GLUFOSINATE-AMMONIUM (175)

EXPLANATION

Glufosinate-ammonium is a herbicide and desiccant. It was first evaluated for residues and toxicology by the 1991 JMPR and subsequently for residues in 1994.

Glufosinate-tolerant crops have now been developed with new use patterns necessitating revised MRLs. The manufacturer has provided many new reports for evaluation dealing with animal and plant metabolism, environmental fate in soil and water, methods of residue analysis, stability in stored analytical samples, supervised residue trials, animal transfer studies, fate of residues in processing and national residue limits.

The delegation of The Netherlands was requested by the 1996 CCPR to send their comments on the definition of the residue to the JMPR (ALINORM 97/24 para 73).

Information was provided to the Meeting by Germany, The Netherlands, Poland and the basic manufacturer.

METABOLISM AND ENVIRONMENTAL FATE

Information was made available on the metabolism and environmental fate of glufosinate in animals, crops, soils and water-sediment systems.

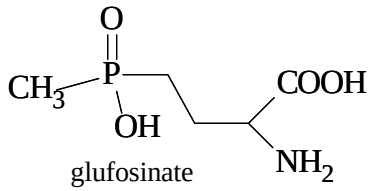
A variety of code numbers and abbreviations have been used for the metabolites and degradation products of glufosinate-ammonium. There are nine different code numbers for glufosinate itself, covering the salts and free acid, and the racemate and separate stereoisomers. Earlier publications used HOE numbers but they have recently been replaced by AEF, AEC or AE numbers. The names, structures and code numbers of glufosinate and its metabolites and degradation products are listed below.

In this evaluation “glufosinate” and the abbreviations NAG, MPP, etc. refer to the compounds without specifying their stereoisomerism or whether they are presents as salts or free acid. Where such extra information is important, e.g. because of molecular weight considerations, extra information will be given, e.g. “glufosinate, expressed as the free acid”.

Structures, names and code numbers of glufosinate, metabolites and degradation products

Glufosinate

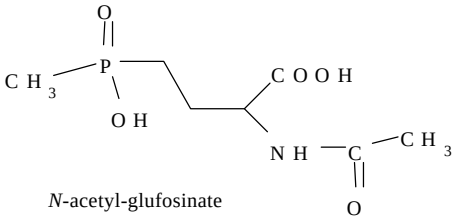
	racemate	L-isomer	R-isomer	MW
free acid	AE F035956	AE F057740	AE F090532	181.1
Ammonium salt	AE F039866	AE F058192	AE F093854	198.2
HCl salt	AE F035125	AE F057742	AE F057741	217.6



glufosinate

N-acetyl-glufosinate (NAG)

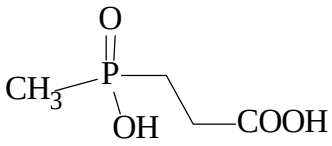
	racemate	L-isomer	R-isomer	MW
free acid	AE F085355	AE F099729	AE F124451	223.2
Disodium salt	AE F098412	AE F099730	AE F124450	267.2



N-acetyl-glufosinate

3-methylphosphinicopropionic acid (MPP)

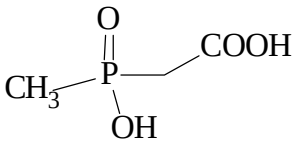
		MW
Free acid	AE F061517	152.1
Disodium salt	AE C527855	196.1



3-methyl-phosphinico-propionic acid

2-methylphosphinicoacetic acid (MPA)

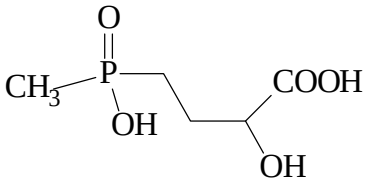
		MW
Free acid	AE F064619	138.1
Disodium salt	AE F159481	182.1



2-methyl-phosphinico-acetic acid

4-methylphosphinico-2-hydroxybutanoic acid (MHB)

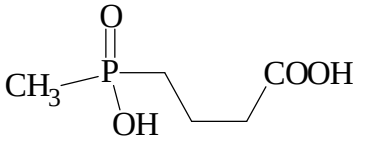
		MW
Free acid	AE F053705	182.1
Disodium salt	AE F042231	226.1



4-methyl-phosphinico-2-hydroxy-butanoic acid

4-methylphosphinicobutanoic acid (MPB)

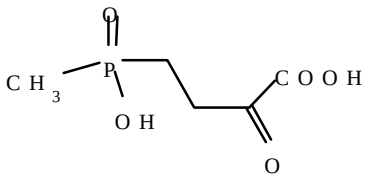
		MW
Free acid	AE F039046	166.1



4-methyl-phosphinico-butanoic acid

4-methylphosphinico-2-oxobutanoic acid

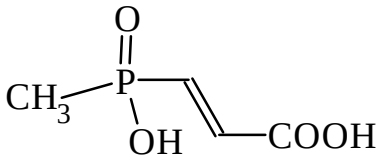
		MW
Free acid	AE F065594	180.1



4-methyl-phosphinico-2-oxo-butanoic acid

3-methylphosphinico-acrylic acid

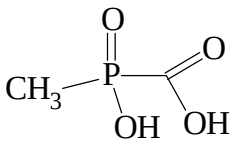
		MW
Free acid	AE 0015081	150.1



AE 0015081
3-methyl-phosphinico acrylic acid

Methylphosphinico-formic acid

		MW
Free acid	AE F130947	124.0



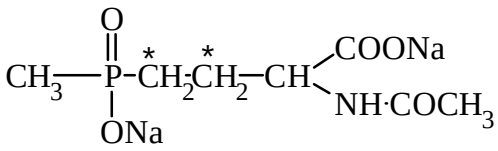
methylphosphinico-formic acid

Animal metabolism

Information on the metabolism of glufosinate-ammonium and NAG (*N*-acetyl-L-glufosinate) in laboratory rats, lactating goats and laying hens was reported. In summary, most of the administered dose of both compounds is rapidly excreted. NAG may be partially metabolized back to glufosinate.

Bremmer and Leist (1997) examined the possible conversion of NAG to glufosinate in rats. Up to 10% deacetylation occurred at a low dose of 3 mg/kg bw as shown by the occurrence of glufosinate in the faeces. The authors concluded however that most of the conversion was caused by bacteria in the colon and rectum although toxicity findings indicate partial bioavailability (Bremmer and Leist, 1998).

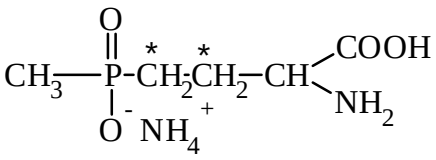
Kellner *et al.* (1993) showed that almost all the radiolabel was excreted in the faeces within 4 days when rats were dosed orally with single doses of [^{14}C]NAG disodium salt at 3 mg/kg body weight. The ^{14}C label was in positions 3 and 4.



Labelled AE F099730

Unchanged NAG was the main source of the radiolabel (85-89%) in faecal extracts from rats dosed orally with [^{14}C]NAG disodium salt at 1000 mg/kg body weight (Lauck-Birkel, 1995a). Small amounts (approx. 1%) of glufosinate were produced.

When [3,4-^{14}C]glufosinate-ammonium was administered in a single oral dose of 500 mg/kg bw to rats, 75% in males and 89% in females of the radiolabel was excreted in the faeces within 48 hours (Lauck-Birkel, 1995b) and 8-11% in the urine. Glufosinate was the principal labelled component in faecal extracts – 72% and 84% of the dose in males and females respectively.



Labelled glufosinate-ammonium

Lauck-Birkel (1996) identified the labelled compounds in urine and faeces from rats dosed orally with [3,4-^{14}C]glufosinate-ammonium at 2 mg/kg bw. The main compound in faecal extracts was glufosinate (77% of the dose); other identified components were NAG (7.5%), MHB (4.3%) and MPP (1.3%). The main components in the urine were glufosinate (4.3% of the dose) and MPP (0.8%).

Tissue, milk and excreta residues were measured in a lactating goat weighing 60 kg dosed orally twice daily for 4 consecutive days by capsule with [3,4-^{14}C]glufosinate at a rate equivalent to 101 ppm glufosinate-ammonium in the dry-weight diet and 3.0 mg/kg bw/day (Huang and Smith, 1995a). The feed intake was 1.8 kg/day. The animal was milked twice daily and slaughtered 15 hours after the final dose.

Most of the administered ^{14}C (69%) was excreted in the faeces with 2.9% in the urine and 11% in the GI tract with contents. Less than 0.1% and 0.02% of the dose was found in the tissues and milk respectively. The levels in the kidneys were higher than in other tissues (Table 1). Levels of ^{14}C reached a plateau in milk by day 2.

The parent compound was the main residue detected in the kidneys, liver and milk, with MPP forming a substantial part of the residue in the kidneys and liver (Table 2).

Table 1. Distribution of ^{14}C in the tissues and milk of a goat dosed twice daily for 4 days with [3,4-^{14}C]glufosinate (Huang and Smith, 1995a).

Sample	^{14}C as glufosinate-ammonium, mg/kg
Kidney	0.61
Liver	0.40
Muscle	0.007
Fat	0.004
Milk day 1, am and pm	0.003 0.009
Milk day 2, am and pm	0.016 0.020
Milk day 3, am and pm	0.022 0.020
Milk day 4, am and pm	0.020 0.014

Table 2. Compounds identified in the tissues and milk of a goat dosed twice daily for 4 days with [3,4-^{14}C]glufosinate (Huang and Smith, 1995a). Residue levels are expressed as glufosinate-ammonium equivalents.

Compound	Kidney		Liver		Milk	
	% of total ^{14}C	mg/kg	% of total ^{14}C	mg/kg	% of total ^{14}C	mg/kg
Glufosinate	49	0.30	53	0.21	49	0.010
NAG, L-isomer	4.2	0.026	not detected		2.2	<0.001
MPP	29	0.18	37	0.15	6.3	0.001
MPA	1.2	0.008	0.4	0.001	5.3	0.001

Tissue, milk and excreta residues were measured in a lactating goat weighing 36 kg dosed orally twice daily for 3 consecutive days by capsule with [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt at a rate equivalent to 84 ppm in the dry-weight diet or 3.0 mg/kg bw/day (Huang and Smith, 1995b). The feed intake was 1.4 kg/day. The animal was milked twice daily and slaughtered 16 hours after the final dose.

Most of the administered ^{14}C was excreted in the faeces (68%) with 7.3% in the urine and 19% in the GI tract with contents. NAG and glufosinate accounted for 52% and 34% of the ^{14}C in the faeces respectively.

Only 0.2% of the administered dose was found in the tissues and blood, with <0.1% in the milk. Levels in the kidneys were higher than in other tissues (Table 3). Levels of ^{14}C reached a plateau in milk by day 2. Glufosinate was the main residue detected in the kidneys, liver and milk, with NAG (the administered material) and MPP forming a substantial part of the residue in the kidneys and liver (Table 4).

Table 3. Distribution of ^{14}C in the tissues and milk of a goat dosed twice daily for 3 consecutive days with [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt (Huang and Smith, 1995b).

Tissue or milk	^{14}C as N-acetyl-L-glufosinate disodium salt, mg/kg
Kidneys	0.93
Liver	0.29
Muscle	0.007
Fat	<0.010
Milk day 1, am and pm	0.005 0.012
Milk day 2, am and pm	0.018 0.020
Milk day 3, am and pm	0.023 0.022

Table 4. Compounds identified in the tissues and milk of a goat dosed twice daily for 3 consecutive days with [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt (Huang and Smith, 1995b). Residue levels are expressed as *N*-acetyl-L-glufosinate disodium salt equivalents.

Compound	Kidneys		Liver		Milk	
	% of total ^{14}C	mg/kg	% of total ^{14}C	mg/kg	% of total ^{14}C	mg/kg
Glufosinate	40	0.37	33	0.095	40	0.009
NAG, L-isomer	32	0.30	19	0.054	9.2	0.002
MPP	20	0.19	21	0.060	14	0.003
MPA	1.6	0.015	2.0	0.006	4.8	0.001

Tissue, eggs and excreta residues were measured in 6 laying hens weighing 1.27-1.67 kg dosed orally twice daily for 14 consecutive days by capsule with [3,4-^{14}C]glufosinate-ammonium at a

rate equivalent to 25 ppm glufosinate-ammonium in the diet (it was not clear whether the feeding level was expressed on a fresh-weight or dry-weight basis) or 2.0 mg/kg bw/day (Huang and Smith, 1995c). The feed intake was 120 g/bird/day. Eggs were collected twice daily and the birds were slaughtered 16 hours after the final dose .

92% of the administered dose was excreted with 1.3% remaining in the GI tract. Glufosinate-ammonium accounted for 81% of the ^{14}C in the faeces.

Less than 0.02% of the administered dose was present in the edible tissues. Levels of ^{14}C as glufosinate-ammonium in the liver, muscle and fat were 0.11, <0.004 and 0.003 mg/kg respectively. Those in the eggs are shown in Table 5. ^{14}C in egg whites reached a plateau by day 6, but in the last 3 days there was again a small increase. The levels in egg yolks increased very slowly throughout the 14 days.

MPP was the main residue identified in the liver with glufosinate-ammonium also a substantial component (Table 6). Glufosinate constituted most of the residue in eggs.

Table 5. ^{14}C in eggs from hens dosed orally twice daily for 14 consecutive days by capsule with [3,4-^{14}C]glufosinate- (Huang and Smith, 1995c).

Collection day	Mean ^{14}C, mg/kg as glufosinate-ammonium	
	Egg white	Egg yolk
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

Table 6. Compounds identified in the tissues and eggs from hens dosed orally twice daily for 14 consecutive days with [3,4-^{14}C]glufosinate-ammonium (Huang and Smith, 1995c). Residues are expressed as glufosinate-ammonium equivalents.

Compound	Liver		Egg white (day 14)		Egg yolk (day 13)	
	% ^{14}C in liver	mg/kg	% ^{14}C in egg white	mg/kg	% ^{14}C in egg yolk	mg/kg
glufosinate	31	0.036	78	0.052	53	0.012
NAG, L-isomer	4.9	0.006	not detected		2.4	0.001
MPP	44	0.050	1.3	0.001	4.1	0.001
MPA	3.5	0.004	not detected		3.1	0.001

Tissue, eggs and excreta residues were measured in 6 laying hens weighing 1.27-1.60 kg dosed orally twice daily for 14 consecutive days by capsule with [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt equivalent to 27 ppm *N*-acetyl-L-glufosinate disodium salt in the diet (it was not clear whether the feeding level was expressed on a fresh- or dry-weight basis) or 2.2 mg/kg bw/day (Huang and Smith, 1995d). The mean feed intake was 116 g/bird/day. Eggs were collected twice daily and the birds were slaughtered 15 hours after the final dose.

86% of the administered dose was excreted with 1.0% remaining in the GI tract. *N*-acetyl-L-glufosinate disodium salt accounted for 73% of the ^{14}C in the faeces with glufosinate and MPP accounting for 13% and 8.6% respectively.

Less than 0.1% of the administered dose was present in the edible tissues and blood. Levels of ^{14}C (as *N*-acetyl-L-glufosinate disodium salt) in the liver, muscle and fat were 0.076, 0.013 and 0.011 mg/kg respectively. Those in eggs are shown in Table 7. Levels in egg whites were slightly above the LOD throughout the study; those in egg yolks increased slowly but steadily throughout the 14 days.

NAG (the administered material) was the main residue identified in liver and egg yolk (Table 8). Glufosinate and MPP were also substantial components of the liver residue. Glufosinate was the main identified residue in egg whites, but the levels in eggs were quite low, making further identification difficult.

Table 7. ^{14}C in eggs from hens dosed orally twice daily for 14 consecutive days with [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt (Huang and Smith, 1995d).

Collection day	Mean ^{14}C level, mg/kg as N-acetyl-L-glufosinate disodium salt	
	Egg white	Egg yolk
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
11	0.012	0.046
12	0.013	0.050
13	0.015	0.049
day 14 sac	0.014	0.052
necropsy	<0.009	0.056

Table 8. Compounds identified in the tissues and eggs from hens dosed orally twice daily for 14 consecutive days with [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt (Huang and Smith, 1995d). Residues are expressed as *N*-acetyl-L-glufosinate disodium salt equivalents.

Compound	Liver		Egg white (day 13)		Egg yolk (necropsy)	
	% ^{14}C	mg/kg	% ^{14}C	mg/kg	% ^{14}C	mg/kg
Glufosinate	15	0.011	14	0.002	2.8	0.002
NAG, L-isomer	27	0.020	5.1	0.001	13	0.007
MPP	17	0.013	2.0	<0.001	2.2	0.001
MPA	not detected		1.1	<0.001	0.6	<0.001

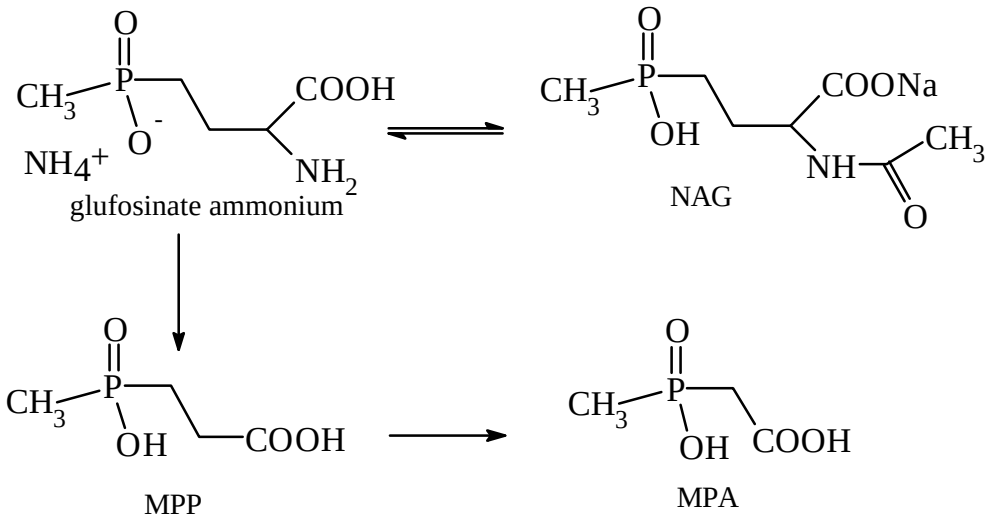
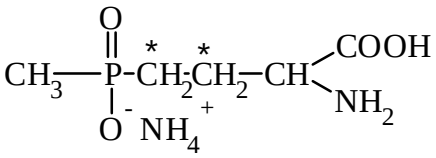


Figure 1. Proposed metabolic pathways of glufosinate and *N*-acetyl glufosinate in ruminants and poultry.

Plant metabolism

Information was reported on the metabolism of glufosinate-ammonium in genetically modified rape (canola), sugar beet, maize, soya and tomatoes. The studies examined the disposition of the residue throughout the plant and its composition. In some cases the metabolism of glufosinate-ammonium in genetically modified and unmodified crops was compared.

Stumpf *et al.* (1995b) placed cut rape plants, genetically modified and unmodified, in a nutrient solution containing 4.7 mg/l of [3,4-^{14}C]glufosinate-ammonium for 6 days. In the genetically modified plants the ^{14}C represented NAG (57%) and glufosinate-ammonium (36%). In the unmodified plants the ^{14}C was mainly due to unchanged glufosinate-ammonium (80%) with 16% MPP. The experiment demonstrated the rapid acetylation of glufosinate in genetically modified rape.



Labelled glufosinate-ammonium

Tshabalala (1993) treated canola plants (*var* 19-2XACS-N3) at the 3-5 leaf stage once with [3,4-^{14}C]glufosinate-ammonium at a rate equivalent to 0.75 kg ai/ha and collected samples for radio-analysis on days 1, 21 and 120. The results are shown in Table 9.

The levels in the top-growth and roots were much the same after 21 days, but at maturity were much higher in the roots than in other plant parts. The residues in the canola seed were investigated by two HPLC systems but the low residue levels made further identification difficult. Glufosinate and MPP were the main constituents with considerably lower levels of NAG.

Table 9. Levels of ^{14}C in canola plants treated once with [3,4-^{14}C]glufosinate-ammonium at 0.75 kg ai/ha (Tshabalala 1993).

Sample	Time since treatment	^{14}C as glufosinate-ammonium, mg/kg
whole plant	1 hour	145
topgrowth	21 days	5.3 3.2
roots	21 days	3.8 5.2
topgrowth	120 days	0.058 0.064 0.024 0.021
roots	120 days	0.22 0.13 0.15 0.19
hulls	120 days	0.12 0.26 0.11 0.076
seed	120 days	0.054 0.11 0.056 0.045

Thalacker (1994) applied [^{14}C]glufosinate-ammonium to glufosinate-tolerant canola and samples were taken after 1 hour and 21 days. After 1-hour 73% of the ^{14}C was in glufosinate and 18% in NAG, and after 21-days 60, 21 and 7% of the ^{14}C corresponded to NAG, glufosinate-ammonium and MPP respectively. Again, genetically modified rape seed (canola) produced NAG very rapidly from glufosinate-ammonium.

In another trial, genetically modified sugar beets were treated twice (22 days interval) with [3,4-^{14}C]glufosinate-ammonium at rates equivalent to 0.6 kg ai/ha (Allan, 1996). The leaves and beets (when formed) were harvested 0, 8 and 15 days after the first treatment and 0, 21 and 146 days after the second. The leaves were rinsed with water to separate surface residues from absorbed residues

The residues after the first treatment are shown in Table 10. The glufosinate isomer composition was unchanged in the surface residue, but in the absorbed residue L-glufosinate was metabolized to NAG (*N*-acetyl-L-glufosinate).

The composition of the residue after the second treatment is shown in Table 11. Identified residues accounted for 93-98% of the total ^{14}C in leaves + rinses and roots. After the first treatment NAG was the main identified residue except in the leaves on the day of treatment, but even after 146 days glufosinate accounted for 19% of the ^{14}C in the roots and 26% in the leaves. The reason is presumably that L-glufosinate is rapidly converted to NAG, but D-glufosinate remains unchanged.

Table 10. Identified residues in genetically modified sugar beets after a single treatment with [3,4-^{14}C]glufosinate-ammonium.

Time after treatment	Sample	Residue as % of total ^{14}C in the sample			
		Glufosinate	D-glufosinate	L-glufosinate	NAG
3 hours	rinse	41	20	21	
8 days	rinse	18	9	9	
15 days	rinse	14	7	7	
3 hours	leaves	45.1	24.6	20.5	9.0
8 days	leaves	35.6	28.4	5.5	39
15 days	leaves	29.3	25.2	3.3	49

Table 11. Identified residues in genetically modified sugar beets after 2 treatments with [3,4-^{14}C]glufosinate-ammonium (Allan, 1996).

Time after 2nd treatment, days	Sample	Glufosinate		MPP		NAG	
		% of ^{14}C in (rinse + leaves) or roots	mg/kg as glufosinate-ammonium	% of ^{14}C in (rinse + leaves) or roots	mg/kg as glufosinate-ammonium	% of ^{14}C in (rinse + leaves) or roots	mg/kg as glufosinate-ammonium
0	rinse	59	12	-	-	-	-
0	leaves	25	5.1	0.4	0.07	13	2.7
0	roots	31	0.62	2.2	0.04	64	1.3
21	rinse	14	1.7	-	-	-	-
21	leaves	28	3.4	1.1	0.13	55	6.8
21	roots	31	2.1	2.0	0.14	63	4.3
146	rinse	2.3	0.05	0.3	0.006	0.2	0.005
146	leaves	24	0.49	2.7	0.055	67	1.4
146	roots	19	0.18	6.0	0.055	68	0.63

In another trial transgenic maize plants were treated twice (40 cm and 60 cm growth stages) with [3,4-^{14}C]glufosinate-ammonium at rates equivalent to 0.50 kg ai/ha (Burnett, 1994). Plants were sampled on the days of treatment and at intervals until 102 days after the second treatment. The levels of ^{14}C as glufosinate-ammonium were 23 and 5.8 mg/kg 1 hour and 5 days after the first treatment respectively and 14.5 and 9.9 mg/kg 1 hour and 5 days after the second.

Glufosinate-ammonium was generally a minor component of the residue whereas the main component in the forage, silage and fodder was NAG and the main component in the grain, cobs and husks was MPP. 73-83% of the residue was identified except in the grain where the very low residue level made further identification difficult.

A sub-sample of the maize forage was analysed by a GLC enforcement analytical method (Czarnecki and Bertrand, 1994), in which samples are extracted with distilled water, and after clean-up which includes ion-exchange chromatography, the residues are derivatized with trimethyl orthoacetate for GLC analysis. The enforcement method and the radiolabel method were in reasonable agreement (Table 13).

Table 12. Identified residues in grain and animal feeding commodities from transgenic maize treated twice with [3,4-^{14}C]glufosinate-ammonium (Burnett, 1994). Forage and silage were sampled 28 and 55 days respectively after the second treatment and other commodities after 102 days.

Compound	Residue components as % of ^{14}C in the sample and as mg/kg glufosinate-ammonium											
	Forage		Silage		Fodder		Grain [1]		Cobs		Husks	
	^{14}C %	mg/kg	^{14}C %	mg/kg	^{14}C %	mg/kg	^{14}C %	mg/kg	^{14}C %	mg/kg	^{14}C %	mg/kg
Glufosinate	13	0.35	11	0.20	9.9	0.20	1.5	0.002	2.6	0.006	2.1	0.018
MPP	12	0.32	12	0.21	11	0.22	33	0.043	44	0.10	41	0.36
NAG	52	1.4	55	0.98	54	1.1	9.1	0.012	20	0.046	19	0.17
MPA	4.6	0.12	3.9	0.070	2.9	0.058	4.4	0.006	12	0.028	11	0.097
Identified	82	2.2	82	1.5	76	1.6	58	0.076	79	0.18	73	0.64
Total		2.6		1.8		2.0		0.13		0.25		0.87

[1] MPB accounted for 9.8% of the ^{14}C in the grain, equivalent to 0.013 mg/kg of glufosinate-ammonium.

Table 13. Comparison between analysis of maize forage from [3,4-^{14}C]glufosinate-ammonium-treated maize by an enforcement GLC method (Czarnecki and Bertrand, 1994) and the ^{14}C HPLC method (Burnett, 1994).

Analyte	Residue, mg/kg as glufosinate-ammonium		
	HPLC ^{14}C	GLC enforcement, not adjusted for recovery	Spike recovery, %, GLC enforcement
Glufosinate	0.366	0.256	113
MPP	0.304	0.185	69.5
NAG	1.43	1.42	105

In another trial transgenic soya bean plants were treated twice at third trifoliate leaf and full bloom growth stages with [3,4-^{14}C]glufosinate-ammonium at rates equivalent to 0.50 kg ai/ha (Rupprecht and Smith, 1994). Forage was sampled just before the second application and the mature crop was harvested 85 days after the second treatment.

NAG was the main residue in all the samples. The levels of MPP were greater than those of glufosinate in the pods and beans. 90-94% of the residue was identified in all the samples. The results are shown in Table 14. They were also reported by Rupprecht *et al*. (1996b).

The samples were extracted with distilled water or acetonitrile + water and analysed by the method of Czarnecki and Bertrand (1994). The GLC and HPLC radiolabel methods produced similar results (Table 15) at the higher levels but at low levels those from the enforcement method were lower.

Table 14. Identified residues in beans, pods, forage and straw from transgenic soya plants treated twice with [3,4-^{14}C]glufosinate-ammonium (Rupprecht and Smith, 1994).

Compound	Identified residue components as % of ^{14}C in the sample and as glufosinate-ammonium, mg/kg							
	Forage		Straw		Pods		Beans	
	^{14}C %	mg/kg	^{14}C %	mg/kg	^{14}C %	mg/kg	^{14}C %	mg/kg
Glufosinate	23	0.45	19	0.58	5.8	0.29	6.2	0.091
MPP	6.5	0.13	14	0.42	22	1.1	16	0.23
NAG	60	1.2	53	1.7	63	3.1	61	0.89
MPA	0.7	0.014	5.7	0.18	2.9	0.142	7.1	0.10
Identified	91	1.8	91	2.8	94	4.6	90	1.3
Total		1.9		3.2		4.7		1.4

Table 15. Comparison between analysis of samples from [3,4-^{14}C]glufosinate-ammonium-treated soya beans by an enforcement GLC method (Czarnecki and Bertrand, 1994) and the ^{14}C HPLC method (Rupprecht and Smith, 1994)

Sample			Residue, mg/kg as glufosinate-ammonium		
	Extract	Method	Glufosinate	NAG	MPP
Forage	water	^{14}C HPLC	0.364	0.962	0.101
Forage	water	GLC	0.321	1.04	0.053
Straw	water	^{14}C HPLC	0.542	1.58	0.404
Straw	water	GLC	0.348	1.89	0.202
Pods	water	^{14}C HPLC	0.250	2.88	1.02
Pods	water	GLC	0.238	3.23	0.781
Beans	water	^{14}C HPLC	0.073	0.687	0.187
Beans	water	GLC	0.034	0.605	0.129
Forage	acetonitrile + water	^{14}C HPLC	0.084	0.195	0.025
Forage	acetonitrile + water	GLC	0.046	0.156	0.022

Köcher and Becker (1991) applied [^{14}C]glufosinate-ammonium at the 7-leaf stage to the leaves of glufosinate-resistant and unmodified tomato plants. After intervals of 1 and four days foliar absorption of the ^{14}C was the same in both types of plant, but translocation of the ^{14}C from the treated leaves to shoots, other leaves and roots was approximately four times as high in the resistant plants.

Stumpf *et al.* (1995a) treated genetically modified tomato plants at the 7-8 leaf stage with [3,4-^{14}C]glufosinate-ammonium at a rate equivalent to 0.8 kg ai/ha. Samples of plants were taken on the day of treatment and subsequently at intervals up to maturity, 74 days after treatment. The composition of the surface residues (in the rinse) and absorbed residues is shown in Table 16.

The major part (83-97%) of the surface residue in the rinse was glufosinate itself, even after long intervals. Glufosinate was very rapidly converted to NAG once absorbed into the tomato leaves and eventually accounted for about half of the residue, which is in accord with the rapid conversion of L-glufosinate and the stability of both D-glufosinate and NAG. The composition of the residue in the stems followed a similar pattern except that NAG accumulated to almost 70% of the residue and glufosinate fell to about 30%.

There were no detectable residues on the tomato fruit surface, probably because the plants were sprayed before any fruit were formed. NAG accounted for about 90% of the residue in the fruit, with glufosinate itself at essentially negligible levels. MPP was a minor residue in the fruit. NAG is evidently sufficiently mobile to translocate to the fruit, but D-glufosinate is not.

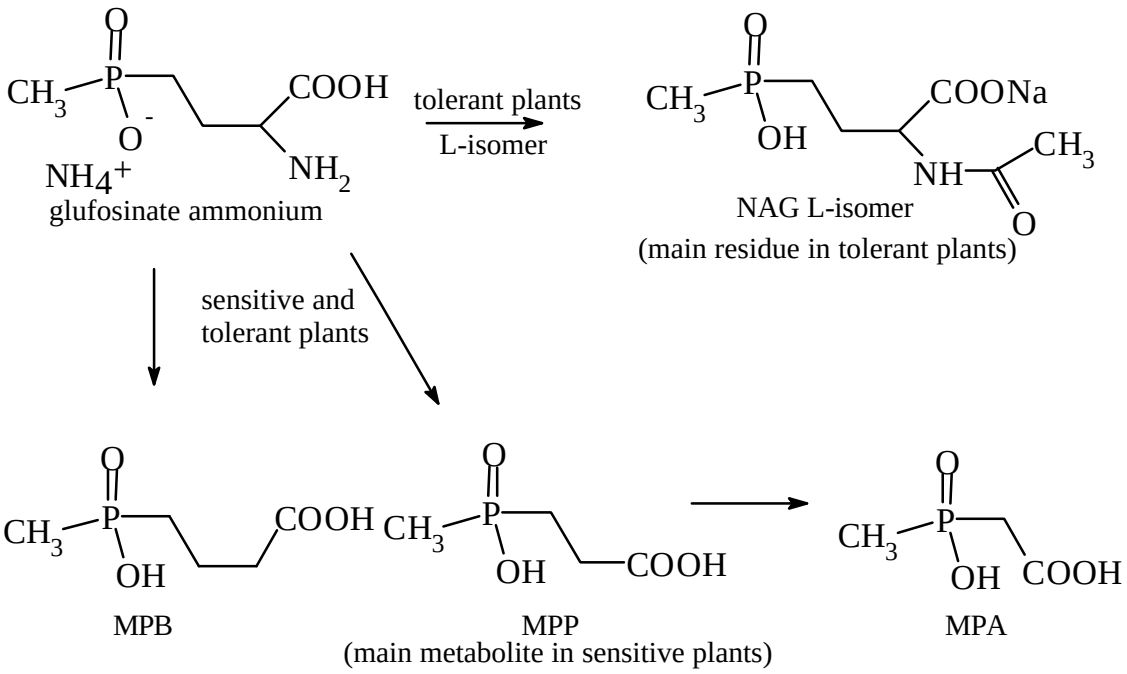
The composition of the glufosinate surface residue on the tomato leaves and stems on day 74 was shown to consist essentially of equal amounts of D- and L-glufosinate. The composition was quite different in the absorbed residue in the leaves (90% D- and 10% L-glufosinate) and in the stems (86% D- and 14% L-glufosinate), showing that within the tissue the L-isomer was more rapidly metabolized

In a separate experiment Stumpf *et al.* (1995a) placed genetically modified and normal tomato plants in solutions of labelled glufosinate for 2 days and examined the uptake and composition of the residue in the plants. The genetically modified plants took up much more glufosinate and converted about half of it to NAG. In the normal tomato plants about 4% was converted to MPP and the D- and L-glufosinate remained approximately equal.

Table 16. Residues in genetically modified tomato plants at the 7-8 leaf stage after treatment with [3,4-^{14}C]glufosinate-ammonium (Stumpf *et al.,* 1995a).

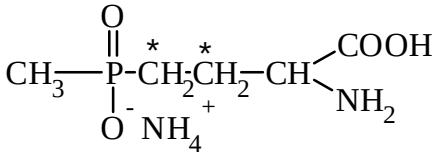
Sample		Days after treatment	% of ^{14}C, mg/kg as glufosinate-ammonium					
			glufosinate-ammonium		MPP		NAG	
			^{14}C %	mg/kg	^{14}C %	mg/kg	^{14}C %	mg/kg
leaves	Rinse	0 (2 h)	94		1.8		0.0	
leaves	Rinse	0 (4 h)	94		1.5		0.0	
leaves	Rinse	1	95		1.5		0.0	
leaves	Rinse	4	94		1.6		0.3	
leaves	Rinse	6	93		1.8		0.6	
leaves	Rinse	32	93		1.4		1.2	
leaves	Rinse	74	86		4.1		3.3	
leaves	Rinse	74	83		7.2		3.9	
leaves	After rinsing	0 (2 h)	69		0.0		27	
leaves	After rinsing	0 (4 h)	61		0.0		36	
leaves	After rinsing	1	64		0.0		33	
leaves	After rinsing	4	52		1.0		42	
leaves	After rinsing	6	53		2.4		41	
leaves	After rinsing	32	57		3.5		37	
leaves	After rinsing	74	44		5.6		51	
leaves	After rinsing	74	42	2.2	2.5	0.13	50	2.6
leaves	Not rinsed	12	30		1.6		65	
leaves	not rinsed	14	17		0.0		83	
leaves	not rinsed	27	0.0		0.0		100	
leaves	not rinsed	32	75		2.7		20	
leaves	not rinsed	74	51	4.4	2.5	0.22	42	3.6
stems	rinse	0 (2 h)	95		1.0		0.0	
stems	rinse	0 (4 h)	97		0.5		0.0	
stems	rinse	1	93		1.9		1.2	
stems	rinse	4	94		1.1		2.0	
stems	rinse	6	94		1.1		2.3	
stems	rinse	32	88		2.1		5.7	
stems	rinse	74	87		2.2		7.4	
stems	rinse	74	92		0.0		7.7	
stems	after rinsing	0 (2 h)	75		0.0		16	
stems	after rinsing	0 (4 h)	61		0.0		39	
stems	after rinsing	1	58		0.0		39	
stems	after rinsing	4	42		0.0		58	
stems	after rinsing	6	45		0.0		53	
stems	after rinsing	32	32		6.4		61	
stems	after rinsing	74	31		-		69	
stems	after rinsing	74	26	0.51	3.8	0.07	67	1.3
stems	not rinsed	12	8.6		0.0		91	
stems	not rinsed	14	7.2		0.0		93	
stems	not rinsed	27	0.0		0.0		100	
stems	not rinsed	32	47		3.6		49	
stems	not rinsed	74	37	0.95	2.6	0.07	57	1.5
fruit	rinse	74	0		0		0	
fruit	after rinsing	74	0.0	-	6.2	0.009	94	0.14
fruit	not rinsed	74	0.0	-	9.5	0.02	85	0.17
fruit, green	not rinsed	32	1.7		4.6		88	
fruit, red	not rinsed	60	0.0		0.0		100	
fruit, red	not rinsed	74	0.0		0.0		100	

Figure 2. Proposed metabolic pathways of glufosinate-ammonium in plants.



Environmental fate in soil

Aerobic degradation


Labelled glufosinate-ammonium

Allan (1995) studied the aerobic degradation of [3,4-^{14}C]glufosinate-ammonium in a sandy loam soil (pH 6.0, organic carbon 1.8%) both when applied directly to the soil at 2 mg/kg and when incorporated as a residue in bushbean leaves which, having been desiccated with labelled glufosinate-ammonium, contained ^{14}C equivalent to 76 mg/kg of glufosinate-ammonium. The bean leaves (1.66 g) were added to 50 g of soil giving a theoretical level of 2.5 mg/kg. The soil was adjusted to 40% of maximum water holding capacity and incubated at 20°C in the dark. The results are shown in Table 17.

Glufosinate disappeared very quickly with a half-life of 3-6 days (Table 18). The main product was MPP, reaching its peak after about 14 days. MPA was also an important product and in the absence of plant material became the main residue after long intervals. NAG was a very minor soil residue.

Table 17. Distribution of ^{14}Cfrom [3,4-^{14}C]glufosinate-ammonium incubated aerobically in a soil in the dark or incorporate-d into the soil as a plant-metabolized residue (Allan 1995).

Days incubated	Residues and evolved $^{14}CO_2$ expressed as % of applied ^{14}C.					
	Soil treatment			Plant incorporation		
	extractable	Unextractable	$^{14}CO_2$	extractable	Unextractable	$^{14}CO_2$
0	106	1.8	-	97	9.7	0
1	80	8.3	0.6	90	14	0.1
3	82	11	1.5	87	17	0.4
7	77	15	5.6	80	20	1.8
14	70	21	11	75	21	4.1
21	61	21	17	69	24	7.6
28	60	20	23	68	28	8.6
41	46	19	26	58	20	9.5
59	32	24	43	32	26	22
90	8.7	25	57	23	29	26
120	5.6	21	62	18	23	31

Table 18. Composition of residues resulting from aerobic incubation of [3,4-^{14}C]glufosinate-ammonium in a soil in the dark or incorporated into the soil as a plant-metabolized residue (Allan 1995).

Days incubated	% of applied ^{14}C							
	Soil treatment				Plant incorporation			
	glufosinate	MPP	NAG	MPA	glufosinate	MPP	NAG	MPA
0	104	-	-	-	75	14	3.2	-
1	77	11	2.3	-	70	18	-	-
3	56	19	2.4	2.1	47	30	4.8	2.2
7	32	31	2.2	6.4	15	52	-	7.3
14	18	37	0.7	11	2.9	62	-	6.3
21	7.8	33	0.4	15	0.6	56	-	9.2
28	3.3	34	-	19	-	61	-	6.9
41	0.9	22	0.3	20	-	55	-	0.3
59	0.5	13	-	17	-	30	-	-
90	0.1	1.4	-	6.8	-	23	-	-
120	0.5	1.6	-	1.6	-	17	-	-

Field dissipation

In a 1-year field dissipation study in California Belcher (1996a) applied glufosinate-ammonium three times to bare ground at rates of 1.7 kg ai/ha between rows in a level vineyard. The soil was a coarse sandy loam. Irrigation supplemented rainfall and followed the growers' standard practices. Soil samples were analysed for glufosinate, MPP and MPA, the main residues. The results are shown in Table 19.

Glufosinate dissipated rapidly, possibly owing to increasing soil moisture and temperature, with calculated half-lives of 15, 7.2 and 2.7 days after the first, second and third applications respectively.

The maximum MPP residues were 0.11, 0.16 and 0.14 mg/kg on days 30, 10 and 5 after the first, second and third applications respectively and the estimated half-lives were 38, 14 and 16 days from these times. The estimated half-lives for MPA were 25, 19 and 7 days for the successive

applications. MPA residues reached their highest levels of 0.06, 0.06 and 0.04 mg/kg on days 60, 10 and 5 after the first, second and third applications respectively.

No residues were detected below the 45-60 cm depth segment. Glufosinate was detected once at the analytical LOD (0.01 mg/kg) after the first application and at 0.02 mg/kg 5 and 10 days after the final application in the 45-60 cm samples. Glufosinate was detectable at low levels 5 and 10 days after application in the 30-45 cm samples, but not at other times. MPP occurred in the 30-45 cm segment on several occasions at 0.01-0.02 mg/kg. MPP was detected only once in the 45-60 cm depth segment, 10 days after the final application at 0.01 mg/kg. MPA was not detected in any sample from 30-45 cm or deeper.

Glufosinate and its soil degradation products have some mobility but their further degradation ensures that travel down the soil profile is limited.

Table 19. Residues of glufosinate and its degradation products at intervals after three applications of glufosinate-ammonium at 1.7 kg ai/ha to bare ground in a level California vineyard (Belcher, 1996a).

Interval after application days	Residues, mg/kg as glufosinate free acid, in soil					
	glufosinate		MPP		MPA	
	soil 0-15 cm	soil 15-30 cm	soil 0-15 cm	soil 15-30 cm	soil 0-15 cm	soil 15-30 cm
Application 1						
0	0.34	<0.01	0.01	<0.01	<0.01	<0.01
5	0.26	<0.01	0.04	<0.01	<0.01	<0.01
10	0.13	<0.01	0.06	<0.01	0.01 <0.01	<0.01
15	0.16	0.05 <0.01	0.07	<0.01	0.02	<0.01
28	0.1	<0.01	0.10	<0.01	0.03	<0.01
47	0.04	<0.01	0.10	<0.01	0.04	<0.01
65	0.02	<0.01	0.05	0.02	0.05	<0.01
93	<0.01	<0.01	0.02	0.02	0.04	<0.01
124	<0.01	<0.01	<0.01	<0.01	0.01	<0.01
Application 2						
0	0.47	<0.01	0.03	<0.01	0.02	<0.01
5	0.10	0.07	0.09	0.03	0.03	<0.01
10	0.02	0.08	0.08	0.04	0.05	0.01
15	0.02	0.03	0.06	0.03	0.05	0.01
30	<0.01	0.02 <0.01	0.03	0.02	0.02	0.01
45	<0.01	0.01	0.01	0.03	0.01 <0.01	0.01 <0.01
59	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
90	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
119	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Application 3						
0	0.41	<0.01	0.02	<0.01	<0.01	<0.01
5	0.03	0.04	0.06	0.04	0.03	0.01
10	0.01	0.01	0.04	0.02	0.02	0.02
15	0.01	<0.01	0.03	0.01	0.01	0.01
30	<0.01	<0.01	0.02	<0.01	<0.01	<0.01
45	<0.01	<0.01	0.02	<0.01	<0.01	<0.01
60	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
93	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Stumpf (1993a) incubated 3.6 mg/kg [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt in a sandy loam soil (pH 5.8, organic carbon 1.1%) in the dark at 20°C under aerobic conditions for 62 days. The results are shown in Table 20.

N-acetyl-L-glufosinate was very rapidly degraded to L-glufosinate, which was then further degraded. The degree of mineralization (40%) and the level of bound residues (21%) in 62 days parallel the 43% mineralization of glufosinate-ammonium and 24% unextractable residues in 59 days reported by Allan (1995), and suggest that the degradation pathway for NAG is through glufosinate.

Table 20. Aerobic soil degradation of [3,4-^{14}C]*N*-acetyl-L-glufosinate disodium salt in soil (Stumpf, 1993c).

Day	Residues and evolved $^{14}CO_2$ expressed as % of applied ^{14}C and as NAG mg/kg										
	$^{14}CO_2$	NAG		L-glufosinate		MPP		MPA		bound residues	
	%	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg	%	mg/kg
0.1	0	47	1.7	47	1.7	2.1	0.08	1.0	0.04	0.7	0.03
1	0.5	8.7	0.31	80	2.9	6.7	0.24	1.3	0.05	1.5	0.05
3	1.0	3.1	0.11	69	2.5	20	0.73	3.4	0.12	1.0	0.04
23	15	1.4	0.05	16	0.57	40	1.4	9.5	0.34	11	0.38
41	24	1.6	0.06	4.7	0.17	39	1.4	9.9	0.36	13	0.44
62	40	0	0	1.5	0.05	27	0.98	9.9	0.36	21	0.75

Stumpf *et al.* (1995c) incubated 0.5 mg/kg [2-^{14}C]2-methylphosphinicoacetic acid (MPA) in a sandy loam (pH 5.8, organic carbon 1.1%) and a loamy sand (pH 4.9, organic carbon 2.4%) in the dark at 20°C under aerobic conditions for 122 days and measured the rate of degradation and mineralization (Table 21). No important degradation products other than CO_2 were identified.

In the sandy loam bound residues accounted for 22% and 25% of the applied ^{14}C on days 28 and 122 respectively. Estimated decline and mineralization half-lives were 24 and 74 days respectively, but the curves were not simple first-order and the rates were slower at longer intervals. The rates in the loamy sand were much slower, with only 20% mineralization after 122 days and a decline half-life of 120-160 days. Bound residues again accounted for about 25% of the applied ^{14}C throughout the study.

Table 21. Aerobic soil degradation of [2-^{14}C]2-methylphosphinicoacetic acid (Stumpf *et al.* 1995c).

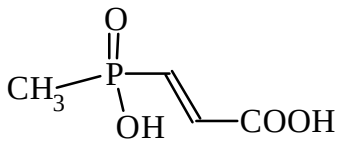
day	loamy sand			sandy loam		
	$^{14}CO_2$	MPA		$^{14}CO_2$	MPA	
	% of applied ^{14}C	% of applied ^{14}C	mg/kg	% of applied ^{14}C	% of applied ^{14}C	mg/kg
0		87	0.47		99	0.54
3	0.7	89	0.48	4.0	90	0.49
7	1.8	86	0.47	6.6	89	0.49
14	4.2	76	0.42	14.5	77	0.42
28	7.9	69	0.38	31	47	0.26
60	17	62	0.34	63	45	0.25
94	23	53	0.29	75	3.8	0.002
122	20	56	0.30	75	3.5	0.002

Stumpf *et al.* (1995d) incubated [3,4-^{14}C]glufosinate-ammonium at 1.9 mg/kg and [3-^{14}C]MPP at 0.9 mg/kg in the same sandy loam soil in the dark at 10°C under aerobic conditions for 120 days. The results are shown in Table 22 and 23.

The estimated half-life for glufosinate-ammonium disappearance was 24 days; the rate of mineralization was slow with an estimated half-life of 300 days or more. MPP and subsequently MPA were major components of the residue after the longer periods of incubation. NAG and

AE F065594 (4-methylphosphinico-2-oxobutanoic acid) and possibly AE F086486 (3-methylphosphinico-3-oxopropionic acid) were identified as minor products.

The estimated half-life for MPP disappearance and mineralization were 86 days and 220 days respectively. MPA was the only product of significance accounting for 40% of the extractable residue by day 120.



AE 0015081
3-methyl-phosphinico
acrylic acid

Stumpf and Zumdick (1998) further investigated the nature of the minor soil product previously identified as AE F086486 and, using LC-MS-MS, identified the compound as 3-methylphosphinicoacrylic acid (AE 0015081). The structure was confirmed by synthesis.

Table 22. Residues resulting from incubation of 1.9 mg/kg [3,4-^{14}C]glufosinate-ammonium in a sandy loam soil in the dark at 10°C under aerobic conditions for 120 days (Stumpf *et al.*, 1995d).

Incubation, days	Residues, % of applied ^{14}C					
	Extractable residue	Glufosinate	MPA	MPP	NAG/ AE F065594	AE F086486[1]
0	94	90	<0.5	1.6	1.8	<0.5
1	93	85	<0.5	5.3	2.5	<0.5
4	91	72	1.9	12	1.8	2.3
7	86	57	3.3	20	2.2	4.0
14	87	42	5.5	32	2.6	4.9
21	87	32	7.9	38	2.5	7.1
30	86	25	9.5	43	1.8	6.8
56	79	9.9	14	45	2.5	6.7
91	69	2.0	19	41	3.1	4.1
120	69	5.4	22	36	2.7	3.9

[1]Subsequently identified as AE 0015081 (Stumpf and Zumdick, 1998).

Table 23. Aerobic degradation of [3-^{14}C]3-methylphosphinicopropionic acid (MPP) incubated at 0.9 mg/kg in a sandy loam soil in the dark at 10°C for 120 days (Stumpf *et al.*, 1995d).

Incubation, days	Residues, % of applied ^{14}C			
	Extractable residue	CO_2	MPP`	MPA
0	97	0	97	<1.0
1	95	0.1	95	<1.0
4	96	0.6	94	1.8
7	90	1.1	87	3.1
14	91	3.0	79	11.4
21	89	5.4	80	9.0
30	90	6.1	76	13.5
56	74	18	49	25
91	68	21	46	22
120	62	32	37	25

Zumdick (1995a) incubated 2.1 mg/kg [3,4-^{14}C]L-*N*-acetyl-glufosinate in a sandy loam and a loamy sand under aerobic conditions for 120 days at 20°C in the dark (Table 24). In a second experiment glufosinate-tolerant tomato leaves containing residues equivalent to 1 mg glufosinate-

ammonium per kg soil were incorporated into the sandy loam and incubated under the same conditions (Table 25). Bound residues accounted for about 14-23% of the applied residue in each of the three incubations from days 28 to 120.

N-acetyl-glufosinate disappeared very quickly, with half-lives of only hours, producing initially L-glufosinate, which was then degraded further to MPP, MPA and CO_2. MPP became the major component of the residue after 1-3 days.

The tolerant-tomato residue comprised mainly glufosinate and *N*-acetyl-glufosinate. After 3 days incubation in the sandy loam soil most of the residue had been converted to MPP, which itself was degraded more slowly to MPA and ultimately to CO_2.

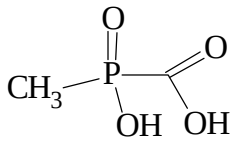
Table 24. Residues resulting from incubation of 2.1 mg/kg [3,4-^{14}C] L-*N*-acetyl-glufosinate in a sandy loam and a loamy sand under aerobic conditions for 120 days at 20°C in the dark (Zumdick 1995a).

Days	Residues, % of applied ^{14}C									
	loamy sand					sandy loam				
	NAG	CO_2	L-glufosinate	MPP	MPA	NAG	CO_2	L-glufosinate	MPP	MPA
0	70	-	20	8.4	0.4	61	-	30	4.7	0.0
0.25	14	0.3	58	13	1.2	29	0.2	58	8.1	0.7
1	15	1.1	23	35	4.7	9.2	0.9	49	22	2.2
2	6.2	3.6	23	38	5.9	4.3	2.3	41	29	3.4
3	5.1	2.8	16	42	7.4	3.1	4.9	24	37	5.9
7	4.1	11	6.3	36	9.6	1.8	1.1	38	34	8.1
14	2.8	21	5.2	27	7.6	1.0	10.5	2.6	39	19
28	1.3	39	2.2	5.2	0.8	0.4	23	1.0	28	18
62	0.5	49	1.3	1.9	0.0	0.2	56	1.0	2.1	1.2
90	0.5	52	1.1	0.7	0.0	0.4	67	1.0	0.7	0.3
120	0.3	72	0.4	0.1	0.0	0.2	66	0.4	0.5	0.1

Table 25. Residues resulting from incubation of [3,4-^{14}C]glufosinate metabolized in tolerant tomato leaves, incorporated at 1 mg glufosinate-ammonium equivalent per kg soil, in a sandy loam under aerobic conditions for 120 days at 20°C in the dark (Zumdick 1995a).

Days	Residues, % of applied ^{14}C				
	glufosinate	CO_2	NAG	MPP	MPA
0	46	-	33	4.1	0.3
0.25	47	0.0	27	4.3	0.4
1	44	0.0	30	8.9	0.6
2	27	0.1	20	27	4.1
3	5.4	0.2	2.9	52	16
7	1.4	5.3	0.4	47	25
14	0.7	5.8	0.0	40	18
28	0.6	16	0.0	31	0.6
62	0.7	48	0.0	6.7	1.2
90	0.2	49	0.0	0.8	0.0
120	0.3	43	0.0	2.4	0.0

Zumdick (1995b) incubated 1 mg/kg [3,4-^{14}C]L-*N*-acetyl-glufosinate in a sandy loam from Germany and a silt loam from Nebraska under aerobic conditions for 120 days at 20°C in the dark (Table 26). The results were consistent with other similar experiments, with the NAG rapidly forming glufosinate, which was in turn converted to MPP and MPA. An additional product (AE F130947, methylphosphinicoformic acid) was separated from the others by the HPLC systems used. Bound residues from days 14 to 90 constituted 14-22% of the applied ^{14}C.



AE F130947
methylphosphinico-formic acid

Table 26. Residues resulting from incubation of 1 mg/kg [3,4-^{14}C] L-*N*-acetyl-glufosinate in a sandy loam and a silt loam under aerobic conditions for 120 days at 20°C in the dark (Zumdick 1995b).

Days	Residues, % of applied ^{14}C											
	sandy loam						silt loam					
	NAG	CO_2	L-glufos	MPP	MPA	130947	NAG	CO_2	L-glufos	MPP	MPA	130947
0.1	91	-	9.7	0.8	0.1	<0.1	85	-	13	1.7	0.1	<0.1
0.25	16	0.2	66	7.3	0.6	0.4	26	0.3	36	11	0.3	0.3
1	7.5	0.8	48	21	1.8	2.0	11	1.7	46	23	3.2	2.8
3	4.7	7.0	26	35	5.2	4.2	3.5	6.3	26	25	11	5.9
7	3.2	16	10	32	9.1	6.3	2.4	15	11	18	21	10
14	1.9	29	4.3	20	12	7.3	0.7	36	2.6	2.8	18	8.7
21	1.2	41	6.1	12	8.8	5.3	0.3	44	2.8	1.9	15	4.5
30	0.7	48	4.2	5.8	5.3	3.9	0.3	59	1.5	0.4	4.4	1.3
59	1.3	64	1.5	1.5	0.2	0.3	<0.1	63	0.8	<0.1	<0.1	<0.1
90	0.3	68	0.6	0.3	<0.1	<0.1		65				
120		77						67				

Stumpf *et al.* (1989) subjected [3,4-^{14}C]glufosinate-ammonium in a thin layer of a microbiologically active sandy loam soil to UV irradiation (xenon arc with 290 nm cut-off filters) for 35 days in a 12 hours light-dark cycle at 25°C and identified the products. The calculated degradation half-life was 35 days, with 7.6% production of $^{14}CO_2$ in that time. Three degradation products were identified: MPP (6-21% of the applied ^{14}C), MPA (0-5%) and NAG (0-10%). Three other products were in too small amounts (0-3% and 4-9%) to be identified. Degradation was caused by microbiological processes and photolysis in the presence of humic acids.

In a rotational crop study Campbell and Bennett (1997) treated bare ground plots in North carolina, Missouri and California twice at 10-day intervals with glufosinate-ammonium at 0.39 and 0.54 kg ai/ha and planted winter wheat 75 and 90 days after the final treatment. No residues of glufosinate or MPP were detected (LOD 0.05 mg/kg) in forage, hay, grain or straw samples. No uptake of glufosinate or MPP should occur in winter wheat following a previous use of glufosinate-ammonium.

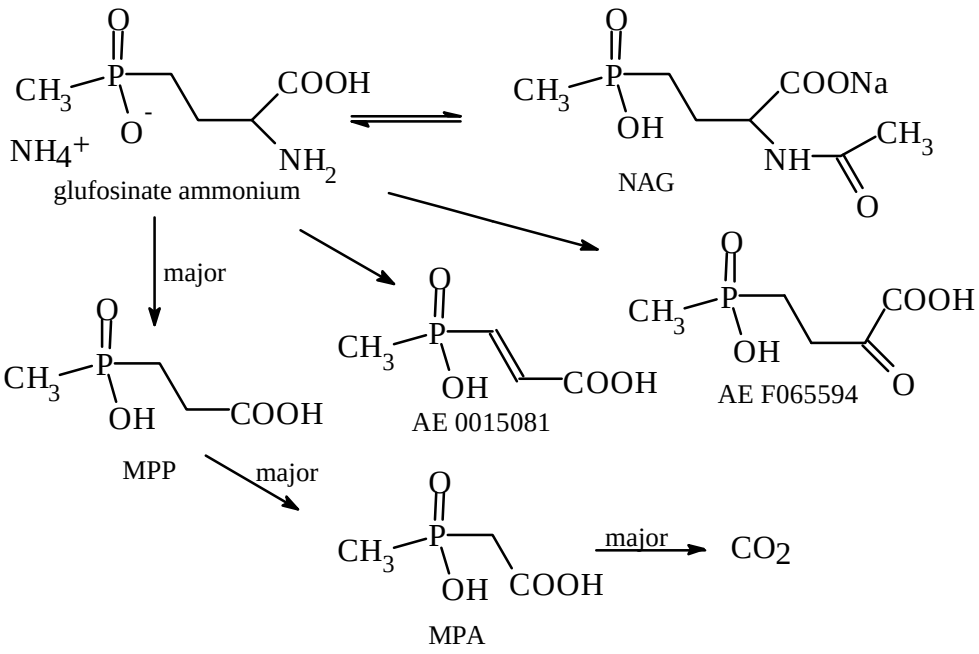
In a confined rotational crop study, Meyer *et al.* (1995) applied [3,4-^{14}C]glufosinate-ammonium to a bare sandy loam soil in a greenhouse in stainless steel tanks at 1.0 kg ai/ha and planted radishes, lettuce and wheat 28 and 119 days later. The intervals represented resowing after crop failure and immediate recropping. Residue levels are shown in Table 27. Product A was not identified, but was characterized as a relatively simple polar unconjugated compound that may be a degradation product taken up by roots. The products were similar in the root, leafy and small cereal grain crops. Levels were much lower after the longer sowing interval. Residues should be undetectable or very low in rotational crops.

Table 27. ^{14}C residues in rotational crops after application of [3,4-^{14}C]glufosinate-ammonium at 1.0 kg ai/ha to a bare sandy loam soil (Meyer *et al.*, 1995).

Commodity	Residues, mg/kg, as glufosinate free acid							
	28 days interval to sowing				119 days interval to sowing			
	total ^{14}C	MPP	MPA	Compound A	total ^{14}C	MPP	MPA	Compound A
Radish top	0.110	0.053	0.006	0.021	0.013[1]			
Radish root	0.090	0.047	0.008	0.024	0.009[1]			
Lettuce	0.079	0.020	0.006	0.013	0.013[1]			
Wheat forage	0.30	0.17	0.019	0.027	0.047[1]			
Wheat straw	0.78	0.35	0.073	0.19	0.14	0.029	0.008	0.035
Wheat grain	0.33	0.11	0.035	0.035	0.12	0.015	<0.001	0.016

[1]Residue too low for characterization.

Figure 3. Proposed aerobic soil degradation pathways of glufosinate-ammonium.



Environmental fate in water/sediment systems

Stumpf and Schink (1992) subjected [3,4-^{14}C]glufosinate-ammonium dissolved in surface water from a gravel pit to UV irradiation for 118 hours (equivalent to 33 days sunlight) at 25°C. Glufosinate suffered little degradation under these conditions, with 3-5% conversion to MPP and 0.2% mineralization. Photolytic degradation of glufosinate in surface waters is a minor degradation route.

Stumpf (1993b) incubated [3,4-^{14}C]glufosinate-ammonium at 0.1 mg/kg in aerobic systems consisting of 180 ml water and 20 g sediment at 20°C and 8°C for 361 days. The sediment was sandy with 0.4% organic carbon.

Glufosinate disappeared from the water/sediment system with half-lives of 3 and 20 days at 20°C and 8°C respectively. Mineralization occurred to the extent of only 25 and 20% at the two temperatures in the 361 days of the study. Within a few days at 20°C and after 29 days at 8°C MPP became the major component of the residue. Seven other products were observed, but five were very minor. Two others amounting to 5% and 20% of the applied dose at various times were not positively identified. At all times most of the total residue was in the aqueous phase.

In sterile (autoclaved) samples incubated for 29 and 120 days up to 50% of the residue was converted to NAG in some samples, but heat resistant bacteria may have been present.

Table 28. Residues resulting from incubation of [3,4-^{14}C]glufosinate-ammonium in a water/sediment system under aerobic conditions at 8°C and 20°C (Stumpf 1993e).

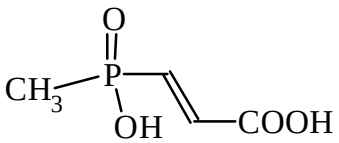
Days	Residues, % of applied ^{14}C									
	20°C					8°C				
	Glufosinate	CO_2	MPP	NAG	MPA	Glufosinate	CO_2	MPP	NAG	MPA
0	95	-	4.4	2.6	0.0	95	-	4.4	2.6	0.0
1	60	0.3	21	14	1.0	84	0.1	7.8	7.0	0.3
4	39	0.8	40	9.8	1.4	61	0.3	15	10.3	0.4
7	24	1.5	49	5.7	3.4	56	0.3	21	14	0.7
14	7.1	4.4	59	3.9	3.2	48	0.7	28	12	1.5
21	0.3	8.2	56	2.4	2.9	45	1.5	32	6.5	2.8
29	0.1	12	53	1.9	3.8	33	1.7	39	3.7	3.5
60	0.0	18	52	0.2	2.9	15	5.4	44	3.3	3.3
90	0.0	21	52	0.1	3.0	2.7	6.7	49	4.2	3.7
120	0.0	17	47	0.0	4.0	0.0	9.0	51	3.0	3.3
238	0.0	23	46	0.0	5.6	0.0	17	45	0.0	2.3
361	0.0	25	48	0.0	5.6	0.0	20	55	0.0	1.4

Stumpf (1994a) incubated [3,4-^{14}C]glufosinate-ammonium at 1 and 0.1 mg/kg in two aerobic systems consisting of 180 ml water and 20 g sediment at 20°C in the dark for 130 days (Tables 29 and 30).

The mineralization in130 days in the gravel pit sediment was 7% at the lower dose and 12% at the higher dose. The half-lives for the degradation of glufosinate itself were 11, 91 and 1.4 days for the loamy river water sediment at 1 mg/kg and the gravel-pit sediment at 1 mg/kg and 0.1 mg/kg respectively. MPP was the main product identified and it was quite persistent in both systems. In all cases most of the residue was in the water phase.

As in a previous experiment (Stumpf, 1993b) a number of minor products were detected but not identified. Two products, sometimes constituting 10-20% of the residue, were further investigated and identified as AE F086486 and possibly AE F130947 (methylphosphinicoformic acid).

In a subsequent study, Stumpf (1994b) used glufosinate labelled in different positions and a higher dose rate to produce sufficient material for positive identification as AE F130947, also identified as a soil degradation study (Zumdick, 1995b).



AE 0015081
3-methyl-phosphinico
acrylic acid

Stumpf and Zumdick (1998) corrected the identification of AE F086486 (3-methylphosphinico-3-oxopropionic acid) to AE 0015081 and confirmed AE F130947.

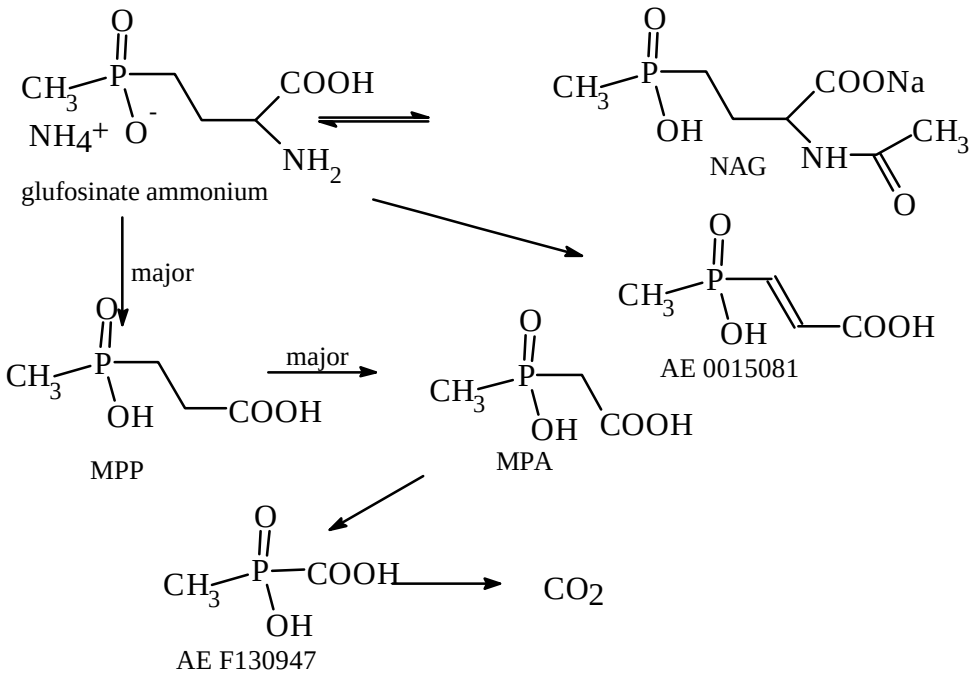
Table 29. Residues resulting from incubation of [3,4-^{14}C]glufosinate-ammonium in a gravel pit water/sediment system at 20°C in the dark for 130 days (Stumpf, 1994a).

Days	Residues, % of applied ^{14}C in whole system									
	gravel-pit sediment, 1 mg/kg					gravel-pit sediment, 0.1 mg/kg				
	Glufosinate	CO_2	MPP	NAG	MPA	Glufosinate	CO_2	MPP	NAG	MPA
0	91	-	2.0	0.7	0.0	89	-	2.9	1.7	0.0
1	86	0.0	6.8	2.0	0.4	59	0.0	23	9.9	0.9
3	74	0.0	15	4.0	0.7	21	0.2	55	7.5	3.5
7	69	0.2	19	4.1	2.4	3.8	1.0	79	1.8	4.7
14	60	0.5	25	3.7	4.1	0.1	2.8	80	0.3	5.7
21	57	0.8	27	2.5	6.2	0.0	4.4	78	0.0	4.6
30	46	1.9	28	3.3	10.0	0.0	6.8	76	0.0	4.1
50	39	2.9	31	4.3	12	0.0	8.6	76	0.0	4.2
77	36	4.3	30	2.9	14	0.0	10.6	74	0.0	4.4
91	37	5.0	30	1.4	14	0.0	12	70	0.0	5.1
130	33	7.1	30	2.1	16	0.0	12	67	0.0	6.8

Table 30. Residues resulting from incubation of [3,4-^{14}C]glufosinate-ammonium in a loamy river water sediment system at 20°C in the dark for 130 days (Stumpf, 1994a).

Days	Residues, % of applied ^{14}C				
	Glufosinate	CO_2	MPP	NAG	MPA
0	91	?	1.3	1.0	0.0
1	87	?	5.4	2.5	0.3
3	72	?	13	6.2	3.4
7	60		19	8.2	5.9
14	46		26	7.6	10.2
21	35		29	4.5	12
30	19		37	1.5	16
50	3.0		41	0.0	20
77	0.0		48	0.0	18
91	0.0		48	0.0	17
130	0.0		45	0.0	16

Figure 4. Proposed aerobic degradation pathways of glufosinate-ammonium in water/sediment systems



METHODS OF RESIDUE ANALYSIS

Analytical methods

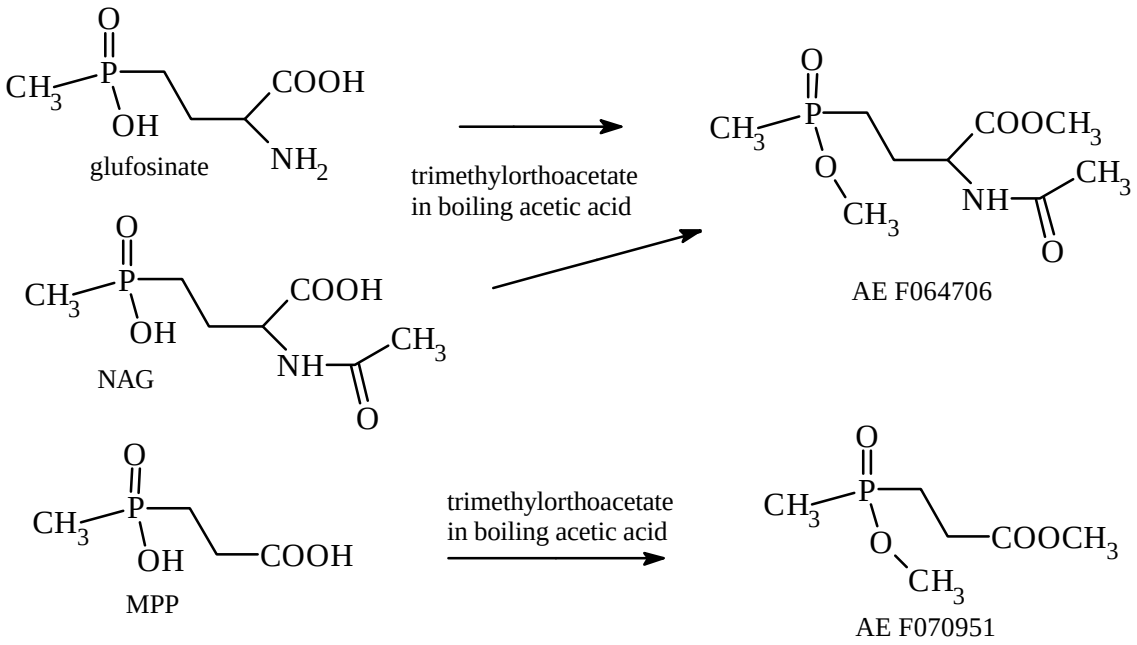
The main components of the residue in genetically modified tolerant crops are glufosinate, NAG (*N*-acetyl-L-glufosinate) and MPP (3-methyl-phosphinico-propionic acid). Analytical methods have been designed to measure the three components separately or, because glufosinate and NAG produce the same derivative in the analytical procedure, to measure glufosinate and NAG combined and MPP separately.

Holzwarth (1995) described the methods used for residue analysis of tolerant crops. Residues are extracted from the finely ground sample with water. The clear extract, after separation from solid material, is passed through an anion exchange resin and the residues are eluted with formic acid. After evaporation of the formic acid the residue is taken up in a 1:1 ethanol/water mixture which is then applied to a cation exchange column. NAG and MPP are eluted with ethanol/water and glufosinate with aqueous ammonia.

After evaporation to dryness the residues from both fractions are taken up in glacial acetic acid and methylated and acetylated with trimethyl orthoacetate in refluxing acetic acid. After solvent exchange and a final silica gel cartridge clean-up the residues are determined by GLC with flame photometric detection.

Variations on the extraction and initial clean-up are needed for samples such as maize oil. The lower limit of determination for crop samples is typically 0.05 mg/kg for each of the three analytes. For fats and milk a mixture of n-propanol and water is used for extraction instead of water.

Figure 5. Derivatization of glufosinate, NAG and MPP.



Czarnecki *et al.* (1989) used a method (HRAV-5A) similar in principle to that described above for the determination of glufosinate and MPP in apples, grapes, soya beans, maize and tree nuts. Czarnecki and Bertrand (1993) described a similar method (HRAV-24) for the determination of glufosinate, MPP and NAG in maize and its processed fractions. Sochor *et al.* (1991) measured residues of glufosinate, MPP and NAG in tomatoes, tobacco and potatoes by the same method and achieved practical LODs of 0.05-0.2 mg/kg.

Czarnecki (1995a) analysed animal commodities for glufosinate and MPP by a similar method but without the cation exchange column clean-up, achieving LODs of 0.02 mg/kg for milk, 0.05 mg/kg for eggs, meat and fat, and 0.1 mg/kg for kidneys and liver. Czarnecki (1995b) included NAG in the same method with the same LODs. NAG and glufosinate appear in the same GLC peak so the LOD is for either compound or the combined residue.

Czarnecki and Bertrand (1994) described a comprehensive procedure for the determination of glufosinate, MPP and NAG in many commodities. The method (AE-24) is essentially as described above with many variations on the extraction depending on the nature of the sample. Recoveries should lie within the range 70-120% at the practical LOD of 0.05 mg/kg for each compound, expressed as glufosinate free acid. The method was determined and used on maize grain, silage, forage, fodder, starch, grits, flour, meal and hulls, and soya bean seed, hay, hulls and meal. The same method was described by Czarnecki (1995c). Bertrand (1994) validated the method (Table 31). Most tests were at a fortification level of 0.05 mg/kg, but recoveries did not seem to depend on concentration. Median recoveries were 95%, 95% and 102% for glufosinate, MPP and NAG respectively.

Bertrand (1994) pointed out that part of the glufosinate added to glufosinate-tolerant plant material could be converted to NAG during recovery testing. When racemic glufosinate was added to transgenic soya bean seed at 0.5 mg/kg, 44-54% was recovered as glufosinate and 33-43% as NAG. When D-glufosinate was used, recoveries of glufosinate were 85-100%. In some transgenic materials it is necessary to use D-glufosinate, which is not subject to enzymic *N*-acetylation, to determine the analytical recovery of glufosinate. The method is not selective for either isomer.

Table 31. Recoveries of glufosinate, MPP and NAG from maize grain, forage, fodder, silage, flour, meal, hulls, starch, and crude oil, and soya bean seeds, hulls, meal, hay/fodder and crude oil (Bertrand, 1994).

Recovery range, %	Number of tests		
	Glufosinate	MPP	NAG
61-70		4	1
71-80	5	13	0
81-90	16	28	4
91-100	17	30	19
101-110	17	23	18
111-120	7	15	8
121-130		4	6
131-140		1	

Castro and Dacus (1994) validated analytical method AE-24 (suitable for enforcement) on commodities from transgenic maize and soya beans. D-glufosinate was used to determine recoveries from soya bean seed. An LOD of 0.05 mg/kg for each compound on each commodity was achieved (Table 32). The time needed to analyse a batch of six samples was about 18 working hours over 3 working days.

Table 32. Recoveries by analytical method AE-24 from transgenic maize and soya bean commodities (Castro and Dacus, 1994).

Commodity	Glufosinate		MPP		NAG	
	spiking levels, mg/kg	Recovery range, %	spiking levels, mg/kg	Recovery range, %	spiking levels, mg/kg	Recovery range, %
Maize grain	0.05 0.25	73-98% (n=4)	0.05 0.25	73-78% (n=4)	0.10 0.50	89-107% (n=4)
Maize forage	0.50 2.5	83-89% (n=4)	0.10 0.50	76-87% (n=4)	3.0 15	101-110% (n=4)
Corn oil, refined	0.05 0.25	57-96% (n=4)	0.05 0.25	70-82% (n=4)	0.05 0.25	113-149% (n=4)
Soya bean seed	0.05 0.25 (D-glufosinate)	65-89% (n=4)	0.10 0.50	80-91% (n=4)	0.20 1.0	85-109% (n=4)

Holzwarth (1996a) determined the suitability of analytical method AE-24 for residues in glufosinate-tolerant maize shoots. Recoveries of spiked analyte over the concentration range 0.05-5 mg/kg were glufosinate 60-99% (n=6), NAG 65-85% (n=6) and MPP 53-99% (n=12). Holzwarth (1996b) also validated method AE-24 for residues in maize grain from plants susceptible to glufosinate-ammonium. Recoveries over the concentration range 0.05-5 mg/kg were: glufosinate 81-95% (n=6), NAG 68-82% (n=6) and MPP 54-94% (n=11).

Snowdon and Taylor (1995b) validated method AE-24 for glufosinate-tolerant maize, achieving LODs of 0.05 mg/kg for each analyte. Recoveries are shown in Table 33.

Table 33. Recoveries by analytical method AE-24 from transgenic maize commodities (Snowdon and Taylor, 1995b).

Commodity	Glufosinate		MPP		NAG	
	spiking levels, mg/kg	Recovery range, %	spiking levels, mg/kg	Recovery range, %	spiking levels, mg/kg	Recovery range, %
Maize shoots	0.05 2.0 50	61-105 (n=7)	0.05 2.0 50	69-85 (n=14)	0.05 2.0 50	76-132 (n=7)
Cobs	0.05 0.50	64-104 (n=5)	0.05 0.50	66-89 (n=10)	0.05 0.50	80-113 (n=5)
Cob/spadix	0.05 0.50	95-131 (n=4)	0.05 0.50	74-105 (n=10)	0.05 0.50	97-122 (n=5)

Czarnecki (1995c) validated analytical method AE-24A for maize and its processed products. The method differs from AE-24 in omitting the cation-exchange column step before derivatization. Consequently, the compounds appear as a single peak and are reported as a combined residue. Recoveries were acceptable for maize grain, forage and fodder.

Niedzwiadek and Bertrand (1995b) validated analytical method AE-24A for residues in glufosinate-tolerant rape seed commodities (Table 34). The LOD was 0.05 mg/kg for each analyte in each sample.

Table 34. Recoveries by method AE-24A from transgenic rape seed commodities (Niedzwiadek and Bertrand 1995b).

Commodity	Glufosinate		MPP		NAG	
	spiking levels, mg/kg	Recovery range, %	spiking levels, mg/kg	Recovery range, %	spiking levels, mg/kg	Recovery range, %
Rape shoot	0.05 0.50 10	92-114 (n=15)	0.05 0.50 10	76-115 (n=30)	0.05 0.50 10	91-119 (n=15)
Rape pod	0.05 0.50 5.0	87-108 (n=15)	0.05 0.50 5.0	71-109 (n=30)	0.05 0.50 5.0	88-120 (n=15
Rape straw	0.05 0.50	95-116 (n=10)	0.05 0.50	71-108 (n=20)	0.05 0.50	101-134 (n=10)
Rape seed	0.05 0.50	76-109 (n=10)	0.05 0.50	82-117 (n=20)	0.05 0.50	99-118 (n=10)

Idstein *et al.* (1987b) extracted glufosinate and MPP from soya beans and derivatized the compounds with trimethyl orthoacetate. After clean-up on a silica gel cartridge, the residues were measured by GLC with a flame photometric detector. Idstein *et al.* (1987a extracted glufosinate and MPP from milk by dialysis and completed the analysis in the same way. Schuld (1988) extracted the residues from hens' eggs with water, washed the aqueous extract with dichloromethane and hexane to remove lipids and then followed a similar procedure.

Sochor *et al.* (1987a) extracted rape seed with water and removed oil with dichloromethane before proceeding with the Idstein method. Sochor *et al.* (1987b) extracted the residues from fats by an initial water/dichloromethane partition before following the remainder of the procedure. Sochor *et al.* (1988) extracted animal organs with water, precipitated high MW co-extractives with acetone and continued with the Idstein method, achieving practical LODs of 0.05-0.1 mg/kg.

The method used for glufosinate in The Netherlands relies on extraction of samples with water and clean-up by dialysis and ion-exchange. The residue is measured by GLC with an FPD after derivatization with trimethyl orthoacetate. Recoveries are 70-80% and the LOD 0.05 mg/kg.

Stability of pesticide residues in stored analytical samples

Information was made available on the frozen storage stability of glufosinate and its metabolites in genetically modified maize and processed commodities, soya bean and processed commodities, dairy

cow tissues and milk, eggs and chicken tissues, susceptible maize grain, and transgenic rape seed and sugar beet root.

Belcher (1995a) determined the stability of glufosinate and its metabolites in genetically modified maize commodities (hulls, grits, flour and refined maize oil) during frozen storage for 12 months (Table 35). The report did not state the storage conditions. Residues were generally stable for 12 months, but glufosinate decreased by 30% and NAG by 19% in refined maize oil.

Table 35. Recoveries of glufosinate, NAG and MPP from maize commodities spiked with 0.5 mg/kg and stored for periods up to 12 months under frozen conditions (Belcher 1995a). Results are means of duplicate samples, uncorrected for analytical recovery.

Storage, months	glufosinate ammonium, racemate				NAG, racemate, free acid				MPP, free acid			
	hulls	grits	flour	oil, refined	hulls	grits	flour	oil, refined	hulls	Grits	flour	oil, refined
0	88%	92%	94%	83%	80%	101%	87%	102%	82%	93%	73%	89%
3	81%	84%	71%	67%	91%	89%	82%	94%	92%	81%	86%	84%
6	76%	74%	83%	68%	90%	93%	93%	85%	87%	86%	85%	79%
12	87%	88%	103%	58%	81%	93%	108%	83%	77%	81%	75%	76%

Belcher (1996b) determined the stability of glufosinate and its metabolites in genetically modified maize grain, fodder and forage during frozen storage for 24 months (Table 36). Residues were generally stable. The report did not include the storage conditions.

Table 36. Recoveries of glufosinate, NAG and MPP from maize commodities spiked with 0.5 mg/kg and stored for periods up to 24 months under frozen conditions (Belcher 1996b). Results are means of duplicate samples, uncorrected for analytical recovery.

Storage, months	glufosinate ammonium, racemate			NAG, racemate, free acid			MPP, free acid		
	grain	fodder	forage	grain	fodder	forage	grain	fodder	forage
0	95%	87%	94%	99%	88%	96%	88%	91%	98%
3	89%	97%	91%	93%	89%	90%	86%	99%	97%
6	91%	91%	83%	109%	86%	100%	90%	91%	93%
12	83%	85%	76%	93%	95%	88%	94%	103%	105%
24	88%	79%	76%	101%	88%	78%	90%	89%	87%

Belcher (1995b) determined the stability of glufosinate and its metabolites in genetically modified soya bean meal, hulls and refined oil during frozen storage for 12 months (Table 37). In some samples of meal and hulls enzyme activity converted some of the added glufosinate to NAG. In these cases the sum of glufosinate and NAG was treated as the remaining residue and used for the calculation in Table 37. Residues were generally stable for the 12 months. The report did not specify the storage conditions.

Table 37. Recoveries of glufosinate, NAG and MPP from soya commodities spiked with 0.5 mg/kg and stored for periods up to 12 months under frozen conditions (Belcher 1995b). Results are means of duplicate samples, uncorrected for analytical recovery.

Storage, months	glufosinate ammonium, racemate			NAG, racemate, free acid			MPP, free acid		
	meal	hulls	oil, refined	meal	hulls	oil, refined	meal	hulls	oil, refined
0	68%	83%	79%	84%	91%	104%	113%	90%	87%
3	86%	86%	77%	92%	82%	86%	101%	96%	96%
6	93%	96%	78%	100%	104%	97%	90%	93%	82%
12	83%	94%	93%	78%	96%	103%	93%	95%	87%

Homogenized eggs, chicken tissue, milk and cow tissue samples were spiked with glufosinate-ammonium, NAG and MPP at 0.25 mg/kg and stored at -12°C to -27°C for 15 months (Crotts and McKinney, 1995, 1996; McKinney and Crotts, 1997). The results are shown in Table 38 and 39.All three compounds were stable for 15 months.

Table 38. Recoveries of glufosinate, NAG and MPP from homogenized eggs and chicken tissues spiked with 0.25 mg/kg and stored for periods up to 15 months under frozen conditions (Crotts and McKinney, 1995; McKinney and Crotts, 1997). Results are means of duplicate samples, uncorrected for analytical recovery.

Storage, months	Glufosinate ammonium, racemate			NAG, L-isomer, disodium			MPP, free acid		
	muscle	Liver	eggs	muscle	liver	eggs	muscle	liver	eggs
0	102%	106%	105%	103%	111%	102%	96%	88%	85%
1	104%	102%	109%	103%	102%	101%	89%	92%	82%
3	79%	81%	83%	76%	91%	81%	86%	87%	79%
15	102%	105%	100%	108%	108%	86%	90%	84%	87%

Table 39. Recoveries of glufosinate, NAG and MPP from milk and cow tissues spiked with 0.25 mg/kg and stored for periods up to 15 months under frozen conditions (Crotts and McKinney, 1996). Results are means of duplicate samples, uncorrected for analytical recovery.

Storage, months	glufosinate ammonium, racemate				NAG, L-isomer, disodium				MPP, free acid			
	kidneys	muscle	liver	whole milk	kidneys	muscle	liver	whole milk	kidneys	muscle	liver	whole milk
14	107			86	112			83	100			82
15		76	97			93	107			81	92	

Werner (1997a-d) spiked homogenized transgenic maize shoots, rape seed and sugar beet roots and susceptible maize grain with glufosinate-ammonium, NAG and MPP at 0.5 mg/kg (expressed as glufosinate free acid) and measured the stability of the residues stored deep-frozen (temperature not stated) for 24 months. Results are shown in Table. All three compounds were stable for 24 months.

Table 40. Recoveries of glufosinate, NAG and MPP from homogenized transgenic maize shoots, susceptible maize grain, transgenic rape seed and sugar beet root stored for periods up to 24 months under frozen conditions (Werner 1997a-d). Results are means of duplicate samples, uncorrected for analytical recovery.

Storage, months	glufosinate ammonium, racemate, % remaining				NAG, L-isomer, disodium, % remaining				MPP, free acid, % remaining			
	maize shoot	maize grain	rape-seed	sugar-beet	maize shoot	maize grain	rape-seed	sugar-beet	maize shoot	maize grain	rape-seed	sugar-beet
0	76	81	96	74	75	59	100	64	65	68	87	67
1		92		74		66		72		74		71
2	67		84		75		91		73		91	
3		80		67		83		67		61		63
4	75		85		60		97		51		90	
6				86	106[1]	68[1]	85[1]	79	108	78	71	80
12			103	94	100[1]	63[1]	88	96	96	73	81	77
15	87	100			68	58			57	84		
18	92	106	92	98	77	89	91	103	77	68	86	86
23			101				97				90	
23							104[1]				106	
24					91[1]	91[1]			86	88		
24	83	113		102	88	97		81	72	86		82

[1]Analyses by method AE-24A. % remaining is calculated as sum of glufosinate + NAG.

Definition of the residue

The current definition of the residue for glufosinate-ammonium is *Sum of glufosinate-ammonium and 3-[hydroxy(methyl)phosphinoyl]propionic acid, expressed as glufosinate (free acid).*

When glufosinate is used on glufosinate-tolerant crops a major part of the residue is *N*-acetyl-glufosinate (NAG). It should be included in the definition of the residue for enforcement because it is generally the main component of the residue and the same GLC derivative is produced in the analytical method from glufosinate and NAG, so unless the compounds are separated before derivatization they both appear in the GLC peak for their common derivative.

A suitable revised definition would be *Sum of glufosinate-ammonium, 3-[hydroxy(methyl)phosphinoyl]propionic acid and N-acetyl-glufosinate, expressed as glufosinate (free acid),* but this definition could not be adopted until *N*-acetyl-glufosinate had been toxicologically evaluated.

USE PATTERN

Glufosinate-ammonium is a non-selective herbicide registered for crop uses, including desiccation, and non-crop uses. It is used to control annual and perennial grasses and broad-leaved weeds in horticultural and agricultural crops. Glufosinate acts as an inhibitor of glutamine synthetase, which leads to poisoning of the plant by ammonia. For weed control in susceptible crops it must be used either before crop emergence or as a directed spray away from foliage.

Glufosinate resistance has been imparted to several agronomic crops by insertion of a gene that enables the plant to detoxify L-glufosinate (the active isomer) by acetylation to *N*-acetyl-L-glufosinate which is not herbicidal.

The use pattern is necessarily different for conventional crops and those with glufosinate tolerance.

Table 41. Registered uses of glufosinate-ammonium. Concentrations and rates are expressed in terms of the active ingredient glufosinate-ammonium.

Crop [1]	Country	Form	Application			Number	Growth stage [2]	PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl			
Almond	USA	120 g/l SL	directed	1.7	min spray vol 187 l/ha	max total 5.1 kg/ha/yr		14
Apple	Poland [4]	150 g/l SL		0.45-0.90	0.04-0.35	2		
Apricot	Poland [4]	200 g/l SL		0.60-1.2	0.06-0.60	2		
Asparagus	Germany [4]	200 g/l SL	spray	0.55	0.14-0.18	1		
Avocado	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Banana	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Banana	Malaysia	150 g/l SL	directed	0.3-0.5	0.11	4		14
Bean	Poland [4]	SL	spray HV	0.45-0.60	0.04-0.30	1	pre-emerg	
Blackberries	Netherlands	200 g/l 150 g/l	directed spray	0.75-1.0		2		-
Canola (tolerant)	Canada	150 g/l SL	foliar	0.30-0.50	min spray vol 110 l/ha	2	early bolting	[3]
Canola (tolerant)	Canada	150 g/l SL	foliar	0.60	min spray vol 110 l/ha	1	early bolting	[3]
Carambola (Starfruit)	Malaysia	150 g/l SL	directed	0.3-0.5	0.11	4		14
Carrot	Germany [4]	200 g/l SL	spray	0.55	0.14-0.18	1	pre-emerg	
Carrot	Poland [4]	SL	spray HV	0.45-0.60	0.04-0.30	1	pre-emerg	
Cashew nut	Malaysia	150 g/l SL	directed	0.3-0.5	0.11	4		14
Cherry	Poland [4]	150 g/l SL		0.45-0.90	0.04-0.35	2		
Currants	Germany [4]	200 g/l SL	spray with screening	0.92	0.15-0.31	1		14
Currants	Netherlands	200 g/l 150 g/l	directed spray	0.75-1.0		2		-
Durian	Malaysia	150 g/l SL	directed	0.3-0.5	0.11	4		14
Dwarf French beans	Germany [4]	200 g/l SL	spray between rows with screening	0.92	0.23-0.31	1		14
Dwarf French beans	Germany [4]	200 g/l SL	spraying	0.46	0.08-0.15	1	desiccation	14
Feijoa	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Field peas	Germany [4]	200 g/l SL	spraying	0.46	0.08-0.15	1	desiccation	14
Fruit trees	Netherlands	200 g/l 150 g/l	directed under trees	0.75-1.0		2		-
Gooseberries	Germany [4]	200 g/l SL	spray with screening	0.92	0.15-0.31	1		14
Gooseberries	Poland [4]	200 g/l SL		0.60-1.2	0.06-0.60	2		
Guava	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Guava	Malaysia	150 g/l SL	directed	0.3-0.5	0.11	4		14
Hazelnut	Italy	120 g/l SL	directed	0.5-1.6	0.08-0.78	max total 2.5 kg/ha/yr		-
Horse beans	Germany [4]	200 g/l SL	spraying	0.46	0.08-0.15	1	desiccation	14

Crop [1]	Country	Form	Application			Number	Growth stage [2]	PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl			
Jack fruit	Malaysia	150 g/l SL	directed	0.3-0.5	0.11	4		14
Kiwifruit	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Lamb's lettuce	Germany [4]	200 g/l SL	spray	0.55	0.14-0.18	1	pre-emerg	
Leek	Germany [4]	200 g/l SL	spray	0.55	0.14-0.18	1	pre-emerg	
Litchi	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Macadamia	USA	120 g/l SL	directed	1.7	min spray vol 187 l/ha	max total 5.1 kg/ha/yr		14
Maize	Germany [4]	200 g/l SL	spray	0.92	0.23-0.31	1	pre-sowing	
Maize	Germany [4]	200 g/l SL	spray, under leaf	0.92	0.23-0.31	1	stage 16-20	
Maize (tolerant)	Germany [5]	200 g/l SL	foliar	0.90	0.23-0.45	1	3-8 leaf	
Maize (tolerant)	Germany [5]	200 g/l SL	foliar	0.45	0.11-0.23	2[1]	8 leaf	
Maize (tolerant)	Canada	200 g/l SL	foliar	0.30-0.50	min spray vol 110 l/ha	2[2]	8-leaf	86 grain 20 (graze)
Maize (tolerant)	Portugal	200 g/l SL	foliar	0.40-0.80	rec spray vol 200-400 l/ha	2	2-4 leaf[3]	
Maize (tolerant)	USA	18.2%		0.20-0.36		2	60 cm	60 (corn forage) 70 (corn grain, fodder)
Maize (tolerant)	USA	200 g/l SL	foliar	0.23-0.41		2	60 cm	70 grain 70 maize fodder 60 maize forage
Mango	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Mango	Malaysia	150 g/l SL	directed	0.3-0.5	0.11	4		14
Onion	Germany [4]	200 g/l SL	spray	0.55	0.14-0.18	1	pre-emerg	
Onion	Poland [4]	SL	spray HV	0.45-0.60	0.04-0.30	1	pre-emerg	
Papaya	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Parsley	Poland [4]	SL	spray HV	0.45-0.60	0.04-0.30	1	pre-emerg	
Passion fruit	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Peach	Poland [4]	200 g/l SL		0.60-1.2	0.06-0.60	2		
Pear	Poland [4]	150 g/l SL		0.45-0.90	0.04-0.35	2		
Pecan	USA	120 g/l SL	directed	1.7	min spray vol 187 l/ha	max total 5.1 kg/ha/yr		14
Pineapple	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Plum	Poland [4]	200 g/l SL		0.60-1.2	0.06-0.60	2		
Potato	Germany [4]	200 g/l SL	spray	0.55	0.14-0.18	1	stage 8-9	
Potato	Germany [4]	200 g/l SL	spraying	0.46	0.08-0.15	1	desiccation	14
Potato	Netherlands	200 g/l 150 g/l	aerial parts, haulm kill	0.45-0.60		1		-
Potato	Netherlands	200 g/l 150 g/l	directed spray	0.45-1.0		1		-
Potato	Netherlands	200 g/l 150 g/l	pre-emergence	0.45-1.0		1		-

[1] Germany, split application on maize, 6 weeks interval.

[2] Maximum rate in the 2nd application is 0.40 kg ai/ha.

[3] For the second application, the maize should have no more than 8-10 leaves.

Crop [1]	Country	Form	Application			Number	Growth stage [2]	PHI, days
			Method	Rate, kg ai/ha	Spray conc. kg ai/hl			
Rambutan	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Rape seed	Poland [4]	SL	spray HV	0.38-0.50	0.04	0.25	desiccation	
Raspberries	Germany [4]	200 g/l SL	spray with screening	0.92	0.15-0.31	1		14
Raspberries	Netherlands	200 g/l 150 g/l	directed spray	0.75-1.0		2		-
Raspberries	Poland [4]	200 g/l SL		0.60-1.2	0.06-0.60	2		
Spinach	Netherlands	200 g/l 150 g/l	aerial parts, haulm kill	0.6-0.8		1		-
Soya beans (tolerant)	USA	200 g/l SL	foliar	0.23-0.41		2	bloom growth	70 seed forage[1]
Strawberries	Germany [4]	200 g/l SL	spray between rows with screening	0.73	0.18-0.24	1	stage 59	42
Strawberries	Germany [4]	200 g/l SL	spray with screening	0.73	0.12-0.24	1	after harvest	
Sugar beets	Germany [4]	200 g/l SL	spray	0.92	0.23-0.31	1	pre-sowing	
Sunflowers	Germany [4]	200 g/l SL	spraying	0.46	0.08-0.15	1	desiccation	14
Tree nuts	Australia	200 g/l SL	directed	0.2-1.0	spray vol 300-1000 l/ha			-
Walnut	Italy	120 g/l SL	directed	0.5-1.6	0.08-0.78	max total 2.5 kg/ha/yr		-
Walnut	USA	120 g/l SL	directed	1.7	min spray vol 187 l/ha	max total 5.1 kg/ha/yr		14
Winter rape	Germany [4]	200 g/l SL	spraying	0.46	0.08-0.15	1	desiccation	14

[1] tolerant: tolerant to glufosinate-ammonium
[2] growth stage at final application.
[3] Do not graze the treated crop or cut for hay; sufficient data are not available to support such use.
[4] Label not provided.
[5] Label not available. Registration document 4574-00 1 Sept 1998 provided.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue data from glufosinate-ammonium supervised residue trials on fruit, tree nuts and field crops are summarized in Tables 42-61.

Table 42	*Tropical fruits -avocado, mango, guava, carambola, papaya*. Australia, Malaysia.
Table 43	*Tree nuts – pecan, walnut, almond, hazel nuts macadamia*. USA, Italy, Australia.
Table 44	*Maize.* France, Germany, Italy, Spain.
Table 45	*Maize*. USA, Argentina.
Table 46	*Maize.* Canada.
Table 47	*Soya beans.* USA.
Table 48	*Rape seed.* France, Germany, UK.
Table 49	*Canola.* Canada.
Table 50	*Canola.* Australia.
Table 51	*Sugar beet root.* France, Germany, UK.
Table 52	*Sugar beet root.* USA.

[1] Do not feed treated green immature growing soya bean plants to livestock.

Table 53	*Maize forage and fodder.* France, Germany, Italy, Netherlands, Spain.
Table 54	*Maize forage and fodder.* USA, Argentina.
Table 55	*Maize forage and fodder.* Canada.
Table 56	*Rape seed forage and fodder.* France, Germany, UK.
Table 57	*Canola forage and fodder.* Australia.
Table 58	*Soya bean forage and fodder.* USA.
Table 59	*Sugar beet tops and leaves.* France, Germany, UK.
Table 60	*Sugar beet tops.* USA.
Table 61	*Almond hulls.* USA.

Where residues were not detected they are recorded as below the limit of determination (LOD), e.g. <0.05 mg/kg. Residue levels of glufosinate and its metabolites are generally expressed as glufosinate free acid. Residues, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOD, to 1 significant figure. Although all trials included control plots residues in control samples are recorded only when they exceeded the LOD. Residues are not corrected for recoveries. Recoveries were mainly in the range 70-120%.

Reports did not generally state whether residues in forage and fodder commodities were expressed on a fresh-weight or dry-weight basis.

Trials were fully reported as well as on summary sheets except the US trials on pecans and walnuts and the Italian trials on hazel-nuts. These trials were all reported on the detailed summary sheets used in Germany.

Glufosinate-ammonium is used as a directed spray for weed control in orchards, so that residues usually only occur in the crop through root uptake from the soil. Glufosinate-ammonium itself is not taken up, but the metabolite 3-methylphosphinicopropionic acid (MPP) can be taken up and translocated by the crop. This metabolite is not herbicidally active, so its residues may occur in the crop when glufosinate-ammonium is used as a directed spray.

In tropical fruit trials in Australia and Malaysia glufosinate-ammonium was applied to weeds around the trees as a directed spray. Plot sizes ranged from 1 tree to 1050 m^2. Intervals of 109 to 468 days of frozen sample storage elapsed before analysis. Residues are expressed on a pulp + peel basis for the avocados and mangoes in the Australian trials, but this does not influence the results which were mainly below the LOD.

Glufosinate-ammonium was used in tree nut trials in the USA with plot sizes ranging from 1 to 9 trees. The method of spraying was not clearly expressed, but was presumably a directed spray. Samples were held in frozen storage for intervals of 400 to 700 days before analysis. The method of application was also not stated for the hazel nut trials in Italy. Samples were held 240 days in frozen storage before analysis. Directed sprays were used in the macadamia trials in Australia. Samples from the year 1992 were analysed 28 days after harvest while those from 1995 were stored for almost a year.

In Canadian trials on resistant maize in 1993-95 glufosinate-ammonium was applied by ground rigs. Two different SL formulations were used, with applications at two rates and at different growth stages.

Glufosinate-ammonium was applied to resistant maize in an extensive set of trials in France from 1993 to 1996. Plot sprayers were used on plots of 11 to 79 m^2. Harvested samples were held in frozen storage for 520-670 days (1993), 310-340 days (1994), 160-230 days (1995) and 50-90 days (1996).

In a similar series of resistant maize trials in Germany from 1994 to 1996, plot sprayers were used on plots of 48-64 m^2. Frozen storage periods were 300-400 days (1994), 150 days (1995) and 30-70 days (1996).

Further European trials on resistant maize were conducted in 1994-96 in Italy, The Netherlands and Spain. Hand-carried plot sprayers were used in Italy and Spain on plots of 40-60 m^2. Information on the sprayers and plot sizes was not available for The Netherlands. Analytical samples were stored frozen for 330-340 days (1994) and 50-150 days (1995-96).

Glufosinate-ammonium was extensively tested on resistant maize in US trials in 1993-1995. In 1993 it was applied by portable and tractor-mounted plot sprayers to plots of 28-186 m^2. In 1995 ground rigs and plot sprayers were used on plots of 110-740 m^2. Harvested samples were held in frozen storage for 280-330 days (1993) and 190-280 days (1995) before analysis. Some of the higher rate treatments in trial ER-93-USA-01 (Virginia and South Dakota) suffered phytotoxicity so grain samples were limited or unavailable for analysis.

Resistant soya beans were determined with glufosinate-ammonium in trials in many States of the USA in 1994-1996. Plots of 70-95 m^2 were sprayed with CO_2 pressurised backpack or plot sprayers in 1994. In 1995 tractor-mounted ground rigs and backpack or bicycle sprayers were used on plots of 40-130 m^2 and in 1996 backpack or plot sprayers were used on 90-280 m^2 plots. Harvested samples were held in frozen storage before analysis for 200-280 days (1994), 100-300 days (1995) and 200-230 days (1996).

Glufosinate-ammonium was determined on resistant rape seed in a supervised residue trial programme in France in 1993, 1994 and 1996. Plot sprayers were used on plots of 18-72 m^2. Periods of frozen storage of samples before analysis were 570-640 days (1993), 380-550 days (1994) and 112 days (1996). In trials on resistant rape seed in Germany (1994 and 1996) and the UK (1993 and 1996) glufosinate-ammonium was applied by plot sprayers to plots of 48-100 m^2 in Germany and 20-80 m^2 in the UK. Frozen storage periods for samples were 1 year and 2 months (1994 and 1996 respectively) in Germany and 600 days and 2 months (1993 and 1995 respectively) in the UK.

Glufosinate-ammonium was applied with boom sprayers to plots of 30-42 m^2 of resistant canola in Australian trials in 1996. Harvested samples were stored for 170-240 days before analysis.

Resistant canola was treated with glufosinate-ammonium in a series of supervised residue trials in Canada in 1993 and 1994. CO_2 powered backpack, plot sprayers and tractor-mounted rigs were used on plots of 24-80 m^2. Samples were held for 170-280 days (1993) and 80 days (1994) in frozen storage before analysis. No field reports were available for trials in report A53770 (Bertrand, 1993). One canola trial (A57514, MacDonald, 1996d) was not evaluated because the data sheets were from a barley trial. It was not clear in some of the canola trials whether the residues were expressed as glufosinate free acid or as the metabolites,.

Glufosinate-ammonium was applied with plot sprayers to plots of 38-76 m^2 of resistant sugar beet in French trials in 1995 and 1996. Harvested samples were stored for 120-300 days under frozen conditions before analysis. In similar trials in the UK in 1996-97 glufosinate-ammonium was applied by plot sprayer or knapsack to 48-64 m^2 plots. Frozen samples were stored for 150-280 days before analysis.

In a US trial programme on resistant sugar beet in 1995 and 1996 glufosinate-ammonium was applied by ground rig, backpack or bicycle sprayer to plots of 17-300 m^2. Storage periods for frozen samples were 240-320 days (1995) and 130-170 days (1996).

Table 42. Glufosinate residues in tropical fruits resulting from supervised trials in Australia and Malaysia. In the Australian trials on avocados and mangoes residues are expressed on a pulp + peel basis. Residues in samples from replicate plots are shown separately. Double-underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

FRUIT, country, year	Application				PHI, days	Residues, mg/kg, as glufosinate free acid		Ref
(variety)	Form	kg ai/ha	kg ai/hl	no.		glufosinate	MPP	
AVOCADO								
Australia (Qld) 1991 (Sharwil)	SL	1.0	0.4	1	7 12 21	<u><u><0.1</u></u> (2) <0.1 (2) <0.1 (2)	<0.1 (2) <0.1 (2) <0.1 (2)	A59027 QD 27/90
Australia (Qld) 1991 (Sharwil)	SL	2.0	0.8	1	7 12 21	<0.1 (2) <0.1 (2) <0.1 (2)	<0.1 (2) <0.1 (2) <0.1 (2)	A59027 QD 27/90
Australia (Qld) 1996 (Fuerte)	SL	0.6	0.33	2	0 14 24 28	<0.05 (2) <0.05 (2) <0.05 (2) <0.05 (2)	<0.05 (2) <0.05 (2) <0.05 (2) <0.05 (2)	A59026 AU QD 26
Australia (Qld) 1996 (Fuerte)	SL	1.2	0.67	2	0 14 24 28	<u><u><0.05</u></u> (2) <0.05 (2) <0.05 (2) <0.05 (2)	<0.05 (2) <0.05 (2) <0.05 (2) <0.05 (2)	A59026 AU QD 26
Australia (Qld) 1995 (Haas)	SL	0.6	0.35 0.30	2	0 14 24 31	<0.05 (2) <0.05 (2) <0.05 (2) <0.05 (2)	<0.05 (2) <0.05 (2) <0.05 (2) <0.05 (2)	A59025 AU QD 24
Australia (Qld) 1995 (Haas)	SL	1.2	0.69 0.60	2	0 14 24 31	<u><u>0.06</u></u> <0.05 <0.05 (2) <0.05 (2) <0.05 (2)	<0.05 (2) <0.05 (2) <0.05 (2) <0.05 (2)	A59025 AU QD 24
MANGO								
Australia (Qld) 1991 (Kensington Pride)	SL	1.0	0.4	1	7 14 20	<u><u><0.1</u></u> <0.1 <0.1	<0.1 <0.1 <0.1	A59030 QD 21/90
Australia (Qld) 1991 (Kensington Pride)	SL	2.0	0.8	1	7 14 20	<0.1 <0.1 <0.1	<0.1 <0.1 <0.1	A59030 QD 21/90
Australia (Qld) 1994-5 (Fascell)	SL	0.6	0.21	2	0 14 21 28	<0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	A59029 AU QD 12
Australia (Qld) 1994-5 (Fascell)	SL	1.2	0.41	2	0 14 21 28	<u><u><0.05</u></u> <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	A59029 AU QD 12
Australia (Qld) 1995 (Kensington Pride)	SL	0.6	0.2	2	0 14 21 28	<0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	A59028 AU QD 13-94
Australia (Qld) 1995 (Kensington Pride)	SL	1.2	0.4	2	0 14 21 28	<u><u><0.05</u></u> <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	A59028 AU QD 13-94
GUAVA								
Malaysia (Perat) 1995 (“with seeds”)	SL	0.50	0.11	4	0 3 7 14 21	<0.05 <0.05 <0.05 <u><u><0.05</u></u> <0.05	<0.05 <0.05 <0.05 <0.05 <0.05	A57294 ER95MYS8800201 02

FRUIT, country, year	Application				PHI, days	Residues, mg/kg, as glufosinate free acid		Ref
(variety)	Form	kg ai/ha	kg ai/hl	no.		glufosinate	MPP	
Malaysia (Perat) 1995 (“with seeds”)	SL	1.0	0.22	4	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	A57294 ER95MYS8800201 03
Malaysia (Perat) 1995 (“seedless”)	SL	0.50	0.11	4	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	A57294 ER95MYS8800202 02
Malaysia (Perat) 1995 (“seedless”)	SL	1.0	0.22	4	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	A57294 ER95MYS8800202 03
CARAMBOLA								
Malaysia (Selangor) 1995 (Bio)	SL	0.50	0.11	3	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	A57295 ER95MYS8800101 02
Malaysia (Selangor) 1995 (Bio)	SL	1.0	0.22	3	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	A57295 ER95MYS8800101 03
Malaysia (Perat) 1995 (Bio)	SL	0.50	0.11	3	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	A57295 ER95MYS8800102 02
Malaysia (Perat) 1995 (Bio)	SL	1.0	0.22	3	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	A57295 ER95MYS8800102 03
PAPAYA								
Australia (Qld) 1996 (Rictor Gold)	SL	0.6	0.26	2	0 14 21 30	<0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	A59285 AU QD 25
Australia (Qld) 1996 (Rictor Gold)	SL	1.2	0.52	2	0 14 21 30	<0.05 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	A59285 AU QD 25

Table 43. Glufosinate residues in tree nuts resulting from supervised trials in Australia, Italy and the USA. Analyses on samples from replicate plots are shown separately. Double-underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

NUT, country, year (variety)	Application				PHI, days	Residues, mg/kg, as glufosinate free acid		Ref
	Form	kg ai/ha	kg ai/hl	No.		glufosinate	MPP	
PECAN								
USA (GA) 1985 (Witchita)	SL	1.7		3	14	<0.05 (3)	<0.05 (3)	A48446 16-GA-85-011
USA (GA) 1985 (Witchita)	SL	3.4		3	14	<0.05 (3)	<0.05 (3)	A48446 16-GA-85-011
USA (LA) 1985 (Cape Fear)	SL	1.7		3	21	<0.05 (3)	<0.05 (3)	A48446 16-LA-85-002
USA (LA) 1985 (Cape Fear)	SL	3.4		3	21	<0.05 (3)	<0.05 (3)	A48446 16-LA-85-002
USA (NM) 1985 (Western Schley)	SL	1.7		3	14	<0.05 (3)	<0.05 (3)	A48446 07-NM-85-032
USA (NM) 1985 (Western Schley)	SL	3.4		3	14	<0.05 (3)	<0.05 (3)	A48446 07-NM-85-032
WALNUT								
USA (CA) 1984 (English)	SL	1.6 +1.2		2	14	<0.05 (3)	<0.05 (3)	A34230
USA (CA) 1984 (English)	SL	3.2 +2.4		2	14	<0.05 (3)	<0.05 (3)	A34231
USA (CA) 1984 (English)	SL	1.6 +2×1.2		3	14	<0.05 (3)	<0.05 (3)	A34232
USA (CA) 1984 (English)	SL	3.2 +2×2.4		3	14	<0.05 (3)	<0.05 (3)	A34233
USA (CA) 1985 (Serr)	SL	1.7		3	14	<0.05 (3)	<0.05 (3)	A35706
USA (CA) 1985 (Serr)	SL	4.5		3	14	<0.05 (3)	<0.05 (3)	A35707
USA (CA) 1985	SL	1.7		3	14	<0.05 (3)	<0.05 (3)	A35708
USA (CA) 1985	SL	3.4		3	14	<0.05 (3)	<0.05 (3)	A35709
USA (CA) 1985 (Serr)	SL	1.7		3	11	<0.05 (3)	<0.05 (3)	A35710
USA (CA) 1985 (Serr)	SL	3.4		3	11	<0.05 (3)	<0.05 (3)	A35711
USA (CA) 1985 (Serr)	SL	1.7		3	14	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-019
USA (CA) 1985 (Serr)	SL	3.4		3	14	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-019
USA (CA) 1985 (Serr)	SL	1.7		3	14	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-024
USA (CA) 1985 (Serr)	SL	3.4		3	14	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-024
USA (CA) 1985 (Serr)	SL	1.7		3	11	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-038

NUT, country, year (variety)	Application				PHI, days	Residues, mg/kg, as glufosinate free acid		Ref
	Form	kg ai/ha	kg ai/hl	No.		glufosinate	MPP	
USA (CA) 1985 (Serr)	SL	3.4		3	11	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-038
ALMOND								
USA (CA) 1985 (Carmel)	SL	1.7		3	15	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-018
USA (CA) 1985 (Carmel)	SL	3.4		3	15	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-018
USA (CA) 1985 (Special)	SL	1.7		3	15	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-018
USA (CA) 1985 (Special)	SL	3.4		3	15	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-018
USA (CA) 1985 (Non-Peril)	SL	1.7		3	14	<0.05 (3)	0.07 0.07 0.07	A48446 07-CA-85-028
USA (CA) 1985 (Non-Peril)	SL	3.4		3	14	<0.05 (3)	0.14 0.22 0.17	A48446 07-CA-85-028
USA (CA) 1985 (Non-Peril)	SL	1.7		3	14	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-037
USA (CA) 1985 (Non-Peril)	SL	3.4		3	14	<0.05 (3)	<0.05 (3)	A48446 07-CA-85-037
HAZEL-NUT								
Italy 1985 (Gentile Del Piemonte)	SL	1.5	0.38	2	19	<0.05	<0.05	A35935
Italy 1985 (Nocchione)	SL	1.5	0.38	2	19	<0.05	<0.05	A35936
Italy 1985 (Gentile Del Piemonte)	SL	1.5	0.38	2	19	<0.05	<0.05	A35937
Italy 1985 (Gentile Del Piemonte)	SL	1.5	0.38	2	19	<0.05	<0.05	A35938
Italy 1985 (Gentile Del Piemonte)	SL	1.5	0.38	2	19	<0.05	<0.05	A35938
MACADAMIA NUT								
Australia 1992 (var 434)	SL	1.0	0.8	4	1 7 14 21	<0.1 (2) <0.1 (2) <0.1 (2) <0.1 (2)	<0.1 (2) <0.1 (2) <0.1 (2) <0.1 (2)	A59031 QDI/92 92/1222
Australia 1992 (var 434)	SL	2.0	1.6	4	1 7 14 21	<0.1 (2) <0.1 (2) <0.1 (2) <0.1 (2)	<0.1 (2) <0.1 (2) <0.1 (2) <0.1 (2)	A59031 QDI/92 92/1222
Australia 1995 (var 344)	SL	1.2	0.63	2	0 16 27	<0.05 (2) <0.05 (2) <0.05 (2)	<0.05 (2) <0.05 (2) <0.05 (2)	A59032 QD20-94 95/3715
Australia 1995 (var 344)	SL	2.4	1.3	2	0 16 27	<0.05 (2) <0.05 (2) <0.05 (2)	<0.05 (2) <0.05 (2) <0.05 (2)	A59032 QD20-94 95/3715

Table 44. Glufosinate residues in maize resulting from supervised trials in France, Germany, Italy and Spain. Double-underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Country, Year, Variety	Application				PHI days	Residues as glufosinate free acid, mg/kg			Ref
	form	kg ai/ha	kg ai/hl	no[1]		glufosinate	MPP	NAG[2]	
France 1993 (LH163×LH164-×F2×T14)	SL	0.45	0.23	2 GS25	145	<0.05	<0.05		A54225 ER93ECN550 FRA000402
France 1993 (LH206×LH74-×F2×T14)	SL	0.45	0.23	2 GS24	145	<0.05	<0.05		A54225 ER93ECN550 FRA000502
France 1993 (MLC 2101/T14)	SL	0.45	0.23	2 GS24	128	<0.05	<0.05		A54225 ER93ECN550 FRA000102
France 1993 (MLC 2101/T14)	SL	0.45	0.23	2 GS24	147	<0.05	<0.05		A54225 ER93ECN550 FRA000202
France 1993 (MLC 2101/T14)	SL	0.45	0.23	2 GS24	145	<0.05	<0.05		A54225 ER93ECN550 FRA000302
France 1994 ((LH82(4)T25)sf (-2)xSH298	SL	0.45	0.23	2 GS25	123	<0.05	<0.05		A54229 ER94ECN550 FRA000202
France 1994 (F6×M1)	SL	0.45	0.23	2 GS25	119	<0.05	<0.05		A54226 ER93ECS550 FRA000102
France 1994 (LH824xT25)AF4x-B73)	SL	0.45	0.15	2 GS25	120	<0.05	<0.05		A54230 ER94ECS550 FRA000102
France 1994 (SH298(LH82(4)T25)sf(2))	SL	0.45	0.23	2 GS25	119	<0.05	<0.05		A54229 ER94ECN550 FRA000102
France 1995 (F2xLH82-T25)	SL	0.45	0.23	2 GS18	100	<0.05	<0.05		A56445 ER95ECN550 FRA000202
France 1995 (F2xLH82-T25)	SL	0.45	0.23	2 GS19	100	<0.05	<0.05		A56445 ER95ECN550 FRA000203
France 1995 (tolerant 039866)	SL	0.45	0.23	2 GS18	106	<0.05	<0.05		A56445 ER95ECN550 FRA000103
France 1995 (tolerant 039866)	SL	0.45	0.23	2 GS18	106	<0.05	<0.05		A56445 ER95ECN550 FRA000102
France 1995 (transgenic hybrid)	SL	0.53	0.27	2 GS18	116	<0.05	<0.05	<0.05	A54760 ER95ECS550 FRA000103
France 1995 (transgenic hybrid)	SL	0.53	0.27	2 GS18	116	<0.05	<0.05	<0.05	A54760 ER95ECS550 FRA000102
France 1995 (transgenic hybrid)	SL	0.45	0.23	2 GS19	119	<0.05	<0.05	<0.05	A54760 ER95ECS550 FRA000203
France 1995 (transgenic hybrid)	SL	0.45	0.23	2 GS19	119	<0.05	<0.05	<0.05	A54760 ER95ECS550 FRA000202
France 1996 (transgenic hybrid)	SL	0.60	0.24	2 GS18	104	<0.05	<0.05		A58191 ER96ECN550 FRA000102
France 1996 (transgenic hybrid)	SL	0.80	0.32	2 GS18	104	<0.05	<0.05		A58191 ER96ECN550 FRA000103

Country, Year, Variety	Application				PHI days	Residues as glufosinate free acid, mg/kg			Ref
	form	kg ai/ha	kg ai/hl	no[1]		glufosinate	MPP	NAG[2]	
France 1996 (transgenic hybrid)	SL	0.45	0.18	2 GS18	104	<0.05	<0.05		A58191 ER96ECN550 FRA000104
France 1996 (transgenic hybrid)	SL	0.60	0.24	2 GS18	105	<0.05	<0.05		A58191 ER96ECN550 FRA000302
France 1996 (transgenic hybrid)	SL	0.80	0.32	2 GS18	105	<0.05	<0.05		A58191 ER96ECN550 FRA000303
France 1996 (transgenic hybrid)	SL	0.45	0.18	2 GS18	105	<0.05	<0.05		A58191 ER96ECN550 FRA000304
France 1996 (transgenic hybrid)	SL	0.60	0.24	2 GS18	110	<0.05	<0.05		A58191 ER96ECN550 FRA000402
France 1996 (transgenic hybrid)	SL	0.80	0.32	2 GS18	110	<0.05	<0.05		A58191 ER96ECN550 FRA000403
France 1996 (transgenic hybrid)	SL	0.45	0.18	2 GS18	110	<0.05	<0.05		A58191 ER96ECN550 FRA000404
France 1996 (transgenic hybrid)	SL	0.45	0.18	2 GS18	129	<0.05	<0.05		A58190 ER96ECS550 FRA000302
France 1996 (transgenic hybrid)	SL	0.60	0.24	2 GS18	129	<0.05	<0.05		A58190 ER96ECS550 FRA000303
France 1996 (transgenic hybrid)	SL	0.80	0.32	2 GS18	129	<0.05	<0.05		A58190 ER96ECS550 FRA000304
France 1996 (transgenic hybrid)	SL	0.60	0.24	2 GS19	103	<0.05	<0.05		A58191 ER96ECN550 FRA000202
France 1996 (transgenic hybrid)	SL	0.80	0.32	2 GS19	103	<0.05	<0.05		A58191 ER96ECN550 FRA000203
France 1996 (transgenic hybrid)	SL	0.45	0.18	2 GS19	103	<0.05	<0.05		A58191 ER96ECN550 FRA000204
France 1996 (transgenic hybrid)	SL	0.45	0.18	2 GS19	130	<0.05	<0.05		A58190 ER96ECS550 FRA000102
France 1996 (transgenic hybrid)	SL	0.60	0.24	2 GS19	130	<0.05	<0.05		A58190 ER96ECS550 FRA000103
France 1996 (transgenic hybrid)	SL	0.80	0.32	2 GS19	130	<0.05	<0.05		A58190 ER96ECS550 FRA000104
France 1996 (transgenic hybrid)	SL	0.45	0.18	2 GS19	140	<0.05	<0.05		A58190 ER96ECS550 FRA000202
France 1996 (transgenic hybrid)	SL	0.60	0.24	2 GS19	140	<0.05	<0.05		A58190 ER96ECS550 FRA000203
France 1996 (transgenic hybrid)	SL	0.80	0.32	2 GS19	140	<0.05	<0.05		A58190 ER96ECS550 FRA000204
Germany 1995 (LH82^4T25sf^4F2)	SL	0.45	0.15	2 GS18	99	<0.05 (2)	<0.05 (2)		A56445 ER95ECN550 DEU010202

Country, Year, Variety	Application				PHI days	Residues as glufosinate free acid, mg/kg			Ref
	form	kg ai/ha	kg ai/hl	no[1]		glufosinate	MPP	NAG[2]	
Germany 1995 (LH82^4T25sf^4SH-298)	SL	0.45	0.15	2 GS18	90	<0.05	<0.05		A56445 ER95ECN550 DEU050102
Germany 1995 (LH82^4T25sf^4xF2)	SL	0.45	0.15	2 GS18	113	<0.05 (2)	<0.05 (2)		A56445 ER95ECN550 DEU010102
Germany 1996 (F2 hybride - LH-82)	SL	0.60	0.20	2 GS18	126	<0.05	<0.05		A58191 ER96ECN550 DEU050102
Germany 1996 (F2 hybride - LH-82)	SL	0.80	0.27	2 GS18	126	<0.05	<0.05		A58191 ER96ECN550 DEU050103
Germany 1996 (F2 hybride - LH-82)	SL	0.45	0.15	2 GS18	126	<0.05	<0.05		A58191 ER96ECN550 DEU050104
Germany 1996 (Facet - transgen)	SL	0.60	0.20	2 GS18	125	<0.05	<0.05		A58191 ER96ECN550 DEU060102
Germany 1996 (Facet - transgen)	SL	0.80	0.27	2 GS18	125	<0.05	<0.05		A58191 ER96ECN550 DEU060103
Germany 1996 (Facet - transgen)	SL	0.45	0.15	2 GS18	125	<0.05	<0.05		A58191 ER96ECN550 DEU060104
Germany 1996 (Facet - transgen)	SL	0.60	0.20	2 GS18	122	<0.05	<0.05		A58191 ER96ECN550 DEU060202
Germany 1996 (Facet - transgen)	SL	0.80	0.27	2 GS18	122	<0.05	<0.05		A58191 ER96ECN550 DEU060203
Germany 1996 (Facet - transgen)	SL	0.45	0.15	2 GS18	122	<0.05	<0.05		A58191 ER96ECN550DEU 060204
Germany 1996 (LH82 T25 SF x F2)	SL	0.60	0.20	2 GS19	137	<0.05	<0.05		A58191 ER96ECN550 DEU010202
Germany 1996 (LH82 T25 SF x F2)	SL	0.80	0.27	2 GS19	137	<0.05	<0.05		A58191 ER96ECN550 DEU010203
Germany 1996 (LH82 T25 SF x F2)	SL	0.45	0.15	2 GS19	137	<0.05	<0.05		A58191 ER96ECN550 DEU010204
Germany 1996 (LH82 T25 SF x Fe)	SL	0.60	0.20	2 GS19	145	<0.05	<0.05		A58191 ER96ECN550 DEU010102
Germany 1996 (LH82 T25 SF x Fe)	SL	0.80	0.27	2 GS19	145	<0.05	<0.05		A58191 ER96ECN550 DEU010103
Germany 1996 (LH82 T25 SF x Fe)	SL	0.45	0.15	2 GS19	145	<0.05	<0.05		A58191 ER96ECN550 DEU010104
Italy 1994 ([B73(4)xT14]sfx - LM82)	SL	0.45	0.15	2 GS23	105	<0.05	<0.05		A54230 ER94ECS550 ITA000102
Italy 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	108	<0.05	<0.05	<0.05	A54760 ER95ECS550 ITA000104
Italy 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	104	<0.05	<0.05	<0.05	A54760 ER95ECS550 ITA000204

Country, Year, Variety	Application				PHI days	Residues as glufosinate free acid, mg/kg			Ref
	form	kg ai/ha	kg ai/hl	no[1]		glufosinate	MPP	NAG[2]	
Italy 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	108	<0.05	<0.05	<0.05	A54760 ER95ECS550 ITA000103
Italy 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	104	<0.05	<0.05	<0.05	A54760 ER95ECS550 ITA000203
Italy 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	108	<0.05	<0.05	<0.05	A54760 ER95ECS550 ITA000102
Italy 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	104	<0.05	<0.05	<0.05	A54760 ER95ECS550 ITA000202
Italy 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS18	100	<0.05	<0.05		A58190 ER96ECS550 ITA000105
Italy 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS18	124	<0.05	<0.05		A58190 ER96ECS550 ITA000205
Italy 1996 (transgenic hybrid)	SL	0.60	0.20	2 GS18	100	<0.05	<0.05		A58190 ER96ECS550 ITA000103
Italy 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS18	100	<0.05	<0.05		A58190 ER96ECS550 ITA000104
Italy 1996 (transgenic hybrid)	SL	0.60	0.20	2 GS18	124	<0.05	<0.05		A58190 ER96ECS550 ITA000203
Italy 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS18	124	<0.05	<0.05		A58190 ER96ECS550 ITA000204
Italy 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS19	104	<0.05	<0.05		A58190 ER96ECS550 ITA000305
Italy 1996 (transgenic hybrid)	SL	0.60	0.20	2 GS19	104	<0.05	<0.05		A58190 ER96ECS550 ITA000303
Italy 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS19	104	<0.05	<0.05		A58190 ER96ECS550 ITA000304
Spain 1994 (LH119-6xT14/LH82)	SL	0.45	0.15	2 GS25	102	<0.05	<0.05		A54230 ER94ECS550 ESP000102
Spain 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	95	<0.05	<0.05	<0.05	A54760 ER95ECS550 ESP000103
Spain 1995 (transgenic hybrid)	SL	0.45	0.15	2 GS18	95	<0.05	<0.05	<0.05	A54760 ER95ECS550 ESP000102
Spain 1996 (transgenic hybrid)	SL	0.45	0.15	2 GS18	95	<0.05	<0.05		A58190 ER96ECS550 ESP000102
Spain 1996 (transgenic hybrid)	SL	0.60	0.20	2 GS18	95	<0.05	<0.05		A58190 ER96ECS550 ESP000103
Spain 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS18	95	<0.05	<0.05		A58190 ER96ECS550 ESP000104
Spain 1996 (transgenic hybrid)	SL	0.45	0.15	2 GS18	96	<0.05	<0.05		A58190 ER96ECS550 ESP000202

Country, Year, Variety	Application				PHI days	Residues as glufosinate free acid, mg/kg			Ref
	form	kg ai/ha	kg ai/hl	no[1]		glufosinate	MPP	NAG[2]	
Spain 1996 (transgenic hybrid)	SL	0.60	0.20	2 GS18	96	<0.05	<0.05		A58190 ER96ECS550 ESP000203
Spain 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS18	96	<0.05	<0.05		A58190 ER96ECS550 ESP000204
Spain 1996 (transgenic hybrid)	SL	0.45	0.15	2 GS18	113	<0.05	<0.05		A58190 ER96ECS550 ESP000302
Spain 1996 (transgenic hybrid)	SL	0.60	0.20	2 GS18	113	<0.05	<0.05		A58190 ER96ECS550 ESP000303
Spain 1996 (transgenic hybrid)	SL	0.80	0.27	2 GS18	113	<0.05	<0.05		A58190 ER96ECS550 ESP000304

[1]. GS: growth stage at final treatment
GS18 8 leaves unfolded
GS19 9 or more leaves unfolded
GS23 5th leaf unfolded
GS24 6th leaf unfolded
GS25 7th leaf unfolded
[2] included in glufosinate-ammonium result.

Table 45. Glufosinate residues in transgenic maize resulting from supervised trials in the USA and Argentina. Analyses of replicate samples are reported individually. Double-underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

Country, Year, Variety	Application				PHI	Residues, mg/kg, as glufosinate free acid[2]			Ref
	form	kg ai/ha	kg ai/hl	No[1]	days	glufosinate-ammonium	MPP	NAG	
USA (NE) 1993 (LH59 X LH51) (LH119)(4) X T14)	SL	1.8 +2.6	1.9 +2.8	2 (60 cm)	96	NQ	NQ	NQ	ER-93-USA-01-NE-02
USA (IA) 1993 (LH216) (LH119) (4) X (T14))	SL	1.5	1.6	1 (60 cm)	100	NQ	NQ	NQ	ER-93-USA-01-IA-02
USA (IA) 1993 (LH216) X (LH119) (4) × T14)	SL	0.36		1 (30 cm)	118	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IA-01
USA (IA) 1993 (LH216) X (LH119) (4) × T14)	SL	0.50		1 (30 cm)	114	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IA-01
USA (IA) 1993 (LH216) X (LH119) (4) × T14)	SL	0.50		1 (60 cm)	114	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IA-01
USA (IL) 1993 (LH 59x LH51)(LH119) (4) X T14)	SL	0.36	0.39	1 (30 cm)	100	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IL-01
USA (IL) 1993 (LH 59x LH51)(LH119) (4) X T14)	SL	0.50	0.54	1 (30 cm)	100	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IL-01
USA (IL) 1993 (LH 59x LH51)(LH119) (4) X T14)	SL	0.50	0.54	1 (60 cm)	95	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IL-01

Country, Year, Variety	Application				PHI	Residues, mg/kg, as glufosinate free acid[2]			Ref
	form	kg ai/ha	kg ai/hl	No[1]	days	glufosinate-ammonium	MPP	NAG	
USA (IL) 1993 (LH 59x LH51)(LH119) (4) X T14)	SL	0.36 +0.50	0.39 +0.54	2 (60 cm)	95	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IL-01
USA (NE) 1993 (LH 59x LH51)(LH119) (4) X T14)	SL	0.36		1 (30 cm)	107	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-NE-01
USA (NE) 1993 (LH 59x LH51)(LH119) (4) X T14)	SL	0.50	0.48	1 (30 cm)	107	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-NE-01
USA (NE) 1993 (LH 59x LH51)(LH119) (4) X T14)	SL	0.50	0.48	1 (60 cm)	95	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-NE-01
USA (IN) 1993 [LH119(4) X T14] X LH216)	SL	0.36	0.39	1 (30 cm)	115	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IN-01
USA (IN) 1993 [LH119(4) X T14] X LH216)	SL	0.50	0.54	1 (30 cm)	115	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IN-01
USA (IN) 1993 [LH119(4) X T14] X LH216)	SL	0.36 +0.50	0.39 +0.54	2 (60 cm)	106	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-IN-01
USA (ND) 1993 (LH85 X LH160) (LH119)(4) X T14)	SL	0.36	0.39	1 (30 cm)	107	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-ND-01
USA (ND) 1993 (LH85 X LH160) (LH119)(4) X T14)	SL	0.50	0.54	1 (30 cm)	107	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-ND-01
USA (ND) 1993 (LH85 X LH160) (LH119)(4) X T14)	SL	0.36 +0.50	0.39 +0.54	2 (60 cm)	97	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-ND-01
USA (ND) 1993 (LH85 X LH160) (LH119)(4) X T14)	SL	0.36	0.39	1 (30 cm)	117	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-ND-01
USA (ND) 1993 (LH85 X LH160) (LH119)(4) X T14)	SL	0.50	0.54	1 (60 cm)	117	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-ND-01
USA (ND) 1993 (LH85 X LH160) (LH119)(4) X T14)	SL	0.36 +0.50	0.39 +0.54	2 (31 cm)	107	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-ND-01
USA (MO) 1993 (LH216) X (LH119(4) X (T14))	SL	0.36	0.39	1 (30 cm)	97	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-MO-01
USA (MO) 1993 (LH216) X (LH119(4) X (T14))	SL	0.36 +0.50	0.39 +0.54	2 (60 cm)	95	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-MO-01
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X T14)	SL	0.36	0.39	1 (30 cm)	119	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-01
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X T14)	SL	0.36	0.39	1 (30 cm)	129	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-01
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X T14)	SL	0.36 +0.50	0.39 +0.54	2 (60 cm)	106	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-01
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X T14)	SL	0.36 +0.50	0.39 +0.54	2 (60 cm)	116	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-01

Country, Year, Variety	Application				PHI	Residues, mg/kg, as glufosinate free acid[2]			Ref
	form	kg ai/ha	kg ai/hl	No[1]	days	glufosinate-ammonium	MPP	NAG	
USA (SD) 1993 (LH85 X LH160) (LH119)(4) X T14))	SL	0.36	0.71	1 (30 cm)	95	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-SD-01
USA (SD) 1993 (LH85 X LH160) (LH119)(4) X T14))	SL	0.50	0.99	1 (30 cm)	95	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-SD-01
USA (SD) 1993 (LH85 X LH160) (LH119)(4) X T14))	SL	0.36 +0.50	0.71 +0.99	2 (60 cm)	95[1]	NQ (3)	NQ (3)	0.07 (3)	ER-93-USA-01-SD-01
USA (VA) 1993 (LH216) (LH119)(4) X T14)	SL	0.36	0.71	1 (30 cm)	106[2]	NQ	NQ	NQ	ER-93-USA-01-VA-01
USA (VA) 1993 (LH216) (LH119)(4) X T14)	SL	0.50	0.99	1 (30 cm)	106[6]	NQ	NQ	NQ	ER-93-USA-01-VA-01
USA (VA) 1993 (LH216) (LH119)(4) X T14)	SL	0.36 +0.50	0.71 +0.99	2 (60 cm)	97[6]	NQ	NQ	0.07	ER-93-USA-01-VA-01
USA (MO) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (12-15 cm)	123	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-MO-02
USA (MO) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (25-30 cm)	118	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-MO-02
USA (MO) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (40-45 cm)	111	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-MO-02
USA (MO) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (55-66 cm)	101	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-MO-02
USA (MO) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (80-90 cm)	97	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-MO-02
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (12-15 cm)	127	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-02
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (25-30 cm)	120	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-02
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (40-45 cm)	114	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-02
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (55-66 cm)	96	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-02
USA (CA) 1993 (LH59 X LH51) (LH119)(4) X (T14))	SL	0.50	0.53	1 (80-90 cm)	96	NQ (3)	NQ (3)	NQ (3)	ER-93-USA-01-CA-02
USA (FL) 1994 (LH51 X LH210) (LH119(4) X T14)	SL	0.40 +0.50	0.43 +0.54	2 (2-14 leaves)	69	NQ	NQ	glu	BK-94R-01-FL-01
USA (IA) 1994 (LH59 X LH51) (LH119(4) X T14))	SL	0.41 +0.53	0.44 +0.57	2 (60 cm)	122	NQ	NQ	glu	BK-94R-01-IA-01

[1] This crop suffered some phytotoxicity and the yield of grain was limited.

[2] Each of the treatments in the Virginia trials suffered from phytotoxicity and unusual weather conditions, resulting in limited collection of samples.

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