94	66 cm	Grain	84	< 0.05	< 0.05	[a]	< 0.05	A57728
Hamburg PA USA (1996) Liberty Link	200SL	2 (20)	0.39 0.50	103 94	123 cm	Grain	115	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.39	94	76-123 cm	Grain	115	< 0.05	< 0.05	[a]	< 0.05	
		1	0.60	94	76-91 cm	Grain	115	< 0.05	< 0.05	[a]	< 0.05	
Chula GA USA (1996) Liberty Link	200SL	2 (16)	0.39 0.50	94 103	91 cm	Grain	94	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.39	103	91 cm	Grain	94	< 0.05	< 0.05	[a]	< 0.05	
		1	0.60	103	91 cm	Grain	94	< 0.05	< 0.05	[a]	< 0.05	
Seven Springs NC USA (1996) Liberty Link	200SL	2 (13)	0.39 0.50	94 94	91 cm	Grain	87	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.39	94	91 cm	Grain	87	< 0.05	< 0.05	[a]	< 0.05	
		1	0.59	94	91 cm	Grain	87	< 0.05	< 0.05	[a]	< 0.05	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Noblesville IN USA (1996) Liberty Link	200SL	2 (8)	0.39 0.50	94 94	91 cm	Grain	121	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.39	94	91 cm	Grain	121	< 0.05	< 0.05	[a]	< 0.05	
		1	0.59	94	91 cm	Grain	121	< 0.05	< 0.05	[a]	< 0.05	
Danville IA USA (1996) Liberty Link	200SL	2 (13)	0.39 0.52	94 94	91 cm	Grain	108	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.40	94	91 cm	Grain	108	< 0.05	< 0.05	[a]	< 0.05	
		1	0.62	94	91 cm	Grain	108	< 0.05	< 0.05	[a]	< 0.05	
New Holland OH USA (1996) Liberty Link	200SL	2 (23)	0.39 0.50	103 103	102 cm	Grain	103	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.39	103	102 cm	Grain	103	< 0.05	< 0.05	[a]	< 0.05	
		1	0.59	103	102 cm	Grain	103	< 0.05	< 0.05	[a]	< 0.05	
Bethany IL USA (1996) Liberty Link	200SL	2 (14)	0.39 0.49	94 94	91 cm	Grain	108	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.38	94	91 cm	Grain	108	< 0.05	< 0.05	[a]	< 0.05	
		1	0.57	94	91 cm	Grain	108	< 0.05	< 0.05	[a]	< 0.05	
Wester City IA USA (1996) Liberty Link	200SL	2 (19)	0.39 0.50	94 94	V9	Grain	114	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.39	94	V9	Grain	114	< 0.05	< 0.05	[a]	< 0.05	
		1	0.59	94	V9	Grain	114	< 0.05	< 0.05	[a]	< 0.05	
Uvalde TX USA (1996) Liberty Link	200SL	2 (14)	0.37 0.50	94 94	91 cm	Grain	85	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.40	94	91 cm	Grain	85	< 0.05	< 0.05	[a]	< 0.05	
		1	0.59	94	91 cm	Grain	85	< 0.05	< 0.05	[a]	< 0.05	
Palm Beach USA (1997) 11962.20	200SL	2 (9)	0.39 0.50	94 94	61 cm	Grain	72	< 0.05	< 0.05	[a]	< 0.05	A57796
		2 (9)	0.40 0.49	94 94	61 cm	Grain	72	< 0.05	< 0.05	[a]	< 0.05	+AMS
Uvalde USA (1997) 11962.20	200SL	2 (14)	0.39 0.49	94 94	61 cm	Grain	87	< 0.05	< 0.05	[a]	< 0.05	A57796
		2 (14)	0.39 0.50	94 94	61 cm	Grain	87	< 0.05	< 0.05	[a]	< 0.05	+ AMS
Oahu USA (1997) 11962.20	200SL	2 (8)	0.39 0.49	94 94	61 cm	Grain	76	< 0.05	< 0.05	[a]	< 0.05	A57796
		2 (8)	0.39 0.49	94 94	61 cm	Grain	76	< 0.05	< 0.05	[a]	< 0.05	+ AMS
Richland IA USA 2000 Glufosinate tolerant	200SL	2 (8)	0.41 0.48	143 138	61 cm	Grain	97	< 0.05	< 0.05	< 0.05	< 0.05	B003263 +AMS
Carlyle IL USA 2000 Glufosinate tolerant	200SL	2 (22)	0.41 0.49	145 143	61 cm	Grain	86	< 0.05	< 0.05	< 0.05	< 0.05	B003263 + AMS
New Holland OH USA 2000 Glufosinate tolerant	200SL	2 (14)	0.41 0.49	144 147	61 cm	Grain	104	< 0.05	< 0.05	< 0.05	< 0.05	B003263 + AMS

MAIZE	Application						Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Rochelle IL USA 2000 Glufosinate tolerant	200SL	2 (15)	0.41 0.49	141 138	61 cm	Grain	116	< 0.05	< 0.05	< 0.05	< 0.05	B003263 + AMS
Ellendale MN USA 2000 Glufosinate tolerant	200SL	2 (11)	0.43 0.49	167 163	61 cm	Grain	121	< 0.05	< 0.05	< 0.05	< 0.05	B003263 + AMS

Transgenic maize 1996 trials first spray broadcast, over-the-top, by ground rig, and the second application by drop nozzles directed to the bottom one-third of the plants. Plots C and D received only drop nozzle applications

Transgenic maize 1997 All applications were made broadcast, over-the-top, by backpack sprayer, in approximately 94 L/ha of spray solution. (no drop nozzles)

Transgenic maize 1995 no drop nozzles

Transgenic maize 1994 two formulations, no drop nozzles

Transgenic maize 1993 no drop nozzles

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Some phytotoxicity was experienced and yield of grain was low

[c] Phytotoxicity and unusually dry weather for the crops which were not irrigated resulted in limited collection of samples

Table 100 Residue decline in glufosinate tolerant maize

MAIZE	Application						Residue (mg/kg)				
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Madera, Madera Co CA USA 1993	1	0.48	93	V3 15 cm	Grain	127	< 0.05	< 0.05	< 0.05	< 0.05	A53691
	1	0.50	85	V4–5 28 cm	Grain	120	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	101	V5 46 cm	Grain	114	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	95	V7 61 cm	Grain	96	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	100	V9–10 86 cm	Grain	96	< 0.05	< 0.05	< 0.05	< 0.05	
Leonard, Shelby Co USA 1993	1	0.48	93	V3 15 cm	Grain	127	< 0.05	< 0.05	< 0.05	< 0.05	A53691
	1	0.50	85	V4–5, 28 cm	Grain	120	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	101	V5 46 cm	Grain	114	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	95	V7 61 cm	Grain	96	< 0.05	< 0.05	0.05	0.05	
	1	0.50	100	V9-10 86 cm	Grain	96	< 0.05	< 0.05	< 0.05	< 0.05	

All applications were made with a 150 SL formulation

Table 101 Residues of glufosinate in glufosinate tolerant rice

RICE	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
West Memphis AR	2 (19)	0.50 0.49	47 47	16	Grain	102	< 0.05	< 0.05	< 0.05	A57819

RICE	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
USA (1996) Glufosinate tolerant Bengal	1	1.00	47		Grain	71	0.98	0.10	1.08	
Rosa USA (1996) Glufosinate	2 (16)	0.52 0.52	47 47	16	Grain	80	0.10	< 0.05	0.10	A57819
tolerant Bengal	1	1.04	47		Grain	62	1.64	0.14	1.78	
Greenville USA (1996)	2 (9)	0.52 0.50	47 47	16	Grain	92	< 0.05	< 0.05	< 0.05	A57819
Glufosinate tolerant Bengal	1	1.00	47		Grain	66	1.07	0.13	1.20	
Pattison USA (1996)	2 (15)	0.53 0.52	47 47	16	Grain	84	< 0.05	< 0.05	< 0.05	A57819
Glufosinate tolerant Bengal	1	1.02	47		Grain	68	0.33	0.06	0.39	
Rosa USA (1997) Glufosinate tolerant Taipei 309	2 (21)	0.50 0.50	94 94	22–23	Grain	97	< 0.05	< 0.05	< 0.05	A59924
Greenville USA (1997) Glufosinate tolerant Taipei 309	2 (23)	0.50 0.53	94 94	22–23	Grain	103	< 0.05	0.05	0.05	A59924
East Bernard USA (1997) Glufosinate tolerant Taipei 309	2 (15)	0.50 0.50	94 94	22–23	Grain	90	< 0.05	0.07	0.07	A59924
Hamilton City USA (1998)	2 (14)	0.50 0.50	94 94	23	Grain	90	< 0.05	0.10	0.10	B002652
Glufosinate tolerant M-202	2 (14)	0.50 0.50	94 94	23	Grain	90	< 0.05	0.07	0.07	+AMS
Live Oak USA (1998) Glufosinate tolerant M-202	2 (24)	0.51 0.51	94 94	23	Grain	89	< 0.05	< 0.05	< 0.05	B002652
Newport USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.50 0.50	93 94	23	Grain	96	0.20	< 0.05	0.20	B002973
Oil Trough USA (1999) Glufosinate tolerant Bengal 62	2 (22)	0.51 0.51	93 94	23	Grain	94	< 0.05	< 0.05	< 0.05	B002973
Proctor USA (1999) Glufosinate tolerant Bengal 62	2 (26)	0.50 0.50	92 95	24	Grain	86	0.08	< 0.05	0.08	B002973

RICE	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Blackfish Lake USA (1999) Glufosinate tolerant Bengal 62	2 (26)	0.50 0.51	95 93	24	Grain	85	0.07	< 0.05	0.07	B002973
Stuttgart USA (1999)	2 (15)	0.50 0.54	100 96	23	Grain	95	< 0.05	< 0.05	< 0.05	B002973
Glufosinate tolerant Bengal 62	2 (13)	0.51 0.52	91 93	22	Grain	106	< 0.05	< 0.05	< 0.05	+AMS
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.50 0.50	95 95	23	Grain	78 84 90 96	0.34 0.43 0.34 0.30	0.09 0.08 0.06 0.06	0.43 0.51 0.40 0.36	B002973
	2 (23)	0.51 0.51	95 95	23	Grain	78 84 90 96	0.24 0.22 0.16 0.20	0.06 0.06 < 0.05 < 0.05	0.30 0.28 0.16 0.20	+AMS
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.50 0.51	93 94	24	Grain	81	0.17	< 0.05	0.17	B002973
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (18)	0.49 0.51	90 94	23	Grain	70	0.66	0.07	0.73	B002973
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (28)	0.51 0.51	95 96	24	Grain	70	0.42	0.05	0.47	B002973
Greenville USA (1999) Glufosinate tolerant Bengal 62	2 (22)	0.50 0.50	94 93	24	Grain	77	0.08	< 0.05	0.08	B002973
Shaw USA (1999) Glufosinate tolerant Bengal 62	2 (29)	0.50 0.50	93 93	23	Grain	79	< 0.05	< 0.05	< 0.05	B002973
Dexter USA (1999) Glufosinate tolerant Bengal 62	2 (32)	0.50 0.50	95 93	24	Grain	80 84 88 92 96	0.09 0.07 0.08 0.07 0.05	0.06 < 0.05 < 0.05 < 0.05 < 0.05	0.15 0.07 0.08 0.07 0.05	B002973
	2 (32)	0.50 0.50	95 95	24	Grain	80 84 88 92 96	0.06 0.06 0.06 0.06 0.06	< 0.05 < 0.05 < 0.05 < 0.05 0.05	0.06 0.06 0.06 0.06 0.11	+AMS
Benton USA (1999) Glufosinate tolerant Bengal 62	2 (17)	0.51 0.52	95 90	24	Grain	86	0.14	< 0.05	0.14	B002973
East Bernard USA (1999)	2 (12)	0.50 0.50	94 103	24	Grain	78	0.29	0.06	0.35	B002973

RICE	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Glufosinate tolerant Bengal 62	2 (12)	0.50 0.50	94 103	24	Grain	78	0.20	0.08	0.28	+AMS
Brookshire USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.51 0.51	94 93	24	Grain	91	0.10	< 0.05	0.10	B002973

[a] Analytical method measures NAG together with glufosinate as a common derivative

All applications were made with a 200SL formulation

GS: BBCH Codes for rice growth stages (Meier, 2001):

- 11: First leaf unfolded
- 12: 2 leaves unfolded
- 13: 3 leaves unfolded
- 14–18: Stages continuous till . . .
- 19: 9 or more leaves unfolded
- 21: Beginning of tillering: first tiller detectable
- 22: 2 tillers detectable
- 23: 3 tillers detectable
- 24–28: Stages continuous till ...
- 29: Maximum number of tillers detectable

Table 102 Residues of glufosinate in tree nuts (directed sprays for weed control)

TREE NUTS	Application						Residue (mg/kg)			
Location, year, variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Macadamia										
Glasshouse Mountains,	1	1.0	127	89	nutmeat	21	< 0.09	< 0.12	< 0.12	A59031
Qld, Australia	1	1.0	127	89	Nutmeat	14	< 0.09	< 0.12	< 0.12	
1992 434	1	1.0	127	89	Nutmeat	7	< 0.09	< 0.12	< 0.12	
	1	1.0	127	89	Nutmeat	1	< 0.09	< 0.12	< 0.12	
	1	2.0	127	89	Nutmeat	21	< 0.09	< 0.12	< 0.12	
	1	2.0	127	89	Nutmeat	14	< 0.09	< 0.12	< 0.12	
	1	2.0	127	89	Nutmeat	7	< 0.09	< 0.12	< 0.12	
	1	2.0	127	89	Nutmeat	1	< 0.09	< 0.12	< 0.12	
Gympie, Qld Australia 1995 334	2 (53)	1.2 1.2	190 190	89	Nutmeat	0 16 27	< 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06	A59032
	2 (53)	2.4 2.4	190 190	89	Nutmeat	0 16 27	< 0.05 < 0.05 < 0.05	< 0.06 < 0.06 < 0.06	< 0.06 < 0.06 < 0.06	
Hazelnut										
Sasso-Marconi Italy 1985	2 (28)	1.5 1.5	400 400	79	Nutmeat	19	< 0.05	< 0.05	< 0.05	A35935
Gentile Del Piemonte	2 (28)	1.5 1.5	400 400	79	Nutmeat	19	< 0.05	< 0.05	< 0.05	A35937
Sasso-Marconi Italy 1985 Nocchione	2 (28)	1.5 1.5	400 400	79	Nutmeat	19	< 0.05	< 0.05	< 0.05	A35936
Pianoro Italy 1985	2 (28)	1.5 1.5	400 400	79	Nutmeat	19	< 0.05	< 0.05	< 0.05	A35938

TREE NUTS	Application						Residue (mg/kg)			
Location, year, variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Gentile Del Piemonte	2 (28)	1.5 1.5	400 400	79	Nutmeat	19	< 0.05	< 0.05	< 0.05	A35939
Walnut										
Stockton, USA 1985 English	2 (103)	1.6 1.2	257 257	n.a.	Nutmeat Shell	14	< 0.05 0.28	< 0.05 0.07	< 0.05 0.35	A34230
	2 (103)	3.2 2.4	257 257	n.a.	Nutmeat Shell	14	< 0.05 0.46	< 0.05 0.07	< 0.05 0.53	A34231
Le Grand USA 1985 English	3 (48 54)	1.6 1.2 1.2	257 257 257	n.a.	Nutmeat Shell Pericarp	14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A34232
	3 (48 54)	3.2 2.4 2.4	257 257 257	n.a.	Nutmeat Shell Pericarp	14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	A34233
Ripon CA USA 1985 Serr	3 (104 101)	1.7 1.7 1.7	380 380 380	89	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
	3 (104 101)	4.5 4.5 4.5	380 380 380	89	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
Fresno CA USA 1985 Serr	3 (88 46)	1.7 1.7 1.7	285 285 285	89	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
	3 (88 46)	3.4 3.4 3.4	285 285 285	89	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
Stockton CA USA 1985 Serr	3 (161 44)	1.7 1.7 1.7	190 190 190	79	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
	3 (161 44)	3.4 3.4 3.4	190 190 190	79	Nutmeat	11	< 0.05	< 0.05	< 0.05	A48446
Almond										
Ripon CA USA 1985 Carmel	3 (135 88)	1.7 1.7 1.7	375 375 375	89	Nutmeat	15	< 0.05	< 0.05	< 0.05	A48446
	3 (135 88)	4.5 4.5 4.5	375 375 375	89	Nutmeat	15	< 0.05	< 0.05	< 0.05	
Ripon USA 1985 Special	3 (135 88)	1.7 1.7 1.7	375 375 375	89	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
	3 (135 88)	4.5 4.5 4.5	375 375 375	89	Nutmeat	14	< 0.05	< 0.05	< 0.05	
Fresno USA 1985 Non- Peril	3 (83 55)	1.7 1.7 1.7	285 285 285	n.a.	Nutmeat	14	< 0.05	0.07	0.07	A48446
	3 (83 55)	3.4 3.4 3.4	285 285 285	n.a.	Nutmeat	14	< 0.05	0.18	0.18	
Escalon USA 1985 Non-Peril	3 (24 55)	1.7 1.7 1.7	190 190 190	79	Nutmeat	15	< 0.05	< 0.05	< 0.05	A48446
	3 (24 55)	3.4 3.4 3.4	190 190 190	79	Nutmeat	15	< 0.05	< 0.05	< 0.05	
Pecan										
Baton Rouge LA USA 1985 Cape Fear	3 (81 82)	1.7 1.7 1.7	475 475 475	89	Nutmeat	21	< 0.05	< 0.05	< 0.05	A48446

TREE NUTS	Application						Residue (mg/kg)			
Location, year, variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	3 (81 82)	3.4 3.4 3.4	475 475 475	89	Nutmeat	21	< 0.05	< 0.05	< 0.05	
Griffin GA USA 1985 Wichita	3 (56 49)	1.7 1.7 1.7	309 309 309	n.a.	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
	3 (56 49)	3.4 3.4 3.4	309 309 309	n.a.	Nutmeat	14	< 0.05	< 0.05	< 0.05	
Las Cruces NM USA 1985 Western Schley	3 (63 125)	1.7 1.7 1.7	285 285 285	n.a.	Nutmeat	14	< 0.05	< 0.05	< 0.05	A48446
	3 (63 125)	3.4 3.4 3.4	285 285 285	n.a.	Nutmeat	14	< 0.05	< 0.05	< 0.05	

All applications were made with a 200 SL formulation

Table 103 Residues of glufosinate in glufosinate tolerant cotton

COTTON SEED	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Pikeville USA (1998) LL-8B-	2 (31)	0.59 0.59	94 94	61	Seed	70	0.57	0.06	0.63	C003708
Cotton-M, Line Cot05	3 (17 14)	0.58 0.59 0.59	93 94 94	61	Seed	70	0.78	0.11	0.89	
Molino USA (1998) LL-8B-	2 (31)	0.52 0.54	82 86	61	Seed	67	0.34	< 0.05	0.34	C003708
Cotton-M, Line Cot05	3 (16 12)	0.52 0.59 0.54	82 98 86	61	Seed	67	0.42	< 0.05	0.42	
Rosa USA (1998) LL-8B-	2 (38)	0.58 0.58	94 92	61	Seed	70	0.45	0.12	0.57	C003708
Cotton-M, Line Cot05	3 (24 14)	0.58 0.58 0.58	94 95 93	61	Seed	70	0.47	0.14	0.61	
Greenville USA (1998)	2 (35)	0.59 0.58	94 93	61	Seed	68	1.08	0.06	1.14	C003708
LL-8B-Cotton-M, Line Cot05	3 (22 13)	0.58 0.58 0.58	94 93 93	61	Seed	68	1.56	0.06	1.62	
West Memphis USA	2 (53)	0.58 0.58	96 95	61	Seed	70	3.10	0.14	3.24	C003708
(1998) LL-8B-Cotton-M, Line Cot05	3 (25 28)	0.58 0.58 0.58	96 93 95	61	Seed	70	2.38	0.15	2.53	
Brookshire USA (1998)	2 (34)	0.58 0.58	93 94	61	Seed	70	0.68	0.10	0.78	C003708
LL-8B-Cotton-M, Line Cot05	3 (18 16)	0.58 0.59 0.58	93 94 93	61	Seed	70	0.59	0.06	0.65	
East Bernard USA (1998)	2 (40)	0.58 0.58	96 92	61	Seed	70	0.15	< 0.05	0.15	C003708
LL-8B-Cotton-M, Line Cot05	3 (15 25)	0.58 0.58 0.58	96 101 92	61	Seed	70	0.20	< 0.05	0.20	
Edmonson USA (1998)	2 (40)	0.56 0.59	94 98	61	Seed	69	0.30	< 0.05	0.30	C003708

COTTON SEED	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
LL-8B-Cotton-M, Line Cot05	3 (21 19)	0.57 0.58 0.59	96 99 98	61	Seed	69	0.20	< 0.05	0.20	
Levelland USA (1998)	2 (40)	0.57 0.58	91 94	61	Seed	70	1.64	0.08	1.72	C003708
LL-8B-Cotton-M, Line Cot05	3 (19 21)	0.57 0.58 0.58	91 93 93	65	Seed	70	2.08	0.17	2.25	
Eakly USA (1998) LL-8B-	2 (22)	0.58 0.60	95 97	61	Seed	70	0.18	0.06	0.24	C003708
Cotton-M, Line Cot05	3 (14 8)	0.58 0.59 0.59	94 97 96	61	Seed	70	0.23	0.78	1.01	
Dill City USA (1998) LL-8B-	2 (21)	0.58 0.58	93 93	61	Seed	69	0.30	< 0.05	0.30	C003708
Cotton-M, Line Cot05	3 (14 7)	0.59 0.58 0.59	93 94 93	61	Seed	69	0.12	0.10	0.22	
Maricopa USA (1998)	2 (35)	0.59 0.58	94 94	61	Seed	76	1.02	0.08	1.10	C003708
LL-8B-Cotton-M, Line Cot05	3 (19 16)	0.60 0.59 0.58	95 94 94	61	Seed	76	1.21	0.09	1.30	
Somerton USA (1998)	2 (36)	0.58 0.59	94 94	61	Seed	70	1.18	0.16	1.34	C003708
LL-8B-Cotton-M, Line Cot05	3 (15 21)	0.57 0.58 0.57	92 93 90	61	Seed	70	1.42	0.19	1.61	
Hickman USA (1998) LL-8B-	2 (41)	0.56 0.57	91 92	61	Seed	70	2.02	0.24	2.26	C003708
Cotton-M, Line Cot05	3 (22 19)	0.57 0.58 0.58	92 93 93	61	Seed	70	1.90	0.18	2.08	
Pikeville USA (2000) LL-8B-Cotton-M, Line Cot05	3 (14 16)	0.59 0.58 0.59	95 101 96	61	Seed	60 70 81 90 99 109	1.28 0.78 0.80 0.88 0.71 0.89	0.34 0.30 0.26 0.21 0.20 0.34	1.62 1.08 1.06 1.09 0.91 1.23	B003882

All applications were made with a 200SL formulation

[a] Analytical method measures NAG together with glufosinate as a common derivative

Note early bloom = GS 61

GS:BBCH growth stages for cotton (Meier, 2001):

- 52: First floral buds visible ("match-head square").
- 55: Floral buds distinctly enlarged
- 59: Petals visible: floral buds still closed
- 60: First flowers opened (sporadically within the population)
- 61: Beginning of flowering (“Early bloom”):
- 5–6: blooms / 25 ft of row (= 5–6 blooms / 7,5 meter of row)
- 65: Full flowering: (“Mid bloom”): 11 and more blooms / 25 ft of row = 11 and more blooms / 7,5 meter of row
- 67: Flowering finishing: majority of flowers faded (“Late bloom”)
- 69: End of flowering

Table 104 Residues of glufosinate in oilseed rape (pre-harvest desiccation)

RAPE SEED	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Hoopte	1	0.6	300	n.a.	Pod	0	15	0.06	15.06	DEU85H40611
Germany (1985) Jet Nuef					Seed	7 14	0.30 0.11	0.07 0.14	0.37 0.25	
Bornheim	1	0.6	300	n.a.	Pod	0	7.8	< 0.05	7.8	DEU85H40621
Germany (1985) Quinta					Seed	7 14	0.89 0.49	0.14 0.14	1.03 0.63	
Hattersheim	1	0.6	300	n.a.	Pod	0	11	0.07	11.07	DEU85H40641
Germany (1985) Jet Nuef					Seed	7 14	0.56 0.13	0.16 0.12	0.72 0.25	
Hoopte	1	0.6	300	87	Pod	0	8.1	0.34	8.44	DEU86H40611
Germany (1986) Jet Nuef					Seed	7 14	0.34 < 0.2	0.28 < 0.2	0.62 < 0.2	
Elbstorf	1	0.6	300	87	Pod	0	17	0.38	17.38	DEU86H40612
Germany (1986) Jet Nuef					Seed	7 14	< 0.2 < 0.2	< 0.2 < 0.2	< 0.2 < 0.2	
Bornheim	1	0.6	300	87	Pod	0	20	0.35	20.35	DEU86H40621
Germany (1986) Lindora					Seed	7 14	1.2 0.48	0.33 0.28	1.53 0.76	
Gersthofen	1	0.6	300	89	Pod	0	18	0.49	18.49	DEU86H40631
Germany (1986) Librador					Seed	7 13	< 0.2 < 0.2	< 0.2 < 0.2	< 0.2 < 0.2	
Hattersheim	1	0.6	300	85	Pod	0	13	< 0.2	13	DEU86H40641
Germany (1986) Jet Nuef					Seed	7 14	< 0.2 0.25	< 0.2 < 0.2	< 0.2 0.25	
Gersthofen	1	0.6	300	87	Pod	0	6.87	< 0.05	6.87	DEU88H42831
Germany (1988) Lirabon					Seed	7 14	0.07 0.09	< 0.05 0.06	0.07 0.15	
Hoopte	1	0.6	300	85	Pod	0	10	0.12	10.12	DEU88H42811
Germany (1988) Ceres					Seed	7 14	0.12 0.12	0.08 0.07	0.20 0.19	
Wesseling	1	0.6	300	87	Pod	0	9.91	< 0.05	9.91	DEU88H42821
Germany (1988) Lirabon					Seed	7 14	0.12 0.14	0.12 0.12	0.24 0.26	
Wicker	1	0.6	300	87	Pod	0	7.53	< 0.05	7.53	DEU88H42841
Germany (1988) Liborius					Seed	7 14	0.05 < 0.05	< 0.05 < 0.05	0.05 < 0.05	

Hattersheim 1985: On day 0 the rape plants were cut and stacked (some pods were taken and prepared). On day 7 half of the seed was extracted from pods by threshing. On day 14 (seed maturity) the remaining seed was extracted from remaining pods.

All applications were made with a 200 SL formulation

GS: BBCH growth stages for oilseed rape (Meier, 2001):

- 83: 30% of pods ripe, seeds dark and hard
- 84: 40% of pods ripe, seeds dark and hard
- 85: 50% of pods ripe, seeds dark and hard
- 86: 60% of pods ripe, seeds dark and hard
- 87: 70% of pods ripe, seeds dark and hard
- 88: 80% of pods ripe, seeds dark and hard
- 89: Fully ripe: nearly all pods ripe, seeds dark and hard
- 97: Plant dead and dry
- 99: Harvested product

Table 105 Residues of glufosinate in glufosinate tolerant canola (oilseed rape)

TOLERANT CANOLA	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Chula USA (1999) Invigor	2 (49)	0.50 0.49	94 94	51–57	Seed	108	< 0.05	< 0.05	< 0.05	B003060
2373	2 (49)	0.50 0.49	94 94	51–57	Seed	108 [a]	< 0.05	< 0.05	< 0.05	+ AMS
Northwood USA (1999)	2 (15)	0.50 0.52	94 94	51–57	Seed	62	0.19	< 0.05	0.19	B003060
Invigor 2373	2 (15)	0.49 0.50	94 94	51–57	Seed	62 [a]	0.16	< 0.05	0.16	+ AMS
Jerome USA (1999)	2 (21)	0.50 0.50	94 94	51–57	Seed	65	0.22	< 0.05	0.22	B003060
Phoenix	2 (21)	0.50 0.50	94 94	51–57	Seed	65 [a]	0.18	< 0.05	0.18	+ AMS
Dayton USA (1999)	2 (24)	0.49 0.52	94 94	51–57	Seed	84	0.06	< 0.05	0.06	B003060
Phoenix	2 (24)	0.50 0.50	94 94	51–57	Seed	84 [a]	0.06	< 0.05	0.06	+ AMS
Walla Walla USA (1999)	2 (28)	0.50 0.50	94 94	34	Seed	41	3.17	0.14	3.31	B003060
Phoenix	2 (28)	0.50 0.50	94 94	34	Seed	41 [a]	7.62	0.28	7.90	+ AMS

[a] In these trials ammonium sulfate was used as an adjuvant while in the other trials no adjuvant was used.

All applications were made with a 200 SL formulation

Table 106 Residues of glufosinate in sunflower (pre-harvest desiccation)

SUNFLOWER	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Gersthofen	1	0.60	300	n.a.	Shoot	14	0.27	0.20	0.47	DEU85H40731
Germany (1985) Bulgar Sun					Seed	14	0.58	0.21	0.79	
Hattersheim	1	0.60	300	n.a.	Shoot	14	0.42	0.31	0.73	DEU85H40741
Germany (1985) Celaflor					Seed	14	0.31	0.12	0.43	
Bornheim Germany (1987) Carlisol	1	0.60	300	80	Seed	14	1.00	0.21	1.21	DEU87H40921
Gersthofen Germany (1987) Frankasol	1	0.60	300	n.a.	Seed	14	1.48	0.82	2.30	DEU87H40931
Kàpolnàsnyèk Hungary (2006) PR63A82	1	0.35	46	87	Seed	0 3 5 10 14	0.39 0.25 0.18 0.20 0.21	0.02 0.01 0.02 0.04 0.04	0.41 0.26 0.20 0.24 0.25	20061252/H1-FPSF
					Seed no shell	14	0.02	< 0.01	0.02	
Vereb Hungary (2006) NK BRIO	1	0.36	48	88	Seed	0 3 5 10 14	0.90 0.67 0.68 0.29 0.34	< 0.01 < 0.01 0.01 0.04 0.04	0.90 0.67 0.69 0.33 0.38	20061252/H1-FPSF
					Seed no shell	14	< 0.01	< 0.01	< 0.01	

SUNFLOWER	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Baracska Hungary (2006) PR64A82	1	0.34	45	88	Seed	0 3 5 10 14	0.32 0.33 0.31 0.47 0.25	< 0.01 < 0.01 0.01 0.02 0.02	0.32 0.33 0.32 0.49 0.27	20061252/H1-FPSF
					Seed no shell	14	< 0.01	< 0.01	< 0.01	
Enying Hungary (2006) PR64H61	1	0.34	44	87	Seed	0 3 5 10 14	0.98 0.86 0.47 0.35 0.38	0.02 0.06 0.06 0.08 0.08	1.00 0.92 0.53 0.43 0.46	20061252/H1-FPSF
					Seed no shell	14	0.19	0.01	0.20	
Füle Hungary (2006) Alexandra	1	0.36	47	87	Seed	0 3 5 10 14	0.05 0.05 0.08 0.05 0.05	< 0.01 < 0.01 0.01 < 0.01 < 0.01	0.05 0.05 0.09 0.05 0.05	20061252/H1-FPSF
					Seed no shell	14	< 0.01	< 0.01	< 0.01	

All applications were made with a 150 SL (Hungary trials) or 200 SL (Germany trials) formulation.

GS:, BBCH growth stages for sunflowers (Meier, 2001):

- 80: Beginning of ripening: seeds on outer third of anthocarp black and hard. Back of anthocarp still green
- 81: Seeds on outer third of anthocarp dark and hard. Back of anthocarp still green
- 83: Dark of anthocarp yellowish-green, bracts still green. Seeds about 50% dry matter
- 85: Seeds on middle third of anthocarp dark and hard. Back of anthocarp yellow, bracts brown edged. Seeds about 60% dry matter
- 87: Physiological ripeness: back of the anthocarp yellow. Bracts marbled brown. Seeds about 75–80% dry matter
- 89: Fully ripe: seeds on inner third of anthocarp dark and hard. Back of anthocarp brown. Bracts brown. Seeds about 85% dry matter

Table 107 Residues of glufosinate in coffee beans (directed spray for weed control)

COFFEE	Application						Residue (mg/kg)[a]			
Location, year variety	No	kg ai/ha	L/ha	GS	Sample [b]	PHI (d)	glufosinate	MPP	Total	Reference
Paulinia, Brazil (2007) Catuai	1	0.60	300	79	Beans	20	< 0.03	< 0.04	< 0.04	RA-1264/07 + lauryl ester sulphate
Uberlandia, Brazil (2007) Catui vermelho	1	0.60	300	79	Beans	20	< 0.03	< 0.04	< 0.04	RA-1265/07

All applications were made with a 200 SL formulation

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Berries were sampled 20 days after application and dried for further 20 days under ambient conditions; thereafter the pulp was removed from the berries using a laboratory-scale machine and the thus obtained beans were stored frozen.

Animal feeds

Table 108 Residues of glufosinate in forage of sweet corn glufosinate tolerant sweet corn

SWEET CORN	Application						Residue (mg/kg)[a]			
Location, year variety	No. (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
East Lansing, WI USA 1997 GH-0937	2 (14)	0.41 0.41	185 185	V8–10	Forage	46	0.74	0.06	0.80	06515 + AMS
Arlington, WI USA 1997 Rodgers 0937	2 (14)	0.41 0.41	191 188	V6–7	Forage	50	0.19	0.05	0.24	06515 + AMS 2nd spray only
	2 (14)	0.41 0.41	191 188	V6–7	Forage	50	0.19	< 0.05	0.19	
Fremont, OH USA 1998 GH 0937 Corn G	2 (14)	0.41 0.41	141 199	–	Forage	47	0.14	< 0.05	0.14	06515 + AMS
Arlington, WI USA 1998 GH 0937	2 (15)	0.41 0.41	186 190	V6	Forage	43	0.63	0.05	0.68	06515 + AMS
Freeville, NY USA 1999 GH 0937	2 (16)	0.43 0.41	283 273	5 collars	Forage	45	0.18	< 0.05	0.18	06953 Tractor oil leak, 10% necrosis
Freeville, NY USA 1999 GH 0937	2 (16)	0.44 0.42	289 277	5–6 collars	Forage	45	0.26	< 0.05	0.26	06953
Gainesville, FL USA 1999 GSS-0966VP	2 (14)	0.42 0.45	379 284	–	Forage	30	0.17	0.14	0.31	06953
	2 (14)	0.44 0.43	395 289	–	Forage	38	0.13	0.05	0.18	06953
Prosser, WA USA 1999 Liberty Link	2 (14)	0.42 0.42	144 145	–	Forage	44	0.34	< 0.05	0.34	06953
Holtville, CA USA 1999 Attribute	2 (14)	0.50 0.50	332 327	–	Forage	45	0.13	< 0.05	0.13	06953
Kimberly, ID USA 1999	2 (14)	0.44 0.43	246 236	–	Forage	46	0.70	0.05	0.75	06953

All applications were made with a 200 SL formulation.

[a] Analytical method measures NAG together with glufosinate as a common derivative

Table 109 Residues of glufosinate in fodder/stover of sweet corn glufosinate tolerant sweet corn

SWEET CORN	Application						Residue (mg/kg)[a]			
Location, year variety	No. (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
East Lansing, WI USA 1997 GH-0937	2 (14)	0.41 0.41	185 185	V8–10	Stover	46	1.62	0.11	1.73	06515 + AMS
Arlington, WI USA 1997 Rodgers 0937	2 (14)	0.41 0.41	191 188	V6–7	Stover	50	0.13	< 0.05	0.13	06515 + AMS 2nd spray only
	2 (14)	0.41 0.41	191 188	V6–7	Stover	50	0.19	< 0.05	0.19	
Fremont, OH USA 1998 GH 0937 Corn G	2 (14)	0.41 0.41	141 199	–	Stover	47	0.11	< 0.05	0.11	06515 + AMS
Arlington, WI USA 1998 GH 0937	2 (15)	0.41 0.41	186 190	V6	Stover	43	1.45	0.12	1.57	06515 + AMS

SWEET CORN	Application						Residue (mg/kg)[a]			
Location, year variety	No. (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Freeville, NY USA 1999 GH 0937	2 (16)	0.43 0.41	283 273	5 collars	Stover	45	1.90	0.05	1.95	06953 Tractor oil leak, 10% necrosis
Freeville, NY USA 1999 GH 0937	2 (16)	0.44 0.42	289 277	5–6 collars	Stover	45	2.14	0.08	2.22	06953
Gainesville, FL USA 1999 GSS-0966VP	2 (14)	0.42 0.45	379 284	–	Stover	30	0.80	0.48	1.28	06953
	2 (14)	0.44 0.43	395 289	–	Stover	38	0.59	0.15	0.74	06953
Prosser, WA USA 1999 Liberty Link	2 (14)	0.42 0.42	144 145	–	Stover	44	1.50	0.10	1.60	06953
Holtville, CA USA 1999 Attribute	2 (14)	0.50 0.50	332 327	–	Stover	45	0.40	0.06	0.46	06953
Kimberly, ID USA 1999	2 (14)	0.44 0.43	246 236	–	Stover	46	3.37	0.18	3.55	06953

The stover samples were dried for up to 13 days before freezing.

All applications were made with a 200 SL formulation.

[a] Analytical method measures NAG together with glufosinate as a common derivative

Table 110 Residues of glufosinate in forage of common (kidney) bean (pre-harvest desiccation and directed applications)

KIDNEY BEAN	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Stelle Germany 1985 Dufix	1	1.00	300	PH	Shoot	0 7 14	2.69 0.74 0.06	0.05 0.11 < 0.05	2.74 0.85 0.06	DEU85H41111
Bornheim Germany 1985 Marona	1	1.00	300	PH	Shoot	0 7 14	1.21 0.47 < 0.05	< 0.05 < 0.05 < 0.05	1.21 0.47 < 0.05	DEU85H41121
Elbstorf Germany 1986 Sperlings Dufrix	1	1.00	300	61	Shoot	0 7 14	0.13 0.16 < 0.05	< 0.05 < 0.05 < 0.05	0.13 0.16 < 0.05	DEU86H40811
Stelle Germany 1986 Hills Maja	1	1.00	300	61	Shoot	0 7 14	0.12 0.15 < 0.05	< 0.05 < 0.05 < 0.05	0.12 0.15 < 0.05	DEU86H40812
Bornheim Germany 1986 Hilds Marona	1	1.00	300	65	Shoot	0 7 14	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	DEU86H40821
Langenhain Germany 1986 Prelude	1	1.00	300	65	Shoot	9 20	0.22 0.19	< 0.05 < 0.05	0.22 0.19	DEU86H40826
Gersthofen Germany 1986 Prelude	1	1.00	300	63	Shoot	0 7 14	0.57 0.14 0.08	< 0.05 < 0.05 < 0.05	0.57 0.14 0.08	DEU86H40831
Hattersheim Germany 1986 Prelude	1	1.00	300	61	Shoot	0 7 14	0.5 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.5 < 0.05 < 0.05	DEU86H40841
Chilly France N 2008 Lingot	2 (27)	0.75 0.75	300 300	13	Green material	0	0.32	0.07	0.39	08-2123
Zwaagdijk-Oost Netherlands 2008 Allure	2 (27)	0.75 0.75	300 300	12	Green material	0	0.81	0.03	0.84	08-2123

KIDNEY BEAN	Application						Residue (mg/kg)			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Toulouse France S 2008 Myka	2 (30)	0.75 0.75	300 300	15	Green material	3	0.02	0.02	0.04	08-2134
Alginet Spain 2008 Cleo	2 (28)	0.75 0.75	300 300	14	Green material	0	0.11	0.04	0.15	08-2134
Fresnoy les Roye France N 2009 Lingot Suisse blanc	2 (28)	0.75 0.75	300 300	14	Green material	0	0.04	0.09	0.13	09-2137
Zwaagdijk Netherlands 2009 Scylla	2 (28)	0.75 0.75	300 300	19	Green material	0	0.58	< 0.01	0.58	09-2137
Toulouse France S 2009 Myka	2 (29)	0.75 0.75	300 300	12	Green material	0	2.1	0.08	2.18	09-2138
Ladispoli Italy 2009 Taylors	2 (28)	0.75 0.75	300 300	12	Green material	0	< 0.01	0.05	0.05	09-2138

PH = pre-harvest desiccation

1985 trials: Application was for pre-harvest desiccation

1986 trials: a directed application to weeds was made around flowering of the crop

2008/2009 trials: The first application was on the whole plot area about 5 days before sowing. The second application was between the plant rows using a spray shield at early post-emergence growth stages (BBCH 12-19).

All applications were made with a 200 SL formulation.

GS: BBCH growth stage codes for Bean (Meier, 2001):

- 61: Beginning of flowering: 10% of flowers open
- 62: 20% of flowers open
- 63: 30% of flowers open
- 64: 40% of flowers open
- 65: Full flowering: 50% of flowers open

Table 111 Residues of glufosinate in straw of common (kidney) bean (pre-harvest desiccation and directed applications)

KIDNEY BEAN	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Stelle Germany 1985 Dufix	1	1.00	300	PH	Straw	35	< 0.05	< 0.05	< 0.05	DEU85H41111
Bornheim Germany 1985 Marona	1	1.00	300	PH	Straw	42	< 0.1	< 0.1	< 0.1	DEU85H41121
Elbstorf Germany 1986 Sperlings Dufrix	1	1.00	300	61	Straw	67	< 0.05	0.63	0.63	DEU86H40811
Stelle Germany 1986 Hills Maja	1	1.00	300	61	Straw	57	< 0.05	< 0.05	< 0.05	DEU86H40812
Bornheim Germany 1986 Hilds Marona	1	1.00	300	65	Straw	69	< 0.05	< 0.05	< 0.05	DEU86H40821
Langenhain Germany 1986 Prelude	1	1.00	300	65	Straw	48	0.07	0.08	0.15	DEU86H40826
Gersthofen Germany 1986 Prelude	1	1.00	300	63	Straw	46	0.17	0.05	0.22	DEU86H40831
Hattersheim Germany 1986 Prelude	1	1.00	300	61	Straw	63	< 0.05	< 0.05	< 0.05	DEU86H40841

PH = pre-harvest desiccation

1985 trials: Application was for pre-harvest desiccation

1986 trials: a directed application to weeds was made around flowering of the crop

All applications were made with a 200 SL formulation.

Table 112 Residues of glufosinate in hulls of common (kidney) bean (pre-harvest desiccation and directed applications)

KIDNEY BEAN	Application						Residue (mg/kg)			
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Stelle Germany 1985 Dufix	1	1.00	300	PH	Hulls	35	< 0.05	< 0.05	< 0.05	DEU85H41111
Bornheim Germany 1985 Marona	1	1.00	300	PH	Hulls	42	< 0.1	< 0.1	< 0.1	DEU85H41121
Elbstorf Germany 1986 Sperlings Dufrix	1	1.00	300	61	Hulls	67	< 0.05	< 0.05	< 0.05	DEU86H40811
Stelle Germany 1986 Hills Maja	1	1.00	300	61	Hulls	57	< 0.05	< 0.05	< 0.05	DEU86H40812
Bornheim Germany 1986 Hilds Marona	1	1.00	300	65	Hulls	69	< 0.05	< 0.05	< 0.05	DEU86H40821
Langenhain Germany 1986 Prelude	1	1.00	300	65	Hulls	48	0.08	0.06	0.14	DEU86H40826
Gersthofen Germany 1986 Prelude	1	1.00	300	63	Hulls	46	0.12	0.07	0.19	DEU86H40831
Hattersheim Germany 1986 Prelude	1	1.00	300	61	Hulls	63	< 0.05	< 0.05	< 0.05	DEU86H40841

PH = pre-harvest desiccation

1985 trials: Application was for pre-harvest desiccation

1986 trials: a directed application to weeds was made around flowering of the crop

All applications were made with a 200 SL formulation.

Table 113 Residues of glufosinate in tops of glufosinate tolerant sugar beet

SUGAR BEET	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Fresno CA USA 1995 Liberty Link	2 (7)	0.60 0.60	102 100	16	Tops	10 15 30 60 139	3.28 2.41 1.20 0.51 0.06	0.24 0.48 1.28 0.78 0.24	3.52 2.89 2.48 1.29 0.30	A55839
	3 (8 7)	0.21 0.20 0.20	98 101 101		Tops	10 15 30 60 139	0.21 0.30 0.26 0.13 < 0.05	< 0.05 0.16 0.54 0.35 0.09	0.21 0.46 0.80 0.48 0.14	
	3 (8 7)	0.42 0.40 0.40	97 101 100		Tops	10 15 30 60 139	0.43 1.12 0.70 0.36 < 0.05	< 0.05 0.45 1.14 0.83 0.23	0.43 1.57 1.84 1.19 0.28	
Fisher MN USA 1995 Liberty Link	2 (7)	0.63 0.60	96 94		Tops	95	0.10	< 0.05	0.10	A55839

SUGAR BEET	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
	3 (10 8)	0.21 0.20 0.21	99 96 99		Tops	95	< 0.05	< 0.05	< 0.05	
	3 (8 7)	0.41 0.43 0.40	97 99 95		Tops	95	< 0.05	< 0.05	< 0.05	
Horace ND USA 1995 Liberty Link	2 (7)	0.60 0.62	94 95	19	Tops	104	0.08	< 0.05	0.08	A55839
	3 (9 6)	0.40 0.41 0.41	93 94 97		Tops	103	0.06	< 0.05	0.06	
	3 (4 10)	0.43 0.43 0.46	99 98 107		Tops	104	0.10	< 0.05	0.10	
Jerome ID USA 1995 Liberty Link	2 (7)	0.86 0.64	100 99		Tops	41	0.31	0.05	0.36	A55839
	3 (4 10)	0.21 0.21 0.22	99 97 103		Tops	41	0.08	< 0.05	0.08	
	3 (4 10)	0.43 0.43 0.46	99 98 107		Tops	41	0.22	< 0.05	0.22	
Conklin MI USA 1996	2 (4)	0.60 0.60	98 93		Tops	109	0.15	0.05	0.20	A57730
Liberty Link	3 (10 7)	0.60 0.35	92 95		Tops	106	0.30	< 0.05	0.30	
		0.60	90							
New Holland OH USA 1996 Liberty Link	2 (8)	0.60 0.60	94 89	18	Tops	83	0.16	< 0.05	0.16	A57730
	3 (8 14)	0.60 0.35	97 94	18	Tops	77	0.46	< 0.05	0.46	
		0.60	90							
Horace ND USA 1996	2 (5)	0.60 0.60	94 94	18	Tops	67	0.24	< 0.05	0.24	A57730
Liberty Link										
	3 (11 10)	0.60 0.35 0.60	94 94 94	18	Tops	62	0.58	< 0.05	0.58	
Scottsbluff NB USA 1996	2 (7)	0.60 0.60	94 94	18	Tops	115	< 0.05	< 0.05	< 0.05	A57730
Liberty Link										
	3 (18 14)	0.60 0.35 0.60	96 94 90	18	Tops	108	< 0.05	< 0.05	< 0.05	
Minot ND USA 1996	2 (7)	0.60 0.60	99 96	18	Tops	73	0.14	< 0.05	0.14	A57730
Liberty Link										
	3 (15 14)	0.60 0.35 0.60	94 94 95		Tops	66	0.24	0.07	0.31	
Eaton CO USA 1996	2 (14)	0.60 0.60	100 101	18	Tops	80	< 0.05	< 0.05	< 0.05	A57730
Liberty Link										
	3 (15 26)	0.60 0.35 0.60	94 96 102	18	Tops	68	0.38	< 0.05	0.38	
Campion CO USA 1996	2 (14)	0.60 0.60	97 99	18	Tops	86	0.06	< 0.05	0.06	A57730

SUGAR BEET	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	Total	Reference
Liberty Link										
	3 (15 26)	0.60 0.35 0.60	92 94 97	18	Tops	81	0.23	< 0.05	0.23	
Fresno CA USA 1996	2 (14)	0.60 0.60	92 95	18	Tops	132	0.06	< 0.05	0.06	A57730
Liberty Link										
	3 (10 19)	0.60 0.35 0.60	96 92 94	18	Tops	122	0.22	0.07	0.29	
Jerome ID USA 1996	2 (4)	0.60 0.60	95 95	19	Tops	128	0.08	< 0.05	0.08	A57730
Liberty Link										
	3 (7 14)	0.60 0.35 0.60	96 94 94		Tops	121	0.31	0.06	0.37	

All applications were made with a 200 SL formulations.

[a] Analytical method measures NAG together with glufosinate as a common derivative

Table 114 Residues of glufosinate in forage of glufosinate tolerant maize

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Webster City, Hamilton Co IA USA 1993	150SL	1	0.36	96	V5	Forage	30	0.07	< 0.05	0.43	0.50	A53691
		1	0.51	95	V5	Forage	30	0.11	< 0.05	0.59	0.70	
		1	0.5	103	V6	Forage	30	0.10	< 0.05	0.69	0.79	
York, York Co NE USA 1993	150SL	1	0.37	96	V6–7	Forage	30	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.51	95	V–-7	Forage	30	< 0.05	< 0.05	0.09	0.09	
		1	0.64	119	V9–10	Forage	30	0.17	< 0.05	1.42	1.59	
East Grand Forks, Polk Co MN USA 1993	150SL	1	0.52	49	V9	Forage	30	0.29	< 0.05	1.39	1.68	A53691
Bethany, Macon Co IL USA (1993)	150SL	1	0.38	97	V6	Forage	30	< 0.05	< 0.05	0.07	0.07	A53691
(LH59 × LH51) (LH119) (4) ×		1	0.53	94	V6	Forage	30	< 0.05	< 0.05	0.10	0.10	
(T14)		1	0.52	92	V8	Forage	30	0.06	< 0.05	0.38	0.44	
		2 (9)	0.36 0.53	93 93	V8, 61 cm	Forage	30	0.13	< 0.05	0.64	0.77	
Sheridan IN USA (1993) (LH216)	150SL	1	0.35	93	V5	Forage	30	0.06	< 0.05	0.40	0.46	A53691
(LH119) (4) × (T14)		1	0.51	96	V5	Forage	30	0.05	< 0.05	0.43	0.48	A53691
		2 (9)	0.38 0.55	100 96	V7, 107 cm	Forage	30	0.25	0.05	1.51	1.81	A53691
Horace ND USA (1993) (LH85 ×	150SL	1	0.35	92	V5–6	Forage	30	< 0.05	< 0.05	0.20	0.20	A53691

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
LH160) (LH119) (4) × (T14)		1	0.49	92	V5–6	Forage	30	0.06	< 0.05	0.29	0.35	
		2 (10)	0.35 0.49	92 91	V6, 48 cm	Forage	30	< 0.05	< 0.05	0.24	0.24	
Leonard MO USA (1993) (LH216)	150SL	1	0.37	105	V3–4	Forage	30	0.06	< 0.05	0.17	0.23	A53691
(LH119) (4) × (T14)		2 (10)	0.34 0.50	98 94	V7, 61 cm	Forage	30	0.24	< 0.05	0.99	1.23	
Madera CA USA (1993) (LH59 ×	150SL	1	0.37	99	V4–5	Forage	30	< 0.05	< 0.05	0.11	0.11	A53691
LH51) (LH119) (4) × (T14)		2 (13)	0.39 0.53	105 102	V5–8, 81 cm	Forage	30	0.12	0.11	0.53	0.76	
Renner Minnehaha Co SD USA	150SL	1	0.38	50	V8	Forage	30	0.07	< 0.05	0.14	0.21	A53691 [b]
(1993) (LH85 × LH160) (LH119) (4) ×		1	0.53	50	V8	Forage	30	0.06	< 0.05	0.30	0.36	
(T14)		2 (13)	0.37 0.52	48 51	V10, 61 cm	Forage	30	0.24	< 0.05	1.15	1.39	
Halifax VA USA (1993) (LH216)	150SL	1	0.37	49	V5	Forage	33	0.05	< 0.05	0.32	0.37	A53691 [c]
(LH119) (4) × (T14)		1	0.53	49	V6–7	Forage	33	0.06	< 0.05	0.38	0.44	
		2 (9)	0.37 0.53	49 49	V6–7, 61 cm	Forage	30	0.37	< 0.05	2.48	2.85	
Molino USA (1994) (LH51	150SL	2(13)	0.39 0.50	94 94	63 cm	Forage	29	2.11	< 0.05	[a]	2.11	A54160
× LH210) (LH119) (4) × (T14)	200SL	2 (13)	0.39 0.50	94 94	63 cm	Forage	29	1.75	< 0.05	[a]	1.75	
Webster City USA (1994) (LH59 ×	150SL	2 (8)	0.42 0.53	92 94	61 cm	Forage	31	0.63	< 0.05	[a]	0.63	A54160
LH51) (LH119) (4) × (T14)	200SL	2 (8)	0.40 0.50	96 93	61 cm	Forage	31	0.52	< 0.05	[a]	0.52	
Richmond USA (1994) (LH59 × LH51)	150SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Forage	31	0.26	< 0.05	[a]	0.26	A54160
(LH119) (4) × (T14)	200SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Forage	31	0.26	< 0.05	[a]	0.26	
East Grand Forks USA	150SL	2 (21)	0.38 0.48	91 90	71 cm	Forage	31	1.12	< 0.05	[a]	1.12	A54160
(1994) (LH74) (LH82) (3) × (T14)	200SL	2 (21)	0.39 0.50	92 94	71 cm	Forage	31	2.38	< 0.05	[a]	2.38	
Clarence USA (1994) (LH59 × LH51)	150SL	2 (13)	0.40 0.53	92 107	66 cm	Forage	30	1.58	< 0.05	[a]	1.58	A54160
(LH119) (4) × (T14)	200SL	2 (13)	0.39 0.56	93 112	66 cm	Forage	30	1.50	< 0.05	[a]	1.50	
Pikeville USA (1994) (LH51 × LH210)	150SL	2 (7)	0.40 0.46	96 94	61 cm	Forage	30	0.21	< 0.05	[a]	0.21	A54160

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
(LH119) (4) × (T14)	200SL	2 (7)	0.38 0.53	96 94	61 cm	Forage	30	0.20	< 0.05	[a]	0.20	
Phelps USA (1994) (LH74) (LH82) (3) ×	150SL	2 (10)	0.38 0.53	91 98	66 cm	Forage	30	3.55	< 0.05	[a]	3.55	A54160
(T14)	200SL	2 (10)	0.40 0.53	97 97	66 cm	Forage	30	1.20	< 0.05	[a]	1.20	
Levelland USA (1994)	150SL	2 (9)	0.38 0.46	92 91	61 cm	Forage	30	0.44	< 0.05	[a]	0.44	A54160
(LH51 × LH210) (LH119) (4) × (T14)	200SL	2 (9)	0.39 0.49	93 92	61 cm	Forage	30	0.52	< 0.05	[a]	0.52	
Ephrata USA (1994) (LH74) (LH82) (3) ×	150SL	2 (13)	0.39 0.52	94 94	61 cm	Forage	30	0.77	0.08	[a]	0.85	A54160
(T14)	200SL	2 (13)	0.40 0.50	94 92	61 cm	Forage	30	0.70	0.08	[a]	0.78	
Delevan USA (1994) (LH85 × LH160)	150SL	2 (9)	0.38 0.55	93 97	61 cm	Forage	30	0.61	< 0.05	[a]	0.61	A54160
(LH119) (4) × (T14)	200SL	2 (9)	0.38 0.50	93 97	61 cm	Forage	30	0.49	< 0.05	[a]	0.49	
Doniphan USA (1995) (LH51) (B73) (5) × (T25)	200SL	2 (9)	0.39 0.50	94 94	66 cm	Forage	32 62 77	1.29 0.96 1.12	< 0.05 < 0.05 0.08	[a]	1.29 0.96 1.20	A57728
Ottawa USA (1995) (LH168) (LH119) (9) × (T14)	200SL	2 (7)	0.39 0.50	94 93	71 cm	Forage	30 61 98	0.72 0.46 0.42	0.08 0.07 0.11	[a]	0.80 0.53 0.53	A57728
Yellow Medicine USA (1995) (LH202) (LH82) (4) × (T25)	200SL	2 (10)	0.39 0.49	94 92	V7–8	Forage	30 60	2.43 1.44	0.06 0.08	[a]	2.49 1.52	A57728
Shelby USA (1995) (LH51) (B73) (5) × (T25)	200SL	2 (10)	0.39 0.52	89 86	61 cm	Forage	10 20 29 40 60 81	4.06 1.50 1.02 0.82 0.49 0.17	0.07 0.05 < 0.05 < 0.05 0.05 0.05	[a]	4.13 1.55 1.02 0.82 0.54 0.22	A57728
York USA (1995) (LH51) (LH119) (9) × (T14)	200SL	2 (8)	0.39 0.52	94 94	61 cm	Forage	20 60 80	0.41 0.33 0.30	< 0.05 < 0.05 < 0.05	[a]	0.41 0.33 0.30	A57728
Fayette USA (1995) (LH51) (LH119) (9) × (T14)	200SL	2 (10)	0.39 0.52	90 94	61 cm	Forage	31 60 88	1.90 1.56 0.62	< 0.05 0.07 0.17	[a]	1.90 1.63 0.79	A57728
Uvalde USA (1995) (LH216) (B73) (5) × (T25)	200SL	2 (7)	0.40 0.50	96 94	66 cm	Forage	10 20 30 40 61 71	3.19 1.62 0.91 0.85 0.72 0.82	0.07 < 0.05 < 0.05 < 0.05 < 0.05 0.07	[a]	3.26 1.62 0.91 0.85 0.72 0.89	A57728
Hamburg PA USA (1996)	200SL	2 (20)	0.39 0.50	103 94	123 cm	Forage	71	0.10	< 0.05	[a]	0.10	A57829
Liberty Link		1	0.39	94	76–123 cm	Forage	71	0.08	< 0.05	[a]	0.08	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
		1	0.60	94	76–91 cm	Forage	71	0.09	< 0.05	[a]	0.09	
Chula GA USA (1996)	200SL	2 (16)	0.39 0.50	94 103	91 cm	Forage	52	0.22	0.48	[a]	0.70	A57829
Liberty Link		1	0.39	103	91 cm	Forage	52	0.06	0.08	[a]	0.14	
		1	0.60	103	91 cm	Forage	52	0.06	0.26	[a]	0.32	
Seven Springs NC USA (1996)	200SL	2 (13)	0.39 0.50	94 94	91 cm	Forage	62	0.11	0.08	[a]	0.19	A57829
Liberty Link		1	0.39	94	91 cm	Forage	62	0.10	< 0.05	[a]	0.10	
		1	0.59	94	91 cm	Forage	62	0.10	< 0.05	[a]	0.10	
Noblesville IN USA (1996)	200SL	2 (8)	0.39 0.50	94 94	91 cm	Forage	91	< 0.05	< 0.05	[a]	< 0.05	A57829
Liberty Link		1	0.39	94	91 cm	Forage	91	< 0.05	< 0.05	[a]	< 0.05	
		1	0.59	94	91 cm	Forage	91	< 0.05	< 0.05	[a]	< 0.05	
Danville IA USA (1996)	200SL	2 (13)	0.39 0.52	94 94	91 cm	Forage	63	0.30	< 0.05	[a]	0.30	A57829
Liberty Link		1	0.40	94	91 cm	Forage	63	0.38	0.06	[a]	0.44	
		1	0.62	94	91 cm	Forage	63	0.17	< 0.05	[a]	0.17	
New Holland OH USA (1996)	200SL	2 (23)	0.39 0.50	103 103	102 cm	Forage	74	0.22	< 0.05	[a]	0.22	A57829
Liberty Link		1	0.39	103	102 cm	Forage	74	0.08	< 0.05	[a]	0.08	
		1	0.59	103	102 cm	Forage	74	0.22	< 0.05	[a]	0.22	
Bethany IL USA (1996)	200SL	2 (14)	0.39 0.49	94 94	91 cm	Forage	92	0.10	< 0.05	[a]	0.10	A57829
Liberty Link		1	0.38	94	91 cm	Forage	92	0.05	< 0.05	[a]	0.05	
		1	0.57	94	91 cm	Forage	92	0.10	< 0.05	[a]	0.10	
Wester City IA USA (1996)	200SL	2 (19)	0.39 0.50	94 94	V9	Forage	86	0.08	< 0.05	[a]	0.08	A57829
Liberty Link		1	0.39	94	V9	Forage	86	0.10	< 0.05	[a]	0.10	
		1	0.59	94	V9	Forage	86	0.18	< 0.05	[a]	0.18	
Uvalde TX USA (1996)	200SL	2 (14)	0.37 0.50	94 94	91 cm	Forage	67	0.07	< 0.05	[a]	0.07	A57829
Liberty Link		1	0.40	94	91 cm	Forage	67	0.05	< 0.05	[a]	0.05	
		1	0.59	94	91 cm	Forage	67	0.12	< 0.05	[a]	0.12	
Palm Beach USA (1997)	200SL	2 (9)	0.39 0.50	94 94	61 cm	Forage	56	0.64	< 0.05	[a]	0.64	A57796
11962.20		2 (9)	0.40 0.49	94 94	61 cm	Forage	56	0.82	< 0.05	[a]	0.82	+ AMS
Uvalde USA (1997)	200SL	2 (14)	0.39 0.49	94 94	61 cm	Forage	55	0.50	< 0.05	[a]	0.50	A57796
11962.20		2 (14)	0.39 0.50	94 94	61 cm	Forage	55	0.28	< 0.05	[a]	0.28	+ AMS
Oahu USA (1997)	200SL	2 (8)	0.39 0.49	94 94	61 cm	Forage	76	1.11	0.05	[a]	1.11	A57796
11962.20		2 (8)	0.39 0.49	94 94	61 cm	Forage	76	0.80	< 0.05	[a]	0.80	+ AMS
Richland IA USA 2000 Glufosinate tolerant	200SL	2 (8)	0.41 0.48	143 138	61 cm	Forage	69	< 0.05	< 0.05	0.06	0.06	B003263 + AMS
Carlyle IL USA 2000 Glufosinate tolerant	200SL	2 (22)	0.41 0.49	145 143	61 cm	Forage	46	0.38	0.06	2.06	2.50	B003263 + AMS
New Holland OH USA 2000 Glufosinate tolerant	200SL	2 (14)	0.41 0.49	144 147	61 cm	Forage	61	< 0.05	< 0.05	< 0.05	< 0.05	B003263 + AMS

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Rochelle IL USA 2000 Glufosinate tolerant	200SL	2 (15)	0.41 0.49	141 138	61 cm	Forage	68	0.12	< 0.05	0.66	0.78	B003263 + AMS
Ellendale MN USA 2000 Glufosinate tolerant	200SL	2 (11)	0.43 0.49	167 163	61 cm	Forage	82	< 0.05	< 0.05	0.06	0.06	B003263 +AMS

Transgenic maize 1996 trials first spray broadcast, over-the-top, by ground rig, and the second application by drop nozzles directed to the bottom one-third of the plants. Plots C and D received only drop nozzle applications

Transgenic maize 1997 All applications were made broadcast, over-the-top, by backpack sprayer, in approximately 94 L/ha of spray solution. (no drop nozzles)

Transgenic maize 1995 no drop nozzles

Transgenic maize 1994 two formulations, no drop nozzles

Transgenic maize 1993 no drop nozzles

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Some phytotoxicity was experienced and yield of grain was low

[c] Phytotoxicity and unusually dry weather for the crops which were not irrigated resulted in limited collection of samples

Table 115 Residues of glufosinate in silage from glufosinate tolerant maize

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Webster City, Hamilton Co IA USA 1993	150SL	1	0.36	96	V5	Silage	60	< 0.05	< 0.05	0.18	0.18	A53691
		1	0.51	95	V5	Silage	60	< 0.05	< 0.05	0.14	0.14	
		1	0.5	103	V6	Silage	60	0.11	< 0.05	0.71	0.82	
York, York Co NE USA 1993	150SL	1	0.37	96	V6–7	Silage	72	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.51	95	V6–7	Silage	72	< 0.05	< 0.05	< 0.05	< 0.05	
		1	0.64	119	V9–10	Silage	60	0.05	< 0.05	0.40	0.45	
East Grand Forks, Polk Co MN USA 1993	150SL	1	0.52	49	V9	Silage	66	0.23	< 0.05	1.20	1.43	A53691
Bethany, Macon Co IL USA (1993) (LH59 × LH51) (LH119) (4) × (T14)	150SL	1	0.38	97	V6	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.53	94	V6	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	
		1	0.52	92	V8	Silage	61	< 0.05	< 0.05	0.18	0.18	
		2 (9)	0.36 0.53	93 93	V8, 61 cm	Silage	61	0.08	< 0.05	0.56	0.64	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Sheridan IN USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.35	93	V5	Silage	69	0.05	< 0.05	0.22	0.27	A53691
		1	0.51	96	V5	Silage	69	0.06	< 0.05	0.19	0.25	A53691
		2 (9)	0.38 0.55	100 96	V7, 107 cm	Silage	60	0.20	0.06	1.42	1.68	A53691
Horace ND USA (1993) (LH85 × LH160) (LH119) (4) × (T14)	150SL	1	0.35	92	V5–6	Silage	80	< 0.05	< 0.05	0.05	0.05	A53691
		1	0.49	92	V5–6	Silage	80	< 0.05	< 0.05	0.05	0.05	
		2 (10)	0.35 0.49	92 91	V6, 48 cm	Silage	72	< 0.05	< 0.05	0.11	0.11	
Leonard MO USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.37	105	V3–4	Silage	73	< 0.05	< 0.05	0.05	0.05	A53691
		2 (10)	0.34 0.50	98 94	V7, 61 cm	Silage	60	0.15	< 0.05	1.04	1.19	
Madera CA USA (1993) (LH59 × LH51) (LH119) (4) × (T14)	150SL	1	0.37	99	V4–5	Silage	73	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		2 (13)	0.39 0.53	105 102	V5–8, 81 cm	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	
Renner Minnehaha Co SD USA (1993) (LH85 × LH160) (LH119) (4) × (T14)	150SL	1	0.38	50	V8	Silage	71	< 0.05	< 0.05	< 0.05	< 0.05	A53691 [b]
		1	0.53	50	V8	Silage	71	< 0.05	< 0.05	0.09	0.09	
		2 (13)	0.37 0.52	48 51	V10, 61 cm	Silage	60	0.07	< 0.05	0.36	0.43	
Halifax VA USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.37	49	V5	Silage	60	< 0.05	< 0.05	0.11	0.11	A53691 [c]
		1	0.53	49	V6–7	Silage	60	0.05	< 0.05	0.22	0.27	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
		2 (9)	0.37 0.53	49 49	V6–7, 61 cm	Silage	61	0.52	0.24	2.30	3.06	
Molino USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2(13)	0.39 0.50	94 94	63 cm	Silage	60	0.70	< 0.05	[a]	0.70	A54160
	200SL	2 (13)	0.39 0.50	94 94	63 cm	Silage	60	0.84	0.06	[a]	0.90	
Webster City USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (8)	0.42 0.53	92 94	61 cm	Silage	60	0.37	< 0.05	[a]	0.37	A54160
	200SL	2 (8)	0.40 0.50	96 93	61 cm	Silage	60	0.40	< 0.05	[a]	0.40	
Richmond USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Silage	60	0.13	< 0.05	[a]	0.13	A54160
	200SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Silage	60	0.15	< 0.05	[a]	0.15	
East Grand Forks USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (21)	0.38 0.48	91 90	71 cm	Silage	60	1.41	< 0.05	[a]	1.41	A54160
	200SL	2 (21)	0.39 0.50	92 94	71 cm	Silage	60	1.64	< 0.05	[a]	1.64	
Clarence USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (13)	0.40 0.53	92 107	66 cm	Silage	60	1.19	< 0.05	[a]	1.19	A54160
	200SL	2 (13)	0.39 0.56	93 112	66 cm	Silage	60	1.20	< 0.05	[a]	1.20	
Pikeville USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2 (7)	0.40 0.46	96 94	61 cm	Silage	58	0.11	< 0.05	[a]	0.11	A54160
	200SL	2 (7)	0.38 0.53	96 94	61 cm	Silage	58	0.12	< 0.05	[a]	0.12	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Phelps USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (10)	0.38 0.53	91 98	66 cm	Silage	60	2.07	< 0.05	[a]	2.07	A54160
	200SL	2 (10)	0.40 0.53	97 97	66 cm	Silage	60	0.73	< 0.05	[a]	0.73	
Levelland USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2 (9)	0.38 0.46	92 91	61 cm	Silage	60	0.32	< 0.05	[a]	0.32	A54160
	200SL	2 (9)	0.39 0.49	93 92	61 cm	Silage	60	0.30	< 0.05	[a]	0.30	
Ephrata USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (13)	0.39 0.52	94 94	61 cm	Silage	60	0.24	0.05	[a]	0.29	A54160
	200SL	2 (13)	0.40 0.50	94 92	61 cm	Silage	60	0.24	0.10	[a]	0.34	
Delevan USA (1994) (LH85 × LH160) (LH119) (4) × (T14)	150SL	2 (9)	0.38 0.55	93 97	61 cm	Silage	60	0.36	< 0.05	[a]	0.36	A54160
	200SL	2 (9)	0.38 0.50	93 97	61 cm	Silage	60	0.19	< 0.05	[a]	0.19	

Transgenic maize 1996 trials first spray broadcast, over-the-top, by ground rig, and the second application by drop nozzles directed to the bottom one-third of the plants. Plots C and D received only drop nozzle applications

Transgenic maize 1997 All applications were made broadcast, over-the-top, by backpack sprayer, in approximately 94 L/ha of spray. (no drop nozzles)

Transgenic maize 1995 no drop nozzles

Transgenic maize 1994 two formulations, no drop nozzles

Transgenic maize 1993 no drop nozzles

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Some phytotoxicity was experienced and yield of grain was low

[c] Phytotoxicity and unusually dry weather for the crops which were not irrigated resulted in limited collection of samples

Table 116 Residues of glufosinate in fodder/stover of glufosinate tolerant maize

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Webster City, Hamilton Co IA USA 1993	150SL	1	0.36	96	V5	Fodder	118	< 0.05	< 0.05	0.18	0.18	A53691
		1	0.51	95	V5	Fodder	118	0.05	< 0.05	0.20	0.25	
		1	0.5	103	V6	Fodder	114	0.13	0.05	0.64	0.82	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
York, York Co NE USA 1993	150SL	1	0.37	96	V6–7	Fodder	107	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.51	95	V6–7	Fodder	107	< 0.05	< 0.05	< 0.05	< 0.05	
		1	0.64	119	V9–10	Fodder	95	0.13	0.10	0.61	0.84	
Bethany, Macon Co IL USA (1993) (LH59 × LH51) (LH119) (4) × (T14)	150SL	1	0.38	97	V6	Fodder	100	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.53	94	V6	Fodder	100	< 0.05	< 0.05	< 0.05	< 0.05	
		1	0.52	92	V8	Fodder	95	< 0.05	< 0.05	0.21	0.21	
		2 (9)	0.36 0.53	93 93	V8, 61 cm	Fodder	95	< 0.05	< 0.05	0.13	0.13	
Sheridan IN USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.35	93	V5	Fodder	115	0.07	0.05	0.16	0.28	A53691
		1	0.51	96	V5	Fodder	115	0.05	0.05	0.13	0.23	A53691
		2 (9)	0.38 0.55	100 96	V7, 107 cm	Fodder	106	0.36	0.16	1.38	1.90	A53691
Horace ND USA (1993) (LH85 × LH160) (LH119) (4) × (T14)	150SL	1	0.35	92	V5–6	Fodder	107	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		1	0.49	92	V5–6	Fodder	107	< 0.05	< 0.05	< 0.05	< 0.05	
		2 (10)	0.35 0.49	92 91	V6, 48 cm	Fodder	97	< 0.05	< 0.05	0.09	0.09	
Leonard MO USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.37	105	V3–4	Fodder	97	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		2 (10)	0.34 0.50	98 94	V7, 61 cm	Fodder	95	0.11	< 0.05	0.53	0.64	
Madera CA USA (1993) (LH59 × LH51) (LH119) (4) × (T14)	150SL	1	0.37	99	V4–5	Fodder	119	< 0.05	< 0.05	< 0.05	< 0.05	A53691
		2 (13)	0.39 0.53	105 102	V5–8, 81 cm	Fodder	106	0.09	0.11	0.58	0.78	
Renner Minnehaha Co SD USA (1993) (LH85 × LH160) (LH119) (4) × (T14)	150SL	1	0.38	50	V8	Fodder	95	< 0.05	< 0.05	0.08	0.08	A53691 [b]
		1	0.53	50	V8	Fodder	95	0.05	< 0.05	0.19	0.24	
		2 (13)	0.37 0.52	48 51	V10, 61 cm	Fodder	95	0.16	0.06	0.19	0.41	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Halifax VA USA (1993) (LH216) (LH119) (4) × (T14)	150SL	1	0.53	49	V6–7	Fodder	106	< 0.05	0.09	0.08	0.17	
		2 (9)	0.37 0.53	49 49	V6–7, 61 cm	Fodder	97	0.30	0.42	0.95	1.67	
Molino USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2(13)	0.39 0.50	94 94	63 cm	Fodder	69	1.02	0.21	[a]	1.43	A54160
	200SL	2 (13)	0.39 0.50	94 94	63 cm	Fodder	69	1.22	0.29	[a]	1.51	
Webster City USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (8)	0.42 0.53	92 94	61 cm	Fodder	122	0.24	< 0.05	[a]	0.24	A54160
	200SL	2 (8)	0.40 0.50	96 93	61 cm	Fodder	122	0.22	< 0.05	[a]	0.22	
Richmond USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Fodder	118	0.12	0.06	[a]	0.18	A54160
	200SL	2 (8)	0.39 0.50	94 94	V6 61 cm	Fodder	118	0.11	0.06	[a]	0.17	
East Grand Forks USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (21)	0.38 0.48	91 90	71 cm	Fodder	114	0.99	0.14	[a]	1.13	A54160
	200SL	2 (21)	0.39 0.50	92 94	71 cm	Fodder	114	2.57	0.23	[a]	2.80	
Clarence USA (1994) (LH59 × LH51) (LH119) (4) × (T14)	150SL	2 (13)	0.40 0.53	92 107	66 cm	Fodder	89	1.52	0.26	[a]	1.78	A54160
	200SL	2 (13)	0.39 0.56	93 112	66 cm	Fodder	89	1.22	0.22	[a]	1.44	
Pikeville USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2 (7)	0.40 0.46	96 94	61 cm	Fodder	92	0.07	< 0.05	[a]	0.07	A54160
	200SL	2 (7)	0.38 0.53	96 94	61 cm	Fodder	92	0.07	0.05	[a]	0.12	
Phelps USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (10)	0.38 0.53	91 98	66 cm	Fodder	88	5.23	0.10	[a]	5.33	A54160
	200SL	2 (10)	0.40 0.53	97 97	66 cm	Fodder	88	1.54	< 0.05	[a]	1.54	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Levelland USA (1994) (LH51 × LH210) (LH119) (4) × (T14)	150SL	2 (9)	0.38 0.46	92 91	61 cm	Fodder	97	0.26	0.07	[a]	0.33	A54160
	200SL	2 (9)	0.39 0.49	93 92	61 cm	Fodder	97	0.24	0.06	[a]	0.30	
Ephrata USA (1994) (LH74) (LH82) (3) × (T14)	150SL	2 (13)	0.39 0.52	94 94	61 cm	Fodder	86	0.56	0.12	[a]	0.68	A54160
	200SL	2 (13)	0.40 0.50	94 92	61 cm	Fodder	86	0.43	0.15	[a]	0.58	
Delevan USA (1994) (LH85 × LH160) (LH119) (4) × (T14)	150SL	2 (9)	0.38 0.55	93 97	61 cm	Fodder	107	0.43	< 0.05	[a]	0.43	A54160
	200SL	2 (9)	0.38 0.50	93 97	61 cm	Fodder	107	0.50	< 0.05	[a]	0.50	
Doniphan USA (1995) (LH51) (B73) (5) × (T25)	200SL	2 (9)	0.39 0.50	94 94	66 cm	Fodder	95	1.23	0.14	[a]	1.37	A57728
Ottawa USA (1995) (LH168) (LH119) (9) × (T14)	200SL	2 (7)	0.39 0.50	94 93	71 cm	Fodder	111	0.50	0.14	[a]	0.64	A57728
Yellow Medicine USA (1995) (LH202) (LH82) (4) × (T25)	200SL	2 (10)	0.39 0.49	94 92	V7–8	Fodder	100	1.23	0.10	[a]	1.33	A57728
Shelby USA (1995) (LH51) (B73) (5) × (T25)	200SL	2 (10)	0.39 0.52	89 86	61 cm	Fodder	90	0.51	0.18	[a]	0.69	A57728
York USA (1995) (LH51) (LH119) (9) × (T14)	200SL	2 (8)	0.39 0.52	94 94	61 cm	Fodder	89	0.53	< 0.05	[a]	0.53	A57728
Fayette USA (1995) (LH51) (LH119) (9) × (T14)	200SL	2 (10)	0.39 0.52	90 94	61 cm	Fodder	117	1.14	0.22	[a]	1.36	A57728
Uvalde USA (1995) (LH216) (B73) (5) × (T25)	200SL	2 (7)	0.40 0.50	96 94	66 cm	Fodder	84	1.55	0.14	[a]	1.69	A57728
Hamburg PA USA (1996) Liberty Link	200SL	2 (20)	0.39 0.50	103 94	123 cm	Stover	136	0.12	< 0.05	[a]	0.12	A57829
		1	0.39	94	76–123 cm	Stover	136	0.09	< 0.05	[a]	0.09	

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
		1	0.60	94	76–91 cm	Stover	136	0.08	< 0.05	[a]	0.08	
Chula GA USA (1996) Liberty Link	200SL	2 (16)	0.39 0.50	94 103	91 cm	Stover	94	0.28	0.80	[a]	1.08	A57829
		1	0.39	103	91 cm	Stover	94	0.06	0.23	[a]	0.29	
		1	0.60	103	91 cm	Stover	94	0.16	0.60	[a]	0.76	
Seven Springs NC USA (1996) Liberty Link	200SL	2 (13)	0.39 0.50	94 94	91 cm	Stover	87	0.10	0.18	[a]	0.28	A57829
		1	0.39	94	91 cm	Stover	87	0.06	< 0.05	[a]	0.06	
		1	0.59	94	91 cm	Stover	87	0.08	< 0.05	[a]	0.08	
Noblesville IN USA (1996) Liberty Link	200SL	2 (8)	0.39 0.50	94 94	91 cm	Stover	121	< 0.05	< 0.05	[a]	< 0.05	A57829
		1	0.39	94	91 cm	Stover	121	< 0.05	< 0.05	[a]	< 0.05	
		1	0.59	94	91 cm	Stover	121	< 0.05	< 0.05	[a]	< 0.05	
Danville IA USA (1996) Liberty Link	200SL	2 (13)	0.39 0.52	94 94	91 cm	Stover	108	0.22	0.06	[a]	0.28	A57829
		1	0.40	94	91 cm	Stover	108	0.09	< 0.05	[a]	0.09	
		1	0.62	94	91 cm	Stover	108	0.13	< 0.05	[a]	0.13	
New Holland OH USA (1996) Liberty Link	200SL	2 (23)	0.39 0.50	103 103	102 cm	Stover	103	0.29	0.06	[a]	0.35	A57829
		1	0.39	103	102 cm	Stover	103	0.40	0.06	[a]	0.46	
		1	0.59	103	102 cm	Stover	103	0.70	0.10	[a]	0.80	
Bethany IL USA (1996) Liberty Link	200SL	2 (14)	0.39 0.49	94 94	91 cm	Stover	108	0.13	< 0.05	[a]	0.13	A57829
		1	0.38	94	91 cm	Stover	108	0.06	< 0.05	[a]	0.06	
		1	0.57	94	91 cm	Stover	108	0.10	< 0.05	[a]	0.10	
Wester City IA USA (1996) Liberty Link	200SL	2 (19)	0.39 0.50	94 94	V9	Stover	114	0.20	< 0.05	[a]	0.20	A57829
		1	0.39	94	V9	Stover	114	0.16	< 0.05	[a]	0.16	
		1	0.59	94	V9	Stover	114	0.29	< 0.05	[a]	0.29	
Uvalde TX USA (1996) Liberty Link	200SL	2 (14)	0.37 0.50	94 94	91 cm	Stover	85	0.15	< 0.05	[a]	0.15	A57829
		1	0.40	94	91 cm	Stover	85	0.12	< 0.05	[a]	0.12	
		1	0.59	94	91 cm	Stover	85	0.22	< 0.05	[a]	0.22	
Palm Beach USA (1997) 11962.20	200SL	2 (9)	0.39 0.50	94 94	61 cm	Stover	72	0.85	0.15	[a]	1.00	A57796
		2 (9)	0.40 0.49	94 94	61 cm	Stover	72	1.16	0.25	[a]	1.41	+AMS
Uvalde USA (1997) 11962.20	200SL	2 (14)	0.39 0.49	94 94	61 cm	Stover	87	1.28	0.07	[a]	1.35	A57796
		2 (14)	0.39 0.50	94 94	61 cm	Stover	87	1.16	< 0.05	[a]	1.16	+AMS
Oahu USA (1997) 11962.20	200SL	2 (8)	0.39 0.49	94 94	61 cm	Stover	76	0.66	0.06	[a]	0.72	A57796
		2 (8)	0.39 0.49	94 94	61 cm	Stover	76	0.44	< 0.05	[a]	0.44	+AMS

MAIZE	Application							Residue (mg/kg)				
Location, year variety	Form	No (int)	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Richland IA USA 2000 Glufosinate tolerant	200SL	2 (8)	0.41 0.48	143 138	61 cm	Stover	97	< 0.05	0.06	0.10	0.16	B003263 +AMS
Carlyle IL USA 2000 Glufosinate tolerant	200SL	2 (22)	0.41 0.49	145 143	61 cm	Stover	86	0.98	0.20	2.76	2.94	B003263 +AMS
New Holland OH USA 2000 Glufosinate tolerant	200SL	2 (14)	0.41 0.49	144 147	61 cm	Stover	104	< 0.05	0.07	0.10	0.17	B003263 +AMS
Rochelle IL USA 2000 Glufosinate tolerant	200SL	2 (15)	0.41 0.49	141 138	61 cm	Stover	116	0.44	0.14	1.33	1.92	B003263 +AMS
Ellendale MN USA 2000 Glufosinate tolerant	200SL	2 (11)	0.43 0.49	167 163	61 cm	Stover	121	< 0.05	< 0.05	< 0.05	< 0.05	B003263 +AMS

Transgenic maize 1996 trials first spray broadcast, over-the-top, by ground rig, and the second application by drop nozzles directed to the bottom one-third of the plants. Plots C and D received only drop nozzle applications

Transgenic maize 1997 All applications were made broadcast, over-the-top, by backpack sprayer, in approximately 94 L/ha. (no drop nozzles)

Transgenic maize 1995 no drop nozzles

Transgenic maize 1994 two formulations, no drop nozzles

Transgenic maize 1993 no drop nozzles

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] Some phytotoxicity was experienced and yield of grain was low

Table 117 Effect of growth stage at application on residues in forage of glufosinate tolerant maize

MAIZE	Application						Residue (mg/kg)				
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Madera, Madera Co CA USA 1993	1	0.48	93	V3 15 cm	Forage	20	< 0.05	< 0.05	< 0.05	< 0.05	A53691
						30	< 0.05	< 0.05	< 0.05	< 0.05	
						40	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	85	V4–5 28 cm	Forage	20	0.23	0.12	1.11	1.46	
						30	0.05	0.05	0.28	0.38	
						40	< 0.05	< 0.05	0.13	0.13	
	1	0.50	101	V5 46 cm	Forage	20	0.24	0.07	1.26	1.57	
						30	0.13	0.05	0.57	0.75	
						40	0.07	< 0.05	0.36	0.43	
	1	0.50	95	V7 61 cm	Forage	20	0.46	0.10	1.59	2.15	
						30	0.36	0.10	1.51	1.97	
						40	0.17	0.07	0.76	1.00	
	1	0.50	100	V9–10 86 cm	Forage	20	0.33	0.06	0.93	1.29	
						30	0.25	0.06	0.76	1.07	
						40	0.16	< 0.05	0.59	0.75	

MAIZE	Application						Residue (mg/kg)				
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Leonard, Shelby Co USA 1993	1	0.48	93	V3 15 cm	Forage	20	< 0.05	< 0.05	< 0.05	< 0.05	A53691
						30	< 0.05	< 0.05	< 0.05	< 0.05	
						40	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	85	V4–5, 28 cm	Forage	20	< 0.05	< 0.05	0.13	0.13	
						30	< 0.05	< 0.05	0.10	0.10	
						40	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	101	V5 46 cm	Forage	20	0.08	< 0.05	0.56	0.64	
						30	0.05	< 0.05	0.23	0.28	
						40	< 0.05	< 0.05	0.10	0.10	
	1	0.50	95	V7 61 cm	Forage	20	0.40	2.64	< 0.05	3.04	
						30	0.33	1.85	0.05	2.23	
						40	0.39	2.00	< 0.05	2.39	
	1	0.50	100	V9-10 86 cm	Forage	20	0.14	< 0.05	0.75	0.89	
						30	0.10	< 0.05	0.72	0.82	
						40	0.10	< 0.05	0.72	0.82	

All applications were made with a 150 SL formulation

Table 118 Effect of growth stage at application on residues in silage of glufosinate tolerant maize

MAIZE	Application						Residue (mg/kg)				
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Madera, Madera Co CA USA 1993	1	0.48	93	V3 15 cm	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	A53691
	1	0.50	85	V4–5 28 cm	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	101	V5 46 cm	Silage	60	< 0.05	< 0.05	0.08	0.08	
	1	0.50	95	V7 61 cm	Silage	60	0.10	0.06	0.35	0.51	
	1	0.50	100	V9–10 86 cm	Silage	60	0.07	< 0.05	0.37	0.44	
Leonard, Shelby Co USA 1993	1	0.48	93	V3 15 cm	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	A53691
	1	0.50	85	V4–5, 28 cm	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	101	V5 46 cm	Silage	60	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	95	V7 61 cm	Silage	60	0.14	0.79	0.37	1.30	
	1	0.50	100	V9-10 86 cm	Silage	60	0.05	< 0.05	0.37	0.42	

All applications were made with a 150 SL formulation

Table 119 Effect of growth stage at application on residues in fodder of glufosinate tolerant maize

MAIZE	Application						Residue (mg/kg)				
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
Madera, Madera Co CA USA 1993	1	0.48	93	V3 15 cm	Fodder	127	< 0.05	< 0.05	< 0.05	< 0.05	A53691

MAIZE	Application						Residue (mg/kg)				
Location, year variety	No	kg ai/ha	L/ha	GS	sample	PHI (d)	glufosinate	MPP	NAG	Total	Reference
	1	0.50	85	V4–5 28 cm	Fodder	120	< 0.05	< 0.05	0.06	0.06	
	1	0.50	101	V5 46 cm	Fodder	114	< 0.05	0.06	0.13	0.19	
	1	0.50	95	V7 61 cm	Fodder	96	0.20	0.11	0.94	1.25	
	1	0.50	100	V9–10 86 cm	Fodder	96	0.20	0.07	1.80	2.07	
Leonard, Shelby Co USA 1993	1	0.48	93	V3 15 cm	Fodder	127	< 0.05	< 0.05	< 0.05	< 0.05	A53691
	1	0.50	85	V4–5, 28 cm	Fodder	120	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	101	V5 46 cm	Fodder	114	< 0.05	< 0.05	< 0.05	< 0.05	
	1	0.50	95	V7 61 cm	Fodder	96	0.20	0.80	< 0.05	1.00	
	1	0.50	100	V9-10 86 cm	Fodder	96	0.09	< 0.05	0.29	0.38	

All applications were made with a 150 SL formulation

Table 120 Residues of glufosinate in straw of glufosinate tolerant rice

RICE	Application						Residue (mg/kg) [a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
West Memphis AR	2 (19)	0.50 0.49	47 47	16	Straw	102	< 0.05	0.06	0.06	A57819
USA (1996) Glufosinate tolerant Bengal	1	1.00	47		Straw	71	3.82	0.31	4.13	
Rosa USA (1996) Glufosinate	2 (16)	0.52 0.52	47 47	16	Straw	80	0.17	0.10	0.27	A57819
tolerant Bengal	1	1.04	47		Straw	62	3.35	0.30	3.65	
Greenville USA (1996)	2 (9)	0.52 0.50	47 47	16	Straw	92	< 0.05	< 0.05	< 0.05	A57819
Glufosinate tolerant Bengal	1	1.00	47		Straw	66	4.35	0.54	4.89	
Pattison USA (1996)	2 (15)	0.53 0.52	47 47	16	Straw	84	< 0.05	0.06	0.06	A57819
Glufosinate tolerant Bengal	1	1.02	47		Straw	68	0.27	1.22	1.49	
Rosa USA (1997) Glufosinate tolerant Taipei 309	2 (21)	0.50 0.50	94 94	22–23	Straw	97	0.16	0.10	0.26	A59924
Greenville USA (1997) Glufosinate tolerant Taipei 309	2 (23)	0.50 0.53	94 94	22–23	Straw	103	0.06	0.06	0.12	A59924
East Bernard USA (1997) Glufosinate tolerant Taipei 309	2 (15)	0.50 0.50	94 94	22–23	Straw	90	< 0.05	0.16	0.16	A59924

RICE	Application						Residue (mg/kg) [a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Hamilton City USA (1998)	2 (14)	0.50 0.50	94 94	23	Straw	90	< 0.05	0.14	0.14	B002652
Glufosinate tolerant M-202	2 (14)	0.50 0.50	94 94	23	Straw	90	< 0.05	0.12	0.12	+ AMS [b]
Live Oak USA (1998) Glufosinate tolerant M-202	2 (24)	0.51 0.51	94 94	23	Straw	89	0.08	0.16	0.24	B002652
Newport USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.50 0.50	93 94	23	Straw	96	0.20	0.14	0.34	B002973
Oil Trough USA (1999) Glufosinate tolerant Bengal 62	2 (22)	0.51 0.51	93 94	23	Straw	94	< 0.05	0.05	0.05	B002973
Proctor USA (1999) Glufosinate tolerant Bengal 62	2 (26)	0.50 0.50	92 95	24	Straw	86	0.05	0.06	0.11	B002973
Blackfish Lake USA (1999) Glufosinate tolerant Bengal 62	2 (26)	0.50 0.51	95 93	24	Straw	85	0.13	0.12	0.25	B002973
Stuttgart USA (1999)	2 (15)	0.50 0.54	100 96	23	Straw	95	< 0.05	0.06	0.06	B002973
Glufosinate tolerant Bengal 62	2 (13)	0.51 0.52	91 93	22	Straw	106	< 0.05	< 0.05	< 0.05	+ AMS
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.50 0.50	95 95	23	Straw	78 84 90 96	0.61 0.29 0.45 0.42	0.32 0.15 0.18 0.23	0.93 0.44 0.43 0.65	B002973
	2 (23)	0.51 0.51	95 95	23	Straw	78 84 90 96	0.24 0.32 0.14 0.28	0.17 0.16 0.10 0.20	0.41 0.48 0.24 0.48	+ AMS [b]
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.50 0.51	93 94	24	Straw	81	0.26	0.11	0.37	B002973
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (18)	0.49 0.51	90 94	23	Straw	70	0.53	0.09	0.62	B002973
Washington USA (1999) Glufosinate tolerant Bengal 62	2 (28)	0.51 0.51	95 96	24	Straw	70	0.87	0.43	1.30	B002973

RICE	Application						Residue (mg/kg) [a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Greenville USA (1999) Glufosinate tolerant Bengal 62	2 (22)	0.50 0.50	94 93	24	Straw	77	0.15	0.15	0.30	B002973
Shaw USA (1999) Glufosinate tolerant Bengal 62	2 (29)	0.50 0.50	93 93	23	Straw	79	< 0.05	0.05	0.05	B002973
Dexter USA (1999) Glufosinate tolerant Bengal 62	2 (32)	0.50 0.50	95 93	24	Straw	80 84 88 92 96	0.10 0.15 0.08 0.09 0.06	0.09 0.14 0.08 0.08 0.06	0.19 0.29 0.18 0.17 0.12	B002973
	2 (32)	0.50 0.50	95 95	24	Straw	80 84 88 92 96	0.06 0.09 0.08 < 0.05 0.06	0.06 0.11 0.08 0.06 0.08	0.12 0.20 0.16 0.06 0.14	+ AMS [b]
Benton USA (1999) Glufosinate tolerant Bengal 62	2 (17)	0.51 0.52	95 90	24	Straw	86	0.51	0.12	0.63	B002973
East Bernard USA (1999)	2 (12)	0.50 0.50	94 103	24	Straw	78	0.34	0.20	0.54	B002973
Glufosinate tolerant Bengal 62	2 (12)	0.50 0.50	94 103	24	Straw	78	0.24	0.16	0.40	+ AMS [b]
Brookshire USA (1999) Glufosinate tolerant Bengal 62	2 (23)	0.51 0.51	94 93	24	Straw	91	0.17	0.18	0.35	B002973

Flood timing 1 = common timing for the trial location.

Flood timing 2 = flood two days after the second application.

Flood timing 3 = flood prior to application, drain for application, re-flood two days later

[a] Analytical method measures NAG together with glufosinate as a common derivative

[b] In these trials ammonium sulfate was used as an adjuvant. They were conducted parallel to trials without adjuvant.

All applications were made with a 200 SL formulation

Table 121 Residues of glufosinate in glufosinate tolerant cotton gin trash

COTTON SEED	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
Greenville USA (1998)	2 (35)	0.59 0.58	94 93	61	Gin trash	68	2.86	0.50	3.36	C003708
LL-8B-Cotton-M, Line Cot05	3 (22 13)	0.58 0.58 0.58	94 93 93	61	Gin trash	68	2.08	0.56	2.64	
West Memphis USA	2 (53)	0.58 0.58	96 95	61	Gin trash	70	3.50	0.55	4.05	C003708
(1998) LL-8B-Cotton-M, Line Cot05	3 (25 28)	0.58 0.58 0.58	96 93 95	61	Gin trash	70	3.72	0.64	4.36	

COTTON SEED	Application						Residue (mg/kg)[a]			
Location, year variety	No (int)	kg ai/ha	L/ha	GS	Sample	PHI (d)	glufosinate	MPP	Total	Reference
East Bernard USA (1998)	2 (40)	0.58 0.58	96 92	61	Gin trash	70	1.28	0.21	1.49	C003708
LL-8B-Cotton-M,Line Cot05	3 (15 25)	0.58 0.58 0.58	96 101 92	61	Gin trash	70	1.58	0.26	1.84	
Edmonson USA (1998)	2 (40)	0.56 0.59	94 98	61	Gin trash	69	0.55	0.10	0.65	C003708
LL-8B-Cotton-M,Line Cot05	3 (21 19)	0.57 0.58 0.59	96 99 98	61	Gin trash	69	0.94	0.16	1.10	
Levelland USA (1998)	2 (40)	0.57 0.58	91 94	61	Gin trash	70	6.79	0.46	7.45	C003708
LL-8B-Cotton-M,Line Cot05	3 (19 21)	0.57 0.58 0.58	91 93 93	65	Gin trash	70	10.2	0.48	10.7	
Eakly USA (1998) LL-8B-	2 (22)	0.58 0.60	95 97	61	Gin trash	70	0.34	0.28	0.62	C003708
Cotton-M, Line Cot05	3 (14 8)	0.58 0.59 0.59	94 97 96	61	Gin trash	70	0.61	4.10	4.71	
Dill City USA (1998) LL-8B-	2 (21)	0.58 0.58	93 93	61	Gin trash	69	0.89	0.25	1.14	C003708
Cotton-M, Line Cot05	3 (14 7)	0.59 0.58 0.59	93 94 93	61	Gin trash	69	0.38	0.69	1.07	

All applications were made with a 200 SL formulation

[a] Analytical method measures NAG together with glufosinate as a common derivative

Rotational crops

Residues of glufosinate in rotational crops were generally < LOQ with finite residues only detected in livestock feeds such as forage and straw.

Table 122 Residues of glufosinate in rotational barley crops

BARLEY	Application					Residue (mg/kg)				
Location, year variety	No (int)	kg ai/ha	GS	sample	DAP (d)	glufosinate	MPP	NAG	Total	Reference
Bellata	3 (14	0.76	pre-	Forage	92	< 0.10	< 0.10	< 0.10	< 0.10	BCS-0205-01
Australia	14)	0.78	seed.	Grain	188	< 0.10	< 0.10	< 0.10	< 0.10	
(2007) Grout		0.78	PBI: 57 d	Straw	188	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (14	0.77	pre-.	Forage	59	< 0.10	< 0.10	< 0.10	< 0.10	
	19)	0.79	seed	Grain	155	< 0.10	< 0.10	< 0.10	< 0.10	
		0.77	PBI: 24 d	Straw	155	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (9	0.77	pre-.	Forage	35	< 0.10	< 0.10	< 0.10	< 0.10	
	15)	0.77	seed	Grain	131	< 0.10	< 0.10	< 0.10	< 0.10	
		0.77	PBI: 0 d	Straw	131	< 0.10	< 0.10	< 0.10	< 0.10	
Cambooya	3 (14	0.73	pre-.	Forage	106	< 0.10	< 0.10	< 0.10	< 0.10	BCS-0205-01
Australia	14)	0.73	seed	Grain	210	< 0.10	< 0.10	< 0.10	< 0.10	
(2007) Binalong		0.76	PBI: 55 d	Straw	210	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (14	0.76	pre-.	Forage	78	< 0.10	< 0.10	< 0.10	< 0.10	
	14)	0.76	seed	Grain	182	< 0.10	< 0.10	< 0.10	< 0.10	

BARLEY	Application					Residue (mg/kg)				
Location, year variety	No (int)	kg ai/ha	GS	sample	DAP (d)	glufosinate	MPP	NAG	Total	Reference
		0.76	PBI: 27 d	Straw	182	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (14	0.76	pre-.	Forage	51	< 0.10	< 0.10	< 0.10	< 0.10	
	14)	0.76	seed	Grain	155	< 0.10	< 0.10	< 0.10	< 0.10	
		0.74	PBI: 0 d	Straw	155	< 0.10	< 0.10	< 0.10	< 0.10	

pre-seed. : before sowing / seeding.

PBI : plant back interval (in days)

DAP: days after sowing or planting

Moisture contents: Bellata: forage 84.7%, straw 8.9%; Cambooya: forage 86.1%, straw 18.5%.

Table 123 Residues of glufosinate in rotational chickpea crops

CHICKPEA	Application					Residue (mg/kg)				
Location, year variety	No (int)	kg ai/ha	GS	sample	DAP (d)	glufosinate	MPP	NAG	Total	Reference
Wee Waa	3 (14	0.78	pre-	Forage	162	< 0.10	< 0.10	< 0.10	< 0.10	BCS-0194-01
Australia	19)	0.79	seed.	Grain	255	< 0.10	< 0.10	< 0.10	< 0.10	
(2007) Genesis 508		0.75	PBI: 108 d	Straw	255	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (14	0.78	pre-.	Forage	126	< 0.10	< 0.10	< 0.10	< 0.10	
	13)	0.76	seed	Grain	219	< 0.10	< 0.10	< 0.10	< 0.10	
		0.78	PBI: 72 d	Straw	219	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (15	0.77	pre-.	Forage	82	< 0.10	< 0.10	< 0.10	< 0.10	
	15)	0.75	seed	Grain	175	< 0.10	< 0.10	< 0.10	< 0.10	
		0.77	PBI: 28 d	Straw	175	< 0.10	< 0.10	< 0.10	< 0.10	
Cambooya	3 (16	0.74	pre-.	Forage	177	< 0.10	< 0.10	< 0.10	< 0.10	BCS-0194-01
Australia	12)	0.74	seed	Grain	281	< 0.10	< 0.10	< 0.10	< 0.10	
(2007) Howzat		0.73	PBI: 115 d	Straw	281	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (15	0.76	pre-.	Forage	134	< 0.10	< 0.10	< 0.10	< 0.10	
	13)	0.76	seed	Grain	238	< 0.10	< 0.10	< 0.10	< 0.10	
		0.77	PBI: 72 d	Straw	238	< 0.10	< 0.10	< 0.10	< 0.10	
	3 (14	0.76	pre-.	Forage	92	< 0.10	< 0.10	< 0.10	< 0.10	
	14)	0.76	seed	Grain	196	< 0.10	< 0.10	< 0.10	< 0.10	
		0.77	PBI: 30 d	Straw	196	< 0.10	< 0.10	< 0.10	< 0.10	

pre-seed: before sowing / seeding.

PBI : plant back interval (in days)

DAP: days after sowing or planting

Moisture contents: Wee Waa: forage 79%, straw 28%; Cambooya: forage 83%, straw 65%.

Table 124 Residues of glufosinate in rotational wheat crops grown after potatoes

ROTATION		Application					Residue (mg/kg)[a]			
Location, year (variety)	crop	No	kg ai/ha	GS	sample	DALT (d)	glufosinate	MPP	Total	Reference
Germansville)	Potato (T)	1	0.47	95		–	–	–	–	B003783
USA (2000)	Winter wheat			PBI : 30 d	Forage	221	< 0.05	< 0.05	< 0.05	
	Hopewell (R1)				Hay	269	< 0.05	< 0.05	< 0.05	

ROTATION		Application					Residue (mg/kg)[a]			
Location, year (variety)	crop	No	kg ai/ha	GS	sample	DALT (d)	glufosinate	MPP	Total	Reference
					Grain	293	< 0.05	< 0.05	< 0.05	
					Straw	293	< 0.05	< 0.05	< 0.05	
Jerome USA	Potato (T)	1	0.45	95			–	–	–	B003783
(2000)	Winter wheat			PBI : 31 d	Forage	231	< 0.05	< 0.05	< 0.05	
	Stephens (R1)				Hay	283	< 0.05	< 0.05	< 0.05	
					Grain	333	< 0.05	< 0.05	< 0.05	
					Straw	333	< 0.05	< 0.05	< 0.05	
Ephrata USA	Potato (T)	1	0.45	48			–	–	–	B003783
(2000)	Winter wheat			PBI : 31 d	Forage	235	< 0.05	< 0.05	< 0.05	
	Stevens (R1)				Hay	258	< 0.05	< 0.05	< 0.05	
					Grain	321	< 0.05	< 0.05	< 0.05	
					Straw	321	< 0.05	< 0.05	< 0.05	

pre-seed: before sowing / seeding.

PBI : plant back interval (in days)

No. = number of applications;

GS = growth stage at last application (BBCH);

DALT = days after last treatment.

(T) : denotes the treated / primary crop

(R1) : denotes the first rotational crop.

[a] Analytical method measures NAG together with glufosinate as a common derivative

Table 125 Residues of glufosinate in rotational crops (mustard green, turnip, wheat)

ROTATIONAL		Application					Residue (mg/kg)				
Location, year	Crop, variety	No	kg ai/ha	GS	sample	DALT (d)	glufosinate	MPP	NAG	Total	Reference
Leland USA	Cotton (T)	3	0.56–0.59								
(2003)	Mustard greens (R1)			PBI 70 d	Leaf	221	< 0.05	< 0.05	[a]	< 0.05	RAGLX002
	Florida										
	Broadleaf										
Leland USA	Cotton (T)	3	0.58–0.59								
(2003)	Turnip			PBI	Tops	221	< 0.05	< 0.05	[a]	< 0.05	
	Purple top			70 d							
	White (R1)				Root	221	< 0.05	< 0.05	[a]	< 0.05	
Leland USA	Cotton (T)	3	0.58								
(2003)	Winter			PBI	Forage	235	< 0.05	< 0.05	[a]	< 0.05	
	Wheat				Hay	264	< 0.05	< 0.05	[a]	< 0.05	
	Cocker				Grain	289	< 0.05	< 0.05	[a]	< 0.05	
	9152 (R1)				Straw	289	< 0.05	< 0.05	[a]	< 0.05	
Leland USA	Cotton (T)	3	0.57–0.58								
(2003)	Spring			PBI	Forage	263	< 0.05	< 0.05	[a]	< 0.05	
	Wheat			213	Hay	283	< 0.05	< 0.05	[a]	< 0.05	
	Alsen (R1)			d	Grain	305	< 0.05	< 0.05	[a]	< 0.05	
					Straw	305	< 0.05	< 0.05	[a]	< 0.05	
Uvalde USA	Cotton (T)	3	0.56–0.59								
(2003)	Mustard greens (R1)			PBI 69 d	Leaf	221	< 0.05	< 0.05	[a]	< 0.05	
	Southern										

ROTATIONAL		Application					Residue (mg/kg)				
Location, year	Crop, variety	No	kg ai/ha	GS	sample	DALT (d)	glufosinate	MPP	NAG	Total	Reference
	giant curl										
Uvalde USA	Cotton (T)	3	0.58–0.60								
(2003)	Turnip			PBI	Tops	236	< 0.05	< 0.05	[a]	< 0.05	
	Purple top										
	White (R1)				Root	236	< 0.05	< 0.05	[a]	< 0.05	
Uvalde USA	Cotton (T)	3	0.58								
(2003)	Winter			PBI	Forage	166	< 0.05	< 0.05	[a]	< 0.05	
	Wheat				Hay	243	< 0.05	< 0.05	[a]	< 0.05	
	Ogallala				Grain	293	< 0.05	< 0.05	[a]	< 0.05	
	(R1)				Straw	293	< 0.05	< 0.05	[a]	< 0.05	
Uvalde USA	Cotton (T)	3	0.57–0.58								
(2003)	Spring			PBI	Forage	238	< 0.05	< 0.05	[a]	< 0.05	
	Wheat				Hay	262	< 0.05	< 0.05	[a]	< 0.05	
	Norlander				Grain	307	< 0.05	< 0.05	[a]	< 0.05	
	(R1)				Straw	307	< 0.05	< 0.05	[a]	< 0.05	
Fresno USA	Cotton (T)	3	0.57–0.58								
(2003)	Mustard greens (R1)			PBI 70 d	Leaf	138	< 0.05	< 0.05	[a]	< 0.05	
	Florida										
	Broadleaf										
Fresno USA	Cotton (T)	3	0.57–0.58								
(2003)	Turnip			PBI	Tops	145	< 0.05	< 0.05	[a]	< 0.05	
	Purple top			70 d							
	Whitee (R1)				Root	145	< 0.05	< 0.05	[a]	< 0.05	
Fresno USA	Cotton (T)	3	0.57–0.58								
(2003)	Winter			PBI	Forage	159	< 0.05	< 0.05	[a]	< 0.05	
	Wheat			70 d	Hay	267	< 0.05	< 0.05	[a]	< 0.05	
	Kronos				Grain	310	< 0.05	< 0.05	[a]	< 0.05	
	(R1)				Straw	310	< 0.05	0.14	[a]	0.14	
Fresno USA	Cotton (T)	3	0.57–0.58								
(2003)	Spring			PBI	Forage	261	< 0.05	< 0.05	[a]	< 0.05	
	Wheat			174	Hay	300	< 0.05	< 0.05	[a]	< 0.05	
	Kronos			d	Grain	324	< 0.05	< 0.05	[a]	< 0.05	
	(R1)				Straw	324	< 0.05	< 0.05	[a]	< 0.05	
Molino USA	Cotton (T)	2	0.86	18							RAGLP003
(2005)	Mustard greens (R1)			PBI 80 d	Leaf	136	< 0.05	< 0.05	< 0.05	< 0.05	
	Florida										
	Broadleaf										
Molino USA	Cotton (T)	2	0.85–86	18							
(2005)	Turnip			PBI	Tops	136	< 0.05	< 0.05	< 0.05	< 0.05	
	Purple top			80 d							
	White globe (R1)				Root	136	< 0.05	< 0.05	< 0.05	< 0.05	
Molino USA	Cotton (T)	2	0.86–0.88	61							
(2005)	Spring			PBI	Forage	171	< 0.05	< 0.05	< 0.05	< 0.05	
	Wheat			78 d	Hay	234	< 0.05	< 0.05	< 0.05	< 0.05	
	Gore				Grain	267	< 0.05	< 0.05	< 0.05	< 0.05	
	(R1)				Straw	267	< 0.05	< 0.05	< 0.05	< 0.05	
Proctor USA	Cotton (T)	2	0.88	55							RAGLP003
(2005)	Mustard, Indian (R1)			PBI 80 d	Leaf	161	< 0.05	< 0.05	< 0.05	< 0.05	

ROTATIONAL		Application					Residue (mg/kg)				
Location, year	Crop, variety	No	kg ai/ha	GS	sample	DALT (d)	glufosinate	MPP	NAG	Total	Reference
	Florida										
	Broadleaf										
Proctor USA	Cotton (T)	2	0.88	55							
(2005)	Turnip			PBI	Tops	187	< 0.05	< 0.05	< 0.05	< 0.05	
	Purple top			80 d							
	(R1)				Root	187	< 0.05	< 0.05	< 0.05	< 0.05	
Proctor USA	Cotton (T)	2	0.88	55							
(2005)	Spring			PBI	Forage	256	< 0.05	< 0.05	< 0.05	< 0.05	
	Wheat			80 d	Hay	267	< 0.05	< 0.05	< 0.05	< 0.05	
	DK 9410				Grain	322	< 0.05	< 0.05	< 0.05	< 0.05	
	(R1)				Straw	322	< 0.05	< 0.05	< 0.05	< 0.05	
Proctor USA	Cotton (T)	2	0.88	55							
(2005)	Spring			PBI	Forage	276	< 0.05	< 0.05	< 0.05	< 0.05	
	Wheat			100	Hay	287	< 0.05	< 0.05	< 0.05	< 0.05	
	DK 9410			d	Grain	342	< 0.05	< 0.05	< 0.05	< 0.05	
	(R1)				Straw	342	< 0.05	< 0.05	< 0.05	< 0.05	
Fresno USA	Cotton (T)	2	0.87–0.89	55							RAGLP003
(2005)	Mustard, Indian (R1)			PBI 80 d	Leaf	174	< 0.05	< 0.05	< 0.05	< 0.05	
	Florida										
	Broadleaf										
Fresno USA	Cotton (T)	2	0.86–0.88	61							
(2005)	Turnip			PBI	Tops	174	< 0.05	< 0.05	< 0.05	< 0.05	
	Purple top			80 d							
	White globe (R1)				Root	174	< 0.05	< 0.05	< 0.05	< 0.05	
Fresno USA	Cotton (T)	2	0.88	61							
(2005)	Spring			PBI	Forage	194	< 0.05	0.10	< 0.05	0.10	
	Wheat			77 d	Hay	264	< 0.05	0.11	< 0.05	0.11	
	Summit				Grain	319	< 0.05	< 0.05	< 0.05	< 0.05	
	(R1)				Straw	319	< 0.05	0.08	< 0.05	0.08	
Fresno USA	Cotton (T)	2	0.88	61							
(2005)	Spring			PBI	Forage	201	< 0.05	< 0.05	< 0.05	< 0.05	
	Wheat			108	Hay	273	< 0.05	< 0.05	< 0.05	< 0.05	
	Summit			d	Grain	319	< 0.05	< 0.05	< 0.05	< 0.05	
	(R1)				Straw	319	< 0.05	0.06	< 0.05	0.06	
Fresno USA	Cotton (T)	2	0.88	61							
(2005)	Spring			PBI	Forage	222	< 0.05	< 0.05	< 0.05	< 0.05	
	Wheat			117	Hay	285	< 0.05	< 0.05	< 0.05	< 0.05	
	Summit			d	Grain	319	< 0.05	< 0.05	< 0.05	< 0.05	
	(R1)				Straw	319	< 0.05	< 0.05	< 0.05	< 0.05	
Cheneyville	Cotton (T)	2	0.89	60							
USA (2005)	Spring			PBI	Forage	190	< 0.05	< 0.05	< 0.05	< 0.05	
	Wheat			117	Hay	239	< 0.05	< 0.05	< 0.05	< 0.05	
	Summit			d	Grain	288	< 0.05	< 0.05	< 0.05	< 0.05	
	(R1)				Straw	288	< 0.05	< 0.05	< 0.05	< 0.05	

PBI = plant back interval

No. = number of applications;

GS = growth stage at last application (BBCH);

DALT = days after last treatment.

(T) : denotes the treated / primary crop

(R1) : denotes the first rotational crop.

[a] Analytical method measures NAG together with glufosinate as a common derivative

FATES OF RESIDUES IN STORAGE AND PROCESSING

In processing

The major residue components present in glufosinate ammonium in treated crops are glufosinate ammonium and its metabolites MPP and NAG. The hydrolytic behaviour of each of these residue components was investigated in order to determine the effect of processing on the nature of residues in processed commodities (Weber 2008 MEF 08/540). The tests were performed under three sets of hydrolysis conditions representative of major food processing operations such as pasteurization, baking/boiling/brewing and sterilization.

The nature and characteristics of the test items used in the hydrolysis tests are described in Table 126. Citrate buffer solutions of pH 4, 5, and 6, respectively, were prepared using commercial citrate buffers and tap drinking water. The test item stock solutions were diluted with these buffer solutions to obtain ca. 1 mg/L buffer solutions of each test item (one buffer solution per pH-value and test item). Aliquots (5 mL) of the test item buffer solutions were filled in crimp cap glass vials and heated at temperatures and for time periods representative of the various food processing operations.

Table 126 Hydrolysis conditions representative of various food processing operations

Simulated process	pH	Nominal temperature	Test period [a]
Pasteurization	4 ± 0.1	90 ± 5 °C (water bath)	20 ± 1 min
Brewing, baking and boiling	5 ± 0.1	100 ± 5 °C (water bath)	60 ± 1 min
Sterilization	6 ± 0.1	120 ± 5 °C (autoclave)	20 ± 1 min

[a] The test periods listed in the table above do not include the heat-up time until reaching the test temperature, and the cooling time to ambient temperature after test termination.

The pH values measured in the test solutions before and after hydrolysis, as well as the radioactivity recoveries are summarised in Table 127. The pH-values were found to remain stable. The amount of ^{14}C recovered in the test solutions after hydrolysis ranged between 100.1% and 103.0% of applied ^{14}C for all test items and all hydrolysis conditions. These results demonstrate that no radioactivity was lost during the tests and no volatile degradation products dissipated from the test systems.

Table 127 Evolution of pH and recovery of radioactivity after hydrolysis under conditions representative of various food processing operations

Simulated process	Test items								
	[3,4-^{14}C] glufosinate			[2,3-^{14}C]-MPP			[3,4-^{14}C]-NAG		
	pH before	pH after	Recovery of ^{14}C	pH before	pH after	Recovery of ^{14}C	pH before	pH after	Recovery of ^{14}C
Pasteurization	3.98	3.96	103.0%	3.99	3.97	100.2%	3.99	3.98	100.5%
Brewing, baking and boiling	4.96	4.95	101.1%	4.96	4.96	101.1%	4.96	4.96	100.7%
Sterilization	5.98	5.95	100.5%	5.99	5.98	100.1%	5.98	5.97	100.1%

In order detect and identify possible degradation products formed during hydrolysis the test solutions of the [3,4-^{14}C] glufosinate hydrochloride salt and the [2,3-^{14}C] 3-methyl-phosphinico propionic acid were analysed by TLC before and after the test while the test solution of the [3,4-^{14}C] N-acetyl glufosinate di-sodium salt was analysed by HPLC. The sensitivity of the TLC and HPLC analyses for the detection of impurities was estimated at about 1% of the total radioactivity. The TLC plates and the HPLC chromatograms did not show the formation of any significant degradation product upon hydrolysis.

Glufosinate-derived residues in food commodities are unlikely to be affected by processing.

The effect of processing on the level of glufosinate-derived residues was also investigated for oranges, plums, grapes, olives, potatoes, sugar beet, soya bean, oilseed rape/ canola, cotton, sunflower, maize/ corn and rice.

As a measure for the transfer of residues into processed products, a processing factor was used, which is defined as

$$F = \frac{\text{Total residue in processed product (mg/kg)}}{\text{Total residue in raw agricultural commodity (mg/kg)}}$$

A concentration of residues takes place when PF > 1.

Oranges

Two trials were performed in Florida and California in order to determine the transfer of glufosinate-derived residues in orange and grapefruit processed commodities (Meikle 1991 A46850). The soil of an orange orchard was treated three times with an SL formulation of glufosinate ammonium at the exaggerated rate of about 8.6 kg ai/ha. The interval between two subsequent applications was about 3 months. Samples for orange processing were collected 14 days after the last treatment. The oranges (ca. 400 kg) were processed into juice, oil, molasses and dried orange peel. The samples of orange fruit and orange processed commodities were analysed for residues parent glufosinate ammonium and its metabolite MPP using the method HRAV-5A with quantification by GC/FPD. The limit of quantification for each glufosinate and MPP was 0.05 mg/kg in all the analysed matrices.

A second processing trial was performed in California during the 2008 growing season in order to determine the transfer of glufosinate-derived residues in orange processed commodities (Lenz 2009 RAGLP023). The soil of an orange orchard was treated three times at the exaggerated rate of 8.4 kg ai/ha. The interval between two subsequent applications was 14 days. A sample of oranges (180 kg) was taken 14 days after the last treatment and processed into peels, peeled fruit, pasteurized juice, orange dried pulp and oil. A batch of oranges was hand peeled to produce both peeled oranges and orange peels. The remaining oranges were washed for 5 minutes and the washed oranges scarified using a modified Hobart Abrasive Peeler to produce scarified fruit and an oil:water emulsion. The scarified fruit was weighed and retained for juice processing. The collected oil-water emulsion was screened (approximately 180 µm) to separate any flavedo (outermost layer of skin = rind) fragments from the oil-water emulsion. The scarified flavedo was set aside for later addition to the shredded peel. The first run oil-water emulsion was processed through the cream separator and centrifuge. The residual emulsion was then frozen, thawed, centrifuged and the oil collected. Juice was extracted from scarified oranges using a juice extractor to produce samples of juice and peel. The collected juice was transferred to the pulper finisher and screened using an approximately 1.2 mm screen to remove vesicular membranes, seeds, segment membranes and peel fragments. The collected rag and seeds were set aside for later addition to the shredded peel. The fresh juice was pasteurized by heating to 88–91 °C for 15 seconds and then rapidly cooling to 38 °C. The peel from the juice extraction was shredded and combined with the scarified flavedo and rag and seeds to generate wet peel.

The residues of parent glufosinate and its metabolites MPP and NAG were determined according to the method GL-001-P07-01 with quantification by LC-MS/MS.

Table 128 Residues of glufosinate in orange processing

	Application				Residue (mg/kg)				
Location, year (variety)	No (int)	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF	Ref
Orange									
Frostproof FL	3 (92	8.73	Fruit	14	< 0.05	0.54	0.59	–	A46850
USA (1990)	91)	8.49	Fruit, washed		< 0.05	0.72	0.77	–	
Hamlin		8.80	Juice		< 0.05	0.50	0.55	0.71	
			Dried peel / pulp		< 0.05	1.65	1.7	2.21	
			Molasses		< 0.05	1.99	2.04	2.65	
			Oil		< 0.05	< 0.05	< 0.05	< 0.13	

	Application				Residue (mg/kg)				
Location, year (variety)	No (int)	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF	Ref
Orange									
Elk Grove, CA	5	4.5 × 5	Fruit	17	< 0.05	< 0.05	< 0.05		A46850 [a]
USA (1985)			Juice		< 0.05	< 0.05	< 0.05		SAI≤ 1566
Washington			Wet peel		< 0.05	< 0.05	< 0.05		
Navel			Dried peel		< 0.05	< 0.05	< 0.05		
			Dried fines		< 0.05	< 0.05	< 0.05		
			Molasses		< 0.05	< 0.05	< 0.05		
			Oil		< 0.05	< 0.05	< 0.05		
Merritt Island	3	1.7	Juice	14	< 0.02	< 0.02			A46850
FL USA (1984)		1.1	Peel		< 0.02	< 0.02			SAI≤ 150
Parson Brown		1.1							
	3	3.4	Juice	14	< 0.02	< 0.02			
		2.2	Peel		< 0.02	< 0.02			
		1.1							
Arroyo Grande USA (2008)	3	8.4–8.6	Fruit	14	< 0.05	< 0.05	< 0.05	-	RAGLP023
Olinda Valencia			Oil		< 0.05	< 0.05	< 0.05	–	
Grapefruit									
Merritt Island	3	1.7	Fruit	14	< 0.02	< 0.02			A46850
FL USA (1984)		1.1	Juice		< 0.02	0.03			
Ruby Red		1.1							
	3	3.4	Fruit	14	< 0.02	< 0.02			
		2.2	Juice		< 0.02	0.03			
		1.1							
Palmetto FL	3	1.7	Juice	14	< 0.02	0.04			A46850
USA (1984)		1.1	Peel		< 0.02	0.02			
White Seedless		1.1							
	3	3.4	Juice	14	< 0.02	0.04			
		2.2	Peel		< 0.02	0.03			
		1.1							

[a] Samples from the Elk Grove, CA trial were stored for up to 1566 days (> 4 yrs) prior to analysis. Stability of residues on storage for this interval has not been demonstrated (note residues in fruit and processed commodities all < 0.05 mg/kg).

Plums

A laboratory scale processing trial was performed in California during the 2008 growing season in order to determine the transfer of glufosinate-derived residues from plums in dried plums (Murphy 2009 RAGLP025). The soil of a plum orchard was treated twice at the exaggerated rate of about 8.4 kg ai/ha. A sample of plums (37 kg) was processed into dried plums (prunes, 39.6% moisture). The plums were dry sorted, removing any fruit that was unsuitable for processing. Plums to be processed (37 kg) were placed in 52–57 °C water for 3–5 minutes to loosen extraneous material and then rinsed with water. Cleaned plums (5.44 kg) were placed in a single layer on racks in a dehydrator and dried at 60–74 °C until prunes were formed (1.33 kg, 39.6% moisture). Prune pits (0.4 kg) were removed manually.

The residues of parent glufosinate and its metabolites MPP and NAG were determined according to the method GL-001-P07-01 with quantification by LC-MS/MS.

Table 129 Residues of glufosinate in plum processing

	Application				Residue (mg/kg)				
country, year (variety)	No (int)	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	NAG	Total	PF
Live Oak USA	2 (28)	8.4	fruit	14	0.06	0.13	< 0.05	0.24	–
(2008) French		8.4	fruit, dried	14	< 0.05	0.33	< 0.05	0.43	1.79

Grapes

Two processing trials were performed to determine the fate of glufosinate-derived residues during processing of grapes into red wine (Helgers 1997a A59103). The grape samples for processing originated from two field residue trials that were performed in 1996 in the northern part of France (Loire Valley) with the grape varieties Groslot and Carbernet Franc. There were three applications to soil at the rate of 0.56 kg ai/ha and at the growth stages BBCH 55, BBCH 69 and BBCH 85. Mature bunches of grapes were collected for processing into red wine 13–14 days after the last application. Grapes were crushed and stemmed to create grape must to which potassium metabisulphite (0.06 g/L) and dried yeast (0.1 g/L) was added. Fermentation proceeded and sugar was added to ensure adequate alcohol content. A wine press was used to separate solids (wet pomace) from raw wine, and demijohns previously seeded with lactic-bacteria were filled with the raw wine. After the malolactic fermentation step was complete, potassium metabisulphite (0.1 g/L) was added and the wine clarified. Dry gelatine was added to improve clarification. The wine was filtered, additional potassium metabisulphite added together with 0.1 g/L tetratartaric acid and the wine bottled.

Analysis was conducted according to the method DFG 651 with quantification by GC/FPD.

However, there were no detectable residues in the grapes or processed commodities and so processing factors could not be derived.

Two processing trials were performed to determine the fate of glufosinate-derived residues during processing of grapes into white wine (Helgers 1997b A59104). The grape samples for processing originated from two field residue trials that were performed in 1996 in the southern part of France with the grape variety Semillon. The two trials were conducted at the same location but according to two different GAPs. The first GAP consisted of three applications of 0.56 kg as/ha of glufosinate ammonium at the growth stages BBCH 55, BBCH 75 and BBCH 85. The second GAP consisted of two applications of 1.12 kg ai/ha of glufosinate ammonium at the growth stages BBCH 75 and BBCH 85. Mature bunches of grapes were collected for processing into white wine 15 days after the last application. Grapes were pressed to produce juice and wet pomace (not used in white wine production). To the juice was added pectolytic enzymes (0.02 g/L), potassium metabisulphite (0.1 g/L) and dried yeast (0.1 g/L). Fermentation proceeded and if required sugar was added to ensure adequate alcohol content. After the alcoholic fermentation step was complete, potassium metabisulphite (0.1 g/L) was added and the wine left to naturally clarify. Dry gelatine was added to improve clarification and additional potassium metabisulphite added to inhibit oxidation. The wine was left in demijohns for 15 days, filtered, re-sulphited and bottled.

Analysis was conducted according to the method DFG 651 with quantification by GC/FPD.

Table 130 Residues of glufosinate in grape processing

	Application				Residue (mg/kg)				
Location, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF	Ref
Langon France (1996)	3	0.56	fruit	0 7 15	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05			A59104
Semillon			must		< 0.05	< 0.05			
			pomace		< 0.05	< 0.05			
			early wine		< 0.05	< 0.05			
			wine		< 0.05	< 0.05			
Langon France (1996)	2	1.12	fruit	0 7 15	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05			A59104
Semillon			must		< 0.05	< 0.05			
			pomace		< 0.05	0.07			
			early wine		< 0.05	< 0.05			
			wine		< 0.05	< 0.05			

An SC formulation containing 250 g/L of glufosinate ammonium and 50 g/L of oxyfluorfen was used.

Olives

A processing trial was performed in California during the 2008 growing season in order to determine the transfer of glufosinate-derived residues from olives in olive oil (Lenz 2009 RAGLP029). The soil of an olive orchard was treated three times with glufosinate ammonium at the exaggerated rate of 8.4 kg ai/ha with olives harvested 14 days after the last application. A sample of olives (18 kg) was processed in olive oil. The olives were cleaned by washing and the cleaned olives warmed in an oven for 19–21 minutes at 24–29 °C before grinding to a paste and malaxing (a process of mixing the paste that coalesces small into larger oil droplets) for 30 minutes. After malaxation, the paste was placed into a filter press to remove oil from the paste. Water that is also released was separated with a centrifuge and separator/funnel. After separation, olive oil was filtered and collected.

The residues of glufosinate and its metabolites MPP and NAG were determined according to the method GL-001-P07-01 with quantification by LC-MS/MS.

Table 131 Residues of glufosinate in olive processing (RAGLP029)

	Application				Residue (mg/kg)				
country, year (variety)	No (int)	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	NAG	Total	PF
Corning USA	3 (14)	8.4	fruit	14	< 0.05	0.13	< 0.05	0.23	–
(2008) Manzanillo			oil		< 0.05	< 0.05	< 0.05	< 0.15	< 0.65

Potatoes

A processing trial was performed in the US in order to determine the fate of glufosinate-derived residues during processing of potato tubers into potato chips and flakes (Deschamps 1997 A57765). Glufosinate ammonium was applied to the potato plants 9 days before harvest at the exaggerated rate of 2.22 kg ai/ha. After harvest, the tuber samples were processed into chips and flakes. About 89.5 kg of potato tubers were used for the two processes overall but taking into account the various steps, about 7.0 kg of potato tubers were required to produce 1.86 kg of potato chips while about 14.1 kg of potato tubers were used to produce 2 kg of flakes.

A 10 kg sample of washed potatoes was peeled using a restaurant style abrasive peeler for 30 seconds. The peeled potatoes were cut into thin 0.16 cm slices and the sliced potatoes were washed with warm water to remove free starch. The slices were drained to remove excess water and fried in oil at an average temperature of 179 °C for 90 seconds. The fried potato chips were drained and salted.

Washed potatoes were batch steam peeled for 45 seconds at 5.6–6.0 kg/cm^2. The potatoes were scrubbed for 30 seconds using an abrasive peeler. The potato peel was collected from the peeling and scrubbing process and the collected peel hydraulically pressed. The pressed peel was blended with the cut trim waste collected and a sample of the combined wet peel and trim was retained.

About 23 kg of peeled potatoes were cut into 1–1.3 cm and spray washed in cold tap water for 30 seconds to remove free starch. The potato slabs were precooked at an average of 74 °C for 20 minutes in a steam jacketed kettle. The precooked potato slabs were cooled to less than 32 °C for 20 minutes. About 13.6 kg of precooked potatoes were steam cooked at 99–100 °C for 40 minutes, mashed and then mixed for 60 seconds with an emulsion of pre-weighed food additives. The wet mash was dried using a drum drier, to a thin sheet. The thin sheet was broken into large flakes prior to completion of the drying process in a fluidized bed dryer. The flakes were passed through a hammer-mill for uniform milling of the finished potato flakes.

Analysis was conducted according to the method HRAV-5A with quantification by GC/FPD.

Table 132 Residues of glufosinate in potato processing

	Application				Residue (mg/kg)				
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	NAG	Total	PF
Ephrata USA (1996) Russet Burbank	1	2.24	tuber	9	0.64	< 0.05		0.69	–
			Chips		1.49	< 0.05		1.54	2.2
			Peel, wet (flakes)		0.36	< 0.05		0.41	0.59
			Flakes		1.96	< 0.05		2.01	2.91

Note: All the RAC and processed commodity samples showed residues of the metabolite MPP < 0.05 mg/kg. These residues were assumed to be equal to 0.05 mg/kg for the calculation of the total residues and for the derivation of the processing factors.

A study was conducted in Europe to determine the transfer of glufosinate-derived residues during industrial processing of potato tubers into potato flakes, potato crisps, and French fries (Pollmann 2004 C041674). There were four trials for each type of processing: one balance trial in which the residues were determined in the raw agricultural commodity, the end-product and various intermediate products and side-products; and three follow-up trials in which the residues were only determined in the raw agricultural commodity and in the final processed food commodity. The potato tubers for the processing trials originated from four supervised field trials performed in France in 2002 using the formulation Glufosinate ammonium was applied to the potato plants at the exaggerated rate of 1.5 kg ai/ha 5 days before harvest. The potatoes were processed using laboratory-scale equipment according to procedures simulating the commercial processes. The amount of potato tubers used for processing was about 15-17 kg for flakes, 4.0-4.4 kg for crisps, and 3.3-3.5 kg for French fries.

Potato flakes

The potatoes were washed and peeled for 4 to 5 min in a potato washing machine, and then cut in about 1 cm thick slices using a cutting machine. The potato pieces that were too small for the next steps of the process were discarded. The potato slices were washed with tap water to remove excessive starch using a rotary drum and band washer prior to blanching for 20 min at 75–76 °C. The blanched potato slices were cooled down and dried at room temperature for 15–25 min on a band drier and then cooked for 18–25 min at 105–115 °C in a steam cooking machine. The cooked slices were mashed and dried at 140–144 °C using a drum dryer. The dried potato plates were crushed by hand to get potato flakes.

Potato crisps

The potatoes were washed and peeled and the peeled potatoes were cut in 1–2 mm thick slices. The potato slices were washed with tap water to remove excessive starch and then dried in an oven at 30–32 °C for about 10 min before deep frying in vegetable oil for 3 min at 180 ± 5 °C. The crisps were cooled before sampling.

Potato fries (French fries)

The potatoes were washed and peeled and then cut into 10 × 10 mm strips. Small strips were not used for frying. The potato strips were blanched for 10 min at 68 to 74 °C and the blanched strips were washed with water to remove excessive starch and then dried in an oven at 28 to 32 °C for 10 min. After drying the potato strips were fried for 3 min at 140 ± 5 °C in a deep fryer using commercially available vegetable oil. After cooling all the French fries were deep frozen and kept frozen for 15–40 hours. Finally the frozen French fries were end-fried in an oven for 15 min at 220 ± 5 °C.

The raw agricultural commodity, the final processed foodstuffs, and the various intermediate and side-products were analysed for residues of glufosinate and its metabolite MPP according to the method 00915 with quantification by LC/MS/MS. No residues were detected in samples of water from washing or blanching the potatoes nor were any detected in oil used for frying.

Table 133 Residues of glufosinate in potato processing

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Chazay	1	1.50	tuber	5	0.13	< 0.05	0.18	–
D'Azergues			Preparation of flakes					
France S (2002)			Peel		< 0.05	< 0.05		
Bintje			Tuber, peeled		0.13	< 0.05	0.18	1
			Flakes		0.27	< 0.05	0.32	1.78
			Preparation of crisps					
			Peel		< 0.05	< 0.05		
			Tuber, washed peeled, sliced		0.08	< 0.05		
			Crisps		0.24	< 0.05	0.29	1.61
			Preparation of fries					
			Peel		< 0.05	< 0.05		
			Tuber, washed peeled, cut		0.08	< 0.05		
			Fried potatoes		0.09	< 0.05		
			French fries		0.11	< 0.05	0.16	0.89
Cestas France S	1	1.50	tuber		0.18	< 0.05	0.23	
(2002) Bintje			flakes		0.74	< 0.05	0.79	3.43
			crisps		0.34	< 0.05	0.39	1.70
			French fries		0.29	< 0.05	0.34	1.48
Davenescourt	1	1.50	tuber		0.12	< 0.05	0.17	
France N (2002)			flakes		0.47	< 0.05	0.52	3.06
Bintje			crisps		0.31	< 0.05	0.36	2.12
			French fries		0.15	< 0.05	0.20	1.18
Fresnoy Lès	1	1.50	tuber		0.51	< 0.05	0.56	
Roye France N			flakes		1.5	< 0.05	1.55	2.77
(2002) Bintje			crisps		0.85	< 0.05	0.90	1.61
			French fries		0.68	< 0.05	0.73	1.30

Note : All the RAC and processed commodity samples showed residues of the metabolite MPP < 0.05 mg/kg. These residues were assumed to be equal to 0.05 mg/kg for the calculation of the total residues and for the derivation of the processing factors.

A study was conducted in Europe to determine the transfer of glufosinate-derived residues during household processing of potato tubers into boiled potatoes, fried potatoes, and baked potatoes (Melrose 2006 RA-3079/05). Four trials were performed for each type of processing. In addition to the raw agricultural commodity and the final ready-to-eat commodity, the residues were also determined in various intermediate fractions and side-products so as to establish a balance of the glufosinate-derived residues. The potato tubers for the processing trials originated from four supervised field trials performed in Germany, the Netherlands and Belgium in 2005 where glufosinate ammonium was applied to the potato plants at the exaggerated rate of 1.5 kg ai/ha 7 days before harvest. The potatoes were processed according to standard household practice. The amount of potato tubers used for processing was about 7 kg for each type of processing. For each type of processed potato, tubers were first washed in lukewarm standing water (ratio potato/water = 1/2 (w/w)) and adhering soil was rubbed off by hand.

Potatoes, boiled (in their jackets)

After addition of sodium chloride (7.5 g/kg potato) and approximately 0.5 to 1 L of water/kg potato, washed potatoes were boiled (in their jackets) at approximately 100 °C for about 25 minutes. A sample of boiled tubers was taken for analysis and cut in small pieces. The remaining cooked tubers were peeled thinly with a kitchen knife. Finally, the cooked and peeled tubers were cut in small pieces.

Potato, fried

Washed tubers were peeled with a peeling knife and cut into thin slices (ca. 0.5 cm). The sliced potatoes were fried portion by portion at medium temperature in a pan with vegetable oil until they were tanned on both sides.

Potato, baked

Washed potatoes were wrapped into aluminium-foil and baked in the oven for 40 to 60 min at about 230 °C. Afterwards, the baked potatoes were removed from the foil. A sample of baked potatoes was taken for analysis and cut into small pieces. The remaining baked potato tubers were peeled thinly with a kitchen knife. Finally, the baked and peeled potatoes were cut into small pieces.

The raw agricultural commodity, the final processed foodstuffs, and the various intermediate and side-products were analysed for residues of glufosinate ammonium and its metabolite MPP according to the method 00915/M001 with quantification by LC/MS/MS.

Table 134 Residues of glufosinate in potato processing

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Monheim	1	1.50	preparation of boiled potatoes					
Germany (2005)			tuber	5	0.27	0.02	0.34	
Cilena			washed potatoes		0.44	0.02	0.47	
			washing water		< 0.01	< 0.01		
			cooked potatoes		0.46	0.01	0.47	0.99
			cooking water		0.02	< 0.01		
			peeled potatoes		0.45	0.01		
			potato peel		0.57	0.02		
			preparation of fried potatoes					
			tuber		0.29	0.02		
			washed potatoes		0.50	0.02		
			washing water		< 0.01	< 0.01		
			peeled potatoes		0.30	0.01		
			potato peel		0.34	0.02		
			fried potatoes		0.67	0.03	0.70	1.48
			preparation of baked potatoes					
			Tuber	5	0.39	0.02		
			washed potato		0.42	0.02		
			washing water		< 0.01	< 0.01		
			baked potatoes		0.59	0.02	0.61	1.29
			peeled potatoes		0.41	0.02	0.43	0.91
			potato peel		0.93	0.04		
Falkenhain	1	1.66	preparation of boiled potatoes					
Germany (2005)			Tuber	5	0.26	0.02	0.28	
Belana			washed potatoes		0.42	0.02	0.47	
			washing water		< 0.01	< 0.01		
			cooked potatoes		0.36	0.01	0.37	0.79
			cooking water		0.03	< 0.01		
			peeled potatoes		0.33	0.02		
			potato peel		0.75	0.04		
			preparation of fried potatoes					
			Tuber	5	0.17	0.01		
			washed potatoes		0.50	0.02		
			washing water		< 0.01	< 0.01		
			peeled potatoes		0.67	0.02		
			potato peel		0.40	0.03		
			fried potatoes		0.90	0.04	0.94	2.01
			preparation of baked potatoes					
			Tuber	5	0.37	0.02		
			washed potato		0.41	0.03		
			washing water		< 0.01	< 0.01		

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
			baked potatoes		0.69	0.03	0.72	1.54
			peeled potatoes		0.54	0.01	0.55	1.18
			potato peel		0.75	0.05		
Zwaagdijk-Oost	1	1.50	preparation of boiled potatoes					
Netherlands			Tuber	5	0.57	0.02	0.53	
(2005)			washed potatoes		0.70	0.03	0.87	
Frieslander			washing water		< 0.01	< 0.01		
			cooked potatoes		0.40	0.01	0.41	0.47
			cooking water		0.12	< 0.01		
			peeled potatoes		0.78	0.01		
			potato peel		0.50	0.02		
			preparation of fried potatoes					
			Tuber	5	0.36	0.01		
			washed potatoes		0.98	0.03		
			washing water		< 0.01	< 0.01		
			peeled potatoes		0.47	< 0.01		
			potato peel		0.65	0.03		
			fried potatoes		1.5	0.04	1.54	1.78
			preparation of baked potatoes					
			Tuber	5	0.62	0.02		
			washed potato		0.84	0.02		
			washing water		< 0.01	< 0.01		
			baked potatoes		0.88	0.03	0.91	1.05
			peeled potatoes		1.4	0.04	1.44	1.66
			potato peel		1.3	0.04		
Villers-Perwin	1	1.50	preparation of boiled potatoes					
Belgium (2005)			Tuber	5	0.63	0.04	0.68	
Bintje			washed potatoes		0.56	0.02	0.91	
			washing water		< 0.01	< 0.01		
			cooked potatoes		0.52	0.02	0.54	0.60
			cooking water		0.11	< 0.01		
			peeled potatoes		0.96	0.04		
			potato peel		0.65	0.04		
			preparation of fried potatoes					
			Tuber	5	0.67	0.04		
			washed potatoes		1.3	0.05		
			washing water		< 0.01	< 0.01		
			peeled potatoes		0.77	0.03		
			potato peel		0.61	0.05		
			fried potatoes		0.82	0.04	0.86	0.95
			preparation of baked potatoes					
			Tuber	5	0.63	0.03		
			washed potato		0.76	0.03		
			washing water		< 0.01	< 0.01		
			baked potatoes		1.1	0.04	1.14	1.26
			peeled potatoes		0.94	0.02	0.96	1.06
			potato peel		1.2	0.05		

Sugar beet

A study was conducted in Europe to determine the transfer of glufosinate-derived residues (glufosinate, MPP and NAG) during processing of glufosinate-tolerant sugar beet (Hees 1997 A57574). The samples for processing originated from three supervised field trials performed in Germany, France and the UK in 1996. In each field trial the glufosinate ammonium was applied twice to the sugar beet plants at the rate of 0.80 kg ai/ha. Samples of sugar beet root (81-102 kg) were taken at normal harvest and processed in raw sugar and by-products such as pressed pulp or molasses.

Beets (80–130 kg) were washed to remove adhering dirt and then sliced to give cossettes of 7 mm thickness. The cossettes were slowly transported from the bottom to the top of a heated trough (inclined at 5°) against the continuous flow of extraction liquid (water) in a speed which allowed the cossettes to remain in the trough for at least 58 minutes (extraction time). The denaturation temperature was between 72 and 76 °C. The resulting raw juice was purified by means of a two-step liming (pre-liming and main liming) and subsequent two-step carbonation while the pulp was pressed to produce pressed pulp and press water. In the pre-liming step the milk of lime was added in portions until a pH-value of 11 was reached. In the main liming step the milk of lime was added in one go. Subsequently CO_2 was introduced under stirring until again a pH-value of 11 was reached. The purified thin juice (clarified juice) was concentrated in a one-step evaporation unit to approximately a fifth of its original volume. The process was carried out at a temperature between 48 and 51 °C and under a vacuum of 0.1 bar. The concentrated juice (thick juice) was further concentrated under vacuum until the metastable super-saturation range was reached and, after the addition of white sugar (200 µm) as seed crystals, was allowed to cool. Subsequently the raw sugar was separated from the green syrup (equivalent to molasses) by means of a discontinuously working sieve-centrifuge. At the end of centrifugation the raw sugar was covered with water for purification purposes.

Sugar beet root, raw sugar, and various intermediate and side-products were analysed for residues according to the method AE-24A with quantification by GC/FPD. This method determines the residues of glufosinate and NAG as a sum (since they have the same GC-derivate) while MPP is determined separately.

Table 135 Residues of glufosinate in sugar beet processing (A57574)

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Tarnow	2	0.800	root	116	0.30	0.05	0.35	
Germany (1996)			pressed pulp		< 0.05	< 0.05	< 0.1	< 0.29
Transgenic			press water		0.06	< 0.05	0.11	0.31
hybrid			raw juice		0.25	< 0.05	0.30	0.86
			clarified juice		0.19	< 0.05	0.24	0.69
			sludge		0.09	< 0.05	0.14	0.40
			concentrated juice		0.72	0.14	0.24	2.46
			raw sugar		< 0.05	< 0.05	< 0.10	< 0.29
			molasses		1.5	0.23	1.73	4.94
Arthies France	2	0.800	root	124	0.06	< 0.05	0.11	
(1996)			pressed pulp		< 0.05	< 0.05	< 0.1	< 0.91
Transgenic			press water		< 0.05	< 0.05	< 0.1	< 0.91
hybrid			raw juice		0.11	< 0.05	0.16	1.45
			clarified juice		0.09	< 0.05	0.14	1.27
			sludge		0.05	< 0.05	0.10	0.91
			concentrated juice		0.55	0.05	0.60	5.45
			raw sugar		< 0.05	< 0.05	< 0.10	< 0.91
			molasses		0.69	0.06	0.75	6.82
Holbeach St	2	0.800	root	111	0.99	< 0.05	1.04	
Matthew United			pressed pulp		0.13	< 0.05	0.18	0.17
Kingdom (1996)			press water		0.18	< 0.05	0.23	0.22
Transgenic			raw juice		0.77	0.05	0.82	0.79
hybrid			clarified juice		0.82	0.05	0.87	0.84
			sludge		0.21	< 0.05	0.26	0.25
			concentrated juice		2.5	0.17	2.67	2.57
			raw sugar		< 0.05	< 0.05	< 0.10	< 0.10
			molasses		3.8	< 0.05	3.85	3.70

Note: All the RAC and processed commodity samples showed residues of the metabolite MPP < 0.05 mg/kg. These residues were assumed to be equal to 0.05 mg/kg for the calculation of the total residues and for the derivation of the processing factors.

A sugar beet processing study was conducted in the USA to investigate the transfer of glufosinate-derived residues from glufosinate-tolerant sugar beet in refined sugar (Brady 1997

A57723). The sugar beet root sample used in the study originated from a field trial performed in California in 1996. The crop received three spray applications at the exaggerated rate of 2.8-3.0 kg ai/ha. Sugar beet roots (ca. 189 kg) were collected at maturity, 136 days after the last application and processed in dried pulp, molasses and refined sugar.

The sugar beets were washed and cut into approximately 5 cm thick pieces before slicing into cossettes (pieces 1–3 mm thick and 3–8 cm long). Sugar was extracted from the cossettes in a series of steam heated kettles (referred to as cells), with a mixture of fresh water and pulp press water. The cells were heated to 65–80 °C (target temperature 70–75 °C). The cossettes and water was transferred counter-current to each other through the series of four vessels. Extracted beet pulp was pressed to recover sugar solution carried out with the pulp and the pressed pulp dried to an average of 1.7% moisture (drying temperature range 78–83 °C). The dried pulp was milled to produce dried beet pulp.

Raw juice from the diffuser was frozen prior to purification. The thawed raw juice was purified in a steam jacketed kettle by addition of lime and CO_2. Temperature was maintained at 80–86 °C and 82–88 °C. The precipitated impurities were coagulated by the addition of settling aid and allowed to settle and clarify. Clear juice was decanted and screened, if necessary, to remove suspended larger particles and the settled sludge was vacuum filtered. Filtrate was combined with the clear decanted liquid. The clarified liquid was further purified by a second carbonation with carbon dioxide gas. Carbonated liquor was vacuum filtered. The clarified juice of 11.4°–11.68° Brix was concentrated to 68.6° and 64.0° Brix in a vacuum evaporator. The concentrated juice was heated to 85 and 84 °C and filtered and frozen for later processing.

The filtered thick juice was removed from frozen storage and warmed to approximately 50 °C. The preheated filtered concentrated thick juice was fed to a vacuum pan and granulator. The massecuite was heated to 72 °C and centrifuged using a perforated bronze basket. Sugar retained in the basket was washed with clean hot water. The initial spin-off syrup (molasses) was also collected from the centrifuge. The washed sugar was stirred and dried under a flow of hot air.

Sugar beet root (raw agricultural commodity) and the processing fractions were analysed for residues using the method BK/04/95 with quantification by GC/FPD. This method determines the residues of glufosinate and NAG as a sum (since they have the same GC-derivate) while MPP is determined separately.

Table 136 Residues of glufosinate in sugar beet processing (A57723)

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Fresno USA	3	2.8-3.0	root	136	0.25	1.00	1.25	
(1996) Liberty			pulp, dry		0.14	0.60	0.74	0.59
Link			molasses		1.58	6.33	7.91	6.32
			refined sugar		< 0.05	< 0.05	< 0.1	< 0.08

Soya bean

A soya bean processing study was conducted in the USA to investigate the fate of glufosinate-derived residues during the preparation of soya bean oil from glufosinate-tolerant soya bean seed (Czarnecki 1995f ER-93-USA-02). The soya bean seed used in the study originated from a field trial performed in Iowa in 1993. The crop was treated at the exaggerated rate of ca. 2.6 kg ai/ha when the crop had reached the six trifoliate growth stage (V6). Mature soya bean seeds were collected at normal harvest, 95 days after application. The soya bean sample (31 kg) was processed into hulls, meal and oil. The whole soya beans are dried and then cleaned by aspiration and screening. The soya beans were mechanically cracked and aspirated to separate the hull and kernel fractions. A sample of hull material was retained. The kernels were heated to 66–74 (max 77 °C) °C, flaked between 0.2–0.3 mm rollers and expanded (extruded) into collets through a 0.95 cm die. A product temperature of 82–113 °C was achieved. After expansion, crude oil was extracted from the collets with hexane (3 × at 49–60 °C). The miscella (crude oil and hexane) was passed through a recovery unit to separate the crude oil and

hexane. The crude oil was heated to 73–90 °C for hexane removal. Residual hexane was removed from the spent collets using a flow of warm air. Sample of spent collets (meal if ground) were collected. The crude oil was refined according to AOCS Method Ca 9b-52 with addition of NaOH and samples of refined oil and soapstock retained.

The soya bean seed (raw agricultural commodity) and the processing fractions were analysed for residues of glufosinate ammonium using the method AE 24 with quantification by GC/FPD. This method allows separate determination of parent glufosinate and its metabolites MPP and NAG.

Table 137 Residues of glufosinate in soya bean processing

	Application				Residue (mg/kg)				
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	NAG	Total	PF
Webster City USA (1993)	1	2.62	seed	95	< 0.05	0.08	0.07	0.20	
Soya bean W98-7			hull		0.06	0.30	0.27	0.63	3.15
			meal		< 0.05	0.12	0.08	0.25	1.22
			oil, crude		< 0.05	< 0.05	< 0.05	< 0.15	< 0.74
			oil, refined		< 0.05	< 0.05	< 0.05	< 0.15	< 0.74

A further soya bean seed processing study was performed in order to determine the residues in aspirated grain fractions, since this fraction had not been analysed during the previous study (Brady 1995 A54283). The raw agricultural commodity used for this new study originated from two field trials conducted in Indiana and Missouri in 1994. The samples (32 kg and 35 kg in the Indiana and Missouri trials, respectively) were dried and the light impurities were aspirated to obtain the aspirated grain fraction The residues of glufosinate and its metabolites MPP and NAG were determined according to the method BK/05/95 with quantification by GC/FPD. This method determines the residues of glufosinate and NAG as a sum (since they have the same GC-derivate) while MPP is determined separately.

Table 138 Residues of glufosinate in soya bean processing (A54283)

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Noblesville USA (1994)	2	1.94–2.58	seed	77	3.44	1.07	4.51	
			aspirated grain fractions		31.4	8.68	40.1	8.89
Clarence USA (1994)	2	2.01–2.58	seed	77	1.44	1.84	3.28	
			aspirated grain fractions		4.24	4.73	8.97	2.73

Due to use-pattern changes a new soya bean processing study was conducted in the USA in 2008 to investigate the fate of glufosinate-derived residues during the preparation of soya bean oil from glufosinate-tolerant soya bean seed (Netzband 2009 RAGLP031). The soya bean seed used in the study originated from a field trial performed in Wisconsin. The crop received three spray applications at the rates of 3.0 kg ai/ha (pre-plant), 3.7 kg ai/ha (three unrolled trifoliates present, BBCH 13) and 2.2 kg ai/ha (beginning flowering BBCH 59-60). Mature soya bean seeds were collected at earliest commercial harvest, 91 days after application. The soya bean sample (27.9 kg) was processed into hulls, meal and oil. As moisture contents of field samples was greater than 13.5% the soya beans were dried in an oven at 54–71 °C to a final moisture content of 10–13.5%. Following drying, samples were cleaned by aspiration and screening. Cleaned whole soya beans were fed into a roller mill to crack the hull and liberate the kernel. After hulling, the material was passed through an aspirator to separate hull and kernel material. Moisture content of the kernel material was determined and adjusted up to 13.5%. Moisture adjusted kernel was allowed to temper for a minimum of twelve hours prior to further processing. Moisture adjusted kernel material was heated to 71–79 °C and flaked in using a flaking roll with a gap setting of 0.2–0.35 mm. Flakes were extruded and turned into collets by direct steam injection and compression (collets exited the processor at 93–121 °C). After

extrusion, the collets were dried in the oven at 66–82 °C for 30–40 minutes. Collets were extracted with 49–60 °C hexane. After 30 minutes, the miscella (crude oil and hexane) was drained and fresh hexane was added to repeat the cycle two more times. Solvent was removed from extracted collets by heating to 99–104 °C in a mixer to give soya bean meal. Miscella was passed through a laboratory vacuum evaporator to separate the crude oil and hexane. Crude oil was heated to 91–96 °C for hexane removal and filtered. Percent free fatty acid (FFA) was determined for the crude oil and based on this sodium hydroxide was added and the mixture heated at 20–24 °C and mixed for 90 minutes under rapid stirring and then for 20 minutes at low stirring and 62–67 °C. The neutralized oil was then centrifuged and the refined oil was decanted and filtered. Refined oil was heated to 40–50 °C and activated bleaching earth added (1.0% by weight of oil), and placed under vacuum. Temperature was increased to 85–100 °C and held for 10 to 15 minutes. After reducing the temperature, the bleached oil was filtered. Bleached oil was deodorised by heating on a steam bath for 28–32 minutes under vacuum with the temperature held between 220–230 °C. During the cooling period a 0.5% citric acid solution was added (1 mL per 100 grams of oil deodorized). Resulting fractions were deodorized oil (RBD oil) and deodorizer distillates. The residues of glufosinate, MPP and NAG were determined according to the method GL-001-P07-01 with quantification by LC-MS/MS.

Table 139 Residues of glufosinate in soya bean processing

	Application				Residue (mg/kg)				
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	NAG	Total	PF
Arkansaw USA	1	2.22–3.73	seed	91	0.12	2.87	0.88	3.87	
(2008) SG0678-LL			hull		2.06	32.52	9.40	43.98	11.4
			meal		< 0.05	< 0.05	< 0.05	< 0.15	< 0.04
			RBD oil [a]		< 0.05	< 0.05	< 0.05	< 0.15	< 0.04

[a] RDB oil: refined, bleached and deodorised oil.

Rape seed

Two rape seed processing trials were conducted in Germany in 1986 (Anon. 1988 DEU86H40641, Anon 1991 DEU86H40621). The product was applied to the rapeseed plants at the rate of 0.6 kg ai/ha for desiccation 14 days before harvest. The harvested seed was processed into crude oil and press cake. The reports did not include details on the processing procedure.

The rapeseed (raw agricultural commodity), the crude oil, and the press cake were analysed for residues of glufosinate ammonium using the method AL11/87 with quantification by GC/FPD.

Table 140 Residues of glufosinate in rape seed processing (DEU86H40621, DEU86H40641)

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Bornheim	1	0.6	seed	14	0.48	0.28	0.76	
Germany (1986)			press cake		0.80	0.14	0.94	1.24
Lindora			oil		< 0.05	< 0.05	< 0.1	< 0.13
Hattersheim	1	0.6	seed	14	0.25	< 0.2	0.45	
Germany (1986)			press cake		0.52	< 0.05	0.57	1.27
Jet Nuef			oil		< 0.05	< 0.05	< 0.1	< 0.22

Canola

A canola (oilseed rape) processing study was conducted in Canada to investigate the fate of glufosinate-derived residues during the preparation of canola oil from canola seed (Gadsby 1991 A46755, Kolodziejczyk 1991 A54754). Glufosinate ammonium was applied by aircraft at the rates of 0.40 kg ai/ha or 0.50 kg ai/ha for desiccation shortly before harvest. The canola seed samples from the

two trials were combined (ca. 1000 kg) and processed to meal, deodorised oil and a number of other intermediate fractions and by-products. The seeds were cleaned, the moisture of seeds was adjusted to 9% and the seed cracked by passing through flaking rolls adjusted to yield particles of 0.2 to 0.25 mm size. Once uniform, the flakes were fed to a cooker prepress. The top tray of the cooker was kept at 70–75 °C and the bottom tray at 90–95 °C. The oil was collected in refrigerated tanks under slight nitrogen purge. The procedure produced press oil and press cake. The press cake (14.7–17% oil content) was subsequently solvent extracted with hexane (50–55 °C) for 90 minutes to produce extracted oil and meal. Oil from the cooker/prepress and solvent extractions was composited (crude oil). Hydratable phospholipids (gums) were precipitated by addition to warm oil (60 °C) of 0.2–0.5% citric acid solution followed by 2% of water. Gums were separated from degummed oil by centrifugation. Free fatty acids were determined and a slight excess of NaOH added to warm oil to saponify the free fatty acids which were separated by centrifugation (soapstock). The oil was washed with water and the wash water removed by centrifugation. Bleaching clay (1% by weight of oil) was added to oil heated to 110 °C, with a contact time of 30 minutes, to produce bleached oil. Hydrogenation of processed oil was carried out using NYSOSEL HK 5 catalyst operating with a hydrogen pressure of 205–215 kPa and temperature 195–205 °C. Steam de-odourisation of oil was conducted at a temperature of 265 °C and reduced pressure of 3–6 mm Hg for 16 min to produce deodorized oil and distillates.

The canola seed (raw agricultural commodity) and the main products or by-products of processing were analysed for residues of glufosinate ammonium using the methods AL37/87 and AL40/87 with quantification by GC/FPD.

Table 141 Residues of glufosinate in oilseed rape seed processing

		Residue (mg/kg)			
country, year (variety)	Sample	glufosinate	MPP	Total	PF
Crossfield Canada (1990) Tobin	seed	1.50	0.45	1.95	
	cleaned seed	1.81	0.52	2.33	
	press oil	< 0.05	< 0.05	< 0.1	< 0.04
	press cake	3.24	0.83	4.07	1.75
	extracted oil	< 0.05	< 0.05	< 0.1	< 0.04
	meal	3.11	0.95	4.06	1.74
	crude oil	< 0.05	< 0.05	< 0.1	< 0.04
	soapstock	< 0.05	< 0.05	< 0.1	< 0.04
	bleached oil	< 0.05	< 0.05	< 0.1	< 0.04
	distillates	< 0.05	< 0.05	< 0.1	< 0.04
	deodorized oil	< 0.05	< 0.05	< 0.1	< 0.04

A canola processing study was conducted in the Canada to investigate the fate of glufosinate-derived residues during the preparation of canola oil from glufosinate-tolerant canola seed (MacDonald 1996 A56386). The canola seed used in the study originated from three field trials performed in Saskatchewan in 1993. Glufosinate ammonium was applied once at the growth stage BBCH 14-16. The three trials were conducted at three different rates: 0.75, 1.5 and 3.75 kg ai/ha. Canola seed was harvested 70 days after application. The seed samples (3.6-4.0 kg per trial) were separately processed into meal, oil and soapstock. The canola samples were dried at 61–71 °C to a moisture content of 7–10% and then cleaned by aspiration and screening. The cleaned seed was moisture-conditioned, flaked (0.38–0.51 mm), heat-conditioned (82–99 °C), and pressed in an expeller for the purpose of liberating a majority of the crude oil. The residual crude oil remaining in the solid material (press cake) exiting the expeller was later extracted with hexane (43–52 °C). Solvent from the extracted press cake (meal) was removed by forcing warm air through the mixture. Additionally some of the meal was toasted by injecting steam as the product was mixed, when the vapour temperature reaches 94–99 °C steam injection was stopped and the mixing and heating continued until a product temperature of 105–114 °C was reached which was maintained for a further 30 minutes. The crude oils recovered from the expeller and solvent extraction were combined and refined. Solvent was removed from the crude oils under vacuum at 75–90 °C. Crude oil was pre-treated with phosphoric acid (85% H_3PO_4, 0.01–0.05% by weight of crude oil) for 30 minutes at 40–45 °C with mixing. A weighed amount of 12 degree Baume NaOH was added to the crude oil, as

calculated on the basis of the amount of free fatty acids present. After NaOH addition the solution was mixed for 20 minutes at 250 RPM and 40–45 °C, and then for an additional 10 minutes at 70 RPM and 65–70 °C. The neutralized oil was allowed to settle for one hour at 60–65 °C. The oil solution was refrigerated overnight (minimum of 12 hours). Refined oil was decanted and filtered. The fraction settling to the bottom of the refrigerated container was soapstock.

The refined oil was further bleached and deodorized. Deodorized oil was prepared by heating under a vacuum to 235–250 °C for 40–60 minutes, cooling to 140–150 °C and adding citric acid solution (0.005%) to the oil at 10 mL/kg of oil. The oil was allowed to cool, under vacuum, to 105–115 °C. The vacuum was then broken and samples of deodorized oil and distillates collected. Bleached oil was produced by heating de-odorised oil to 40–50 °C prior to adding activated earth (2% by weight of oil). As the solution was mixed, a vacuum was applied, and the temperature was increased to 85–100 °C. The temperature was maintained, while continuously mixing, for 10 to 15 minutes. At the end of this period, the temperature was reduced to 58–68 °C, the vacuum broken and bleached oil recovered by filtering the mixture.

The canola seed (raw agricultural commodity) and the main products or by-products of processing were analysed for residues of glufosinate ammonium using the method AE-24 with quantification by GC/FPD. This method allows separate determination of glufosinate and each of the metabolites NAG and MPP.

Table 142 Residues of glufosinate in oilseed rape processing (A56386)

	Application				Residue (mg/kg)				
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	NAG	Total	PF
Indian Head	1	0.75	seed	70	< 0.05	< 0.05	0.06	0.16	
Canada (1993)			meal, untoasted		< 0.05	< 0.05	0.17	0.27	1.69
HCN92			meal, toasted		< 0.05	< 0.05	0.21	0.31	1.94
			oil, crude		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			oil, refined		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			oil, bleached		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			oil, deodorised		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			soapstock		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
Indian Head	1	1.50	seed	70	< 0.05	< 0.05	0.06	0.16	
Canada (1993)			meal, untoasted		< 0.05	< 0.05	0.22	0.32	2.00
HCN92			meal, toasted		< 0.05	0.05	0.29	0.39	2.44
			oil, crude		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			oil, refined		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			oil, bleached		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			oil, deodorised		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
			soapstock		< 0.05	< 0.05	< 0.05	< 0.15	< 0.94
Indian Head	1	3.75	seed	70	< 0.05	< 0.05	0.21	0.31	
Canada (1993)			meal, untoasted		< 0.05	0.11	0.60	0.76	2.45
HCN92			meal, toasted		< 0.05	0.10	0.64	0.79	2.55
			oil, crude		< 0.05	< 0.05	< 0.05	< 0.15	< 0.48
			oil, refined		< 0.05	< 0.05	< 0.05	< 0.15	< 0.48
			oil, bleached		< 0.05	< 0.05	< 0.05	< 0.15	< 0.48
			oil, deodorised		< 0.05	< 0.05	< 0.05	< 0.15	< 0.48
			soapstock		< 0.05	< 0.05	0.08	< 0.15	< 0.48

Note The residues of glufosinate, MPP and/or NAG < 0.05 mg/kg (LOQ) were assumed to be equal to 0.05 mg/kg for the calculation of the total residues and for the derivation of the processing factors.

Cotton

A cotton processing study was conducted in the USA to investigate the fate of glufosinate-derived residues during the preparation of cotton oil from glufosinate-tolerant cotton seed (Brady 1998 A67536). The cotton seed used in the study originated from a field trial performed in Arkansas in 1997. The crop was treated twice at the exaggerated rate of 2.6 kg ai/ha. The first treatment was

carried out at the 4-leaf stage and the second treatment at the beginning of bloom. The cotton was mechanically harvested at maturity, 76 days after the second application. The raw cotton seed (66 kg) was ginned to separate seeds (35 kg) and lint (17 kg) and the cotton seed was further processed into hulls, meal and oil.

The seed cotton was dried and burrs, sticks and other plant parts (gin trash) removed. After extraction, the lint cotton was saw ginned to remove the lint. After delinting, approximately 3% of the lint remains with the seed. A huller was used to mechanically crack the seed and screen the material to separate most of the hull material from the kernel which was then heated to 77–87 °C and held for 15 to 30 minutes at that temperature. After heating, the kernel material was flaked in a flaking roll with a gap setting of 0.2–0.3 mm. The flaked kernel material was extruded (0.95 cm die). As the material moved through the expander, steam was injected directly on the product so that the exiting temperature range of the resulting collets was 82–113 °C. Collets were dried at 54–71 °C for 30–40 minutes and then solvent extracted with 49–60 °C hexane. After 30 minutes, the hexane was drained and fresh hexane added to repeat the cycle two more times. The final two washes were for 15 minutes each. After the final draining, warm air was forced through the spent collets to remove residual hexane to give meal. The remaining miscella was adjusted to an approximate 60:40 ratio (crude oil to hexane) and the mixture was miscella refined. After refining the refined oil was passed through the evaporator to remove residual hexane.

The cotton seed (raw agricultural commodity) and the main products or by-products of processing were analysed for residues of glufosinate ammonium using the method BK/05/95 with quantification by GC/FPD. This method determines the residues of glufosinate and NAG as a sum (since they have the same GC-derivate) while MPP is determined separately.

Table 143 Residues of glufosinate in cotton seed processing (A67536)

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Crittenden USA (1997)	2	2.39–2.42	seed	76	4.24	0.86	5.10	
			meal		5.50	0.86	6.36	1.25
			hull		4.78	1.16	5.94	1.16
			oil, refined		< 0.05	< 0.05	< 0.1	< 0.02

Sunflowers

Three sunflower processing trials were conducted in the northern part of France in 1987 (Werner 1989abc A39984, A39986, A39987). Glufosinate ammonium was applied to the sunflower plants at the exaggerated rate of 1.0 kg ai/ha for desiccation between 2 and 5 weeks before harvest. The harvested seeds were processed into crude oil and soapstock. The reports do not include details on the processing procedure.

The sunflower seed (raw agricultural commodity), the crude oil, and the soapstock were analysed for residues of glufosinate using the method AL11/87 with quantification by GC/FPD.

Table 144 Residues of glufosinate in sunflower seed processing (A39984, A39986, A39987)

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Averdon France	1	1.0	seed	35	2.7	0.72	3.42	
(1987) Rodeo			soapstock		5.46	1.72	7.18	2.1
			oil, crude		0.06	< 0.05	< 0.11	< 0.03
Villeneuve St.	1	1.0	seed	12	0.69	0.51	1.20	
Martin France			soapstock		1.77	1.13	2.90	2.42
(1987) Frankasol			oil, crude		< 0.05	< 0.05	< 0.10	< 0.08
Reims France	1	1.0	seed	13	0.7	0.75	1.45	

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
(1987) Frankasol			soapstock		2.11	1.82	3.93	2.71
			oil, crude		< 0.05	< 0.05	< 0.10	< 0.07

Maize

A first maize processing study was conducted in the USA to investigate the fate of glufosinate residues during wet and dry milling of glufosinate-tolerant maize (Brady 1996 A54284). The maize grain used in the study originated from two field trials performed in 1993 in Iowa (Webster City) and Nebraska (York). In both trials glufosinate ammonium was applied at exaggerated application rates. In the Iowa trial treatment was performed at 1.5 kg ai/ha when the crop height was 61 cm. In the Nebraska trial, a first treatment was performed at 1.79 kg is/ha when the crop height was 30 cm, followed by a second treatment at 2.58 kg ai/ha when the crop height was 61 cm. Mature maize grain was collected at normal harvest, 100 and 96 days after the last application, respectively. The harvested maize grain samples were processed by dry and wet milling. The total quantity of grain used for milling (dry + wet) was approximately 51 kg and 139 kg in the Iowa and Nebraska trials, respectively.

Whole corn was dried in an oven at 54–71 °C to a moisture content of 10–15%. The light impurities were separated using an aspirator. After aspiration, the samples were screened to separate large and small foreign particles (screenings) from the corn. The light impurities from aspiration were classified as chaff and grain dust by sieving. The whole corn grain was moisture conditioned to 20–22% and allowed to "temper" for 2–2.5 hours. After tempering, the corn grain was impact milled and allowed to cool to approximately 32 °C after removal from the oven. The resulting cornstock was passed over a 3.2 mm shaker screen. Material above the screen was further processed into large grits, germ, and hull (bran). Material through the screen was separated into medium and small grits, coarse meal, meal, and flour.

The material above the 3.2 mm screen was passed through an aspirator to separate the hull material and hull material with attached germ from the large grits and germ. The hull material and hull material with attached germ was aspirated at a lower setting to separate the hull material from the hull material with attached germ. Hull material with attached germ was passed through a mill and aspirated to separate the hull from the germ. The hull material was combined. Large grits and germ from the first aspiration were separated on a gravity separator. The germs were combined and dried at 54–71 °C to 7–10% moisture.

The material passing through the 3.2 mm shaker screen was separated using a sample sifter (screen sizes: 2.0, 1.4 mm, 0.52, and 0.25 mm). Material on top of the 2.0 mm screen is medium grits; material on top of the 1.4 mm screen is small grits; material on top of the 0.52 mm screen is coarse meal; material on top of the 0.25 mm screen is meal; and material through the 0.25 mm screen is flour.

The germ was moisture conditioned to 12% and heated to 88–104 °C. The material was flaked in a flaking roll with a gap setting of 0.2 to 0.3 mm, and pressed in an expeller to liberate some of the crude oil. Resulting fractions were expelled crude oil and press cake with residual crude oil. The oil remaining in the press cake was extracted with hexane (49–60 °C). After 30 minutes, the hexane was drained and fresh hexane added to repeat the cycle two more times. After the final draining, warm air was forced through the extracted press cake to remove residual hexane. Miscella (crude oil and hexane) was passed through a recovery unit to separate the crude oil and hexane. Crude oil was heated to 73–90 °C for hexane removal. The crude oil recovered from the expeller and solvent extraction was combined, sampled, and refined according AOCS method Ca9a52. After refining, the refined oil and soapstock were separated.

The maize grain (raw agricultural commodity) and the main products, by-products and intermediate fractions of processing were analysed for residues of glufosinate ammonium using the

method AE-24 with quantification by GC/FPD. This method allows separate determination of glufosinate and each of the metabolites NAG and MPP.

Table 145 Residues of glufosinate in maize processing (A54284)

	Application				Residue (mg/kg)				
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	NAG	Total	PF
Webster City	1	1.57	grain	100	< 0.05	< 0.05	< 0.05		
USA			wet milling						
			Starch		< 0.05	< 0.05	< 0.05		
			Hulls		< 0.05	< 0.05	< 0.05		
			oil, refined		< 0.05	< 0.05	< 0.05		
			oil, crude		< 0.05	< 0.05	< 0.05		
			dry milling						
			flour		< 0.05	< 0.05	< 0.05		
			hulls		< 0.05	< 0.05	< 0.05		
			Grits		< 0.05	< 0.05	< 0.05		
			meal		< 0.05	< 0.05	< 0.05		
			oil, crude		< 0.05	< 0.05	< 0.05		
			oil, refined		< 0.05	< 0.05	< 0.05		
York USA (1993)	2	1.80-2.58	Grain	96	< 0.05	< 0.05	0.05		
(LH59 × LH51)			wet milling						
(LH119) (4) ×			Starch		< 0.05	< 0.05	< 0.05		
(T14)			Hulls		< 0.05	< 0.05	< 0.05		
			oil, refined		< 0.05	< 0.05	< 0.05		
			oil, crude		< 0.05	< 0.05	< 0.05		
			dry milling						
			flour		< 0.05	< 0.05	0.05		
			hulls		< 0.05	< 0.05	0.10		
			Grits		< 0.05	< 0.05	0.05		
			meal		< 0.05	< 0.05	0.05		
			oil, crude		< 0.05	< 0.05	< 0.05		
			oil, refined		< 0.05	< 0.05	< 0.05		

Note : The residues of glufosinate, MPP and/or NAG < 0.05 mg/kg (LOQ) were assumed to be equal to 0.05 mg/kg for the calculation of the total residues and for the derivation of the processing factors.

A further maize grain processing study was performed in order to determine the residues in aspirated grain fractions as this fraction had not been analysed during the previous study (Brady 1995 A53690). The glufosinate-tolerant maize grain used for this new study originated from a field trial conducted in Missouri in 1994 using an exaggerated rate. After harvest, the maize grain sample (116 kg) was sent to a processing facility where it was dried. The light impurities were aspirated to obtain the aspirated grain fraction.

Analysis was performed according to the method BK/05/95 with quantification by GC/FPD. This method determines the residues of glufosinate and NAG as a sum (since they have the same GC-derivate) while MPP is determined separately. A first analysis was performed in 1995. In 1996 the aspirated grain fraction was further separated in fractions of different particle size (< 425 µm, 425-850 µm, 850-1180 µm, and 1180-2030 µm), each fraction was analysed separately and the residues in aspirated grain were calculated based on a standard distribution of particle size: 50% of particles < 425 µm, 25% of particles in the range 425-850 µm, 15% of particles in the range 850-1180 µm, and 10% of particles in the range 1180-2030 µm.

Table 146 Residues of glufosinate in maize processing (A53690)

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Clarence, Shelby	2	1.22–1.55	grain	89	0.26	0.06	0.32	

USA (1994) [a]			aspirated grain fractions	89	3.11	0.75	3.86	12.06
Clarence, Shelby	2	2.01–2.62	grain	89	0.34	0.06	0.40	
USA (1994) [b]			aspirated grain fractions	89	2.96	0.62	3.58	8.85

[a] Initial analyses (1995)

[b] Re-analyses (1996).

Rice

A processing study was conducted in the USA to investigate the fate of glufosinate-derived residues during milling of glufosinate-tolerant rice (Brady 1998 C000898). The rice sample used in the study originated from a field trial performed in Mississippi in 1997 The crop received two spray applications at the exaggerated rate of ca. 1.53 kg ai/ha. The treatments were conducted at the growth stages BBCH 12-14 and BBCH 22-23. Rice grain (*ca*. 66 kg) was collected at normal harvest, 103 days after the last application, and milled to produce hulls, bran and polished rice. The rice samples (RAC) were dried in an oven at between 43–60 °C to a final moisture content of 11–14%. The light impurities were separated using an aspirator, after which the samples were screened to separate large and small foreign particles from the rice.

The whole rice was passed through a rice dehuller to remove and separate the hull from the brown rice. Hull accounts for 18–24% of the starting rice. During de-hulling, the goal is to produce brown rice with a trace or no annulled rice.

The brown rice was decorticated in an abrasion mill. After decorticating, the sample was classified with a sifter equipped with a 14 TMS screen. Material on top of the screen is white milled rice while material passing through the screen is bran. Decorticating was repeated until the total amount of bran was 11–17% of the starting brown rice weight.

Rice grain (raw agricultural commodity) and the milling fractions were analysed for residues using the method BK/04/95 with quantification by GC/FPD. This method determines the residues of glufosinate and NAG as a sum (since they have the same GC-derivate) while MPP is determined separately.

Table 147 Residues of glufosinate in rice processing

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ae/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
Greenville USA (1997)	2	1.52-1.54	grain	103	< 0.05	0.15	0.20	
Transgenic			grain, polished		< 0.05	0.06	0.11	0.55
			hull		< 0.05	0.32	0.37	1.85
			bran		< 0.05	0.12	0.17	0.87

Note: The residues of glufosinate & NAG and/or MPP < 0.05 mg/kg (LOQ) were assumed to be equal to 0.05 mg/kg for the calculation of the total residues and for the derivation of the processing factors.

A second processing study with a more critical use-pattern was conducted in the USA to investigate the fate of glufosinate-derived residues during milling of glufosinate-tolerant rice (Brady 2000 B002989). The rice sample used in the study originated from a field trial performed in Texas in 1999. The crop received two spray applications at the exaggerated nominal rate of 2.5 kg ai/ha. The treatments were conducted at the growth stages BBCH 12-14 and BBCH 23-24. Rice grain (ca. 66 kg) was collected at normal harvest, 78 days after the last application, and milled to produce hulls, bran and polished rice. The rough rice samples were dried in an oven at 43–60 °C to a final moisture content of 13.1%. The light impurities were separated by aspiration after which the sample was screened to separate large and small foreign particles (screening) from the rice. The whole rice was passed through a rice huller to remove and separate the hull from the brown rice. Hull accounted for approximately 18% of the starting rough rice.

The brown rice was decorticated in an abrasion mill. After decortication, the bran was separated from the polished rice with a sample sifter equipped with a 14 TMS screen. Material on top of the screen was polished rice while material passing through the screen was bran. Decortication removed the bran which accounted for 11–17% of the starting brown rice weight.

Rice grain (raw agricultural commodity) and the milling fractions were analysed for residues using the method BK/01/99 with quantification by GC/FPD. This method determines the residues of glufosinate and NAG as a sum (since they have the same GC-derivate) while MPP is determined separately.

Table 148 Residues of glufosinate in rice processing

	Application				Residue (mg/kg)			
country, year (variety)	No	kg ai/ha	Sample	PHI (d)	glufosinate	MPP	Total	PF
East Bernard USA (1999)	2	2.50–2.51	grain	78	0.42	0.30	0.72	
Bengal 62			grain, polished		0.50	0.18	0.68	0.94
			hull		0.67	0.98	1.65	2.29
			bran		0.32	0.21	0.53	0.74

Note: Due to the low recoveries at the 0.05 mg/kg fortification level the results for MPP in polished grain are not fully supported by validation data. They were not corrected for the recoveries obtained at the 0.05 mg/kg fortification level.

Table 149 Overview of processing factors for glufosinate-derived residues

Raw commodity	Processed commodity	Individual processing factors	Best estimate processing factor
Orange	Juice	0.71	0.7
	Dried peel / pulp	2.21	2.2
	Molasses	2.65	2.7
	Oil	< 0.13	< 0.13
Plum	Dried fruit	1.79	1.8
Grape	Wine	ND ND ND ND	–
Olive	Oil	< 0.65	< 0.65
Potato	Chips	2.2	2.2
	Flakes	1.78 2.77 2.91 3.43 3.06	2.9
	Crisps	1.61 1.61 1.70 2.12	1.7
	French fries	0.89 1.18 1.30 1.48	1.2
	Boiled potatoes	0.47 0.60 0.79 0.99	0.7
	Fried potatoes	0.95 1.48 1.78 2.01	1.6
	Baked potatoes	1.05 1.26 1.29 1.54	1.3
Sugar beet	Pressed pulp	0.17 < 0.29 < 0.91	< 0.29
	Dried pulp	0.59	0.6
	Molasses	3.70 4.94 6.32 6.82	5.6
	Raw or refined sugar	< 0.08 < 0.10 < 0.29 < 0.91	< 0.2
Soya bean	Aspirated grain fraction	2.73 8.89	5.8
	Hulls	3.15, 11.4	7.3
	Meal	1.22	1.2
	Oil	< 0.04 < 0.74	< 0.74
Rape/canola	Press cake	1.24, 1.27, 1.75	1.27
	Meal	1.74, 1.94, 2.44, 2.55	2.2
	Oil	< 0.13 < 0.22 < 0.48 < 0.94 < 0.94	< 0.48
Cottonseed	Hulls	1.16	1.2
	Meal	1.25	1.2
	Oil	< 0.02	< 0.02
Sunflower seed	Oil	< 0.03 < 0.07 < 0.08	< 0.07
Maize	Starch (wet milling)	ND ND	–
	Hulls (wet milling)	ND ND	–
	Oil (wet milling)	ND ND	–
	Hulls (dry milling)	ND ND	–

Raw commodity	Processed commodity	Individual processing factors	Best estimate processing factor
	Flour (dry milling)	ND ND	–
	Meal (dry milling)	ND ND	–
	Oil (dry milling)	ND ND	–
	Aspirated grain fraction	8.85 12.06	10.5
Rice grain	Hull	1.85 2.29	2.1
	Bran	0.74 0.87	0.8
	Polished grain	0.60 0.94	0.8

ND: not determined. Denotes processing trials in which no processing factor could be derived because the residues were found to be < LOQ in both the raw agricultural commodity and the processed fractions.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Two sets of livestock feeding studies were conducted. In the first set, the dairy cows and laying hens were dosed with a mixture of glufosinate ammonium and MPP. These residue components are representative of the glufosinate ammonium uses for non-selective weed control and desiccation in non-genetically modified conventional crops. While the residues of MPP in treated crops are usually low, the residues of parent glufosinate ammonium may be present at higher levels (following desiccation uses). Therefore, the mixture administered to the animals consisted mainly of glufosinate ammonium with a lower amount of MPP. In the second set of studies the dairy cows and laying hens were dosed with a mixture of parent glufosinate ammonium and NAG. NAG is specific to genetically modified glufosinate-tolerant crops and represents the major portion of the residue in these crops.

Lactating dairy cows

A dairy cow feeding study was conducted with lactating Holstein Friesian cows (454-723 kg body weight) in which cows were dosed for 28 days using a maize meal pre-mix that was fortified with the appropriate amount of glufosinate ammonium and MPP to give feed levels of 4, 12 and 40 ppm glufosinate equivalents (Krebs 1989 A40885). The daily feed consisted of 9.1 kg/cow/d maize silage, 2.6 hay, 1.8 sugar beet chip and 1.8 kg/cow/day supplementary feed. Cows producing more than 13 L milk/day received an additional 0.5 kg concentrated feed stuff per litre of milk. Individual cow milk production ranged from 8.4 to 28 L/day/cow. Milk was collected daily up to day 6 and then on days 9, 13, 16, 20, 23 and 27 (retained separately for each animal). After 28 days, three cows from each group were sacrificed. Remaining animals from the middle and high dose groups were kept on a control diet for a further week to monitor the change in tissue residue levels after that time. Samples of blood, muscle, diaphragm muscle, fat (including perirenal, omental and mediastinal deposits), liver and kidney were taken from each animal.

Table 150 Residues of glufosinate and MPP in milk and tissues of cows dosed for 28 days with a mixture of glufosinate and MPP

Commodity	Residue (mg/kg glufosinate acid equivalents)					
	3 ppm glufosinate + 1 ppm MPP		9 ppm glufosinate + 3 ppm MPP		30 ppm glufosinate + 10 ppm MPP	
	glufosinate	MPP	glufosinate	MPP	glufosinate	MPP
Milk [c]	< 0.02	< 0.02	< 0.02 [a]	< 0.02	< 0.02 [b]	< 0.02
Muscle	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05
Diaphragm muscle	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10
Blood	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05

Commodity	Residue (mg/kg glufosinate acid equivalents)					
	3 ppm glufosinate + 1 ppm MPP		9 ppm glufosinate + 3 ppm MPP		30 ppm glufosinate + 10 ppm MPP	
	glufosinate	MPP	glufosinate	MPP	glufosinate	MPP
Liver	0.13 < 0.10 < 0.10	1.5 1.3 0.62	< 0.10 < 0.10 < 0.10	3.9 3.9 4.2	< 0.10 0.10 < 0.10	10.7 9.0 7.1
Kidney	< 0.10 < 0.10 < 0.10	0.34 0.41 0.38	< 0.10 < 0.10 < 0.10	0.77 2.0 1.5	< 0.10 < 0.10 0.13	4.8 3.8 7.4
Fat	< 0.05 0.06 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	0.06 < 0.05 0.08	< 0.05 < 0.05 < 0.05	0.16 0.07 0.08

The nominal 1 × dose level was set at 3 ppm glufosinate + 1 ppm of MPP in feed (dry matter). Based on an average body weight of ca. 600 kg and a daily feed intake of 15 kg (dry matter), this is equivalent to 0.075 mg/kg bw/d of glufosinate and 0.025 mg/kg bw/d of MPP.

[a] For one cow, residues of 0.03 mg/kg were determined on day 0 and day 1.

[b] For one cow, residues of 0.02 mg/kg were determined on day 9. An apparent residue level of 0.03 mg/kg of glufosinate was also found in one control milk sample.

[c] For milk, the levels reported in the table are the maximum levels observed throughout the 28 day dosing period. Exceptions are indicated in the following annotations since they were not dose-related.

Table 151 Residues of glufosinate and MPP in milk and tissues of cows dosed for 28 days with a mixture of glufosinate ammonium and MPP and then kept on a control diet for 7 days

Commodity	Residue (mg/kg)			
	9 ppm glufosinate + 3 ppm MPP		30 ppm glufosinate + 10 ppm MPP	
	glufosinate	MPP	glufosinate	MPP
Milk [a]	< 0.02	< 0.02	< 0.02	< 0.02
Muscle	< 0.05	< 0.05	< 0.05	< 0.05
Diaphragm muscle	< 0.10	< 0.10	< 0.10	< 0.10
Blood	< 0.05	< 0.05	< 0.05	< 0.05
Liver	< 0.10	0.51	< 0.10	0.86
Kidney	< 0.10	0.24	< 0.10	0.21
Fat	< 0.05	< 0.05	< 0.05	< 0.05

[a] For milk, the levels reported in the table are the maximum levels observed during the 7 days control diet period.

An additional dairy cow feeding study was conducted in which Holstein dairy cows (426–595 kg bw) were dosed once daily (after morning milking) for 28 days using gelatine capsules that contained the appropriate amount of glufosinate and NAG (Czarnecki 1996c A54503). The three treated groups received doses of glufosinate ammonium and NAG equivalent to a residue in the feed of 9.1 ppm (1×), 27.3 ppm (3×), and 91.1 ppm (10×) of glufosinate free acid. Average feed consumption during dosing was 21.7 kg/day while average milk production was 17.8, 14.9 and 14.0 L/d for the low, middle and high dose groups respectively. Milk was collected daily and after 28 days all animals were sacrificed within 24 hours of the final dose. Samples of muscle (meat - from the hind quarter), liver, fat (perinephric), and kidney were taken from each cow.

Table 152 Residues of glufosinate/NAG and MPP in milk and tissues of cows dosed for 28 days with a mixture of glufosinate and NAG

Commodity	Residue (mg/kg)					
	1.4 ppm glufosinate + 7.7 ppm NAG		4.2 ppm glufosinate + 23.1 ppm NAG		14 ppm glufosinate + 77 ppm NAG	
	Glufosinate/NAG	MPP	Glufosinate/NAG	MPP	Glufosinate/NAG	MPP
Milk [a]	< 0.02	< 0.02	0.03	< 0.02	0.07 [a]	< 0.02 [a]
Muscle	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05
Liver	< 0.10 < 0.10	< 0.10 < 0.10	< 0.10 < 0.10	< 0.10 < 0.10	< 0.10 < 0.10	0.28 0.29

Commodity	Residue (mg/kg)					
	1.4 ppm glufosinate + 7.7 ppm NAG		4.2 ppm glufosinate + 23.1 ppm NAG		14 ppm glufosinate + 77 ppm NAG	
	Glufosinate/NAG	MPP	Glufosinate/NAG	MPP	Glufosinate/NAG	MPP
	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	0.25
Kidney	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10	< 0.10 < 0.10 < 0.10	0.11 0.14 0.15	< 0.10 < 0.10 0.13
Fat	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05	< 0.05 < 0.05 < 0.05

The nominal 1 × dose level was set at 1.4 ppm glufosinate ammonium + 7.7 ppm of NAG in feed (dry matter). Based on an average body weight of ca. 500 kg and a daily feed intake of ca. 21 kg (dry matter), this is equivalent to 0.059 mg/kg bw/d of glufosinate and 0.325 mg/kg bw/d of NAG.

[a] For milk, the levels reported in the table are the maximum levels observed throughout the 28 day dosing period. Exceptions are on two consecutive sampling dates the milk of one cow was found to contain 0.23 mg/kg and 0.14 mg/kg of glufosinate residues. The same milk samples contained 0.03 mg/kg of MPP residues. These residues were associated with reduced feed consumption (ca. 50%) together with reduced milk production (ca. 50%), during this isolated period

Laying hens

A hen feeding study was conducted in which laying Shaver Starcross hens (ca. 33-week old) were dosed for 28 days using a maize meal pre-mix that was fortified with the appropriate amount of glufosinate ammonium and MPP to give feed levels of 4.5, 13.5 and 45 ppm glufosinate equivalents (Sochor 1989a A40697). All hens were fed once per day. Eggs were collected daily and pooled per dose group. After 28 days, 10 hens from each group were sacrificed with the remaining five hens per dose group were kept on a control diet for a further week to monitor the change in tissue residue levels after that time. Samples of blood, muscle (from the breast), fat (from the abdominal cavity), liver and kidney were taken from each bird and pooled per dose group.

The egg samples were analysed within 4 days of sampling while the tissue samples were stored for a maximum of 61 days before analysis. Due to the short storage period of egg samples the storage stability of residues in eggs was not investigated. The storage stability of glufosinate and MPP residues in tissues was investigated using control tissue samples, which were fortified with glufosinate and MPP about two weeks after sampling, stored under the same conditions as the treated samples and analysed alongside the treated samples. Although the treated samples were stored for a longer period than the fortified samples (about 14 days longer), it may be reasonably concluded that the residues of glufosinate and MPP in the treated samples remained stable during storage.

Average feed consumption was 114, 116 and 120 g/day for the 1×, 3× and 10× groups respectively while average egg weights were 63.4, 59.2 and 62.7 g respectively for the same groups. Lay efficiency was 77, 89 and 87% for the three dose groups. Within the 1 × dose group one hen laid only one egg and one hen did not lay any eggs over the entire trial period. After slaughtering the samples from these two hens were pooled, but were kept separate from those of the remaining eight hens and were analysed separately.

Table 153 Residues of glufosinate and MPP in eggs and tissues of hens dosed for 28 days with a mixture of glufosinate ammonium and MPP

Commodity	Residue (mg/kg)					
	3.5 ppm glufosinate + 1 ppm MPP		10.5 ppm glufosinate + 3 ppm MPP		35 ppm glufosinate + 10 ppm MPP	
	glufosinate	MPP	glufosinate	MPP	glufosinate	MPP
Eggs [a]	< 0.05	< 0.05	< 0.05	< 0.05	0.07 [b]	< 0.05
Muscle	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Blood	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Liver	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10
Kidney	< 0.05	0.69	0.07	2.0	0.23	7.8
Fat	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05

The nominal 1 × dose level was set at 3.5 ppm glufosinate ammonium + 1 ppm of MPP in feed (dry matter). Based on an average body weight of ca. 1.7 kg and a daily feed intake of 120 g (dry matter), this is equivalent to 0.25 mg/kg bw/d of glufosinate and 0.07 mg/kg bw/d of MPP.

[a] For eggs, the levels reported in the table are the maximum levels observed throughout the 28 day dosing period. For tissues only one residue level is available per dose group since the samples were pooled per dose group.

[b] The maximum of 0.07 mg/kg was reached between day 7 and day 13. Theafter the residues of glufosinate in eggs decreased again to levels close to or below the LOQ of 0.05 mg/kg

Table 154 Residues of glufosinate and MPP in eggs and tissues of hens dosed for 28 days with a mixture of glufosinate ammonium and MPP and then kept on a control diet for 7 days

Commodity	Residue (mg/kg)					
	3.5 ppm glufosinate+1 ppm MPP		10.5 ppm glufosinate+3 ppm MPP		35 ppm glufosinate+10 ppm MPP	
	glufosinate	MPP	glufosinate	MPP	glufosinate	MPP
Eggs [a]	< 0.05	< 0.05	< 0.05	< 0.05	0.05	< 0.05
Muscle	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Blood	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Liver	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10	< 0.10
Kidney	< 0.05	0.05	< 0.05	0.19	< 0.05	0.66
Fat	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05

The nominal 1 × dose level was set at 3.5 ppm glufosinate ammonium + 1 ppm of MPP in feed (dry matter). Based on an average body weight of ca. 1.7 kg and a daily feed intake of 120 g (dry matter), this is equivalent to 0.25 mg/kg bw/d of glufosinate and 0.07 mg/kg bw/d of MPP.

[a]For eggs, the levels reported in the table are the maximum levels observed during the 7days control diet period.

An additional poultry feeding study was conducted in which White leghorn laying hens (ca. 30-week old, 1.43–1.51 kg bw) were dosed for 28 days using gelatine capsules that contained the appropriate amount of glufosinate and NAG (Czarnecki 1997 A54485). The doses were equivalent to feed levels of 0.44, 1.31and 4.4 ppm glufosinate equivalents. Eggs were collected daily and pooled per subgroup. After 28 days, 12 hens from each group were sacrificed and the remaining eight hens were kept on a control diet for either one or two further weeks to monitor the change in tissue residue levels after that time. Samples of muscle (from the breast and thigh), liver, fat (subcutaneous plus abdominal), and skin were taken from each bird and pooled per subgroup. These storage intervals are covered by storage stability data generated during the study, which demonstrated that the residues of glufosinate, NAG and MPP remain stable for up to 15 months in frozen poultry eggs, muscle and liver.

Average feed consumption was 146, 145 and 146 g/day for the 1×, 3× and 10× groups respectively while lay efficiency was 90, 94 and 91% for the three dose groups.

Table 155 Residues of glufosinate/NAG and MPP in eggs and tissues of hens dosed for 28 days with a mixture of glufosinate ammonium and NAG

Commodity	Measured residue level (mg/kg)[a]					
	0.07 ppm glufosinate + 0.37 ppm NAG		0.21 ppm glufosinate + 1.1 ppm NAG		0.7 ppm glufosinate + 3.7 ppm NAG	
	glufosinate/NAG	MPP	glufosinate/NAG	MPP	glufosinate/NAG	MPP
Eggs	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Muscle	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 < 0.05 < 0.05[b] < 0.05[c]	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]
Liver	< 0.10 < 0.10 < 0.10 < 0.10 [b] < 0.10 [c]	< 0.10 < 0.10 < 0.10 < 0.10[b] < 0.10[c]	< 0.10 < 0.10 < 0.10 < 0.10 [b] < 0.10 [c]	< 0.10 < 0.10 < 0.10 < 0.10 [b] < 0.10 [c]	< 0.10 < 0.10 < 0.10 < 0.10 [b] < 0.10 [c]	< 0.10 < 0.10 < 0.10 < 0.10 [b] < 0.10 [c]
Skin	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05	< 0.05 < 0.05

Commodity	Measured residue level (mg/kg)[a]					
	0.07 ppm glufosinate + 0.37 ppm NAG		0.21 ppm glufosinate + 1.1 ppm NAG		0.7 ppm glufosinate + 3.7 ppm NAG	
	glufosinate/NAG	MPP	glufosinate/NAG	MPP	glufosinate/NAG	MPP
	< 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05[b] < 0.05[c]	< 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 [b] < 0.05[c]	< 0.05 < 0.05 [b] < 0.05 [c]
Fat	< 0.05 < 0.05 < 0.05 < 0.05[b] < 0.05[c]	< 0.05 < 0.05 < 0.05 < 0.05[b] < 0.05[c]	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 < 0.05 < 0.05[b] < 0.05[c]	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]	< 0.05 < 0.05 < 0.05 < 0.05 [b] < 0.05 [c]

The actual 1 × dose level was about 0.07 ppm glufosinate ammonium + 0.37 ppm of NAG in feed (dry matter). Based on an average body weight of ca. 1.45 kg and a daily feed intake of 150 g (dry matter), this is equivalent to 0.007 mg/kg bw/d of glufosinate and 0.038 mg/kg bw/d of NAG.

[a] For eggs, the residues reported in the table are the maximum residues observed throughout the study for all five subgroups at each dose level while for tissues the individual results for the five subgroups at each dose level are given.

[b] Residue levels in tissues of hens dosed for 28 days with a mixture of glufosinate ammonium and NAG and then kept on a control diet for 7 days.

[c] Residue levels in tissues of hens dosed for 28 days with a mixture of glufosinate ammonium and NAG and then kept on a control diet for 14 days.

APPRAISAL

Glufosinate is a non-selective contact herbicide with uses on many crops, both conventional and glufosinate tolerant. Glufosinate has been evaluated several times by the JMPR with the initial evaluation in 1991 and the latest in 1999. Glufosinate-ammonium was scheduled at the Forty-third Session of the CCPR (2011) for periodic re-evaluation of toxicology and residues by the 2012 JMPR.

Glufosinate is a synthetic version of the natural product, phosphinothricin, and exists as a racemic mixture (i.e., 50:50 D- and L-glufosinate). The D-enantiomer is not herbicidally active. The L-enantiomer of glufosinate acts by inhibition of glutamine synthetase thereby causing accumulation of toxic levels of ammonium ion and indirectly stopping photosynthesis. Two genes for acetyltransferase, *bar* and *pat*, that were isolated from *Streptomyces hygroscopicus* and *Streptomyces viridochromogenes*, respectively, have been used to produce glufosinate-tolerant crops. For the current evaluation data have been submitted covering herbicide use on conventional and crops genetically modified by inclusion of the *pat* and *bar* gene to be glufosinate tolerant. These crops inactivate L-glufosinate by converting it to *N*-acetyl-glufosinate (NAG).

The Meeting received information on the metabolism of glufosinate and NAG (the main metabolite expected to be formed in tolerant plants) in animals, on glufosinate metabolism in conventional crops, and on crops genetically modified to contain the *pat* or *bar* genes and be glufosinate tolerant, methods of residue analysis, freezer storage stability, GAP information, supervised residue trials on conventional and glufosinate tolerant crops, fate of residue during storage and processing, and livestock feeding studies.

Metabolites referred to in the appraisal were addressed by their common names,

NAG N-acetyl-glufosinate;

MPP 3-methyl-phosphinico-propionic acid;

MPA 2-methyl-phosphinico-acetic acid;

MPB 4-methyl-phosphinico -butanoic acid;

MHB 4-methyl-phosphinico-hydroxy-butanoic acid;

PPO 4-methyl-phosphinico-2-oxo-butanoic acid.

Animal metabolism

Metabolism of glufosinate in cattle and hens involves formation of MPP and the slow transformation to MPA. NAG is metabolized by de-acetylation to form glufosinate with subsequent transformation proceeding as for glufosinate.

In one study a lactating goat was orally treated twice daily for 4 consecutive days with [3,4-^{14}C]- glufosinate at a dose equivalent to 101 ppm in the feed. Approximately 84% of the administered dose was recovered with the majority in the excreta (69% faeces, 3% urine) or gastrointestinal tract (12%). The radioactivity in the tissues ranged from 0.004 in fat to 0.61 mg/kg glufosinate equivalents in kidney. TRR values in milk were 0.003 to 0.022 mg/kg glufosinate equivalents during the dosing period with plateau levels reached after two days of dosing.

Major components of the ^{14}C residues were unchanged glufosinate (kidney 49% TRR, liver 53% TRR, milk 49% TRR) and MPP (kidney 29% TRR, liver 36% TRR and milk 6.3% TRR). The minor metabolites MPA and NAG individually accounted for no more than 5.3% TRR in kidney, liver and milk.

In a separate study a lactating goat was dosed twice a day with NAG at a dose equivalent to 84 ppm in the feed for 3 consecutive days. Most of the administered dose was recovered in the faeces (68%), urine (7.3%) and gastrointestinal tract (19%). Radioactive residues were highest in kidney (0.93 mg/kg NAG equivalents) with lowest levels in fat and muscle (< 0.01 mg eq./kg). ^{14}C levels in milk were 0.023 mg/kg NAG equivalents by the third day of dosing. Major components of the ^{14}C residues were glufosinate (kidney 40% TRR, liver 33% TRR, milk 40% TRR), unchanged NAG (kidney 32% TRR, liver 19% TRR, milk 9% TRR) and MPP (kidney 20% TRR, liver 21% TRR, milk 14% TRR). MPA was detected as a minor metabolite < 5% TRR.

Laying hens were orally treated twice daily for 14 consecutive days with [3,4-^{14}C]-glufosinate at a dose equivalent to 24.5 ppm in the feed and were sacrificed 15–16 hours after the last dose. The majority (92%) of the dose was eliminated in the excreta with 1.3% of the dose recovered from the GIT. Radioactivity in tissues ranged from 0.11 mg/kg glufosinate equivalents in kidney to 0.003 mg/kg glufosinate equivalents in fat and < 0.004 mg/kg glufosinate equivalents in muscle. The ^{14}C levels in egg whites and yolks reached 0.067 and 0.024 mg/kg glufosinate equivalents respectively by 14 days of dosing.

Unchanged glufosinate was the main residue component (liver 31% TRR, yolk 53% TRR, whites 78% TRR). MPP was a significant component of ^{14}C residues in liver (44% TRR) but a minor component in eggs (< 5%). MPA and NAG were also detected but at levels that represented < 5% TRR in liver and eggs.

The metabolism of NAG was studied in laying hens dosed orally twice daily for 14 consecutive days with [^{14}C]-NAG at a dose equivalent to 27 ppm in the feed and sacrificed 15 hours after the last dose. Most of the administered dose was recovered in the excreta (86%) with an additional portion (1%) found in the gastrointestinal tract at sacrifice. Radioactive residues in egg white reached a plateau by day 9 of dosing with a maximum level of 0.015 mg/kg NAG equivalents while egg yolk reached a plateau by day 12 with a maximum residue of 0.056 mg/kg NAG equivalents. In tissues at sacrifice, ^{14}C residues were highest in liver (0.076 mg/kg NAG equivalents) and much lower in muscle and fat (0.013 and 0.011 mg/kg NAG equivalents respectively).

NAG was a major component of the ^{14}C residues (liver 27% TRR, egg yolk 13% TRR, egg white 5% TRR) together with glufosinate (liver 15% TRR, egg yolk 3% TRR, egg white14% TRR) and MPP (liver 17% TRR, egg yolk 2.2% TRR, egg white 2% TRR). MPA was only detected as a minor metabolite and was present at < 1.1%TRR.

Metabolism in laboratory animals (rat) was summarized and evaluated by the WHO panel of the JMPR in the present meeting.

The metabolism of glufosinate and NAG in ruminants and laying hens is qualitatively the same as observed in rats.

Plant metabolism

Glufosinate-ammonium is used for three different situations:

- Directed sprays for weed control (crop not intentionally treated)
- Use as a crop desiccant to facilitate crop harvest (crop treated)
- Selective use in genetically modified glufosinate-tolerant crops (crop treated)

Plant metabolism studies were conducted with glufosinate-ammonium to investigate these three situations.

Directed sprays to weeds present in conventional crops

For weed control in conventional crops where the crop is not treated, metabolism studies were conducted in apple, grape, lettuce, corn/maize, wheat and potato as well as in artificial systems such as excised roots and leaves and cell cultures of a variety of weed and non-weed species.

The main metabolite observed in excised shots and leaves from conventional plants was MPP with lower amounts of MHB also detected.

When L-glufosinate was applied to conventional tobacco, alfalfa and carrot plants the main metabolites identified were MPP, MPB and MHB.

MPP was the only compound (89% TRR) detected in apples harvested 14 weeks after the soil under the trees was treated at 1.5 kg ai/ha with [2-^{14}C]-glufosinate-ammonium.

There was limited translocation of ^{14}C from soil-applied ^{14}C-glufosinate in a two year trial with grapevines where a single application was made in the first year and two in the second, all at 1.5 kg ai/ha. Levels of ^{14}C in grapes reached a maximum of 0.008 mg eq./kg and were too low to enable identification of components.

In a study where lettuce plants were transplanted in hydroponic solutions to which [3-^{14}C]-glufosinate-ammonium had been added, MPP represented about 90% of the TRR in leaves.

Following pre-crop emergent application of [3,4-^{14}C]-glufosinate-ammonium to potatoes at a rate equivalent to 1.0 kg ai/ha, 90% of the TRR in tubers at harvest was MPP while MPP represented > 80% TRR in leaves, stems and new sprouts.

In studies identifying residues in corn/maize following pre-crop emergent use of [3,4-^{14}C]-glufosinate-ammonium at 1.0 to 1.9 kg ai/ha, only low levels of ^{14}C were found in grain that precluded identification of components. Fodder had higher ^{14}C levels which were mostly MPP (60% TRR) with the remaining attributed to incorporation of ^{14}C into natural components (hemicellulose, lignin, cellulose, proteins and starch).

MPP was also the major component of the solvent extracted residue in wheat when [2-^{14}C]-glufosinate-ammonium is applied to soil at 1.2 kg ai/ha prior to crop emergence (straw 70%; husks 106%; grain 86%).

In summary, following directed application to soil and weeds, glufosinate related residues in conventional plants are almost entirely MPP.

Crop desiccation

The use as a pre-harvest desiccant was investigated in potato, rape and beans following foliar spray application to the crop. When applied to conventional crops for desiccation the residues consist primarily of glufosinate and its metabolite MPP. Since the plants are senescent at the time of application or die quickly after application, metabolism is essentially stopped and translocation from the treated parts of the crops into other plant parts such as seeds and roots is reduced. Following use as a crop desiccant, glufosinate was the major component of the ^{14}C residue in potato leaves and tubers, bean leaves, hulls and seeds and rape leaves, hulls and seeds accounting for more than 80% of TRR in combined surface rinses and solvent extracts. MPP was also detected but generally represented less than 10% of the TRR.

Glufosinate-tolerant crops

The use as a selective herbicide on tolerant plants was investigated following foliar application to glufosinate-tolerant crops such as corn/maize, rape, soya bean, rice, tomato and cotton.

The metabolic fate of [3,4-^{14}C]-glufosinate-ammonium in glufosinate tolerant tomato plants was examined following a single foliar application at 0.8 kg ai/ha at the 7–8 leaf growth stage. Extracts of rinsed leaves (absorbed ^{14}C) contained almost equal amounts of NAG and glufosinate and together accounted for > 90% TRR in the extracts. MPP was present as a minor component (< 10% TRR). Residues in fruit that developed after application were almost completely due to NAG.

The metabolic fate of [3,4-^{14}C]-glufosinate-ammonium in glufosinate-tolerant (*pat* gene) soya bean plants was examined in two studies where plants were sprayed with two applications at about 0.5 kg ai/ha. Soya bean plants were harvested at typical forage stage and at maturity (PHI 84–85 days). In soya forage the major components of the ^{14}C residues were NAG (26–60%) and glufosinate (18–23%) with MPP and MPA present at < 10% TRR. For straw, pods, husks and beans at harvest, NAG (28–63%), glufosinate (11–18%) and MPP (10–22%) were major components of ^{14}C with smaller amounts of MPA also observed (1.3–7.1%).

In a study of [3,4-^{14}C]-glufosinate-ammonium metabolism in glufosinate-tolerant sugar beet plants were sprayed twice, at 5 weeks after sowing and 22 days later, at 0.6 kg ai/ha. At harvest, 146 days after the last application, the major components of ^{14}C identified in leaves and roots were NAG (67–68%) and glufosinate (19–24%) with low levels of MPP (2.7–6%).

The metabolic fate of [^{14}C]-glufosinate-ammonium in tolerant (*pat* gene) corn/maize plants was examined following application at 0.5 kg ai/ha when plants were 40 cm high and 10 days later when 60 cm high. ^{14}C residues in forage at 28 days after treatment were 2.6 mg/kg glufosinate equivalents with major components NAG (52%), glufosinate (13%) and MPP (12%). Maize fodder at harvest contained 0.2 mg eq./kg comprising NAG (54%), MPP (11%) and glufosinate (10%) as the main ^{14}C residue components. In grain at harvest, ^{14}C residues were 0.13 mg/kg glufosinate equivalents comprising MPP (37%), NAG (19%), MPB (9.8%) and MPA (4.4%) with glufosinate present but only at very low levels (< 1.5%). In a comparison of crops made tolerant through incorporation of the *pat* or *bar* genes the same metabolite profile was observed for both modifications.

The metabolic fate of [^{14}C]-glufosinate-ammonium in tolerant rice plants was examined following application at 0.5 kg ai/ha when plants were at the 2–4 leaf stage and then at the 2–4 tiller stage. Plants were managed under two flooding regimes, the first was with flooding 2 days prior to the first treatment and the second regime was flooding one day after the second treatment. There were no significant differences in metabolite profiles between the two management regimes. ^{14}C residues in forage at 18 days after the last treatment were 2.0–2.6 mg/kg glufosinate equivalents with major components NAG (54–64%), glufosinate (8.1%) and MPP (7.8–9%). Straw at harvest (184 days after the last application) contained 9.5–13.1 mg eq./kg comprising NAG (60%), MPP (10–13%) and glufosinate (18%) as the main components. In grain at harvest ^{14}C residues were 1.1–1.4 mg/kg glufosinate equivalents comprising MPP (68–72%), NAG (11%), MPB (9.8%) and glufosinate (5–6%) with MPA also present but only at very low levels (0.6–0.8%).

In an additional study tolerant (*bar* gene) and conventional cotton were treated at the 10 leaf stage with a commercial formulation of glufosinate-ammonium at 0.5 kg ai/ha, and selected individual leaves spotted with a solution of ^{14}C-glufosinate-ammonium. Tolerant cotton showed high levels of metabolism with NAG accounting for 72% TRR at 72 hours after application while in conventional cotton glufosinate accounted for 73% TRR at 72 hours.

The metabolic fate of [^{14}C]-glufosinate-ammonium in tolerant rape plants was examined in several studies. Following application to tolerant rape at the three leaf growth stage at 0.75 kg ai/ha, ^{14}C residues in foliage at 21 days were 4.3 mg/kg glufosinate equivalents with major components NAG (60%), glufosinate (21%) and MPP (6.7%). In seeds at harvest (120 days after application) ^{14}C residues were 0.04–0.11 mg/kg glufosinate equivalents comprising glufosinate (0–14%) and MPP (3-45%) with NAG present but only at very low levels (< 2%). Following two applications at 0.8 kg ai/ha to tolerant (*pat* gene) rape at the 5–6 leaf growth stage and when plants were 50 cm high,

residues in forage 154 days after the first application were 0.2 mg eq./kg and with major components NAG (71%) and glufosinate (7.9%). At harvest 102 days after the last application, ^{14}C residues in straw and hulls were 12.7 and 7.1 mg eq./kg respectively with major components NAG (57–77%) and glufosinate (21–31%). Lower levels of ^{14}C were detected in seed (0.5 mg eq./kg) with NAG (32%) the major component and glufosinate (6.2%) and MPP (9.7%) minor components together with trace amounts of MPA and MPB. In a comparison of crops made tolerant through incorporation of the *pat* or *bar* genes, the same metabolite profile was observed in shoots sampled 14 and 28 days after treatment for both modifications.

The pathway of glufosinate-ammonium in tolerant plants genetically modified to contain the *pat* or *bar* genes is rapid deactivation of L-glufosinate through N-acetylation to form NAG. D-glufosinate is not acetylated but rather undergoes slow metabolism to form MPP, MPB and MPA.

The metabolism of glufosinate-ammonium by plants is well understood for the three different situations. The metabolism of glufosinate-ammonium in conventional (glufosinate-susceptible) crops following directed application to weeds or as a pre-harvest desiccation use is in principle identical for the five standard crop groups: fruit crops, cereal/grass crops, leafy crops, root crops and pulses and oilseeds. For these crop groups major residue components consist of parent glufosinate and the metabolite MPP. In case of glufosinate-tolerant crops, NAG, MPP and glufosinate are the major components of the residue.

Environmental fate

The Meeting received information on soil aerobic metabolism, soil photolysis and aqueous hydrolysis properties of [^{14}C]-glufosinate-ammonium. Studies were also received on the behaviour of [^{14}C]-glufosinate-ammonium in a rotational crop situation.

Glufosinate-ammonium residues are not persistent in soils and residues in soils resulting from approved uses should not contribute significantly to the residues in succeeding crops.

In soil incubation studies under aerobic conditions in the dark at 20 °C, glufosinate disappeared with a half-life of 1–25 days. Glufosinate was not significantly metabolized in sterile soil demonstrating the importance of microbial metabolism in its soil degradation. Under aerobic soil incubation, the first metabolite formed was MPP followed by MPA, which mostly disappeared with half-lives in the range of 13–22 days (n = 4) and 18 days (n = 1) respectively. After 120 days, 17–81% of the applied dose was mineralised with 20–38% remaining unextracted with the solvents used. Further analysis of the unextracted portion of ^{14}C demonstrated incorporation into humin, fulvic acid and humic acid fractions present in the soil.

In a soil photolysis study with application of ^{14}C-glufosinate-ammonium on the surface of a sterilised sandy loam soil, glufosinate was stable to continuous irradiation for 120 hours suggesting photolysis has negligible effect on degradation when compared to metabolism.

Glufosinate-ammonium was also stable to hydrolysis in aqueous solutions at pH 5, 7 and 9 suggesting hydrolysis plays a negligible role in the degradation of glufosinate when compared to metabolism.

In a confined rotational crop study with wheat, lettuce and radish, a plot of sandy loam soil was treated with [^{14}C]-glufosinate-ammonium at the equivalent of 1.0 kg ai/ha and crops sown 28, 119 and 300/364 days. The proportion of ^{14}C residue attributed to glufosinate-related compounds decreased with increasing plant-back interval (PBI). In wheat straw and grain sown at a PBI of 119 days, MPP represented 22% TRR in straw and 12% in grain while MPA represented 6% TRR in straw and 0.3% in grain. Natural products accounted for the remaining TRR.

In summary, glufosinate residues in soil should contribute little to residue levels in rotational crops.

Methods of analysis

The Meeting received description and validation data for analytical methods for residue analysis of glufosinate and its metabolites MPP and NAG in various plant and animal commodities. Most of the analytical methods for the determination of glufosinate-derived residues developed prior to 2006 follow the same general principle. The relevant residues are usually extracted in water. Thereafter, the aqueous extract is concentrated to dryness and reacted with trimethyl orthoacetate in the presence of acetic acid. After silica gel purification the obtained derivatives are measured by GC/FPD or more recently by LC-MS/MS. LOQs are typically 0.01–0.05 mg/kg for glufosinate and MPP.

When reacted with trimethyl orthoacetate in the presence of acetic acid, glufosinate and NAG yield the same derivative. Therefore, glufosinate and NAG are usually determined together as a sum. However, if the two compounds are separated before derivatisation by means of cation exchange clean-up it is possible to determine the residues of glufosinate and NAG separately. In some methods glufosinate, NAG and MPP were measured separately with LOQs typically in the range 0.01 to 0.05 mg/kg.

With the availability of new liquid chromatography-columns specially designed for highly polar character and comparatively low molecular weight compounds such as glufosinate and its metabolites, it is possible to quantify glufosinate, MPP and NAG using LC/MS/MS without prior derivatisation (LOQs 0.01–0.02 mg/kg).

In another method, derivatisation of glufosinate was carried out with a mixture of o-phthalic dialdehyde and mercapto-propionic acid in the presence of sodium borate. The derivative of glufosinate is quantified by LC-MS/MS while the metabolites MPP and NAG are measured by LC-MS/MS without prior derivatisation as in the previous approach (LOQs 0.01 mg/kg for each analyte for plant and animal matrices).

Multi-residue methods are currently not validated for glufosinate and its metabolites.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of glufosinate-ammonium and its metabolites MPP and NAG in samples of commodities from conventional crops stored frozen. NAG is not expected to be present in commodities from conventional crops.

Glufosinate, NAG and MPP are stable for at least 24 months in peaches when stored frozen. Glufosinate and MPP (NAG not studied) residues are stable for at least 6 months in kiwifruit, 12 months in almond, 20 months in blueberries, 24 months in apple, orange, maize grain and soya bean seeds from conventional crops stored frozen.

Glufosinate, NAG and MPP residues are stable for at least 24 months in tolerant sugar beet roots and tops and for at least 9 months in sugar beet processed fractions, for at least 24 months in soya bean seed and hay and at least 12 months for soya bean processed fractions, at least 23 months for rape seed, at least 24 months in maize forage and grain and at least 12 months in maize processed fractions, at least 26 months for sweet corn forage, at least 30 months for sweet corn ears, and at least 12 months for rice grain.

In animal commodities, glufosinate-ammonium, NAG and MPP were stable for at least 14 months in cow milk, muscle, liver and kidney. Glufosinate, NAG and MPP were stable for at least 15 months in hen eggs, muscle and liver.

The periods of demonstrated stability cover the frozen storage intervals used in the residue studies.

Definition of the residue

Livestock may be exposed to residues present in feeds prepared from conventional and glufosinate-tolerant crops. Residues in conventional crops are principally glufosinate and MPP while in tolerant crops they are glufosinate and NAG. Metabolism of glufosinate in goats and hens involves formation of MPP and the slow transformation to MPA. NAG is metabolized by de-acetylation to form

glufosinate with subsequent metabolism proceeding as for glufosinate. The major components of the residue in livestock are glufosinate, MPP and NAG and should be included in the residue definition for compliance with MRLs and estimation of dietary intake in animal commodities.

In the metabolism studies, residues in muscle and fat were too low to identify the proportions of the individual components required to determine relative partitioning between fat and muscle. In the metabolism study for NAG in lactating goats, radioactive residues in whole milk were 0.005–0.023 mg eq./kg, skim milk 0.007–0.023 mg eq./kg and cream 0.022–0.046 mg eq./kg with the profile of components similar in all three products. The data for milk suggest NAG partitions slightly more into the fat than aqueous portions of milk. In eggs, the concentration of glufosinate was greater in whites when compared to yolks while for NAG the opposite occurred. In the lactating cow feeding study with glufosinate and MPP, higher concentrations of glufosinate and MPP were found in fat compared to muscle. The combined residues of glufosinate (glufosinate +MPP+NAG) are borderline fat-soluble. The Meeting decided that residues of glufosinate are not fat-soluble.

Glufosinate ammonium is used on crops for three different situations:

- Directed sprays for weed control (crop not intentionally treated)
- Use as a crop desiccant to facilitate crop harvest (crop treated)
- Selective use in genetically modified glufosinate-tolerant crops (crop treated)

The main metabolite observed in studies with conventional crops (directed sprays for weed control, pre-emergent or pre-sowing applications) is MPP representing greater than 80% of the ^{14}C residue not attributed to natural products in apples, grapes, potatoes, wheat and corn fodder. However, the residue levels are generally low, less than 0.1 mg/kg, with only occasional higher residues detected.

Following use as a crop desiccant, glufosinate was the major component of the residue in potato leaves and tubers, bean leaves, hulls and seeds and rape leaves, hulls and seeds accounting for more than 80% of TRR. MPP was also detected but generally represented less than 10% of the TRR.

In the case of foliar applications to glufosinate-tolerant crops, glufosinate, NAG and MPP are the major residue components. NAG accounted for 19–77% TRR in soya, maize, rape, rice and sugar beet foliage (forage, fodder, straw), 68% TRR in sugar beet roots, 11–63% TRR in grain and husks (soya, maize, rice, rapeseed). Glufosinate accounted for 8–31% TRR in soya, maize, rape, rice and sugar beet foliage (forage, fodder, straw), 19–24% TRR in sugar beet roots, < 2–18% TRR in grain and husks (soya, maize, rice, rapeseed). MPP generally represented no more than 12% TRR in foliage but was a significant component of the residue in grains at 3–72% TRR.

In plants, the majority of glufosinate-related residues in conventional and tolerant crops are accounted for in the previous residue definition; the sum of glufosinate, MPP and NAG. Although NAG is not a significant residue in conventional crops, some analytical methods do not distinguish between residues of glufosinate and NAG and so NAG should be included in the residue definition for compliance for conventional and tolerant crops.

The toxicological evaluation of glufosinate-ammonium concluded that the ADI and ARfD apply to glufosinate, NAG, MPP and MPA. MPA is a minor component of the residue and does not contribute significantly to dietary intake. The Meeting decided it was not necessary to include MPA in the residue definition for estimation of dietary intake.

Based on the above the Meeting confirmed the previous residue definition for compliance with MRLs and estimation of dietary intake as follows:

Definition of the residue for compliance with MRL and estimation of dietary intake (for animal and plant commodities): *sum of glufosinate, 3-[hydroxy(methyl)phosphinoyl]propionic acid (MPP) and N-acetyl-glufosinate (NAG), calculated as glufosinate (free acid).*

The residue is not fat-soluble.

Results of supervised residue trials on crops

The Meeting received supervised residue trial data for glufosinate-ammonium on citrus fruit, pome fruit, stone fruit, small fruit and berries including grapes, tropical fruit (inedible and edible peel), bulb onions, glufosinate-tolerant sweet corn, leafy vegetables, legume vegetables, pulses including glufosinate tolerant soy beans, carrots, potatoes, glufosinate tolerant sugar beet, asparagus, glufosinate tolerant maize, glufosinate tolerant rice, tree nuts, glufosinate tolerant cotton, conventional rape, glufosinate tolerant rape, sunflower and coffee as well as for some animal feed commodities.

Residues reported in supervised trials consist of glufosinate and NAG, often reported as the sum, and MPP. In metabolism studies, one of the three components comprises the majority of the combined total when all three are present. It is reasonable to assume when all three components are below the LOQ, the combined total is also below or close to the LOQ. Also when one component is above and the others below the LOQ, the combined residue is assumed to be equal to the residue of the main component. This is illustrated below.

Glufosinate	MPP	NAG	Total residues (glufosinate+MPP+NAG)
< 0.05	< 0.05	< 0.05	< 0.05 or where LOQs differ, the highest LOQ
< 0.05	< 0.05	0.06	0.06
0.05	< 0.05	0.09	0.14
< 0.05	0.06	< 0.05	0.06

The OECD calculator was used as a tool in the estimation of the maximum residue level from the selected residue data set obtained from trials conducted according to GAP. As a first step, the Meeting reviewed all relevant factors related to each data set in arriving at a best estimate of the maximum residue level using expert judgement. Then, the OECD calculator was employed. If the statistical calculation spreadsheet suggested a different value from that recommended by the JMPR, a brief explanation of the deviation was provided.

Some use patterns for field crops include the possibility of pre-planting or pre-emergent applications together with post-emergent applications. Often the supervised trials for these field crops did not include pre-emergent application. However, as metabolism and rotational crop studies suggest the contribution from pre-emergent applications are negligible in comparison to the contribution from post-emergent applications, the lack of a pre-emergent application should not affect the decision as to whether the trials approximate critical GAP.

As no data were available for almond hulls, broad bean (dry) and peas (dry) the previous recommendations for these commodities are withdrawn.

Citrus fruits (application to weeds)

Field trials involving citrus orchards where glufosinate-ammonium was applied to weeds were conducted in Brazil, Europe and the USA and were available to the Meeting.

The GAP for citrus in Brazil is application directed to weeds at 0.4 kg ai/ha with a PHI of 40 days. In the trials matching this GAP total glufosinate residues (glufosinate + NAG + MPP) in ranked order were (n = 2): < 0.04 (2) mg/kg.

In Portugal, glufosinate ammonium is applied to weeds in citrus orchards at up to 1.5 kg ai/ha with a 0 day PHI. None of the trials from Europe matched the GAP of Portugal and an approved use on citrus in the USA was not available.

As the use pattern is application to weeds and not the crop, trials from different regions of the world can be used to support maximum residue level recommendations based on GAP from another region. The Meeting noted that total residues were < 0.05 (21) mg/kg in the trials on citrus (oranges, mandarin, grapefruit and lemons) conducted in the USA that utilized rates higher than permitted in Portugal (3×1.7 kg ai/ha USA versus 1–2 × 0.4–1.5 kg ai/ha in Portugal). Additionally, no residues were detected in trials in Europe that were conducted at rates lower than the maximum Portugal rate (2 sprays the last at 0.75 to 1.0 kg ai/ha). One trial from Brazil conducted at a much lower rate (0.4 kg ai/ha) reported very high residues at 10, 20 and 30 days after application with glufosinate the major

component of the residue in fruit suggesting application to the fruit. This trial was not used in maximum residue estimation. The Meeting concluded that residues above LOQ are not expected in citrus fruit following application of glufosinate-ammonium to weeds growing in orchards.

The Meeting estimated a maximum residue level of 0.05 mg/kg for citrus fruit to replace its previous recommendation of 0.1 mg/kg. The Meeting estimated an STMR of 0.05 mg/kg and an HR of 0.05 mg/kg for citrus fruit.

Pome fruits (application to weeds)

Field trials involving apples and pears conducted in Brazil, Europe and the USA were made available to the Meeting.

Apples

The GAP for apples in Brazil is application directed to weeds at 0.4 kg ai/ha with a PHI of 7 days. In the trials matching this GAP the total glufosinate residues were (n = 4): < 0.04 (4) mg/kg.

In Germany, glufosinate-ammonium is applied to weeds in apple orchards as two applications at up to 1.5 (spring) + 1 (spring/summer) kg ai/ha with a 14 day PHI. No residues (total glufosinate) were observed in 10 trials from Europe that approximated maximum GAP for Germany (n = 10): < 0.05 (8) and < 0.06 (2) mg/kg.

GAP in the USA is application to weeds at a maximum rate of 1.7 kg ai/ha with a maximum annual rate of 5 kg ai/ha/year. The PHI is 14 days. No residues (total glufosinate) were detected (< 0.05 mg/kg) in 10 trials where application rates ranged from 1 × 1.1 kg ai/ha to 3 × 4.5 kg ai/ha. The Meeting decided that residues of glufosinate are not expected in apples following directed application to orchard weeds.

Pears

Trials on pears with application to weeds were conducted in the USA but use of glufosinate-ammonium in pear orchards is not listed on USA labels. The use pattern in Germany allows glufosinate-ammonium to be applied to weeds in pear orchards as two applications at up to 1.5 (spring) + 1 (spring/summer) kg ai/ha with a 14 day PHI. Trials in the USA were assessed against the GAP of Germany. Total residues in six trials that approximated maximum GAP for Germany (n = 6) were: < 0.05 (5) and 0.08 mg/kg.

In 25 of 26 trials on apples and pears that approximated the GAP of Germany or the USA, total residues were < LOQ. The residue value of 0.08 mg/kg in pear was due to MPP, likely taken up from soil. In the apple metabolism study where the soil beneath a tree was treated at 1.5 kg ai/ha, total residues in fruit were 0.1 mg/kg at harvest, fourteen weeks after the soil treatment. Occasional residues due to uptake of the soil degradation product MPP are expected, as are occasional residues of glufosinate due to spray directed to weeds inadvertently contacting fruit. As location of the trials should not be an important factor in the residues, the Meeting decided to combine the results for apples and pears and estimated a maximum residue level of 0.1 mg/kg for glufosinate residues in pome fruit replacing its previous recommendation of 0.05* mg/kg.

The STMR and HR for pome fruit are 0.05 and 0.08 mg/kg respectively.

Stone fruits (application to weeds)

Field trials involving application to weeds under apricot, cherry, peach, plum and nectarine trees were made available from Brazil, Europe and the USA.

In Brazil, weeds under peach and nectarine trees may be treated at 0.4 kg ai/ha with a PHI of 7 days. In the Netherlands weeds in fruit tree orchards may be sprayed at up to 1 kg ai/ha, no PHI required. In Canada weeds in peach and plum orchards may be sprayed at up to 0.75 kg ai/ha, maximum 1 kg ai/ha/year with a PHI of 40 days. In Portugal and Germany (except peach), weeds under stone fruit trees may be sprayed at up to 1.5 kg ai/ha with PHIs of not required for Portugal and

14 days for Germany. Use on stone fruit in the UK as at rates up to 0.75 kg ai/ha with no PHI required.

The location of the trial sites should not be a significant influence on residues in fruit when glufosinate-ammonium is applied to weeds. In a large number of trials from various locations total residues above the LOQ were infrequent. Occasional residues due to uptake of the soil degradation product MPP might be expected as are occasional residues of glufosinate due to spray directed to weeds inadvertently contacting fruit. Total residues in fruit from trials carried out with application rates ranging from 1 × 0.4 to 2 × 1.7 kg ai/ha were (n = 42): apricot < 0.02, 0.05; cherry 0.02, < 0.05 (7); peach and nectarine < 0.02 (2), < 0.05 (19), 0.08; plum < 0.01 (5), 0.01 (3), < 0.05 (4), 0.06 and 0.07 mg/kg.

The Meeting estimated a maximum residue level of 0.15 mg/kg for stone fruit to replace its previous recommendation of 0.05* mg/kg. The Meeting estimated an STMR of 0.05 mg/kg and an HR 0.08 mg/kg.

Berries and other small fruit (application to weeds)

Currants

Glufosinate-ammonium is approved for application using spray shields to weeds between currant bushes in the Netherlands (1 kg ai/ha, no PHI required), the UK (soft fruit 0.75 kg ai/ha, no PHI required) and the USA (1.7 kg ai/ha, maximum 3.4 kg ai/ha/year, PHI 14 days). In twelve trials conducted in France, Germany and the UK where glufosinate-ammonium was applied as two sprays at 1 kg ai/ha, as two sprays with one at 1.6 kg ai/ha and the other at 1 kg ai/ha or as three sprays at 0.75 kg ai/ha, total residues in currants at PHIs of 0 to 28 days were (n = 11): < 0.01_s (3), 0.01_s, < 0.02, $\underline{0.02}_s$, 0.05, 0.08_s, 0.12, 0.43_s and 0.48 mg/kg. Trials indicated with an "s" utilized a spray shield to reduce crop contamination. The major component of the samples with total residues 0.43 and 0.48 mg/kg were glufosinate and MPP respectively. Residues in currants appear to be from both uptake of MPP from soil and inadvertent contamination when spraying weeds. A trial with a total residue of 2.4 mg/kg was not used as the level was much higher than observed in the other trials with the residue due almost entirely to glufosinate. The magnitude of the residue was considered too high to represent good practice in application of glufosinate.

The Meeting estimated a maximum residue level of 1 mg/kg for currants to replace its previous recommendation of 0.5 mg/kg.

The Meeting estimated an STMR of 0.02 mg/kg and an HR of 0.48 mg/kg for currants.

Grapes

Trials were available from Brazil, Europe and the USA. The use pattern in Argentina is application to weeds at up to 1.6 kg ai/ha, no PHI required while Brazil allows application to weeds at 0.4 kg ai/ha with a PHI of 7 days. In Australia application to weeds is at up to 1 kg ai/ha no PHI required. Germany allows an application to weeds at 1.5 kg ai/ha in spring and one in summer at 1 kg ai/ha with a 14 day PHI. In the USA application to weeds is at up to 1.4 kg ai/ha with a maximum of 5 kg ai/ha/year and a PHI of 14 days. Residue data were available from 30 different trial locations with data presented for berries in accordance with the Codex Classification or for bunches. Where both berries and bunches were analysed only values for berries were considered.

In trials from Brazil matching GAP of that country total residues were: < 0.04 (3) mg/kg.

In trials from Europe approximating the GAP of Germany total residues were (n = 14): < 0.01 (3), < 0.02 (3) $\underline{0.02}$ (2), < 0.05 (4), 0.06 and 0.12 mg/kg.

Total residues in grapes from US trials that approximated the GAP of the USA were (n = 5): < 0.05 (5) mg/kg.

The Meeting considered the number of trials available from Brazil and the USA too few and utilized the trials from Europe approximating German GAP to estimate a maximum residue level of

0.15 mg/kg for grapes. The Meeting estimated an STMR of 0.02 mg/kg and an HR of 0.12 mg/kg for grapes.

Strawberries

Trials were available from Finland, France and Germany. GAP in strawberry in Australia is application to weeds at 1 kg ai/ha with no PHI required, Germany as a single shielded application to weeds at 0.8 kg ai/ha (pre-flowering of strawberry plants) with a 42 day PHI and UK one to two applications to weeds at up to 0.75 kg ai/ha with no PHI required.

Total residues in three trials approximating German GAP (but not using spray shields) were: < 0.05 (3) mg/kg.

Total residues in 8 trials approximating UK GAP, conducted as one to four sprays at 0.75 kg ai/ha with harvest 4 days after last application, were (n = 8): < 0.01_s, < 0.01_s, 0.02_s, 0.02, 0.02, 0.03, 0.06 and 0.15 mg/kg. Trials indicated with an "s" utilized a spray shield to reduce crop contamination. Although the number of applications was greater than recommended, earlier applications should not contribute significantly to residues as the interval between sprays was 21–101 days compared to a typical interval between flowering and harvest of about 28 days.

The Meeting utilized trials approximating UK GAP to estimate a maximum residue level of 0.3 mg/kg for strawberries. The Meeting estimated an STMR of 0.02 mg/kg and an HR of 0.15 mg/kg for strawberries.

Blueberries

In trials conducted in the USA according to US GAP (application to weeds at 1.7 kg ai/ha, maximum 3.4 kg ai/ha/year, PHI 14 days) total residues in blueberries were (n = 5): < 0.05 (4) and 0.06 mg/kg. The individual component responsible for the highest residue was glufosinate, possibly reflecting inadvertent exposure to spray during application. An additional trial with residues < 0.05 mg/kg had residues in control samples of 0.07 mg/kg and was therefore not considered. Inadvertent contamination of blueberries with glufosinate may occur when spraying weeds between bushes. The Meeting estimated a maximum residue level of 0.1 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.06 mg/kg for blueberries.

Gooseberries

GAP in Germany is application using spray shields to weeds between bushes at 1×1 kg ai/ha, PHI 14 days, in the UK (soft fruit) application to weeds at 0.75 kg ai/ha with no PHI required and in the USA application to weeds at1.7 kg ai/ha with a maximum of 3.4 kg ai/ha/year and a PHI of 14 days). In three trials from Germany where weeds were sprayed at 2 × 1.5 kg ai/ha and approximating the GAP of the USA total residues were (n = 3): < 0.02 (3) mg/kg.

Inadvertent contamination of gooseberries with glufosinate may occur when spraying weeds between bushes. The Meeting estimated an STMR of 0.02 mg/kg, an HR of 0.02 mg/kg and a maximum residue level of 0.1 mg/kg for gooseberries.

Raspberries

Trials were conducted on raspberry bushes in France, Germany and Italy. GAP in Australia is application to weeds at up to 1 kg ai/ha with no PHI required; Canada application to weeds at 1 kg ai/ha with no PHI required; Germany application using spray shields to weeds at 1 kg ai/ha with a PHI of 14 days; in the Netherlands application using spray shields to weeds at 1 kg ai/ha with no PHI required; and in the UK (soft fruit) application to weeds at 0.75 kg ai/ha with no PHI required. Total residues, after application at 3 × 0.75 kg ai/ha approximating the GAP of the UK were: < 0.01, 0.03 and 0.03 mg/kg. A spray shield was used in these trials to minimise crop contamination.

Inadvertent contamination of raspberries with glufosinate may occur when spraying weeds between raspberry plants. The Meeting estimated an STMR of 0.03 mg/kg, an HR of 0.03 mg/kg and a maximum residue level of 0.1 mg/kg for raspberries.

The Meeting agreed to withdraw its previous recommendation for berries and other small fruits (except currants) of 0.1 mg/kg.

Assorted tropical and sub-tropical fruit, edible peel (application to weeds)

Carambola

In Malaysia, weeds under carambola trees may be sprayed at 0.5 kg ai/ha, PHI 14 days. In two trials approximating GAP of Malaysia total residues were: < 0.05 (2) mg/kg.

Olives

Trials on olives were available from Europe and the USA. GAP in Portugal and Spain is application to weeds at up to 1.5 kg ai/ha, no PHI required in Portugal and 21 days in Spain while in Australia application to weeds of up to 1 kg ai/ha is permitted with no PHI required. In four trials from Europe where weeds in orchards were sprayed with two applications, the first at 1.1 and the second at 0.75 kg ai/ha, total residues at a PHI of 0 days were < 0.01 (2), 0.01 and 0.03 mg/kg. In three trials from the USA where weeds were treated at 3 × 1.5 kg ai/ha, total residues were all < 0.05 mg/kg 14 days after the last application.

Following the use of glufosinate-ammonium as a directed spray to control weeds in orchards residues of glufosinate are generally not expected as glufosinate is not readily taken up by roots. Occasional residues of MPP, the main degradation product in soil, are expected as MPP is translocated through the crop. Inadvertent contamination of fruit by small amounts of glufosinate spray may also occur. The Meeting agreed that recommendations for maximum residue levels for assorted tropical and sub-tropical fruit, edible peel should accommodate the occasional residues that occur by the routes discussed above and that a level of 0.1 mg/kg would be adequate to accommodate such residues.

The Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.05 mg/kg and a maximum residue level of 0.1 mg/kg for assorted tropical and subtropical fruit, edible peel.

Assorted tropical and sub-tropical fruit, inedible peel (application to weeds)

Banana

Glufosinate-ammonium is permitted to be used for weed control in banana plantations in Australia (1 kg ai/ha, PHI 0 days), Brazil (0.4 kg ai/ha, PHI 10 days), Columbia (0.3 kg ai/ha, PHI 0 days), Malaysia (0.5 kg ai/ha, PHI 14 days), Mexico (0.56 kg ai/ha, PHI 0 days), Philippines (2 kg ai/ha, PHI 0 days), Portugal (1.5 kg ai/ha, PHI 0 days) and Taiwan (1 kg ai/ha PHI 0 days).

Residues do not show a decline typical for application to fruit or foliage of bananas but rather following application to weeds and soil residues often only develop in fruit following a significant interval between spraying weeds and harvest of bananas, presumably due to uptake of MPP or D-glufosinate from soil. The Meeting decided to use the highest residue observed at any interval after application to weeds. In addition in many of the trials multiple applications were made at relatively long intervals between sprays (23 to 137 days) with harvest between sprays. The conditions preceding each harvest are sufficiently different to be considered as independent trials. Total residues in trials from Central and South America and the Philippines where weeds were treated at 1 × 0.3 kg ai/ha to 5 × 2 kg ai/ha + 8 × 1.2 kg ai/ha were (highest of pulp or peel if whole fruit not measured) (n = 86): 0.02, < 0.04 (2), 0.04, < 0.05 (56), 0.05 (3), < 0.06 (3), 0.06 (8), 0.07 (3), 0.08 (4), 0.09, 0.10, 0.11 (2) and 0.13 mg/kg.

In two trials from Brazil conducted at a much lower rate (0.4 kg ai/ha), residues at 0 days after a single application at 0.4 kg ai/ha were 0.49 and 0.53 mg/kg with residues also observed at 3 and 7 days after application. As the high residue levels and the observation that glufosinate was the major component of the residue in fruit suggest direct application to the banana plants the Meeting could not be sure the residues represent inadvertent contamination. These two trials were not used for maximum residue estimation.

Using the available data, the Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.13 mg/kg and a maximum residue level of 0.2 mg/kg to for bananas to confirm its previous recommendation of 0.2 mg/kg. The Meeting noted that for banana the IESTI accounts for 110% of the ARfD. The residues arise from uptake of glufosinate related residues from the soil. No suitable alternative GAP was identified that would resolve the intake concern.

Kiwifruit

Trials were available from Italy and the USA. GAP in Australia is application to weeds at 1 kg ai/ha with no PHI required and in Japan 1.4 kg ai/ha with a PHI of 21 days. Weeds under "woody crops" in Spain may be treated at 0.45–1.5 kg ai/ha, no PHI required. Total residues in nine trials conducted approximating the GAP of Spain with applications ranging from two sprays at 1.4 and 1.0 kg ai/ha to three sprays at 2.0 kg ai/ha were: < 0.01 (2), < 0.05 (4), 0.07, 0.10 and 0.37 mg/kg.

Using GAP of Spain, the Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.37 mg/kg and a maximum residue level of 0.6 mg/kg to for kiwifruit. The Meeting noted that for kiwifruit the IESTI accounts for 110% of the ARfD. No residue data were identified that complied with an alternative GAP that would resolve the intake concern.

Avocado

In two trials approximating GAP in Australia for application to weeds in avocado orchards (1 kg ai/ha, no PHI required) total residues were: < 0.06 (2) mg/kg.

Guava

In two trials approximating GAP in Malaysia for weed control in guava orchards (0.5 kg ai/ha, PHI 14 days) total residues were: < 0.05 (2) mg/kg.

Mango

In two trials approximating GAP in Australia for weed control in mango orchards (1 kg ai/ha, no PHI required) total residues were: < 0.05 (2) mg/kg.

Papaya

In one trial approximating GAP in Australia for weed control in papaya orchards (1 kg ai/ha, no PHI required) total residues were: < 0.06 mg/kg.

As noted earlier, following the use of glufosinate-ammonium as a directed spray to control weeds in orchards residues of glufosinate are generally not expected as glufosinate is not readily taken up by roots. Occasional residues of MPP, the main degradation product in soil, are expected as MPP is translocated through the crop. Inadvertent contamination of fruit by small amounts of glufosinate spray may also occur. The Meeting agreed that recommendations for maximum residue levels for assorted tropical and sub-tropical fruit, inedible peel should accommodate the occasional residues that occur by the routes discussed above and that a level of 0.1 mg/kg would be adequate to accommodate such residues for fruit other than banana and kiwifruit.

The Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.05 mg/kg and a maximum residue level of 0.1 mg/kg for assorted tropical and subtropical fruit, inedible peel (except banana and kiwifruit).

The Meeting agreed to withdraw its previous recommendation for assorted tropical and sub-tropical fruits-inedible peel (except banana) of 0.05* mg/kg.

Onion, bulb (pre-crop emergence or pre-sowing)

Use on glufosinate-ammonium on onions for control of weeds when applied pre-emergent or pre-sowing of onions is permitted in Canada (GAP 0.75 kg ai/ha, no PHI required). In twenty trials approximating Canada GAP total residues were: < 0.01 (6) and < 0.05 (14) mg/kg. The Meeting

estimated an STMR of 0.05 mg/kg, an HR of 0.05 mg/kg and a maximum residue level of 0.05 mg/kg for bulb onions to confirm its previous recommendation of 0.05 mg/kg.

Sweet corn (tolerant)

Residue trials from the USA were received for use on glufosinate tolerant sweet corn however there is no associated GAP in the USA.

Corn salad (pre-crop emergence)

In Germany glufosinate-ammonium is approved for control of weeds in corn salad (lambs lettuce) when applied prior to emergence of the crop. The application rate is 0.6 kg ai/ha with no PHI required. Four trials were available from Germany on corn salad that matched GAP with total residues < 0.05 (4) mg/kg. The Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.05 mg/kg and a maximum residue level of 0.05 mg/kg for corn salad to replace its previous recommendation of 0.05* mg/kg.

Lettuce (application to weeds)

Residue trials were available from Brazil and various countries from Europe following one directed spray to weeds or one spray pre-planting or a combination of the two. GAP was available for Brazil (directed application to weeds at 0.4 kg ai/ha, PHI 7 days, protect plants with plastic cups), Canada (pre-crop emergence, pre-crop sowing application to weeds at 0.75 kg ai/ha, no PHI required), Japan (directed application to weeds at 0.7 kg ai/ha, PHI 60 days) and Portugal (application to weeds pre-sowing/pre-emergence/pre-planting at 0.75 kg ai/ha, no PHI required). The Meeting decided to combine the dataset to recommend a maximum residue level for lettuce (leaf and head).

Total residues in trials approximating GAP of Brazil (0.4 kg ai/ha, PHI 7 days) were: < 0.04 (4) mg/kg.

Total residues from Europe approximating GAP of Portugal and following a single application to weeds at 0.75 kg ai/ha prior to planting the crop were: < 0.05 (7), 0.10 and 0.29 mg/kg.

The Meeting used the data from Europe to estimate an STMR of 0.05 mg/kg, an HR of 0.29 mg/kg and a maximum residue level of 0.4 mg/kg for both leaf lettuce and head lettuce. The Meeting noted that for lettuce the IESTI accounts for 180% of the ARfD. No suitable alternative GAP was identified that would resolve the intake concern.

Legume vegetables

Common bean (pre-crop emergence or application to weeds)

Glufosinate-ammonium is permitted to be used as a spray directed to weeds (Portugal 0.75 kg ai/ha, no PHI required; Germany 1 kg ai/ha, using a screen to protect the crop PHI 14 days; France 0.75 kg ai/ha no PHI required) and also as a spray to control weeds prior to crop emergence (the Netherlands 0.6 kg ai/ha, no PHI required).

Total residues in beans harvested immature (pods and/or immature seeds) from trials that did not use a screen to protect the crop from accidental contamination but otherwise complied with German GAP (1.0 kg ai/ha) were (n = 7): < 0.05 (7) mg/kg. If a protective screen were used during application of glufosinate-ammonium, lower total residues would be expected.

The Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.05 mg/kg and a maximum residue level of 0.05* mg/kg for common bean (pods and/or immature seeds) confirming its previous recommendation of 0.05 * mg/kg.

Pulses

Common beans (dry) (pre-harvest desiccation)

Residue data from trials in beans were made available from Brazil for pre-harvest desiccation use. The use pattern in Brazil is 0.4 kg ai/ha with a PHI of 5 days. The UK use is for pre-harvest desiccation at 0.45 kg ai/ha with a 7 day PHI.

In seven trials conducted in Brazil approximating Brazil GAP total residues were < 0.04 (4) and < 0.05 (3) mg/kg.

Common beans (dry) (directed application to weeds)

Directed sprays in the Portugal are permitted at up to 0.75 kg ai/ha with no PHI required and in Germany at up to 1 kg ai/ha using spray shields with a 14 day PHI. In eight trials conducted in Germany, total residues following a directed application at 1 kg ai/ha and after a 14 day PHI were: < 0.05 (8). In eight trials in Europe, total residues following two directed sprays at 0.75 kg ai/ha residues were: < 0.01 (8) at 62 to 122 days after the last application.

Rotational crop metabolism studies gave detectable residues suggesting the STMR should not be zero.

Residues were less than the limit of quantification following both pre-harvest desiccation and directed sprays to weeds. Utilising the dataset from Brazil, the Meeting estimated an STMR of 0.04 mg/kg and a maximum residue level of 0.05 mg/kg for common beans, dry to replace its previous recommendation of 2 mg/kg.

Soya beans, tolerant

The Meeting received field trials performed in the USA involving glufosinate tolerant soya beans. GAP for USA is for (1) one application pre-planting or pre-emergence at 0.59–0.74 kg ai/ha with additional applications from post-emergence to the early bloom growth stage at 0.45–0.59 kg ai/ha with a maximum seasonal rate of 1.3 kg ai/ha/year or (2) post-emergence only with applications from post-emergence to the early bloom growth stage at 0.41–0.50 kg ai/ha with a maximum seasonal rate of 0.91 kg ai/ha/year. The PHI is 70 days. Post-emergent application leads to higher residues. In trials approximating critical GAP in the USA total residues in soya bean seeds were (n = 24): 0.22, 0.32, 0.39, 0.43, 0.51, 0.51, 0.56, 0.68, 0.70, 0.71, 0.78, 0.81, 0.84, 0.89, 0.96, 1.0, 1.1, 1.2, 1.2, 1.3, 1.3, 1.6, 1.7 and 1.9 mg/kg.

The Meeting estimated an STMR of 0.825 mg/kg and a maximum residue level of 3 mg/kg for soya bean, dry to replace its previous recommendation of 2 mg/kg.

Carrots (pre-crop emergence)

In Canada glufosinate-ammonium is approved for pre-emergent weed control in carrot crops (GAP: 0.41–0.75 kg ai/ha, PHI not required). The Netherlands permits the pre-crop emergence use of glufosinate-ammonium for weed control at a rate of 0.6 kg ai/ha and France allows bare soil cultivation at 0.75 kg ai/ha. In seventeen trials conducted in Europe with pre-crop emergence application at 0.6–0.75 kg ai/ha residues were: < 0.05 (17) mg/kg. Confined rotation crop studies suggest the residue is not zero.

The Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.05 mg/kg and a maximum residue level of 0.05* mg/kg for carrots to confirm its previous recommendation of 0.05* mg/kg.

Potato (pre-harvest desiccation)

Glufosinate-ammonium is approved for pre-crop emergence or pre-harvest desiccation of potato crops in various countries. Use of glufosinate-ammonium prior to crop emergence does not lead to significant residues compared to use for pre-harvest desiccation. The Meeting agreed to use trials involving pre-harvest desiccation, with or without an application prior to crop emergence, to estimate

a maximum residue level. Pre-harvest desiccation use-patterns approved in various countries include Brazil (GAP 0.4 kg ai/ha, PHI 10 days), France (GAP 2 × 0.38 kg ai/ha, PHI 14 days), Germany (GAP: 0.5 kg ai/ha at BBCH 90, PHI 14 days), Mexico (GAP 0.6 kg ai/ha), the Netherlands (GAP: 0.45 kg ai/ha or 0.38 kg ai/ha if crop is flailed), Portugal (GAP: 0.45 kg ai/ha, PHI 14 days), the UK (GAP:0.45 kg ai/ha, PHI 7 days) and the USA (GAP:0.42 kg ai/ha, PHI 9 days).

In trials conducted according to the GAP of Brazil total residues were: < 0.04 (4), < 0.05 (3) mg/kg.

In residue trials involving pre-harvest desiccation and approximating GAP of France total residues were: < 0.01, < 0.05 (6), 0.07, 0.08, 0.09, 0.11, 0.15, 0.16 (2) and 0.22 mg/kg.

In residue trials involving pre-harvest desiccation and approximating GAP of Germany total residues were: < 0.01 (3), 0.01, < 0.05, 0.06, 0.12, 0.21, 0.27 and 0.34 mg/kg.

In residue trials involving pre-harvest desiccation and approximating GAP of USA total residues were: < 0.05 (4), 0.06 (2), 0.1 (2), 0.11, 0.12 (2), 0.14, 0.16, 0.18, 0.24, 0.26, 0.28, 0.32, 0.38 and 0.62 mg/kg.

Using the residue data from the USA, the Meeting estimated an STMR of 0.12 mg/kg, an HR of 0.62 mg/kg and a maximum residue level of 0.8 mg/kg. The Meeting noted that for potatoes the IESTI accounts for 320% of the ARfD.

An alternative GAP was available from Brazil for pre-harvest desiccation at an application rate of 0.4 kg ai/ha with a PHI of 10 days. Total residues were < 0.04 (4), < 0.05 (3) mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg for potatoes and noted in estimating STMR and HR values of 0.05 and 0.05 mg/kg respectively that occasional finite residues are expected.

Sugar beet, tolerant

In the USA glufosinate-ammonium is approved for use on glufosinate tolerant sugar beet. GAP for USA is for either (i) a pre-planting or pre-emergence application at 0.59–0.74 kg ai/ha or (ii) post-emergence application from post-emergence from the cotyledon stage up to the 10-leaf stage (BBCH 19) at 0.29–0.61 kg ai/ha with a maximum seasonal rate of 1.2 kg ai/ha/year and a PHI of 60 days. Post-emergent application leads to higher residues. In trials approximating critical GAP in the USA total residues in sugar beet roots were (n = 13): 0.06, 0.11, 0.14, 0.20, 0.27, 0.27, 0.28, 0.31, 0.39, 0.42, 0.54, 0.62 and 0.67 mg/kg.

The Meeting estimated an STMR of 0.28 mg/kg and a maximum residue level of 1.5 mg/kg for sugar beet to replace its previous recommendation of 0.05 (*) mg/kg.

Asparagus (pre-crop emergence)

Trials were available following application pre-crop emergence for weed control in asparagus. In Canada pre-crop emergence application is permitted with an application rate of 0.4–0.75 kg ai/ha with a PHI not required while the Netherlands permits pre-emergence application to crops at 0.6 kg ai/ha with application no later than 3 days before crop emergence (PHI 3 days). GAP in Germany is application at prior to crop emergence at 0.6 kg ai/ha with no PHI required.

The trials were assessed against the GAP of Canada providing total residues of 0.01, < 0.05 (6), 0.16 and 0.27 mg/kg.

The Meeting estimated an STMR of 0.05 mg/kg, an HR of 0.27 mg/kg and a maximum residue level of 0.4 mg/kg for asparagus to replace its previous recommendation of 0.05* mg/kg.

Maize, tolerant

The Meeting received field trials performed in the USA involving glufosinate tolerant maize. GAP for USA is for (i) pre-planting or pre-emergence application at 0.59–0.74 kg ai/ha with a maximum seasonal rate of 0.74 kg ai/ha or (ii) post-emergence application from post-emergence until corn is 61 cm tall or in the V-7 growth stage (i.e., 7 developed collars) at 0.41–0.5 kg ai/ha with a maximum

seasonal rate of 0.91 kg ai/ha/year and a PHI of 70 days. Post-emergent application leads to higher residues. In trials approximating critical GAP in the USA total residues in maize grain were (n = 32): < 0.05 (27), 0.05, 0.06 and 0.07 (3) mg/kg.

The Meeting estimated an STMR of 0.05 mg/kg and a maximum residue level of 0.1 mg/kg for maize confirming its previous recommendation of 0.1 mg/kg.

Rice, tolerant

The Meeting received field trials performed in the USA involving glufosinate tolerant rice. GAP for USA is for (i) pre-planting or pre-emergence application at 0.59–0.74 kg ai/ha with a maximum seasonal rate of 0.74 kg ai/ha or (ii) applications post-emergence from the 1-leaf stage through the mid-tillering stage of crop development at 0.41–0.5 kg ai/ha with a maximum seasonal rate of 0.91 kg ai/ha/year and a PHI of 70 days. Post-emergent application leads to higher residues. The Meeting considered the growth stage at last application to be an important factor in considering whether or not trials were conducted according to critical GAP. In trials approximating critical GAP in the USA total residues in rice grain (with husk) were (n = 20): < 0.05 (5), 0.05, 0.07, 0.07, 0.08, 0.08, 0.10, 0.10, 0.14, 0.15, 0.17, 0.20, 0.35, 0.47, 0.51 and 0.73 mg/kg.

The Meeting estimated an STMR of 0.09 mg/kg and a maximum residue level of 0.9 mg/kg for rice.

Tree nuts (application to weeds)

Application to tree nuts is for weed control in the orchard including under the tree canopy. The use pattern in the USA is application to weeds at 0.84–1.68 kg ai/ha with a maximum seasonal rate of 5.0 kg ai/ha and a PHI of 14 days. Application to tree nuts in Australia is at 0.2–1.0 kg ai/ha and in the UK at 0.45–0.75 kg ai/ha, both with a PHI not required. In trials approximating GAP, total residues in tree nuts were:

Almonds: < 0.05, < 0.05, < 0.05, 0.07 mg/kg (GAP of USA)

Hazelnut: < 0.05, < 0.05, < 0.05 mg/kg (two times GAP of UK)

Macadamia: < 0.06, < 0.12 mg/kg (GAP of Australia)

Pecan: < 0.05, < 0.05, < 0.05 mg/kg (GAP of USA)

Walnut: < 0.05, < 0.05, < 0.05, < 0.05, < 0.05 mg/kg (GAP of USA).

Residues for the sum of glufosinate and NAG were: Almonds: < 0.05 (4) mg/kg (GAP of USA), Hazelnut: < 0.05 (3) mg/kg (two times GAP of the UK), Macadamia: < 0.05, < 0.09 mg/kg (GAP of Australia), Pecan: < 0.05 (3) mg/kg (GAP of USA) and Walnut: < 0.05 (5) mg/kg (GAP of the USA).

The Meeting considered the four almond, three pecan, five walnut, two macadamia and three hazelnut trials as a group. The Meeting estimated an STMR of 0.05 mg/kg and a maximum residue level of 0.1 mg/kg for tree nuts confirming its previous recommendation.

Cotton seed, tolerant

The Meeting received field trials performed in the USA involving glufosinate tolerant cotton. GAP for USA is for (i) pre-planting or pre-emergence application at 0.59 kg ai/ha and post-emergence use from emergence up to the early bloom stage at 0.45–0.59 kg ai/ha with a maximum seasonal rate of 1.8 kg ai/ha/year or (ii) pre-planting or pre-emergence application at 0.561–0.88 kg ai/ha and post-emergence use from emergence up to the early bloom stage at 0.45–0.59 kg ai/ha with a maximum seasonal rate of 1.5 kg ai/ha/year or (iii) post-emergence use from emergence up to the early bloom stage at 0.29–0.58 kg ai/ha with a maximum seasonal rate of 1.2 kg ai/ha/year. The PHI is 70 days. Post-emergent application leads to higher residues. In trials approximating critical GAP in the USA total residues in cotton seed were (n = 14): 0.15, 0.24, 0.30, 0.30, 0.34, 0.57, 0.63, 0.78, 1.1, 1.1, 1.3, 1.7, 2.3 and 3.2 mg/kg.

The Meeting estimated an STMR of 0.705 mg/kg and a maximum residue level of 5 mg/kg for cotton seed.

Rape seed, conventional (pre-harvest desiccation) and tolerant

Glufosinate-ammonium is approved for pre-harvest desiccation of conventional rape in Germany (GAP: 0.5 kg ai/ha, PHI 14 days) and the UK (GAP: 0.45 kg ai/ha, PHI 7 days). Total residues in rape seeds from trials conducted in Germany approximating German GAP were (n = 12): < 0.05, 0.15, 0.19, < 0.2, < 0.2, < 0.2, 0.25, 0.25, 0.25, 0.26, 0.63 and 0.76 mg/kg.

The Meeting received field trials performed in the USA involving glufosinate tolerant rape. GAP for USA is for (i) pre-planting or pre-emergence application at 0.59–0.74 kg ai/ha with a maximum seasonal rate of 0.74 kg ai/ha or (ii) application post-emergence from the cotyledon stage up to the early bolting stage of crop development at 0.47–0.5 kg ai/ha with a maximum seasonal rate of 0.99 kg ai/ha/year and a PHI of 65 days. Post-emergent application leads to higher residues. In trials approximating GAP in the USA total residues in rape seeds were (n = 5): < 0.05, 0.06, 0.19, 0.22 and 7.9 mg/kg.

The Meeting considered five trials inadequate to estimate a maximum residue level for rape when glufosinate-ammonium is used on tolerant crops and decided to use the trials for pre-harvest desiccation of conventional crops to estimate an STMR of 0.225 mg/kg and a maximum residue level of 1.5 mg/kg for rape seed replacing its previous recommendation of 5 mg/kg.

Sunflower seed (pre-harvest desiccation)

Glufosinate–ammonium is approved for pre-harvest desiccation of sunflowers in Germany (GAP: 0.5 kg ai/ha, PHI 14 days). Total residues in trials from Germany approximating German GAP were: 0.43, 0.79, 1.2 and 2.3 mg/kg (all at 0.6 kg ai/ha). Additional trials from Hungary had total residues: 0.05, 0.25, 0.38, 0.27 and 0.46 mg/kg (all at rates 0.34–0.36 kg ai/ha)

The Meeting noted that the number of trials available from Germany is inadequate for the purpose of estimating a maximum residue level and the trials from Hungary do not match a relevant GAP. However, as the application of glufosinate-ammonium is for desiccation, the Meeting considered it valid to scale the residues in both sets of trials to the German GAP application rate. Total residues (scaled to an application rate of 0.5 kg ai/ha) in sunflower were (n = 9): 0.07, 0.36, 0.36, 0.40, 0.53, 0.66, 0.68, 1.0 and 1.9 mg/kg.

The Meeting estimated an STMR of 0.53 mg/kg and a maximum residue level of 3 mg/kg for sunflower seed replacing its previous recommendation of 5 mg/kg.

Coffee beans (application to weeds)

In two trials from Brazil residues in coffee beans were < 0.04 (2). The Meeting considered two trials inadequate to estimate a maximum residue level. However, the Meeting considered there is sufficient other evidence, including from other fruiting trees and shrubs, to conclude the use pattern should not lead to residues above the LOQ although occasional residues of MPP through uptake from soil may occur and decided to estimate an STMR of 0.04 mg/kg and a maximum residue level of 0.1 mg/kg for coffee beans.

Animal feeds

Sweet corn forage, tolerant

The current Meeting received trials on glufosinate tolerant sweet corn forage and stover performed in USA. No GAP for the USA was available.

Bean forage and fodder (Common bean) (pre-crop emergence or application to weeds in crop)

Residue levels occurring in shoots and green material of conventional beans were evaluated. Glufosinate-ammonium is permitted to be used as a spray directed to weeds growing in bean crops

(Portugal 0.75 kg ai/ha, no grazing restriction; Germany 1 kg ai/ha, using a screen to protect the crop, no grazing restriction; France 0.75 kg ai/ha no grazing restriction) and also as a spray to control weeds prior to crop emergence (the Netherlands 0.6 kg ai/ha, no grazing restriction).

In considering the available trials it was agreed that shoots at 14 or more days after application at beginning of flowering would be representative of forage. Total residues approximating GAP of France and Portugal (0.75 kg ai/ha) were (n = 8): < 0.05, < 0.05, < 0.05, < 0.05, < 0.05, 0.06, 0.08 and 0.19 mg/kg. The Meeting estimated a median and a highest residue level of 0.05 mg/kg and 0.19 mg/kg respectively for bean forage (both on an as received basis).

Residue levels occurring in straw of conventional beans following directed application use were also available to the Meeting. In eight trials conducted in Germany, total residues following a directed application at 1 kg ai/ha with harvest 39 to 65 days after application were: < 0.05 (4), < 0.1, 0.15, 0.22 and 0.63 mg/kg (on an as received basis). The Meeting estimated a median residue of 0.075 mg/kg, a highest residue of 0.63 mg/kg (both on an as received basis) and a maximum residue level of 1 mg/kg for bean fodder (on a dry weight basis).

Miscellaneous fodder and forage crops

Sugar beet tops, tolerant

The Meeting received trials on glufosinate tolerant sugar beet) performed in the USA.

GAP for USA is for either (i) a pre-planting or pre-emergence application at 0.59–0.74 kg ai/ha or (ii) post-emergence application from post-emergence from the cotyledon stage up to the 10-leaf stage (BBCH 19) at 0.29–0.61 kg ai/ha with a maximum seasonal rate of 1.2 kg ai/ha/year and a PHI of 60 days. There is a restriction on the use of treated glufosinate tolerant sugar beet tops for livestock feed: Do not graze the treated crop or cut for hay. The Meeting did not receive GAP relevant to the use of sugar beet tops for livestock feed.

Straw, forage and fodder of cereal grains and grasses

Maize forage and stover, tolerant

For the current evaluation the Meeting received field trials involving glufosinate tolerant maize performed in USA. GAP for USA is for (i) pre-planting or pre-emergence application at 0.59–0.74 kg ai/ha with a maximum seasonal rate of 0.74 kg ai/ha or (ii) post-emergence application from post-emergence until corn is 61 cm tall or in the V-7 growth stage, (i.e., 7 developed collars) at 0.41–0.5 kg ai/ha with a maximum seasonal rate of 0.91 kg ai/ha/year and a PHI of 60 days for forage and 70 days for fodder. Post-emergent application leads to higher residues.

Total residues in maize forage from trials approximating USA critical GAP were (n = 13): < 0.05, 0.06, 0.33, 0.50, 0.53, 0.54, 0.78, 0.82, 0.89, 1.1, 1.2, 1.5 and 1.6 mg/kg (fresh weight basis). The Meeting estimated median and highest residues of 0.78 and 1.6 mg/kg respectively.

Total residues in maize silage from trials approximating critical GAP in the USA were: < 0.05, 0.11, 0.12, 0.15, 0.32, 0.34, 0.36, 0.40, 0.43, 0.90, 1.2, 1.2, 1.6, 1.7 and 2.1 mg/kg (fresh weight basis). The Meeting estimated median and highest residues of 0.40 and 2.1 mg/kg respectively, both on a fresh weight basis.

Total residues in maize fodder/stover (fresh weight basis) from trials approximating USA GAP were (n = 31): < 0.05, 0.09, 0.12, 0.13, 0.16, 0.17, 0.18, 0.24, 0.33, 0.41, 0.50, 0.53, 0.64, 0.68, 0.69, 0.72, 0.78, 1.3, 1.4, 1.4, 1.4, 1.4, 1.5, 1.7, 1.8, 1.9, 1.9, 2.8, 2.9 and 5.3 mg/kg. The Meeting estimated median and highest residues of 0.72 and 5.3 mg/kg respectively, both on a fresh weight basis and a maximum residue level of 8 mg/kg for maize fodder.

Rice, tolerant

The Meeting received field trials performed in the USA involving glufosinate tolerant rice. GAP for USA is for (i) pre-planting or pre-emergence application at 0.59–0.74 kg ai/ha with a maximum

seasonal rate of 0.74 kg ai/ha or (ii) application post-emergence from the 1-leaf stage through the mid-tillering stage of crop development at 0.41–0.5 kg ai/ha with a maximum seasonal rate of 0.91 kg ai/ha/year and a PHI of 70 days. Post-emergent application leads to higher residues.

In the trials matching the critical GAP, glufosinate total residues in rice straw on an as received basis were (n = 21): 0.05 (2), 0.06, 0.11, 0.12 (2), 0.14, 0.16, 0.24, 0.25, 0.26, 0.29, 0.30, 0.34, 0.35, 0.37, 0.54, 0.62, 0.63, 0.93 and 1.3 mg/kg.

The Meeting estimated median and highest residues of 0.26 and 1.3 mg/kg respectively together with a maximum residue level of 2 mg/kg for rice straw.

Cotton (tolerant) gin trash, tolerant

The Meeting received field trials performed in the USA involving glufosinate tolerant cotton. GAP in the USA is (i) pre-planting or pre-emergence application at 0.59 kg ai/ha and post-emergence use from emergence up to the early bloom stage at 0.45–0.59 kg ai/ha with a maximum seasonal rate of 1.8 kg ai/ha/year or (ii) pre-planting or pre-emergence application at 0.561–0.88 kg ai/ha and post-emergence use from emergence up to the early bloom stage at 0.45–0.59 kg ai/ha with a maximum seasonal rate of 1.5 kg ai/ha/year or (iii) post-emergence use from emergence up to the early bloom stage at 0.29–0.58 kg ai/ha with a maximum seasonal rate of 1.2 kg ai/ha/year. Post-emergent application leads to higher residues. In trials approximating critical GAP in the USA total residues in cotton gin trash were (n = 7): 0.62, 0.65, 1.1, 1.5, 3.4, 4.0 and 7.4 mg/kg (on an as received basis).

The Meeting estimated a median residue of 1.5 mg/kg for cotton gin trash.

Rotational crop residues

Soil residues of glufosinate, MPP and NAG are not persistent and the maximum single spray application rate for weeds in non-permanent crops of 1 kg ai/ha can be used (after adjusting for likely interception of spray by crops and weeds) as a basis for estimating likely residues in soil that might transfer to rotational (follow) crops. If it is assumed weeds and crop present at the time of application intercept 50% of the spray, the amount reaching the soil would be equivalent to an application rate to bare soil of 0.5 kg ai/ha. Trials on residues in follow crops were made available to the Meeting. At plant-back intervals of 69 to 174 days after application at 0.6 to 0.9 kg ai/ha to cotton, residues in follow crops (mustard greens, turnips) were < LOQ except for some wheat forage, straw and hay samples for which total residues ranged from < 0.05 to 0.14 mg/kg. Residues were also < 0.05 mg/kg in wheat commodities grown following a potato crop treated at 0.45 kg ai/ha and at plant back intervals of 30 days.

In the confined rotation crop study where crops were rotated into soil previously treated with ^{14}C-glufosinate-ammonium at the equivalent of 1 kg ai/ha, total residues (sum of glufosinate, MPP and NAG) were < 0.05 mg/kg in radish tops and lettuce at a plant back interval of 28 days and 0.15, 0.38 and 0.12 mg/kg in wheat forage, straw and grain respectively. At longer plant back intervals residues were all < 0.05 mg/kg.

The Meeting considered residues in rotational crops above the LOQ would be unlikely.

Fate of residues during processing

The Meeting received information on the nature of residues under simulated processing condition on the fate of incurred residues of glufosinate during the processing of oranges, plums, grapes, olives, potatoes, sugar beet, soy bean, oilseed rape/canola, cotton seed, sunflower seed, maize and rice. A study of the nature of the residue of glufosinate, NAG and MPP under simulated processing conditions (pasteurization, baking/brewing/boiling, sterilization) showed glufosinate, MPP and NAG are stable.

Summary of selected processing factors for glufosinate

Raw commodity	Processed commodity	Individual PF	Best estimate PF	$STMR_{RAC}$ (mg/kg)	$STMR_{RAC}$ × PF (mg/kg)
Orange	Juice	0.71	0.71	0.05	0.036

Raw commodity	Processed commodity	Individual PF	Best estimate PF	$STMR_{RAC}$ (mg/kg)	$STMR_{RAC}$ × PF (mg/kg)
	Dried peel / pulp	2.21	2.21		0.11
	Molasses	2.65	2.65		0.13
	Oil	< 0.13	< 0.13		< 0.0065
Plum	Dried fruit	1.79	1.79	0.05	0.090
Olive	Oil	< 0.65	< 0.65	0.05	< 0.0325
Potato	Chips	2.2	2.2	0.05	0.11
	Flakes	1.78 2.77 2.91 3.43 3.06	2.91		0.146
	Crisps	1.61 1.61 1.70 2.12	1.655		0.083
	French fries	0.89 1.18 1.30 1.48	1.24		0.062
	Boiled potatoes	0.47 0.60 0.79 0.99	0.695		0.035
	Fried potatoes	0.95 1.48 1.78 2.01	1.63		0.082
	Baked potatoes	1.05 1.26 1.29 1.54	1.275		0.064
Sugar beet	Dried pulp	0.59	0.59	0.28	0.168
	Molasses	3.70 4.94 6.32 6.82	5.63		1.568
	Raw or refined sugar	< 0.08 < 0.10 < 0.29 < 0.91	< 0.195		0.056
Soya bean	Aspirated grain fraction	2.73 8.89	5.81	0.825	4.78
	Hulls	3.15, 11.4	7.275		6.02
	Meal	1.22	1.22		0.99
	Oil	< 0.04 < 0.74	< 0.74		< 0.61
Rape/canola	Meal	1.74, 1.94, 2.44, 2.55	2.19	0.225	0.495
	Oil	< 0.13 < 0.22 < 0.48 < 0.94 < 0.94	< 0.48		< 0.108
Cottonseed	Hulls	1.16	1.16	0.705	0.818
	Meal	1.25	1.25		0.881
	Oil	< 0.02	< 0.02		< 0.014
Sunflower seed	Oil	< 0.03 < 0.07 < 0.08	< 0.07	0.53	< 0.037
Maize	Aspirated grain fraction	8.85 12.06	10.455	0.05	0.52275
Rice	Hull	1.85 2.29	2.07	0.09	0.1863
	Bran	0.74 0.87	0.805		0.0724
	Polished grain	0.60 0.94	0.77		0.0693

Residues are not expected in oils obtained from treated crops and did not concentrate in bran.

Residues in sugar beet molasses were much higher than in sugar beets. The Meeting estimated a maximum residue level of 8 mg/kg based on the recommended maximum residue level for sugar beet (1.5 mg/kg) and a processing factor of 5.63.

Residues in prunes were also higher than in fresh plums and the Meeting estimated a maximum residue level of 0.3 mg/kg for dried prunes based on the recommended maximum residue level for stone fruit (0.15 mg/kg) and a processing factor of 1.79.

Residues in animal commodities

Farm animal feeding studies

The Meeting received information on the residue levels arising in tissues and milk when dairy cows were fed a diet containing glufosinate and MPP at total dietary levels of 4, 12 and 40 ppm for the sum of glufosinate and MPP expressed as glufosinate free acid for 28 consecutive days. The ratio of glufosinate to MPP in the feed was 3:1. Apart from milk of a single dosed cow on days 0, 1 (0.03 mg/kg) and 9 (0.02 mg/kg) and a control milk sample (0.03 mg/kg), no residues were detected in milk (LOQ of 0.02 mg/kg) samples analysed at all dose levels and time intervals. The highest total residues (mean in brackets) in liver, kidney, fat and muscle from the highest dose animals were 10.7 (9.0), 7.5 (5.4), 0.16 (0.10) and < 0.05 (< 0.05) mg/kg respectively.

In an additional study dairy cows were fed glufosinate and NAG at total dietary levels of 9.1, 27.3 and 91 ppm for the sum of glufosinate and NAG expressed as glufosinate free acid for 28 consecutive days. The ratio of glufosinate to NAG in the dose was 1:5.5. Milk residues for the highest

dose group were 0.07 mg/kg. Milk from one animal had much higher residues on two consecutive days that coincided with a dramatic reduction in feed consumption. These results were excluded from the analysis. The highest total residues (mean in brackets) in liver, kidney, fat and muscle from the highest dose animals were 0.13 (0.11), 0.29 (0.27), < 0.05 (< 0.05) and < 0.05 (< 0.05) mg/kg respectively.

The Meeting also received information on the residue levels arising in tissues and eggs, when laying hens were fed a diet containing glufosinate and MPP at total dietary levels of 4.5, 13.5 and 45 ppm glufosinate and MPP (both as glufosinate free acid) for 28 consecutive days. The ratio of glufosinate to MPP in the feed was 3.5:1. Residues in eggs for the highest feed group reached a maximum of 0.07 mg/kg at between day 7 and 13 of exposure. The mean total residues in liver, fat and muscle from the highest dose animals were < 0.10, 8.0, < 0.05 and < 0.05 mg/kg respectively.

In an additional study laying hens were dosed with glufosinate and NAG at levels equivalent to 0.44, 1.3 and 4.4 ppm in the feed for the sum of glufosinate and NAG expressed as glufosinate free acid for 28 consecutive days. The ratio of glufosinate to NAG in the dose was 1:5.3. Egg residues were < 0.05 mg/kg for all dose groups. Similarly, residues in liver, kidney, fat and muscle were all < LOQ (< 0.05 mg/kg for muscle, kidney, skin and fat and < 0.1 mg/kg for liver).

Animal commodity maximum residue levels

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

Potential cattle feed items include: soya bean hulls, cotton gin by-products, sugar beet molasses, cottonseed meal, maize forage/silage, soya bean meal, soya bean seed, potato culls, maize aspirated grain fractions, rice straw.

Potential poultry feed items include: soya bean hulls, cotton seed meal, maize forage/silage, soya bean meal, soya bean seed, potato culls, rice grain, rice bran/pollard, bean seed, maize grain.

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

	US-Canada		EU		Australia		Japan	
	max	mean	Max	mean	max	Mean	max	Mean
Beef cattle	2.4	1.8	4.7[a]	2.4[c]	4.3	1.8	1.3	0.9
Dairy cattle	2.7	1.4	4.0	2.3[d]	4.4[b]	1.8	2.5	1.5
Poultry Broiler	0.5	0.5	1.2	1.2[f]	0.7	0.7	0.4	0.4
Poultry Layer	0.5	0.5	1.4[e]	0.9[g]	0.7	0.7	0.4	0.4

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

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

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

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

[e] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs.

[f] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat.

[g] Highest mean poultry dietary burden suitable for STMR estimates for poultry eggs.

Animal commodity maximum residue levels

Two lactating dairy cow feeding studies were available to the Meeting. The first addressed feed consisting of ingredients selected from conventional crops and therefore with residues dominated by glufosinate (crop desiccation uses) and MPP (weed control uses). Estimates of highest and median residues made using the first feeding study are relevant if glufosinate is the major component of the total glufosinate residue in the feed. A review of metabolism and residue studies available for feed items that contribute most to the dietary burden for dairy and beef cattle showed that glufosinate is the

major component of the total residues in livestock feed. Therefore the first feeding study is used to estimate residues in meat, edible offal and milk.

The calculations used to estimate highest total residues for use in estimating maximum residue levels, STMR and HR values are shown below.

Glufosinate + MPP feeding study	Feed level	Residues	Feed level	Residues (mg/kg) in			
	(ppm) for milk residues	(mg/kg) in milk	(ppm) for tissue residues	Muscle	Liver	Kidney	Fat
Maximum residue level beef or dairy cattle							
Feeding study [a]	4.0 12	< 0.02 0.02	4.0 12	< 0.05 < 0.05	1.63 4.2	0.41 2.0	0.06 0.08
Dietary burden and high residue	4.4	< 0.02	4.7	< 0.05	1.85	0.55	0.062
STMR beef or dairy cattle							
Feeding study [b]	4.0	< 0.02	4.0	< 0.05	1.18	0.38	0.05
Dietary burden and residue estimate	2.3	< 0.012	2.4	< 0.03	0.708	0.228	0.03

[a] highest residues for tissues and mean residues for milk

[b] mean residues for tissues and mean residues for milk

The Meeting estimated the following STMR values: milk 0.012 mg/kg; muscle 0.03 mg/kg; edible offal 0.708 mg/kg for liver and 0.228 mg/kg for kidney and fat 0.03 mg/kg. The following HR values are also estimated: milk 0.02 mg/kg; muscle 0.05 mg/kg; edible offal 1.85 mg/kg for liver and 0.55 mg/kg for kidney and fat 0.062 mg/kg.

The Meeting estimated the following maximum residue levels: milk 0.02 mg/kg; meat (mammalian except marine mammals) 0.1 mg/kg and edible offal 3 mg/kg to replace its previous recommendations of: milk 0.02* mg/kg; meat (mammalian except marine mammals) 0.05* mg/kg and edible offal 0.1* mg/kg. The Meeting noted that for cattle liver the IESTI accounts for 140–170% of the ARfD.

The corresponding calculations for poultry are provided below.

	Feed level	Residues	Feed level	Residues (mg/kg) in			
	(ppm) for egg residues	(mg/kg) in egg	(ppm) for tissue residues	Muscle	Liver	Skin	Fat
Maximum residue level broiler or laying hen							
Feeding study [a]	4.5	< 0.05	4.5	< 0.05	< 0.1	< 0.05	< 0.05
Dietary burden and residue estimate	1.4	< 0.016	1.4	< 0.016	< 0.031	< 0.016	< 0.016
STMR broiler or laying hen							
Feeding study [b]	4.5	< 0.05	4.5	< 0.05	< 0.1	< 0.05	< 0.05
Dietary burden and residue estimate	0.9	< 0.01	1.2	< 0.013	< 0.027	< 0.013	< 0.013

[a] highest residues for tissues and mean residues for egg

[b] mean residues for tissues and mean residues for egg

For poultry no residues are expected. The Meeting estimated the following maximum residue levels for poultry commodities: poultry meat 0.05* mg/kg; poultry edible offal 0.1* mg/kg and eggs 0.05* mg/kg.

The mean dietary burden of poultry is 0.9 ppm for layers and 1.4 ppm for broilers. The Meeting estimated the following STMR values: poultry meat 0 mg/kg; poultry fat 0 mg/kg; poultry edible offal (based on liver) 0 mg/kg and eggs 0 mg/kg.

RECOMMENDATIONS FURTHER WORK OR INFORMATION

On the basis of the data obtained from supervised residue 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 MRL and estimation of dietary intake (for animal and plant commodities): *sum of glufosinate, 3-[hydroxy(methyl)phosphinoyl]propionic acid (MPP) and N-acetyl-glufosinate (NAG), calculated as glufosinate (free acid)*

The residue is not fat soluble.

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
AM 0660	Almond hulls	W	0.5		
VS 0621	Asparagus	0.4	0.05 (*)	0.05	0.27
FI 0030	Assorted tropical and sub- tropical fruits - inedible peel (except banana)	W	0.05 (*)		
FI 0030	Assorted tropical and sub- tropical fruits - inedible peel (except banana and kiwifruit)	0.1		0.05	0.05
FT0026	Assorted tropical and sub- tropical fruits - edible peel	0.1		0.05	0.05
FI 0327	Banana	0.2	0.2	0.05	0.13
AL 0061	Bean fodder	1		0.075 fw	0.63 fw
FB 0018	Berries and other small fruits (except currants)	W	0.1	0.03	
FB 0020	Blueberries	0.1		0.05	0.06
VD 0523	Broad bean (dry)	W	2		
VR 0577	Carrot	0.05	0.05 (*)	0.05	0.05
FC 0001	Citrus fruits	0.05	0.1	0.05	0.05
VD 0526	Common bean (dry)	0.05	2	0.04	
SB 0716	Coffee beans	0.1		0.04	
VP 0526	Common bean (pods and/or immature seeds)	0.05 (*)	0.05 (*)	0.05	0.05
VL 0470	Corn salad	0.05	0.05 (*)	0.05	0.05
SO 0691	Cotton seed	5		0.705	
FB 0021	Currants, Black, Red, White	1	0.5	0.02	0.48
MO 0105	Edible offal (mammalian)	3	0.1 (*)	0.228 K 0.55 L	0.708 K 1.85 L
PE 0112	Eggs	0.05 (*)	0.05 (*)	0	0.02
FB 0268	Gooseberry	0.1		0.02	0.02
FB 0269	Grapes	0.15		0.02	0.12
FI 0341	Kiwifruit	0.6		0.05	0.37
VL 0482	Lettuce, Head	0.4		0.05	0.29
VL 0483	Lettuce, Leaf	0.4		0.05	0.29
GC 0645	Maize	0.1	0.1	0.05	
AS 0645	Maize fodder (dry)	8	10	0.72fw	5.3fw
AF 0645	Maize forage	W	5	0.78 fw	1.6 fw
MM 0095	Meat (from mammals other than marine mammals)	0.05	0.05 (*)	0.03 M 0.03 F	0.05 M 0.062 F
ML 0106	Milks	0.02 (*)	0.02 (*)	0.01	0.02
VA 0385	Onion, Bulb	0.05	0.05	0.05	0.05
VD 0072	Peas (dry)	W	3		
FP 0009	Pome fruits	0.1	0.05 (*)	0.05	0.08
VR 0589	Potato	0.1	0.5	0.05	0.05
PM 0110	Poultry meat	0.05 (*)	0.05 (*)	0	0.02
PO 0111	Poultry, Edible offal of	0.1 (*)	0.1 (*)	0	0.04
DF 0014	Prunes	0.3		0.09	
SO 0495	Rape seed	1.5	5	0.225	
OC 0495	Rape seed oil, Crude	0.05 (*)	0.05 (*)		
FB 0272	Raspberries, Red, Black	0.1		0.03	0.03

Commodity		Recommended MRL (mg/kg)		STMR or STMR-P (mg/kg)	HR, HR-P, highest residue (mg/kg)
CCN	Name	New	Previous		
GC 0349	Rice	0.9		0.09	
AS 0649	Rice straw and fodder, dry	2		0.26 fw	1.3 fw
VD 0541	Soya bean (dry)	3	2	0.825	
FS 0012	Stone fruits	0.15	0.05 (*)	0.05	0.08
FB 0275	Strawberry	0.3		0.02	0.15
VR 0596	Sugar beet	1.5	0.05 (*)	0.28	
DM 0596	Sugar beet molasses	8		1.24	
SO 0702	Sunflower seed	3	5	0.47	
OC 0702	Sunflower seed oil, crude	0.05 (*)	0.05 (*)		
TN 0085	Tree nuts	0.1	0.1	0.05	0.05

fw = fresh weight basis

Commodity		STMR or STMR-P (mg/kg)	highest residue (mg/kg)
CCN	Name		
AL 1030	Bean forage (green)	0.05 fw	0.19 fw
	Cotton gin trash	1.5	
	Maize aspirated grain fractions	0.52275	29
	Rape seed meal	0.4928	
AB 0596	Sugar beet pulp dry	0.168	

fw = fresh weight basis

DIETARY RISK ASSESSMENT

Long-term intake

The WHO Panel of the 2012 JMPR established an Acceptable Daily Intake (ADI) of 0–0.01 mg/kg bw for glufosinate.

The evaluation of glufosinate resulted in recommendations for maximum residue levels and STMR values for raw and processed commodities. Where data on consumption were available for the listed food commodities, dietary intakes were calculated for the 13 GEMS/Food Consumption Cluster Diets. The results are shown in Annex 3 of the 2012 JMPR Report.

The IEDIs in the thirteen Cluster Diets, based on the estimated STMRs were 6–10% of the maximum ADI (0.01 mg/kg bw). The Meeting concluded that the long-term intake of residues of glufosinate from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The WHO Panel of the 2012 JMPR established an Acute Reference Dose (ARfD) of 0.01 mg/kg bw for glufosinate.

For bananas, kiwifruit, lettuce, soya bean (dry) and cattle liver, the IESTI represented 110, 110, 180, 120 and 170% respectively of the ARfD of 0.01 mg/kg bw. The results are shown in Annex 4 of the 2012 JMPR Report. Since MPP represents the majority of the residue in bananas, kiwifruit, lettuce and cattle liver, and because MPP is of lower toxicity than glufosinate, these exceedances are unlikely to present a public health concern. Although the IESTI for soya beans represented 120% of the ARfD, MPP represents about 15% of the residues. The Meeting concluded that the short-term intake of residues of glufosinate resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

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