STUDY TITLE

Magnitude of the Residue of Glufosinate-Ammonium in Transgenic Field Corn Tolerant to Glufosinate-Ammonium

DATA REQUIREMENTS

OPPTS 860.1500

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STUDY COMPLETED ON

January 10, 2002

PERFORMING LABORATORY

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LABORATORY STUDY ID

000290

STATEMENT OF NO DATA CONFIDENTIALITY CLAIMS

Compound: Glufosinate-Ammonium

Title: Magnitude of the Residue of Glufosinate-Ammonium in Transgenic Field Corn Tolerant to Glufosinate-Ammonium

No claim of confidentiality is made for any information contained in this study on the basis of its falling within the scope of FIFRA Section 10 (d)(1)(A)(B), or (C).*

Company: Dow AgroSciences LLC

Company Agent: P. L. Hunst

Title: Regulatory Manager

Signature: Penny L. Hunst

Date: 8/8/01

*In the United States, the above statement supersedes all other statements of confidentiality that may occur elsewhere in this report.

THIS DATA MAY BE CONSIDERED CONFIDENTIAL IN COUNTRIES OUTSIDE THE UNITED STATES.

STATEMENT OF COMPLIANCE WITH GOOD LABORATORY PRACTICE STANDARDS

Title: Magnitude of the Residue of Glufosinate-Ammonium in Transgenic Field Corn Tolerant to Glufosinate-Ammonium

Study Initiation Date:	17-May-2000	Study Completion Date:	10-Jan-2002
Experimental Start Date:	24-May-2000	Experiment Termination Date:	21-Apr-2001

This report represents data generated after the effective date of the EPA FIFRA Good Laboratory Practice Standards.

United States Environmental Protection Agency
Title 40 Code of Federal Regulations Part 160
FEDERAL REGISTER, August 17, 1989

Organisation for Economic Co-Operation and Development
ISBN 92-64-12367-9, Paris 1982

All aspects of this study were conducted in accordance with the requirements for Good Laboratory Practice Standards, 40 CFR 160, with the following exceptions: Documentation is incomplete by GLP standards for weather/meteorological data, irrigation data, crop and pesticide history, plot preparation, pesticide maintenance data, weights used for scale measurement (000290IA1), sample weights (000290IL1) and equipment maintenance logs (000290MN1). In addition, copies provided as analytical raw data were not stamped as verified copies, and the specific details of the storage stability sample creation were not documented.

Penny K. Hunst — 1/10/2002
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Penny K. Hunst — 1/10/2002
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Dow AgroSciences Quality Assurance Unit
Good Laboratory Practice Statement Page

Compound: Glufosinate Ammonium **Study ID:** 000290

Title: Magnitude of the Residue of Glufosinate-Ammonium in Transgenic Field Corn Tolerant to Glufosinate-Ammonium

Study Initiation Date: 5/17/2000 **Study Completion Date:** 1/10/2002

GLP Quality Assurance Inspections

Date of GLP Inspection(s)	Date Reported to the Study Director and to Management	Phases of the Study which received a GLP Inspection by the Quality Assurance Unit
5/17/2000	5/17/2000	Protocol review
5/24/2000	6/7/2000 & 6/13/2000	Planting/000290MN1 (Warnke Research Services-WRS)
5/26/2000	6/9/2000 & 7/18/2000	Planting/000290OH1 (Ag Consultants, Inc. – ACI)
5/31/2000	6/5/2000	Planting/000290IA1 (Bennett Agricultural Research & Consulting-BARC)
6/21/2000	6/23/2000	Calibration/000290IA1 (BARC)
6/21/2000	6/23/2000	Cry1F protein expression determination/000290IA1 (BARC)
6/28/2000	9/5/2000 & 9/7/2000	Application #1/000290OH1 (ACI)
7/6/2000	7/10/2000	Application/000390IN1/(Heartland Technologies-HLD)
7/6/2000	7/24/2000 & 7/30/2000	2nd Liberty application/000290IA2/(Land O'Lakes Research Farm-LOL)
7/12/2000	7/19/2000	Facility Inspection of HLD
7/12/2000	7/19/2000	Post application and pre-sampling/000290IN1 (HLD)
7/18/2000	7/26/2000	Facility Inspection of Ag Consultants, Inc.
7/18/2000	7/26/2000	Check field study notebook, test plot, inspect equipment maintenance records/000290OH1 (ACI)
9/6/2000	9/13/2000	Forage sampling, field IP audit, HLD
9/6/2000	9/19/2000 & 9/21/2000	Forage sampling/000290IL1/(Alvey Agricultural Research -AAR)
9/7/2000	9/14/2000	Forage sampling/000290IA2 (LOL)
9/13/2000	9/19/2000	Facility inspection (AAR)
9/14/2000	9/26/2000	Sampling/000290MN1 (WRS)

10/3,5/10/2000	11/13/2000 & 11/14/2000	Anion Exchange col. Cleanup, filtering, concentration, test system receipt, and test system preparation/(McKenzie/Wright Laboratories-MWL)
11/1/2000	11/13/2000 & 11/14/2000	Review of raw data/000290MN1 (Warnke)
11/10/2000	11/13/2000	Field databook/000290IA1 (BARC)
11/14/2000	11/15/2000 & 11/17/2000	Final data audit/000290IL1 (AAR)
11/15/2000	11/15/2000 & 5/11/2001	Raw data/final notebook review/000290 (LOL)
11/27/2000	12/18/2000 &12/17/2000	Final Data/000290OH1/(ACI)
12/28/2000	12/28/2000 & 5/22/2001	Trial notebook review/000290IN1/(HLD)
3/26/2001	4/4/2001	Facility Inspection – LOL
3/27/2001	4/4/2001	Facility Inspection – BARC
4/11/2001	4/18/2001	Facility Inspection – WRS
5/18/2001	5/18/2001 & 5/24/2001	Analytical report review/(MWL)
6/11,13/2001	6/19/2001 & 6/20/2001	Final report review/000290/(MWL)
12/6, 12, 13/2001	1/10/2002	Final report audit

QUALITY ASSURANCE STATEMENT:

The Quality Assurance Unit has reviewed the final study report and has determined that the report reflects the raw data generated during the conduct of this study.

Patricia A. Houtman D. Keyes — 10-January-2002 1/10/02

Patricia A. Houtman/David Keyes
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Quality Assurance

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Magnitude of the Residue of Glufosinate-Ammonium in Transgenic Field Corn Tolerant to Glufosinate-Ammonium

ABSTRACT

Glufosinate-ammonium, the active ingredient in Liberty herbicide, is registered for use on corn tolerant to glufosinate-ammonium for the control of a broad spectrum of emerged broadleaf weeds, annual, and perennial grasses. Tolerances for residues of glufosinate-ammonium and its metabolites (expressed as glufosinate acid equivalence) have been established at 4.0 μg/g for field corn forage, 0.2 μg/g for field corn grain, and 6.0 μg/g for field corn stover.

The transgenic field corn used for this study had been genetically modified by the insertion of a gene isolated from *Bacillus thuringiensis (Bt)* which expresses the Cry1F protein which imparted insecticidal resistance, and the gene, phosphinothricin acetyltransferase (PAT) which imparted herbicidal resistance to glufosinate-ammonium, the active ingredient in Liberty herbicide.

Liberty herbicide was applied to transgenic field corn two times for a total application rate of 62 ounces/A (0.81 lb ai/A) per growing season, the maximum label use rate. The first application of 34 oz (0.44 lb ai/A) was applied as an over-the-top foliar broadcast spray to transgenic corn at 10-15 inches tall (approximate V4-V5 growth stage). The second application of 28 oz (0.37 lb ai/A) was applied as an over-the-top foliar broadcast spray or ground application to transgenic corn at 22-48 inches tall (approximate V7-V9 growth stage). The formulation was commercially available Liberty herbicide, a soluble liquid, that contained approximately 19.5% w/w glufosinate-ammonium as the active ingredient. Applications were made in 14.7-25.6 gal/A using water as a diluent and contained ammonium sulfate at a rate of 2.5-3.9 lb. a.i./A.

Field trials were conducted at 6 sites (IL, IN, MN, OH, and 2 in IA) in the EPA region 5 starting in May 2000 and ending in October 2000. Samples of raw agricultural commodities (grain and

stover) were collected at normal harvest times. Forage was collected at the late dough/early dent stage. No decline samples were collected.

Samples were analyzed by McKenzie/Wright Laboratories for residues of glufosinate-ammonium and two metabolites by Aventis CropScience method RAM number BK/05/95 with modifications, which employs measurement by gas chromatography with flame photometric detection in the phosphorous mode (GC/FPD). The limits of detection (LOD) and quantitation (LOQ) for the analytes and matrices were 0.015-0.020 µg/g and 0.050-0.066 µg/g, respectively.

Field trial average residues of glufosinate-ammonium and its metabolites were 0.06-1.40 µg/g in forage, ND-<0.05 µg/g in grain, and 0.06-2.20 µg/g in stover. The magnitude of residue of glufosinate acid equivalence in transgenic field corn forage, grain, and stover did not exceed the current established tolerances. Field residues observed were approximately 4 times lower than the established tolerances.

The following table summarizes the residues of glufosinate-ammonium and its metabolites (expressed as glufosinate acid equivalence) found in transgenic field corn forage, grain, and stover after treatment with Liberty herbicide at 62 ounces/a (0.81 lb ai/a) per growing season.

State	EPA Region	PHI[a] (Days)	Application Rate (lb a.i./A)	Residue[b] (µg/g)
Forage				
Current Tolerance				**4.0**
IA	5	62	0.797	0.37
IA	5	63	0.808	0.14
IN	5	62	0.818	0.46
OH	5	61	0.786	0.26
IL	5	47	0.803	1.40
MN	5	69	0.806	(0.062)[c]
			Mean =	0.45
Grain				
Current Tolerance				**0.2**
IA	5	89	0.807	ND[d]
IA	5	98	0.802	ND
IN	5	104	0.804	ND
OH	5	104	0.814	ND
IL	5	87	0.830	(0.052)
MN	5	101	0.804	ND
			Mean =	ND
Stover				
Current Tolerance				**6.0**
IA	5	89	0.807	0.07
IA	5	98	0.802	0.14
IN	5	104	0.804	0.45
OH	5	104	0.814	0.21
IL	5	87	0.830	2.20
MN	5	101	0.804	0.06
			Mean =	0.52

[a] Pre-harvest interval in days.
[b] Residue values corrected for average set recovery. Average of duplicate samples taken.
[c] ( ) = value between the LOD and LOQ, a region of greater uncertainty.
[d] ND = Not detected. Less than the LOD.

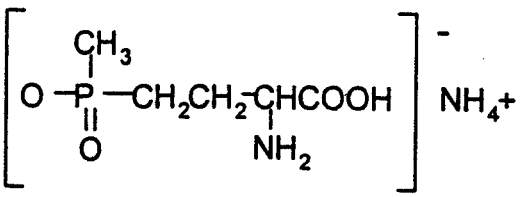
INTRODUCTION

Corn has been modified by the insertion of genes encoding for the insecticidally active protein from *Bacillus thuringiensis (Bt)* designated Cry1F, and the herbicide tolerant protein phosphinothricin acetyltransferase (PAT) which inactivates glufosinate-ammonium. Glufosinate-ammonium, the active ingredient in Liberty herbicide, is registered for use on corn tolerant to glufosinate-ammonium for the control of a broad spectrum of emerged broadleaf weeds, annual, and perennial grasses (1).

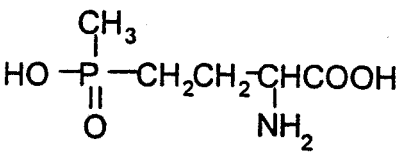
This study was performed to verify that the magnitude of residues of glufosinate-ammonium and its metabolites (expressed as glufosinate acid equivalence) did not exceed the tolerance levels of 4.0 μg/g for field corn forage, 0.2 μg/g for field corn grain, and 6.0 μg/g for field corn stover. Liberty herbicide was applied at the maximum label rate for use with transgenic corn encoded to express PAT protein. This study was not required by the EPA but was designed to conform as closely as possible to EPA Pesticide Assessment Guideline OPPTS 860.1500 with the exception of the number of site locations and no decline study.

The maximum label use rate for Liberty herbicide on corn is 34 ounces (0.44 lb ai/A) followed by 28 ounces (0.37 lb ai/A) for a total of 62 ounces (0.81 lb ai/A) per growing season. The minimum post harvest interval is 60 days for forage, and 70 days for grain and fodder (stover). The first application is to be applied at the V-3 to V-4 stage of growth while the second application is to be applied V-6 to V-7 stage of growth or up to 36 inches. Applications are to be over-the-top broadcast applications or directed broadcast application using drop nozzle from emergence until the corn is 24" tall (V-7) and/or as a ground application and drop nozzle for corn 24" to 36" tall (2). This application regimen protects the crops from possible phytotoxicity damage which may result as the crop matures.

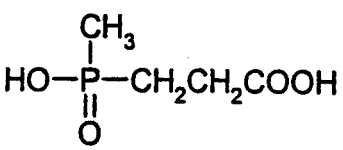
The chemical structures, common names, IUPAC names, and CAS numbers for glufosinate-ammonium, the free acid, and its metabolites are summarized below.



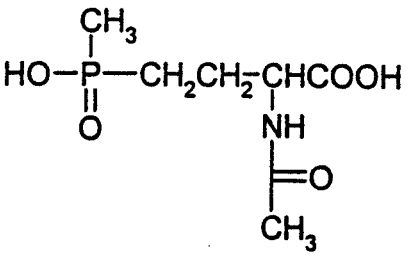
Glufosinate-ammonium
IUPAC Name: Ammonium-DL-homoalanin-4-yl-(methyl)-phosphinate
Aventis CropScience Identification Number: HOE-039866
CAS No. 77182-82-2



Glufosinate
IUPAC Name: DL-homoalanin-4-yl-(methyl)phosphinic acid
Free Acid of Glufosinate-ammonium
Aventis CropScience Identification Number: HOE-035956
CAS No. 51276-47-2



IUPAC Name: 3-Methylphosphinico-propionic acid
Metabolite of Glufosinate-ammonium (referred to as M1)
Aventis CropScience Identification Number: HOE-061517
CAS No. 15090-23-0



IUPAC Name: DL-2-acetamino-4-methylphosphinico-butanoic acid
Common Name: N-Acetyl Glufosinate
Metabolite of Glufosinate-ammonium (referred to as M2)
Aventis CropScience Identification Number: HOE-085355
CAS No. Not Applicable

EXPERIMENTAL

Test Substance

Liberty herbicide (TSN 102208, lot no. LJ02081402), containing a concentration of 19.5 % w/w glufosinate-ammonium (~200 g a.i./L), was used in this study (3).

Trial Location and General Site Data

A total of 6 field trials for the magnitude of residue study of glufosinate-ammonium in corn were conducted from May to October 2000. The sites were located within major growing regions for corn within the United States (Figure 1). Site information is shown in Table 1.

Site Preparation and Trial Layout

Untreated (Treatment 01) and treated plots (Treatments 02 and 03) were established at each site with a buffer zone of at least 50 ft between the control plot and the treated plots, and at least 20

feet between the treated plots. A minimum of 660 ft separated the transgenic plots from any non-transgenic corn. Each plot measurement was a minimum of 0.01 acre that was large enough to produce a representative sample without sampling plot end rows or border rows. In addition, the plot was sufficiently large to allow accurate application of the pesticide in a manner that represents a commercial application technique. Plots were maintained using standard local cultural practices that included maintenance chemical applications of fertilizers and approved pesticides. Field data with details of planting information and maintenance chemicals are given in Table 2.

Seed Source and Confirmation of Protein Expression

The field corn seed, provided by Mycogen Seeds, was from TC1507, lot number PR00203. This seed produced plants capable of expressing the Cry1F protein for insecticidal activity and the PAT protein for herbicidal resistance.

The presence of the Cry1F protein was confirmed using a lateral flow test kit obtained from Strategic Diagnostic, Inc (SDI). A minimum of 12 plants per plot were randomly selected at the V3 through V5 stage (crop height from 6 inches to 14 inches). All the plants tested positive for the Cry1F protein. The presence of the PAT protein was confirmed by the plants being tolerant (no phytotoxicity) to the Liberty herbicide applications.

Application

The spray mixtures were prepared according to label instructions except for tank mixtures prepared by site location 000290IA2. For each application (except for site location 000290IA2), a measured amount of test substance was added to the spray tank while approximately one-half of the required amount of water was being agitated. Ammonium sulfate was then added at a rate of 2.5-3.9 lb. a.i./A (target rate of 3.0 lb. a.i./A) to the spray tank and the contents were agitated. The measuring device was rinsed with water to ensure complete transfer of the test substance to

the spray tank and the rinses were added to the spray tank. After agitating, the spray tank was filled to the required volume with water and mixed. For tank mixes prepared at site 000290IA2, all of the water was added followed by the ammonium sulfate. The mixture was agitated for approximately 1 minute, then all of the test substance was added, and then the mixture was agitated for an additional minute. Amounts of test substance and total volume of water used in the final spray mixture for each site are shown in Appendix A.

Applications to corn less than 24" tall were made as an over-the-top foliar broadcast spray in 14.7-20.2 gal/A using typical tractor-mounted or backpack sprayer ground application equipment. Applications to corn greater than 24" tall were made as a ground application in 14.9-25.6 gal/A, using drop nozzles to avoid spraying into whorl or leaf axils, using typical tractor-mounted or back sprayer ground application equipment. Details of the application information are shown in Table 3.

All application rates were within the protocol specification, ranging from 97 to 104% of the target application rate. Calculations of the application rates are summarized in Appendix A.

The crop vigor was good at all sites for all application times. No phytotoxicity was observed at any application time, except for one application. Site 000290IA1 exhibited a small amount of phytotoxicity after application. For the first application, a slight discoloration of approximately 20% of the plants was observed with one plant dying in treatment 03 (grain and stover sampling plot).

No precipitation occurred after the applications for a minimum of at least one day. Liberty herbicide is rainfast 4 hours after application to most weed species.

Climatological Conditions

Climatological data during the crop growing season for each of the field sites are presented in Table 4. For comparison, the table includes historical data of the temperature and precipitation from weather stations near these sites (4).

The average temperatures of all sites were similar to the historical temperatures with the maximum variations being 3.6 °F less than the historical temperature (IA2, Webster City, June 2000) to 4.7 °F greater than the historical temperature (IA1, Richland, September 2000).

The amount of rainfall received at the sites varied significantly in cases from the historical rainfall. For IA1 (Richland, Iowa), the rainfall for the month of June was 191% above the historical while the month of August was only 47% of the historical rainfall. For IA2 (Webster City, Iowa), the rainfall for the entire study was below the historical rainfall, ranging from 51 to 80% of the historical rainfall. For IN1 (Noblesville, Indiana) the rainfall for June, August and September, ranged between 168 to 198% above the historical rainfall. For OH1 (New Holland, Ohio), the rainfall for June, July and September ranged from 161 to 211% above the historical rainfall while the month of August was only 57% of the historical rainfall. For IL1 (Carlyle, Illinois), all the months were above the historical rainfall, ranging from 123 to 367% of the historical rainfall. For MN1 (Geneva, Minnesota), the rainfall for the months of June, July, and August was above the historical rainfall, ranging from 120 to 251% of the historical rainfall while September and October were approximately 45% of the historical rainfall.

No crop damage was noted for any of the sites due to weather conditions.

Sample Collection

Samples were collected first from the untreated (control) plot, followed by the sample collection from the treated plots. A single composite sample was collected from the control plot while duplicate composite samples were taken independently from the treated plot. Each sample was

collected from at least 12 randomly selected areas over the entire plot, excluding the end or border rows (except in Indiana). For the control forage sample at site 000290IN1, stalks containing ears of immature corn were taken from the border rows due to deer grazing on the immature corn ears of the untreated plot. The border rows were sampled to insure sufficient ears remaining for grain harvest.

Forage Sample Collection

Forage samples were collected 47 to 69 days after the last application (Treatment 2) at the late dough to early dent stage. Composite sample weights of forage were between 6.5-9.5 lb, as shown in Table 5.

Forage samples were collected by randomly selecting 12 stalks per sample from the plot. Stalks were hand cut approximately 2 inches above the soil. Three groups, 4 stalks per group, were sectioned into three equal sections—top, middle, and bottom. One section from each group was composited to comprise the sample. The remainder of the stalks was discarded back into the plot. For some sites, only the section to be composited was removed from the plot, leaving the remaining 2/3 of the stalk in the plot. After compositing, the stalk sections were further cut into smaller pieces using hand sheers, a knife, or shredded directly into labeled polyethylene-lined cloth residue sampling bags.

Grain and Stover Sample Collection

Grain and stover samples were collected 87 to 104 days after the last application (Treatment 3) at crop maturity. Composite sample weights were between 2.2-6.5 lb for grain, and between 2.0-5.2 lb for stover, as shown in Table 5.

Grain and stover samples were collected by randomly selecting 12 stalks per sample from the plot. The ears of corn were removed from the stalks, and 12 ears were mechanically shelled directly into the residue sampling bags, or were shelled by hand, winnowed, mixed by hand. The sample size stipulated by the protocol was transferred to labeled residue sample bags. The stover

sample stalks were sectioned and composited as described for the forage, except for site 000290IL1. The entire stalks at this site were transferred to labeled residue bags.

Sample Handling and Shipping

Each sample was labeled with a unique number which was used for identification and tracking throughout the study. Samples of forage, grain, and stover were put in freezers or coolers with dry ice within about 2 hours of collection. Samples were maintained frozen until shipped by freezer truck, or overnight delivery in insulated boxes with dry ice.

ANALYTICAL

Sample Receipt, Storage, and Preparation

Unique Dow AgroSciences sample numbers were used to track the samples throughout shipment, receipt, preparation, storage, and analysis. Samples were identified in groups based upon matrix, test site location, and collection date. Table 6 summarizes the dates of sampling, shipment from the field, receipt at Dow AgroSciences, preparation at Dow AgroSciences, shipment to and receipt at McKenzie/Wright Laboratories, and analysis.

Upon receipt at Dow AgroSciences, the samples were inspected for physical condition, checked for identification with the pre-assigned unique sample numbers, and then logged into Dow AgroSciences Sample Tracking and Reporting (DSTAR) system. All samples were received frozen. The samples were stored in temperature-monitored freezers, being removed only for sample preparation and shipment to McKenzie/Wright laboratories. Prior to the preparation date, the samples were maintained frozen as the bulk field samples.

Frozen samples of forage and stover were ground using an Agvise Model 2001 hammermill equipped with a 1/4-inch screen, while samples of grain were ground using an Agvise Model 2001 hammermill equipped with a 3/16- or 1/4-inch screen. Samples were maintained frozen throughout the grinding process by treating them with liquid nitrogen before grinding. After appropriate mixing of each sample, two subsamples of at least 200 g each were transferred to high density polyethylene (HDPE) freezer containers. One subsample was designated for analysis while the second was stored as a long-term retainer sample. All movements of samples at Dow AgroSciences were documented within the DSTAR system.

The analytical samples were shipped by overnight delivery in insulated boxes with dry ice to McKenzie/Wright laboratories. Upon receipt at McKenzie/Wright, the samples were inspected for physical condition and were checked for identification with the Dow AgroSciences sample numbers. All samples were received frozen. The samples were stored frozen in temperature-monitored freezers, being removed only for sample analysis.

The temperature of the freezers used for sample storage at the Dow AgroSciences and McKenzie/Wright facilities averaged approximately -20 °C and -15 °C, respectively. No significant deviations from this temperature were observed over the storage period between sample receipt and analysis.

All sample analyses for residues of glufosinate-ammonium and its metabolites were conducted between October 23, 2000 and April 21, 2001.

Residue Sample Analysis

Samples were analyzed by McKenzie/Wright Laboratories in Phoenix, Arizona. All samples were analyzed using Aventis CropScience analytical method, RAM number BK/05/95 with modifications (5). A summary of the analytical procedure used by the contract facility is provided in Appendix B, section ANALYTICAL METHODOLOGY. The complete method is attached in Appendix C. Residues of glufosinate-ammonium and its metabolites were expressed

as glufosinate acid equivalents. The total residue concentration was determined by summing the individual residues and reporting as glufosinate acid equivalents. Details of the sample analysis, method recoveries, and calculations to generate the residue results are described in Appendix B.

Storage Stability

Storage stability was determined by fortifying control forage, grain, and stover with glufosinate-ammonium and its metabolites. The control material used for fortification was processed at Dow AgroSciences. These controls were then shipped to McKenzie/Wright for frozen storage stability sample preparation. At McKenzie/Wright, portions of control forage sample numbers 32887101 and 33060401 were composited into a single sample for use as the bulk control. Grain sample number 33064701 and stover sample number 33065501 were also used as bulk control samples. Dates of control shipment to the contract facility and compositing are presented in Table 6.

Details of the frozen storage stability samples preparation and analysis is described in Appendix B, section LABORATORY SAMPLE HANDLING, Storage Stability Study Set-up. Residue results of the storage stability samples are summarized in Table 7. Residues of glufosinate-ammonium and its metabolites were found to be stable in forage, grain, and stover with 90% or greater of residues remaining after 77, 59, and 91 days, respectively, when stored frozen at approximately -15 °C.

As shown in Table 6, the forage, grain, and stover field samples were stored for a maximum of 72, 61, and 85 days, respectively, prior to analysis. Based on the results of the frozen storage stability samples, the residues of glufosinate-ammonium and its metabolites were stable during this time period. The storage stability for the grain was 2 days less than the maximum storage time of the field samples. With 93% or greater stability showing at day 59 for all analytes in grain, residue stability at 61 days should be acceptable with no significant loss during 2 days.

Statistical Analysis

Statistical analysis used to evaluate the analytical data included the calculation of average or mean, standard deviation, and regression analysis. These are shown in Appendix B.

RESULTS AND DISCUSSION

Usage According to Labeling

All aspects of this study were performed according to labeling instructions with the following exceptions.

At test site 000290IA2, the tank mixes were not prepared according to labeling. The actual spray tank mixing procedure used was previously discussed in the Section EXPERIMENTAL-Application.

At test site 000290IA2, the second application was applied to corn 42-48" tall (instead of maximum 36" tall) due to rain and poor soil drying which prevented the equipment from entering the field to make the second application. During this same time period, temperatures were favorable which resulted in rapid crop growth. No crop injury was observed after the second application.

At test site 000290IL1, the forage sample was harvested at 47 days post harvest interval (PHI) instead of 60 days PHI. The late dough/early dent stage occurred at an earlier time than the other test sites. This lower PHI represents a worse-case scenario than a 60 day PHI so results were used to support this study.

Analytical Method Performance

A summary of the recoveries is presented in the following table. Detailed summaries of the recoveries are presented in Appendix B, Table 4. Recovery results indicated that the method was suitable for defining residues of glufosinate-ammonium (Parent) and its metabolites (M1 and M2) in the samples. Representative chromatograms of calibration standards, reagent blank, field control samples, fortified controls, and field treated samples are shown in Appendix B, Figures 1-13.

Matrix and Compound		Fortification Range (µg/g)	LOD[a]	LOQ[b]	Mean	Standard Deviation	No. Analyses
Forage							
Parent	HOE-039866	0.05-4.0	0.015	0.050	98	13	13
M1	HOE-061517	0.05-4.0	0.015	0.050	100	11	13
M2	HOE-085355	0.05-4.0	0.019	0.063	71	12	13
Grain							
Parent	HOE-039866	0.05-0.20	0.019	0.064	95	11	14
M1	HOE-061517	0.05-0.20	0.019	0.064	105	11	14
M2	HOE-085355	0.05-0.20	0.015	0.050	78	5	14
Stover							
Parent	HOE-039866	0.05-6.0	0.015	0.050	88	7	13
M1	HOE-061517	0.05-6.0	0.015	0.050	100	8	13
M2	HOE-085355	0.05-6.0	0.018	0.061	75	11	13

[a] LOD = Limit of detection. For Parent and M1, the LOD was calculated using results from both analytes since the method cannot determine the analytes separately.

[b] LOQ = Limit of quantitation. For Parent and M1, the LOQ was calculated using results from both analytes since the method cannot determine the analytes separately.

Residue Results

Forage samples were collected at the late dough/early dent stage with PHI from 47 to 69 days. As shown in Table 8, total residues of glufosinate acid equivalent ranged from <0.06 to 1.50 µg/g in the forage samples. Grain and stover samples were collected at crop maturity with PHI from 87 to 104 days. Residues of glufosinate acid equivalents ranged from ND to

<0.06 µg/g in the grain samples, and from <0.05 to 2.61 µg/g in the stover samples as shown in Tables 9 and 10, respectively.

CONCLUSION

The magnitude of residue of glufosinate-ammonium and its metabolites (reported as glufosinate acid equivalence) in transgenic field corn forage, grain, and stover did not exceed the current established tolerances of 4.0 µg/g for forage, 0.2 µg/g for grain, and 6.0 µg/g for stover. Field residues observed were approximately 4 times lower than the established tolerances. The maximum label use rate of 62 oz was applied to the corn in two applications. The crops were harvested at the normal harvest times for the transgenic crop variety. The transgenic corn used for this study contained a gene for Cry1F protein which imparted insecticidal resistance, and a gene for PAT which imparted herbicidal resistance to glufosinate-ammonium, the active ingredient in Liberty herbicide.

ARCHIVING

Final report, field data books, and all raw data (verified and signed copies) associated with this study are filed in the Dow AgroSciences facility archives, Indianapolis, Indiana.

ACKNOWLEDGEMENT

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REFERENCES

1. Environmental Protection Agency 40 CFR Part 180 [OPP-300945; FRL-6391-5] RIN 2070-AB78, Federal Register, November 4, 1999 (Volume 64, Number 213), pages 60112-60121.

2. Liberty Herbicide Specimen Label, Aventis CropScience USA Company, L-SL-5M-(991207)-Rev. 12/99.

3. Certificate of Analysis No. RCD-47/99-B (Revision 1), Analysis reference number 243-03.28, June 15, 1999, Aventis CropScience.

4. Owenby, J. R.; Ezell, D. S., "Monthly Station Normals of Temperature, Precipitation, and Heating and Cooling Degree Days, 1961-1990", Climatography of the United States No. 81, NOAA, National Climatic Data Center, Ashville, North Carolina, 1992.

5. Czarnecki, J. J.; Bertrand, N., "Gas Chromatographic Determination of HOE-039866 (Glufosinate-ammonium) and its Metabolites as Residues in Glufosinate Resistant Corn and Soybean RAC and Processed Commodities", Aventis CropScience analytical method RAM Number BK/05/95, 1995, unpublished method of Aventis CropScience.

Table 1. Field Site Information

State	EPA Region	Field Investigator Facility Address	Site location Experiment Number	Soil Type	Treated Plot Size, ft
IA	5	David Bennett Bennett Ag Research & Consulting 1109 Ivy Avenue Richland, IA 52585	Richland, IA Jefferson County 000290IA1	Silty clay loam	Two replicates of 10 x 25 (0.0115 A)
IA	5	Paul Clayton Land O'Lakes Research Farm 1025 190th St. Webster City, IA 50595	Webster City, IA Hamilton County 000290IA2	Loam	Two replicates of 10 x 40 (0.0184 A)
IN	5	Kevin Kiser Heartland Technologies, Inc 12491 E. 136th Street Noblesville, IN 46060	Noblesville, IN Hamilton County 000290IN1	Silty clay loam	Two replicates of 10 x 33 (0.0152 A)
OH	5	John Gruber Ag Consultants, Inc. 270 South Main New Holland, OH 43145	New Holland, OH Fayette County 000290OH1	Silt loam	Two replicates of 10 x 30[a] (0.0138 A)
IL	5	Bill Tarter Alvey Ag Research 19300 Marydale Road Carlyle, IL 62231	Carlyle, IL Clinton County 000290IL1	Silt loam	Two replicates of 10 x 25 (0.0115 A)
MN	5	Jon Warnke Warnke Research Services, LLC 215 East Main Geneva, MN 56035	Geneva, MN Freeborn County 000290MN1	Clay loam	Two replicates of 10 x 25 (0.0115 A)

[a] The actual plot size was 10 ft x 50 ft but the plot size the test substance was applied to was 10 ft x 30 ft.

Table 2. Planting and Maintenance Chemical Information

Site ID	Planting Date Plant/Row Spacing	Maintenance Chemicals			
		Product	Applied[a]	Rate	Purpose
IA1	Planted May 31, 2000	Harness Xtra	01-June	2.9 lb ai/A acetochlor 1.1 lb ai/A atrazine	Herbicide
	10 in. plant spacing 30 in. row spacing	Nitrogen	01-June	51 GPA (180 lb N/A)	Fertilizer
IA2	Planted May 25, 2000 9.8 in. plant spacing 30 in. row spacing	46-0-0 (urea) Dual II	May 17 April 26	326 lb/A 2.4375 lb ai/A	Fertilizer Herbicide
IN1	Planted May 26, 2000 10 in. plant spacing 30 in. row spacing	22-11-16 Bicep 6 SC	May 26 May 31	680 lb/A 2.4 qt/A product	Fertilizer Herbicide
OH1	Planted May 26, 2000 10 in. plant spacing 30 in. row spacing	Laddock SL 46-0-0	June 23 June 26	1.67 pt/A product 150 lb/A	Herbicide Fertilizer
IL1	Planted June 7, 2000 6 in. plant spacing 30 in. row spacing	Bicep Lite II Warrior T	May 13 May 13	0.25 lb ai/A 0.025 lb ai/A	Herbicide Insecticide
MN1	Planted May 24, 2000 10 in. plant spacing 30 in. row spacing	Surpass 6.4EC Atrazine 4L	May 25 May 25	1.6 lb ai/A 1.0 lb ai/A	Herbicide Herbicide

[a] Date applied in was in the year 2000.

Table 3. Application Information

Site ID.	Applicator Description	Treat. No.	Appl. No.	Date Applied (2000)	Approx. Crop Stage, Crop Height, (BBCH)
IA1	Tractor mounted research side boom, 6-nozzle, CO_2 pressurized sprayer with 8002 flat fan nozzles on 20-inch spacing, 90° to row, 18 inches above target.	02	1	June 21	NR[a], 10-12 inches (15)
		03	1	June 21	NR, 10-12 inches (15)
		02	2	June 29	V7, 22 inches (17)
	Tractor mounted research side boom, 5-nozzle, CO_2 pressurized sprayer with 15004 double outlet flat nozzles on 30-inch spacing, 90° to row, 12 inches above soil.	03	2	July 7	V9, 34 inches (19)
IA2	LOL research plot sprayer #1, 6-nozzle, Compressed air sprayer with DG80015VS flat fan nozzles on 20-inch spacing, 90° to row, 18 inches above target.	02	1	June 22	V4, 14 inches (14)
		03	1	June 22	V4, 14 inches (14)
	JD high clearance sprayer with research spray boom #1, 5-nozzle, nitrogen pressurized sprayer with 150015 double outlet flat fan nozzles on 30-inch spacing, down and to side of row, 18 inches above soil.	02	2	July 6	V9, 42 inches (19)
		03	2	July 6	V9, 48 inches (19)
IN1	Tractor mounted sprayer, 10 ft offset, 8-nozzle, nitrogen pressurized sprayer with XR11002VS flat fan tee jet nozzles on 15-inch spacing, down direction, 14 inches above target.	02	1	June 19	NR, 12 inches (14)
		03	1	June 19	NR, 12 inches (14)
	Drop nozzle sprayer, 4-nozzle, nitrogen pressurized sprayer with XR11002VS flat fan tee jet nozzles on 30-inch spacing, 45° with ground, 14 inches above target.	02	2	July 6	V8, 36 inches (18)
		03	2	July 6	V8, 36 inches (18)

Table 3. (Cont.) Application Information

Site ID.	Applicator Description	Treat. No.	Appl. No.	Date Applied (2000)	Approx. Crop Stage, Crop Height,(BBCH)
OH1	R&D 10 ft research boom, 6-nozzle, Compressed air sprayer with 8002 flat fan nozzles on 20-inch spacing, down between rows, 18 inches above target.	02 03	1 1	June 28 June 28	V4, 12 inches (14) V4, 12 inches (14)
	Single nozzle boom, Compressed air sprayer with 8002E even flat fan nozzle, down between rows, 12 inches above target.	02 03	2 2	July 12 July 12	V8, 30-38 inches (18) V8, 30-38 inches (18)
IL1	Tractor mounted sidemount boom, 6-nozzle, compressed air sprayer with 8003 tee jet flat fan nozzles on 20-inch spacing, parallel to boom, 18 inches above target.	02 03	1 1	June 30 June 30	V4, 12 inches (14) V4, 12 inches (14)
	Tractor mounted sidemount boom, 6-nozzle, compressed air sprayer with 15003 tee jet flat fan even nozzles on 30-inch spacing, parallel to boom, 18 inches above weeds.	02 03	2 2	July 21 July 21	V7, 36 inches (17) V7, 36 inches (17)
MN1	Backpack sprayer, 6-nozzle, nitrogen pressurized sprayer with 8002 flat fan nozzles on 20-inch spacing, down Direction, 18 inches above target.	02 03	1 1	June 21 June 21	V4, 12-15 inches (14) V4, 12-15 inches (14)
	Single drop nozzle, nitrogen pressurized sprayer with 15004 split nozzle, down direction, 4-6 inches above soil.	02 03	1 1	July 7 July 7	V9, 36 inches[b] (19) V9, 36 inches[b] (19)

[a] NR = Not recorded in field raw data.
[b] Extended leaf.

Table 4. Climatological Data

Site Location	Month	Temperature, °F Average, 2000 Max	Average, 2000 Min	Historical[a] Max	Historical[a] Min	Precipitation (in.) 2000	Precipitation (in.) Hist.	Irrigation (in.) 2000
Richland, IA (Fairfield, IA)[c]	May[b]	89.6	69.0	71.9	50.3	0.03	5.54	0
	Jun	79.8	59.6	81.6	61.0	8.06	4.23	0
	Jul	83.6	63.4	85.6	65.4	4.19	4.80	0
	Aug	86.5	74.4	84.0	63.1	1.73	3.71	0
	Sep	80.2	53.1	75.5	52.4	3.50	3.08	0
	Oct[b]	68.3	55.2	65.3	43.1	1.91	2.55	0
Webster City, IA (Webster City, IA)	May[b]	69.9	52.4	70.9	48.1	2.61	5.22	0
	Jun	76.6	54.6	80.2	58.6	5.70	7.09	0
	Jul	81.2	62.4	82.5	61.7	3.33	4.82	0
	Aug	81.3	62.2	80.6	59.3	4.48	5.06	0
	Sep	78.4	50.7	74.7	49.8	1.34	2.61	0
	Oct[b]	60.3	34.8	63.6	38.5	0.70	2.21	0
Noblesville, IN (Indianapolis, IN)	May[b]	74.3	54.7	72.4	52.4	1.17	4.97	0
	Jun	80.0	59.7	81.7	62.1	7.54	4.49	1.50
	Jul	82.0	59.9	85.5	65.7	3.07	4.02	0
	Aug	81.3	60.6	83.9	64.0	5.53	3.30	0
	Sep	75.4	52.9	77.5	55.1	5.24	2.67	0
	Oct[b]	66.2	43.2	66.1	44.1	2.48	2.83	0
New Holland, OH (Fayette County, OH)	May[b]	73.3[d]	54.3[d]	71.4	51.3	1.65	4.99	0
	Jun	81.6[d]	62.0[d]	79.3	61.0	5.85	3.45	0
	Jul	81.4	62.3	82.7	57.1	6.65	4.13	0
	Aug	81.5	61.5	81.4	62.2	2.05	3.58	0
	Sep	72.7[e]	52.1[e]	75.9	54.5	4.30	2.04	0
	Oct[b]	71.0[e]	47.3[e]	67.0	54.5	2.00	2.41	0
Carlyle, IL[f] (Carlyle, IL)	Jun[b]	82.2	64.8	82.2	62.8	9.60	4.98	0
	Jul	90.9	55.6	86.1	65.9	6.70	3.83	0
	Aug	96.3	62.8	84.9	64.1	8.00	2.18	0
	Sep	92.1	36.5	79.1	53.4	3.62	2.94	0
	Oct[b]	68.2	46.6	68.2	41.4	0.00	2.87	0

Table 4. (Cont.) Climatological Data

Site Location	Month	Temperature, °F Average, 2000 Max	Average, 2000 Min	Historical[a] Max	Historical[a] Min	Precipitation (in.) 2000	Precipitation (in.) Hist.	Irrigation (in.) 2000
Geneva, MN[g]	May[b]	68	52	67.6	46.6	1.35	3.73	0
(Albert Lea, MN)	Jun	76	53	79.5	58.2	10.50	4.18	0
	Jul	80	61	80.3	60.5	9.00	4.80	0
	Aug	80	58	79.2	59.2	5.60	4.67	0
	Sep	74	48	71.0	49.0	1.25	3.05	0
	Oct[b]	60	33	59.1	36.0	0.90	1.97	0

[a] Historical data is from NOAA (30-year average) weather station.
[b] Partial data reported for the average 2000 data and covered only the duration of the study.
[c] Site locations in parenthesis represent the NOAA weather stations used for historical data and are located within about 20 miles of the test site.
[d] 2000 data is from the Circleville, Ohio, NOAA weather station which is approximately 20 miles from the test site.
[e] Missing data on some days during the month; average temperatures or total precipitation do not reflect missing data.
[f] 2000 temperature and rainfall data reported for the test site is obtained from the Alvey Agricultural Research weather station located about 5 miles from the site.
[g] 2000 temperature reported for the test site is obtained from the Warnke Research Services weather station located about 2.5 miles from the site. The rainfall data was obtained at the test site.

Table 5. Sampling Data of Corn Forage, Grain, and Stover for Residue Study of Glufosinate-ammonium

Site ID	Crop	Sample Group No. (SGN)	Date, 2000		PHI[b] (days)	Sample Size, lb.
			Application[a]	Sampling		
IA1	Forage	328871	Jun 27	Aug 28	62	8.5-9.5
	Grain	328898	Jul 07	Oct 04	89	6.0-6.5
	Stover	328901	Jul 07	Oct 04	89	3.0-3.5
IA2	Forage	328928	Jul 06	Sep 07	63	9.0-9.8
	Grain	328936	Jul 06	Oct 12	98	2.2-2.4
	Stover	328944	Jul 06	Oct 12	98	2.3-2.4
IN1	Forage	331767	Jul 6	Sep 06	62	7.0-7.5
	Grain	331775	Jul 6	Oct 18	104	3.0
	Stover	331783	Jul 6	Oct 18	104	4.0
OH1	Forage	330639	Jul 12	Sep 11	61	7.0-8.0
	Grain	330647	Jul 12	Oct 24	104	5.0-6.0
	Stover	330655	Jul 12	Oct 24	104	2.0
IL1	Forage	330604	Jul 21	Sep 06	47	6.5-8.2
	Grain	330612	Jul 21	Oct 16	87	4.1-5.2
	Stover	330620	Jul 21	Oct 16	87	4.2-5.2
MN1	Forage	329045	Jul 07	Sep 14	69	7.0-8.2
	Grain	329053	Jul 07	Oct 16	101	5.5-6.2
	Stover	329061	Jul 07	Oct 16	101	2.2-2.6

[a] Last application date.
[b] Pre-harvest interval: Days from last application to harvest.

Table 6. Summary of Sampling, Shipping, Receiving, and Analysis Dates, and Length of Frozen Storage

DAS[a] Sample Group Number	Field Site Location	Sampling Date	Shipping Date[b]	Receipt Date[c]	Sample Preparation Date[d]	McKenzie/Wright Laboratories			Length Frozen Storage (days)[h]
						Shipping Date[e]	Receipt Date[f]	Analysis Date[g]	
Forage									
328871	000290IA1	28-Aug-00	07-Sep-00	15-Sep-00	15-Sep-00	25-Sep-00	26-Sep-00	23-Oct-00	56
328928	000290IA2	07-Sep-00	21-Sep-00	26-Sep-00	26-Sep-00	10-Oct-00	11-Oct-00	13-Nov-00	67
330604	000290IL1	06-Sep-00	06-Sep-00	07-Sep-00	08-Sep-00	25-Sep-00	26-Sep-00	23-Oct-00	47
329045	000290MN1	14-Sep-00	21-Sep-00	26-Sep-00	26-Sep-00	10-Oct-00	11-Oct-00	13-Nov-00	60
330639	000290OH1	11-Sep-00	26-Sep-00	02-Oct-00	03-Oct-00	10-Oct-00	11-Oct-00	17-Nov-00	67
331767	000290IN1	06-Sep-00	20-Sep-00	28-Sep-00	03-Oct-00	10-Oct-00	11-Oct-00	17-Nov-00	72
Grain									
328898	000290IA1	04-Oct-00	11-Oct-00	20-Oct-00	25-Oct-00	07&13-Nov-00	08&14-Nov-00	04-Dec-00	61
328936	000290IA2	12-Oct-00	17-Oct-00	20-Oct-00	25-Oct-00	07&13-Nov-00	08&14-Nov-00	30-Nov-00	49
330612	000290IL1	16-Oct-00	19-Oct-00	20-Oct-00	25-Oct-00	07&13-Nov-00	08&14-Nov-00	04-Dec-00	49
329053	000290MN1	16-Oct-00	17-Oct-00	20-Oct-00	25-Oct-00	13-Nov-00	14-Nov-00	04-Dec-00	49
330647	000290OH1	24-Oct-00	06-Nov-00	07-Nov-00	08-Nov-00	06-Dec-00	07-Dec-00	11-Dec-00	48
331775	000290IN1	18-Oct-00	07-Nov-00	28-Nov-00	29-Nov-00	06-Dec-00	07-Dec-00	11-Dec-00	54
Stover									
328901	000290IA1	04-Oct-00	11-Oct-00	20-Oct-00	26-Oct-00	07&13-Nov-00	08&14-Nov-00	28-Dec-00	85
328944	000290IA2	12-Oct-00	17-Oct-00	20-Oct-00	26-Oct-00	07&13-Nov-00	08&14-Nov-00	28-Dec-00	77
330620	000290IL1	16-Oct-00	19-Oct-00	20-Oct-00	26-Oct-00	07&13-Nov-00	08&14-Nov-00	03-Jan-01	79
329061	000290MN1	16-Oct-00	17-Oct-00	20-Oct-00	26-Oct-00	07-Nov-00	08-Nov-00	03-Jan-01	79
330655	000290OH1	24-Oct-00	06-Nov-00	07-Nov-00	08-Nov-00	06-Dec-00	07-Dec-00	08-Jan-01	76
331783	000290IN1	18-Oct-00	07-Nov-00	28-Nov-00	29-Nov-00	06-Dec-00	07-Dec-00	08-Jan-01	82

Table 6. (Cont.) Summary of Sampling, Shipping, Receiving, and Analysis Dates, and Length of Frozen Storage

DAS[a] Sample Group Number	Field Site Location	Sampling Date	Shipping Date[b]	Receipt Date[c]	Sample Preparation Date[d]	McKenzie/Wright Laboratories			Length Frozen Storage (days)[h]
						Shipping Date[e]	Receipt Date[f]	Analysis Date[g]	
Frozen Storage Stability									
342408	000290FSS	27-Nov-00[i]	NA[j]	NA	NA	10-Oct-00[k]	11-Oct-00[k]	12-Feb-01	77
342416	000290FSS	12-Jan-01[i]	NA	NA	NA	10-Jan-01[k]	11-Jan-01[k]	12-Mar-01	59
342424	000290FSS	16-Jan-01[i]	NA	NA	NA	10-Jan-01[k]	11-Jan-01[k]	17-Apr-01	91

[a] DAS = Dow AgroSciences

[b] Date on which samples were shipped from the field to Dow AgroSciences.

[c] Date on which samples were received at Dow AgroSciences from the field.

[d] Date on which the bulk samples were prepared at Dow AgroSciences.

[e] Date on which samples were shipped from Dow AgroSciences to McKenzie/Wright Laboratories.

[f] Date on which samples were received at McKenzie/Wright Laboratories from Dow AgroSciences.

[g] Date on which the last analysis was begun by the addition of the extraction solution.

[h] Length of frozen storage from the sampling date to analysis date.

[i] Date frozen storage stability samples were fortified (corresponds to Day 0).

[j] NA = Not applicable.

[k] Refers to bulk samples used to prepare frozen storage stability samples. Sample numbers of the bulk controls were 32887101 and 33060401 for forage, 33064701 for grain, and 33178301and 33065501 for stover.

Table 7. Storage Stability of Glufosinate-ammonium and its Metabolites in Corn Forage, Grain, and Stover

Frozen Storage Day	Dow AgroSciences Sample Number	Sampling Date[a]	Glufosinate Acid Equivalent - P + M1					Glufosinate Acid Equivalent - M2				
			Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]	Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]
Forage Frozen Storage Stability												
0	34240801	27-Nov-00	NA[f]	97	ND[g]	ND						
	34240802	27-Nov-00	1.00	97	1.042	1.07	107					
	34240802	27-Nov-00	1.00	97	0.900	0.93	93					
	34240802	27-Nov-00	1.00	97	1.058	1.09	109					
						Parent =	103					
0	34240801	27-Nov-00	NA	104	ND	ND		NA	60	ND	ND	
	34240803	27-Nov-00	1.00	104	1.010	0.97	97	1.00	60	0.595	1.00	100
	34240803	27-Nov-00	1.00	104	1.223	1.17	117	1.00	60	0.767	1.29	129
	34240803	27-Nov-00	1.00	104	1.222	1.17	117	1.00	60	0.733	1.23	123
						M1 =	111				M2 =	117
77	34240801	12-Feb-01	NA	98	ND	ND						
	34240802	12-Feb-01	1.00	98	0.937	0.95	95					
	34240802	12-Feb-01	1.00	98	0.941	0.96	96					
	34240802	12-Feb-01	1.00	98	1.035	1.05	105					
						Parent =	99					
77	34240801	12-Feb-01	NA	106	0.028	(0.026)[h]		NA	76	ND	ND	
	34240803	12-Feb-01	1.00	106	1.072	1.01	101	1.00	76	0.691	0.92	92
	34240803	12-Feb-01	1.00	106	1.006	0.95	95	1.00	76	0.686	0.91	91
	34240803	12-Feb-01	1.00	106	1.077	1.01	101	1.00	76	0.725	0.96	96
						M1 =	99				M2 =	93

Table 7. (Cont.) Storage Stability of Glufosinate-ammonium and its Metabolites in Corn Forage, Grain, and Stover

Frozen Storage Day	Dow AgroSciences Sample Number	Sampling Date[a]	Glufosinate Acid Equivalent - P + M1					Glufosinate Acid Equivalent - M2				
			Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]	Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]
Grain Frozen Storage Stability												
0	34241601	12-Jan-01	NA	90	ND	ND						
	34241602	12-Jan-01	0.20	90	0.175	0.19	97					
	34241602	12-Jan-01	0.20	90	0.196	0.22	109					
	34241602	12-Jan-01	0.20	90	0.181	0.20	101					
						Parent =	102					
0	34241601	12-Jan-01	NA	98	ND	ND		NA	82	ND	ND	
	34241603	12-Jan-01	0.20	98	0.216	0.22	110	0.20	82	0.182	0.22	111
	34241603	12-Jan-01	0.20	98	0.229	0.23	116	0.20	82	0.182	0.22	111
	34241603	12-Jan-01	0.20	98	0.182	0.18	92	0.20	82	0.136	0.17	83
						M1 =	106				M2 =	102
59	34241601	12-Mar-01	NA	88	ND	ND						
	34241602	12-Mar-01	0.20	88	0.166	0.19	94					
	34241602	12-Mar-01	0.20	88	0.170	0.19	97					
	34241602	12-Mar-01	0.20	88	0.180	0.20	102					
						Parent =	95					
59	34241601	12-Mar-01	NA	99	ND	ND		NA	75	ND	ND	
	34241603	12-Mar-01	0.20	99	0.172	0.17	87	0.20	75	0.142	0.19	95
	34241603	12-Mar-01	0.20	99	0.182	0.18	92	0.20	75	0.145	0.19	97
	34241603	12-Mar-01	0.20	99	0.178	0.18	90	0.20	75	0.135	0.18	91
						M1 =	90				M2 =	94

Table 7. (Cont.) Storage Stability of Glufosinate-ammonium and its Metabolites in Corn Forage, Grain, and Stover

Frozen Storage Day	Dow AgroSciences Sample Number	Sampling Date[a]	Glufosinate Acid Equivalent - P + M1					Glufosinate Acid Equivalent - M2				
			Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]	Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]
Stover Frozen Storage Stability												
0	34242401	16-Jan-01	NA	86	ND	ND						
	34242402	16-Jan-01	1.50	86	1.125	1.31	87					
	34242402	16-Jan-01	1.50	86	1.313	1.53	102					
	34242402	16-Jan-01	1.50	86	1.288	1.50	100					
						Parent =	96					
0	34242401	16-Jan-01	NA	95	ND	ND		NA	66	ND	ND	
	34242403	16-Jan-01	1.50	95	1.470	1.54	103	1.50	66	1.074	1.62	108
	34242403	16-Jan-01	1.50	95	1.443	1.52	101	1.50	66	1.172	1.77	118
	34242403	16-Jan-01	1.50	95	1.347	1.42	94	1.50	66	1.033	1.56	104
						M1 =	99				M2 =	110
91	34242401	17-Apr-01	NA	85	ND	ND						
	34242402	17-Apr-01	1.50	85	1.276	1.50	100					
	34242402	17-Apr-01	1.50	85	1.163	1.37	91					
	34242402	17-Apr-01	1.50	85	1.371	1.62	108					
						Parent =	100					

Table 7. (Cont.) Storage Stability of Glufosinate-ammonium and its Metabolites in Corn Forage, Grain, and Stover

Frozen Storage Day	Dow AgroSciences Sample Number	Sampling Date[a]	Glufosinate Acid Equivalent - P + M1					Glufosinate Acid Equivalent - M2				
			Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]	Fort. Level, µg/g[b]	Percent Recovery[c]	Uncorrected µg/g	Corrected[d] µg/g	Percent Remaining[e]
Stover Frozen Storage Stability (Cont.)												
91	34242401	17-Apr-01	NA	98	ND	ND		NA	78	ND	ND	
	34242403	17-Apr-01	1.50	98	1.521	1.55	103	1.50	78	1.167	1.49	100
	34242403	17-Apr-01	1.50	98	1.424	1.45	97	1.50	78	1.112	1.42	95
	34242403	17-Apr-01	1.50	98	1.392	1.42	94	1.50	78	1.049	1.34	90
						M1 =	98				M2 =	95

[a] Date on which analysis was begun by the addition of the extraction solution.
[b] Fort. Level = Fortification level - Concentration the frozen storage stability samples were fortified at.
[c] Percent recovery of the daily fortified samples analyzed with the storage stability samples.
[d] Corrected by the average daily fortified recovery samples analyzed within the same sample analysis set and standard curve injection.
[e] Percent remaining using recovery corrected concentrations and the theoretical fortification level.
[f] NA = Not applicable. Control sample was not fortified.
[g] ND = Not detected. Less than the LOD.
[h] ( ) = Value between the LOD and the LOQ.

Table 8. Residues of Glufosinate Acid Equivalents in Corn Forage After Treatment with Liberty Herbicide

Site ID	PHI[a] (days)	Sample Number[b]	Analysis Date[c]	Glufosinate Acid Equivalent Metabolite 2				Glufosinate Acid Equivalent Parent + Metabolite 1				Total Glufosinate Acid Equivalent[h] (µg/g)
				% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	
000290IA1	62	32887101	23-Oct-00	52	ND[i]	ND	ND	92	ND	ND	ND	ND[j]
		32887101	23-Oct-00	61	ND	ND	ND	103	ND	ND	ND	
		32887101	23-Oct-00	--[k]	--	--	--	107	0.0269	0.026	(0.026)[l]	
		32887102	23-Oct-00	52	ND	ND	ND	92	0.3720	0.406	0.41	0.41
		32887103	23-Oct-00	52	ND	ND	ND	92	0.2983	0.325	0.33	0.33
											Average =	0.37
000290IA2	63	32892801	13-Nov-00	71	ND	ND	ND	115	ND	ND	ND	ND[j]
		32892801	13-Nov-00	87	ND	ND	ND	100	ND	ND	ND	
		32892802	13-Nov-00	71	0.0413	0.058	(0.058)	115	0.0997	0.087	0.09	0.15
		32892803	13-Nov-00	71	0.0260	0.037	(0.037)	115	0.1018	0.089	0.09	0.13
											Average =	0.14
000290IN1	62	33176701	17-Nov-00	84	ND	ND	ND	89	ND	ND	ND	ND
		33176702	17-Nov-00	84	0.0396	0.047	(0.047)	89	0.3827	0.429	0.43	0.48
		33176703	17-Nov-00	84	0.0336	0.040	(0.040)	89	0.3963	0.444	0.44	0.45[m]
		33176703	17-Nov-00	84	0.0287	0.034	(0.034)	89	0.3324	0.373	0.37	
											Average =	0.46
000290OH1	61	33063901	17-Nov-00	69	ND	ND	ND	90	ND	ND	ND	ND[j]
		33063901	17-Nov-00	84	ND	ND	ND	89	ND	ND	ND	
		33063902	17-Nov-00	69	0.0199	0.029	(0.029)	90	0.1848	0.205	0.21	0.23
		33063903	17-Nov-00	69	0.0236	0.034	(0.034)	90	0.2269	0.252	0.25	0.29
											Average =	0.26

Table 8. (Cont.) Residues of Glufosinate Acid Equivalents in Corn Forage After Treatment with Liberty Herbicide

Site ID	PHI[a] (days)	Sample Number[b]	Analysis Date[c]	Glufosinate Acid Equivalent Metabolite 2				Glufosinate Acid Equivalent Parent + Metabolite 1				Total Glufosinate Acid Equivalent[h] (µg/g)
				% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	
000290IL1	47	33060401	23-Oct-00	61	ND	ND	ND	104	ND	ND	ND	ND
		33060402	23-Oct-00	61	0.0435	0.071	0.071	104	1.2704	1.222	1.22	1.30
		33060403	23-Oct-00	61	0.0304	0.050	(0.050)	104	1.5187	1.460	1.46	1.50[m]
		33060403	23-Oct-00	61	0.0327	0.054	(0.054)	104	1.4621	1.406	1.41	
											Average =	1.40
000290MN1	69	32904501	13-Nov-00	87	0.0205	0.024	(0.024)	100	ND	ND	ND	(0.024)
		32904502	13-Nov-00	87	0.0224	0.026	(0.026)	100	0.0404	0.040	(0.040)	(0.066)
		32904503	13-Nov-00	87	0.0277	0.032	(0.032)	100	0.0324	0.032	(0.032)	(0.059)[m]
		32904503	13-Nov-00	87	0.0251	0.029	(0.029)	100	0.0251	0.025	(0.025)	
											Average =	(0.062)

[a] Pre-harvest interval: days from application to harvest.
[b] Application rate was 62 oz/A (0.81 lb a.i./A) from Treatment No. 3; all sample numbers with last two digits of 01 were control samples, untreated with Liberty herbicide.
[c] Date on which analysis was begun by the addition of the extraction solution.
[d] % Avg. Curve Recovery = Average set and calibration curve recovery of daily fortified recovery samples analyzed with treated samples.
[e] Uncorr. = Uncorrected.
[f] Corrected for the average curve recovery of fortified samples.
[g] Calculated with more digits than displayed.
[h] Total glufosinate acid equivalent, µg/g = Glufosinate acid equivalent, µg/g (parent + M1 + M2).
[i] ND = Not detected. Less than the LOD of 0.019 µg/g for M2 and 0.015 µg/g for P + M1.
[j] Average of a single sample injected multiple times.
[k] -- = Not applicable.
[l] ( ) = Value between the LOD and the LOQ of 0.019 µg/g and 0.063 µg/g for M2 and between 0.015 µg/g and 0.050 µg/g for P + M1.
[m] Average of duplicate samples.

Table 9. Residues of Glufosinate Acid Equivalents in Corn Grain After Treatment with Liberty Herbicide

Site ID	PHI[a] (days)	Sample Number[b]	Analysis Date[c]	Glufosinate Acid Equivalent Metabolite 2				Glufosinate Acid Equivalent Parent + Metabolite 1				Total Glufosinate Acid Equivalent[h] (µg/g)
				% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	
000290IA1	89	32889801	30-Nov-00	73	ND[i]	ND	ND	108	ND	ND	ND	ND[j]
000290IA1		32889801	30-Nov-00	78	ND	ND	ND	114	ND	ND	ND	
000290IA1		32889802	30-Nov-00	73	ND	ND	ND	108	ND	ND	ND	ND
000290IA1		32889803	04-Dec-00	79	ND	ND	ND	101	ND	ND	ND	ND
											Average =	ND
000290IA2	98	32893601	30-Nov-00	78	ND	ND	ND	114	ND	ND	ND	ND
000290IA2		32893602	30-Nov-00	78	ND	ND	ND	114	ND	ND	ND	ND
000290IA2		32893603	30-Nov-00	78	ND	ND	ND	114	ND	ND	ND	ND[k]
000290IA2		32893603	30-Nov-00	78	ND	ND	ND	114	ND	ND	ND	
											Average =	ND
000290IN1	104	33177501	11-Dec-00	72	ND	ND	ND	99	ND	ND	ND	ND
000290IN1		33177502	11-Dec-00	72	ND	ND	ND	99	ND	ND	ND	ND
000290IN1		33177503	11-Dec-00	72	ND	ND	ND	99	0.0158	0.016	ND	ND[k]
000290IN1		33177503	11-Dec-00	72	ND	ND	ND	99	ND	ND	ND	
											Average =	ND
000290OH1	104	33064701	11-Dec-00	87	ND	ND	ND	95	ND	ND	ND	ND[j]
000290OH1		33064701	11-Dec-00	72	ND	ND	ND	99	ND	ND	ND	
000290OH1		33064702	11-Dec-00	87	ND	ND	ND	95	ND	ND	ND	ND
000290OH1		33064703	11-Dec-00	87	ND	ND	ND	95	ND	ND	ND	ND
											Average =	ND

Table 9. (Cont.) Residues of Glufosinate Acid Equivalents in Corn Grain After Treatment with Liberty Herbicide

Site ID	PHI[a] (days)	Sample Number[b]	Analysis Date[c]	Glufosinate Acid Equivalent Metabolite 2				Glufosinate Acid Equivalent Parent + Metabolite 1				Total Glufosinate Acid Equivalent[h] (µg/g)
				% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	
000290IL1	87	33061201	04-Dec-00	78	ND	ND	ND	107	ND	ND	ND	ND[j]
000290IL1		33061201	04-Dec-00	79	ND	ND	ND	101	ND	ND	ND	
000290IL1		33061202	04-Dec-00	78	0.0203	0.026	(0.026)[l]	107	0.0304	0.029	(0.029)	(0.055)
000290IL1		33061203	04-Dec-00	78	0.0158	0.020	(0.020)	107	0.0301	0.028	(0.028)	(0.048)
											Average =	(0.052)
000290MN1	101	32905301	04-Dec-00	79	ND	ND	ND	101	ND	ND	ND	ND
000290MN1		32905302	04-Dec-00	79	ND	ND	ND	101	ND	ND	ND	ND
000290MN1		32905303	04-Dec-00	79	ND	ND	ND	101	ND	ND	ND	ND[k]
000290MN1		32905303	04-Dec-00	79	ND	ND	ND	101	ND	ND	ND	
											Average =	ND

[a] Pre-harvest interval: days from application to harvest.
[b] Application rate was 62 oz/A (0.81 lb a.i./A) from Treatment No. 3; all sample numbers with last two digits of 01 were control samples, untreated with Liberty herbicide.
[c] Date on which analysis was begun by the addition of the extraction solution.
[d] % Avg. Curve Recovery = Average set and calibration curve recovery of daily fortified recovery samples analyzed with treated samples.
[e] Uncorr. = Uncorrected.
[f] Corrected for the average curve recovery of fortified samples.
[g] Calculated with more digits than displayed.
[h] Total glufosinate acid equivalent, µg/g = Glufosinate acid equivalent, µg/g (parent + M1 + M2).
[i] ND = Not detected. Less than the LOD of 0.015 µg/g for M2 and 0.019 µg/g for P + M1.
[j] Average of a single sample injected duplicate times.
[k] Average of duplicate samples.
[l] ( ) = Value between the LOD and the LOQ of 0.015 µg/g and 0.05 µg/g for M2 and 0.019 µg/g and 0.064 µg/g for P + M1.

Table 10. Residues of Glufosinate Acid Equivalents in Corn Stover After Treatment with Liberty Herbicide

Site ID	PHI[a] (days)	Sample Number[b]	Analysis Date[c]	Glufosinate Acid Equivalent Metabolite 2				Glufosinate Acid Equivalent Parent + Metabolite 1				Total Glufosinate Acid Equivalent[h] (µg/g)
				% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	
000290IA1	89	32890101	28-Dec-00	82	ND[i]	ND	ND	92	ND	ND	ND	ND[j]
		32890101	28-Dec-00	70	ND	ND	ND	94	ND	ND	ND	
		32890102	28-Dec-00	82	ND	ND	ND	92	0.0391	0.042	(0.042)[k]	(0.042)
		32890103	28-Dec-00	82	ND	ND	ND	92	0.0875	0.095	0.09	0.09
											Average =	0.07
000290IA2	98	32894401	28-Dec-00	70	ND	ND	ND	94	ND	ND	ND	ND
		32894402	28-Dec-00	70	0.0106	0.015	ND	94	0.1157	0.123	0.12	0.12
		32894403	28-Dec-00	70	0.0151	0.022	(0.022)	94	0.1252	0.133	0.13	0.16[l]
		32894403	28-Dec-00	70	0.0156	0.022	(0.022)	94	0.1348	0.144	0.14	
											Average =	0.14
IN1	104	33178301	08-Jan-01	58	ND	ND	ND	85	ND	ND	ND	ND[j]
		33178301	08-Jan-01	70	ND	ND	ND	95	ND	ND	ND	
		33178302	08-Jan-01	58	0.0328	0.056	(0.056)	85	0.4369	0.512	0.51	0.57
		33178303	08-Jan-01	58	0.0295	0.051	(0.051)	85	0.2407	0.282	0.28	0.33
											Average =	0.45
OH1	104	33065501	08-Jan-01	70	ND	ND	ND	95	ND	ND	ND	ND
		33065502	08-Jan-01	70	0.0415	0.059	(0.059)	95	0.1382	0.146	0.15	0.21
		33065503	08-Jan-01	70	0.0491	0.070	0.07	95	0.1436	0.152	0.15	0.22[l]
		33065503	08-Jan-01	70	0.0444	0.063	0.06	95	0.1397	0.148	0.15	
											Average =	0.21

Table 10. (Cont.) Residues of Glufosinate Acid Equivalents in Corn Stover After Treatment with Liberty Herbicide

Site ID	PHI[a] (days)	Sample Number[b]	Analysis Date[c]	Glufosinate Acid Equivalent Metabolite 2				Glufosinate Acid Equivalent Parent + Metabolite 1				Total Glufosinate Acid Equivalent[h] (µg/g)
				% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	% Avg. Curve Recovery[d]	Uncorr.[e] µg/g	Corrected[f,g] µg/g	Reported[g] µg/g	
IL1	87	33062001	03-Jan-01	90	ND	ND	ND	103	ND	ND	ND	ND
		33062002	03-Jan-01	90	0.1816	0.202	0.20	102	2.4570	2.409	2.41	2.61
		33062003	03-Jan-01	90	0.1350	0.150	0.15	102	1.7310	1.697	1.70	1.80[l]
		33062003	03-Jan-01	90	0.1326	0.147	0.15	102	1.6454	1.613	1.61	
											Average =	2.20
MN1	101	32906101	03-Jan-01	80	ND	ND	ND	102	ND	ND	ND	ND[j]
		32906101	03-Jan-01	90	ND	ND	ND	NA	ND	ND	ND	
		32906101	03-Jan-01	NA	ND	ND	ND	102	0.0290	0.028	(0.028)	
		32906102	03-Jan-01	80	ND	ND	ND	102	0.0611	0.060	0.06	0.06
		32906103	03-Jan-01	80	ND	ND	ND	102	0.0707	0.069	0.07	0.07
											Average =	0.06

[a] Pre-harvest interval: days from application to harvest.
[b] Application rate was 62 oz/A (0.81 lb a.i./A) from Treatment No. 3; all sample numbers with last two digits of 01 were control samples, untreated with Liberty herbicide.
[c] Date on which analysis was begun by the addition of the extraction solution.
[d] % Avg. Curve Recovery = Average set and calibration curve recovery of daily fortified recovery samples analyzed with treated samples.
[e] Uncorr. = Uncorrected.
[f] Corrected for the average curve recovery of fortified samples.
[g] Calculated with more digits than displayed.
[h] Total glufosinate acid equivalent, µg/g = Glufosinate acid equivalent, µg/g (parent + M1 + M2).
[i] ND = Not detected. Less than the LOD of 0.015 µg/g for M2 and 0.019 µg/g for P + M1.
[j] Average of a single sample injected multiple times.
[k] ( ) = Value between the LOD and the LOQ of 0.015 µg/g and 0.05 µg/g for M2 and 0.018 µg/g and 0.061 µg/g for P + M1.
[l] Average of duplicate samples.

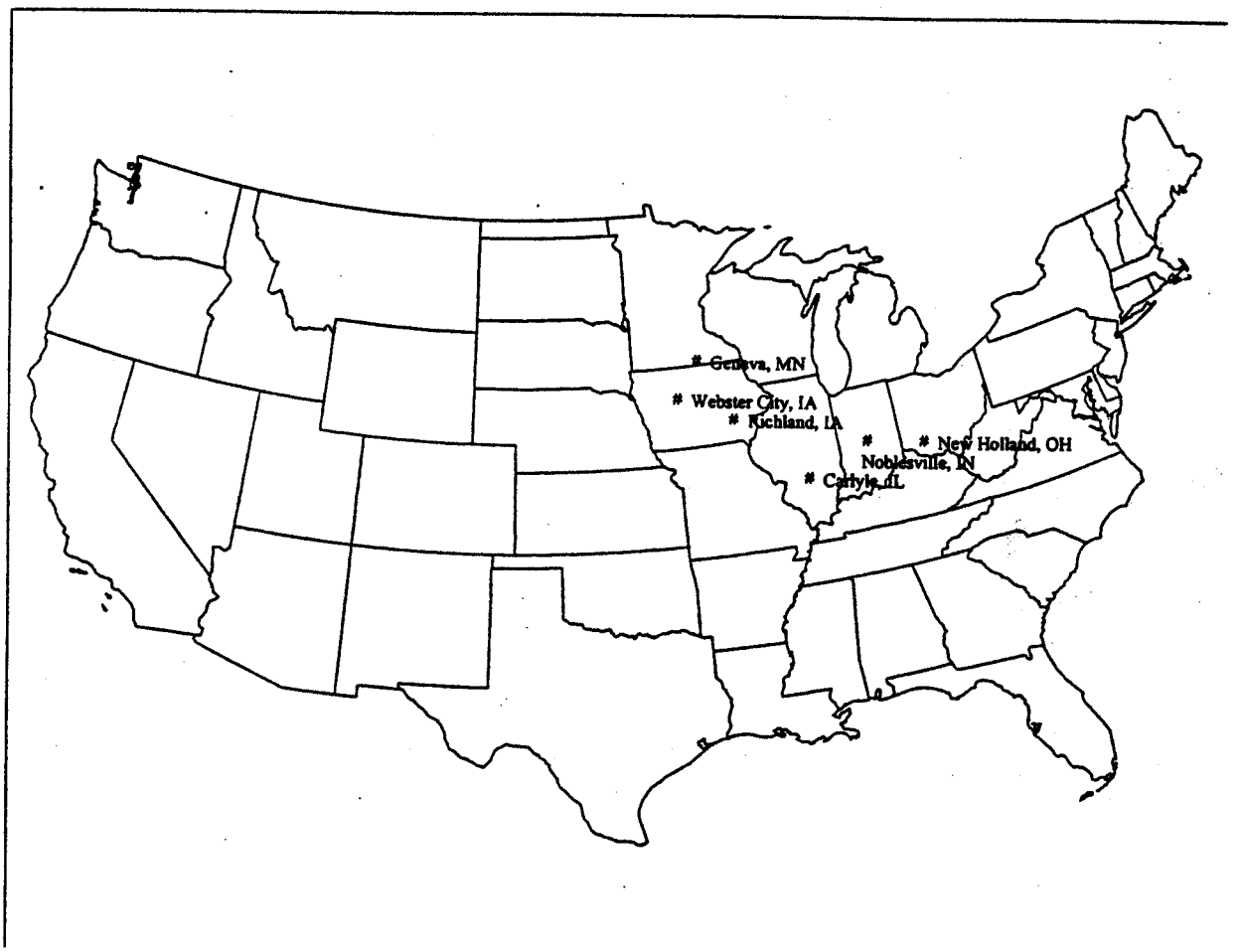

Figure 1. Site Locations for Magnitude of Residue Study of Glufosinate-ammonium in Transgenic Corn

APPENDIX A—Application Rate Calculations

Calculations:

Microsoft Excel (Version 97 SR-2(f)) was used for calculations. Any slight variations observed between the displayed values and other calculated values are due to difference in rounding.

$$\text{Spray vol, gal/A} = \frac{\text{(calib. vol., mL) (pass time, sec) (138.1)}}{\text{(calib. time, sec) (distance, ft) (nozzle spacing, in)}}$$

$$138.1 = \text{(43560 sq ft/A) (12 in./ft) (1 gal/3785.4 mL)}$$

$$\text{Actual applic. rate, lb. a.i./A} = \frac{\text{(amt. form., mL) (spray rate, gal./A) (3.7854, L/gal.) (200 g ai/L)}}{\text{(total mix. vol., mL) (453.6 g/lb.)}}$$

$$\text{Assay} = 200 \text{ g a.i./L}$$

$$\text{\% of target applic. rate} = \frac{\text{actual applic. rate, lb. a.i./A}}{\text{target applic. rate, lb. a.i./A}} \times 100$$

$$\text{lb/A ammonium sulfate (liquid)} = \frac{\text{(mL AMS) (3.33 lb AMS/gal) (spray rate, gal./A)}}{\text{(total mix volume, mL)}}$$

$$\text{lb/A ammonium sulfate (powder)} = \frac{\text{(g AMS) (3785.4 mL/gal) (spray rate, gal./A)}}{\text{(total mix volume, mL) (453.6 g/lb)}}$$

Conversion units:

1 gal = 3785.4 mL
1 A = 43560 sq ft
1 ft = 12 in.

Appendix A. Application Rate Calculations for Liberty Herbicide (Glufosinate-ammonium) in Transgenic Field Corn

Site ID	Treat. No.	Appl. No.	Amt. Form. mL[a]	AMS[b] mL or g[a]	Total mix. vol. mL[a]	Dist. ft.[a]	Pass time sec.[a]	Calib. vol. mL[a]	Calib time sec.[a]	Nozzle space in.[a]	AMS lb./A	Spray rate gal./A	Actual applic. rate lb. ai/A	Target applic. rate lb. ai/A[c]	Percent of target applic.
IA1	2	1	52.0	61.4	3002.0	25	4.12	391.0	30	20	2.5	14.8	0.429	0.44	97
	3	1	52.0	61.4	3002.0	25	4.19	391.0	30	20	2.6	15.1	0.436	0.44	99
	2	2	31.0	50.5	2501.0	25	6.22	310.8	30	20	3.0	17.8	0.368	0.37	100
	3	2	26.0	43.4	2996.0	25	6.37	656.0	30	30	3.1	25.6	0.371	0.37	100
IA2	2	1	55.4	75.0	4184.4	40	13.11	313.1	35	20	3.0	20.2	0.447	0.44	102
	3	1	55.4	75.0	4184.4	40	13.00	313.1	35	20	3.0	20.1	0.444	0.44	101
	2	2	152.1	249.9	13867.1	40	20.26	296.0	35	30	3.0	19.7	0.361	0.37	98
	3	2	152.1	249.9	13867.1	40	20.08	296.0	35	30	2.9	19.5	0.358	0.37	97
IN1	2	1	50.0	67.7	2823.0	33	5.35	305.3	30	15	3.0	15.2	0.449	0.44	102
	3	1	50.0	67.7	2823.0	33	5.19	305.3	30	15	2.9	14.7	0.436	0.44	99
	2	2	50.0	82.2	3428.0	33	11.83	271.3	30	30	3.0	14.9	0.363	0.37	98
	3	2	50.0	82.2	3428.0	33	11.99	271.3	30	30	3.0	15.1	0.368	0.37	99
OH1	2	1	29.2	39.5	1679.2	50	10.90	292.5	30	20	2.9	14.7	0.426	0.44	97
	3	1	29.2	39.5	1679.2	50	11.60	292.5	30	20	3.1	15.6	0.453	0.44	103
	2	2	16.6	27.2	1181.6	30	6.74	300.0	30	20.2	3.0	15.4	0.360	0.37	97
	3	2	16.6	27.2	1181.6	30	6.76	300.0	30	20.2	3.0	15.4	0.361	0.37	98
IL1	2	1	50.0	170.0[d]	2930.0	25	3.70	447.0	30	20	2.9	15.2	0.434	0.44	99
	3	1	50.0	170.0[d]	2930.0	25	3.90	447.0	30	20	3.1	16.0	0.457	0.44	104
	2	2	67.0	141.0	6935.0	25	8.80	423.9	30	30	3.9	22.9	0.369	0.37	100
	3	2	67.0	141.0	6935.0	25	8.90	423.9	30	30	3.9	23.2	0.373	0.37	101
MN1	2	1	16.2	22.0	1000.0	25	5.65	156.5	15	20	3.0	16.3	0.440	0.44	100
	3	1	16.2	22.0	1000.0	25	5.62	156.5	15	20	3.0	16.2	0.438	0.44	100
	2	2	18.1	29.8	2000.0	25	5.69	346.7	15	30	3.0	24.2	0.366	0.37	99
	3	2	18.1	29.8	2000.0	25	5.69	346.7	15	30	3.0	24.2	0.366	0.37	99

[a] Raw data obtained from field data books (some values were rounded to the nearest decimal place).
[b] AMS = ammonium sulfate. Target rate of 3.0 lb/A.
[c] Target rate is specified in the protocol (000290).
[d] AMS is in liquid form.

APPENDIX B—Analytical Summary—McKenzie/Wright Laboratories

ANALYTICAL REPORT

TITLE

Magnitude of the Residue of Glufosinate Ammonium in Transgenic Field Corn Tolerant to Glufosinate Ammonium

SUMMARY AUTHOR

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McKenzie/Wright Laboratories, L.L.C.

STUDY DIRECTOR

Cynthia K. Robb
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ANALYTICAL REPORT COMPLETION DATE

June 14, 2001

ANALYTICAL LABORATORY

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LABORATORY PROJECT IDENTIFICATION

Dow AgroSciences LLC Protocol No. 000290

GOOD LABORATORY PRACTICES COMPLIANCE STATEMENT

We, the undersigned, hereby certify that the data contained in this report were generated in compliance with Good Laboratory Practice Standards (40 CFR Part 160) applicable to analytical testing facilities.

Melinda Lalko

Melinda Lalko
Principal Investigator
McKenzie/Wright Laboratories, L.L.C.

21 Jun 01
Date

M. C. Wright

M. C. Wright
President
McKenzie/Wright Laboratories, L.L.C.

23 Jul 01
Date

QUALITY ASSURANCE STATEMENT

The Quality Assurance Unit of McKenzie/Wright Laboratories, L.L.C., has reviewed this report package for Study Number 000290, "Magnitude of the Residue of Glufosinate Ammonium in Transgenic Field Corn Tolerant to Glufosinate Ammonium". The Quality Assurance Unit has conducted the following inspections of this study and has submitted written reports of these inspections to the Study Director, his or her management, and to the Sponsor's Representative.

Date Inspected	Phase	Date reported to: Study Director & Management		McKenzie Management
03 Oct 00	Analytical Method • Filtering • Anion Exchange Column Clean-up • Filtering	13 Nov 00	14 Nov 00	27 Oct 00
05 Oct 00	Analytical Method • Silica Gel Column Clean-up	13 Nov 00	14 Nov 00	27 Oct 00
10 Oct 00	Analytical Method • Concentration	13 Nov 00	14 Nov 00	27 Oct 00
	Raw/Supporting Data Review • Test System Receipt Documentation • Test System Preparation Documentation	13 Nov 00	14 Nov 00	27 Oct 00
13 Mar 01	Analytical Method • Silica Gel Column Clean-up	18 May 01	18 May 01*	17 May 01
15 Mar 01	Raw/Supporting Data Review • Standard Preparation Documentation	18 May 01	18 May 01*	17 May 01
03 Apr 01	Raw/Supporting Data Review • Study Notebook • Standard Preparation Documentation	18 May 01	18 May 01*	17 May 01
17 Apr 01	Analytical Method • Fortification • Extraction • Anion Exchange Column Clean-up • Filtering • Concentration	18 May 01	18 May 01*	17 May 01

QUALITY ASSURANCE STATEMENT
(Continued)

Date Inspected	Phase	Date reported to: Study Director & Management		McKenzie Management
18 May 01	Analytical Report	18 May 01	18 May 01*	18 May 01
11, 13 Jun 01	Final Analytical Report	14 Jun 01	14 Jun 01*	14 Jun 01

SQA.

Quality Assurance

June 28, 2001
Date

* These are the dates that the inspection report was sent to the Study Director.

SIGNATURE PAGE

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SUMMARY

Grain, forage and stover samples of transgenic field corn, resistant to glufosinate ammonium (Ammonium-DL-homoalanin-4-yl-(methyl)-phosphinate), were analyzed for residues of parent glufosinate ammonium (referred to as P or HOE-039866) and its major metabolites: disodium L-2-acetamido-4-methylphosphinato-butyrate (referred to as M1 or HOE-099730) and 3-methylphosphinico-propionic acid (referred to as M2 or HOE-061517).

The analyses were performed using AgrEvo (now Aventis CropSciences) analytical method BK/05/95 by agreement with Dow AgroSciences for the conduct of this study. The gas chromatographic results for this analysis are in terms of quantification of two derivative compounds. The first derivative is [methyl-4-(methoxymethyl)phosphinoyl-2-acetamido-butyrate] (referred to as HOE-064706) and the second is [methyl-3-(methoxymethyl)phosphinoyl-propionate] (referred to as HOE-070951). HOE-064706 is the result of derivatizing a common moiety of P and metabolite M1. The second gas chromatographic target analyte, HOE-070951, is the derivative of metabolite M2. By this method all glufosinate related residue data for a sample is collected in a single chromatographic run. The residues are quantified using a separate standard curve generated for the two derivatives produced by the method and the results are calculated and reported as free glufosinate ammonium acid equivalents (referred to as HOE-035956).

Fortified corn forage, grain and stover samples were analyzed with their respective sample sets. Separate fortifications were made with P and the two metabolites, M1 and M2. Fortifications for corn forage ranged from 0.05 µg/g to 4.00 µg/g with mean recovery of parent at 98% and of metabolite M1 at 100% and of metabolite M2 at 71%. Fortifications for corn grain ranged from 0.05 µg/g to 0.20 µg/g with mean recovery of parent at 95% and of metabolite M1 at 105% and of metabolite M2 at 78%. Fortifications for corn stover ranged from 0.05 µg/g to 6.00 µg/g with mean recovery of parent at 88% and of metabolite M1 at 100% and of metabolite M2 at 75%.

A frozen storage stability study was conducted to determine the stability of glufosinate ammonium and its metabolites in/on corn grain, forage and stover. The study was set up to cover a period of frozen storage that represented, at minimum, the longest interval of frozen storage between harvest of the sample and analysis of the sample. According to this definition, frozen storage intervals for all samples ranged between 47 and 85 days. The frozen storage stability study intervals tested ranged from 59 to 91 days. Glufosinate and its two metabolites, M1 and M2 were found to have been stable during frozen storage in all three matrices.

INTRODUCTION

Samples of a transgenic variety of field corn resistant to glufosinate ammonium treatment were collected during the year 2000 season in association with Dow AgroSciences study protocol number 000290 [1]. The samples were first shipped for compositing and homogenization processing to Dow AgroSciences LLC in Zionsville, IN. Representative subsamples of the thoroughly homogenized samples were shipped to McKenzie/Wright Laboratories, L.L.C. in Phoenix, AZ for analysis of the samples. This report describes the analytical phase of the study performed at McKenzie/Wright Laboratories, L.L.C. and presents the analytical results obtained.

LABORATORY SAMPLE HANDLING

Samples had been assigned unique Dow AgroSciences sample numbers, which were used by McKenzie/Wright Laboratories to track the samples throughout receipt, storage, and analysis. Samples were identified in groups based upon the collection time and trial site location. Fully homogenized samples were received frozen from Dow AgroSciences and were placed into frozen storage upon receipt. The temperature of the freezer used for sample storage at McKenzie/Wright Laboratories' facility averaged < -15 °C during the course of the sample analyses. No deviation above this temperature was observed over the storage period between sample receipt and analysis.

Table 1 summarizes the dates of receipt from Dow AgroSciences and the dates of analysis for each sample group.

Analysis of treated study samples was conducted between October 23, 2000 and January 9, 2001. Storage stability sample analyses were conducted between November 27, 2000 and April 21, 2001.

Storage Stability

As shown in Table 1, field-collected corn forage, grain and stover were held in frozen storage prior to analysis for a maximum of 72, 61 and 85 days, respectively. Frozen storage stability analyses were conducted to determine the stability of P and its two metabolites, M1 and M2. The storage interval tested for corn forage was 77 days. The storage interval tested for corn grain was 59 days and the storage interval tested for corn stover was 91 days.

Frozen storage stability in corn forage, grain and stover of glufosinate ammonium and its two metabolites, M1 and M2 was determined according to instructions provided by the Study Director in Amendment 5 to the Study Protocol. The storage stability study was set up by fortifying a bulk 300 g sample of each corn matrix with either P or a mixture of M1 and M2 metabolites. Corn forage was fortified at a concentration of 1.00 µg/g parent and each metabolite. Corn grain was fortified at 0.20 µg/g parent and each metabolite. Corn stover was fortified at a concentration of 1.50 µg/g parent and each metabolite. The fortified samples were mixed in a plastic bag and then the matrix was transferred to high density polyethylene (HDPE) containers supplied by the Sponsor for storage in the freezer.

Once the storage stability sample fortification was completed, a unique sample number was assigned and used to track the sample throughout the course of the study. On the day of fortification (Day 0), three fortified storage stability samples along with one control, one reagent blank and two freshly fortified samples were analyzed for parent. In addition, 3 fortified storage

stability samples and two freshly fortified control samples were analyzed for M1 and M2. The remaining fortified samples and controls were stored frozen under monitored conditions in a walk-in freezer held at < -15 °C. After the appropriate interval of days of frozen storage, samples fortified with either parent or a mixture of M1 and M2 metabolites were weighed and analyzed in triplicate along with one control and duplicate fresh fortifications of the parent and metabolites. Table 2 summarizes the results of the stability analyses.

ANALYTICAL METHODOLOGY

Analytical Standards

The analytical standard of glufosinate ammonium used for sample fortifications was supplied by Aventis CropSciences as code number AE F039866 00 1B99 0006 Batch No. 26880-125-M29 with a stated purity of 99.2%. Purity determination date was 14-Aug-98 and stability at – 20 °C was reported to be 48 months giving an expiry date of 14-Aug-02 [2].

The analytical standard of glufosinate ammonium metabolite M1 used for sample fortifications was supplied by Aventis CropSciences as code number AE F099730 00 1B96 0001 Batch No. H 1693 with a stated purity of 95.2%. Purity determination date was 18-May-00 and stability at – 20 °C was reported to be 24 months giving an expiry date of 19-May-02 [3].

The analytical standard of glufosinate ammonium metabolite M2 used for sample fortifications was supplied by Aventis CropSciences as code number AE F061517 00 1C97 0002 Batch No. KA 183+H 239,246,248, with a stated purity of 97.9%. The purity determination date was 23-Feb-00 and stability at – 20 °C was reported to be 48 months giving an expiry date of 23-Feb-04 [4].

The analytical standard used as the target gas chromatographic analyte for the derivatized glufosinate ammonium and metabolite M1 was methyl-4-(methoxymethyl)phosphinoyl-2-acetamido-butyrate. It was supplied Aventis CropSciences as code number AE F064706 00 1B99 0003 Batch No. 30099-014A, with a stated purity of 98.9%. Purity determination date

was 20-Jul-98 and stability at – 20 °C was reported to be 48 months giving an expiry date of 20-Jul-02 [5].

The analytical standard used as the target gas chromatographic analyte for the derivatized glufosinate ammonium metabolite M2 was methyl-3-(methoxymethyl)phosphinoylpropionate. It was supplied by Aventis CropSciences as code number AE F070951 00 1B99 0001 Batch No. WB 111, with a stated purity of 98.8%. Purity determination date was 12-Aug-99 and stability at – 20 °C was reported to be 48 months giving an expiry date of 12-Aug-03 [6].

Aventis CropSciences Analytical Method RAM Number BK/05/95

Residues of glufosinate ammonium and metabolites M1 and M2 were determined using the Aventis CropSciences analytical method RAM Number BK/05/95 [7].

For analysis, each 12.5 g sample was extracted once with 200 mL of distilled water by stirring at room temperature for one half hour. The sample was then brought to a total volume of 500 mL with water before filtering through Whatman 934-AH filter paper using a Büchner funnel and suction. A 100 mL aliquot of the filtered extract was then cleaned up using an anion exchange resin (BioRad AG 1X8) in the hydroxyl form.

The anion exchange clean-up column was prepared by converting 100 g of resin to the hydroxyl form by stirring it in 1 L of 1M sodium hydroxide until the pH was approximately 10 –11 to pH paper. The resin was then filtered and washed with approximately 4 L of distilled water until the filtrate was pH 5.5 – 7.5. Each sample clean-up column consisted of 12 mL of slurry packed resin in a 10.5 mm I.D. glass column, using distilled water for transfer and packing of the resin bed. A 100 mL aliquot of the sample extract was then eluted through the resin by gravity flow at a flow rate of one to two drops per second. The resin was then washed with 50 mL of distilled water and all eluates were discarded. The analytes were then eluted from the resin with approximately 100 mL of 50% formic acid into a 250 mL round bottom flask containing a few glass beads.

The formic acid extract was evaporated to dryness using a Büchi rotary evaporator with a waterbath set at ca. 50 °C. After the sample had gone to dryness 10 mL of distilled water was added and the sample was again evaporated to dryness.

The sample residue was dissolved by adding 3 mL of glacial acetic acid and sonicating with a swirling action for approximately one minute or until all sample residue was visibly dislodged from the sides of the flask and dissolved. The sample was then derivatized by adding 12 mL of trimethylorthoacetate and setting the sample to reflux for 4.5 hours.

After the reflux period the sample was allowed to cool to room temperature, usually overnight. When the sample was at room temperature, 15 mL of toluene was added and the sample reconstituted by evaporating the sample to approximately 2 mL using a Büchi rotary evaporator with a waterbath set at ca. 45 °C. The addition of 15 mL aliquots of toluene followed by evaporation to 0.5 to 1 mL was repeated two more times. The final toluene evaporation was continued to a final volume of ca. 1 mL without allowing the sample to go to dryness. The sample was then brought to 2 - 3 mL with toluene in preparation for an additional silica gel solid phase extraction clean-up step (SI-SPE).

The 500 mg SI-SPE cartridge was preconditioned by eluting approximately 10 mL of degassed methyl acetate/toluene (1:1 v/v) through each cartridge without allowing the cartridge bed to go dry. Elution was assisted using a vacuum box assembly designed for multiple SPE cartridge elutions.

To prepare the sample for SI-SPE clean-up, 4 mL ± 0.1 mL of methyl acetate was measured into the sample and the solution sonicated. The entire sample was then drawn up using a needle connected to a Luer® tipped 10 mL glass syringe. The volume was measured and adjusted to 8 mL with toluene. The empty sample flask was saved for additional transfer washes.

The sample in the syringe was transferred to the SI-SPE cartridge by inverting the syringe so that the needle pointed upwards, replacing the needle with a 0.45 µm disposable filter and loading the contents of the syringe through the filter with pressure on the syringe plunger so that the sample was delivered uniformly onto the top of the SI-SPE cartridge. The sample was allowed to elute through the SI-SPE cartridge to waste, being careful not to let the silica bed go dry. The filter connected to the syringe was replaced by the needle, and the empty sample flask was rinsed with 10 mL of methyl acetate. The methyl acetate rinse was transferred from the flask into the syringe using the needle previously used for the initial transfer and then added to the SI-SPE cartridge through the disposable filter as previously described. The rinse eluate was discarded and the silica gel bed was then allowed to go dry. The derivative compounds, HOE-064706 and HOE-070951 were eluted from the cartridge using 5.5 mL of methanol/methyl acetate (1:1 v/v), and the eluate was collected in a clean 10 mL screw top test tube. The test tube containing the sample was removed from the SPE vacuum box and placed into a N-Evap Analytical Concentrator with a water bath temperature set at ca. 50 °C where the sample volume was reduced under nitrogen to approximately 0.5 to 1 mL. The sample was then quantitatively transferred to an appropriate volumetric flask using methyl acetate, and submitted for instrumental analysis.

Instrumental analysis of the samples was performed by capillary gas chromatography with phosphorus mode flame photometric detection. Typical gas chromatographic conditions are summarized in Table 3. Typical chromatograms of calibration standards, controls, fortified controls, and treated samples from the analyses of corn forage, corn grain and corn stover can be found in Figures 1-13j.

Method Recovery

Method performance, in terms of recovery of P and metabolites M1 and M2 from corn forage, grain and stover, was determined at the time of analysis of treated samples by fortifying the appropriate corn control matrix with known amounts of the analytes and analyzing as described above. Fortified recoveries of P, M1 and M2 in corn forage were analyzed over a range of

0.05 µg/g to 4.00 µg/g glufosinate acid equivalents with a mean recovery of 98% for the parent, 100% for M1 and 71% for M2. Fortified recoveries of P, M1 and M2 in corn grain were analyzed over a range of 0.05 µg/g to 0.20 µg/g glufosinate acid equivalents with a mean recovery of 95% for the parent, 105% for M1 and 78% for M2. Fortified recoveries of P, M1 and M2 in corn stover were analyzed over a range of 0.05 µg/g to 6.00 µg/g glufosinate acid equivalents with a mean recovery of 88% for the parent, 100% for M1 and 75% for M2. Detailed summaries of the recoveries of P and metabolites M1 and M2 are presented in Table 4.

Limits of Detection and Quantification

The target limit of detection (LOD) of 0.015 µg/g and limit of quantification (LOQ) of 0.05 µg/g for the analysis of glufosinate ammonium and its metabolites in corn forage, grain and stover were confirmed by fortifying the respective control samples with each analyte at levels equivalent to the target LOD and LOQ. Fortifications at the target LOD were used only to confirm instrumental detection at 0.015 µg/g.

The target LOD and LOQ for each analyte in each matrix were statistically evaluated using the recovery results obtained from respective control samples fortified with each analyte at the target LOQ. In addition, using the recovery for all fortifications of P and M1 at the target LOQ, the LOD and LOQ for P + M1 was calculated. This combined LOD and LOQ is used to report residues found as the derivative HOE-064706, because it is not possible to determine if incurred residues in field treated samples result from residues of P or M1 individually. For all LOD and LOQ calculations, the standard deviation, *s*, of the amount found in the recovery samples was determined and the LOD and LOQ were calculated as equal to 3*s* and 10*s*, respectively [8]. The target LOD's and LOQ's were statistically supported if the calculated values were equivalent to or less than the target values. In these cases the target LOD and LOQ were used to evaluate and report the corresponding study data. If the calculated LOD's and LOQ's were greater than the target values, the target values were not supported statistically and the calculated values were used to evaluate and report the corresponding data.

The following table summarizes the statistical evaluation of the data for each matrix and analyte.

Matrix and Analyte	Fortification (Target) Concentration µg/g		Concentration Found at Target LOQ, µg/g		Calculated Limit Values, µg/g		Limits Used for Study Evaluations, µg/g	
	LOD	LOQ	Mean	St Dev, s	LOD	LOQ	LOD	LOQ
Forage								
P	0.015	0.05	0.0525	0.0059	0.018	0.059	0.018	0.059
M1	0.015	0.05	0.0521	0.0019	0.006	0.019	0.015	0.050
P + M1	0.015	0.05	0.0523	0.0042	0.013	0.042	0.015	0.050
M2	0.015	0.05	0.0386	0.0063	0.019	0.063	0.019	0.063
Grain								
P	0.015	0.05	0.0514	0.0057	0.017	0.057	0.017	0.057
M1	0.015	0.05	0.0560	0.0066	0.020	0.066	0.020	0.066
P + M1	0.015	0.05	0.0537	0.0064	0.019	0.064	0.019	0.064
M2	0.015	0.05	0.0382	0.0024	0.007	0.024	0.015	0.050
Stover								
P	0.015	0.05	0.0464	0.0041	0.012	0.041	0.015	0.050
M1	0.015	0.05	0.0526	0.0035	0.010	0.035	0.015	0.050
P + M1	0.015	0.05	0.0495	0.0049	0.015	0.049	0.015	0.050
M2	0.015	0.05	0.0384	0.0061	0.018	0.061	0.018	0.061

The target LOQ recoveries used for the above table calculations are presented in Table 4. The spreadsheet for each analytical set lists the target LOD and target LOQ of 0.015 µg/g and 0.05 µg/g, respectively. However, data reporting on the spreadsheet is in accordance with the procedures described above, where the LOD and LOQ for P + M1 was used to report incurred residues found as the derivative HOE-064706 and the LOD and LOQ for M2 was used for reporting incurred residues found as the derivative HOE-070951.

CALCULATIONS

Determination of Percent Recovery of Analytes from Corn Forage, Grain and Stover

Because all fortification and GC standard concentrations are reported in terms of glufosinate free acid equivalents, it is not necessary to apply a molecular weight conversion factor within these calculations to account for the molecular weight difference between the fortification standards and the derivatized standards used for gas chromatographic quantification.

All calculations were performed using Microsoft's Excel Spreadsheet program. The spreadsheets, prepared by the Study Director, were preprogrammed and preformatted to accept data values from the analytical procedure and instrumental analysis so that residue values and percent recovery for each analytical set could be more easily and uniformly determined. Calculations were performed by the spreadsheet without rounding intermediate values in the calculations. However, intermediate values in the calculation are reported in the spreadsheet as rounded values. Therefore, calculation checks that start with one of the intermediate values reported in the spreadsheet, may produce slightly different end values than those shown as reported residues or reported percent recovery. Hand calculated results exactly matching those reported in the spreadsheet may be obtained by starting the calculation with the sample peak area count and preserving at least six significant figures in the intermediate values.

Data was input to the spreadsheet from the sample work-up portion of the analytical procedure, including sample weight, aliquot and final volume for analysis. Results of the instrumental analysis were then input to complete the calculation of residue concentrations and percent recovery.

From the instrumental analysis, a multilevel calibration response curve was generated for analysis of each sample set by injecting a mixed standard of HOE-099730 and HOE-061517 ranging in concentrations from 0.015 µg/mL to 0.30 µg/mL glufosinate free acid equivalents

(0.03 µg/g to 0.60 µg/g sample equivalent concentration). The detector response for each analyte was determined by measuring peak areas in the standard and sample solutions

The equation for the calibration curve for both analytes was calculated by the Excel spreadsheet using the program's "Intercept", "Slope" and "RSQ" mathematical functions to operate on the full range of analyte concentrations for the standards (in µg/mL) as the X values, and the corresponding range of peak area responses for that analyte as the Y value. These functions returned the values for the calibration curve parameter and constant, using a linear regression model of the following mathematical form:

(1) $Y = mX + b$

where: Y = Peak Area
X = Analyte Concentration (µg/mL)
b = The Y intercept of X
m = The slope of the linear regression

To calculate the analyte concentration (µg/mL) from the standard calibration curve, equation (1) was rearranged to give:

(2) $X = (Y - b)/m$

The concentrations for each analyte in each sample analyzed were calculated by the spreadsheet, which was programmed to insert into Equation (2) the values for Y, b and m determined from the set calibration curve calculation on another part of the spreadsheet. Equation (2) was set up as the spreadsheet cell equation at the cell location designated for reporting the analyte concentration.

The analyte concentration in µg/g was then calculated using the following equation:

(3) Analyte Conc. (µg/g) = Analyte Concentration (µg/mL) x MF (mL/g)

Where the Method Factor: $MF = \left(\frac{EV \times FV \times DF}{AV \times SM}\right)$

EV = Extraction Volume, 500 mL
FV = Final Volume, 5.0 mL
DF = Dilution Factor, variable
AV = Aliquot Volume, 100 mL
SM = Sample Mass, 12.5 g

For example, using the data from control corn forage sample 32887101 fortified with 0.05 µg/g P (see Figure 4a for chromatogram and Figures 14, injection 13 from spreadsheet data), along with equation (2), the standard curve generated for that set of samples yielded values from Equation (1) for *m* and *b* of 18670.87399 and - 54.09573, respectively for the P + M1 analyte, HOE 064706. Using Equation (2) to interpolate the sample peak area count of 398.91422 for this fortification gave the concentration of glufosinate free acid equivalent as follows:

$$\text{Glufosinate Concentraion µg/mL Fortified Sample} = \frac{398.91422 - (-54.9573)}{18670.87399}$$

$$= 0.0243 \text{ µg/mL glufosinate acid equiv.}$$

The analyte concentration was found using Equation (3) where the method factor of 2 mL/g was calculated using a dilution factor of 1x. Equation (3) then gives for this control fortification:

$$\text{Glufosinate Acid Equivalents} = 0.0243 \times 2$$

$$= 0.0485 \text{ µg/g}$$

The percent recovery of each fortified sample was then calculated by dividing the concentration found by the concentration added and multiplying the result by 100. For example, continuing with the data from control corn forage sample 32887101 fortified with 0.05 µg/g P (Figures 14, injection 13):

$$(4) \qquad \% \text{ Recovery} = \frac{\text{Concentration Found}}{\text{Concentration Added}} \times 100$$

$$= \frac{0.0485 \text{ µg/g}}{0.050 \text{ µg/g}} \times 100$$

$$= 97\%$$

In one set, the reinjection of corn stover set 2S, the control sample displayed some low level interferences (LOD level or below). The analyte peak areas of the fortified samples were adjusted for the interferences observed in the control sample by simple subtraction of the µg/g control residue found from the µg/g found for the fortified sample prior to determining percent recovery for the fortification.

Determination of the Residues of the Analytes in Corn Forage, Grain and Stover

The residue concentrations for the P + M1 metabolite were determined separately from residue concentrations for M2 in the treated samples by separate quantification of the derivatized analytes, HOE-064706 and HOE-070951, respectively. For treated samples the residues found were then corrected for the average recovery of the set for the two derivatized analytes. This correction is recorded as the corrected value of µg/g found. The total glufosinate acid equivalent residue for the sample was then determined by simply adding the recovery corrected residue value determined for P + M1 and the recovery corrected residue value determined for M2.

Calculation of these separate residues was performed using Equations (2) and (3) as described in the above section for determination of fortification recoveries. The recovery correction was performed using the following calculation:

$$(5) \qquad \text{Corrected } \mu g/g = \frac{\mu g/g \text{ Found}}{\text{Average Recovery for Analytical Set}} \times 100$$

As an example of the calculations leading to the report of total glufosinate acid equivalent for a treated sample, the calculations for treated corn forage sample 33060402 is given below. The example chromatogram for this sample is found in Figures 5c and 5d and the spreadsheets, which correspond to the two chromatograms, may be found in Figures 15 and 16, respectively.

The residue concentration for parent glufosinate plus M1 could not be determined from the initial chromatographic analysis because the peak area response for the common analyte, HOE-064706, exceeded the range of the standard calibration curve. However, the concentration of metabolite M2 could be determined from this initial analysis as follows:

From Equation (1) $m = 23143.03053$
$b = 40.46943$

Therefore from Equation (2):

$$\text{M2 Solution Concentration (as Glufosinate Acid Equivalents)} = \frac{(544.29163 - (40.46943))}{23143.03053}$$

$$= 0.0218 \ \mu g/mL$$

and from Equation (3) we have:

$$\mu g/g \text{ Glufosinate Acid Equivalent Found Metabolite M2} = 0.0218 \times 2$$

$$= 0.0435 \ \mu g/g \text{ acid equivalents found}$$

And finally, from Equation (5) and an average recovery of 61% from the calibration curve for this part of the field sample set, the corrected metabolite M2 residue was calculated as follows:

$$\text{Corrected } \mu g/g \text{ M2} = \frac{0.0435 \ \mu g/g \text{ Glufosinate acid equivalents found}}{61\%} \times 100$$

$$= 0.0713 \ \mu g/g \text{ corrected Glufosinate acid equivalents}$$

A second instrumental analysis was performed on a 4x dilution of sample 33060402 in order to quantify the parent plus M1 derivative concentration. From Equation 1 the slope and intercept, *m* and *b* for the reinjection set of samples were found to be 17644.17473 and –108.57411, respectively. Inserting these values and the peak area of 2693.42456 for the diluted sample into Equation (2) yields the following:

$$\text{Glufosinate + M1 Solution Concentration (as Glufosinate Acid Equivalents)} = \frac{(2693.42456 - (-108.57411))}{17644.17473}$$

$$= 0.1588 \ \mu g/mL \text{ Glufosinate plus M1}$$

And From Equation (3), using a 4x dilution rate we have:

$$\mu g/g \text{ Glufosinate Acid Equivalent Found (Parent + M1)} = 0.1588 \ \mu g/mL \times 8$$

$$= 1.2704 \ \mu g/g \text{ acid equivalents found}$$

Correcting for an average recovery of 107% for the analytical set gives the following:

$$\text{Corrected } \mu g/g \text{ Parent + M1} = \frac{1.2704 \ \mu g/g \text{ Glufosinate acid equivalents found}}{107\%} \times 100$$

$$= 1.1873 \ \mu g/g \text{ corrected Glufosinate acid equivalents}$$

The total glufosinate acid equivalent residue for corn forage sample 33060402 is then calculated by adding the corrected results for the M2 analysis calculated above (0.0713 µg/g) to the results obtained for the parent + M1 (1.1873 µg/g) analysis, as follows:

Total Glufosinate acid equivalent residue (Sample 33060402) = 1.1873 µg/g + 0.0713 µg/g

= 1.259 µg/g acid equivalents

STATISTICS

Statistical methods of mean, standard deviation, and linear regression were used to analyze the data.

ANALYTICAL DATA

Preparation of the samples was performed by sets. There were a total of 15 sets of samples analyzed; 3 each for forage, grain and stover field samples, and 2 each for frozen storage stability of forage, grain and stover samples. The average preparation set for field samples was 15 samples, consisting of 1 reagent blank, 1 control, 7 fortified controls, and 6 field samples. The average preparation set of 7 storage stability samples consisted 1 reagent blank, 1 control, 2 fortified controls, and 3 storage stability samples.

The instrument stability was not adequate to produce a quality standard calibration curve over the extended run time required by the large sized sample set. Therefore the field sample preparation sets were analyzed by splitting the analysis of the 15 samples so that approximately half the samples and fortifications were analyzed by an initial standard curve, called "Curve 1", while the remaining samples and fortifications were quantified using a trailing standard curve, called "Curve 2". Samples analyzed by Curve 1 were typically the reagent blank, one control sample, four fortified controls and two to four study samples. Curve 2 analysis typically consisted of 1 control, 3 fortified controls and 3 to four study samples. This analytical instrument strategy resulted in producing two subsets of instrumental quantification, or curve data, for each set of field samples prepared. The average fortified control recovery used to correct study field sample residues for method efficiency were derived from the two individual calibration curve data sets, not from the overall average recovery for the preparation set. The storage stability sets did not require splitting the analysis into two different curves because of the fewer number of samples extracted per set.

Data from each preparation set and calibration curve set was summarized in the form of data spreadsheets. Data spreadsheets have been grouped together based on matrix, analytes, analysis set identification and standard curve. These spreadsheets are presented in Figures 14. through 45. Data presented include the peak areas of the analytes, average set recoveries, and values of the calibration curves. Calculated sample analysis results are expressed as uncorrected values and corrected concentrations (values corrected for average analytical set recovery). In addition, the spreadsheets provide the values for the LOD and LOQ that were applied to the reported sample results. The residue concentration was reported as ND if the corrected concentration was below the LOD. The residue concentration was reported as the corrected concentration in parentheses, ( ), if the corrected concentration was less than the LOQ but greater than or equal to the LOD. The residue concentration was reported as the corrected concentration, if the corrected concentration was equal to or greater than the LOQ. Residue concentrations above the LOQ are reported with a relative standard deviation of less than ± 20% at the 95% confidence level[8]. Residue concentrations reported below the LOD and LOQ have a higher degree of uncertainty than values above the LOQ.

Definitions of terms and abbreviations used in the spreadsheets can be found in Table 5. Analytical data for the field study samples are shown in Figures 14. through 33. Results for the storage stability sample analyses are shown in Figures 34. through 45.

DISCUSSION

Certain samples required dilution and reanalysis in order to quantify P + M1. Corn forage samples 33060402 and 33060403 required a 4x dilution, to bring chromatographic responses for HOE-064706 (P + M1) within the valid detector response range. The 1.0 µg/g fortification on control 32887101, which was prepared with the analysis set and analyzed within the calibration curve set, was also reinjected with the diluted samples in order to verify the continued integrity of analyte recovery for the reinjected set. The percent recovery found for this reinjection of the fortified control was within 20% of the original recovery for this fortified control, demonstrating stability of the extract solution. The average curve set recovery of the original injection was used

to correct the P + M1 residue value of the diluted sample. This recovery value of the reinjected fortified sample was not used in evaluating the overall recovery for the corn forage matrix because it was a reinjection.

Corn stover sample 33062002 required a 20x dilution and sample 33062003 required a 10x dilution to bring responses for HOE-064706 (Parent + M1) within the valid detector response range. These dilutions were analyzed along with a reinjection of the 1.0 µg/g M1 + M2 fortification to control sample 32906101. The percent recovery found for this reinjection of the fortified control was within 20% of the original recovery. The fortified control sample reinjected with the diluted treated samples was not originally analyzed in the same standard curve as the undiluted treated samples. However, it was prepared and extracted with the undiluted treated samples, and therefore demonstrates the stability of the sample solutions over the time interval required for reanalysis of the dilutions. The average curve set recovery from the fortified control sample was used to correct the P + M1 residue value of the diluted samples. This recovery value of the reinjected fortified sample was not used in evaluating the overall recovery for the corn stover matrix because it was a reinjection of the same fortified sample.

REFERENCES

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3. Assay of Analytical Standard AE F099730, Certificate of Analysis Number AZ 08471, 2000, Aventis CropSciences.

4. Assay of Analytical Standard AE F061517, Certificate of Analysis Number AZ 08304, 2000, Aventis CropSciences.

5. Assay of Analytical Standard AE F064707, Certificate of Analysis Number AZ 07484, 1998, Aventis CropSciences.

6. Assay of Analytical Standard AE F070951, Certificate of Analysis Number AZ 08044, 1999, Aventis CropSciences

7. Czarnecki, J. J., Unpublished Analytical Method No. BK/05/95, "Gas Chromatographic Determination of HOE-039866 (Glufosinate-Ammonium) and Its Metabolites as Residues in Glufosinate-Resistant Corn and Soybean RAC and Processed Commodities", June 12, 1995.

8. Keith, L. H.; Crummett, W.; Deegan, J., Jr.; Libby, R. A.; Taylor, J. K.; Wentler, G., *Anal. Chem.* 1983, *55*, 2210-2218

Table 1. Summary of Sampling, Receiving and Analysis Dates

Dow AgroSciences Sample Group Number	Sample Description	Site Location	Sampling Date	Receipt Date from Dow AgroSciences	Analysis Extraction Date[a]	Sample Frozen Storage Interval[b]
328871	Corn Forage	000290IA1	28-Aug-00	26-Sep-00	23-Oct-00	56
328928	Corn Forage	000290IA2	07-Sep-00	11-Oct-00	13-Nov-00	67
330604	Corn Forage	000290IL1	06-Sep-00	26-Sep-00	23-Oct-00	47
329045	Corn Forage	000290MN1	14-Sep-00	11-Oct-00	13-Nov-00	60
330639	Corn Forage	000290OH1	11-Sep-00	11-Oct-00	17-Nov-00	67
331767	Corn Forage	000290IN1	06-Sep-00	11-Oct-00	17-Nov-00	72
328898	Corn Grain	000290IA1	04-Oct-00	8 & 14-Nov-00	04-Dec-00	61
328936	Corn Grain	000290IA2	12-Oct-00	8 & 14-Nov-00	30-Nov-00	49
330612	Corn Grain	000290IL1	16-Oct-00	8 & 14-Nov-00	04-Dec-00	49
329053	Corn Grain	000290MN1	16-Oct-00	14-Nov-00	04-Dec-00	49
330647	Corn Grain	000290OH1	24-Oct-00	7-Dec-00	11-Dec-00	48
331775	Corn Grain	000290IN1	18-Oct-00	7-Dec-00	11-Dec-00	54
328901	Corn Stover	000290IA1	04-Oct-00	8 & 14-Nov-00	28-Dec-00	85
328944	Corn Stover	000290IA2	12-Oct-00	8 & 14-Nov-00	28-Dec-00	77
330620	Corn Stover	000290IL1	16-Oct-00	8 & 14-Nov-00	03-Jan-01	79
329061	Corn Stover	000290MN1	16-Oct-00	8-Nov-00	03-Jan-01	79
330655	Corn Stover	000290OH1	24-Oct-00	7-Dec-00	08-Jan-01	76
331783	Corn Stover	000290IN1	18-Oct-00	7-Dec-00	08-Jan-01	82

[a] Date sample was extracted for analysis.

[b] Interval calculated from sampling date to sample extraction date.

Table 2. Frozen Storage Stability of Glufosinate Ammonium and Metabolites M1 and M2 in Corn Forage, Grain and Stover

McKenzie/Wright Analysis Set	DAS Control Sample Identification	Date Analysis Began	Glufosinate Acid Equivalent - P			Glufosinate Acid Equivalent - M1			Glufosinate Acid Equivalent - M2		
			µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery
Day 0 FSS -Forage	34240801	27-Nov-00	1.0	1.07	107	1.0	0.97	97	1.0	1.00	100
Day 0 FSS -Forage	34240801	27-Nov-00	1.0	0.93	93	1.0	1.17	117	1.0	1.29	129
Day 0 FSS -Forage	34240801	27-Nov-00	1.0	1.09	109	1.0	1.17	117	1.0	1.23	123
Day X FSS-Forage	34240801	12-Feb-01	1.0	0.95	95	1.0	1.01	101	1.0	0.92	92
Day X FSS-Forage	34240801	12-Feb-01	1.0	0.96	96	1.0	0.95	95	1.0	0.91	91
Day X FSS-Forage	34240801	12-Feb-01	1.0	1.05	105	1.0	1.01	101	1.0	0.96	96

McKenzie/Wright Analysis Set	DAS Control Sample Identification	Date Analysis Began	Glufosinate Acid Equivalent - P			Glufosinate Acid Equivalent - M1			Glufosinate Acid Equivalent - M2		
			µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery
Day 0 FSS-Grain	34241601	12-Jan-01	0.2	0.194	97	0.2	0.220	110	0.2	0.222	111
Day 0 FSS-Grain	34241601	12-Jan-01	0.2	0.217	108	0.2	0.232	116	0.2	0.222	111
Day 0 FSS-Grain	34241601	12-Jan-01	0.2	0.202	101	0.2	0.185	92	0.2	0.166	83
Day X FSS-Grain	34241601	12-Mar-01	0.2	0.188	94	0.2	0.174	87	0.2	0.191	96
Day X FSS-Grain	34241601	12-Mar-01	0.2	0.193	96	0.2	0.184	92	0.2	0.194	97
Day X FSS-Grain	34241601	12-Mar-01	02	0.204	102	0.2	0.180	90	0.2	0.181	90

McKenzie/Wright Analysis Set	DAS Control Sample Identification	Date Analysis Began	Glufosinate Acid Equivalent - P			Glufosinate Acid Equivalent - M1			Glufosinate Acid Equivalent - M2		
			µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery
Day 0 FSS-Stover	34242401	16-Jan-01	1.5	1.31	87	1.5	1.54	103	1.5	1.62	108
Day 0 FSS-Stover	34242401	16-Jan-01	1.5	1.53	102	1.5	1.52	101	1.5	1.77	118
Day 0 FSS-Stover	34242401	16-Jan-01	1.5	1.50	100	1.5	1.42	95	1.5	1.56	104
Day X FSS-Grain	34242401	17-Apr-01	1.5	1.50	100	1.5	1.55	103	1.5	1.49	99
Day X FSS-Grain	34242401	17-Apr-01	1.5	1.37	91	1.5	1.45	97	1.5	1.42	95
Day X FSS-Grain	34242401	17-Apr-01	1.5	1.62	108	1.5	1.42	95	1.5	1.34	89

Table 3 Typical Operating Parameters for Gas Chromatograph with Flame Photometric Detector

Parameter:	Condition:
Instrument Type:	HP 6890 (Now Agilent 6890)
Detector Type:	Flame Photometric operated in Phosphorus mode
Capillary Column	Restek RTX-Wax; MKL No. CC-RTXwax-260-00
Column Size:	Capillary 15m x 0.53 mm ID x 1.0 µm
Temperatures:	
Detector Temp:	230 °C
Oxidizer Flow:	100 mL/min (air)
Hydrogen Flow	150 mL/min
Oven:	145 °C for 1.5 min
Ramp1:	2.5 °C/min
Temperature:	150 °C for 0 min
Ramp 2:	5 °C/min
Temperature:	240 °C for 5 min
Run Time	26.5 minutes
Flows:	
Carrier:	157 cm/sec helium
Inlet Purge Flow:	74.5 mL/minute at 0.5 minutes
Inlet Pressure:	9.79 psi
Make-up:	Nitrogen 30 mL/min
Retention Time:	HOE-070951 = ca. 5.5 minutes
Retention Time:	HOE-064706 = ca. 19.7 minutes
Injection Volume:	5 µL
Data System:	HP Chemstation A.05.04
Method:	GLUFOSIN.M
Data Subdirectory	C:\HPCHEM\2\DATA\GLUFOSIN

NOTE: The raw data identifies all equipment used in the study and exact parameters used for each analysis. The equipment and parameters listed here are representative.

Table 4. Recovery of Glufosinate Ammonium and Metabolites M1 and M2 from Fortified Corn Control Samples

McKenzie/Wright Analysis Set	DAS Control Sample Identification	Extraction Date	Glufosinate Acid Equivalent - P			Glufosinate Acid Equivalent - M1			Glufosinate Acid Equivalent - M2		
			µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery
Forage											
1F	32887101	23-Oct-00	0.05	0.0485	97%	1.00	0.8143	81%	1.00	0.5223	52%
1F	32887101	23-Oct-00	0.05	0.0484	97%	0.05	0.0531	106%	0.05	0.0308	62%
1F	32887101	23-Oct-00	1.00	0.9737	97%	0.05	0.0545	109%	0.05	0.0302	60%
2F	32892801	13-Nov-00	0.05	0.0565	113%	4.00	4.2695	107%	4.00	2.8276	71%
2F	32892801	13-Nov-00	0.05	0.0625	125%	0.05	0.0522	104%	0.05	0.0421	84%
2F	32892801	13-Nov-00	4.00	3.5888	90%	0.05	0.0529	106%	0.05	0.0446	89%
3F	33063901	17-Nov-00	0.05	0.0514	103%	0.50	0.3594	72%	0.50	0.3437	69%
3F	33063901	17-Nov-00	0.05	0.0476	95%	0.05	0.0504	101%	0.05	0.0423	85%
3F	33063901	17-Nov-00	0.50	0.3416	68%	0.05	0.0493	99%	0.05	0.0414	83%
Day 0 FSS-Forage	34240801	27-Nov-00	1.00	1.0069	101%	1.00	1.1011	110%	1.00	0.5854	59%
Day 0 FSS-Forage	34240801	27-Nov-00	1.00	0.9332	93%	1.00	0.9840	98%	1.00	0.6055	61%
Day X FSS-Forage	34240801	12-Feb-01	1.00	1.0133	101%	1.00	1.0500	105%	1.00	0.7598	76%
Day X FSS-Forage	34240801	12-Feb-01	1.00	0.9563	96%	1.00	1.0744	107%	1.00	0.7511	75%
				Average Recovery	98%		Average Recovery	100%		Average Recovery	71%
				Standard Deviation	13%		Standard Deviation	11%		Standard Deviation	12%

Table 4. Recovery of Glufosinate Ammonium and Metabolites M1 and M2 from Fortified Corn Control Samples (Continued)

McKenzie/Wright Analysis Set	DAS Control Sample Identification	Extraction Date	Glufosinate Acid Equivalent - P			Glufosinate Acid Equivalent - M1			Glufosinate Acid Equivalent - M2		
			µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery
Grain											
1G	32889801	30-Nov-00	0.05	0.0544	109%	0.20	0.1895	95%	0.20	0.1464	73%
1G	32889801	30-Nov-00	0.05	0.0602	120%	0.05	0.0621	124%	0.05	0.0389	78%
1G	32889801	30-Nov-00	0.20	0.1879	94%	0.05	0.0622	124%	0.05	0.0394	79%
2G	33061201	04-Dec-00	0.05	0.0507	101%	0.10	0.1134	113%	0.10	0.0779	78%
2G	33061201	04-Dec-00	0.05	0.0524	105%	0.05	0.0562	112%	0.05	0.0386	77%
2G	33061201	04-Dec-00	0.10	0.0760	76%	0.05	0.0576	115%	0.05	0.0399	80%
3G	33064701	11-Dec-00	0.05	0.0462	92%	0.08	0.0823	103%	0.08	0.0693	87%
3G	33064701	11-Dec-00	0.05	0.0444	89%	0.05	0.0537	107%	0.05	0.0388	78%
3G	33064701	11-Dec-00	0.08	0.0795	99%	0.05	0.0444	89%	0.05	0.0334	67%
Day 0 FSS-Grain	34241601	12-Jan-01	0.20	0.1711	86%	0.20	0.2004	100%	0.20	0.1600	80%
Day 0 FSS-Grain	34241601	12-Jan-01	0.20	0.1788	89%	0.20	0.1954	98%	0.20	0.1686	84%
Day 0 FSS-Grain	34241601	12-Jan-01	0.20	0.1898	95%	0.20	0.1948	97%	0.20	0.1635	82%
Day X FSS-Grain	34241601	12-Mar-01	0.20	0.1819	91%	0.20	0.1963	98%	0.20	0.1550	78%
Day X FSS-Grain	34241601	12-Mar-01	0.20	0.1701	85%	0.20	0.1987	99%	0.20	0.1433	72%
				Average Recovery	95%		Average Recovery	105%		Average Recovery	78%
				Standard Deviation	11%		Standard Deviation	11%		Standard Deviation	5%

Table 4. Recovery of Glufosinate Ammonium and Metabolites M1 and M2 from Fortified Corn Control Samples (Continued)

McKenzie/Wright Analysis Set	DAS Control Sample Identification	Extraction Date	Glufosinate Acid Equivalent - P			Glufosinate Acid Equivalent - M1			Glufosinate Acid Equivalent - M2		
			µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery	µg/g Added	µg/g Found	Percent Recovery
Stover											
1S	32890101	28-Dec-00	0.05	0.0442	88%	6.00	5.9108	99%	6.00	4.9382	82%
1S	32890101	28-Dec-00	0.05	0.0451	90%	0.05	0.0534	107%	0.05	0.0399	80%
1S	32890101	28-Dec-00	6.00	4.8412	81%	0.05	0.0470	94%	0.05	0.0300	60%
2S	32906101	03-Jan-01	0.05	0.0512	102%	1.00	1.0063	101%	1.00	0.8018	80%
2S	32906101	03-Jan-01	0.05	0.0519	104%	0.05	0.0560	112%	0.05	0.0459	92%
2S	32906101	03-Jan-01	1.00	0.8386	84%	0.05	0.0563	113%	0.05	0.0442	88%
3S	33178301	08-Jan-01	0.05	0.0441	88%	4.00	3.3481	84%	4.00	2.3283	58%
3S	33178301	08-Jan-01	0.05	0.0420	84%	0.05	0.0522	104%	0.05	0.0345	69%
3S	33178301	08-Jan-01	4.00	3.1232	78%	0.05	0.0508	102%	0.05	0.0356	71%
Day 0 FSS-Stover	34242401	16-Jan-01	1.50	1.3085	87%	1.50	1.3945	93%	1.50	0.9587	64%
Day 0 FFS-Stover	34242401	16-Jan-01	1.50	1.2704	85%	1.50	1.4616	97%	1.50	1.0268	68%
Day X FFS-Stover	34242401	17-Apr-01	1.50	1.2935	86%	1.50	1.5412	103%	1.50	1.2167	81%
Day X FFS-Stover	34242401	17-Apr-01	1.50	1.2530	84%	1.50	1.4103	94%	1.50	1.1266	75%
			Average Recovery		88%	Average Recovery		100%	Average Recovery		75%
			Standard Deviation		7%	Standard Deviation		8%	Standard Deviation		11%

Table 5. Definitions Associated with Sample Analysis Spreadsheets

Runsheet, results, and method factor pages.

(P) = Fortification made with Parent Glufosinate ammonium (HOE-039886)

(M1 + M2) = Fortifications made with metabolite HOE-099730 and HOE-061517, respectively

DUP = a sample set up as a duplicate of another sample being analyzed

M2 = Metabolite HOE-061517 detected as derivative HOE-070951

P + M1 = Parent Glufosinate ammonium (HOE-039886) and metabolite HOE-090730 detected as the common derivative analyte, HOE-064706

Glufosinate Acid Equivalent = All compound concentrations are expressed based on their molecular weight equivalency to the molecular weight of glufosinate free acid, thus avoiding the need for molecular weight ratio calculations as part of the calculations to determine results of the chromatographic analysis

µg/mL Extract = (Peak Area - Intercept)/Slope

Method Factor = [(Final Volume (mL)) * (Extraction Volume (mL))]/ [(Aliquot Volume (mL)) * (Sample Weight (g))] * Dilution Factor

Found µg/g = (µg/mL Extract*Method Factor).

Corrected µg/g = [(Found µg/g)*(100/Average Curve Recovery)].

Reported µg/g = The µg/g reported at the end of the study, after the LOD and LOQ have been calculated.

NA = Not Applicable to this sample.

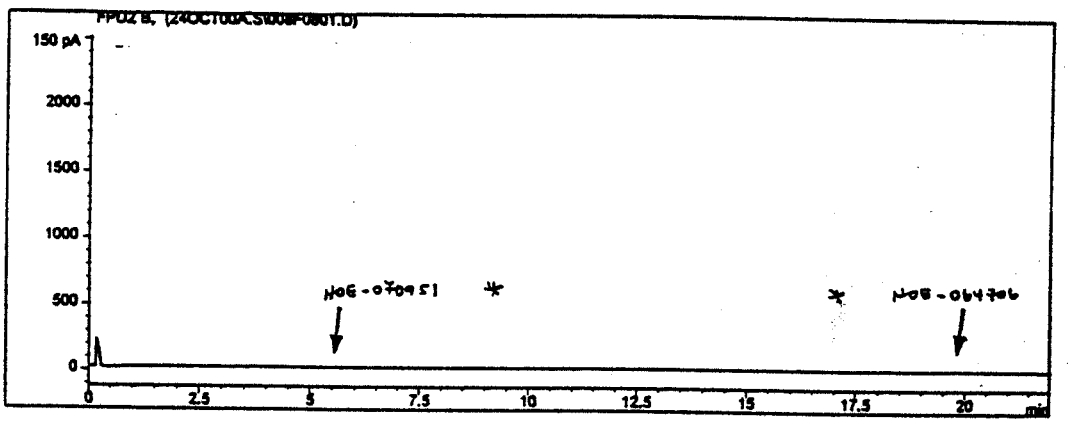
ND = Not Detected. Less than the LOD

( ) = Concentration is between the LOD and LOQ. Concentration has a greater degree of uncertainty.

Percent Recovery =[(µg/g of M2 or (P+M1) control corrected)/(µg/g M2 or (P+M1) found)] * 100

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\008F0801.D                Reagent blank
    Reagent blank
    500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  1F

    ================================================================================
    Injection Date  : 10/24/00 9:46:55 PM              Seq. Line :   8
    Sample Name     : Reagent blank                         Vial :   8
    Acq. Operator   : MYL                                    Inj :   1
                                                       Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/24/00 2:23:24 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/26/00 11:28:43 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```




```
    ================================================================================
                              External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, October 26, 2000 11:25:27 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type      Area      Amt/Area     Amount    Grp   Name
     [min]           150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.540           -           -          -          HOE-070951
     19.798           -           -          -          HOE-064706

    Totals :                                  0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found
```

* the retention time and compound name were added on 26 Oct 00.
MYL 07 Jun 01

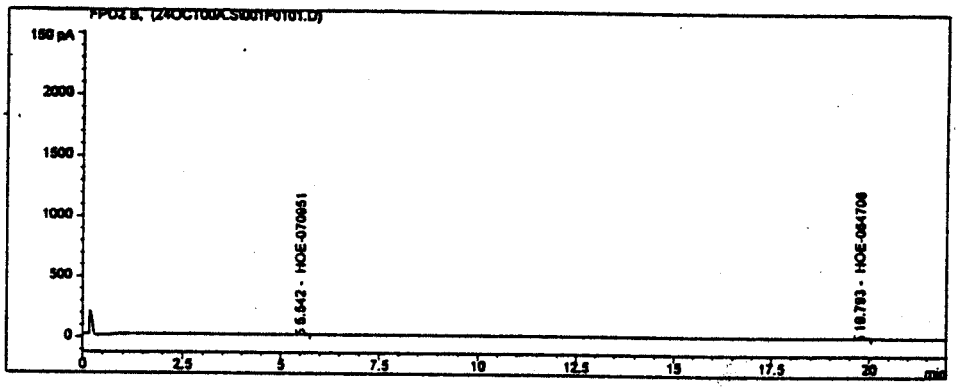
Reagent Blank, Set 1F

Analyzed in Set 1F, Curve 1, Injection No. 8

Figure 1. Typical Chromatogram of a Reagent Blank Sample from the Analysis for Residues of Glufosinate Ammonium in Corn Forage, Grain and Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\001F0101.D                0.015 µg/mL
    0.015 µg/mL HOE-064706 & 070951
    G8G915Sep00-0.015MIX
    #000290          Set #  1F

================================================================================
Injection Date   : 10/24/00 6:14:33 PM              Seq. Line :   1
Sample Name      : 0.015 µg/mL                          Vial :   1
Acq. Operator    : MYL                                   Inj :   1
                                                   Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 10/24/00 2:23:24 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 10/26/00 11:28:43 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, October 26, 2000 11:25:27 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type      Area      Amt/Area     Amount    Grp   Name
 [min]          150 pA*s                 [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.542 BB       352.42188 4.19868e-5 1.47971e-2     HOE-070951
 19.793 BB       270.22275 6.27523e-5 1.69571e-2     HOE-064706

Totals :                                3.17542e-2

 Results obtained with enhanced integrator!
================================================================================

6890 EC-FPD 10/26/00 11:30:34 AM MYL                        Page 1 of 2
```

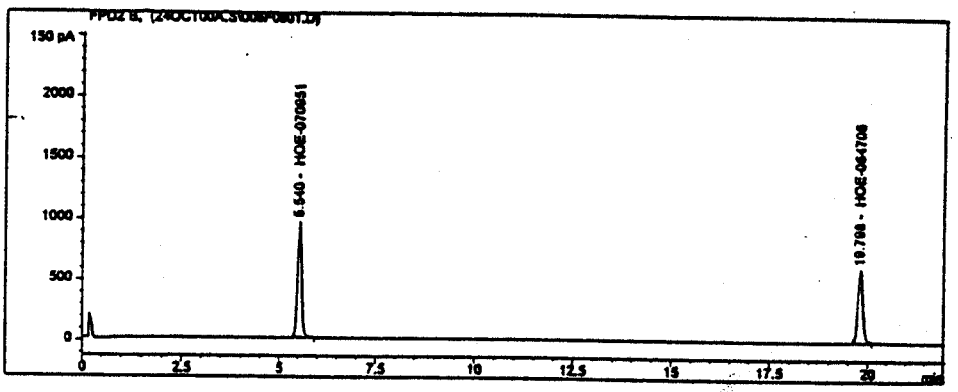
0.015 µg/mL Each HOE-070951 and HOE-064706 Derivative Calibration Standards

Each Equivalent to 0.03 µg/g Glufosinate as the Free Acid (HOE-035956)
Analyzed in Set 1F, Curve 1, Injection No. 1

Figure 2a. Typical Chromatogram of a Low Level Mixed Calibration Standard from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\006F0601.D                0.30 µg/mL
    0.30 µg/mL HOE-064706 & 070951
    G8G915Sep00-0.30MIX
    #000290          Set #  1F

================================================================================
Injection Date  : 10/24/00 8:46:10 PM              Seq. Line :   6
Sample Name     : 0.30 µg/mL                          Vial :   6
Acq. Operator   : MYL                                  Inj :   1
                                                  Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 10/24/00 2:23:24 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 10/26/00 11:28:43 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, October 26, 2000 11:25:27 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]         150 pA*s                [µg/mL]
-------|------|----------|----------|---------|--|-----------------
  5.540 BB     7153.68799 4.19996e-5 3.00452e-1    HOE-070951
 19.798 BBA    5626.27832 5.41109e-5 3.04443e-1    HOE-064706

Totals :                              6.04894e-1

 Results obtained with enhanced integrator!
================================================================================


6890 EC-FPD 10/26/00 11:31:26 AM MYL                        Page 1 of 2
```

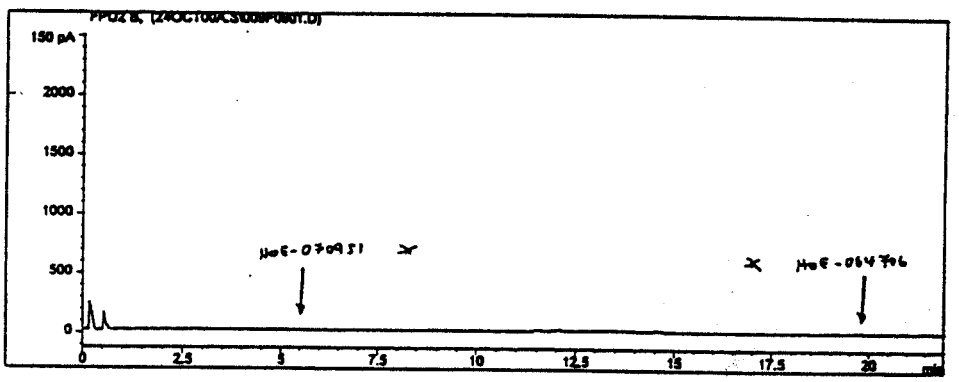
0.30 µg/mL Each HOE-070951 and HOE-064706 Derivative Calibration Standards

Each Equivalent to 0.60 µg/g Glufosinate as the Free Acid (HOE-035956)
Analyzed in Set 1F, Curve 1, Injection No. 6

Figure 2b. Typical Chromatogram of a High Level Mixed Calibration Standard from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\009F0901.D                UTC 32887101
  corn forage control   32887101
  12.5 g sample weight; 500 mL extraction volume; 100 mL
  aliquot; 5.0 mL final volume in methyl acetate
  #000290          Set #  1F

  Injection Date   : 10/24/00 10:17:21 PM          Seq. Line :   9
  Sample Name      : UTC 32887101                      Vial :   9
  Acq. Operator    : MYL                               Inj :   1
                                                   Inj Volume : 5 µl
  Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
  Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
  Last changed     : 10/24/00 2:23:24 PM by MYL
  Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
  Last changed     : 10/26/00 11:28:43 AM by MYL
  GC Determination of Glufosinate-Ammonium and its Metabolites
```




```
                        External Standard Report

  Sorted By             :      Signal
  Calib. Data Modified  :      Thursday, October 26, 2000 11:25:27 AM
  Multiplier            :      1.0000
  Dilution              :      1.0000

  Signal 1: FPD2 B,

  RetTime  Type     Area      Amt/Area    Amount    Grp   Name
   [min]          150 pA*s               [µg/mL]
  -------|------|----------|----------|----------|--|-----------------
    5.540           -           -           -          HOE-070951
   19.798           -           -           -          HOE-064706

  Totals :                                  0.00000

   Results obtained with enhanced integrator!
  1 Warnings or Errors :

  Warning : Calibrated compound(s) not found

6890 EC-FPD 10/26/00 11:32:00 AM MYL                          Page 1 of 2
```

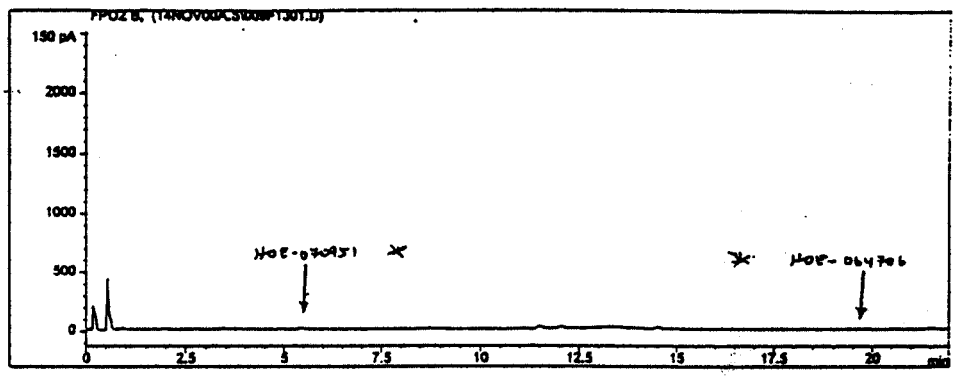
* The retention time and compound name were added on 26 Oct 00. MYL 07 Jun 01

Control Corn Forage Sample 32887101
Analyzed in Set 1F, Curve 1, Injection No. 9

Figure 3a. Typical Chromatograms of Control Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\14NOV00A.S\009F1301.D          UTC 32892801
    corn forage control   32892801
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  2F

    ================================================================
    Injection Date  : 11/14/00 11:53:03 PM          Seq. Line :  13
    Sample Name     : UTC 32892801                       Vial :   9
    Acq. Operator   : MYL                                Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\14NOV00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/31/00 10:06:58 AM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 11/16/00 3:03:31 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      11/16/00 2:30:16 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.530           -           -          -          HOE-070951
     19.793           -           -          -          HOE-064706

    Totals :                                  0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

    6890 EC-FPD 11/16/00 3:07:56 PM MYL                    Page 1 of 2
```

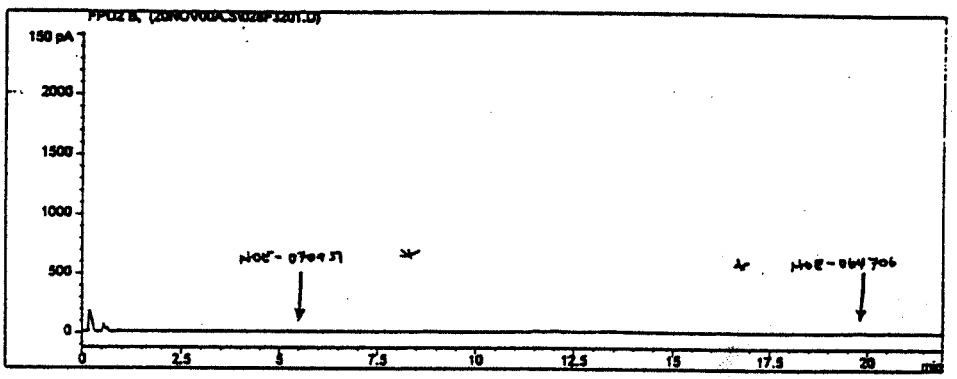
* The retention time areas and compound names were added on 16 Nov 00.
MYL 07 Jun 01

Control Corn Forage Sample 32892801
Analyzed in Set 2F, Curve 1, Injection No. 9

Figure 3b. Typical Chromatograms of Control Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\20NOV00A.S\028F3201.D          UTC 33063901
    corn forage control   33063901
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  3F

    ================================================================
    Injection Date  : 11/21/00 10:10:53 AM          Seq. Line :  32
    Sample Name     : UTC 33063901                       Vial :  28
    Acq. Operator   : MYL                                Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\20NOV00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 11/20/00 5:57:08 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 11/21/00 5:34:35 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      11/21/00 5:34:26 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|---------|----------|----------|--|-----------------
      5.524           -           -          -          HOE-070951
     19.794           -           -          -          HOE-064706

    Totals :                                  0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

    6890 EC-FPD 11/21/00 5:37:40 PM MYL                    Page 1 of 2
```

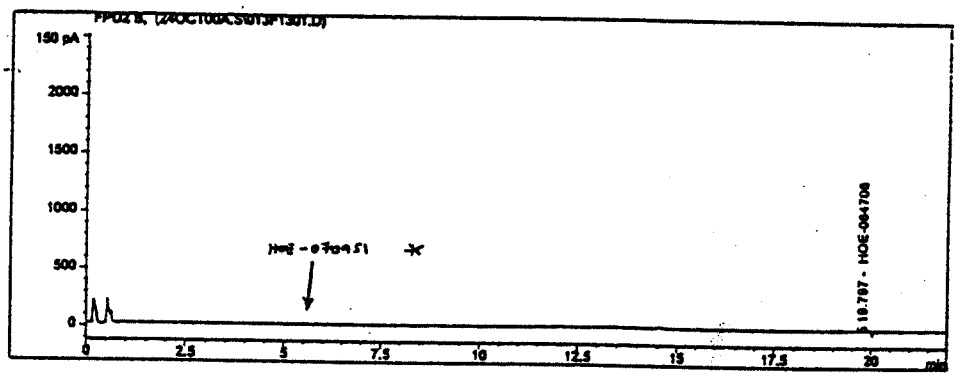
the retention time areas and compound names were added on 21 Nov 00. MYL 07 Jun 01

Control Corn Forage Sample 33063901
Analyzed in Set 3F, Curve 2, Injection No. 28

Figure 3c. Typical Chromatograms of Control Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\013F1301.D                0.050 µg/g P
    corn forage control + 0.050 µg/g HOE-039866
    control 32887101
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1F

    ================================================================
    Injection Date  : 10/25/00 12:18:41 AM          Seq. Line :  13
    Sample Name     : 0.050 µg/g P                      Vial :  13
    Acq. Operator   : MYL                               Inj :   1
                                                    Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/24/00 2:23:24 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/26/00 11:28:43 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, October 26, 2000 11:25:27 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type      Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.540             -           -          -          HOE-070951
     19.797 BP      398.91422 5.98239e-5 2.38646e-2        HOE-064706

    Totals :                                 2.38646e-2

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

6890 EC-FPD 10/26/00 11:32:43 AM MYL                          Page 1 of 2
```

* the retention time and compound name were added on 26 Oct 00. MYL 07 Jun 01

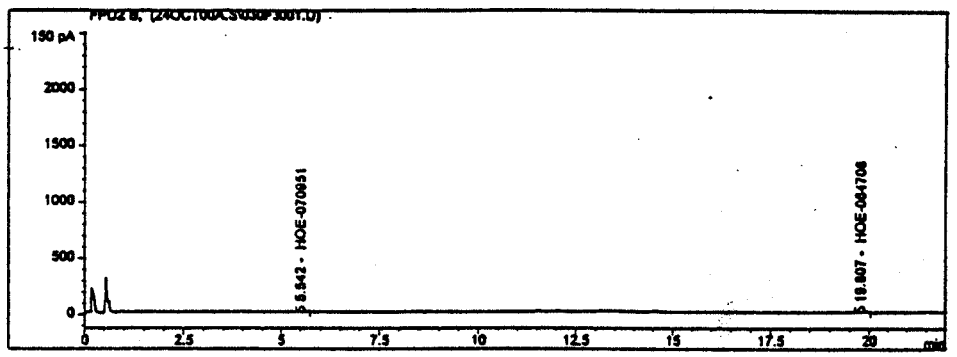
Control Corn Forage Sample 32887101
Fortified at 0.05 µg/g HOE-039866 (P)

0.0485 µg/g Equivalent Glufosinate Found as Free Acid, 97% Recovery
Analyzed in Set 1F, Curve 1, Injection No. 13

Figure 4a. Typical Chromatograms of Control Corn Forage Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\030F3001.D          0.050 µg/g M1M2
    corn forage control + 0.050 µg/g HOE-099730 + HOE-06151
    7
    control 32887101
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1F

    ================================================================
    Injection Date  : 10/25/00 8:54:08 AM          Seq. Line :  30
    Sample Name     : 0.050 µg/g M1M2                   Vial :  30
    Acq. Operator   : MYL                               Inj :   1
                                                  Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/24/00 2:23:24 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/26/00 11:59:21 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, October 26, 2000 11:58:28 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|---------|---------|---------|--|-----------------
      5.542 BB      396.63062 3.94338e-5 1.56406e-2    HOE-070951
     19.807 BB      466.91818 5.66671e-5 2.64589e-2    HOE-064706

    Totals :                              4.20995e-2

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 10/26/00 12:04:04 PM MYL                        Page 1 of 2
```

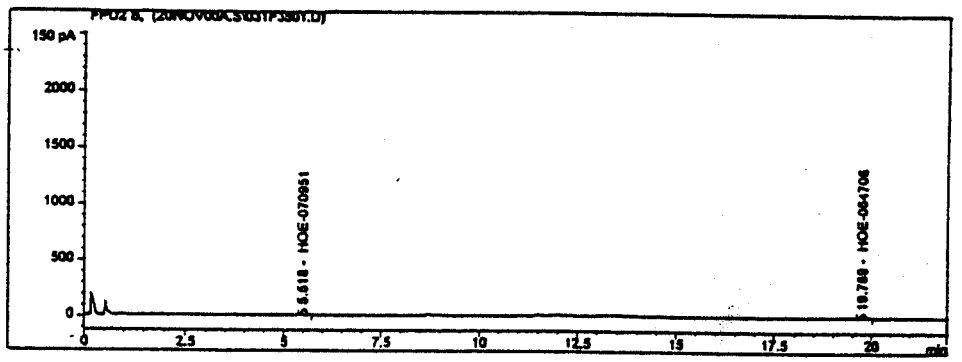
Control Corn Forage Sample 32887101
Fortified at 0.05 µg/g HOE-099730 (M1) + 0.05 µg/g HOE-061517 (M2)

HOE-099730 Fort. (M1) - 0.0531 µg/g Equivalent Glufosinate Found as Free Acid, 106% Recovery
HOE-061517 Fort. (M2) - 0.0308 µg/g Equivalent Glufosinate Found as Free Acid, 62% Recovery
Analyzed in Set 1F, Curve 2, Injection No. 30

Figure 4b. Typical Chromatograms of Control Corn Forage Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\20NOV00A.S\031F3501.D          0.050µg/gMIM2DUP
corn forage control + 0.050 µg/g HOE-099730 + HOE-06151
7
control 33063901                                  DUP
12.5 g sample weight; 500 mL extraction volume; 100 mL
aliquot; 5.0 mL final volume in methyl acetate
#000290          Set #  3F

================================================================================
Injection Date  : 11/21/00 11:41:54 AM          Seq. Line :  35
Sample Name     : 0.050µg/gMIM2DUP                   Vial :  31
Acq. Operator   : MYL                                 Inj :   1
                                                Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\20NOV00A.S
Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 11/20/00 5:57:08 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 11/21/00 5:34:35 PM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                          External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      11/21/00 5:34:26 PM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.518 BB      415.93152 4.87269e-5 2.02671e-2     HOE-070951
 19.789 BB      369.94287 6.51992e-5 2.41200e-2     HOE-064706

Totals :                                4.43870e-2

 Results obtained with enhanced integrator!
================================================================================

6890 EC-FPD 11/21/00 5:38:13 PM MYL                          Page 1 of 2
```

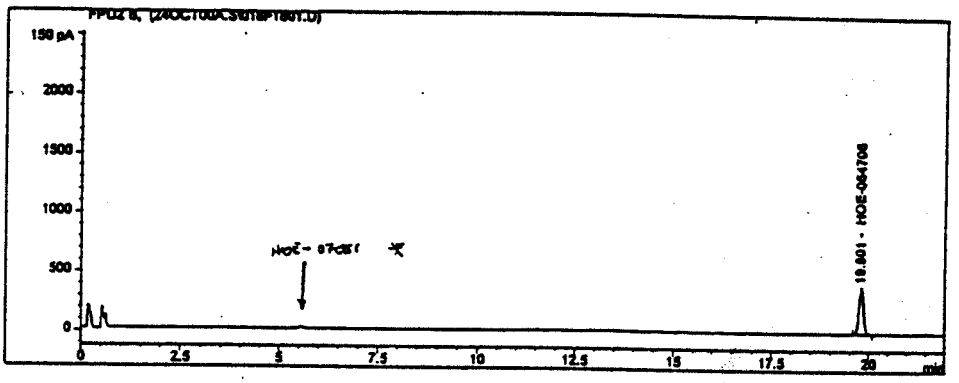
Control Corn Forage Sample 33063901
Fortified at 0.05 µg/g HOE-099730 (M1) + 0.05 µg/g HOE-061517 (M2)

HOE–099730 Fort. (M1) - 0.0493 µg/g Equivalent Glufosinate Found as Free Acid, 99% Recovery
HOE-061517 Fort. (M2) - 0.0414 µg/g Equivalent Glufosinate Found as Free Acid, 83% Recovery
Analyzed in Set 3F, Curve 2, Injection No. 31

Figure 4c. Typical Chromatograms of Control Corn Forage Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\018F1801.D  Sample Name: 32887102
   corn forage  TRT   32887102
   12.5 g sample weight; 500 mL extraction volume; 100 mL
   aliquot; 5.0 mL final volume in methyl acetate
   #000290           Set #  1F

================================================================================
Injection Date   : 10/25/00 2:50:09 AM              Seq. Line :  18
Sample Name      : 32887102                            Vial :  18
Acq. Operator    : MYL                                  Inj :   1
                                                   Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 10/24/00 2:23:24 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 10/26/00 11:28:43 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, October 26, 2000 11:25:27 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]         150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.540          -           -           -          HOE-070951
 19.801 BB     3418.53394 5.43924e-5 1.85942e-1     HOE-064706

Totals :                                1.85942e-1

 Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

6890 EC-FPD 10/26/00 11:33:36 AM MYL                        Page 1 of 2
```

* The retention time and compound name were added on 26 Oct 00. MYL 07 Jun 01

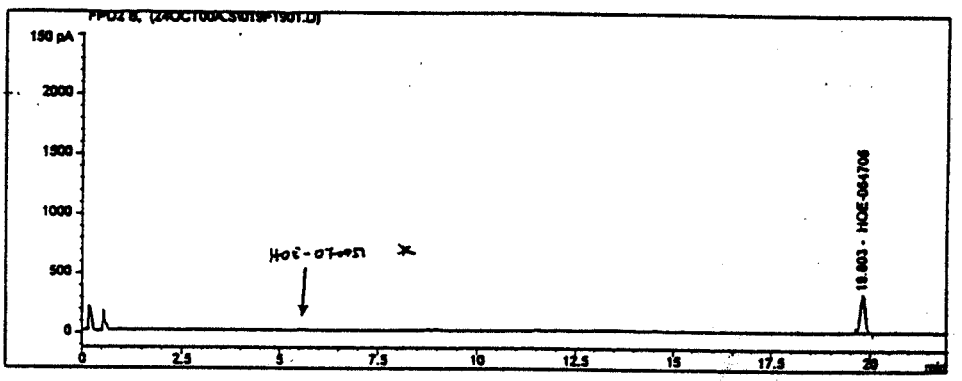
Treated Corn Forage Sample 32887102
Analyzed in Set 1F, Curve 1, Injection No. 18

Residues of P + M1 Equivalent to 0.3720 µg/g Glufosinate as Uncorrected Free Acid
Residues Metabolite M2 Found Equivalent to Non-Detection
Total Residues of Glufosinate Reported as 0.41 µg/g Free Acid, Corrected for Recovery

Figure 5a. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\019F1901.D  Sample Name: 32887103
corn forage  TRT   32887103
12.5 g sample weight; 500 mL extraction volume; 100 mL
aliquot; 5.0 mL final volume in methyl acetate
#000290           Set #  1F

================================================================================
Injection Date   : 10/25/00 3:20:31 AM              Seq. Line :  19
Sample Name      : 32887103                            Vial :  19
Acq. Operator    : MYL                                  Inj :   1
                                                   Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 10/24/00 2:23:24 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 10/26/00 11:28:43 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```

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================================================================================
                          External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, October 26, 2000 11:25:27 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.540              -          -          -          HOE-070951
 19.803 BP     2730.40259 5.45733e-5 1.49007e-1        HOE-064706

Totals :                                1.49007e-1

 Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found
```

* The retention time and compound name were added on 26 Oct 00. MYL 07 Jun 01

```
6890 EC-FPD 10/26/00 11:33:47 AM MYL                    Page 1 of 2
```

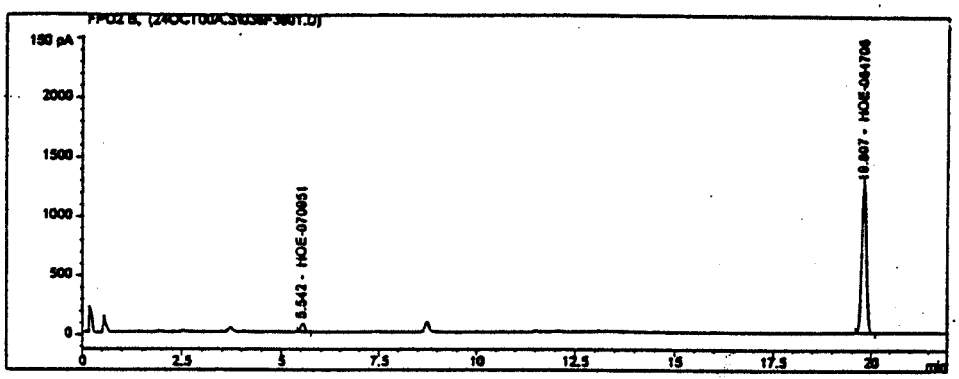
Treated Corn Forage Sample 32887103
Analyzed in Set 1F, Curve 1, Injection No. 19

Residues of P + M1 Equivalent to 0.2983 µg/g Glufosinate as Uncorrected Free Acid
Residues Metabolite M2 Found Equivalent to Non-Detection
Total Residues of Glufosinate Reported as 0.33 µg/g Free Acid, Corrected for Recovery

Figure 5b. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\036F3601.D  Sample Name: 33060402
    corn forage TRT   33060402
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  1F

    ================================================================
    Injection Date  : 10/25/00 11:55:54 AM          Seq. Line :  36
    Sample Name     : 33060402                          Vial :  36
    Acq. Operator   : MYL                               Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/24/00 2:23:24 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/26/00 11:59:21 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, October 26, 2000 11:58:28 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type      Area     Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.542 BB       544.29163 4.04429e-5 2.20127e-2    HOE-070951
     19.807 BBA     1.13041e4  5.54528e-5 6.26845e-1    HOE-064706

    Totals :                              6.48858e-1

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 10/26/00 12:05:08 PM MYL                        Page 1 of 2
```

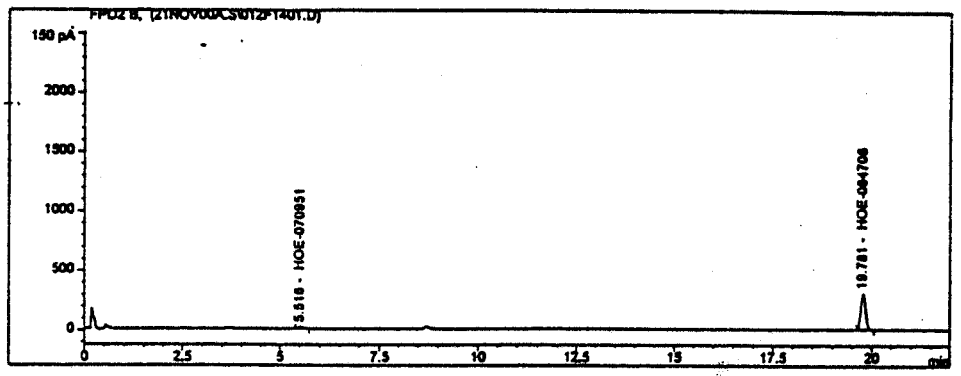
Treated Corn Forage Sample 33060402 Standard Volume (Method Factor 2)
Analyzed in Set 1F, Curve 2, Injection No. 36

Residues of P + M1 Above Standard Calibration Range (For P + M1 values see Figure 5d.)
Residues of M2 Found Equivalent to 0.0435 µg/g Glufosinate as Uncorrected Free Acid
For Total Residues of Glufosinate as Recovery Corrected Free Acid see Figure 5d.)

Figure 5c. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\21NOV00A.S\012F1401.D  Sample Name: 33060402
    corn forage TRT   33060402          4X Dilution
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1F (re-injection)

================================================================================
Injection Date  : 11/22/00 12:51:58 AM          Seq. Line :  14
Sample Name     : 33060402                         Vial :  12
Acq. Operator   : MYL                               Inj :   1
                                                Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\21NOV00A.S
Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 11/21/00 5:44:32 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 11/24/00 8:14:32 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      11/24/00 8:14:22 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.516 PB      137.57076 5.92684e-5 8.15361e-3    HOE-070951
 19.781 BP     2693.42456 5.88751e-5 1.58576e-1    HOE-064706

Totals :                              1.66729e-1

 Results obtained with enhanced integrator!
================================================================================
```

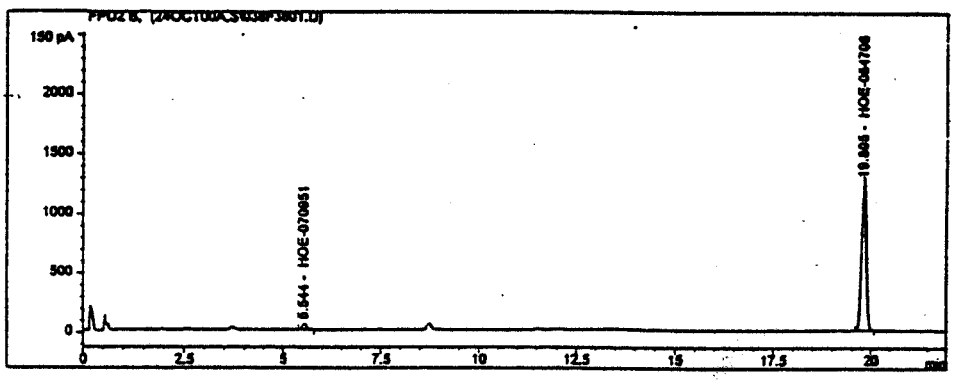

Treated Corn Forage Sample 33060402 Diluted 4x (Method Factor 8)
Analyzed in Set 1F (reinjection), Injection No. 12

Residues of Parent + M1 Equivalent to 1.2704 µg/g Glufosinate as Uncorrected Free Acid
Residues of M2 Not Determined at this Dilution (For M2 values see Figure 5c.)
Total Residues of Glufosinate Reported as 1.29 µg/g Free Acid, Corrected for Recovery

Figure 5d. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\24OCT00A.S\038F3801.D  Sample Name: 33060403
    corn forage TRT   33060403
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  1F

    ================================================================
    Injection Date  : 10/25/00 12:56:29 PM          Seq. Line :  38
    Sample Name     : 33060403                          Vial :  38
    Acq. Operator   : MYL                               Inj :   1
                                                    Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\24OCT00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/24/00 2:23:24 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/26/00 11:59:21 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, October 26, 2000 11:58:28 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.544 BP       391.76801 3.93876e-5 1.54308e-2    HOE-070951
     19.805 BB      1.10630e4  5.54540e-5 6.13488e-1    HOE-064706

    Totals :                              6.28919e-1

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 10/26/00 12:05:28 PM MYL                    Page 1 of 2
```

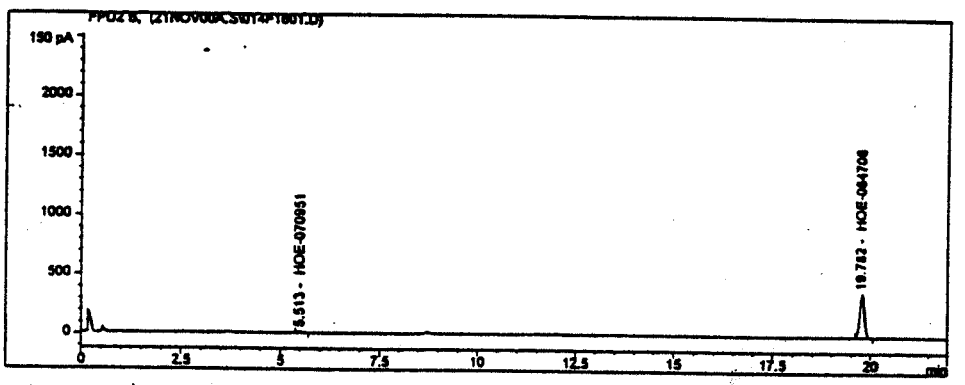
Treated Corn Forage Sample 33060403 Standard Volume (Method Factor 2)
Analyzed in Set 1F, Curve 2, Injection No. 38

Residues of P + M1 Above Standard Calibration Range (For P + M1 values see Figure 5f.)
Residues of M2 Found Equivalent to 0.0304 µg/g Glufosinate as Uncorrected Free Acid
For Total Residues of Glufosinate as Recovery Corrected Free Acid see Figure 5f.

Figure 5e. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\21NOV00A.S\014F1601.D  Sample Name: 33060403
    corn forage TRT   33060403          4X Dilution
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1F (re-injection)

================================================================================
Injection Date   : 11/22/00 1:52:46 AM              Seq. Line :  16
Sample Name      : 33060403                            Vial :  14
Acq. Operator    : MYL                                  Inj :   1
                                                   Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\21NOV00A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 11/21/00 5:44:32 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 11/24/00 8:14:32 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      11/24/00 8:14:22 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]         150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|------------------
  5.513 BB      118.40089 6.18849e-5 7.32723e-3     HOE-070951
 19.782 BP     3240.98315 5.85475e-5 1.89752e-1     HOE-064706

Totals :                              1.97079e-1

 Results obtained with enhanced integrator!
================================================================================

6890 EC-FPD 11/24/00 8:17:39 AM MYL                        Page 1 of 2
```

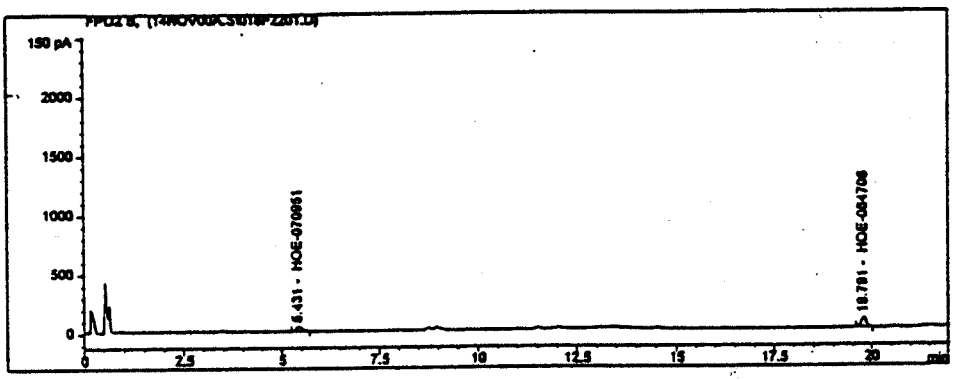
Treated Corn Forage Sample 33060403 Diluted 4x (Method Factor 8)
Analyzed in Set 1F (reinjection), Injection No. 14

Residues of Parent + M1 Equivalent to 1.5187 µg/g Glufosinate as Uncorrected Free Acid
Residues of M2 Not Determined at this Dilution (For M2 values see Figure 5e)
Total Residues of Glufosinate Reported as 1.51 µg/g Free Acid, Corrected for Recovery

Figure 5f. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\14NOV00A.S\018F2201.D  Sample Name: 32892802
    corn forage  TRT   32892802
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  2F

    ================================================================
    Injection Date  : 11/15/00 4:26:14 AM          Seq. Line :  22
    Sample Name     : 32892802                          Vial :  18
    Acq. Operator   : MYL                               Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\14NOV00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/31/00 10:06:58 AM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 11/16/00 3:03:31 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      11/16/00 2:30:16 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
      [min]          150 pA*s               [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.431 BP      427.91351 4.76087e-5 2.03724e-2    HOE-070951
     19.791 BB      743.94611 6.58887e-5 4.90176e-2    HOE-064706

    Totals :                              6.93900e-2

     Results obtained with enhanced integrator!
    ================================================================

    6890 EC-FPD 11/16/00 3:41:05 PM MYL                  Page 1 of 2
```

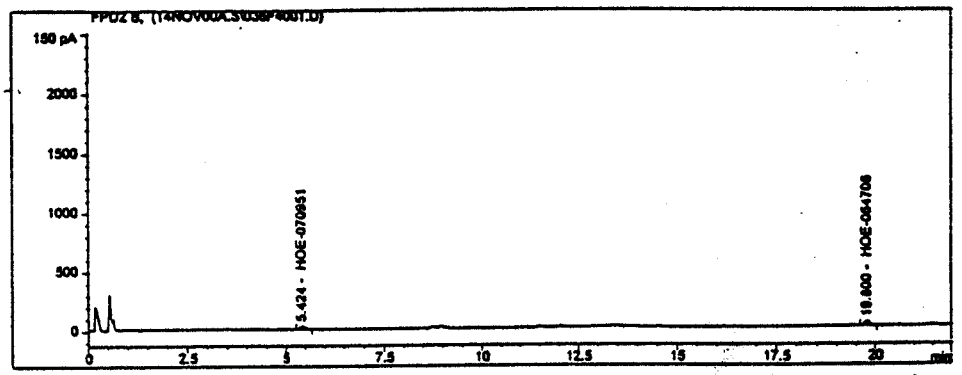
Treated Corn Forage Sample 32892802
Analyzed in Set 2F, Curve 1, Injection No. 18

Residues of Parent + M1 Equivalent to 0.0997 µg/g Glufosinate as Uncorrected Free Acid
Residues Metabolite M2 Found Equivalent to 0.0413 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as 0.15 µg/g Free Acid, Corrected for Recovery

Figure 5g. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\14NOV00A.S\036F4001.D  Sample Name: 32904502
    corn forage TRT   32904502
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  2F

    ================================================================================
    Injection Date  : 11/15/00 1:32:21 PM          Seq. Line :  40
    Sample Name     : 32904502                          Vial :  36
    Acq. Operator   : MYL                               Inj :   1
                                                  Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\14NOV00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 10/31/00 10:06:58 AM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 11/16/00 3:58:46 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```




```
    ================================================================================
                              External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      11/16/00 3:58:28 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
      [min]         150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.424 BB      257.52646 4.36148e-5 1.12320e-2     HOE-070951
     19.800 BP      346.79144 5.82054e-5 2.01851e-2     HOE-064706

    Totals :                             3.14171e-2

     Results obtained with enhanced integrator!
    ================================================================================


    6890 EC-FPD 11/16/00 4:04:04 PM MYL                          Page 1 of 2
```

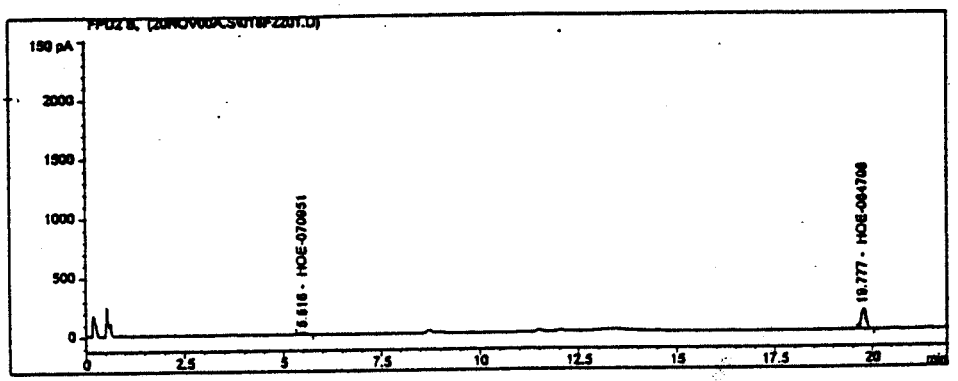
Treated Corn Forage Sample 32904502
Analyzed in Set 2F, Curve 2, Injection No. 36

Residues of Parent + M1 Equivalent to 0.0404 µg/g Glufosinate as Uncorrected Free Acid
Residues Metabolite M2 Found Equivalent to 0.0224 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as 0.07 µg/g Free Acid, Corrected for Recovery

Figure 5h. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\20NOV00A.S\018F2201.D  Sample Name: 33063902
    corn forage  TRT   33063902
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  3F

    ================================================================
    Injection Date  : 11/21/00 5:07:17 AM          Seq. Line :  22
    Sample Name     : 33063902                         Vial :  18
    Acq. Operator   : MYL                               Inj :   1
                                                  Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\20NOV00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 11/20/00 5:57:08 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 11/24/00 8:37:39 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Friday, November 24, 2000 8:35:20 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.516 BP      208.62703 4.73137e-5 9.87091e-3     HOE-070951
     19.777 BP     1535.17053 5.99393e-5 9.20170e-2     HOE-064706

    Totals :                             1.01888e-1

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 11/24/00 8:42:25 AM MYL                          Page 1 of 2
```

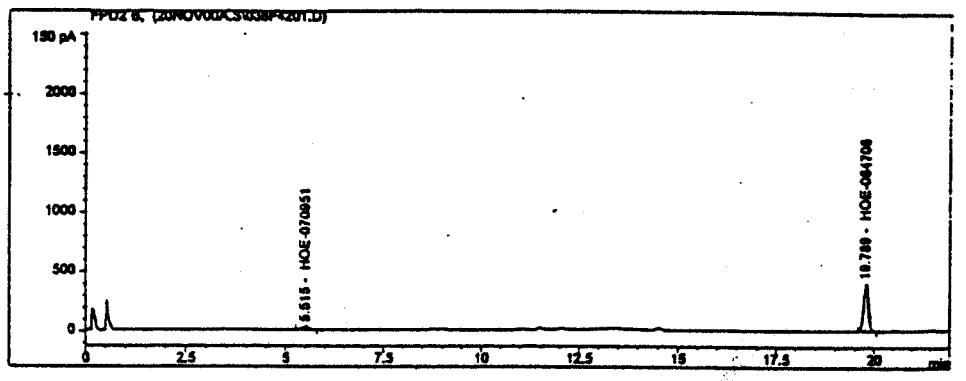
Treated Corn Forage Sample 33063902
Analyzed in Set 3F, Curve 1, Injection No. 18

Residues of Parent + M1 Equivalent to 0.1848 µg/g Glufosinate as Uncorrected Free Acid
Residues Metabolite M2 Found Equivalent to 0.0199 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as 0.23 µg/g Free Acid, Corrected for Recovery

Figure 5i. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\20NOV00A.S\038F4201.D  Sample Name: 33176703
    corn forage TRT   33176703
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  3F

    ================================================================
    Injection Date   : 11/21/00 3:14:31 PM          Seq. Line :  42
    Sample Name      : 33176703                        Vial :  38
    Acq. Operator    : MYL                              Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\20NOV00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 11/20/00 5:57:08 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 11/21/00 5:34:35 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      11/21/00 5:34:26 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type      Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.515 BP       325.06122 5.03979e-5 1.63824e-2    HOE-070951
     19.789 BB      3463.20679 5.72062e-5 1.98117e-1    HOE-064706

    Totals :                              2.14499e-1

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 11/21/00 5:39:28 PM MYL                        Page 1 of 2
```

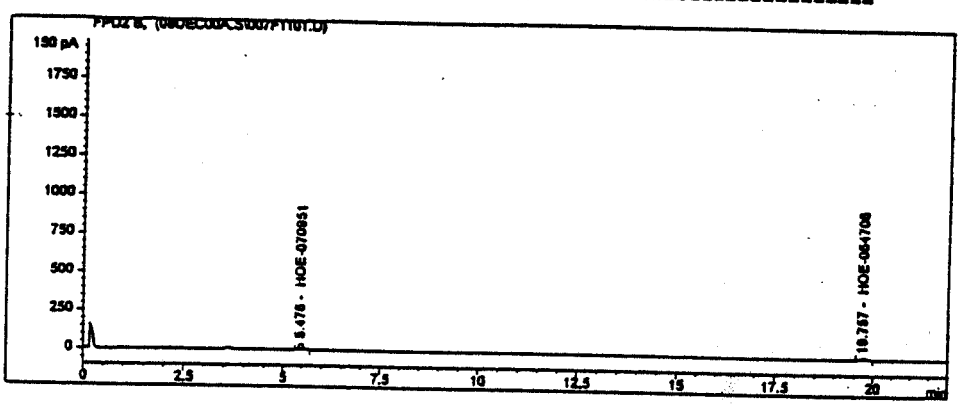
Treated Corn Forage Sample 33176703
Analyzed in Set 3F, Curve 2, Injection No. 38

Residues of Parent + M1 Equivalent to 0.3963 µg/g Glufosinate as Uncorrected Free Acid
Residues Metabolite M2 Found Equivalent to 0.0336 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as 0.48 µg/g Free Acid, Corrected for Recovery

Figure 5j. Typical Chromatograms of Treated Corn Forage from the Analysis for Residues of Glufosinate Ammonium in Corn Forage

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\08DEC00A.S\007F1101.D                0.015 µg/mL
    0.015 µg/mL HOE-064706 & 070951
    G8G913Nov00-0.015MIX
    #000290          Set #  1G

================================================================================
Injection Date   : 12/8/00 9:54:42 PM                    Seq. Line :   11
Sample Name      : 0.015 µg/mL                               Vial :    7
Acq. Operator    : MYL                                        Inj :    1
                                                        Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\08DEC00A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 12/8/00 4:47:08 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 12/11/00 4:47:53 PM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```

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================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Monday, December 11, 2000 4:47:04 PM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type      Area      Amt/Area    Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.476 PB       346.68015 4.43531e-5 1.53764e-2     HOE-070951
 19.757 BBA      200.24312 7.94717e-5 1.59137e-2     HOE-064706

Totals :                                3.12900e-2

 Results obtained with enhanced integrator!
================================================================================

6890 EC-FPD 12/11/00 4:58:20 PM MYL                          Page 1 of 2
```

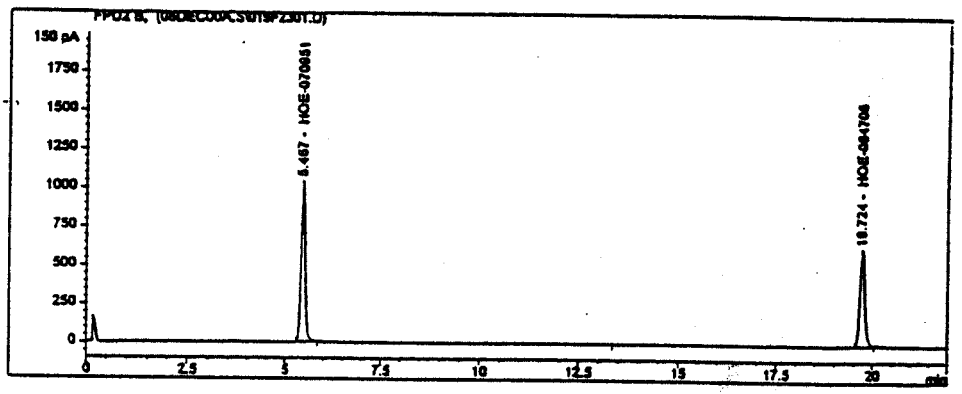
0.015 µg/mL Each HOE-070951 and HOE-064706 Derivative Calibration Standards

Each Equivalent to 0.03 µg/g Glufosinate as the Free Acid (HOE-035956)
Analyzed in Set 1G, Curve 1, Injection No. 7

Figure 6a. Typical Chromatogram of a Low Level Mixed Calibration Standard from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

Data File C:\HPCHEM\2\DATA\GLUFOSIN\08DEC00A.S\019F2301.D 0.30 µg/mL
0.30 µg/mL HOE-064706 & 070951
G8G913Nov00-0.30MIX
#000290 Set # 1G

Injection Date : 12/9/00 3:58:45 AM Seq. Line : 23
Sample Name : 0.30 µg/mL Vial : 19
Acq. Operator : MYL Inj : 1
Inj Volume : 5 µl
Sequence File : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\08DEC00A.S
Acq. Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed : 12/8/00 4:47:08 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed : 12/11/00 4:47:53 PM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites



External Standard Report

Sorted By : Signal
Calib. Data Modified : Monday, December 11, 2000 4:47:04 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FPD2 B,

RetTime [min]	Type	Area 150 pA*s	Amt/Area	Amount [µg/mL]	Grp	Name
5.467	BB	7790.77441	3.93834e-5	3.06827e-1		HOE-070951
19.724	BBA	6049.28271	5.26313e-5	3.18382e-1		HOE-064706
Totals :				6.25209e-1		

Results obtained with enhanced integrator!

6890 EC-FPD 12/11/00 5:00:30 PM MYL Page 1 of 2

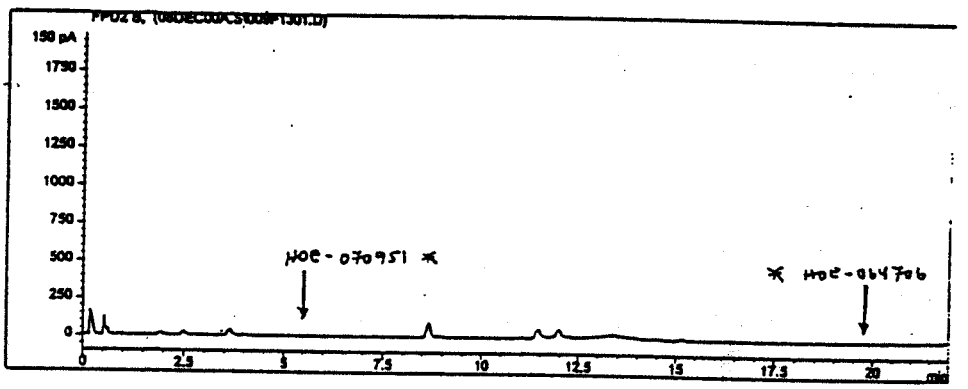
0.30 µg/mL Each HOE-070951 and HOE-064706 Derivative Calibration Standards

Each Equivalent to 0.60 µg/g Glufosinate as the Free Acid (HOE-035956)
Analyzed in Set 1G, Curve 1, Injection No. 19

Figure 6b. Typical Chromatogram of a High Level Mixed Calibration Standard from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

Data File C:\HPCHEM\2\DATA\GLUFOSIN\08DEC00A.S\009F1301.D UTC 32889801
corn forage control 32889801
12.5 g sample weight; 500 mL extraction volume; 100 mL aliquot; 5.0 mL final volume in methyl acetate
#000290 Set # 1G
MYL 05 Jun 01

Injection Date : 12/8/00 10:55:23 PM Seq. Line : 13
Sample Name : UTC 32889801 Vial : 9
Acq. Operator : MYL Inj : 1
Inj Volume : 5 µl
Sequence File : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\08DEC00A.S
Acq. Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed : 12/8/00 4:47:08 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed : 12/11/00 4:47:53 PM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites



External Standard Report

Sorted By : Signal
Calib. Data Modified : Monday, December 11, 2000 4:47:04 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FPD2 B,

RetTime [min]	Type	Area 150 pA*s	Amt/Area	Amount [µg/mL]	Grp	Name
5.467		-	-	-		HOE-070951
19.724		-	-	-		HOE-064706
Totals :				0.00000		

Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

* The retention time areas and compound name were added on 11 Dec 00. MYL 04 Jun 01

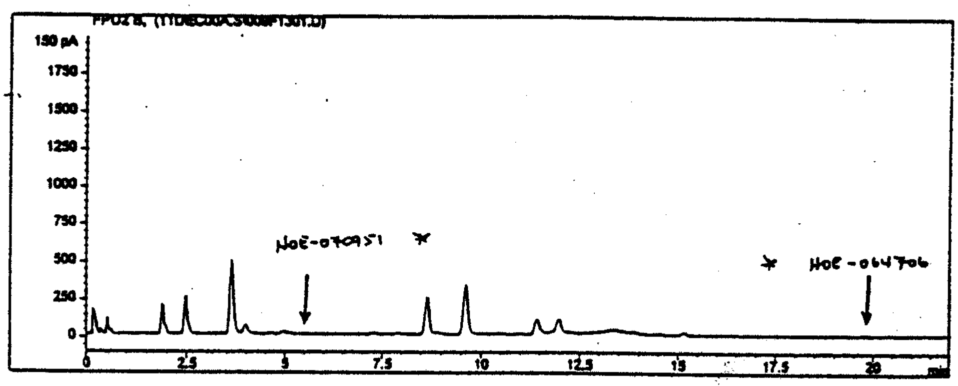
6890 EC-FPD 12/11/00 4:58:42 PM MYL Page 1 of 2

Control Corn Grain Sample 32889801
Analyzed in Set 1G, Curve 1, Injection No. 9

Figure 7a. Typical Chromatograms of Control Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

Data File C:\HPCHEM\2\DATA\GLUFOSIN\11DEC00A.S\009F1301.D UTC 33061201
corn ~~forage~~ control 33061201
12.5 g sample weight; 500 mL extraction volume; 100 mL aliquot; 5.0 mL final volume in methyl acetate
#000290 Set # 2G

```
Injection Date  : 12/12/00 2:01:49 AM          Seq. Line :  13
Sample Name     : UTC 33061201                      Vial :   9
Acq. Operator   : MYL                                Inj :   1
                                              Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\11DEC00A.S
Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/11/00 6:25:18 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/14/00 8:18:21 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
```



External Standard Report

```
Sorted By             :  Signal
Calib. Data Modified  :  Thursday, December 14, 2000 8:11:17 AM
Multiplier            :  1.0000
Dilution              :  1.0000
```

Signal 1: FPD2 B,

RetTime [min]	Type	Area 150 pA*s	Amt/Area	Amount [µg/mL]	Grp	Name
5.472		-	-	-		HOE-070951
19.745		-	-	-		HOE-064706

Totals : 0.00000

* the retention time arrow and compound name were added on 14 Dec 00. MYL

Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

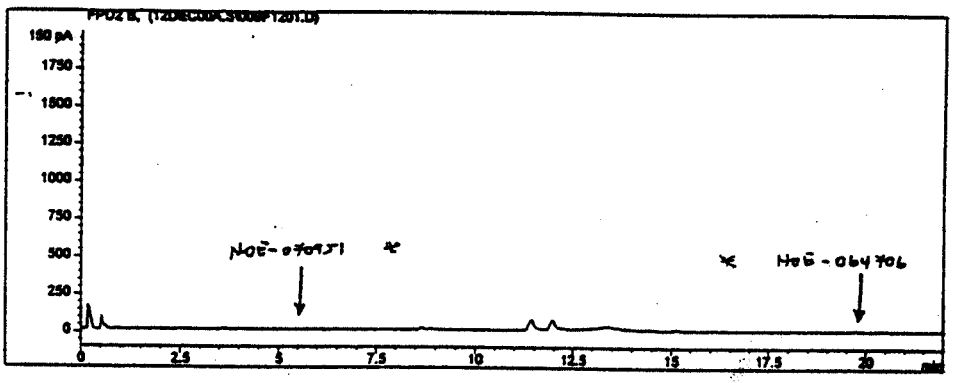
6890 EC-FPD 12/14/00 8:22:34 AM MYL Page 1 of 2

Control Corn Grain Sample 33061201
Analyzed in Set 2G, Curve 1, Injection No. 9

Figure 7b. Typical Chromatograms of Control Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\12DEC00A.S\009F1201.D          UTC 33064701
  corn grain control   33064701
  12.5 g sample weight; 500 mL extraction volume; 100 mL
  aliquot; 5.0 mL final volume in methyl acetate
  #000290          Set #  3G

================================================================================
Injection Date  : 12/13/00 1:00:38 AM              Seq. Line :  12
Sample Name     : UTC 33064701                          Vial :   9
Acq. Operator   : MYL                                    Inj :   1
                                                  Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\12DEC00A.S
Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/11/00 6:25:18 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/14/00 8:59:50 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, December 14, 2000 8:51:39 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.465          -           -           -          HOE-070951
 19.723          -           -           -          HOE-064706

Totals :                                  0.00000

 Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

6890 EC-FPD 12/14/00 9:02:36 AM MYL                        Page 1 of 2
```

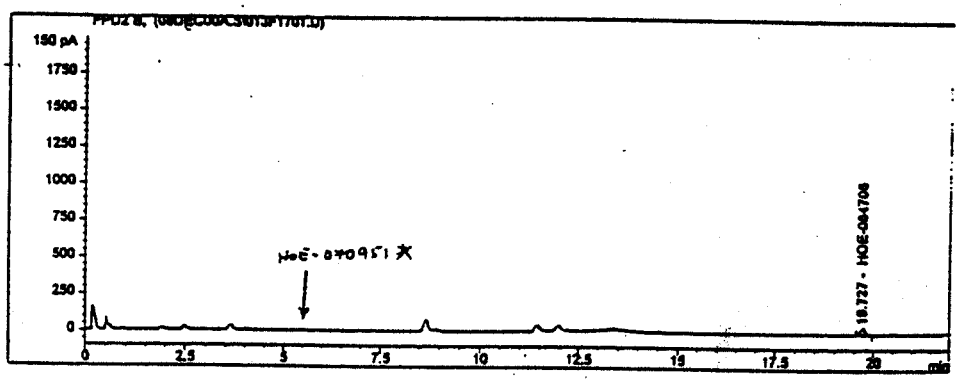
* the retention time arrow and compound name were added on 14 Dec 00. MYL 04 Jun 01

Control Corn Grain Sample 33064701
Analyzed in Set 3G, Curve 1, Injection No. 9

Figure 7c. Typical Chromatograms of Control Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

Data File C:\HPCHEM\2\DATA\GLUFOSIN\08DEC00A.S\013F1701.D 0.050 µg/g P
corn ~~foliage~~ control + 0.050 µg/g HOE-039866
control 32889801
12.5 g sample weight; 500 mL extraction volume; 100 mL aliquot; 5.0 mL final volume in methyl acetate
#000290 Set # 1G
MYL 05Jan01

Injection Date : 12/9/00 12:56:47 AM Seq. Line : 17
Sample Name : 0.050 µg/g P Vial : 13
Acq. Operator : MYL Inj : 1
Inj Volume : 5 µl
Sequence File : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\08DEC00A.S
Acq. Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed : 12/8/00 4:47:08 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed : 12/11/00 4:47:53 PM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites



External Standard Report

Sorted By : Signal
Calib. Data Modified : Monday, December 11, 2000 4:47:04 PM
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: FPD2 B,

RetTime [min]	Type	Area 150 pA*s	Amt/Area	Amount [µg/mL]	Grp	Name
5.467		-	-	-		HOE-070951
19.727	BB	401.04263	6.55728e-5	2.62975e-2		HOE-064706
Totals :				2.62975e-2		

Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

* The retention time and compound name were added on 11 Dec 00. MYL 04 Jan 01

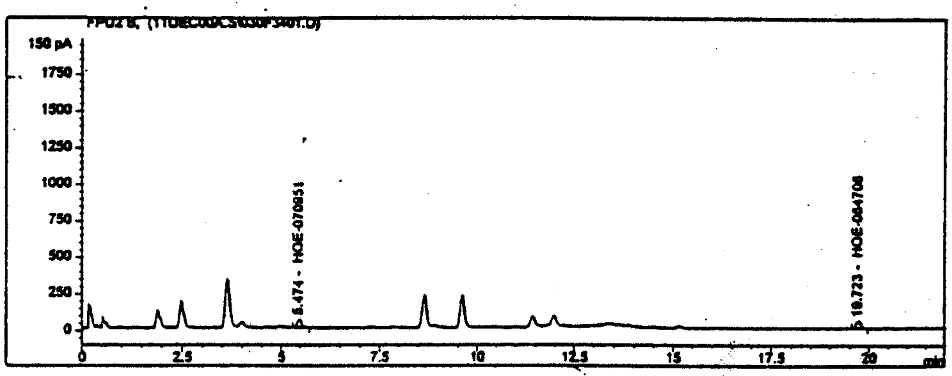
6890 EC-FPD 12/11/00 4:59:26 PM MYL Page 1 of 2

Control Corn Grain Sample 32889801
Fortified at 0.05 µg/g HOE-039866 (P)

0.0544 µg/g Equivalent Glufosinate Found as Free Acid, 109% Recovery
Analyzed in Set 1G, Curve 1, Injection No. 13

Figure 8a. Typical Chromatograms of Control Corn Grain Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\11DEC00A.S\030F3401.D          0.050 µg/g M1M2
     corn [struck out] control + 0.050 µg/g HOE-099730+HOE-061517
     control 33061201
     12.5 g sample weight; 500 mL extraction volume; 100 mL
     aliquot; 5.0 mL final volume in methyl acetate
     #000290           Set #  2G
=====================================================================
Injection Date    : 12/12/00 12:39:24 PM                Seq. Line :  34
Sample Name       : 0.050 µg/g M1M2                          Vial :  30
Acq. Operator     : MYL                                       Inj :   1
                                                       Inj Volume : 5 µl
Sequence File     : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\11DEC00A.S
Acq. Method       : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed      : 12/11/00 6:25:18 PM by MYL
Analysis Method   : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed      : 12/14/00 8:32:40 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
=====================================================================
```




```
=====================================================================
                     External Standard Report
=====================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, December 14, 2000 8:31:28 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area     Amt/Area     Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
--------|------|----------|----------|----------|--|------------------
  5.474 PB      438.89932 4.34723e-5 1.90800e-2     HOE-070951
 19.723 BP      472.92410 5.81766e-5 2.75131e-2     HOE-064706

Totals :                             4.65931e-2

 Results obtained with enhanced integrator!
=====================================================================

6890 EC-FPD 12/14/00 8:41:12 AM MYL                          Page 1 of 2
```

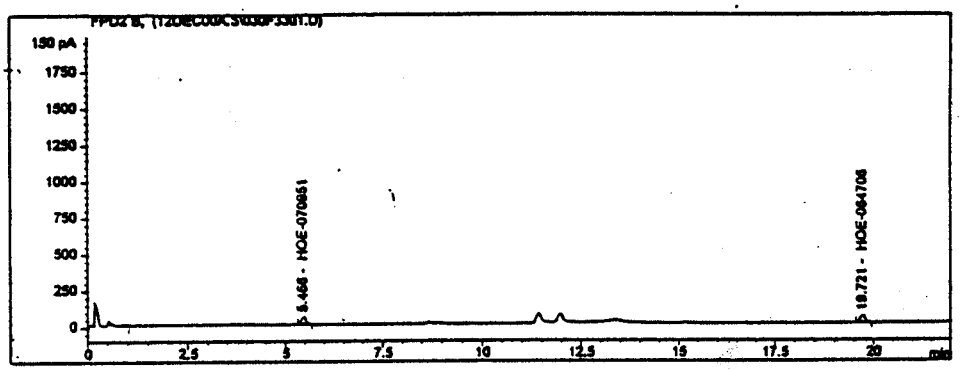
Control Corn Grain Sample 33061201
Fortified at 0.05 µg/g HOE-099730 (M1) + 0.05 µg/g HOE-061517 (M2)

HOE-099730 Fort. (M1) - 0.0562 µg/g Equivalent Glufosinate Found as Free Acid, 112% Recovery
HOE-061517 Fort. (M2) - 0.0386 µg/g Equivalent Glufosinate Found as Free Acid, 77% Recovery
Analyzed in Set 2G, Curve 2, Injection No. 30

Figure 8b. Typical Chromatograms of Control Corn Grain Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\12DEC00A.S\030F3301.D          0.050 µg/g M1M2
    corn grain control + 0.050 µg/g HOE-099730+HOE-061517
    control 33064701
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  3G

    ================================================================
    Injection Date  : 12/13/00 11:37:42 AM          Seq. Line :  33
    Sample Name     : 0.050 µg/g M1M2                    Vial :  30
    Acq. Operator   : MYL                                Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\12DEC00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/11/00 6:25:18 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/14/00 9:19:21 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```

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    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, December 14, 2000 9:11:32 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.466 BB      426.75156 4.51072e-5 1.92496e-2     HOE-070951
     19.721 BB      457.08905 5.80570e-5 2.65372e-2     HOE-064706

    Totals :                              4.57868e-2

     Results obtained with enhanced integrator!
    ================================================================

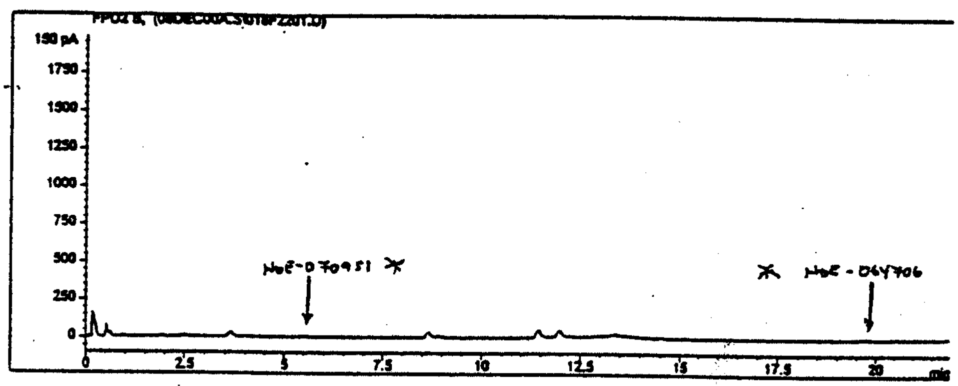
6890 EC-FPD 12/14/00 9:23:25 AM MYL                        Page 1 of 2
```

Control Corn Grain Sample 33064701
Fortified at 0.05 µg/g HOE-099730 (M1) + 0.05 µg/g HOE-061517 (M2)

HOE-099730 Fort. (M1) - 0.0537 µg/g Equivalent Glufosinate Found as Free Acid, 107% Recovery
HOE-061517 Fort. (M2) - 0.0388 µg/g Equivalent Glufosinate Found as Free Acid, 78% Recovery
Analyzed in Set 3G, Curve 2, Injection No. 30

Figure 8c. Typical Chromatograms of Control Corn Grain Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\08DEC00A.S\018F2201.D  Sample Name: 3288980
    corn forage (A) TRT    32889802
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290              Set #  1G
    (A) MYL 05 Jan 01
    ===================================================================
    Injection Date   : 12/9/00 3:28:21 AM               Seq. Line :  22
    Sample Name      : 32889802                               Vial :  18
    Acq. Operator    : MYL                                     Inj :   1
                                                        Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\08DEC00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/8/00 4:47:08 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/11/00 4:47:53 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ===================================================================
```

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    ===================================================================
                         External Standard Report
    ===================================================================

    Sorted By             :         Signal
    Calib. Data Modified  :         Monday, December 11, 2000 4:47:04 PM
    Multiplier            :         1.0000
    Dilution              :         1.0000

    Signal 1: FPD2 B,
```

RetTime [min]	Type	Area 150 pA*s	Amt/Area	Amount [µg/mL]	Grp	Name
5.467		-	-	-		HOE-070951
19.724		-	-	-		HOE-064706

Totals : 0.00000

* The retention time arrows and compound name were added on 11 Dec 00. MYL 05 Jan 01

Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

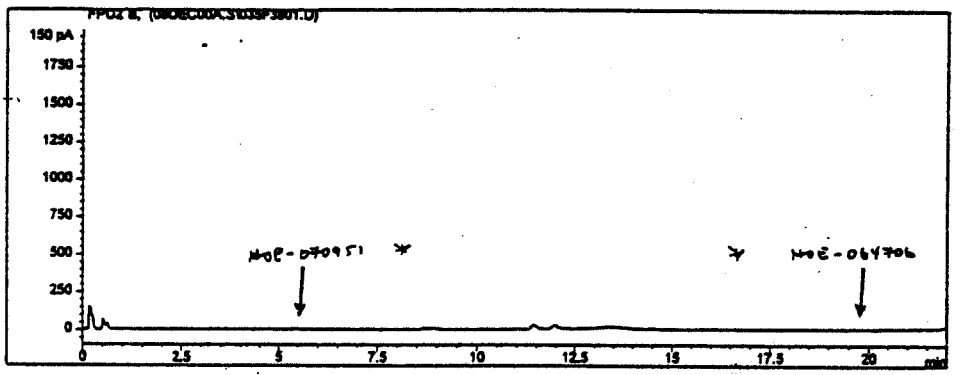
6890 EC-FPD 12/11/00 5:00:18 PM MYL — Page 1 of 2

Treated Corn Grain Sample 32889802
Analyzed in Set 1G, Curve 1, Injection No. 18

Residues of P + M1 Equivalent to Non-Detection
Residues Metabolite M2 Found Equivalent to Non-Detection
Total Residues of Glufosinate Reported as ND

Figure 9a. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\08DEC00A.S\035F3901.D  Sample Name: 32893602
    corn forage TRT   32893602
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  1G
    MYL 05Jan01
    ================================================================================
    Injection Date  : 12/9/00 12:04:31 PM          Seq. Line :  39
    Sample Name     : 32893602                        Vial :  35
    Acq. Operator   : MYL                              Inj :   1
                                                  Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\08DEC00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/8/00 4:47:08 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/11/00 5:50:07 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```




```
    ================================================================================
                              External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Monday, December 11, 2000 5:49:06 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]         150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.473           -           -          -          HOE-070951
     19.732           -           -          -          HOE-064706

    Totals :                                  0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

    6890 EC-FPD 12/11/00 6:03:23 PM MYL                        Page 1 of 2
```

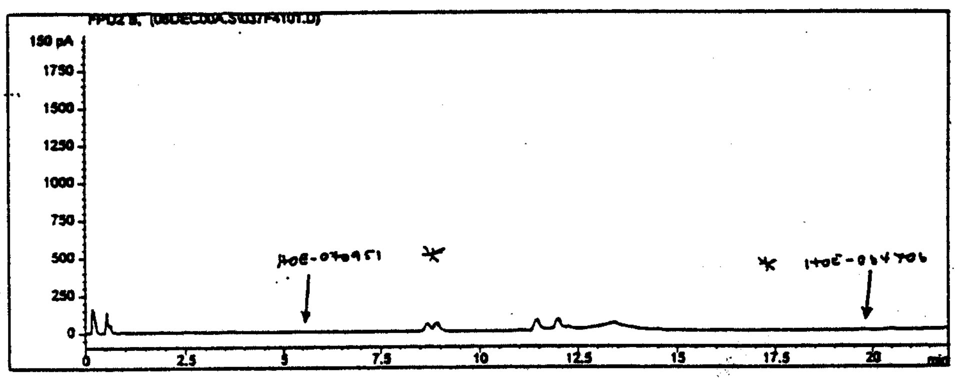
the retention time areas and compound name were added on 11 Dec 00. MYL 04Jan01

Treated Corn Grain Sample 32893602
Analyzed in Set 1G, Curve 2, Injection No. 35

Residues of P + M1 Equivalent to Non-Detection
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate Reported as ND

Figure 9b. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\08DEC00A.S\037F4101.D  Sample Name: 32893603
    corn forage grain TRT   32893603
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set # 1G
    @MYL 05Jan01
    ==================================================================
    Injection Date  : 12/9/00 1:05:12 PM              Seq. Line :  41
    Sample Name     : 32893603                             Vial :  37
    Acq. Operator   : MYL                                   Inj :   1
                                                    Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\08DEC00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/8/00 4:47:08 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/11/00 5:50:07 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ==================================================================
```




```
    ==================================================================
                          External Standard Report
    ==================================================================

    Sorted By            :        Signal
    Calib. Data Modified :        Monday, December 11, 2000 5:49:06 PM
    Multiplier           :        1.0000
    Dilution             :        1.0000

    Signal 1: FPD2 B,
```

RetTime [min]	Type	Area 150 pA*s	Amt/Area	Amount [µg/mL]	Grp	Name
5.473		-	-	-		HOE-070951
19.732		-	-	-		HOE-064706

```
    Totals :                                 0.00000
    Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found
```

* the retention time arrow and compound name were added on 11 Dec 00. MYL 04 Jun 01

6890 EC-FPD 12/11/00 6:03:46 PM MYL   Page 1 of 2

Treated Corn Grain Sample 32893603
Analyzed in Set 1G, Curve 2, Injection No. 37

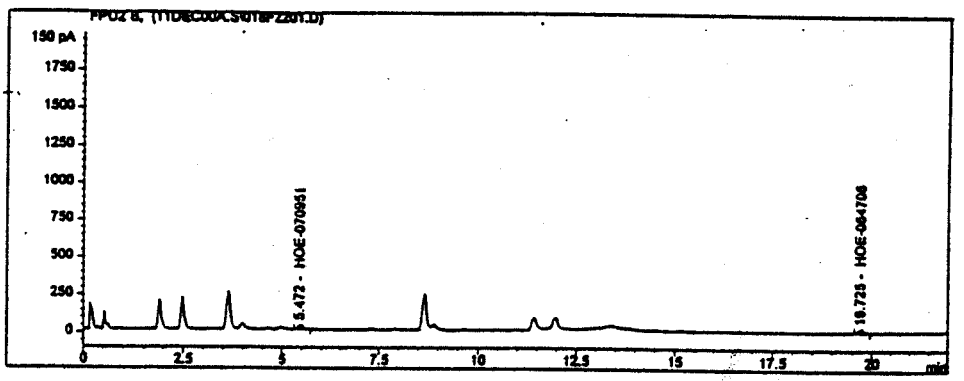
Residues of P + M1 Equivalent to Non-Detection
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate Reported as ND

Figure 9c. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

Data File C:\HPCHEM\2\DATA\GLUFOSIN\11DEC00A.S\018F2201.D Sample Name: 33061202
corn ~~forage~~ ② TRT 33061202
12.5 g sample weight; 500 mL extraction volume; 100 mL
aliquot; 5.0 mL final volume in methyl acetate
#000290 Set # 2G
④ Wt of Sample

Injection Date	: 12/12/00 6:35:11 AM	Seq. Line	: 22
Sample Name	: 33061202	Vial	: 18
Acq. Operator	: MYL	Inj	: 1
		Inj Volume	: 5 µl
Sequence File	: C:\HPCHEM\2\SEQUENCE\GLUFOSIN\11DEC00A.S		
Acq. Method	: C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M		
Last changed	: 12/11/00 6:25:18 PM by MYL		
Analysis Method	: C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M		
Last changed	: 12/14/00 8:18:21 AM by MYL		

GC Determination of Glufosinate-Ammonium and its Metabolites



External Standard Report

Sorted By	:	Signal
Calib. Data Modified	:	Thursday, December 14, 2000 8:11:17 AM
Multiplier	:	1.0000
Dilution	:	1.0000

Signal 1: FPD2 B,

RetTime [min]	Type	Area 150 pA*s	Amt/Area	Amount [µg/mL]	Grp	Name
5.472	PB	222.04556	4.52215e-5	1.00412e-2		HOE-070951
19.725	BB	243.58322	6.10683e-5	1.48752e-2		HOE-064706
Totals :				2.49164e-2		

Results obtained with enhanced integrator!

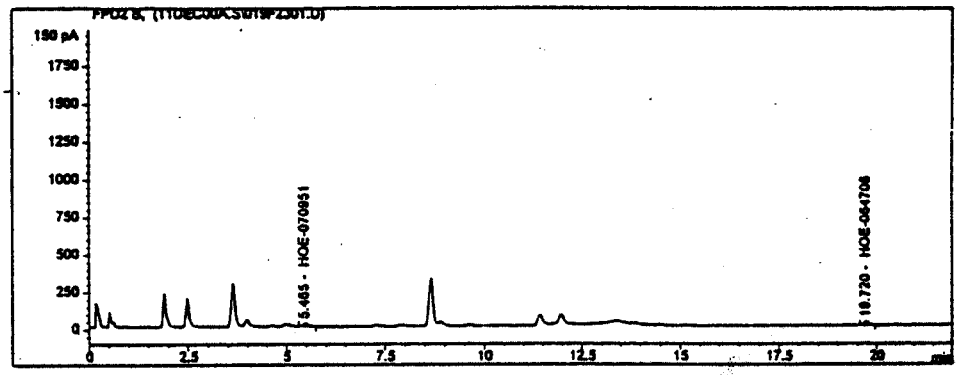
6890 EC-FPD 12/14/00 8:24:10 AM MYL Page 1 of 2

Treated Corn Grain Sample 33061202
Analyzed in Set, 2G, Curve 1, Injection No. 18

Residues of P + M1 Equivalent to 0.0304 µg/g Glufosinate as Uncorrected Free Acid
Residues Metabolite M2 Equivalent to 0.0203 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as (0.055 µg/g) Free Acid, Corrected for Recovery

Figure 9d. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\11DEC00A.S\019F2301.D  Sample Name: 33061203
   corn ~~forage~~ TRT   33061203
   12.5 g sample weight; 500 mL extraction volume; 100 mL
   aliquot; 5.0 mL final volume in methyl acetate
   #000290          Set #  2G
   50 µL of gln 01
   ================================================================
   Injection Date  : 12/12/00 7:05:31 AM          Seq. Line :  23
   Sample Name     : 33061203                        Vial :  19
   Acq. Operator   : MYL                             Inj :   1
                                                 Inj Volume : 5 µl
   Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\11DEC00A.S
   Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
   Last changed    : 12/11/00 6:25:18 PM by MYL
   Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
   Last changed    : 12/14/00 8:18:21 AM by MYL
   GC Determination of Glufosinate-Ammonium and its Metabolites
   ================================================================
```



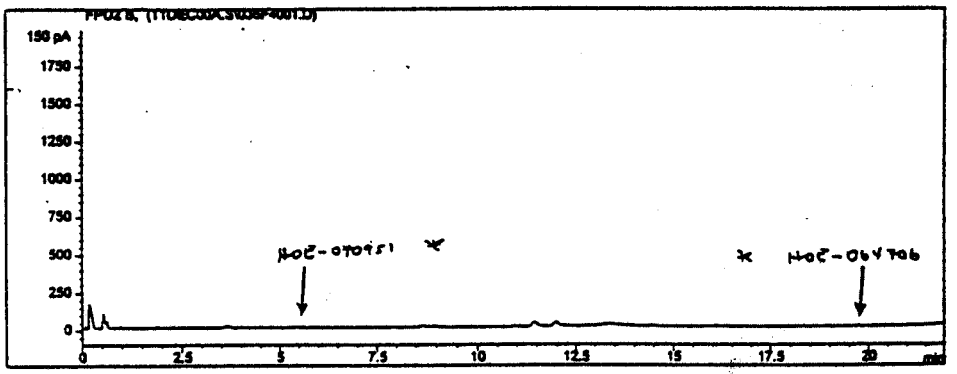

```
   ================================================================
                        External Standard Report
   ================================================================

   Sorted By             :      Signal
   Calib. Data Modified  :      Thursday, December 14, 2000 8:11:17 AM
   Multiplier            :      1.0000
   Dilution              :      1.0000

   Signal 1: FPD2 B,

   RetTime  Type     Area      Amt/Area    Amount   Grp   Name
    [min]          150 pA*s                [µg/mL]
   -------|------|----------|----------|----------|--|-----------------
     5.465 PP      168.29135 4.62440e-5 7.78247e-3    HOE-070951
    19.720 BB      240.85057 6.11536e-5 1.47289e-2    HOE-064706

   Totals :                             2.25114e-2

    Results obtained with enhanced integrator!
   ================================================================

   6890 EC-FPD 12/14/00 8:24:21 AM MYL                  Page 1 of 2
```

Treated Corn Grain Sample 33061203
Analyzed in Set 2G, Curve 1, Injection No. 19

Residues of P + M1 Equivalent to 0.0301 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to 0.0158 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as (0.048 µg/g) Free Acid, Corrected for Recovery

Figure 9e. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\11DEC00A.S\036F4001.D  Sample Name: 32905302
corn forage TRT   32905302
12.5 g sample weight; 500 mL extraction volume; 100 mL
aliquot; 5.0 mL final volume in methyl acetate
#000290           Set #  2G
MYL 01-Jun-01
================================================================================
Injection Date  : 12/12/00 3:41:17 PM            Seq. Line :  40
Sample Name     : 32905302                         Vial :  36
Acq. Operator   : MYL                               Inj :   1
                                                 Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\11DEC00A.S
Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/11/00 6:25:18 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/14/00 8:32:40 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                        External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, December 14, 2000 8:31:28 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount   Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|---------|----------|----------|--|------------------
  5.467           -           -          -          HOE-070951
 19.726           -           -          -          HOE-064706

Totals :                                  0.00000

 Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

6890 EC-FPD 12/14/00 8:42:16 AM MYL                          Page 1 of 2
```

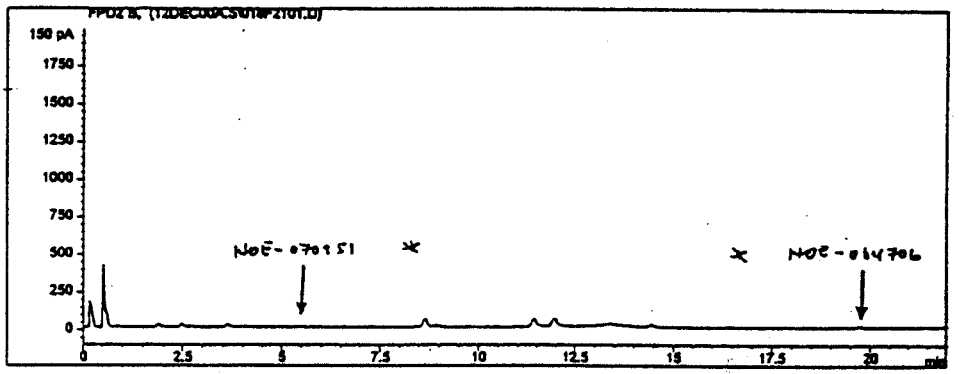
* the retention time arrow and compound name was added on 14 Dec 00. MYL 01 Jun 01

Treated Corn Grain Sample 32905302
Analyzed in Set 2G, Curve 2, Injection No. 36

Residues of P + M1 Equivalent to Non-Detection
Residues of Metabolite M2 Equivalent to Non-Detection Limit
Total Residues of Glufosinate were Reported as ND

Figure 9f. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\12DEC00A.S\018F2101.D  Sample Name: 33064702
    corn grain  TRT    33064702
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290            Set #  3G

    ================================================================================
    Injection Date   : 12/13/00 5:33:48 AM              Seq. Line :  21
    Sample Name      : 33064702                            Vial :  18
    Acq. Operator    : MYL                                  Inj :   1
                                                       Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\12DEC00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/11/00 6:25:18 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/14/00 8:59:50 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```




```
    ================================================================================
                              External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, December 14, 2000 8:51:39 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type      Area      Amt/Area     Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.465            -           -          -          HOE-070951
     19.723            -           -          -          HOE-064706

    Totals :                                   0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

    6890 EC-FPD 12/14/00 9:04:13 AM MYL                          Page 1 of 2
```

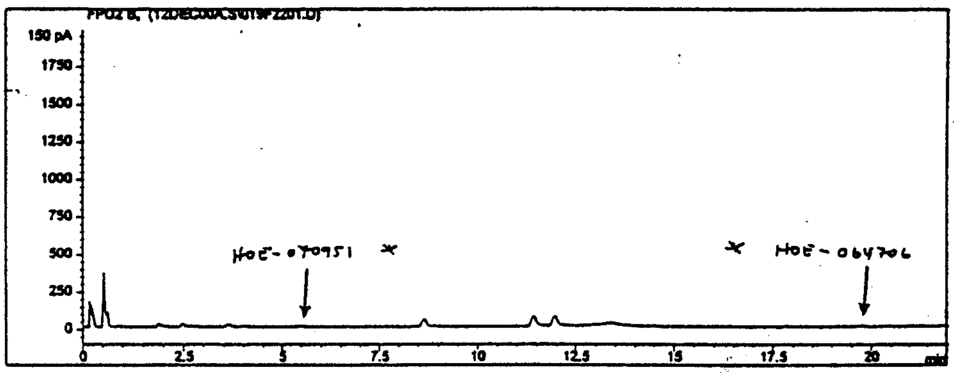
* The retention time area and compound name were added on 14 Dec 00. MYL 04 Jun 01

Treated Corn Grain Sample 33064702
Analyzed in Set 3G, Curve 1, Injection No. 18

Residues of P + M1 Equivalent to Non-Detection
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate were Reported as ND

Figure 9g. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\12DEC00A.S\019F2201.D  Sample Name: 33064703
    corn grain  TRT    33064703
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  3G

    =====================================================================
    Injection Date   : 12/13/00 6:04:06 AM            Seq. Line :  22
    Sample Name      : 33064703                            Vial :  19
    Acq. Operator    : MYL                                  Inj :   1
                                                     Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\12DEC00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/11/00 6:25:18 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/14/00 8:59:50 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    =====================================================================
```




```
    =====================================================================
                         External Standard Report
    =====================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, December 14, 2000 8:51:39 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type    Area      Amt/Area    Amount    Grp   Name
     [min]         150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.465           -          -          -          HOE-070951
     19.723           -          -          -          HOE-064706

    Totals :                              0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

6890 EC-FPD 12/14/00 9:04:26 AM MYL                          Page 1 of 2
```

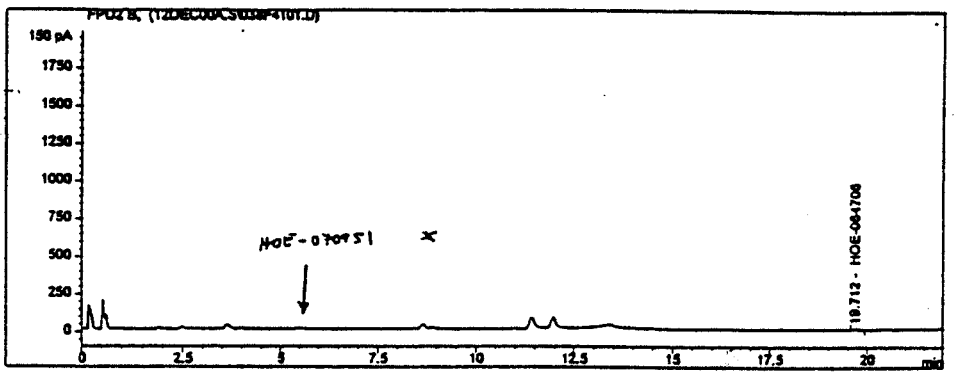
* the retention time arrows and compound name were added on 14 Dec 00 MYL 04Jan01

Treated Corn Grain Sample 33064703
Analyzed in Set 3G, Curve 1, Injection No. 19

Residues of P + M1 Equivalent to Non-Detection
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate were Reported as ND

Figure 9h. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\12DEC00A.S\038F4101.D  Sample Name: 33177503
    corn grain TRT   33177503
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  3G

================================================================================
Injection Date  : 12/13/00 3:40:07 PM            Seq. Line :  41
Sample Name     : 33177503                            Vial :  38
Acq. Operator   : MYL                                  Inj :   1
                                                 Inj Volume : 5 µl
Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\12DEC00A.S
Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/11/00 6:25:18 PM by MYL
Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed    : 12/14/00 9:19:21 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                          External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, December 14, 2000 9:11:32 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type      Area      Amt/Area    Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|-----------------
  5.463            -           -           -          HOE-070951
 19.712 BP      103.59633 7.30192e-5 7.56452e-3        HOE-064706

Totals :                                7.56452e-3

 Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

6890 EC-FPD 12/14/00 9:24:53 AM MYL                          Page 1 of 2
```

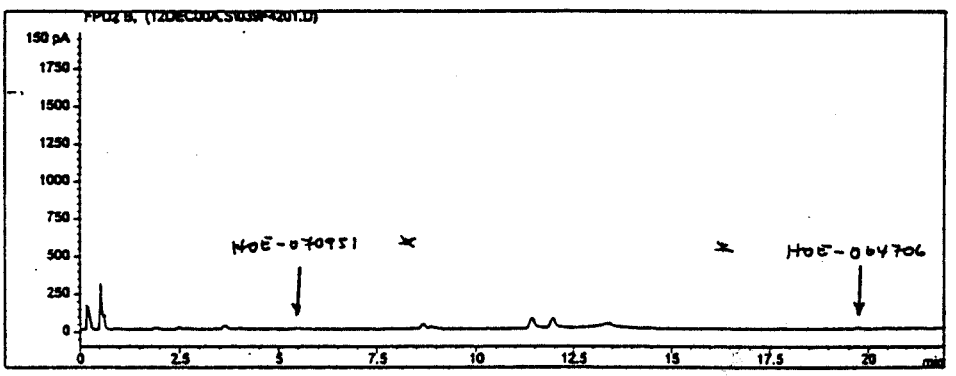
Ⓐ the retention time and compound name were added on 14 Dec 00. Ⓐ MYL 04 Jun 01

Treated Corn Grain Sample 33177503
Analyzed in Set 3G, Curve 2, Injection No. 38

Residues of P + M1 Equivalent to 0.0158 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate Reported as ND, Corrected for Recovery

Figure 9i. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\12DEC00A.S\039F4201.D          33177503  DUP
  corn grain TRT   33177503   DUP
  12.5 g sample weight; 500 mL extraction volume; 100 mL
  aliquot; 5.0 mL final volume in methyl acetate
  #000290          Set #  3G

  ================================================================
  Injection Date  : 12/13/00 4:10:21 PM          Seq. Line :  42
  Sample Name     : 33177503  DUP                    Vial :  39
  Acq. Operator   : MYL                              Inj :   1
                                                 Inj Volume : 5 µl
  Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\12DEC00A.S
  Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
  Last changed    : 12/11/00 6:25:18 PM by MYL
  Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
  Last changed    : 12/14/00 9:19:21 AM by MYL
  GC Determination of Glufosinate-Ammonium and its Metabolites
  ================================================================
```




```
  ================================================================
                        External Standard Report
  ================================================================

  Sorted By             :      Signal
  Calib. Data Modified  :      Thursday, December 14, 2000 9:11:32 AM
  Multiplier            :      1.0000
  Dilution              :      1.0000

  Signal 1: FPD2 B,

  RetTime  Type     Area      Amt/Area    Amount   Grp   Name
   [min]         150 pA*s                [µg/mL]
  -------|------|----------|----------|----------|--|-----------------
    5.463           -           -          -          HOE-070951
   19.725           -           -          -          HOE-064706

  Totals :                                 0.00000

   Results obtained with enhanced integrator!
  1 Warnings or Errors :

  Warning : Calibrated compound(s) not found

  6890 EC-FPD 12/14/00 9:25:04 AM MYL                    Page 1 of 2
```

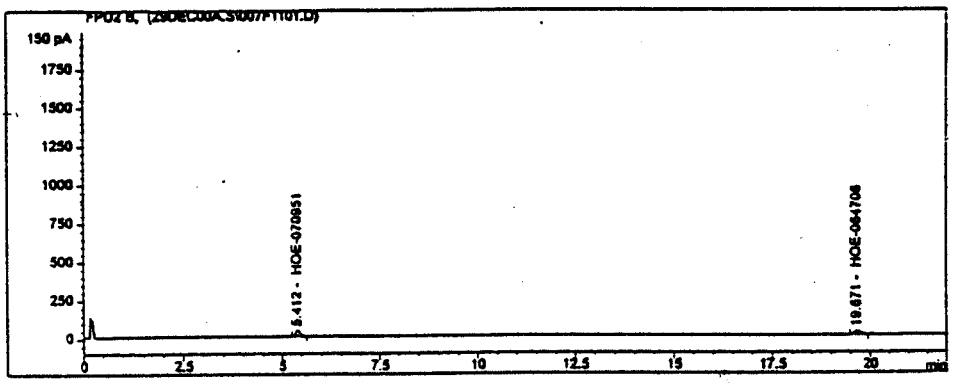
* the retention time arrow and compound name were added on 14 Dec 00. MYL 04 Jan 01

Treated Corn Grain Sample ID. 33177503 - Duplicate Analysis
Analyzed in Set 3G, Curve 2, Injection No. 39

Residues of P + M1 Equivalent to Non-Detection
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate Reported as ND

Figure 9j. Typical Chromatograms of Treated Corn Grain from the Analysis for Residues of Glufosinate Ammonium in Corn Grain

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\007F1101.D                0.015 µg/mL
    0.015 µg/mL HOE-064706 & 070951
    G8G913Nov00-0.015MIX
    #000290            Set #  1S

    ================================================================================
    Injection Date  : 12/30/00 12:35:44 AM          Seq. Line :  11
    Sample Name     : 0.015 µg/mL                       Vial :   7
    Acq. Operator   : MYL                               Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/29/00 6:48:38 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/3/01 5:26:35 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```



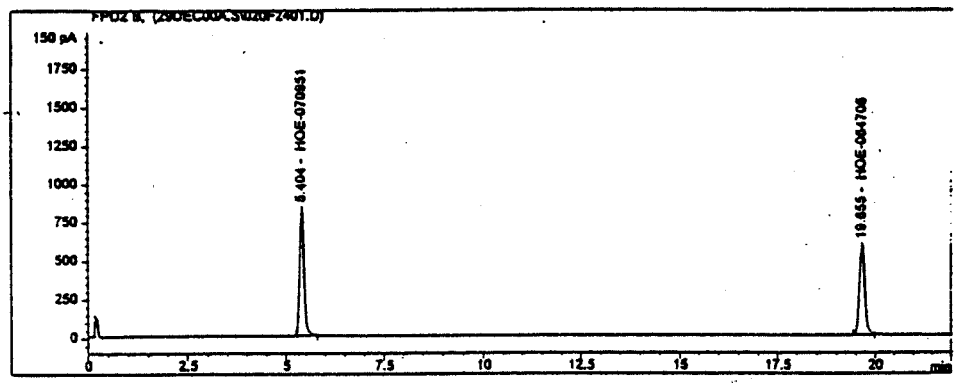

```
    ================================================================================
                                External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Wednesday, January 03, 2001 5:26:03 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]         150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.412 BB      308.65936 4.39927e-5 1.35788e-2     HOE-070951
     19.671 BP      261.64352 5.57595e-5 1.45891e-2     HOE-064706

    Totals :                             2.81679e-2

     Results obtained with enhanced integrator!
    ================================================================================


6890 EC-FPD 1/3/01 5:29:54 PM MYL                                  Page 1 of 2
```

0.015 µg/mL Each HOE-070951 and HOE-064706 Derivative Calibration Standards

Each Equivalent to 0.03 µg/g Glufosinate as the Free Acid (HOE-035956)
Analyzed in Set 1S, Curve 1, Injection No. 7

Figure 10a. Typical Chromatogram of a Low Level Mixed Calibration Standard from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\020F2401.D                0.30 µg/mL
    0.30 µg/mL HOE-064706 & 070951
    G8G91JNov00-0.30MIX
    #000290            Set #  1S

    ================================================================
    Injection Date   : 12/30/00 7:10:29 AM          Seq. Line :  24
    Sample Name      : 0.30 µg/mL                       Vial :  20
    Acq. Operator    : MYL                              Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/29/00 6:48:38 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/3/01 5:26:35 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



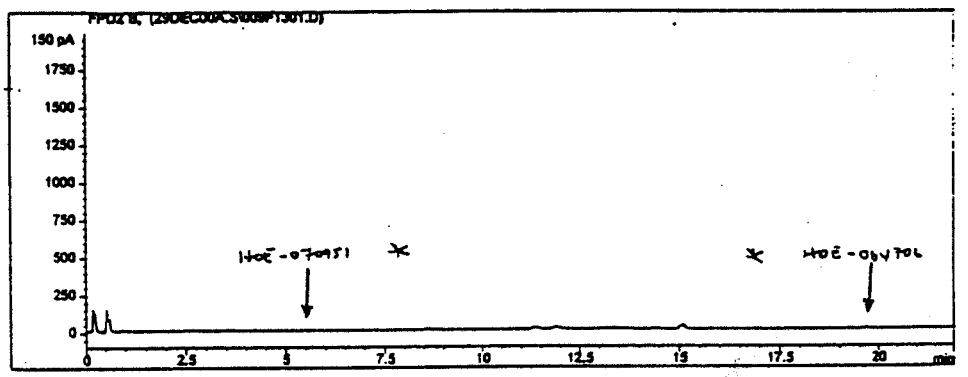

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Wednesday, January 03, 2001 5:26:03 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.404 BB     6564.93213 4.55666e-5 2.99142e-1     HOE-070951
     19.655 BBA    5797.39014 5.24072e-5 3.03825e-1     HOE-064706

    Totals :                              6.02967e-1

     Results obtained with enhanced integrator!
    ================================================================


6890 EC-FPD 1/3/01 5:32:15 PM MYL                          Page 1 of 2
```

0.30 µg/mL Each HOE-070951 and HOE-064706 Derivative Calibration Standards

Each Equivalent to 0.60 µg/g Glufosinate as the Free Acid (HOE-035956)
Analyzed in Set 1S, Curve 1, Injection No. 20

Figure 10b. Typical Chromatogram of a High Level Mixed Calibration Standard from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\009F1301.D          UTC 32890101
    corn stover control   32890101
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1S

    ================================================================
    Injection Date   : 12/30/00 1:36:28 AM          Seq. Line :  13
    Sample Name      : UTC 32890101                      Vial :   9
    Acq. Operator    : MYL                               Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/29/00 6:48:38 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/3/01 5:26:35 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



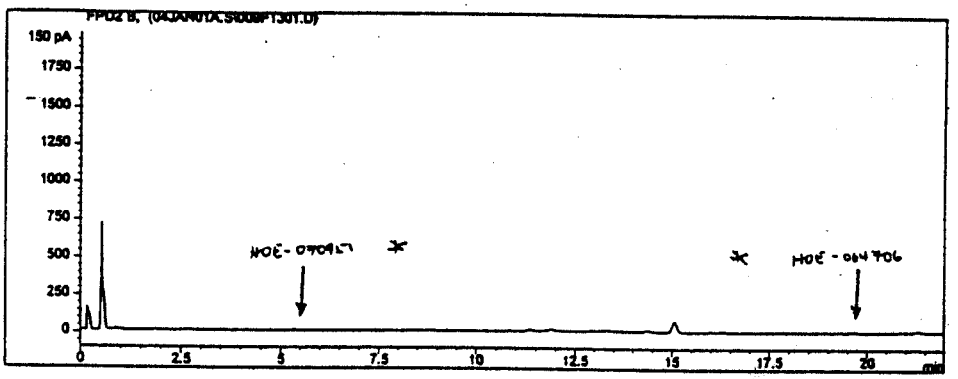

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Wednesday, January 03, 2001 5:26:03 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.404          -           -           -          HOE-070951
     19.655          -           -           -          HOE-064706

    Totals :                                  0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

    6890 EC-FPD 1/3/01 5:30:17 PM MYL                        Page 1 of 2
```

* The retention time and compound name were added on 03 Jan 01. MYL 04 Jan 01 04 Jan 01

Control Corn Stover Sample 32890101
Analyzed in Set 1S, Curve 1, Injection No. 9

Figure 11a. Typical Chromatograms of Control Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\04JAN01A.S\009F1301.D          UTC 32906101
    corn stover control   32906101
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  2S

    ================================================================
    Injection Date  : 1/4/01 9:34:17 PM              Seq. Line :  13
    Sample Name     : UTC 32906101                       Vial :   9
    Acq. Operator   : MYL                                Inj :   1
                                                    Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\04JAN01A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/4/01 1:47:57 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/5/01 2:01:58 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



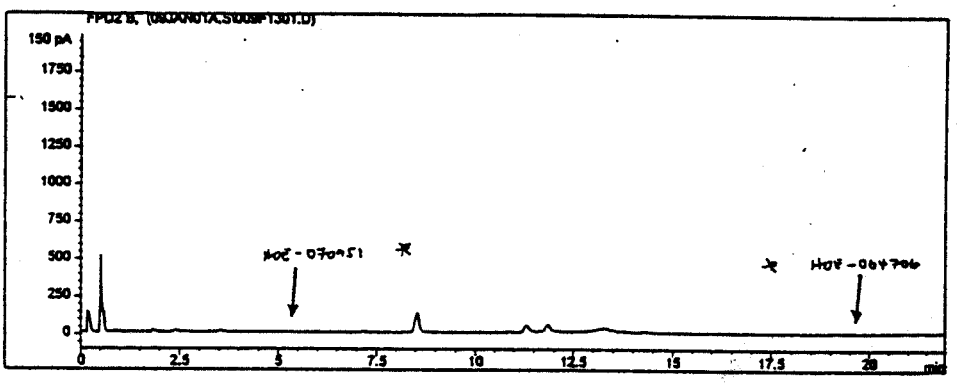

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Friday, January 05, 2001 2:01:26 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.392           -           -          -         HOE-070951
     19.648           -           -          -         HOE-064706

    Totals :                                 0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

6890 EC-FPD 1/5/01 2:13:04 PM MYL                          Page 1 of 2
```

* the retention time and compound name were added on 05Jan01. MYL 04 Jan 01

Control Corn Stover Sample 32906101
Analyzed in Set 2S, Curve 1, Injection No. 9

Figure 11b. Typical Chromatograms of Control Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\09JAN01A.S\009F1301.D          UTC 33178301
    corn stover control   33178301
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  3S

    ================================================================
    Injection Date   : 1/10/01 3:08:55 AM              Seq. Line :  13
    Sample Name      : UTC 33178301                         Vial :   9
    Acq. Operator    : MYL                                   Inj :   1
                                                       Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\09JAN01A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/9/01 4:27:42 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/11/01 7:54:35 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



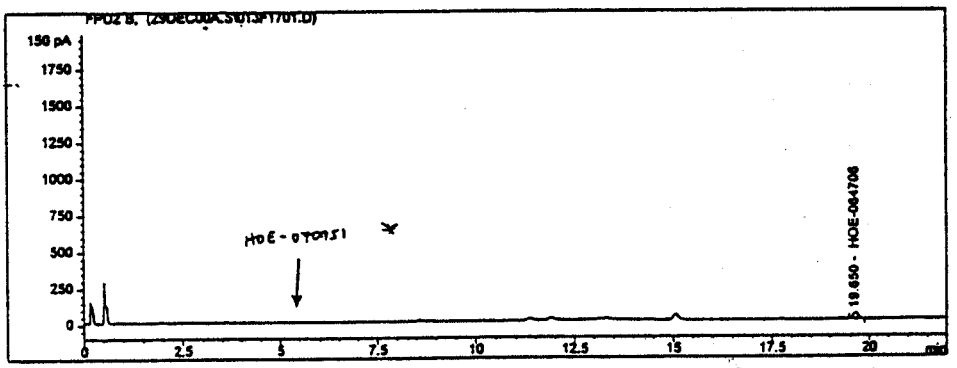

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, January 11, 2001 7:54:09 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.362           -           -          -          HOE-070951
     19.593           -           -          -          HOE-064706

    Totals :                                 0.00000

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

6890 EC-FPD 1/11/01 7:58:18 AM MYL                          Page 1 of 2
```

The retention time arrow and compound name were added on 11 Jan 01 MYL

Control Corn Stover Sample 33178301
Analyzed in Set 3S, Curve 1, Injection No. 9

Figure 11c. Typical Chromatograms of Control Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\013F1701.D          0.050 µg/g P
    corn stover control + 0.050 µg/g HOE-039866
    control 32890101
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1S

    ================================================================
    Injection Date  : 12/30/00 3:37:59 AM          Seq. Line :  17
    Sample Name     : 0.050 µg/g P                      Vial :  13
    Acq. Operator   : MYL                               Inj :   1
                                                  Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/29/00 6:48:38 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/3/01 5:26:35 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Wednesday, January 03, 2001 5:26:03 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.404                -          -          -       HOE-070951
     19.650 BB       402.30057 5.45320e-5 2.19383e-2      HOE-064706

    Totals :                                 2.19383e-2

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

6890 EC-FPD 1/3/01 5:31:01 PM MYL                              Page 1 of 2
```

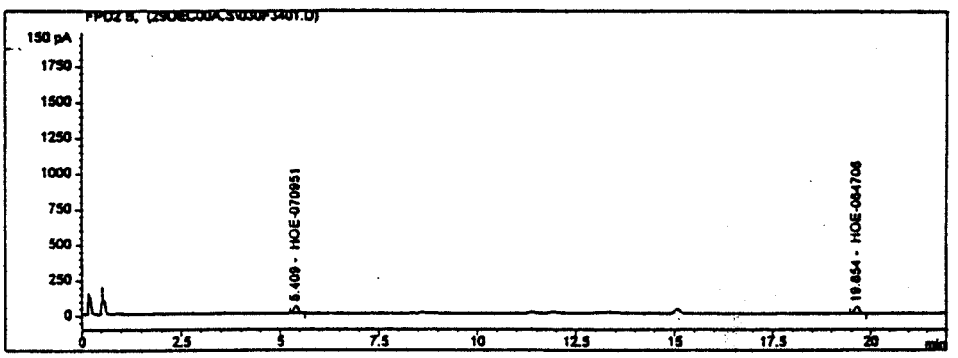
+ the retention time arrow and compound name were added on 03 Jan 01.
MYL 04 Jan 01

Control Corn Stover Sample 32890101
Fortified at 0.05 µg/g HOE-039866 (P)

0.0442 µg/g Equivalent Glufosinate Found as Free Acid, 88% Recovery
Analyzed in Set 1S, Curve 1, Injection No. 13

Figure 12a. Typical Chromatograms of Control Corn Stover Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\030F3401.D          0.050 µg/g M1M2
    corn stover control + 0.050 µg/g HOE-099730 + HOE-06151
    7
    control 32890101
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1S

    ================================================================
    Injection Date  : 12/30/00 12:13:39 PM          Seq. Line :  34
    Sample Name     : 0.050 µg/g M1M2                    Vial :  30
    Acq. Operator   : MYL                                Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 12/29/00 6:48:38 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/3/01 5:43:53 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



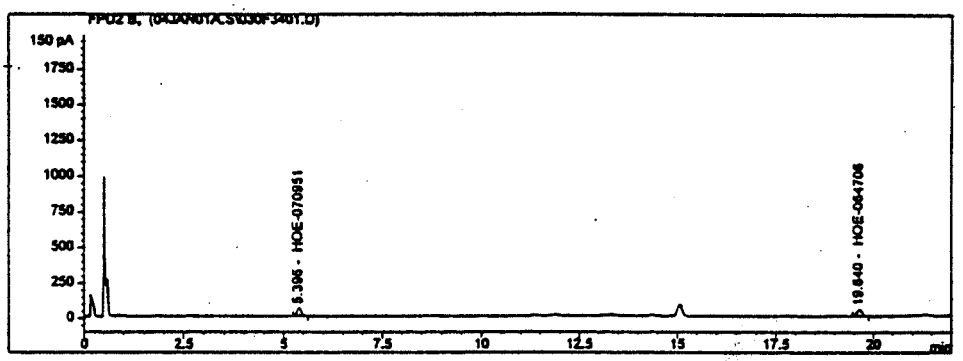

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Wednesday, January 03, 2001 5:43:39 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.409 BB      426.83585 4.65634e-5 1.98749e-2    HOE-070951
     19.654 BB      451.86087 5.79724e-5 2.61955e-2    HOE-064706

    Totals :                             4.60704e-2

     Results obtained with enhanced integrator!
    ================================================================

    6890 EC-FPD 1/3/01 5:47:48 PM MYL                    Page 1 of 2
```

Control Corn Stover Sample 32890101
Fortified at 0.05 µg/g HOE-099730 (M1) + 0.05 µg/g HOE-061517 (M2)

HOE-099730 Fort. (M1) - 0.0534 µg/g Equivalent Glufosinate Found as Free Acid, 107% Recovery
HOE-061517 Fort. (M2) - 0.0399 µg/g Equivalent Glufosinate Found as Free Acid, 80% Recovery
Analyzed in Set 1S, Curve 2, Injection No. 30

Figure 12b. Typical Chromatogram of Control Corn Stover Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\04JAN01A.S\030F3401.D          0.050 µg/g M1M2
    corn stover control + 0.050 µg/g HOE-099730 + HOE-06151
    7
    control 32906101
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  2S

    ================================================================
    Injection Date   : 1/5/01 8:09:21 AM              Seq. Line :  34
    Sample Name      : 0.050 µg/g M1M2                    Vial :  30
    Acq. Operator    : MYL                                 Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\04JAN01A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/4/01 1:47:57 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/5/01 2:59:14 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



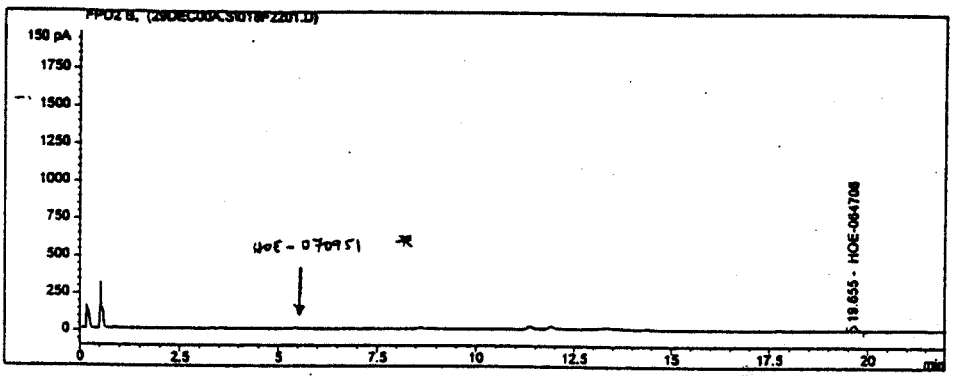

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Friday, January 05, 2001 2:57:11 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.396 BB      462.55539 4.83362e-5 2.23582e-2    HOE-070951
     19.640 BB      464.21286 5.86444e-5 2.72235e-2    HOE-064706

    Totals :                             4.95816e-2

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 1/5/01 3:10:43 PM MYL                          Page 1 of 2
```

Control Corn Stover Sample 32906101
Fortified at 0.05 µg/g HOE-099730 (M1) + 0.05 µg/g HOE-061517 (M2)

HOE-099730 Fort. (M1) - 0.0560 µg/g Equivalent Glufosinate Found as Free Acid, 112% Recovery
HOE-061517 Fort. (M2) - 0.0459 µg/g Equivalent Glufosinate Found as Free Acid, 92% Recovery
Analyzed in Set 2S, Curve 2, Injection No. 30

Figure 12c. Typical Chromatograms of Control Corn Stover Fortified at 0.05 µg/g from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\018F2201.D  Sample Name: 32890102
    corn stover  TRT   32890102
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1S

================================================================================
Injection Date   : 12/30/00 6:09:44 AM            Seq. Line :  22
Sample Name      : 32890102                           Vial :  18
Acq. Operator    : MYL                                Inj :   1
                                                  Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 12/29/00 6:48:38 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 1/3/01 5:26:35 PM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```




```
================================================================================
                          External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Wednesday, January 03, 2001 5:26:03 PM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type     Area      Amt/Area    Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|------------------
  5.404              -           -          -        HOE-070951
 19.655 BP      353.56769 5.48467e-5 1.93920e-2       HOE-064706

Totals :                                1.93920e-2

 Results obtained with enhanced integrator!
1 Warnings or Errors :

Warning : Calibrated compound(s) not found

6890 EC-FPD 1/3/01 5:31:54 PM MYL                          Page 1 of 2
```

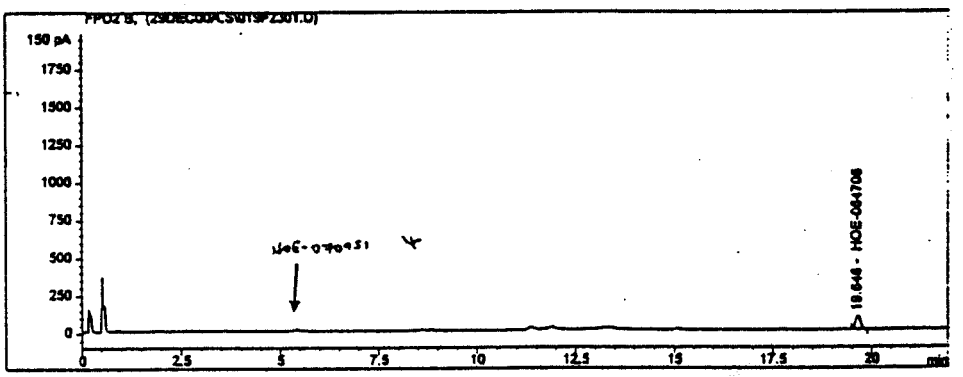
* The retention time and area and compound name were added on 03 Jan 01. ① MYL 04 Jan 01

Treated Corn Stover Sample 32890102
Analyzed in Set 1S, Curve 1, Injection No. 18

Residues of P + M1 Equivalent to 0.0391 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate Reported as (0.042 µg/g) Free Acid, Corrected for Recovery

Figure 13a. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\019F2301.D  Sample Name: 32890103
    corn stover  TRT   32890103
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  1S

    ================================================================================
    Injection Date   : 12/30/00 6:40:04 AM            Seq. Line :  23
    Sample Name      : 32890103                          Vial :  19
    Acq. Operator    : MYL                               Inj :   1
                                                    Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/29/00 6:48:38 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/3/01 5:26:35 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```




```
    ================================================================================
                              External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Wednesday, January 03, 2001 5:26:03 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type      Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.404            -           -          -          HOE-070951
     19.646 BP      817.22162 5.33728e-5 4.36174e-2     HOE-064706

    Totals :                                4.36174e-2

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

    6890 EC-FPD 1/3/01 5:32:04 PM MYL                          Page 1 of 2
```

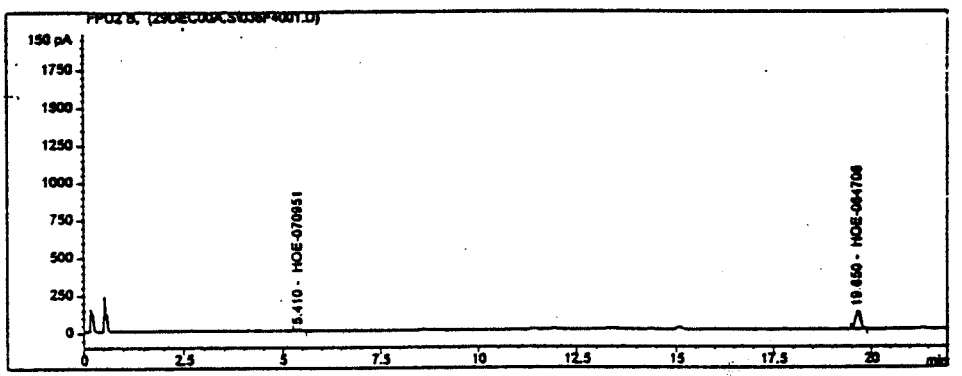
* the retention time and compound name were added on 03 Jan 01 MYL 04 Jan 01

Treated Corn Stover Sample 32890103
Analyzed in Set 1S, Curve 1, Injection No. 19

Residues of P + M1 Equivalent to 0.0875 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to Non-Detection
Total Residues of Glufosinate Reported as 0.09 µg/g Free Acid, Corrected for Recovery

Figure 13b. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\29DEC00A.S\036F4001.D  Sample Name: 32894402
    corn stover TRT   32894402
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  1S

    ================================================================================
    Injection Date   : 12/30/00 3:15:36 PM              Seq. Line :  40
    Sample Name      : 32894402                            Vial :  36
    Acq. Operator    : MYL                                  Inj :   1
                                                       Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\29DEC00A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 12/29/00 6:48:38 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/3/01 5:43:53 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```



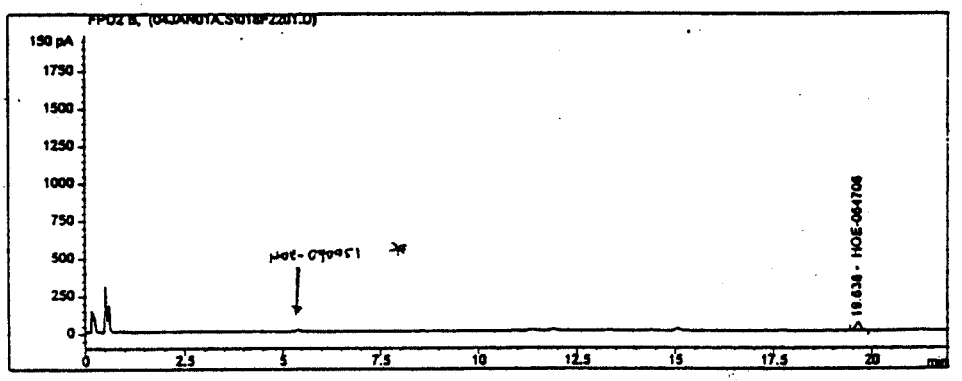

```
    ================================================================================
                              External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Wednesday, January 03, 2001 5:43:39 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.410 BB      107.26668 4.87275e-5 5.22684e-3     HOE-070951
     19.650 BP     1062.07422 5.40834e-5 5.74406e-2     HOE-064706

    Totals :                              6.26674e-2

     Results obtained with enhanced integrator!
    ================================================================================


6890 EC-FPD 1/3/01 5:48:54 PM MYL                                  Page 1 of 2
```

Treated Corn Stover Sample 32894402
Analyzed in Set 1S Curve 2, Injection No. 36

Residues of P + M1 Equivalent to 0.1157 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to 0.0106 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as 0.12 µg/g Free Acid, Corrected for Recovery

Figure 13c. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\04JAN01A.S\018F2201.D  Sample Name: 32906102
    corn stover  TRT   32906102
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  2S

    ================================================================
    Injection Date   : 1/5/01 2:06:29 AM              Seq. Line :  22
    Sample Name      : 32906102                           Vial :  18
    Acq. Operator    : MYL                                 Inj :   1
                                                     Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\04JAN01A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/4/01 1:47:57 PM by MYL
    Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/5/01 2:01:58 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Friday, January 05, 2001 2:01:26 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type      Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.392                -          -          -      HOE-070951
     19.638 BP       506.90616 5.90878e-5 2.99520e-2     HOE-064706

    Totals :                                 2.99520e-2

     Results obtained with enhanced integrator!
    1 Warnings or Errors :

    Warning : Calibrated compound(s) not found

    6890 EC-FPD 1/5/01 2:14:40 PM MYL                      Page 1 of 2
```

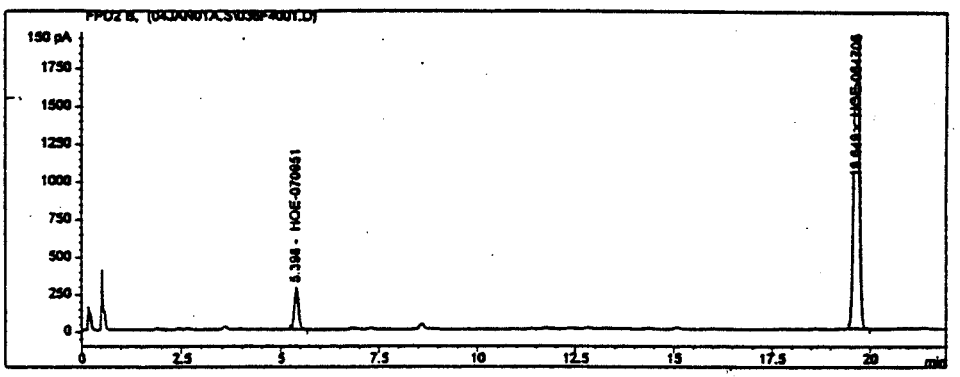
* the retention time and compound name added on 05-Jan-01. MYL 04 Jan 01

Treated Corn Stover Sample 32906102
Analyzed in Set 2S, Curve 1, Injection No. 18

Residues of P + M1 Equivalent to 0.0611 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 were at Non-Detection Limit
Total Residues of Glufosinate Reported as 0.06 µg/g Free Acid, Corrected for Recovery

Figure 13d. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\04JAN01A.S\036F4001.D  Sample Name: 33062002
    corn stover TRT   33062002
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  2S

================================================================================
Injection Date   : 1/5/01 11:10:44 AM              Seq. Line :  40
Sample Name      : 33062002                            Vial :  36
Acq. Operator    : MYL                                  Inj :   1
                                                   Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\04JAN01A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 1/4/01 1:47:57 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 1/5/01 2:59:14 PM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```



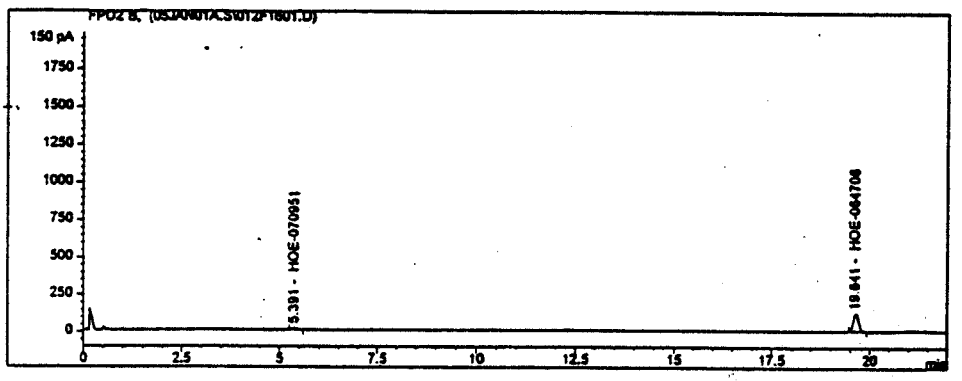

```
================================================================================
                          External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Friday, January 05, 2001 2:57:11 PM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type      Area      Amt/Area    Amount   Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|------------------
  5.398 BB     2148.64648 4.20738e-5 9.04017e-2    HOE-070951
 19.649 BBA    2.34441e4  4.84499e-5    1.13587    HOE-064706

Totals :                                1.22627

 Results obtained with enhanced integrator!
================================================================================

6890 EC-FPD 1/5/01 3:11:47 PM MYL                          Page 1 of 2
```

Treated Corn Stover Sample 33062002 (Method Factor 2)
Analyzed in Set 2S, Curve 2, Injection No. 36

Residues of P + M1 Exceeded the Calibration Standard Range (For P + M1 values see Figure 13f.)
Residues Metabolite M2 Found Equivalent to 0.1816 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported in Figure 13f.

Figure 13e. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\05JAN01A.S\012F1601.D  Sample Name: 33062002
    corn stover TRT   33062002          20X DILUTION
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  2S (reinj)

    ================================================================================
    Injection Date  : 1/6/01 2:29:53 AM                Seq. Line :  16
    Sample Name     : 33062002                              Vial :  12
    Acq. Operator   : MYL                                    Inj :   1
                                                       Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\05JAN01A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/5/01 5:05:45 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/9/01 2:59:25 PM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================================
```



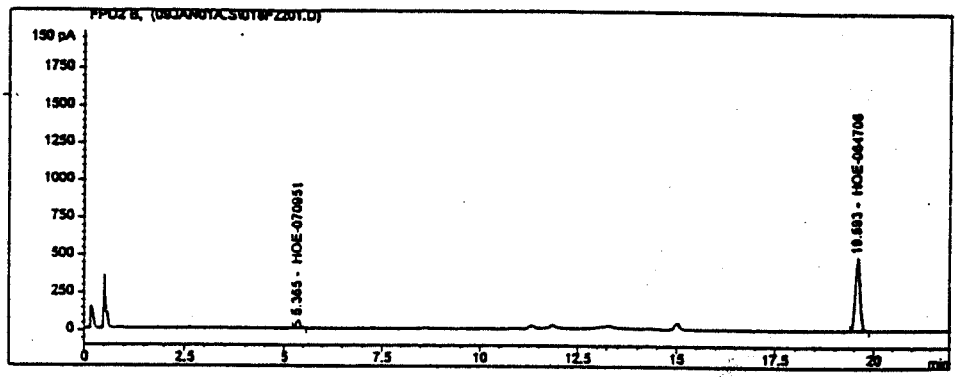

```
    ================================================================================
                              External Standard Report
    ================================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Tuesday, January 09, 2001 2:54:35 PM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.391 BB      112.33689 5.29669e-5 5.95014e-3    HOE-070951
     19.641 BB     1136.13892 5.37767e-5 6.10978e-2    HOE-064706

    Totals :                              6.70479e-2

    Results obtained with enhanced integrator!
    ================================================================================

    6890 EC-FPD 1/9/01 3:06:47 PM MYL                          Page 1 of 2
```

Treated Corn Stover Sample 33062002 – 20x Dilution (Method Factor 40)
Analyzed in Set 2S (reinjected), Injection No. 12

Residues of P + M1 Equivalent to 2.4570 µg/g Glufosinate as Uncorrected Free Acid
For Residues of Metabolite M2 Equivalent to Glufosinate as the Uncorrected Free Acid, See Figure 13e.
Total Residues of Glufosinate Reported as 2.51 µg/g as Free Acid, Corrected for Recovery

Figure 13f. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\09JAN01A.S\018F2201.D  Sample Name: 33178302
    corn stover  TRT   33178302
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  3S

================================================================================
Injection Date   : 1/10/01 7:42:03 AM              Seq. Line :  22
Sample Name      : 33178302                            Vial :  18
Acq. Operator    : MYL                                  Inj :   1
                                                   Inj Volume : 5 µl
Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\09JAN01A.S
Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 1/9/01 4:27:42 PM by MYL
Analysis Method  : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
Last changed     : 1/11/01 7:54:35 AM by MYL
GC Determination of Glufosinate-Ammonium and its Metabolites
================================================================================
```



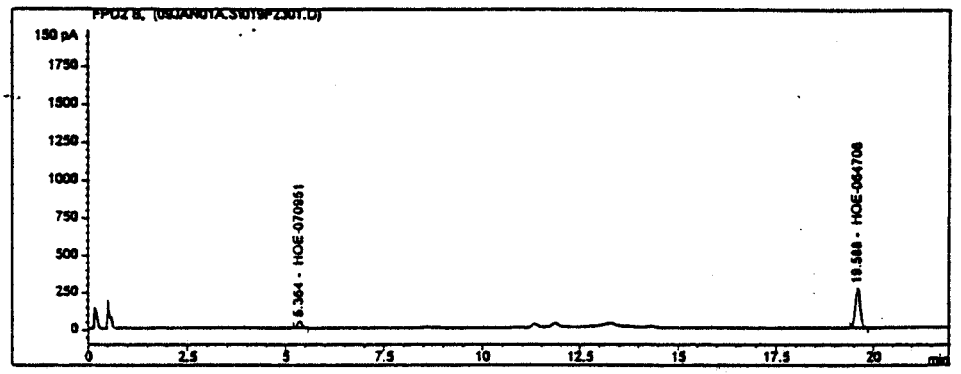

```
================================================================================
                          External Standard Report
================================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, January 11, 2001 7:54:09 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: FPD2 B,

RetTime  Type      Area      Amt/Area    Amount    Grp   Name
 [min]          150 pA*s                [µg/mL]
-------|------|----------|----------|----------|--|------------------
  5.365 BB       407.59619 4.01451e-5 1.63630e-2     HOE-070951
 19.593 BB      4336.39014 5.03790e-5 2.18463e-1     HOE-064706

Totals :                                2.34826e-1

 Results obtained with enhanced integrator!
================================================================================

6890 EC-FPD 1/11/01 7:59:56 AM MYL                          Page 1 of 2
```

Treated Corn Stover Sample 33178302
Analyzed in Set 3S, Curve 1, Injection No. 18

Residues of P + M1 Equivalent to 0.4369 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to 0.0328 µg/g Glufosinate as Uncorrected Free Acid.
Total Residues of Glufosinate Reported as 0.57µg/g Free Acid, Corrected for Recovery

Figure 13g. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\09JAN01A.S\019F2301.D  Sample Name: 33178303
    corn stover  TRT   33178303
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290          Set #  3S

    ================================================================
    Injection Date  : 1/10/01 8:12:26 AM            Seq. Line :  23
    Sample Name     : 33178303                          Vial :  19
    Acq. Operator   : MYL                                Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\09JAN01A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/9/01 4:27:42 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/11/01 7:54:35 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



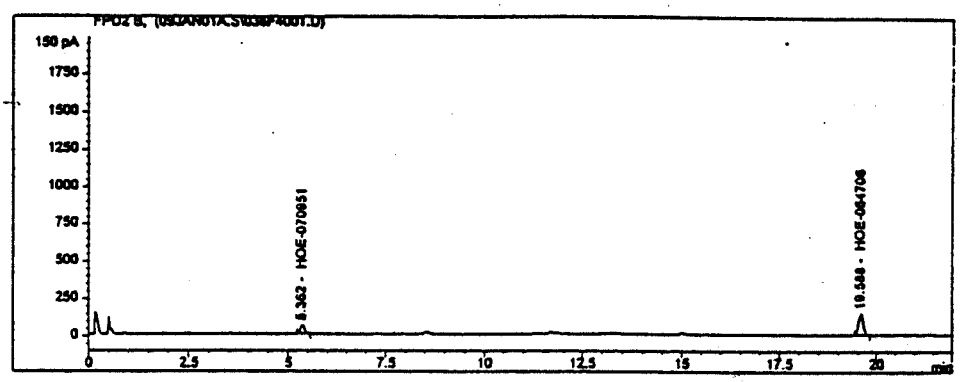

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, January 11, 2001 7:54:09 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount    Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.364 BP      365.53592 4.02475e-5 1.47119e-2    HOE-070951
     19.588 BB     2367.93506 5.07519e-5 1.20177e-1    HOE-064706

    Totals :                             1.34889e-1

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 1/11/01 8:00:06 AM MYL                        Page 1 of 2
```

Treated Corn Stover Sample 33178303
Analyzed in Set 3S, Curve 1, Injection No. 19

Residues of P + M1 Equivalent to 0.2407 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to 0.0295 µg/g Glufosinate as Uncorrected Free Acid
Total Residues of Glufosinate Reported as 0.33 µg/g Free Acid, Corrected for Recovery

Figure 13h. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\09JAN01A.S\036F4001.D  Sample Name: 33065502
    corn stover TRT   33065502
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  3S

    ================================================================
    Injection Date   : 1/10/01 4:48:14 PM              Seq. Line :  40
    Sample Name      : 33065502                            Vial :  36
    Acq. Operator    : MYL                                  Inj :   1
                                                      Inj Volume : 5 µl
    Sequence File    : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\09JAN01A.S
    Acq. Method      : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/9/01 4:27:42 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed     : 1/11/01 8:14:26 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```



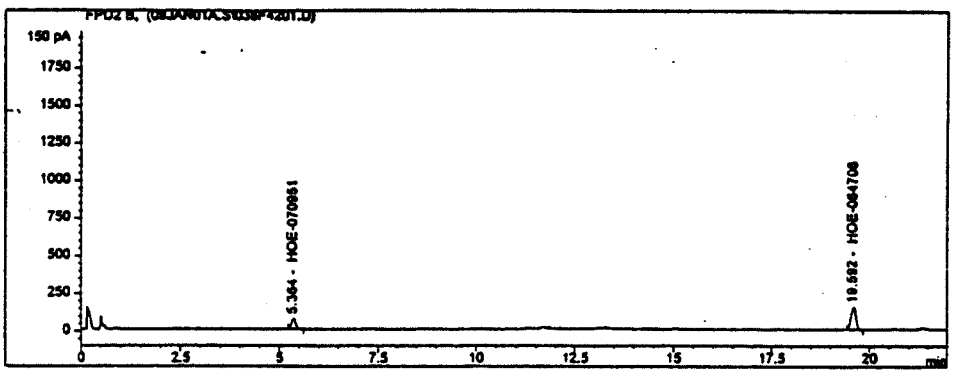

```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, January 11, 2001 8:13:14 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]          150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|------------------
      5.362 BB      446.69067 4.52850e-5 2.02284e-2     HOE-070951
     19.588 BB     1276.16785 5.35636e-5 6.83562e-2     HOE-064706

    Totals :                              8.85845e-2

     Results obtained with enhanced integrator!
    ================================================================

6890 EC-FPD 1/11/01 8:19:38 AM MYL                        Page 1 of 2
```

Treated Corn Stover Sample 33065502
Analyzed in Set 3S, Curve 2, Injection No. 36

Residues of P + M1 Equivalent to 0.1382 µg/g Glufosinate as Uncorrected Free Acid
Residues of Metabolite M2 Equivalent to 0.0415 µg/g Glufosinate as Uncorrected Free Acid.
Total Residues of Glufosinate Reported as 0.21 µg/g Free Acid, Corrected for Recovery

Figure 13i. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover

```
Data File C:\HPCHEM\2\DATA\GLUFOSIN\09JAN01A.S\038F4201.D  Sample Name: 33065503
    corn stover TRT   33065503
    12.5 g sample weight; 500 mL extraction volume; 100 mL
    aliquot; 5.0 mL final volume in methyl acetate
    #000290           Set #  3S

    ================================================================
    Injection Date  : 1/10/01 5:48:50 PM            Seq. Line :  42
    Sample Name     : 33065503                          Vial :  38
    Acq. Operator   : MYL                               Inj :   1
                                                   Inj Volume : 5 µl
    Sequence File   : C:\HPCHEM\2\SEQUENCE\GLUFOSIN\09JAN01A.S
    Acq. Method     : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/9/01 4:27:42 PM by MYL
    Analysis Method : C:\HPCHEM\6890GC\METHODS\GLUFOSIN.M
    Last changed    : 1/11/01 8:14:26 AM by MYL
    GC Determination of Glufosinate-Ammonium and its Metabolites
    ================================================================
```




```
    ================================================================
                          External Standard Report
    ================================================================

    Sorted By             :      Signal
    Calib. Data Modified  :      Thursday, January 11, 2001 8:13:14 AM
    Multiplier            :      1.0000
    Dilution              :      1.0000

    Signal 1: FPD2 B,

    RetTime  Type     Area      Amt/Area    Amount   Grp   Name
     [min]         150 pA*s                [µg/mL]
    -------|------|----------|----------|----------|--|-----------------
      5.364 BB      546.37372 4.40096e-5 2.40457e-2    HOE-070951
     19.592 BP     1331.90112 5.33640e-5 7.10755e-2    HOE-064706

    Totals :                              9.51212e-2

     Results obtained with enhanced integrator!
    ================================================================


6890 EC-FPD 1/11/01 8:19:59 AM MYL                        Page 1 of 2
```

Treated Corn Stover Sample 33065503
Analyzed in Set 3S, Curve 2, Injection No. 38

Residues of P + M1 Equivalent to 0.1436 µg/g Glufosinate as Uncorrected Free Acid
For Residues of Metabolite M2 Equivalent to 0.0491 µg/g Glufosinate as Uncorrected Free Acid.
Total Residues of Glufosinate Reported as 0.22 µg/g Free Acid, Corrected for Recovery

Figure 13j. Typical Chromatograms of Treated Corn Stover from the Analysis for Residues of Glufosinate Ammonium in Corn Stover.

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Laiko
Analytical Set I.D.: 1F curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 23 Oct 00
Injection Date: 24-25 Oct 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 32887101	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32887101 + 0.015 µg/g (P)	11	NA	129.65146	NA	0.00984	2.0	NA	NA	NA	0.0197	NA	NA	NA
Control 32887101 + 0.05 µg/g (P)	13	NA	398.91422	NA	0.0243	2.0	NA	NA	NA	0.0485	NA	NA	NA
Control 32887101 + 0.05 µg/g (P) DUP	14	NA	397.30096	NA	0.0242	2.0	NA	NA	NA	0.0484	NA	NA	NA
Control 32887101 + 1 µg/g (M1 + M2)	16	621.96265	706.12360	0.0261	0.0407	20	0.5223	NA	NA	0.8143	NA	NA	NA
Sample 32887102	18	ND	3418.53394	ND	0.186	2.0	ND	ND	ND	0.3720	0.406	0.41	0.41
Sample 32887103	19	ND	2730.40259	ND	0.149	2.0	ND	ND	ND	0.2983	0.325	0.33	0.33

Recorded By: Melinda Laiko Date: 27 Apr 01

Reviewed By: S.D.L Date: 10 May 01

Figure 14. Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 1F, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 1F curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 23 Oct 00
Injection Date: 24-25 Oct 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
1	0.015	352.42188	270.22275
2	0.020	468.65839	349.46939
3	0.050	1067.36951	779.21362
4	0.100	2318.10986	1680.61792
5	0.200	4891.05127	3619.39087
6	0.300	7153.68799	5626.27832
7	0.015	365.70352	289.63541
10	0.020	489.81329	344.14365
12	0.050	1274.61560	976.28564
15	0.100	2654.89453	1979.17004
17	0.200	4282.68164	3220.15332
20	0.300	7301.33740	5795.36768
	Slope	23809.29980	18670.87399
	Intercept	0.13368	-54.09573
	fit	0.99496	0.99221

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32887101 + 0.015 µg/g (P)	0.015	NA	NA	NA	0.0197	0.0197	NA
Control 32887101 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0485	0.0485	97
Control 32887101 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0484	0.0484	97
Control 32887101 + 1 µg/g (M1 + M2)	1.0	0.5223	0.5223	52	0.8143	0.8143	81
			Average Recovery =	52			92
			Std. Dev. =	NA			9

Recorded By: Melinda Latko Date: 27 Aug 01

Reviewed By: [signature] Date: 10 May 01

Figure 14 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 1F, Curve 1

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1F curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 23 Oct 00
Injection Date: 25 Oct 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 32887101	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32887101 + 0.05 µg/g (M1 + M2)	30	396.63062	466.91818	0.0154	0.0266	2.0	0.0308	NA	NA	0.0531	NA	NA	NA
Control 32887101 + 0.05 µg/g (M1+M2) DUP	31	390.43732	479.56549	0.0151	0.0273	2.0	0.0302	NA	NA	0.0545	NA	NA	NA
Control 32887101 + 1 µg/g (P)	33	NA	866.57849	NA	0.0487	20	NA	NA	NA	0.9737	NA	NA	NA
Sample 33060401	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 33060402	36	544.29163	1.13041e+4	0.0218	0.627	2.0	0.0435	0.071	0.07	ESR	ESR	ESR	#VALUE!
Sample 33060403	38	391.76801	1.10630e+4	0.0152	0.613	2.0	0.0304	0.050	(0.050) *	ESR	ESR	ESR	#VALUE!
Sample 33060403 DUP	39	418.76096	1.20708e+4	0.0163	0.669	2.0	0.0327	0.054	(0.054) *	ESR	ESR	ESR	#VALUE!

ESR = exceeded standard range

"# Value!" is printed under the column of Total Glufosinate acid because no values are reported for M1. M1 values are reported in set 1F (re-inj) at method factor 8.0 (4X dilution).

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Lalko Date: 15 May 01

Reviewed By: SDJ. L Date: 15 May 01

Figure 15. Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 1F, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 1F curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 23 Oct 00
Injection Date: 25 Oct 00

IIA. Calibration Data — Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas P+M1
21	0.015	362.98401	279.24341
22	0.020	513.45709	369.05801
23	0.050	1225.95911	845.28784
24	0.100	2383.50317	1818.59436
25	0.200	4837.70410	3601.52710
26	0.300	7013.99219	5467.58691
27	0.015	349.70993	253.14159
29	0.020	466.13965	356.74670
32	0.050	1278.39526	940.07751
34	0.100	2207.78589	1578.27002
37	0.200	4718.18213	3839.55127
40	0.300	6833.77246	5241.15137
	Slope	23143.03053	18059.75313
	Intercept	40.46943	-12.63547
	fit	0.99874	0.99668

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	P + M1 Found µg/g	P + M1 Control Corrected µg/g	P + M1 Percent Recovery
Control 32887101 + 0.05 µg/g (M1 + M2)	0.050	0.0308	0.0308	62	0.0531	0.0531	106
Control 32887101 + 0.05 µg/g (M1+M2) DUP	0.050	0.0302	0.0302	60	0.0545	0.0545	109
Control 32887101 + 1 µg/g (P)	1.0	NA	NA	NA	0.9737	0.9737	97
			Average Recovery =	61			104
			Std. Dev. =	1			6

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. Date: 10 May 01

Figure 15 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 1F, Curve 2

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1F (re-inj)
Compound: Parent and Metabolites

Analysis Initiation Date: 23 Oct 00
Injection Date: 21-22 Nov 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

1A. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected* µg/g	Reported µg/g	
Control 32887101	8	ND	128.49985	ND	0.0134	2.0	ND	ND	ND	0.0269	0.026	(0.026)	(0.026)
Control 32887101 + 1 µg/g (P)	10	NA	863.43658	NA	0.0551	20	NA	NA	NA	1.1018	NA	NA	NA
Sample 33060402	12	137.57076	2693.42456	0.00855	0.159	8.0	0.0684	#VALUE!	#VALUE!	1.2704	1.222	1.22	#VALUE!
Sample 33060403	14	118.40089	3240.98315	0.00772	0.190	8.0	0.0618	#VALUE!	#VALUE!	1.5187	1.460	1.46	#VALUE!
Sample 33060403 DUP	16	128.37512	3116.19019	0.00815	0.183	8.0	0.0652	#VALUE!	#VALUE!	1.4621	1.406	1.41	#VALUE!

* The average recovery in set 1F curve 2 was used to calculate the corrected values.

"# Value!" is printed under the columns of corrected and reported M2 and Total Glufosinate acid because a control spiked with M2 was not re-injected in this set. Re-injection was needed only for samples in set 1F curve 2 where M1 values exceeded the standard range.

Recorded By: Melinda Lalko Date: 26 Jan 01
Reviewed By: SDD. L Date: 10 May 01

Figure 16. Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 1F, Reinjection Set

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 1F (re-inj)
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 23 Oct 00
Injection Date: 21-22 Nov 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P + M1
1	0.015	333.04620	229.82973
2	0.020	423.25891	261.09802
3	0.050	1101.18237	711.40131
4	0.100	2166.46387	1500.89771
5	0.200	4343.89111	3145.76099
6	0.300	6824.90283	4959.88623
7	0.015	326.59094	243.10457
9	0.020	449.52182	326.57397
11	0.050	1170.13013	792.54407
13	0.100	2163.71851	1561.64136
15	0.200	4627.76611	3565.85840
17	0.300	7179.60889	5571.03369
	Slope	23242.80600	17644.17473
	Intercept	-61.04688	-108.57411
	fit	0.99758	0.99133

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32887101 + 1 µg/g (P)	1.0	NA	NA	NA	1.1018	1.0749	107
			Average Recovery =	NA			107
			Std. Dev. =	NA			NA

Average Recovery in set 1F curve 2 = 104

Recorded By: Melinda Latko Date: 26 Apr 01

Reviewed By: SDD.L Date: 10 May 01

Figure 16 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 1F, Reinjection Set

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Laiko
Analytical Set I.D.: 2F curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 13 Nov 00
Injection Date: 14-15 Nov 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 32892801	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32892801 + 0.015 µg/g (P)	11	NA	138.51863	NA	0.0149	2.0	NA	NA	NA	0.0297	NA	NA	NA
Control 32892801 + 0.05 µg/g (P)	13	NA	370.65085	NA	0.0283	2.0	NA	NA	NA	0.0565	NA	NA	NA
Control 32892801 + 0.05 µg/g (P) DUP	14	NA	422.61789	NA	0.0313	2.0	NA	NA	NA	0.0625	NA	NA	NA
Control 32892801 + 4 µg/g (M1 + M2)	16	1575.70947	1729.19788	0.0707	0.107	40	2.8276	NA	NA	4.2695	NA	NA	NA
Sample 32892802	18	427.91351	743.94611	0.0207	0.0498	2.0	0.0413	0.058	(0.058) *	0.0997	0.087	0.09	0.15
Sample 32892803	19	251.93517	762.47430	0.0130	0.0509	2.0	0.0260	0.037	(0.037)	0.1018	0.089	0.09	0.13

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Laiko Date: 15 May 01

Reviewed By: SDD. C Date: 15 May 01

Figure1 17. Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 2F, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 2F curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 13 Nov 00
Injection Date: 14-15 Nov 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
1	0.015	336.03262	232.51443
2	0.020	358.86996	232.85110
3	0.050	1101.34705	647.69836
4	0.100	2256.36572	1411.69800
5	0.200	3952.90771	2679.12427
6	0.300	7001.01416	4920.65967
7	0.015	344.52338	209.72705
10	0.020	433.92300	280.09457
12	0.050	1215.61487	910.72913
15	0.100	2218.84399	1647.47192
17	0.200	4739.28174	3558.35376
20	0.300	6917.75244	5562.72119
	Slope	22938.99020	17313.77811
	Intercept	-45.82833	-118.85271
	fit	0.99359	0.97879

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32892801 + 0.015 µg/g (P)	0.015	NA	NA	NA	0.0297	0.0297	NA
Control 32892801 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0565	0.0565	113
Control 32892801 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0625	0.0625	125
Control 32892801 + 4 µg/g (M1 + M2)	4.0	2.8276	2.8276	71	4.2695	4.2695	107
			Average Recovery =	71			115
			Std. Dev. =	NA			9

Recorded By: Melinda Lalko Date: 24 Apr 01

Reviewed By: SDD. L Date: 10 May 01

Figure17 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 2F, Curve 1

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 2F curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 13 Nov 00
Injection Date: 15 Nov 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 32892801	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32892801 + 0.05 µg/g (M1 + M2)	30	480.47806	448.68588	0.0210	0.0261	2.0	0.0421	NA	NA	0.0522	NA	NA	NA
Control 32892801 + 0.05 µg/g (M1+M2) DUP	31	509.49094	454.83209	0.0223	0.0264	2.0	0.0446	NA	NA	0.0529	NA	NA	NA
Control 32892801 + 4 µg/g (P)	33	NA	1553.43835	NA	0.0897	40	NA	NA	NA	3.5888	NA	NA	NA
Sample 32904501	35	235.71022	ND	0.0103	ND	2.0	0.0205	0.024	(0.024)	ND	ND	ND	(0.024)
Sample 32904502	36	257.52646	346.79144	0.0112	0.0202	2.0	0.0224	0.026	(0.026)	0.0404	0.040	(0.040)	(0.066) *
Sample 32904503	38	317.82205	277.39673	0.0139	0.0162	2.0	0.0277	0.032	(0.032)	0.0324	0.032	(0.032)	(0.064) *
Sample 32904503 DUP	39	288.09930	213.86168	0.0126	0.0126	2.0	0.0251	0.029	(0.029)	0.0251	0.025	(0.025)	(0.054) *

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Latko Date: 13 Jun 01
Reviewed By: [signature] Date: 13 Jun 01

Figure 18. Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 2F, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 2F curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 13 Nov 00
Injection Date: 15 Nov 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
21	0.015	355.87607	274.93811
22	0.020	437.69873	345.32788
23	0.050	1088.00098	685.22803
24	0.100	2153.81543	1568.41309
25	0.200	4632.75195	3601.13354
26	0.300	6893.35645	5203.91650
27	0.015	322.60080	225.44475
29	0.020	454.06851	333.40973
32	0.050	1139.15271	890.50525
34	0.100	2312.18848	1807.66772
37	0.200	4821.95752	3944.54858
40	0.300	6523.57666	4853.73779
	Slope	22700.01805	17361.49425
	Intercept	3.00163	-4.24801
	fit	0.99709	0.98903

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32892801 + 0.05 µg/g (M1 + M2)	0.050	0.0421	0.0421	84	0.0522	0.0522	104
Control 32892801 + 0.05 µg/g (M1+M2) DUP	0.050	0.0446	0.0446	89	0.0529	0.0529	106
Control 32892801 + 4 µg/g (P)	4.0	NA	NA	NA	3.5888	3.5888	90
			Average Recovery =	87			100
			.Std. Dev. =	4			9

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. Date: 10 May 01

Figure 18 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 2F, Curve 2

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 3F curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 17 Nov 00
Injection Date: 20-21 Nov 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 33063901	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33063901 + 0.015 µg/g (P)	11	NA	121.17823	NA	0.0113	2.0	NA	NA	NA	0.0226	NA	NA	NA
Control 33063901 + 0.05 µg/g (P)	13	NA	372.19745	NA	0.0257	2.0	NA	NA	NA	0.0514	NA	NA	NA
Control 33063901 + 0.05 µg/g (P) DUP	14	NA	339.00708	NA	0.0238	2.0	NA	NA	NA	0.0476	NA	NA	NA
Control 33063901 + 0.5 µg/g (M1 + M2)	16	3829.47583	3057.50659	0.172	0.180	2.0	0.3437	NA	NA	0.3594	NA	NA	NA
Sample 33063902	18	208.62703	1535.17053	0.00997	0.0924	2.0	0.0199	0.029	(0.029)	0.1848	0.205	0.21	0.23
Sample 33063903	19	249.95937	1902.58350	0.0118	0.113	2.0	0.0236	0.034	(0.034)	0.2269	0.252	0.25	0.29

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. L Date: 10 May 01

Figure 19. Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 3F, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 3F curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 17 Nov 00
Injection Date: 20-21 Nov 00

IIA. Calibration Data

Calibration Curve Formula: $Y = mX + b$

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas P+M1
1	0.015	335.93912	240.25816
2	0.020	445.76224	316.26938
3	0.050	1076.65259	724.62750
4	0.100	2149.67529	1516.42749
5	0.200	4381.16846	3266.57056
6	0.300	6194.58594	4647.70752
7	0.015	340.48657	196.10674
10	0.020	425.07733	298.82834
12	0.050	1105.59131	816.30048
15	0.100	2269.26416	1761.71399
17	0.200	4541.83447	3417.24390
20	0.300	7202.99902	5775.74854
	Slope	22364.90898	17438.22775
	Intercept	-14.24073	-76.04745
	fit	0.99190	0.98242

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	P + M1 Found µg/g	P + M1 Control Corrected µg/g	P + M1 Percent Recovery
Control 33063901 + 0.015 µg/g (P)	0.015	NA	NA	NA	0.0226	0.0226	NA
Control 33063901 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0514	0.0514	103
Control 33063901 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0476	0.0476	95
Control 33063901 + 0.5 µg/g (M1 + M2)	0.50	0.3437	0.3437	69	0.3594	0.3594	72
			Average Recovery =	69			90
			Std. Dev. =	NA			16

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD. Date: 10 May 01

Figure 19 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 3F, Curve 1

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Laiko
Analytical Set I.D.: 3F curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 17 Nov 00
Injection Date: 21 Nov 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 33063901	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33063901 + 0.05 µg/g (M1 + M2)	30	427.23486	379.36829	0.0212	0.0252	2.0	0.0423	NA	NA	0.0504	NA	NA	NA
Control 33063901 + 0.05 µg/g (M1+M2) DUP	31	415.93152	369.94287	0.0207	0.0247	2.0	0.0414	NA	NA	0.0493	NA	NA	NA
Control 33063901 + 0.5 µg/g (P)	33	NA	2975.35913	NA	0.171	2.0	NA	NA	NA	0.3416	NA	NA	NA
Sample 33176701	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 33176702	36	395.13004	3341.96509	0.0198	0.191	2.0	0.0396	0.047	(0.047)	0.3827	0.429	0.43	0.48
Sample 33176703	38	325.06122	3463.20679	0.0168	0.198	2.0	0.0336	0.040	(0.040)	0.3963	0.444	0.44	0.48
Sample 33176703 DUP	39	267.70001	2893.56152	0.0144	0.166	2.0	0.0287	0.034	(0.034)	0.3324	0.373	0.37	0.41

Recorded By: Melinda Laiko Date: 27 Apr 01

Reviewed By: [signature] Date: 10 May 01

Figure 20. Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 3F, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 3F curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 17 Nov 00
Injection Date: 21 Nov 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas P+M1
21	0.015	352.47849	250.46956
22	0.020	408.68643	267.19684
23	0.050	1068.95837	751.43854
24	0.100	2218.63623	1635.08899
25	0.200	4711.54590	3523.39380
26	0.300	6908.31641	5125.89600
27	0.015	327.72165	217.86287
29	0.020	438.74921	306.29602
32	0.050	1033.98779	771.84344
34	0.100	2172.51685	1677.57935
37	0.200	4647.65918	3791.19263
40	0.300	7001.88281	5272.08447
	Slope	23443.24529	17829.45716
	Intercept	-68.84223	-69.66782
	fit	0.99941	0.99686

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	P + M1 Found µg/g	P + M1 Control Corrected µg/g	P + M1 Percent Recovery
Control 33063901 + 0.05 µg/g (M1 + M2)	0.050	0.0423	0.0423	85	0.0504	0.0504	101
Control 33063901 + 0.05 µg/g (M1+M2) DUP	0.050	0.0414	0.0414	83	0.0493	0.0493	99
Control 33063901 + 0.5 µg/g (P)	0.50	NA	NA	NA	0.3416	0.3416	68
			Average Recovery =	84			89
			Std. Dev. =	1			18

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: [illegible] Date: 10 May 01

Figure 20 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Forage Set 3F, Curve 2

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1G curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 30 Nov 00
Injection Date: 08-09 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 32889801	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32889801 + 0.015 µg/g (P)	11	NA	139.66031	NA	0.0137	2.0	NA	NA	NA	0.0275	NA	NA	NA
Control 32889801 + 0.05 µg/g (P)	13	NA	401.04263	NA	0.0272	2.0	NA	NA	NA	0.0544	NA	NA	NA
Control 32889801 + 0.05 µg/g (P) DUP	14	NA	457.30057	NA	0.0301	2.0	NA	NA	NA	0.0602	NA	NA	NA
Control 32889801 + 0.2 µg/g (M1 + M2)	16	1818.48572	1713.92505	0.0732	0.0947	2.0	0.1464	NA	NA	0.1895	NA	NA	NA
Sample 32889802	18	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: [signature] Date: 10 May 01

Figure 21. Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 1G, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1G curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 30 Nov 00
Injection Date: 08-09 Dec 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
1	0.015	358.71588	234.65688
2	0.020	495.53845	306.79703
3	0.050	1249.24268	848.99084
4	0.100	2318.51709	1566.65845
5	0.200	4825.16113	3472.77686
6	0.300	7467.15332	5437.93945
7	0.015	346.68015	200.24312
10	0.020	461.46552	302.47797
12	0.050	1226.83130	825.49042
15	0.100	2502.38916	1816.57727
17	0.200	5350.71094	4033.40405
19	0.300	7790.77441	6049.28271
	Slope	25582.02958	19432.23115
	Intercept	-54.51671	-127.23847
	fit	0.99731	0.99151

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32889801 + 0.015 µg/g (P)	0.015	NA	NA	NA	0.0275	0.0275	NA
Control 32889801 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0544	0.0544	109
Control 32889801 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0602	0.0602	120
Control 32889801 + 0.2 µg/g (M1 + M2)	0.20	0.1464	0.1464	73	0.1895	0.1895	95
			Average Recovery =	73			108
			Std. Dev. =	NA			13

Recorded By: Melinda LaKo Date: 27 Apr 01

Reviewed By: SDD.L Date: 10 May 01

Figure 21 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 1G, Curve 1

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1G curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 30 Nov 00
Injection Date: 09 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 32889801	27	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32889801 + 0.05 µg/g (M1 + M2)	29	492.92816	510.00388	0.0195	0.0310	2.0	0.0389	NA	NA	0.0621	NA	NA	NA
Control 32889801 + 0.05 µg/g (M1+M2) DUP	30	499.14178	510.94968	0.0197	0.0311	2.0	0.0394	NA	NA	0.0622	NA	NA	NA
Control 32889801 + 0.2 µg/g (P)	32	NA	1663.67102	NA	0.0939	2.0	NA	NA	NA	0.1879	NA	NA	NA
Sample 32893601	34	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32893602	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32893603	37	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32893603 DUP	38	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND

Recorded By: Melinda Lalko Date: 27 Apr 01
Reviewed By: [signature] Date: 10 May 01

Figure 22. Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 1G, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1G curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 30 Nov 00
Injection Date: 09 Dec 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
20	0.015	346.07266	251.30055
21	0.020	476.12979	312.61224
22	0.050	1161.79321	710.00476
23	0.100	2622.07520	1738.28809
24	0.200	4848.16504	3450.99365
25	0.300	6872.65723	4934.45215
26	0.015	371.44324	259.90274
28	0.020	467.47934	314.40546
31	0.050	1274.05786	871.36151
33	0.100	2435.20581	1783.01562
36	0.200	5103.52051	3901.22363
39	0.300	7670.67090	5889.26758
	Slope	24405.61743	18340.63489
	Intercept	17.79791	-59.15348
	fit	0.99472	0.98656

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32889801 + 0.05 µg/g (M1 + M2)	0.050	0.0389	0.0389	78	0.0621	0.0621	124
Control 32889801 + 0.05 µg/g (M1+M2) DUP	0.050	0.0394	0.0394	79	0.0622	0.0622	124
Control 32889801 + 0.2 µg/g (P)	0.20	NA	NA	NA	0.1879	0.1879	94
			Average Recovery =	78			114
			Std. Dev. =	1			17

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD. C Date: 10 May 01

Figure 22 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 1G, Curve 2

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 2G curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 04 Dec 00
Injection Date: 11-12 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 33061201	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33061201 + 0.015 µg/g (M1 + M2)	11	116.33845	156.75554	0.00573	0.0105	2.0	0.0115	NA	NA	0.0211	NA	NA	NA
Control 33061201 + 0.05 µg/g (P)	13	NA	433.98593	NA	0.0254	2.0	NA	NA	NA	0.0507	NA	NA	NA
Control 33061201 + 0.05 µg/g (P) DUP	14	NA	449.50815	NA	0.0262	2.0	NA	NA	NA	0.0524	NA	NA	NA
Control 33061201 + 0.1 µg/g (M1 + M2)	16	907.12982	1020.33759	0.0389	0.0567	2.0	0.0779	NA	NA	0.1134	NA	NA	NA
Sample 33061202	18	222.04556	243.58322	0.0102	0.0152	2.0	0.0203	0.026	(0.026)	0.0304	0.029	(0.029)	(0.055) *
Sample 33061203	19	168.29133	240.85057	0.00791	0.0150	2.0	0.0158	0.020	(0.020)	0.0301	0.028	(0.028)	(0.048) *

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Latko Date: 15 May 01
Reviewed By: SDD. Date: 17 May 01

Figure 23. Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 2G, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 2G curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 04 Dec 00
Injection Date: 11-12 Dec 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
1	0.015	338.04492	274.65076
2	0.020	452.19110	333.26349
3	0.050	1123.06641	883.85962
4	0.100	2320.93652	1699.92139
5	0.200	4861.52490	3713.47363
6	0.300	6924.33447	5347.92627
7	0.015	337.03183	241.17935
10	0.020	455.56165	337.92523
12	0.050	1239.80505	977.31006
15	0.100	2453.40161	1931.92920
17	0.200	4477.04736	3451.84619
20	0.300	7400.49072	5946.54834
	Slope	23812.93829	18704.93643
	Intercept	-20.02408	-40.49412
	fit	0.99705	0.99369

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 33061201 + 0.015 µg/g (M1 + M2)	0.015	0.0115	0.0115	NA	0.0211	0.0211	NA
Control 33061201 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0507	0.0507	101
Control 33061201 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0524	0.0524	105
Control 33061201 + 0.1 µg/g (M1 + M2)	0.10	0.0779	0.0779	78	0.1134	0.1134	113
			Average Recovery =	78			107
			Std. Dev. =	NA			6

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDL Date: 10 May 01

Figure 23 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 2G, Curve 1

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 2G curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 04 Dec 00
Injection Date: 12 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 33061201	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33061201 + 0.05 µg/g (M1 + M2)	30	438.89932	472.92410	0.0193	0.0281	2.0	0.0386	NA	NA	0.0562	NA	NA	NA
Control 33061201 + 0.05 µg/g (M1+M2) DUP	31	455.22076	487.01196	0.0200	0.0288	2.0	0.0399	NA	NA	0.0576	NA	NA	NA
Control 33061201 + 0.1 µg/g (P)	33	NA	669.06946	NA	0.0380	2.0	NA	NA	NA	0.0760	NA	NA	NA
Sample 32905301	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32905302	36	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32905303	38	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32905303 DUP	39	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32889803	40	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND

Recorded By: Melinda Lalko Date: 05-Jan 01
Reviewed By: Jonathan Gunter Date: 23 Jan 01

Figure 24. Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 2G, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 2G curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 04 Dec 00
Injection Date: 12 Dec 00

IIA. Calibration Data — Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
21	0.015	342.94711	271.71729
22	0.020	461.02414	342.06750
23	0.050	1160.56458	835.06104
24	0.100	2423.14673	1841.23035
25	0.200	4863.53711	3726.24487
26	0.300	7472.03662	5806.02588
27	0.015	335.20367	264.39963
29	0.020	464.95218	329.31311
32	0.050	1244.38635	943.11688
34	0.100	2496.45776	1902.60938
37	0.200	4844.04736	3791.15283
41	0.300	7442.56006	6093.77734
	Slope	24848.76770	19817.20803
	Intercept	-40.99567	-83.57158
	fit	0.99972	0.99794

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 33061201 + 0.05 µg/g (M1 + M2)	0.050	0.0386	0.0386	77	0.0562	0.0562	112
Control 33061201 + 0.05 µg/g (M1+M2) DUP	0.050	0.0399	0.0399	80	0.0576	0.0576	115
Control 33061201 + 0.1 µg/g (P)	0.10	NA	NA	NA	0.0760	0.0760	76
			Average Recovery =	79			101
			Std. Dev. =	2			22

Recorded By: Melinda Lalko Date: 05 Jan 01

Reviewed By: Samantha L Ginter Date: 23 Jan 01

Figure 24 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 2G, Curve 2

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 3G curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 11 Dec 00
Injection Date: 12-13 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 33064701	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33064701 + 0.015 µg/g (P)	11	NA	149.31908	NA	0.00910	2.0	NA	NA	NA	0.0182	NA	NA	NA
Control 33064701 + 0.05 µg/g (P)	13	NA	415.43991	NA	0.0231	2.0	NA	NA	NA	0.0462	NA	NA	NA
Control 33064701 + 0.05 µg/g (P) DUP	14	NA	398.58221	NA	0.0222	2.0	NA	NA	NA	0.0444	NA	NA	NA
Control 33064701 + 0.08 µg/g (M1 + M2)	16	784.88281	759.13043	0.0346	0.0412	2.0	0.0693	NA	NA	0.0823	NA	NA	NA
Sample 33064702	18	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 33064703	19	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND

Recorded By: Melinda Lalko Date: 15 Dec 00

Reviewed By: Samantha L. Sauter Date: 23 Jan 01

Figure 25. Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 3G, Curve 1

Method: Aventis CropSciences RAM DK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 3G curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 11 Dec 00
Injection Date: 12-13 Dec 00

IIA. Calibration Data — Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas P+M1
1	0.015	350.02005	337.71622
2	0.020	443.70001	373.48779
3	0.050	1176.83118	942.72101
4	0.100	2228.09644	1802.06592
5	0.200	4810.93506	3761.10815
6	0.300	7099.26367	5617.38672
7	0.015	346.68497	276.67990
10	0.020	418.55884	325.33752
12	0.050	1138.03528	857.15204
15	0.100	2306.76099	1872.38049
17	0.200	4810.00391	3880.79150
20	0.300	7133.01514	5727.27686
	Slope	23933.47312	19022.13870
	Intercept	-43.91272	-23.85216
	fit	0.99959	0.99926

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	P + M1 Found µg/g	P + M1 Control Corrected µg/g	P + M1 Percent Recovery
Control 33064701 + 0.015 µg/g (P)	0.015	NA	NA	NA	0.0182	0.0182	NA
Control 33064701 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0462	0.0462	92
Control 33064701 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0444	0.0444	89
Control 33064701 + 0.08 µg/g (M1 + M2)	0.080	0.0693	0.0693	87	0.0823	0.0823	103
			Average Recovery =	87			95
			Std. Dev. =	NA			7

Recorded By: Melinda Latko Date: 23 Jan 01

Reviewed By: Samantha L. Gaster Date: 23 Jan 01

Figure 25 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 3G, Curve 1

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 3G curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 11 Dec 00
Injection Date: 13 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 33064701	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33064701 + 0.05 µg/g (M1 + M2)	30	426.75156	457.08905	0.0194	0.0269	2.0	0.0388	NA	NA	0.0537	NA	NA	NA
Control 33064701 + 0.05 µg/g (M1+M2) DUP	31	363.82111	370.49939	0.0167	0.0222	2.0	0.0334	NA	NA	0.0444	NA	NA	NA
Control 33064701 + 0.08 µg/g (P)	33	NA	697.80023	NA	0.0398	2.0	NA	NA	NA	0.0795	NA	NA	NA
Sample 33177501	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 33177502	36	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 33177503	38	ND	103.59633	ND	0.00792	2.0	ND	ND	ND	0.0158	0.016	ND *	ND *
Sample 33177503 DUP	39	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Latko Date: 04 Jun 01

Reviewed By: [signature] Date: 13 Jun 01

Figure 26. Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 3G, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 3G curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 11 Dec 00
Injection Date: 13 Dec 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas P+M1
21	0.015	347.50888	269.94949
22	0.020	440.29193	323.61462
23	0.050	1105.12073	853.05109
24	0.100	2277.71436	1744.01025
25	0.200	4587.97607	3534.26953
26	0.300	6864.06641	5327.59570
27	0.015	334.69171	227.74142
29	0.020	418.85114	339.91260
32	0.050	1149.49963	876.37250
34	0.100	2330.67480	1895.90027
37	0.200	4697.48730	3884.25098
40	0.300	7043.74023	5763.22314
	Slope	23282.39643	18664.47805
	Intercept	-24.93833	-44.20361
	fit	0.99963	0.99622

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	P + M1 Found µg/g	P + M1 Control Corrected µg/g	P + M1 Percent Recovery
Control 33064701 + 0.05 µg/g (M1 + M2)	0.050	0.0388	0.0388	78	0.0537	0.0537	107
Control 33064701 + 0.05 µg/g (M1+M2) DUP	0.050	0.0334	0.0334	67	0.0444	0.0444	89
Control 33064701 + 0.08 µg/g (P)	0.080	NA	NA	NA	0.0795	0.0795	99
			Average Recovery =	72			99
			Std. Dev. =	8			9

Recorded By: Melinda Lalko Date: 17 Jan 01

Reviewed By: Samantha L. Gunter Date: 23 Jan 01

Figure 26 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Grain Set 3G, Curve 2

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Laiko
Analytical Set I.D.: 1S curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 28 Dec 00
Injection Date: 29-30 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

1A. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 32890101	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32890101 + 0.015 µg/g (P)	11	NA	140.88049	NA	0.00844	2.0	NA	NA	NA	0.0169	NA	NA	NA
Control 32890101 + 0.05 µg/g (P)	13	NA	402.30057	NA	0.0221	2.0	NA	NA	NA	0.0442	NA	NA	NA
Control 32890101 + 0.05 µg/g (P) DUP	14	NA	410.94708	NA	0.0225	2.0	NA	NA	NA	0.0451	NA	NA	NA
Control 32890101 + 6 µg/g (M1 + M2)	16	2716.71533	2809.65210	0.123	0.148	40	4.9382	NA	NA	5.9108	NA	NA	NA
Sample 32890102	18	ND	353.56769	ND	0.0195	2.0	ND	ND	ND	0.0391	0.042	(0.042)	(0.042)
Sample 32890103	19	ND	817.22162	ND	0.0438	2.0	ND	ND	ND	0.0875	0.095	0.09	0.09

Recorded By: *Melinda Laiko* Date: 26 Apr 01

Reviewed By: *[signature]* Date: 30 Apr 01

Figure 27. Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 1S, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1S curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 28 Dec 00
Injection Date: 29-30 Dec 00

IIA. Calibration Data

Calibration Curve Formula: $Y = mX + b$

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
1	0.015	319.42435	311.54056
2	0.020	424.15204	385.08557
3	0.050	1107.54321	903.99261
4	0.100	2296.19653	1896.60291
5	0.200	4675.42529	3925.53662
6	0.300	6337.64111	5562.24805
7	0.015	308.65936	261.64352
10	0.020	415.03482	337.14828
12	0.050	1070.38159	873.83118
15	0.100	2120.56812	1875.09839
17	0.200	4519.95215	3862.04346
20	0.300	6564.93213	5797.39014
	Slope	21898.73836	19154.65875
	Intercept	13.21993	-20.81010
	fit	0.99717	0.99882

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32890101 + 0.015 µg/g (P)	0.015	NA	NA	NA	0.0169	0.0169	NA
Control 32890101 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0442	0.0442	88
Control 32890101 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0451	0.0451	90
Control 32890101 + 6 µg/g (M1 + M2)	6.000	4.9382	4.9382	82	5.9108	5.9108	99
			Average Recovery =	82			92
			Std. Dev. =	NA			5

Recorded By: Melinda Lalko Date: 26 Apr 01

Reviewed By: Kate Koltavy Date: 30APR01

Figure 27 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 1S, Curve 1

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 1S curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 28 Dec 00
Injection Date: 30 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 32890101	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32890101 + 0.05 µg/g (M1 + M2)	30	426.83585	451.86087	0.0199	0.0267	2.0	0.0399	NA	NA	0.0534	NA	NA	NA
Control 32890101 + 0.05 µg/g (M1+M2) DUP	31	319.82153	389.86514	0.0150	0.0235	2.0	0.0300	NA	NA	0.0470	NA	NA	NA
Control 32890101 + 6 µg/g (P)	33	NA	2299.33008	NA	0.121	40	NA	NA	NA	4.8412	NA	NA	NA
Sample 32894401	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 32894402	36	107.26668	1062.07422	0.00528	0.0578	2.0	0.0106	0.015	ND *	0.1157	0.123	0.12	0.12
Sample 32894403	38	156.51620	1155.40369	0.00754	0.0626	2.0	0.0151	0.022	(0.022)	0.1252	0.133	0.13	0.16
Sample 32894403 DUP	39	162.25996	1248.69275	0.00780	0.0674	2.0	0.0156	0.022	(0.022)	0.1348	0.144	0.14	0.17

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Latko Date: 15 May 01

Reviewed By: SDD. C Date: 17 May 01

Figure 28. Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 1S, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 1S curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 28 Dec 00
Injection Date: 30 Dec 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
21	0.015	339.02551	287.34760
22	0.020	415.62262	325.60617
23	0.050	1018.96686	827.30133
24	0.100	2145.06055	1822.26819
25	0.200	4087.29712	3561.18408
26	0.300	6556.25244	5703.63428
27	0.015	350.82785	281.71301
29	0.020	389.50424	332.02646
32	0.050	1073.48950	891.75574
34	0.100	2259.04517	1881.19214
37	0.200	4697.79150	4123.32568
40	0.300	6467.71045	5942.04248
	Slope	21822.39285	19582.39845
	Intercept	-8.00703	-70.70739
	fit	0.99662	0.99588

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32890101 + 0.05 µg/g (M1 + M2)	0.050	0.0399	0.0399	80	0.0534	0.0534	107
Control 32890101 + 0.05 µg/g (M1+M2) DUP	0.050	0.0300	0.0300	60	0.0470	0.0470	94
Control 32890101 + 6 µg/g (P)	6.0	NA	NA	NA	4.8412	4.8412	81
			Average Recovery =	70			94
			Std. Dev. =	14			13

Recorded By: Melinda Lalko Date: 26 Apr 01

Reviewed By: Kate Hokamp Date: 30 APR 01

Figure 28 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 1S, Curve 2

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 2S curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 03 Jan 01
Injection Date: 04-05 Jan 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 32906101	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32906101 + 0.015 µg/g (P)	11	NA	118.54986	NA	0.0107	2.0	NA	NA	NA	0.0215	NA	NA	NA
Control 32906101 + 0.05 µg/g (P)	13	NA	409.32986	NA	0.0256	2.0	NA	NA	NA	0.0512	NA	NA	NA
Control 32906101 + 0.05 µg/g (P) DUP	14	NA	416.06781	NA	0.0259	2.0	NA	NA	NA	0.0519	NA	NA	NA
Control 32906101 + 1 µg/g (M1 + M2)	16	2307.63818	2370.88770	0.100	0.126	8.0	0.8018	NA	NA	1.0063	NA	NA	NA
Sample 32906102	18	ND	506.90616	ND	0.0306	2.0	ND	ND	ND	0.0611	0.060	0.06	0.06
Sample 32906103	19	ND	600.45319	ND	0.0354	2.0	ND	ND	ND	0.0707	0.069	0.07	0.07

Recorded By: Melinda Lalko Date: 26 Apr 01

Reviewed By: Kati Kolstad Date: 27 APR 01

Figure 29. Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 2S, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 2S curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 03 Jan 01
Injection Date: 04-05 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
1	0.015	343.59900	250.59619
2	0.020	432.45258	298.51373
3	0.050	1080.87695	769.92505
4	0.100	2258.57495	1756.02490
5	0.200	4388.67578	3346.24121
6	0.300	7161.82617	5760.76416
7	0.015	349.23615	244.43176
10	0.020	460.46976	348.17685
12	0.050	1152.10657	940.22021
15	0.100	2392.34668	1973.07239
17	0.200	4682.60156	4077.25317
20	0.300	6889.80518	5955.64355
	Slope	23320.08440	19577.16860
	Intercept	-29.66203	-91.65482
	fit	0.99827	0.99263

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32906101 + 0.015 µg/g (P)	0.015	NA	NA	NA	0.0215	0.0215	NA
Control 32906101 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0512	0.0512	102
Control 32906101 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0519	0.0519	104
Control 32906101 + 1 µg/g (M1 + M2)	1.0	0.8018	0.8018	80	1.0063	1.0063	101
			Average Recovery =	80			102
			Std. Dev. =	NA			2

Recorded By: Melinda LaRo Date: 26 Apr 01

Reviewed By: Kate Kolberg Date: 27APR01

Figure 29 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 2S, Curve 1

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 2S curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 03 Jan 01
Injection Date: 05 Jan 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 32906101	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 32906101 + 0.05 µg/g (M1 + M2)	30	462.55539	464.21286	0.0230	0.0280	2.0	0.0459	NA	NA	0.0560	NA	NA	NA
Control 32906101 + 0.05 µg/g (M1+M2) DUP	31	440.94138	467.39914	0.0221	0.0281	2.0	0.0442	NA	NA	0.0563	NA	NA	NA
Control 32906101 + 1 µg/g (P)	33	NA	2063.67603	NA	0.105	8.0	NA	NA	NA	0.8386	NA	NA	NA
Sample 33062001	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 33062002	36	2148.64648	2.34441e+4	0.0908	1.13	2	0.1816	0.202	0.20	ESR	ESR	ESR	#VALUE!
Sample 33062003	38	1569.73962	1.57270e+4	0.0675	0.761	2	0.1350	0.150	0.15	ESR	ESR	ESR	#VALUE!
Sample 33062003 DUP	39	1539.53918	1.60518e+4	0.0663	0.777	2	0.1326	0.147	0.15	ESR	ESR	ESR	#VALUE!

ESR = exceeded standard range

"# Value!" is printed under the column of Total Glufosinate acid because no values are reported for M1. M1 values are reported in set 2S (reinj) at method factor of 40 (20X dilution) for sample 33062002 and 20 (10X dilution) for sample 33062003.

Recorded By: Melinda Latko Date: 26 Apr 01

Reviewed By: Kate Ichlang Date: 27 Apr 01

Figure 30. Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 2S, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 2S curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 03 Jan 01
Injection Date: 05 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
21	0.015	360.83362	288.05698
22	0.020	445.10388	351.74054
23	0.050	1051.61938	800.34344
24	0.100	2357.07886	1921.72229
25	0.200	4776.65771	3811.11572
26	0.300	7071.47168	5802.17969
27	0.015	352.58325	259.37625
29	0.020	450.57965	374.74762
32	0.050	1181.01355	949.69031
34	0.100	2113.35889	1750.68359
37	0.200	4758.05078	4200.89844
40	0.300	7840.55127	6585.80957
	Slope	24860.00301	20815.98003
	Intercept	-108.27513	-118.46068
	fit	0.99454	0.99155

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32906101 + 0.05 µg/g (M1 + M2)	0.050	0.0459	0.0459	92	0.0560	0.0560	112
Control 32906101 + 0.05 µg/g (M1+M2) DUP	0.050	0.0442	0.0442	88	0.0563	0.0563	113
Control 32906101 + 1 µg/g (P)	1.0	NA	NA	NA	0.8386	0.8386	84
			Average Recovery =	90			103
			Std. Dev. =	2			16

Recorded By: Melinda Latko Date: 26 Apr 01

Reviewed By: Kat Mahany Date: 27 Apr 01

Figure 30 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 2S, Curve 2

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 2S (reinj)
Compound: Parent and Metabolites

Analysis Initiation Date: 03 Jan 01
Injection Date: 05-06 Jan 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected ** µg/g	Reported µg/g	
Control 32906101	8	ND	223.72673	ND	0.0145	2.0	ND	ND	ND	0.0290	0.028	(0.028)	(0.028)
Control 32906101 + 1 µg/g (M1 + M2)	10	2484.77295	2599.68237	0.105	0.137	8.0	0.8437	NA	NA	1.0937	NA	NA	NA
Sample 33062002	12	112.33689	1136.13892	0.00617	0.0614	40	- *	#VALUE!	#VALUE!	2.4570	2.409	2.41	#VALUE!
Sample 33062003	14	161.18622	1624.57727	0.00822	0.0865	20	- *	#VALUE!	#VALUE!	1.7310	1.697	1.70	#VALUE!
Sample 33062003 DUP	16	161.05275	1541.40686	0.00821	0.0823	20	- *	#VALUE!	#VALUE!	1.6454	1.613	1.61	#VALUE!

* M2 values are reported in set 2S curve 2 at method factor 2 (no dilution).

"# Value!" is printed under the columns of corrected and reported M2 and Total Glufosinate acid because there are no M2 values reported for these samples. Re-injection was needed only for samples in set 2S curve 2 where M1 values exceeded the standard range.

** The average recovery in set 2S curve 1 was used to calculate the corrected values.

Recorded By: Melinda Lalko Date: 26 Apr 01

Reviewed By: Kat Worley Date: 30 APR 01

Figure 31. Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 2S, Reinjection Set

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 2S (reinj)
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 03 Jan 01
Injection Date: 05-06 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P + M1
1	0.015	336.26212	238.89143
2	0.020	425.97623	300.14630
3	0.050	1137.65930	794.61053
4	0.100	2378.71436	1774.15002
5	0.200	4754.23584	3624.35571
6	0.300	6774.15283	5533.68799
7	0.015	354.80261	362.78976
9	0.020	456.12976	380.96301
11	0.050	1185.01428	977.04907
13	0.100	2219.90259	1906.08215
15	0.200	4827.63818	3922.57764
17	0.300	7463.78809	6122.38477
	Slope	23894.85152	19440.92941
	Intercept	-35.13920	-58.03208
	fit	0.99641	0.99430

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 32906101 + 1 µg/g (M1 + M2)	1.0	0.8437	0.8437	84	1.0937	1.0647	106
			Average Recovery =	84			106
			Std. Dev. =	NA			NA

*Average Recovery in set 2S curve 1 = 102

* The recovery value was originally 101% of expected (see set 2S curve 1). Reanalysis was 106% of expected indicating no degradation of the samples occurred. Because of this, the samples will be corrected by the average recovery of the original run from 2S curve 1 (set recovery was from).

Recorded By: Melinda Latko Date: 26 Apr 01

Reviewed By: Kati Hartung Date: 30APR01

Figure 31 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 2S, Reinjection Set

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 3S curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 08 Jan 01
Injection Date: 09-10 Jan 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 33178301	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33178301 + 0.015 µg/g (P)	11	NA	ND	NA	ND	2.0	NA	NA	NA	ND	NA	NA	NA
Control 33178301 + 0.05 µg/g (P)	13	NA	396.14438	NA	0.0220	2.0	NA	NA	NA	0.0441	NA	NA	NA
Control 33178301 + 0.05 µg/g (P) DUP	14	NA	375.40524	NA	0.0210	2.0	NA	NA	NA	0.0420	NA	NA	NA
Control 33178301 + 4 µg/g (M1 + M2)	16	2955.64526	3312.37646	0.116	0.167	20	2.3283	NA	NA	3.3481	NA	NA	NA
Sample 33178302	18	407.59619	4336.39014	0.0164	0.218	2.0	0.0328	0.056	(0.056) *	0.4369	0.512	0.51	0.57
Sample 33178303	19	365.53592	2367.93506	0.0148	0.120	2.0	0.0295	0.051	(0.051) *	0.2407	0.282	0.28	0.33

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Lalko Date: 15 May 01
Reviewed By: SDD.C Date: 17 May 01

Figure 32. Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 3S, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 3S curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 08 Jan 01
Injection Date: 09-10 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
1	0.015	358.51065	264.46719
2	0.020	472.67645	337.76025
3	0.050	1286.49976	939.52380
4	0.100	2585.81958	2040.33521
5	0.200	5131.73828	4066.49658
6	0.300	7689.88184	6048.88672
7	0.015	362.96826	267.19208
10	0.020	459.38300	333.10593
12	0.050	1278.67773	921.72961
15	0.100	2484.21777	1918.76392
17	0.200	5194.67139	3967.75195
20	0.300	7474.60986	5825.74170
	Slope	25482.40700	20062.08335
	Intercept	-10.93692	-46.10827
	fit	0.99940	0.99908

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 33178301 + 0.015 µg/g (P)	0.015	NA	NA	NA	ND	ND	NA
Control 33178301 + 0.05 µg/g (P)	0.050	NA	NA	NA	0.0441	0.0441	88
Control 33178301 + 0.05 µg/g (P) DUP	0.050	NA	NA	NA	0.0420	0.0420	84
Control 33178301 + 4 µg/g (M1 + M2)	4.0	2.3283	2.3283	58	3.3481	3.3481	84
			Average Recovery =	58			85
			Std. Dev. =	NA			2

Recorded By: Melinda Latko Date: 26 Jan 01

Reviewed By: Kat Hartwig Date: 30 JAN 01

Figure 32 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 3S, Curve 1

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: 3S curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 08 Jan 01
Injection Date: 10 Jan 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P + M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 33178301	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 33178301 + 0.05 µg/g (M1 + M2)	30	355.56882	389.87732	0.0173	0.0261	2.0	0.0345	NA	NA	0.0522	NA	NA	NA
Control 33178301 + 0.05 µg/g (M1+M2) DUP	31	370.09741	375.30139	0.0178	0.0254	2.0	0.0356	NA	NA	0.0508	NA	NA	NA
Control 33178301 + 4 µg/g (P)	33	NA	3069.88135	NA	0.156	20	NA	NA	NA	3.1232	NA	NA	NA
Sample 33065501	35	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Sample 33065502	36	446.69067	1276.16785	0.0207	0.0691	2.0	0.0415	0.059	(0.059) *	0.1382	0.146	0.15	0.21
Sample 33065503	38	546.37372	1331.90112	0.0246	0.0718	2.0	0.0491	0.070	0.07	0.1436	0.152	0.15	0.22
Sample 33065503 DUP	39	485.19760	1291.52673	0.0222	0.0699	2.0	0.0444	0.063	0.06	0.1397	0.148	0.15	0.21

* Data is reported according to calculated LOD/LOQ in the final report rather than the target LOD/LOQ indicated at the top of the page.

Recorded By: Melinda Lalko Date: 04 Jan 01
Reviewed By: SDD. L Date: 13 Jan 01

Figure 33. Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 3S, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: 3S curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 08 Jan 01
Injection Date: 10 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P+M1
21	0.015	369.49429	270.90073
22	0.020	470.19397	306.87024
23	0.050	1170.49573	795.79346
24	0.100	2365.70996	1693.09595
25	0.200	5016.05859	3698.78638
26	0.300	7706.64258	5916.38623
27	0.015	342.12247	238.34424
29	0.020	481.88474	382.93506
32	0.050	1237.45642	873.82483
34	0.100	2501.87842	1870.91455
37	0.200	5048.40723	4026.62036
40	0.300	8004.94385	6381.40039
	Slope	26185.06608	20604.75177
	Intercept	-96.52102	-147.71979
	fit	0.99858	0.99451

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P + M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 33178301 + 0.05 µg/g (M1 + M2)	0.050	0.0345	0.0345	69	0.0522	0.0522	104
Control 33178301 + 0.05 µg/g (M1+M2) DUP	0.050	0.0356	0.0356	71	0.0508	0.0508	102
Control 33178301 + 4 µg/g (P)	4.0	NA	NA	NA	3.1232	3.1232	78
			Average Recovery =	70			95
			Std. Dev. =	2			14

Recorded By: Melinda Latko Date: 26Apr01

Reviewed By: Kath Kohlberg Date: 30APR01

Figure 33 (Cont). Summary Spreadsheets of Glufosinate Residue Data for Corn Stover Set 3S, Curve 2

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: Day0 FSS-Forage curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 27 Nov 00
Injection Date: 07-08 Dec 00

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 34240801	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34240801 + 1 µg/g (P)	11	NA	797.92621	NA	0.0503	20	NA	NA	NA	1.0069	NA	NA	NA
Control 34240801 + 1 µg/g (P) DUP	12	NA	730.72253	NA	0.0467	20	NA	NA	NA	0.9332	NA	NA	NA
Sample 34240802	14	NA	829.88025	NA	0.0521	20	NA	NA	NA	1.0420	1.074	1.07	1.07
Sample 34240802 DUP	16	NA	700.71033	NA	0.0450	20	NA	NA	NA	0.9002	0.928	0.93	0.93
Sample 34240802 TRIP	18	NA	844.44794	NA	0.0529	20	NA	NA	NA	1.0579	1.091	1.09	1.09

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD.L Date: 03 May 01

Figure 34. Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Forage Day 0, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: Day0 FSS-Forage curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 27 Nov 00
Injection Date: 07-08 Dec 00

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P
1	0.015	373.61243	206.92241
2	0.020	475.58289	280.53497
3	0.050	1234.53845	695.44366
4	0.100	2530.35767	1604.53564
5	0.200	4989.60205	3272.05176
6	0.300	7766.05713	5344.62598
7	0.015	343.71072	204.92004
10	0.020	482.99063	314.54169
13	0.050	1246.55664	805.65204
15	0.100	2484.56616	1707.60901
17	0.200	5060.83301	3579.72681
19	0.300	7762.30615	5519.47900
	Slope	25852.26579	18229.15642
	Intercept	-55.57418	-119.82511
	fit	0.99964	0.99706

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34240801 + 1 µg/g (P)	1.0	NA	NA	NA	1.0069	1.0069	101
Control 34240801 + 1 µg/g (P) DUP	1.0	NA	NA	NA	0.9332	0.9332	93
			Average Recovery =	NA			97
			Std. Dev. =	NA			5

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD.L Date: 03 May 01

Figure 34 (Cont). Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Forage Day 0, Curve 1

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: Day0 FSS-Forage curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 27 Nov 00
Injection Date: 08 Dec 00

	M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	M1	M2	M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 34240801	27	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34240801 + 1 µg/g (M1 + M2)	29	719.05353	949.53693	0.0293	0.0551	20	0.5854	NA	NA	1.1011	NA	NA	NA
Control 34240801 + 1 µg/g (M1+M2) DUP	30	744.19525	842.61798	0.0303	0.0492	20	0.6055	NA	NA	0.9840	NA	NA	NA
Sample 34240803	32	731.42627	866.70575	0.0298	0.0505	20	0.5953	1.000	1.00	1.0104	0.969	0.97	1.97
Sample 34240803 DUP	34	944.83618	1061.27087	0.0383	0.0612	20	0.7667	1.288	1.29	1.2234	1.173	1.17	2.46
Sample 34240803 TRIP	36	902.66467	1060.39978	0.0366	0.0611	20	0.7328	1.231	1.23	1.2224	1.173	1.17	2.40

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD.L Date: 03 May 01

Figure 35. Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Forage Day 0, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: Day0 FSS-Forage curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 27 Nov 00
Injection Date: 08 Dec 00

IIA. Calibration Data — Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas M1
20	0.015	340.05624	206.83403
21	0.020	458.03235	261.80316
22	0.050	1234.05273	766.91394
23	0.100	2515.50952	1624.92578
24	0.200	4975.27246	3314.97314
25	0.300	7314.67139	4862.91699
26	0.015	343.50037	186.65099
28	0.020	456.81577	315.04874
31	0.050	1213.65271	970.30157
33	0.100	2542.41797	2013.01062
35	0.200	5126.78467	3865.81519
37	0.300	7476.98096	5965.41943
	Slope	24901.29772	18270.46226
	Intercept	-9.75256	-56.32664
	fit	0.99932	0.98045

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	M1 Found µg/g	M1 Control Corrected µg/g	M1 Percent Recovery
Control 34240801 + 1 µg/g (M1 + M2)	1.0	0.5854	0.5854	59	1.1011	1.1011	110
Control 34240801 + 1 µg/g (M1+M2) DUP	1.0	0.6055	0.6055	61	0.9840	0.9840	98
			Average Recovery =	60			104
			Std. Dev. =	1			8

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD.C Date: 03 May 01

Figure 35 (Cont). Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Forage Day 0, Curve 2

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: DayX FSS-Forage curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 12 Feb 01
Injection Date: 13-14 Feb 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 34240801	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34240801 + 1 µg/g (P)	11	NA	837.76990	NA	0.0507	20	NA	NA	NA	1.0133	NA	NA	NA
Control 34240801 + 1 µg/g (P) DUP	12	NA	783.36896	NA	0.0478	20	NA	NA	NA	0.9563	NA	NA	NA
Sample 34240802	14	NA	765.06201	NA	0.0469	20	NA	NA	NA	0.9371	0.952	0.95	0.95
Sample 34240802 DUP	16	NA	769.25775	NA	0.0471	20	NA	NA	NA	0.9415	0.956	0.96	0.96
Sample 34240802 TRIP	18	NA	858.84601	NA	0.0518	20	NA	NA	NA	1.0354	1.051	1.05	1.05

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD.C Date: 03 May 01

Figure 36. Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Forage Day 77, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: DayX FSS-Forage curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 12 Feb 01
Injection Date: 13-14 Feb 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P
1	0.015	366.01172	235.87779
2	0.020	469.24194	263.83859
3	0.050	1172.25525	750.06519
4	0.100	2459.19922	1606.69263
5	0.200	4877.53906	3642.40625
6	0.300	7426.85449	5445.15137
7	0.015	382.16196	242.58398
10	0.020	448.91293	262.60730
13	0.050	1182.20459	843.32159
15	0.100	2433.21118	1696.30237
17	0.200	5047.94092	3857.83838
19	0.300	7714.19287	5744.27148
	Slope	25297.02488	19077.71940
	Intercept	-56.43316	-128.79322
	fit	0.99914	0.99721

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34240801 + 1 µg/g (P)	1.0	NA	NA	NA	1.0133	1.0133	101
Control 34240801 + 1 µg/g (P) DUP	1.0	NA	NA	NA	0.9563	0.9563	96
			Average Recovery =	NA			98
			Std. Dev. =	NA			4

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD.L Date: 03 May 01

Figure 36 (Cont). Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Forage Day 77, Curve 1

Protocol: 000290
Matrix: Corn Forage
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: DayX FSS-Forage curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 12 Feb 01
Injection Date: 14 Feb 01

	M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	M1	M2	M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 34240801	27	ND	128.71297	ND	0.0141	2.0	ND	ND	ND	0.0281	0.026	(0.026)	(0.026)
Control 34240801 + 1 µg/g (M1 + M2)	29	915.64838	912.40820	0.0380	0.0539	20	0.7598	NA	NA	1.0782	NA	NA	NA
Control 34240801 + 1 µg/g (M1+M2) DUP	30	904.05554	936.43079	0.0376	0.0551	20	0.7511	NA	NA	1.1026	NA	NA	NA
Sample 34240803	32	824.61371	906.42236	0.0346	0.0536	20	0.6915	0.915	0.92	1.0721	1.009	1.01	1.92
Sample 34240803 DUP	34	817.23358	841.44452	0.0343	0.0503	20	0.6859	0.908	0.91	1.0060	0.947	0.95	1.86
Sample 34240803 TRIP	36	868.68225	910.83875	0.0362	0.0538	20	0.7245	0.959	0.96	1.0766	1.013	1.01	1.97

Recorded By: Melinda Lalko Date: 27 Apr 01
Reviewed By: [signature] Date: 03 May 01

Figure 37. Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Forage Day 77, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: DayX FSS-Forage curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Forage
Analysis Initiation Date: 12 Feb 01
Injection Date: 14 Feb 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas M1
20	0.015	372.48947	252.30106
21	0.020	455.29135	277.90665
22	0.050	1196.61499	791.54993
23	0.100	2496.52051	1637.11841
24	0.200	5031.38330	3510.72827
25	0.300	7857.82275	5700.24414
26	0.015	365.75668	248.54536
28	0.020	479.19434	283.87045
31	0.050	1198.32129	819.73859
33	0.100	2487.11279	1774.94055
35	0.200	5388.30566	3875.41992
37	0.300	8034.81445	6001.11523
	Slope	26665.20778	19671.52717
	Intercept	-97.30892	-148.04281
	fit	0.99880	0.99585

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	M1 Found µg/g	M1 Control Corrected µg/g	M1 Percent Recovery
Control 34240801 + 1 µg/g (M1 + M2)	1.0	0.7598	0.7598	76	1.0782	1.0500	105
Control 34240801 + 1 µg/g (M1+M2) DUP	1.0	0.7511	0.7511	75	1.1026	1.0744	107
			Average Recovery =	76			106
			Std. Dev. =	1			2

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: [signature] Date: 03 May 01

Figure 37 (Cont). Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Forage Day 77, Curve 2

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: Day0 FSS-Grain curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 12 Jan 01
Injection Date: 15-16 Jan 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 34241601	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34241601 + 0.2 µg/g (P)	11	NA	1448.56995	NA	0.0856	2.0	NA	NA	NA	0.1711	NA	NA	NA
Control 34241601 + 0.2 µg/g (P) DUP	12	NA	1518.11096	NA	0.0894	2.0	NA	NA	NA	0.1788	NA	NA	NA
Sample 34241602	14	NA	1480.53809	NA	0.0873	2.0	NA	NA	NA	0.1746	0.194	0.19	0.19
Sample 34241602 DUP	16	NA	1672.30664	NA	0.0978	2.0	NA	NA	NA	0.1956	0.217	0.22	0.22
Sample 34241602 TRIP	18	NA	1542.42432	NA	0.0907	2.0	NA	NA	NA	0.1814	0.202	0.20	0.20
Control 34241601 + 0.2 µg/g (P) addn	19	NA	1618.98657	NA	0.0949	2.0	NA	NA	NA	0.1898	NA	NA	NA

Recorded By: Melinda Lalko Date: 27 Apr 01
Reviewed By: SDD.L Date: 03 May 01

Figure 38. Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Grain Day 0, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: Day0 FSS-Grain curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 12 Jan 01
Injection Date: 15-16 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P
1	0.015	344.54816	231.66705
2	0.020	443.57797	259.61234
3	0.050	1116.79980	745.90936
4	0.100	2285.58179	1656.67102
5	0.200	4620.98291	3397.46655
6	0.300	6747.67676	5045.44336
7	0.015	323.97098	209.27542
10	0.020	427.84232	262.60776
13	0.050	1122.48328	801.70587
15	0.100	2275.62305	1625.07349
17	0.200	4564.64453	3682.19946
20	0.300	7311.46777	5734.26758
	Slope	23447.27108	18269.95754
	Intercept	-44.79684	-114.82855
	fit	0.99759	0.99312

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34241601 + 0.2 µg/g (P)	0.20	NA	NA	NA	0.1711	0.1711	86
Control 34241601 + 0.2 µg/g (P) DUP	0.20	NA	NA	NA	0.1788	0.1788	89
Control 34241601 + 0.2 µg/g (P) addn	0.20	NA	NA	NA	0.1898	0.1898	95
			Average Recovery =	NA			90
			Std. Dev. =	NA			5

Recorded By: Melinda Lalko Date: 27 Jan 01

Reviewed By: SDD. L Date: 03 May 01

Figure 38 (Cont). Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Grain Day 0, Curve 1

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: Day0 FSS-Grain curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 12 Jan 01
Injection Date: 16 Jan 01

	M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	M1	M2	M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 34241601	28	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34241601 + 0.2 µg/g (M1 + M2)	30	1865.97559	1789.99768	0.0800	0.100	2.0	0.1600	NA	NA	0.2004	NA	NA	NA
Control 34241601 + 0.2 µg/g (M1+M2) DUP	31	1968.76550	1741.79712	0.0843	0.098	2.0	0.1686	NA	NA	0.1954	NA	NA	NA
Sample 34241603	33	2126.86768	1939.37256	0.0909	0.108	2.0	0.1817	0.222	0.22	0.2162	0.220	0.22	0.44
Sample 34241603 DUP	35	2125.99316	2057.97827	0.0908	0.114	2.0	0.1817	0.222	0.22	0.2286	0.232	0.23	0.45
Sample 34241603 TRIP	37	1579.06396	1614.04932	0.0681	0.0910	2.0	0.1362	0.166	0.17	0.1820	0.185	0.18	0.35
Control 34241601 + 0.2 µg/g (M1 + M2) addn	38	1907.46619	1736.19312	0.0817	0.0974	2.0	0.1635	NA	NA	0.1948	NA	NA	NA

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. L Date: 03 May 01

Figure 39. Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Grain Day 0, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Laiko
Analytical Set I.D.: Day0 FSS-Grain curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 12 Jan 01
Injection Date: 16 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	M1
21	0.015	344.98303	253.01065
22	0.020	.453.69644	279.85590
23	0.050	1074.91492	763.30048
24	0.100	2206.06055	1587.08826
25	0.200	4578.77344	3548.40723
26	0.300	7237.89795	5562.42139
27	0.015	330.92981	228.83835
29	0.020	430.26868	285.02908
32	0.050	1215.61365	867.28320
34	0.100	2374.49170	1796.25452
36	0.200	4841.67822	3782.90942
39	0.300	7150.54199	5708.61572
	Slope	24031.62090	19025.24283
	Intercept	-56.95585	-116.79737
	fit	0.99895	0.99787

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34241601 + 0.2 µg/g (M1 + M2)	0.20	0.1600	0.1600	80	0.2004	0.2004	100
Control 34241601 + 0.2 µg/g (M1+M2) DUP	0.20	0.1686	0.1686	84	0.1954	0.1954	98
Control 34241601 + 0.2 µg/g (M1 + M2) addn	0.20	0.1635	0.1635	82	0.1948	0.1948	97
			Average Recovery =	82			98
			Std. Dev. =	2			2

Recorded By: Melinda Laiko Date: 27 Apr 01

Reviewed By: SDD. L Date: 03 May 01

Figure 39 (Cont). Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Grain Day 0, Curve 2

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: DayX FSS-Grain curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 12 Mar 01
Injection Date: 13-14 Mar 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 34241601	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34241601 + 0.2 µg/g (P)	11	NA	1439.47253	NA	0.0910	2.0	NA	NA	NA	0.1819	NA	NA	NA
Control 34241601 + 0.2 µg/g (P) DUP	12	NA	1339.85083	NA	0.0851	2.0	NA	NA	NA	0.1701	NA	NA	NA
Sample 34241602	14	NA	1304.27271	NA	0.0829	2.0	NA	NA	NA	0.1659	0.188	0.19	0.19
Sample 34241602 DUP	16	NA	1338.74451	NA	0.0850	2.0	NA	NA	NA	0.1700	0.193	0.19	0.19
Sample 34241602 TRIP	18	NA	1421.79846	NA	0.0899	2.0	NA	NA	NA	0.1798	0.204	0.20	0.20

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. L Date: 03 May 01

Figure 40. Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Grain Day 59, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: DayX FSS-Grain curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 12 Mar 01
Injection Date: 13-14 Mar 01

IIA. Calibration Data

Calibration Curve Formula: $Y = mX + b$

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P
1	0.015	350.93588	234.29762
2	0.020	440.83612	253.70114
3	0.050	1054.19714	654.25732
4	0.100	2207.59229	1509.35327
5	0.200	4732.17090	3209.35254
6	0.300	7045.97021	4983.39258
7	0.015	321.28555	203.86414
10	0.020	428.20221	272.59036
13	0.050	1132.72729	758.75604
15	0.100	2312.71948	1596.58972
17	0.200	4947.67725	3256.10278
19	0.300	7599.99072	5035.99414
	Slope	24643.08763	16860.59873
	Intercept	-98.89375	-94.23072
	fit	0.99716	0.99904

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34241601 + 0.2 µg/g (P)	0.20	NA	NA	NA	0.1819	0.1819	91
Control 34241601 + 0.2 µg/g (P) DUP	0.20	NA	NA	NA	0.1701	0.1701	85
			Average Recovery =	NA			88
			Std. Dev. =	NA			4

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. L Date: 03 May 01

Figure 40 (Cont). Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Grain Day 59, Curve 1

Protocol: 000290
Matrix: Corn Grain
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: DayX FSS-Grain curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 12 Mar 01
Injection Date: 14 Mar 01

	M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	M1	M2	M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 34241601	27	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34241601 + 0.2 µg/g (M1 + M2)	29	1755.60742	1563.77051	0.0775	0.0982	2.0	0.1550	NA	NA	0.1963	NA	NA	NA
Control 34241601 + 0.2 µg/g (M1+M2) DUP	30	1620.18323	1583.85730	0.0716	0.0994	2.0	0.1433	NA	NA	0.1987	NA	NA	NA
Sample 34241603	32	1607.43542	1355.70911	0.0711	0.0858	2.0	0.1422	0.191	0.19	0.1716	0.174	0.17	0.36
Sample 34241603 DUP	34	1636.31042	1444.56958	0.0723	0.0911	2.0	0.1447	0.194	0.19	0.1822	0.184	0.18	0.38
Sample 34241603 TRIP	36	1526.67883	1407.86279	0.0676	0.0889	2.0	0.1352	0.181	0.18	0.1778	0.180	0.18	0.36

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD. C Date: 03 May 01

Figure 41. Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Grain Day 59, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: DayX FSS-Grain curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Grain
Analysis Initiation Date: 12 Mar 01
Injection Date: 14 Mar 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	M1
20	0.015	330.82883	213.25795
21	0.020	409.53772	257.89444
22	0.050	1095.79895	669.64062
23	0.100	1979.81299	1479.31226
24	0.200	4782.00537	3242.84326
25	0.300	6585.97021	4896.92139
26	0.015	339.36157	216.13158
28	0.020	445.33209	275.69022
31	0.050	1085.14148	776.06219
33	0.100	2391.43066	1647.33203
35	0.200	4777.78418	3224.27588
37	0.300	6993.76807	5089.26465
	Slope	23068.75976	16797.17777
	Intercept	-32.28573	-85.29226
	fit	0.99589	0.99853

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34241601 + 0.2 µg/g (M1 + M2)	0.20	0.1550	0.1550	78	0.1963	0.1963	98
Control 34241601 + 0.2 µg/g (M1+M2) DUP	0.20	0.1433	0.1433	72	0.1987	0.1987	99
			Average Recovery =	75			99
			Std. Dev. =	4			1

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. L Date: 03 May 01

Figure 41 (Cont). Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Grain Day 59, Curve 2

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: Day0 FSS-Stover curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 16 Jan 01
Injection Date: 17-18 Jan 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 34242401	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34242401 + 1.5 µg/g (P)	11	NA	2358.95337	NA	0.131	10	NA	NA	NA	1.3085	NA	NA	NA
Control 34242401 + 1.5 µg/g (P) DUP	12	NA	2286.66089	NA	0.127	10	NA	NA	NA	1.2704	NA	NA	NA
Sample 34242402	14	NA	2011.48914	NA	0.113	10	NA	NA	NA	1.1252	1.309	1.31	1.31
Sample 34242402 DUP	16	NA	2366.75024	NA	0.131	10	NA	NA	NA	1.3126	1.527	1.53	1.53
Sample 34242402 TRIP	18	NA	2319.52734	NA	0.129	10	NA	NA	NA	1.2877	1.498	1.50	1.50

Recorded By: Melinda Lalko Date: 27 Apr 01
Reviewed By: SDD.C Date: 03 May 01

Figure 42. Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Stover Day 0, Curve 1

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Laiko
Analytical Set I.D.: Day0 FSS-Stover curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 16 Jan 01
Injection Date: 17-18 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P
1	0.015	316.78629	248.52669
2	0.020	426.96469	273.85590
3	0.050	1038.46399	776.95251
4	0.100	2129.63843	1606.72253
5	0.200	4335.58740	3465.84668
6	0.300	6817.75391	5634.80762
7	0.015	311.18173	228.82213
10	0.020	422.19370	271.67889
13	0.050	1063.54272	795.66449
15	0.100	2294.56104	1781.99829
17	0.200	4755.39746	3872.15454
19	0.300	6666.24219	5554.42383
	Slope	22691.44195	18958.10946
	Intercept	-42.41349	-121.76299
	fit	0.99815	0.99720

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34242401 + 1.5 µg/g (P)	1.5	NA	NA	NA	1.3085	1.3085	87
Control 34242401 + 1.5 µg/g (P) DUP	1.5	NA	NA	NA	1.2704	1.2704	85
			Average Recovery =	NA			86
			Std. Dev. =	NA			2

Recorded By: Melinda Laiko Date: 27 Apr 01

Reviewed By: SDD. L Date: 03 May 01

Figure 42 (Cont). Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Stover Day 0, Curve 1

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: Day0 FSS-Stover curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 16 Jan 01
Injection Date: 18 Jan 01

	M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	M1	M2	M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 34242401	27	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34242401 + 1.5 µg/g (M1 + M2)	29	2080.77563	2345.72827	0.0959	0.139	10	0.9587	NA	NA	1.3945	NA	NA	NA
Control 34242401 + 1.5 µg/g (M1+M2) DUP	30	2229.61621	2462.55615	0.103	0.146	10	1.0268	NA	NA	1.4616	NA	NA	NA
Sample 34242403	32	2331.92090	2477.34204	0.107	0.147	10	1.0737	1.622	1.62	1.4701	1.544	1.54	3.17
Sample 34242403 DUP	34	2546.84448	2430.90479	0.117	0.144	10	1.1721	1.771	1.77	1.4434	1.516	1.52	3.29
Sample 34242403 TRIP	36	2242.60327	2263.59229	0.103	0.135	10	1.0328	1.560	1.56	1.3473	1.415	1.42	2.98

Recorded By: Melinda Latko Date: 27 Aug 01

Reviewed By: [signature] Date: 03 May 01

Figure 43. Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Stover Day 0, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: Day0 FSS-Stover curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 16 Jan 01
Injection Date: 18 Jan 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	M1
20	0.015	312.27158	230.99808
21	0.020	408.64932	266.15338
22	0.050	1072.86011	756.37579
23	0.100	2197.26123	1656.16956
24	0.200	4363.84766	3197.62061
25	0.300	6450.58154	5132.56494
26	0.015	311.57455	211.56190
28	0.020	446.95135	272.54810
31	0.050	1085.28235	817.12140
33	0.100	2154.62891	1630.10254
35	0.200	4319.95312	3415.51074
37	0.300	6641.17773	5279.95801
	Slope	21839.51050	17409.54581
	Intercept	-12.92416	-82.03273
	fit	0.99965	0.99830

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			M1		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34242401 + 1.5 µg/g (M1 + M2)	1.5	0.9587	0.9587	64	1.3945	1.3945	93
Control 34242401 + 1.5 µg/g (M1+M2) DUP	1.5	1.0268	1.0268	68	1.4616	1.4616	97
			Average Recovery =	66			95
			Std. Dev. =	3			3

Recorded By: Melinda LaOrs Date: 27 Apr 01

Reviewed By: [signature] Date: 03 May 01

Figure 43 (Cont). Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Stover Day 0, Curve 2

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: DayX FSS-Stover curve 1
Compound: Parent and Metabolites

Analysis Initiation Date: 17 Apr 01
Injection Date: 20 Apr 01

	P + M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - P			Total Glufosinate Acid Equivalent (µg/g)
		M2	P + M1	M2	P + M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Reagent Blank	8	ND	ND	ND	ND	NA	ND	ND	ND	ND	ND	ND	ND
Control 34242401	9	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34242401 + 1.5 µg/g (P)	11	NA	2120.75122	NA	0.129	10	NA	NA	NA	1.2935	NA	NA	NA
Control 34242401 + 1.5 µg/g (P) DUP	12	NA	2052.49121	NA	0.125	10	NA	NA	NA	1.2530	NA	NA	NA
Sample 34242402	14	NA	2091.97144	NA	0.128	10	NA	NA	NA	1.2764	1.504	1.50	1.50
Sample 34242402 DUP	16	NA	1901.33655	NA	0.116	10	NA	NA	NA	1.1633	1.370	1.37	1.37
Sample 34242402 TRIP	18	NA	2251.19604	NA	0.137	10	NA	NA	NA	1.3708	1.615	1.62	1.62

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD.C Date: 03 May 01

Figure 44. Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Stover Day 91, Curve 1

Method: Avenlis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: DayX FSS-Stover curve 1
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 17 Apr 01
Injection Date: 20 Apr 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas	
		M2	P
1	0.015	333.14911	221.66309
2	0.020	437.66257	291.79614
3	0.050	1149.61401	726.28125
4	0.100	2341.72681	1532.87500
5	0.200	4665.00684	3181.84790
6	0.300	7241.32715	4872.13770
7	0.015	336.04733	196.73389
10	0.020	459.70392	330.47586
13	0.050	1176.07068	815.63489
15	0.100	2272.97925	1592.80701
17	0.200	4919.50439	3480.79028
19	0.300	7226.24707	5136.04346
	Slope	24227.64389	16854.54090
	Intercept	-52.73608	-59.30288
	fit	0.99940	0.99734

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2			P		
		Found µg/g	Control Corrected µg/g	Percent Recovery	Found µg/g	Control Corrected µg/g	Percent Recovery
Control 34242401 + 1.5 µg/g (P)	1.5	NA	NA	NA	1.2935	1.2935	86
Control 34242401 + 1.5 µg/g (P) DUP	1.5	NA	NA	NA	1.2530	1.2530	84
			Average Recovery =	NA			85
			Std. Dev. =	NA			2

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD.C Date: 03 May 01

Figure 44 (Cont). Summary Spreadsheets of Glufosinate Storage Stability Data for Corn Stover Day 91, Curve 1

Protocol: 000290
Matrix: Corn Stover
Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Lalko
Analytical Set I.D.: DayX FSS-Stover curve 2
Compound: Parent and Metabolites

Analysis Initiation Date: 17 Apr 01
Injection Date: 20-21 Apr 01

	M1	M2
LOD (µg/g)	0.015	0.015
LOQ (µg/g)	0.050	0.050

IA. Results

Sample I.D.	Injection number	Peak Areas		µg/mL Extract		Method Factor	Glufosinate Acid Equivalent - M2			Glufosinate Acid Equivalent - M1			Total Glufosinate Acid Equivalent (µg/g)
		M2	M1	M2	M1		Found µg/g	Corrected µg/g	Reported µg/g	Found µg/g	Corrected µg/g	Reported µg/g	
Control 34242401	27	ND	ND	ND	ND	2.0	ND	ND	ND	ND	ND	ND	ND
Control 34242401 + 1.5 µg/g (M1 + M2)	29	2974.61841	2621.54150	0.122	0.154	10	1.2167	NA	NA	1.5412	NA	NA	NA
Control 34242401 + 1.5 µg/g (M1+M2) DUP	30	2749.93262	2392.32568	0.113	0.141	10	1.1266	NA	NA	1.4103	NA	NA	NA
Sample 34242403	32	2851.33691	2586.01245	0.117	0.152	10	1.1673	1.494	1.49	1.5209	1.546	1.55	3.04
Sample 34242403 DUP	34	2712.83521	2416.57837	0.111	0.142	10	1.1117	1.423	1.42	1.4241	1.448	1.45	2.87
Sample 34242403 TRIP	36	2557.58569	2361.16968	0.105	0.139	10	1.0494	1.344	1.34	1.3925	1.415	1.42	2.76

Recorded By: Melinda Lalko Date: 27 Apr 01

Reviewed By: SDD.C Date: 03 May 01

Figure 45. Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Stover Day 91, Curve 2

Method: Aventis CropSciences RAM BK/05/95
Analyst(s): Melinda Latko
Analytical Set I.D.: DayX FSS-Stover curve 2
Compound: Parent and Metabolites

Protocol: 000290
Matrix: Corn Stover
Analysis Initiation Date: 17 Apr 01
Injection Date: 20-21 Apr 01

IIA. Calibration Data

Calibration Curve Formula: Y = mX + b

Injection number	Standard Concentration (µg/mL)	Peak Areas M2	Peak Areas M1
20	0.015	341.70273	225.22621
21	0.020	453.15283	292.46707
22	0.050	1106.65405	711.64276
23	0.100	2356.73511	1648.44604
24	0.200	4760.37402	3284.18164
25	0.300	7320.17871	5266.69189
26	0.015	343.65152	213.40002
28	0.020	454.77121	288.93091
31	0.050	1184.01404	791.24677
33	0.100	2523.10620	1777.06311
35	0.200	5050.32568	3362.58081
37	0.300	7555.16309	5203.20117
	Slope	24929.15713	17505.62892
	Intercept	-58.59301	-76.46944
	fit	0.99884	0.99863

IIIA. Procedural Recoveries

Sample I.D.	µg/g Added	M2 Found µg/g	M2 Control Corrected µg/g	M2 Percent Recovery	M1 Found µg/g	M1 Control Corrected µg/g	M1 Percent Recovery
Control 34242401 + 1.5 µg/g (M1 + M2)	1.5	1.2167	1.2167	81	1.5412	1.5412	103
Control 34242401 + 1.5 µg/g (M1+M2) DUP	1.5	1.1266	1.1266	75	1.4103	1.4103	94
			Average Recovery =	78			98
			Std. Dev. =	4			6

Recorded By: Melinda Latko Date: 27 Apr 01

Reviewed By: SDD. L Date: 03 May 01

Figure 45 (Cont). Summary Spreadsheets of M1 and M2 Storage Stability Data for Corn Stover Day 91, Curve 2

APPENDIX C—Aventis CropScience Analytical Method RAM Number BK/05/95

Method Modification

Protocol Number:	000290
Method Title:	Gas Chromatographic Determination of HOE-039866 (Glufosinate-Ammonium) and its Metabolites as Residues in Glufosinate-Resistant Corn and Soybean RAC and Processed Commodities (RAM Number BK/05/95)
Effective Date:	13 Nov 00
Modification Number:	1
Requirement:	1. Page 16, Section 6.2.1, 6th paragraph, states to "transfer a 100.0 mL aliquot (V2) to a 250 mL flat bottom flask." 2. Page 17, Section 6.4.2 states to "Add 12 (±1) mL of trimethylorthoacetate reagent and a few glass beads. Mix by swirling with sonication. [See also Section 11.0 (Derivatization).] When mixing is complete, reflux the reaction mixture for 4.5 hours."
Description:	1. A 150 mL beaker was used instead of a 250 mL flat bottom flask. 2. The volume of trimethylorthoacetate reagent that is added into the 250mL flat bottom flask containing the sample is increased from 12 mL to 18 mL. The water passing through the reflux condensers are chilled during the 4.5 hours of reflux.
Reason:	1. The aliquot solution can be easily transferred to the anion exchange column with the use of a beaker. 2. The average recovery values for M2 in Set 1F fell below the 70% limit. The above changes were done with the intention of increasing M2 recoveries in future extraction sets.
Effect:	There should be no detrimental effect on the study.

Approved by:

Cynthia K. Robb — Feb 19, 2001

Cynthia K. Robb
Study Director
Dow AgroSciences

Date

Melinda Lalko — 13 Feb 01

Melinda Lalko
Principal Investigator
McKenzie/Wright Laboratories, LLC

Date

AgrEvo

A company of Hoechst and NOR-AM

Analytical Method

Department: Residue Chemistry

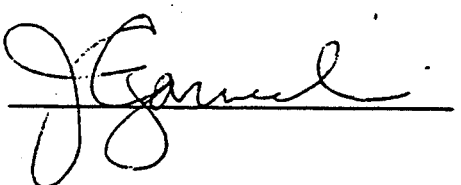
Date: June 12, 1995

RAM Number: BK/05/95

Cross References: HRAV-5A, AE-24, AE-24A

Title: Gas Chromatographic Determination of HOE-039866 (Glufosinate-Ammonium) and Its Metabolites as Residues in Glufosinate-Resistant Corn and Soybean RAC and Processed Commodities

Submitted and Approved by: J. J. Czarnecki, Ph.D.
Manager, Residue Applications
AgrEvo Research Center

Signed:  Date: 9-15-95

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GAS CHROMATOGRAPHIC DETERMINATION OF HOE-039866 (GLUFOSINATE-AMMONIUM) AND ITS METABOLITES AS RESIDUES IN GLUFOSINATE RESISTANT CORN AND SOYBEAN RAC AND PROCESSED COMMODITIES

AGREVO ANALYTICAL METHOD: BK/05/95

Basis of Method: HRAV-5A (glufosinate enforcement procedure)
AE-24
AE-24A

Issued: June 12 , 1995

Written by: J. J. Czarnecki, Ph.D.
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DATA REQUIREMENT

Guideline 171-4(c)

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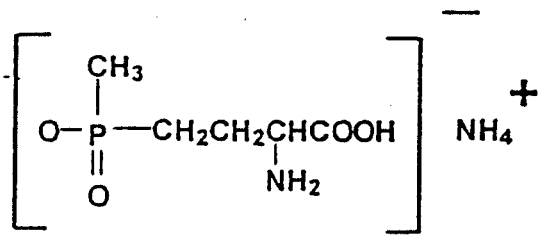
1.0 Chemical Information:

1.1 HOE-039866 (Parent):

Chemical Name: Ammonium-DL-homoalanin-4-yl-(methyl)-phosphinate (IUPAC, English).
Common Name: Glufosinate-ammonium

Structure:



HOE-039866

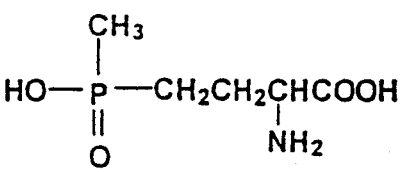
Molecular Formula: $C_5H_{15}N_2O_4P$
Molecular Weight: 198.2 g/mole
CAS Number: CA 77182-82-2

1.2 Free Acid of HOE-039866:

1.21 HOE-035956
Chemical Name: DL-homoalanin-4-yl-(methyl)phosphinic acid (IUPAC, English)
Common Name: Glufosinate

1.22 HOE-090532 (Purified D-isomer form of HOE-035956):
Chemical Name: D-homoalanin-4-yl-(methyl)phosphinic acid (IUPAC, English)
Common Name: M-Glufosinate

Structure:



HOE-035956
HOE-090532

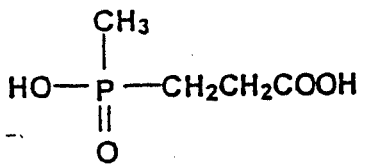
Molecular Formula: $C_5H_{12}NO_4P$
Molecular Weight: 181.1 g/mole
CAS Number: CA 51276-47-2 (HOE-035956)

1.0 Chemical Information (Continued):

1.3 HOE-061517 (Metabolite of HOE-039866):

Chemical Name: 3-Methylphosphinico-propionic acid (IUPAC, English)

Structure:



HOE-061517

Molecular Formula: $C_4H_9O_4P$
Molecular Weight: 152.1 g/mole
CAS Number: CA 15090-23-0

1.4 N-Acetyl Glufosinate (Metabolite of HOE-039866):

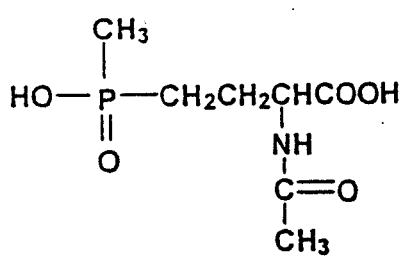
1.41 HOE-085355

Chemical Name: DL-2-acetamido-4-methylphosphinico-butanoic acid (IUPAC, English)

1.42 HOE-099730 (Purified L-isomer form of HOE-085355):

Chemical Name: Disodium L-2-acetamido-4-methylphosphinico-butyrate (IUPAC, English)

Structure:



HOE-085355 (free acid, shown above)
HOE-099730 (disodium salt)

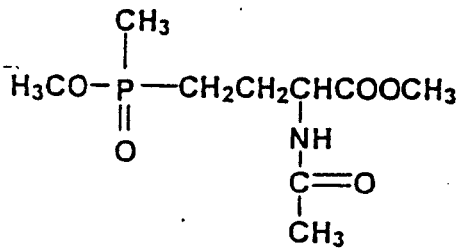
Molecular Formula: $C_7H_{14}NO_5P$ (free acid)
Molecular Weight: 223.2 g/mole (free acid)
Molecular Weight: 267.1 g/mole (disodium salt)

1.0 Chemical Information (Continued):

1.5 HOE-064706 (Derivative of HOE-039866/-035956/-090532/-085355/-099730):

Chemical Name: Methyl-4-(methoxymethyl)phosphinoyl-2-acetamido-butyrate (IUPAC, English)

Structure:



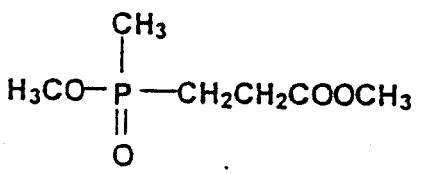
HOE-064706

Molecular Formula: $C_9H_{18}NO_5P$
Molecular Weight: 251.2 g/mole

1.6 HOE-070951 (Derivative of HOE-061517):

Chemical Name: Methyl-3-(methoxymethyl)phosphinoyl-propionate (IUPAC, English)

Structure:



HOE-070951

Molecular Formula: $C_6H_{13}O_4P$
Molecular Weight: 180.1 g/mole

2.0 Principle of the Method:

Residues of glufosinate (ammonium) and its principal metabolites *in glufosinate resistant field corn and soybeans* are extracted from finely ground sample material into distilled water. Corn or soybean oil is not extracted; residues are derivatized in the sample matrix.

The aqueous extract is filtered (or centrifuged) to remove non-dissolved solids.

For all matrices except oil, an aliquot of the filtrate is passed through anion exchange resin (hydroxide form) to remove potentially interfering co-extracted sample matrix components. Such components, which are not bound by the resin, are water washed through the column. The parent and metabolite compounds are then eluted from the anion exchange resin using a formic acid solution.

The column eluate containing HOE-039866 (or HOE-090532), HOE-061517 and HOE-099730 (or HOE-085355) is evaporated to dryness using a rotary evaporator.

The sample residue is derivatized under reflux for approximately 4.5 hours using trimethylorthoacetate in glacial acetic acid. After a solvent exchange procedure, the solution is passed through a silica gel SPE cartridge for additional clean up.

Thus, the analytical solution contains the derivatives:

- HOE-064706 [methyl-4-(methoxymethyl)phosphinoyl-2-acetamido-butyrate] from the combined residue of HOE-039866 (or HOE-090532) and HOE- 099730 (or HOE-085355) *and*
- HOE-070951 [methyl-3-(methoxymethyl)phosphinoyl-propionate] from the residue of HOE-061517.

Quantitative determination of HOE-064706 and/or HOE-070951 is accomplished using GC/FPD, i.e., gas chromatography with flame photometric detection (P-mode).

Residues are expressed as HOE-035956 (glufosinate free acid) equivalents.

The following corn and soybean samples have been successfully analyzed using this methodology:

Corn:	grain, forage, silage, fodder, aspirated grain fractions (dust), starch, oil, grits, meal, and flour
Soybeans:	seed, forage, hay, aspirated grain fractions (dust), meal, hulls, and oil

2.0 Principle of the Method (Continued):

The analytical standards listed below are useful for determination of procedural recoveries:

HOE-039866 [ammonium-DL-homoalanin-4-yl-(methyl)phosphinate] - active ingredient/parent,

HOE-090532 [D-homoalanin-4-yl-(methyl)phosphinic acid] - purified "D" isomer of the parent compound (free acid form),

HOE-061517 [3-methylphosphinico-propionic acid] - metabolite,

HOE-099730 [disodium L-2-acetamido-4-methylphosphinico-butyrate] - metabolite, as purified "L" isomer form, and.

HOE-085355 [DL-2-acetamido-4-methylphosphinico-butanoic acid] - metabolite, as racemic mixture.

Note that reference compounds HOE-039866 and HOE-090532 represent options for use in determining parent compound recovery. Likewise, reference compounds HOE-085355 and HOE-099730 represent options for determining recovery of the N-acetyl glufosinate metabolite. The following procedures will assume utilization of HOE-039866 and HOE-099730 reference standards for this purpose.

3.0 Equipment:

[Note: Unless otherwise indicated, functionally equivalent equipment may be substituted for the equipment listed below.]

1. Mill: Retsch grinder equipped with a 2 mm sieve.
2. Food processor: Robot-Coupe.
3. Balances: Mettler Model BB-2440, (for sample preparation); Mettler Model AE 240, (for standard preparation).
4. Microliter Syringes: 10, 100, 250, 500 microliter (µL), Hamilton.
5. Flat Bottom Flask: 250 mL capacity [VWR: 29114-045].

5a. Round Bottom Flask: 50 mL capacity [VWR 29127-036].

6. Reflux Condenser: High efficiency, 300 mm, equipped with a 24/40 ground glass joint [VWR: 23114-042].
7. Hot Plate: Series 730 PMC Dataplate [Fisher Scientific: 11-495-137].

3.0 Equipment (Continued)

8. Erlenmeyer Flasks: 500 mL [VWR: 29140-566].

9. Collection Tubes, Glass: 16mm X 100 mm, for collection of silica gel column fractions [Fisher Scientific: 14-961-29].

10. Volumetric Pipettes, Glass: Class A; 0.10, 0.50, 1.0, 2.0 mL capacity.

11. Luer Stopcocks: Varian/Analytichem International; used with SPE silica gel cartridges.

12. Ultrasonic Bath: Bransonic [VWR: 21812-119].

13. Rotary Flash Evaporator: Buchi, RE-121A [VWR: 27558-354], with a water bath operating at 45-60°C (see also item no. 31).

14. Millex SR Filters: Millipore No. SLR 025NS; or "Baker" Disposable Filtration Columns, J.T. Baker, Inc., Cat. No. 7121-06.

15. Vacuum Flask: 500 mL [Fisher 10-180E].

16. Disposable Pasteur Pipettes, Glass: 9" x 7 mm O.D.

17. Syringes: Polypropylene, 10 mL capacity, equipped with a Luer tip [Aldrich Chemical Co.] - ***unless all glass, substitution not recommended***; avoid use of rubber-tipped syringe plunger.

18. Magnetic Stirrer: Variomag Multipoint [Cole-Parmer: G-0465-20].

19. Magnetic Stir Bars: TFE, [VWR: 58948150].

20. N-Evap Analytical Concentrator: Organomation Associates (see also item no. 31).

21. Gas Chromatograph: Varian 3400 GC equipped with a Flame Photometric Detector operating in the Phosphorus mode, or equivalent instrumentation. [Note: According to the application, the GC system is configured for megabore capillary column operation, with SPI injector.] Use of an autosampler device is optional but recommended for optimum reproducibility and efficiency.

22. Gas Chromatography Column : 1.0 µm DB-WAX, fused silica, 15 meter x 0.53 mm megabore column with cross linked (Carbowax 20M) stationary phase, [J & W Scientific: J1257012]. Note: The commercially available 30 meter column may be divided into two shorter column segments. Typically, a column of 10-15 meter length is adequate for these applications.

3.0 Equipment (Continued)

23. Vacuum Box Apparatus: Vac Elut SPS 24 Vacuum Box [Varian/Analytichem International: No. A16500-2]; for use with silica gel SPE cartridges. [Note: Use of a vacuum box apparatus is optional. Any equivalent apparatus or device which enables operation of the SPE cartridge system may be utilized to provide a uniform and controllable cartridge elution (drip) rate.]

24. Glass Column: with stopcock and reservoir, 10.5 mm id., 250 mm length; [Lab Glass: LG-4565-T-100], used for anion exchange column chromatography.

25. Silica Gel SPE Cartridge: 500 mg cartridge with reservoir, [Varian/Analytichem International: No. LR01304].

26. Buchner Type Funnel: 7 cm; for vacuum filtration [Fisher Scientific: 10-356B].

27. Filter Paper: Whatman No. 934-AH, 1.5µ, [e.g., Fisher Scientific: 09-873F (7 cm)], or sized to fit the Buchner funnel (Item 26).

28. Graduated Cylinders: various capacities, up to 500 mL.

29. Centrifuge: Laboratory bench top model, capable of 600-700 x G (at 2,000 - 3,000 rpm).

30. Centrifuge Tubes/Bottles: Various capacities as required. [Note: a graduated tube (10 - 20-mL capacity) is used in Section 11.0]

31. Speed Vac Concentrator: Savant SC110 (or equivalent), for use in place of a rotary evaporator or N-Evaporator, as appropriate, within the method.

4.0 Reagents/Chemical Supplies:

a) Formic Acid: Fisher Scientific (88% w/w), ACS Reagent Grade.

b) Methyl Acetate: (99%) AR.

c) Ethyl Acetate: pesticide quality.

d) Glacial Acetic Acid: AR.

e) Trimethylorthoacetate: Aldrich (99%) [reagent must be dry]

4.0 Reagents/Chemical Supplies (Continued):

f) Methanol: pesticide quality.

g) Toluene: pesticide quality.

h) Denatured Ethanol: Mallinckrodt Cat. No. 7018; or

Ethanol/Methanol (85/15 v/v): pure reagents mixed to obtain a 85/15 volume ratio. [Generally available as a prepared commercial reagent.]

i) Denatured Ethanol/Methyl Acetate (1/1 v/v): Mix equal volumes of denatured ethanol and methyl acetate reagents.

j) Methanol/Methyl Acetate (1/1 v/v): Mix pure reagents to a 1/1 volume ratio of Methanol/Methyl Acetate.

k) Toluene/Methyl Acetate (1/1 v/v): Mix pure reagents to a 1/1 volume ratio of Toluene/Methyl Acetate.

l) Formic Acid (50% w/w): Dilute 570 mL of Formic Acid reagent (88%) to 1 L (final volume) with distilled water. Always add the concentrated acid to 300-400 mL of water before diluting to final volume.

m) Ammonium Hydroxide (0.015 M): Dilute 1 mL of ammonia solution [BDH No. ACS 033-037 (28-30% NH_3)] to 100 mL with distilled water.

n) HOE-039866: Ammonium DL-homoalanin-4-yl-(methyl)phosphinate, analytical standard.

o) HOE-090532: D-homoalanin-4-yl-(methyl)phosphinic acid, analytical standard.

p) HOE-061517: 3-Methylphosphinico-propionic acid, analytical standard.

q) HOE-085355: DL-2-acetamido-4-methylphosphinico-butanoic acid, analytical standard.

r) HOE-099730: Disodium L-2-acetamido-4-methylphosphinico-butyrate, analytical standard.

4.0 Reagents/Chemical Supplies (Continued):

s) HOE-064706: Methyl-4-(methoxymethyl)phosphinoyl-2-acetamido-butyrate, analytical standard.

t) HOE-070951: Methyl-3(methoxymethyl)phosphinoyl propionate, analytical standard.

u) Silica Gel (Activated): VWR Scientific 26668-109, for drying tubes

v) Anion Exchange Resin: Bio-Rad (140-1431) AG 1X8 ion exchange resin, 8% cross linking, 50-100 dry mesh, in the hydroxide form (see also item 'x' below).

w) Preparation of Anion Exchange Resin: Mix 100 g of Anion Exchange Resin (w) with 1 L of 1M Sodium Hydroxide (y) in a 1-2 L beaker and gently stir using a Teflon-coated spatula for approximately 2 minutes. Let stand for 30 minutes or until the resin solution is pH 10-11 (as indicated using pH paper). The solution must be stirred slowly to avoid crushing the resin. Filter the resin through a coarse fritted disc Buchner funnel apparatus (to remove excess Sodium Hydroxide) and continue to wash the resin with distilled water until the pH of the filtrate is no longer basic (pH 5.5-7.5). Typically, for 100 g of resin, about 3-5 liters of distilled water are required.

x) Sodium Hydroxide (1M): Dissolve 40 grams of Sodium Hydroxide [VWR 6720-1] in distilled water to a final volume of 1 liter.

[NOTE: Analytical Standards 4.0(n), 4.0 (o), 4.0(p), 4.0(q), 4.0(r), 4.0(s), and 4.0(t) are available from AgrEvo USA Company, AgrEvo Research Center, Pikeville, NC.]

5.0 Preparation of Standard Solutions:

[Note: The calibration and fortification standard amounts in the examples below are given for pure (100%) materials. Actual amounts used should be adjusted for certified percent purity. Store all solutions in a refrigerator. Store pure standard materials in a freezer.]

5.1 Calibration Solutions:

HOE-064706 and HOE-070951:

Weigh 139 (±1) mg of HOE-064706 and 100 (±1) mg of HOE-070951 into separate 100 mL volumetric flasks. Dilute each to volume with pure methanol. These solutions (Stock Solutions A1 and A2) contain 1.0 mg of HOE-064706/mL and 1.0 mg of HOE-070951/mL, respectively, expressed as HOE 035956 equivalents. They must be prepared fresh every six months.

Transfer a 2.0-mL aliquot from both Stock Solution A1 and Stock Solution A2 to the same 100 mL volumetric flask. Dilute to volume with pure methyl acetate. This solution (Solution B) contains 20.0 µg of HOE-064706 and HOE-070951/mL, expressed as HOE-035956 equivalents. It must be prepared fresh every six months.

Make dilutions of Solution B every month (or as needed) for calibration of the gas chromatograph. A typical set of dilutions for GC calibration is shown in Table I.

[Note: To ensure accuracy and verify reference solution stability, freshly prepared calibration standard solutions are to be cross-checked with corresponding, previously prepared calibration solutions using side-by-side GC/FPD analysis (e.g., just prior to the expiration date of the solution). The cross-check results (i.e., GC/FPD analyte response) should agree to within ±10% (rel.). Otherwise, standard preparation accuracy should be re-verified and/or appropriate problem solving measures implemented. In addition, ongoing monitoring of GC/FPD calibration (response) factors is also recommended, to provide an indication of analytical system control, including adequate calibration standard stability.]

GC Calibration: [See also Sections 7.2 and 7.3.]

The calibration solutions shown in Table I (below) have been found to provide an adequate linear working range for the GC/FPD analysis. Other standard concentrations may be used according to specific analysis requirements, alternate dilution schemes, etc. However, the working range of the calibration curve should not exceed an approximate factor of 10 to 20, i.e., from the lowest to highest standard concentration utilized.

5.0 Preparation of Standard Solutions (Continued):

5.1 Calibration Solutions (Continued):

TABLE I TYPICAL SERIES OF GC CALIBRATION STANDARDS	
Amount of Stock Solution B (µL)	**Final Concentration in 100.0 mL Final Volume (ng/µL)**
75	0.015
100	0.020
250	0.050
500	0.100
1000	0.200
1500	0.300
Note: *Final concentrations are expressed as HOE-035956 (glufosinate free acid) equivalents.*	

5.2 Fortification Solutions:

HOE-039866:

Weigh 109 (±1) mg of HOE-039866 into a 100 mL volumetric flask. Dilute to volume with 0.015M ammonium hydroxide. This solution [Stock Solution C] contains 1.0 mg of HOE-039866, expressed as HOE-035956 equivalents. It must be prepared fresh every six months.

HOE-061517:

Weigh 84 (±1) mg of HOE-061517 into a 100 mL volumetric flask. Dilute to volume with 0.015 M ammonium hydroxide. This solution (Stock Solution D) contains 1.0 mg of HOE-061517/mL, expressed as HOE-035956 equivalents. It must be prepared fresh every six months.

HOE-099730:

Weigh 147 (±1) mg of HOE-099730 into a 100 mL volumetric flask. Dilute to volume with 0.015 M ammonium hydroxide. This solution (Stock Solution E) contains 1.0 mg of HOE-099730/mL, expressed as HOE-035956 equivalents. It must be prepared fresh every six months.

5.0 Preparation of Standard Solutions (Continued):

5.2 Fortification Solutions (Continued):

Combined Standards:

Transfer 1.0-mL aliquots of Stock Solutions C and D to the same 100 mL volumetric flask. Dilute to volume with 0.015 M ammonium hydroxide (or with denatured ethanol/methyl acetate (1/1) for fortification of oil samples). This solution [Stock Solution F] contains 10.0 µg of HOE-039866 and HOE-061517/mL, expressed as HOE-035956 equivalents. It must be prepared fresh every six months.

Transfer 1.0-mL aliquots of Stock Solutions D and E to the same 100 mL volumetric flask. Dilute to volume with 0.015 M ammonium hydroxide (or with denatured ethanol/methyl acetate (1/1) for fortification of oil samples). This solution (Stock Solution G) contains 10.0 µg of HOE-061517 and HOE-099730/mL, expressed as HOE-035956 equivalents. It must be prepared fresh every six months.

The 10.0 µg/mL stock solutions of HOE-039866 plus HOE-061517 or HOE-061517 plus HOE-099730 is typically used to fortify control samples for recovery purposes.

Note: For determination of procedural recoveries, fortify glufosinate resistant (transgenic) sample matrices with solutions containing HOE-039866 plus HOE-061517 or HOE-099730 plus HOE-061517.

Additional fortification solutions may be prepared as required to cover the range of residues found in actual field samples. Stock solutions and/or fortification solutions may be prepared to have standard concentration levels which differ from those specified above (see also Section 11.0).

6.0 Analytical Procedure:

6.1 Sample Preparation:

Analytical samples (bulk solids) are to be finely ground using a Retsch grinder (or equivalent) or homogenized using a food processor (or equivalent) with dry ice (as necessary). It is important that the laboratory sample be finely ground (e.g., to a particle size $\leq$ 2 mm diameter) and homogeneous before subjecting it to the analysis procedures described below. Liquid analytical samples (e.g., oils) or fine powders (e.g., flour, dust, etc.), which are homogeneous, require no additional preparation.

6.2 Extraction: [For oil samples, proceed to Section 6.2.2.]

6.2.1 Extraction: All Matrices Except Oil

Into a 500-mL Erlenmeyer flask, accurately weigh 12.5 (±0.1) g of the analytical sample ($W_{S)}$, prepared as described in Section 6.1.

6.0 Analytical Procedure (Continued):

6.2.1 Extraction: All Matrices Except Oil (continued)

Add 200 (±1) mL of distilled water and a magnetic stirring bar. [Note: See also Section 11.0.]

Place the flask containing the sample and distilled water onto a magnetic stirring plate. Stir vigorously at room temperature for 0.5 hour.

Transfer the entire contents of the extraction flask to a clean 500-mL graduated cylinder. Add sufficient distilled water to achieve a 500-mL final extraction volume (V1).

{Note: Centrifuge a portion of the *soybean seed, hulls, or meal* extract [e.g., 600-700 x G (2,000 - 3,000 rpm)] in a 250-mL centrifuge bottle. Remove the top fatty layer using a Pasteur pipette or filter as described below, }

Suction filter the mixture through Whatman 934-AH filter paper (or equivalent) contained in a Buchner funnel, collecting the aqueous filtrate in a clean 1-L filtering flask.

- **Except for grain "dust" samples,** transfer a 100.0-mL aliquot (V2) to a 250-mL flat bottom flask. Proceed to Section 6.3.
- **For grain dust,** transfer a 50.0-mL aliquot (V2) to a 250-mL flat bottom flask. Adjust the volume to 100.0-mL by adding 50.0 mL of distilled water. Proceed to Section 6.3.

6.2.2 Extraction: Corn and Soybean Oil

Into a 250-mL flat bottom flask, weigh 2.5 (±0.1) g (W_S) of oil. [For determination of procedural recovery, a (control) oil sample is fortified at this point, using a standard solution prepared in 1/1 ethanol/methyl acetate. See Section 5.2 above.] Proceed to Section 6.4.1 (derivatization step).

[Note: It is recommended to fortify oil samples using ≤ 0.2 mL of standard solution. If a volume ≥ 0.2 mL is used, evaporate the fortification solution solvent (e.g., using a rotary evaporator) before carrying out the derivatization step.]

6.3 Extract Clean Up Using Anion Exchange: [See also Section 11.0.]

Use anion exchange resin which has been conditioned as described in the Reagents Section 4.0. Slurry pack a calibrated[a] glass chromatography column with resin (~10.5 mm id), using distilled water to assist in transferring and packing the resin into the column. The following volumes of resin (± 0.5 mL) are used according to sample type:

- Corn Samples: **12 mL**
- Soybean Samples: **18 mL**

6.0 Analytical Procedure (Continued):

6.3 Extract Clean Up Using Anion Exchange: (continued)

[[a]Note: For convenience, calibrate the glass chromatography column prior to use. Add a measured volume of water to an empty column (containing a glass wool plug) and mark the volume level on the column exterior accordingly.]

6.3.1 Load the column with the 100.0-mL (total) aqueous extract aliquot from Section 6.2 [Note: V2 = 100.0 mL (50.0 mL for grain dust)]. Allow this solution to pass through the resin (gravity flow). At this point, wash the resin with 50 (± 5) mL of distilled water. Regulate the flow of column effluent as required using the column stopcock valve such that a flow rate of approximately one to two drops per second is attained. Discard this column eluate which contains only residual sample matrix. Proceed to Section 6.3.2.

6.3.2 Elute both the parent compound (HOE-039866) and metabolite compounds (HOE-061517 and HOE-099730) using 100 (±10) mL of 50% formic acid, collecting the eluate directly into a clean 250-mL flat bottom flask. As above, use a drip rate of about one to two drops per second.

Evaporate the formic acid eluate to dryness using a rotary evaporator (50-55°C). Operate the rotary evaporator at a moderately slow speed, which avoids excessive dispersion of the sample inside of the flat bottom flask. Add 10 (±1) mL of water to the dried sample extract and again evaporate to dryness as above to remove all residual formic acid. Proceed to Section 6.4.2 (derivatization step).

6.4 Derivatization:

6.4.1 Derivatization: Oil Samples

Add 6.0 (±0.5) mL of glacial acetic acid to the oil samples from Section 6.2.1. Sonicate at room temperature for approximately one minute. Add 24 (± 1) mL of trimethylorthoacetate reagent and a few glass beads. Mix by swirling with sonication. When mixing is complete, reflux the reaction mixture for 4.5 hours. Proceed to Section 6.4.3.

6.4.2 Derivatization: All Samples (except oil)

Add 3 (±0.5) mL of glacial acetic acid to the flat bottom flask containing the sample residue (from Section 6.3.2). Sonicate at room temperature for approximately one minute (or until all visible sample residue is dissolved and/or dislodged from the wall of the flask). Add 12 (±1) mL of trimethylorthoacetate and a few glass beads. Mix by swirling with sonication. [See also Section 11.0 (Derivatization).] When mixing is complete, reflux the reaction mixture for 4.5 hours. Proceed to Section 6.4.3.

6.4.3 After the reflux period, allow the sample to cool to room temperature. The procedure may be stopped at this point. Disconnect the flask from the reflux condenser. Add 15 (±1) mL of toluene. The procedure may also be stopped at this point (capping the flask for storage at room temperature). Otherwise, proceed to Section 6.5.

6.0 Analytical Procedure (Continued):

6.5 Reconstitution:

The cooled derivatized sample mixture (from Section 6.4.3) is reconstituted as follows. Evaporate the contents of the flat bottom flask to a final volume of approximately 2 mL (using a rotary evaporator operating with a water bath temperature of 45°C or an equivalent technique, e.g., Speed Vac apparatus). Add successive 15 (±1) mL portions of toluene and continue to repeat the evaporation procedure. At a minimum, three 15-mL portions of toluene are added and evaporated to an approximate final volume of 0.5 - 1 mL, to remove all traces of the derivatization solution. **It is important that the solution is not evaporated to dryness during the reconstitution procedure. The derivatized parent and/or metabolite compounds may be lost should this occur.**

The final volume of the extract is adjusted to *no more than* 4 mL of toluene.

6.6 Post Derivatization Clean Up:

The derivatized sample extract undergoes additional clean up prior to GC/FPD analysis. This final clean-up step is carried out using commercially available silica gel solid phase extraction (SPE) cartridges.

The SPE clean-up technique requires verification of the retention and elution characteristics of the silica gel material(s) utilized. The characterization procedure is described in Section 6.6.2.

6.6.1 Silica Gel SPE Cartridge Clean Up:

Silica Gel SPE cartridges are typically used in conjunction with a vacuum box assembly as described in the Equipment Section (3.0). In addition, always refer to the manufacturer's operating instructions for the particular device utilized. See also Section 6.6.2.

Immediately before use, the SPE cartridges are conditioned by passing ~10 mL of degassed (sonicated) methyl acetate/toluene (1/1 v/v) through each cartridge. Do not allow the cartridges to become dry at this point.

Add 4.0 (± 0.1) mL of methyl acetate to the toluene extract from Section 6.5. The solution is then sonicated to dislodge any residual sample material which may adhere to the flask.

To load the SPE cartridge, attach a 100 mm syringe needle to a 10 mL disposable (polypropylene) syringe [see Equipment Section 3.0 (item 17)] and draw the sample into the syringe. Adjust the final volume in the syringe to 8.0 (±0.2) mL with toluene, achieving a 1/1 methyl acetate/toluene solution. Save the empty flask at this point.

Invert the syringe (plunger end down), remove and save the needle. Attach a 0.45-µm disposable filter to the tip of the syringe. Load the contents of the syringe onto the top of the SPE cartridge using the syringe/disposable filter apparatus. Elute to waste but do not allow the cartridge to become dry.

6.0 Analytical Procedure (Continued):

6.6.1 Silica Gel SPE Cartridge Clean Up: (continued)

Wash the (saved) empty flask with 10 mL of pure methyl acetate. Draw this final wash solution into the disposable syringe (filter removed and saved) via the re-attached 100 mm syringe needle. Reconnect the filter assembly (needle removed) and apply this wash solution to the SPE cartridge. Elute to waste; the cartridge is dried using vacuum suction at this point. Proceed to Section 6.6.1a. [Note also alternate procedure: Section 11.0.]

6.6.1a Elute the HOE-064706 and/or HOE-070951 analytes with 5.5 (±0.1) mL of methanol/methyl acetate (1/1 v/v) solvent.

Collect approximately 5 mL of eluate in a clean collection tube (e.g., Item 9, Section 3.0) which is typically contained in the vacuum box assembly. Transfer the eluate into a 50-mL round bottom flask (see Note below). Wash the tube with approximately 10 mL of methyl acetate. Add the wash to the round bottom flask. Proceed to Section 6.6.3.

[Note: If solvent exchange (6.6.3) is to be carried out by an alternate technique (e.g., using an N-evap concentrator), a more appropriate collection vessel may be used in place of the round bottom flask.]

6.6.2 Silica Gel SPE Cartridge Characterization:

Before analyses are conducted, the SPE cartridges must be characterized. Such characterization is necessary to account for possible variability in silica gel raw materials as well as to verify the conditioning and elution procedures described above.

To characterize column performance, add appropriate aliquots of HOE-070951 and HOE-064706 standard solutions to a control sample (not previously fortified) which has undergone procedural work-up through the derivatization/reconstitution steps (Section 6.5). The fortification level of HOE-070951 and HOE-064706 should provide a representative amount of test compound (i.e., 200-250 ng in a 5.0 mL final GC/FPD test solution volume). Continue the analytical procedure (Section 6.6.1) and observe the "recovery" of the test compounds after GC/FPD analysis (see below). The methanol/methyl acetate solvent strength and/or elution volume may need to be optimized to maximize recovery (90-100%) for both test substances. If this is necessary, adjust the final eluate volume accordingly (Section 6.6.3) to obtain a 5.0 mL (typical) volume in methyl acetate solvent. [Note that the GC/FPD calibration standards are prepared in methyl acetate.]

6.0 Analytical Procedure (Continued):

6.6.3 Solvent Exchange:

Using an N-Evap Analytical Concentrator operating at 50°C or a rotary evaporator with a water bath operating at 35°C (or equivalent apparatus); evaporate the silica gel column eluate to approximately 0.5 - 1.0 mL. **[Caution: Care must be taken to avoid excessive evaporation; do not evaporate to a volume < 0.5 mL at any point.]**

Using methyl acetate, quantitatively transfer this solution to an appropriate (e.g., 5.0 mL) volumetric flask or graduated tube and adjust to final volume (V3) with methyl acetate. Typically, a final volume of 5.0 (±0.1) mL (2.5 mL for grain dust) provides adequate sensitivity for GC/FPD analysis.

[Note: The volume of the solution for GC analysis may be reduced to compensate for particular GC/FPD sensitivity limitations. However, avoid excessive evaporation (i.e., to volumes < 0.5 mL) to minimize evaporative losses of derivatized compounds.]

7.0 Determination by Gas Chromatography:

The derivatized parent [HOE-039866 → HOE-064706] and derivatized metabolites [HOE-099730 → HOE-064706 and HOE-061517 → HOE-070951] are determined using gas chromatography with flame photometric detection (P-mode). A "megabore" (0.53 mm id) fused silica capillary column application is used. Note that the exact final volume (V3) of the GC test solution must be known, typically 5.0 (±0.1) mL in methyl acetate solvent.

7.1 Gas Chromatography Instrumentation:

A Varian Model 3400 Gas Chromatograph equipped with a Flame Photometric Detector operating in the phosphorus selective mode (P-mode) is adequate for determination of 0.05 ppm levels of the test compounds. Other GC/FPD instrumentation systems which are found to have equivalent or superior performance [e.g., signal-to-noise (S/N), repeatability, and sensitivity] may be utilized. For method validation, data acquisition was carried out using the IBDH (In-Board Data Handling) capabilities of the Varian instrument.

7.1.1 Chart Recorder/Electrometer::

Operate in the linear mode (for phosphorus). For manual peak height measurement, attenuate as required to obtain chart recorder peak heights ≥10 mm (S/N ≥3) for a level corresponding to 0.05 ppm of each test compound, determined as HOE-035956 equivalents. When using an electronic integrator, the S/N ratio must also be ≥ 3 for the chromatography peak(s) of interest, with a minimum peak height of 5 mm displayed on the chromatogram.

7.0 Determination by Gas Chromatography (Continued):

7.1.2 Limit of Quantitation (LOQ):

The validated LOQ of this method is 0.05 ppm of each test compound (quantified as HOE-035956 equivalents). At this level: i) fortification recoveries should fall within a range of 70% to 120%, ii) the analyte GC/FPD signal should be ≥ 3 times the background GC/FPD signal, and iii) the intra-laboratory reproducibility as indicated by the relative standard deviation (n ≥ 3) obtained from replicated analyses should fall within 20% (rel.) of the averaged result.

7.2 DB-Wax Megabore Capillary Column Chromatography:

Column: 15 meter x 0.53 mm id fused silica (megabore) column with 1.0 micron DB-Wax stationary phase (J&W Scientific).

Temperatures:

Inlet:	225°C
Detector:	230°C

Column Temperature Program (e.g.):

Initial Temp:	145°C
Initial Time:	1.5 min.
Rate1:	2.5°C/min.
Final Temp1:	150°C
Rate2:	5°C/min.
Final Temp2:	240°C
Final Time:	5 min.

Gas Flows:

Carrier:	20 mL/min. (Helium)
Make-up:	10 mL/min. (Nitrogen)
Detector:	85-100 mL/min. (Hydrogen)
	70-120 mL/min. (Air)

Injection Volume: 5 µL (typical) - Vinj

Approximate Retention Time: 6 - 7 min. (HOE-070951)
21 - 22 min. (HOE-064706)

7.0 Determination by Gas Chromatography (Continued):

7.2 DB-Wax Megabore Capillary Column Chromatography (Continued):

[Note: The above component retention times are estimated for a 15-meter DB-Wax GC column which is operated as described above. Other operating conditions may be used according to the specifications of alternative instrumentation and/or laboratory variables (including GC column length, supplier, etc.). Although permissible within the scope of this methodology, alternative GC/FPD operating conditions must, of course, produce adequate performance (i.e., reproducible retention times, sensitivity, S/N, and component resolution). However, the use of megabore fused silica GC column lengths > 15 meters is not recommended.]

Representative chromatograms are included in Appendix I.

7.3 GC/FPD Calibration:

The GC/FPD response is calibrated as follows using the representative GC/FPD operating conditions outlined in Section 7.2 (above).

Determine the GC/FPD response in peak area (or height) units for HOE-064706 and/or HOE-070951 for a series of analytical standards prepared as described in Section 5.1.

The lowest level analytical standard must correspond to a sample residue level of approximately 50% to 70% of the 0.05 ppm quantitation limit.

For example, assuming a 12.5 g sample (V1 = 500.0 mL), a sample aliquot of 100.0 mL (V2), a final volume (V3) of 5.0 mL, and a 5 µL injection volume (Vinj), successful GC/FPD analysis of a 0.025 ng/µL standard is required to approximate 0.05 ppm of test compound. [All concentration levels are expressed as HOE-035956 equivalents.]

During each analytical sequence, a standard calibration curve is drawn and a linear least squares regression calculation is carried out:

$$Y = mX + b$$

$$X = \frac{Y - b}{m}$$

where:

- Y = analyte (GC/FPD) peak area (or height) from the injected sample
- m = slope of the regression line (X coefficient)
- X = amount of analyte found in the sample (ng)
- b = constant (Y-intercept)

7.0 Determination by Gas Chromatography (Continued):

For verification of stable GC/FPD response, construct a standard curve which includes the levels of interest. [See Note below]. Typical calibration curves are provided for HOE-064706 and HOE-070951 in Appendix II. Every 2 to 3 sample injections within an analytical sequence are to be followed by an analytical standard. This practice serves as an ongoing "quality control" check of detector sensitivity/drift and component retention time (column) stability. Always analyze a calibration standard before the first analytical sample and after the last analytical sample within a sequence.

Residue results must not be determined by extrapolation of calibration data outside of the concentration range of the analyzed calibration standards (using ±10% tolerance, typically).

[Note: Before standards and/or samples are analyzed within a sequence, two to three sample injections into the GC/FPD are recommended. These preliminary pre-sequence analyses are carried out to condition the GC/FPD system (e.g., to mask active sites).[2,3] Instrument response should be stabilized such that the GC/FPD peak areas (or heights) from replicate injections ($n \geq 3$) agree to within ±10% relative.]

7.4 Sample Analysis:

Inject an appropriate aliquot (typically ≤ 5 µL) of the prepared sample as obtained from Section 6.6. Note the injection volume (Vinj). For practical considerations, the use of equivalent injection volumes for standards and samples is highly recommended.

Compute the integrated (or otherwise accurately measured) peak areas (or heights) obtained for HOE-064706 and/or HOE-070951 in the chromatogram. Compare the component peak area (or height) value with the analytical standard amount found on the corresponding calibration curve (Section 7.3). Both samples and standards must be analyzed under stabilized GC conditions and within the same analytical sequence.

For purposes of this example calculation, the amount (Amt) of analyte found from the calibration curve is expressed as nanograms.

8.0 Calculation of the Residue:

8.1 Percent Recovery: Sample residue is determined as follows:

Analyte Measured (GC/FPD)	Residue Compound Determined
HOE-070951	HOE-061517 - Metabolite
HOE-064706	HOE-039866 - Parent or HOE-099730 - Metabolite

[Note that the GC/FPD signal (chromatography peak) corresponding to the reference substance HOE-064706 determines combined residue of both parent and N-acetyl glufosinate metabolite.]

8.0 Calculation of the Residue: (Continued)

Following the GC/FPD analysis described in Section 7.4, determine the residue of test compound in the sample as follows:

Parts per million (ppm) of HOE-035956 (free acid) equivalents = Amt ÷ B

where: Amt = ng (X) of HOE-064706 or HOE-070951 found from the standard curve, expressed as HOE-035956 equivalents (See 7.3)

B = mg of sample injected

$$= \frac{Ws\ (g) * V2\ (mL)}{V1\ (mL)} * \frac{Vinj\ (\mu L)}{V3\ (mL)}$$

Percent recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{ppm found as HOE-035956 equivalents} * 100}{\text{ppm added as HOE-035956 equivalents}}$$

9.0 Quality Control Procedures:

9.1 Laboratory Fortifications:

To assure the quality of the laboratory data, laboratory fortifications are to be run along with each set of residue samples. These fortified samples should cover the range of expected residues in the set and at least 10% of the set must be laboratory fortifications. Spiking of quality control samples should be directly onto the analytical sample, e.g., prior to extraction. Preparation of fortification standards is described in Section 5.2. Generally, spiking volumes may range from 100 µL to 1 mL using the 10 µg/mL spiking solutions of HOE-039866 (or HOE-090532) + HOE-061517 or HOE-061517 + HOE-099730 (or HOE-085355). Spiking volumes ≤ 0.2 mL are recommended for oil samples.

For example, 0.50 mL of Stock Solution F [10.0 µg/mL (each) of HOE-039866 and HOE-061517, expressed as HOE-035956 equivalents] added to 12.5 g of (control) sample corresponds to 0.40 ppm of HOE-035956 (free acid) equivalents of each compound, in the form of HOE-039866 (parent) and HOE-061517 (metabolite).

9.2 Sample Storage:

All residue samples should be stored frozen until analysis. After sampling for analysis, the remaining sample should be promptly re-frozen and stored until authorized for disposal.

10.0 Confirmatory Techniques:

At least two sources of information provide confidence in the identification the GC/FPD peaks assigned to the derivatized parent and metabolite test compounds. First, analysis cross-checks carried out using four different GC columns have provided results consistent with the assigned GC peaks.(1)

Secondly, capillary GC/MS analysis has been shown to yield characteristic EI mass spectra for both the derivatized parent and metabolite compounds. The respective capillary GC/MS retention times of the verified test compounds were found to be in line with GC/FPD chromatography retention times. In addition, GC/MS operation in the Selected Ion Monitoring mode can be used to verify characteristic relative mass abundance ratios using the following ions: m/z 149 and 165 (for HOE-070951) and m/z 150 and 192 (for HOE-064706). A Hewlett Packard 5890A GC interfaced to a Model 5970 Mass Selective Detector has been utilized successfully for such confirmatory analytics.(4)

11.0 Modifications and/or Potential Problem Areas:

Fortification:

For QC and/or troubleshooting purposes, samples should be fortified with reference standards containing either the parent (e.g., HOE-039866) or the N-acetyl metabolite (e.g., HOE-099730). If added as a mixture, *recovery* as individual components cannot be determined because these substances are derivatized to a common moiety (HOE-064706). As a result, combined residue of parent and N-acetyl metabolite are determined from the measurement of one chromatography peak. Fortification solutions containing either the parent compound or its N-acetyl glufosinate metabolite (prepared as described in Section 5.2) are therefore recommended.

Extraction:

With consideration of potential sample variability, the *sample moisture content is generally accounted for* by adjusting the final sample extraction volume (V1) to 500 (± 1) mL. This step, combined with the use of a 12.5-g sample of homogeneous material, also minimizes bulk volume contribution from the sample itself (e.g., as may be significant for samples of hulls). Equivalent procedures which adequately account for sample moisture content as well as bulk sample volume may be utilized on a case-by-case basis. For example, a 25.0-g corn grain sample can be successfully analyzed, using a 25.0-mL aliquot (V2) taken from a 250.0-mL (V1) total extract volume. However, it is essential that the sample extract aliquot (V2) which is carried through the procedure accurately reflects the analyzed sample mass. Indications of the moisture content of representative samples are provided below:

11.0 Modifications and/or Potential Problem Areas: (Continued)

COMMODITY	PERCENT DRY MATTER
Field Corn	
Grain	88
Forage	40
Fodder (stover)	83
Milled Products	85
Soybeans	
Seed	89
Meal	92
Hulls	90
Forage	35
Hay	85
Ref: Updated Livestock Feeds Table for Subdivision O (Residue Chemistry) of the Pesticide Assessment Guidelines (Proposed Changes; Issued June 2, 1994), US Environmental Protection Agency.	

Anion Exchange Columns

As required, the elution profiles of the anion exchange columns may be experimentally verified (or calibrated) using the appropriate reference substances, including C-14 materials (if available). In any case, column profiles should be established with fortified matrix extracts rather than solvent blanks. This will appropriately account for extracted sample matrix components which may affect column characteristics.

On occasion, very dry hay samples may require anion exchange clean up of a 50.0-mL extract aliquot (V2), i.e., instead of a 100.0-mL aliquot. In this case, the procedure used for the ***grain dust*** samples should be followed. This troubleshooting option is recommended, as may be required, to optimize procedural recovery of parent and metabolite compounds.

Derivatization:

Variable and/or low derivatization yields may be the result of spurious water contamination of the derivatization mixture. All reagents and glassware used to carry out the derivatization reaction must be dry. Typically, water contamination in this step more adversely affects the recovery of HOE-061517 (i.e., compared to the recovery of parent compound).

Certain sample matrices, i.e., corn grain, soybean seed and meal, may require additional time for sonication prior to derivatization (Section 6.4). Sonication should continue until all sample residue dissolves, including material which may adhere to the wall of the derivatization flask. In some cases, a five-minute sonication with acetic acid followed by a five-to-ten minute sonication after addition of trimethylorthoacetate. The sample should be capped at all times during this procedure.

11.0 Modifications and/or Potential Problem Areas: (Continued)

Silica Gel Clean-up Procedure

An alternate technique for loading the silica gel SPE cartridge (Section 6.6.1) is described below:

Transfer the toluene extract (Section 6.5) to a 10-mL graduated cylinder, adjust the volume to 4.0 mL with toluene, and return this solution to the original flask. Add 4.0 mL of methyl acetate. Filter this solution through a disposable filtration column (see Section 3.0, item no. 14), which is "piggy-backed" onto the silica gel clean-up column. Rinse the flask with 10 mL of methyl acetate and elute this solution through the filtration column and SPE cartridge. Both the loading and rinse solutions are eluted to waste. Remove the filtration column and dry the SPE cartridge using vacuum suction as described in the method. Proceed to Section 6.6.1a.

Chromatography:

Because analyses are carried out in the presence of some residual matrix components, a gradual deterioration of GC column performance may be experienced. For megabore fused silica capillary GC columns, performance may often be restored by cutting and discarding the first six to twelve inches of GC column (injector end). Otherwise, the use of a retention gap of pure fused silica may be advantageous.

It is also important to utilize a gas chromatography system (i.e., considering injector, column, and detector components) which has been adequately conditioned (to mask active sites) just prior to initiating a sample analysis sequence. See "Note" in Section 7.3. Further, GC instrument problems may individually or collectively contribute to poor quantitative data. Factors to consider in this regard include i) contaminated carrier gas (e.g., with oxygen), ii) excessive GC column stationary phase bleed, iii) non-optimized column connections (e.g., fractured column ends, exposed external column coating material, etc.), and/or iv) minor gas leaks.

12.0 Safety:

Derivatization reactions (Section 6.4), evaporation steps, etc. should be carried out in a fume hood or high efficiency condensers must be used (to prevent escape of reagent fumes into the laboratory).

14.0 Method Flow Diagram:

AQUEOUS EXTRACTION

[All corn and soybean samples except oil]
12.5 g (1 X 200 mL) → Dilute to 500 mL (aq) → [Centrifuge soybean seed, hulls, or meal extract]

↓

VACUUM FILTRATION

Filter → Take 100-mL aliquot of filtrate [For "dust" samples, take 50-mL aliquot → Dilute to 100 mL (aq)]

↓

ANION EXCHANGE COLUMN CLEAN UP

[Resin Volume: Corn Samples (12 mL); Soybean Samples (18 mL)]
Load with 100-mL (total) extract aliquot → Wash with 50 mL distilled water → Elute with 100 mL formic acid (50% aq) → Rotovap to dryness

↓

DERIVATIZATION

Oil Samples	All Other Samples
Sonicate 2.5 g sample in 6.0 mL glacial acetic acid → Add 24 mL trimethylorthoacetate → Sonicate → Reflux 4.5 h. [Optional Stopping Point]	Add 3 mL glacial acetic acid → Sonicate → Add 12 mL trimethylorthoacetate → Reflux 4.5 h. [Optional Stopping Point]

↓

SILICAL GEL SPE CARTRIDGE CLEAN UP

Solvent exchange into toluene → Add 4.0 mL methyl acetate → Adjust volume to 8.0 mL with toluene → Load onto SPE cartridge (via 0.45 µm filter) → Wash with methyl acetate → Elute with 1/1 methanol/methyl acetate

↓

GC/FPD DETERMINATION

Solvent exchange into methyl acetate → Analyze final extract using gas chromatography with flame photometric detector (P-mode): LOQ = 0.05 ppm glufosinate free acid equivalents

METHOD APPENDIX I

Representative Chromatograms

Reference: ABC/Pan-Ag Division Study No. 94354

Note: Figures are shown to illustrate typical analysis results. Therefore, the test samples represent a variety of field trial locations. Chromatograms are reduced and/or annotated (as necessary) for clarity of presentation.

Figure 1 Standards: HOE-070951 Plus HOE-064706
0.015 µg/mL

File Name: F3001 (Reference: Method Appendix II)

Standard ID: IGC-1A (Reference: Method Appendix III)

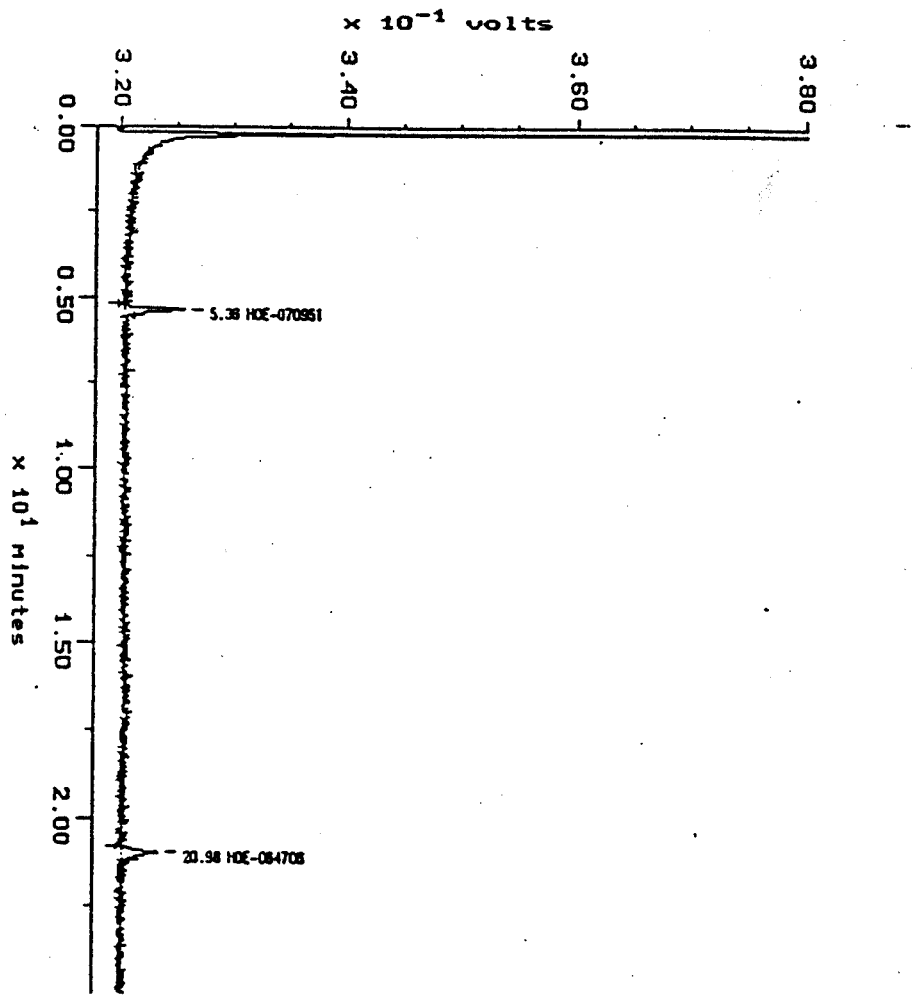


Figure 2 Standards: HOE-070951 Plus HOE-064706
0.020 µg/mL

File Name: F3002 (Reference: Method Appendix II)

Standard ID: IGC-2A (Reference: Method Appendix III)

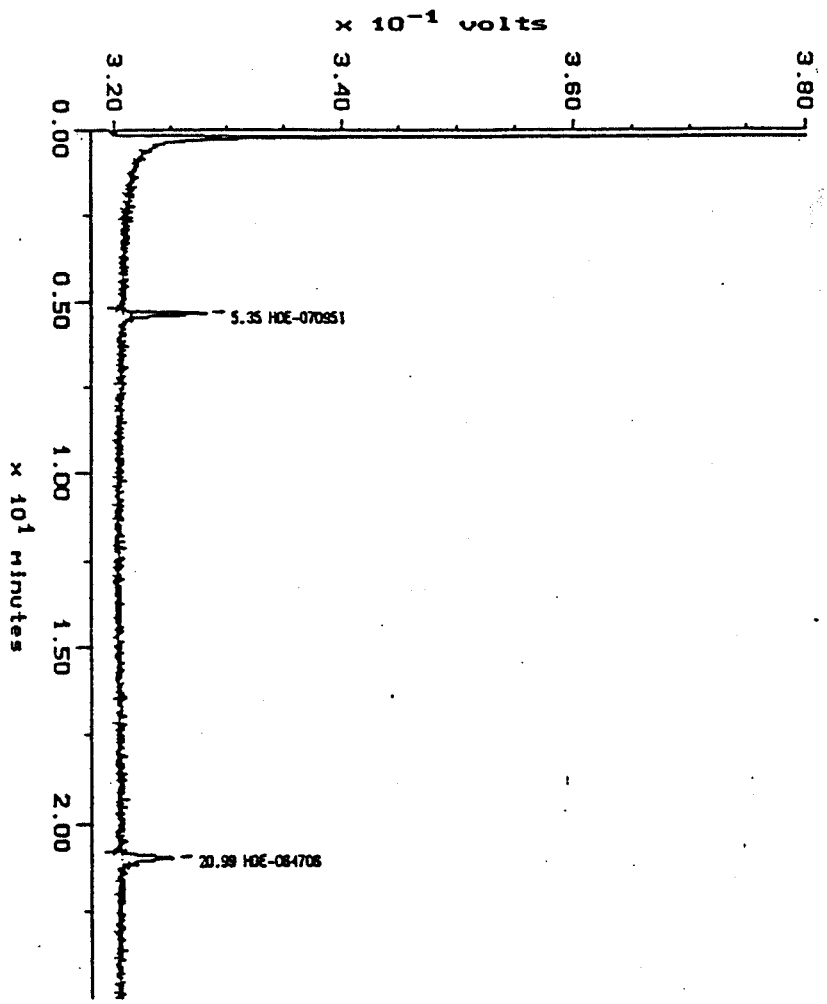


Figure 3 Standards: HOE-070951 Plus HOE-064706
0.050 µg/mL

File Name: F3005 (Reference: Method Appendix II)

Standard ID: IGC-3A (Reference: Method Appendix III)

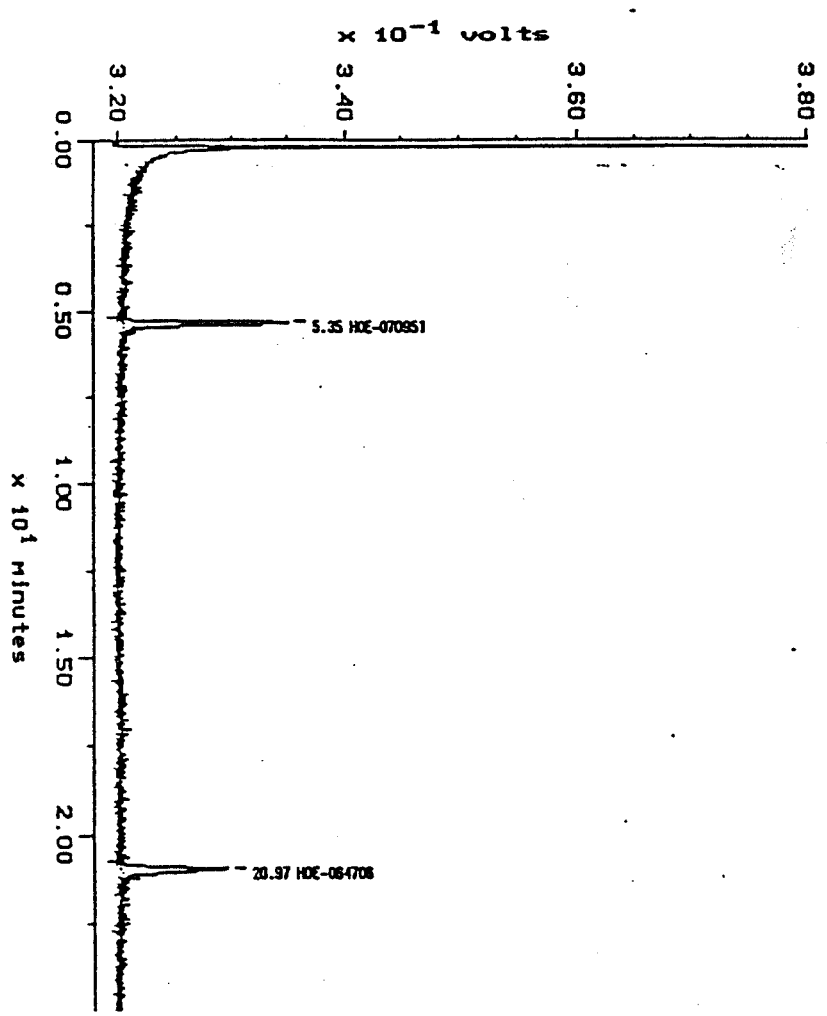


Figure 4 Standards: HOE-070951 Plus HOE-064706
0.100 µg/mL

File Name: F3008 (Reference: Method Appendix II)

Standard ID: IGC-4A (Reference: Method Appendix III)

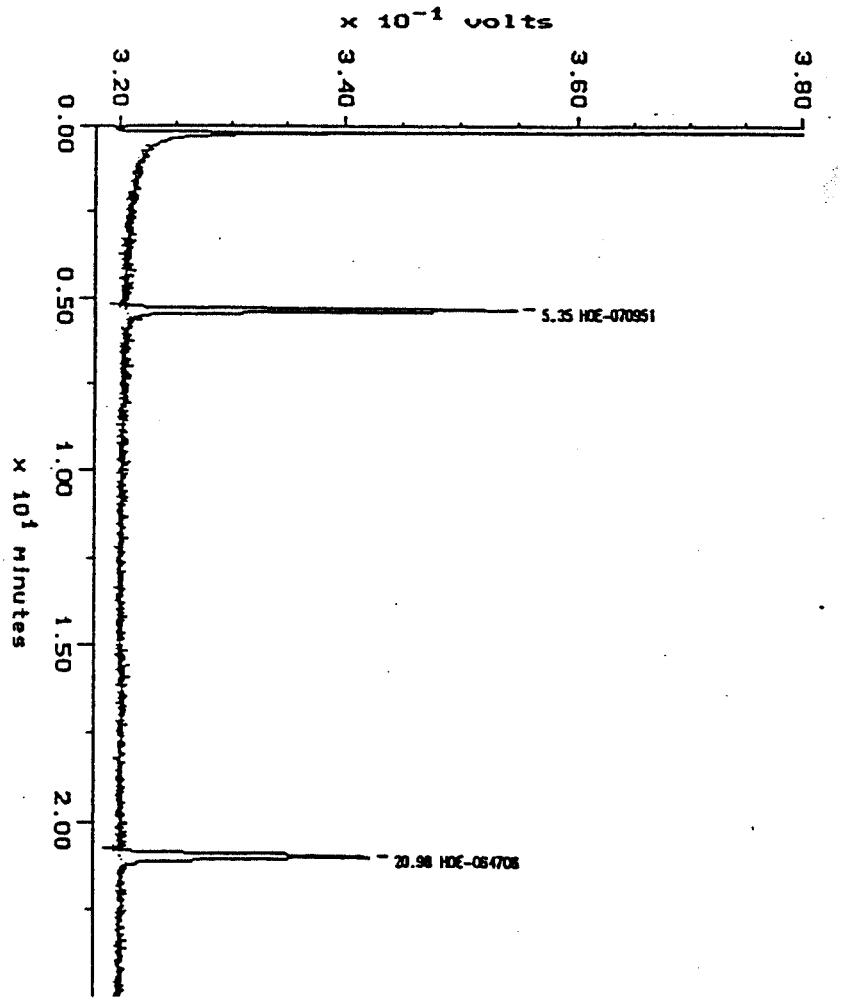


Figure 5 Standards: HOE-070951 Plus HOE-064706
0.200 µg/mL

File Name: F3011 (Reference: Method Appendix II)

Standard ID: IGC-5A (Reference: Method Appendix III)

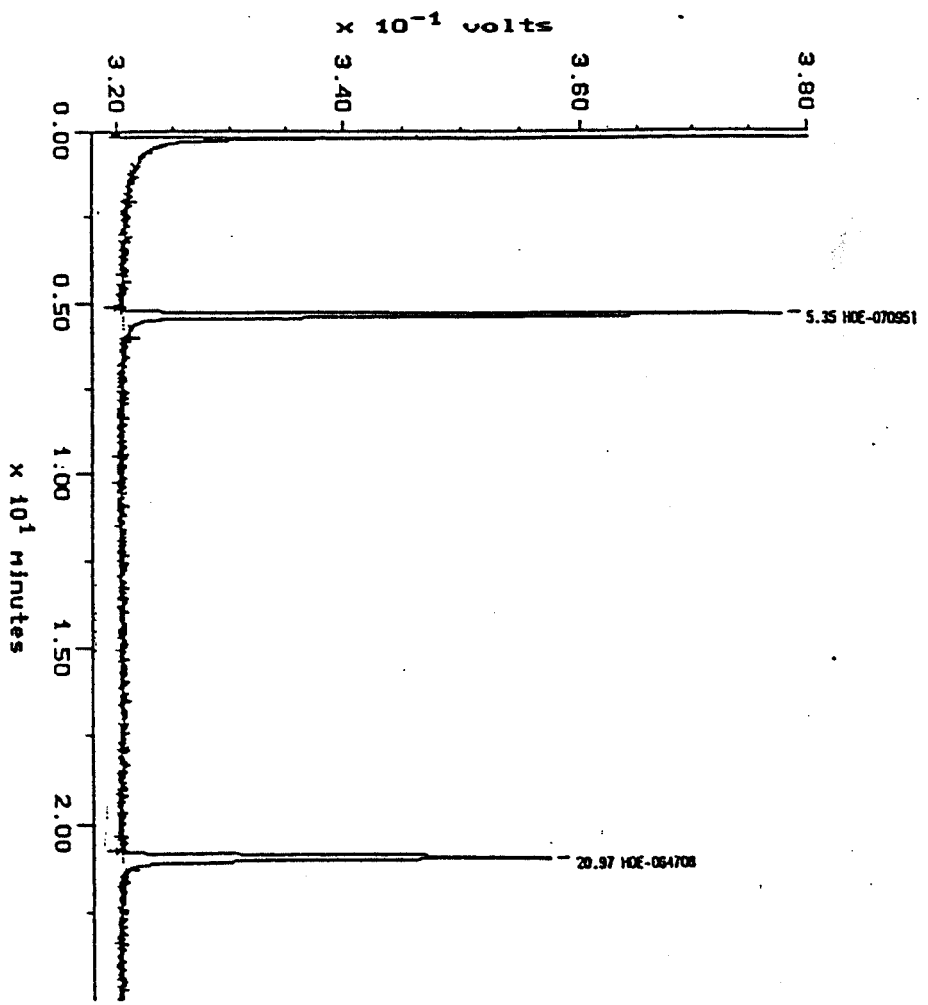


Figure 6 Standards: HOE-070951 Plus HOE-064706
0.300 µg/mL

File Name: F3014 (Reference: Method Appendix II)

Standard ID: IGC-6A (Reference: Method Appendix III)

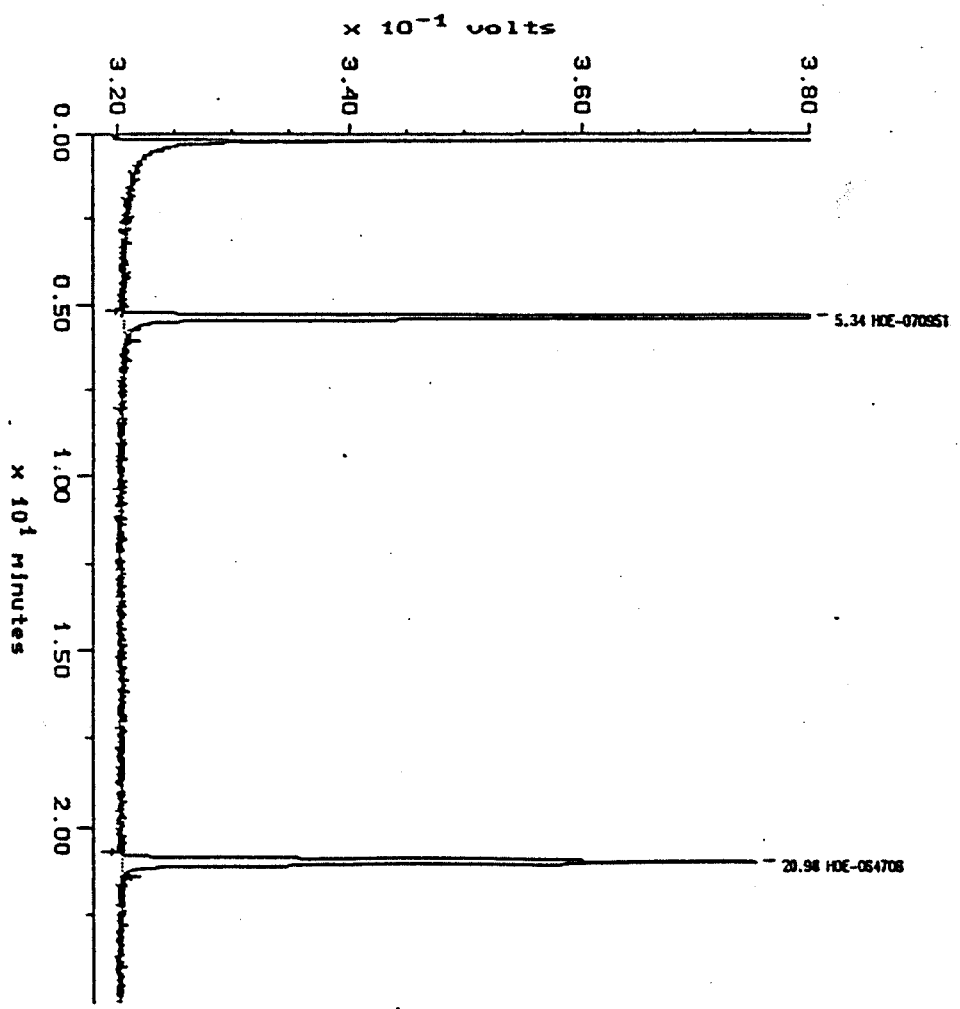


Figure 7 Control Corn Forage
<0.05 ppm HOE-061517 or
HOE-039866/HOE-099930 found

Sample Lab ID: 354F3-1 (Reference: Method Appendix III)

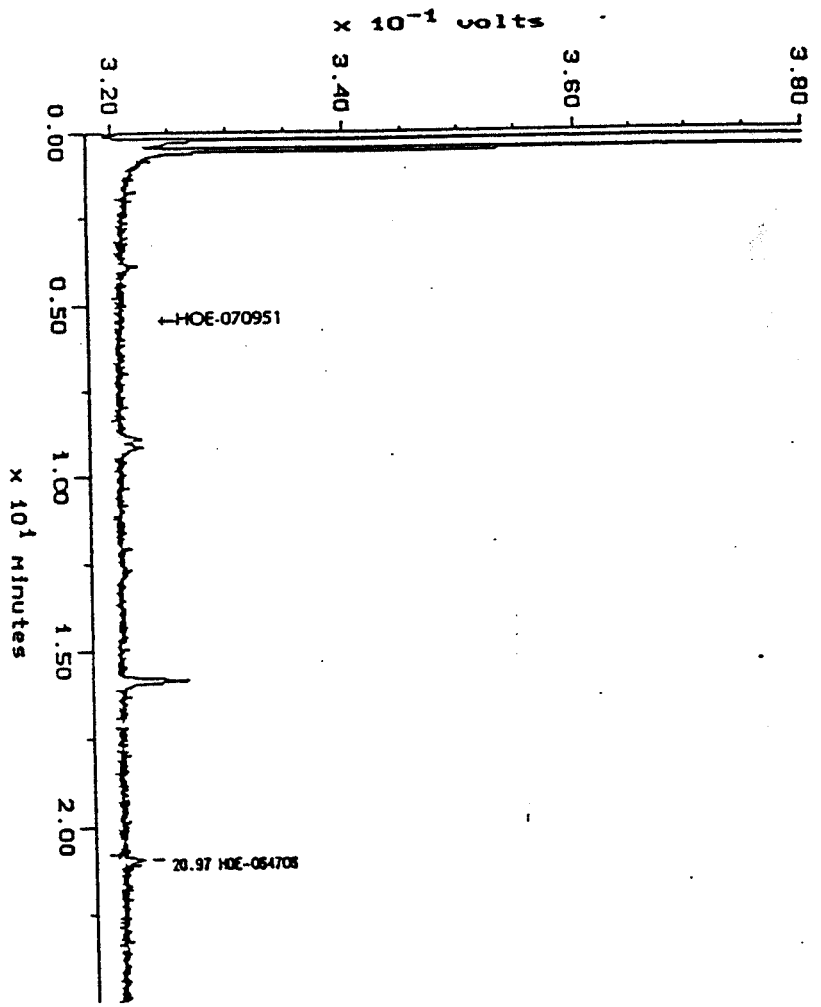


Figure 8 Control Corn Forage
Fortified at 0.05 ppm with HOE-061517
and at 0.0502 ppm with HOE-099730
Recovery = 85.8% for HOE-061517, 98.6% for HOE-099730

Sample Lab ID: 354F3-2 (Reference: Method Appendix III)

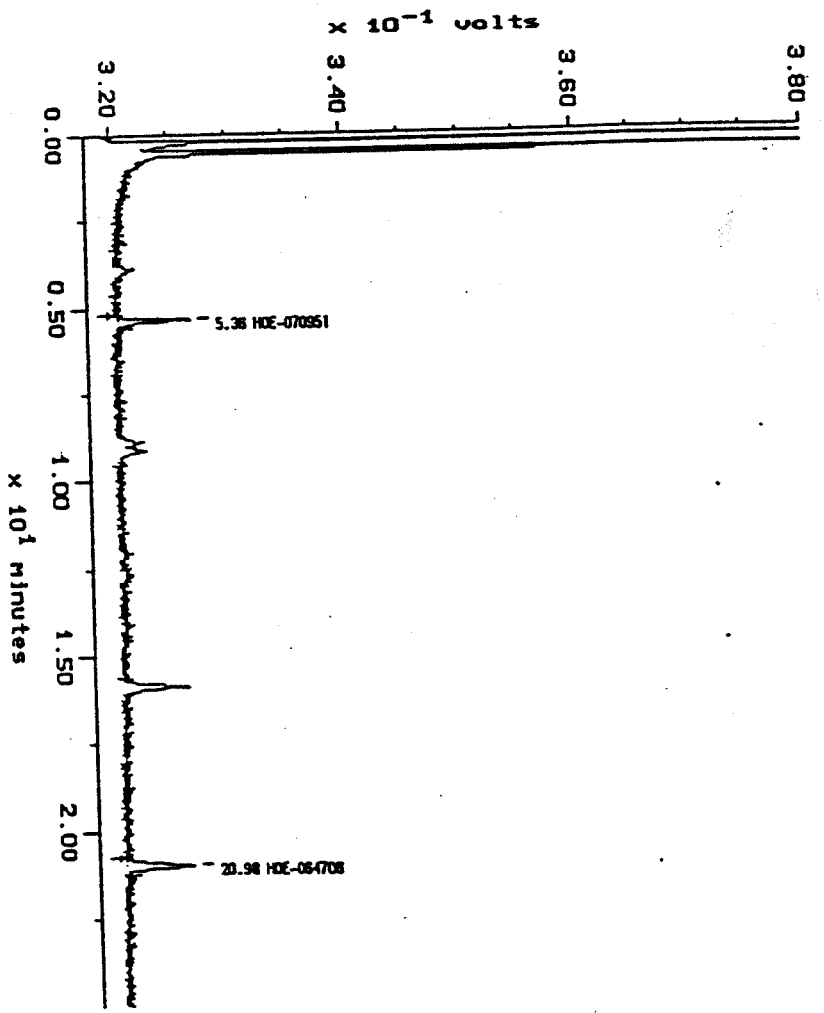


Figure 9 Control Corn Forage
Fortified at 0.10 ppm with HOE-061517
and at 1.01 ppm with HOE-039866
Recovery = 83.3% for HOE-061517, 87.7% for HOE-039866

Sample Lab ID: 354F3-8 (Reference: Method Appendix III)

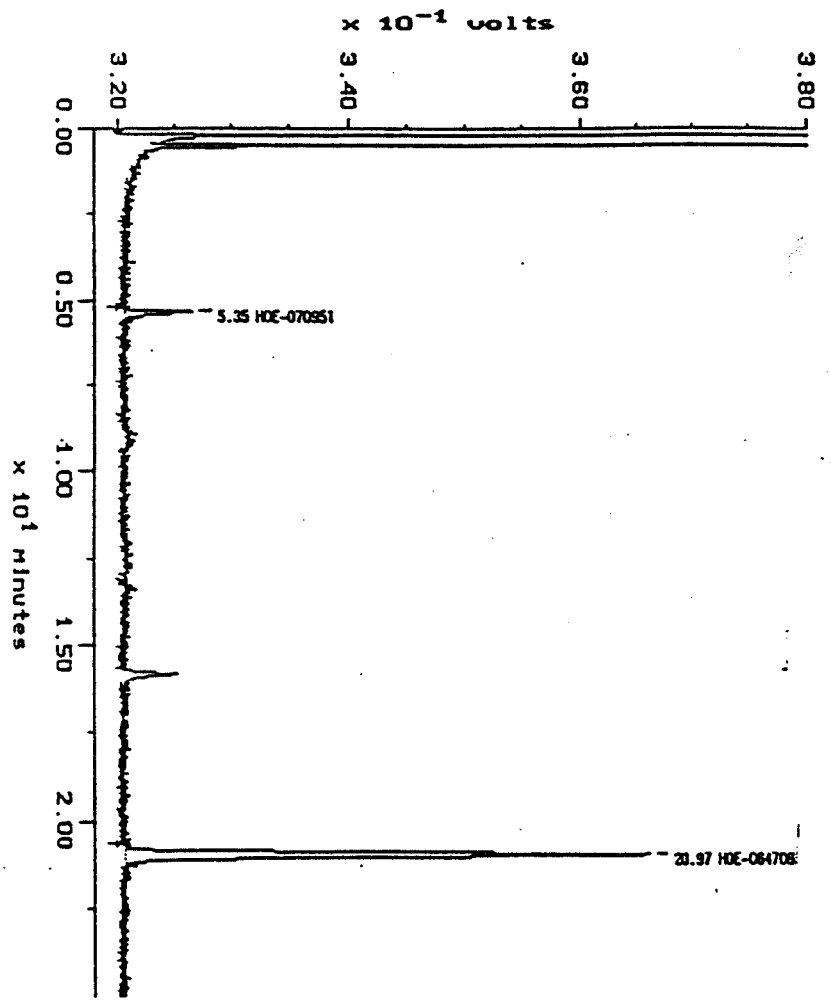


Figure 10 Treated Corn Forage
<0.05 ppm of HOE-061517
and 0.40 ppm of HOE-039866/HOE-099730 found

Sample Lab ID: 354F3-9 (Reference: Method Appendix III)

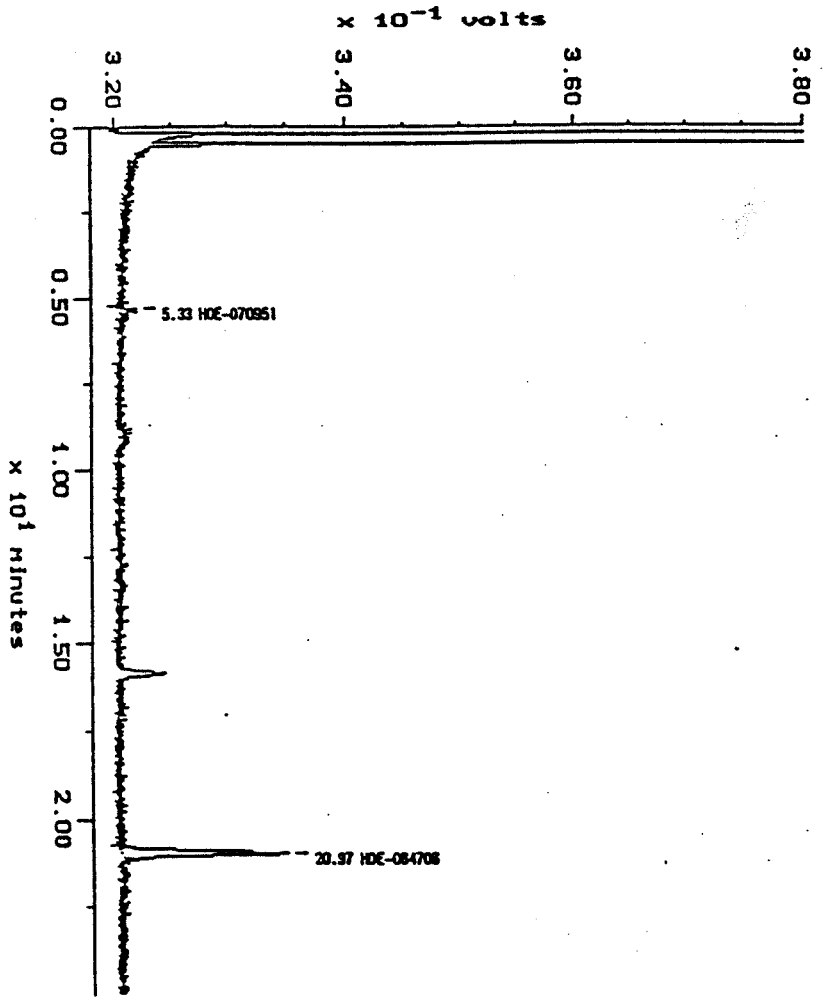


Figure 11 Control Corn Silage
<0.05 ppm HOE-061517 or HOE-039866/HOE-099730 found

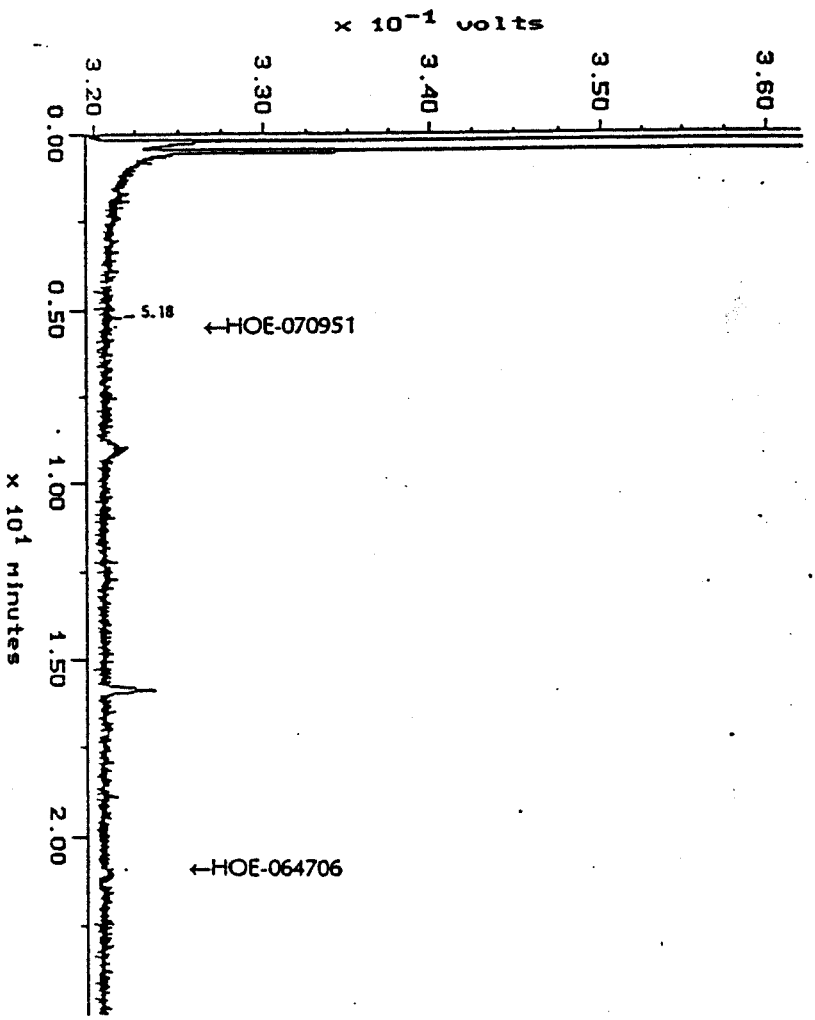


Figure 12 Control Corn Silage
Fortified at 0.20 ppm with HOE-061517
and at 2.02 ppm with HOE-039866
Recovery = 88% for HOE-061517, 93% for HOE-039866

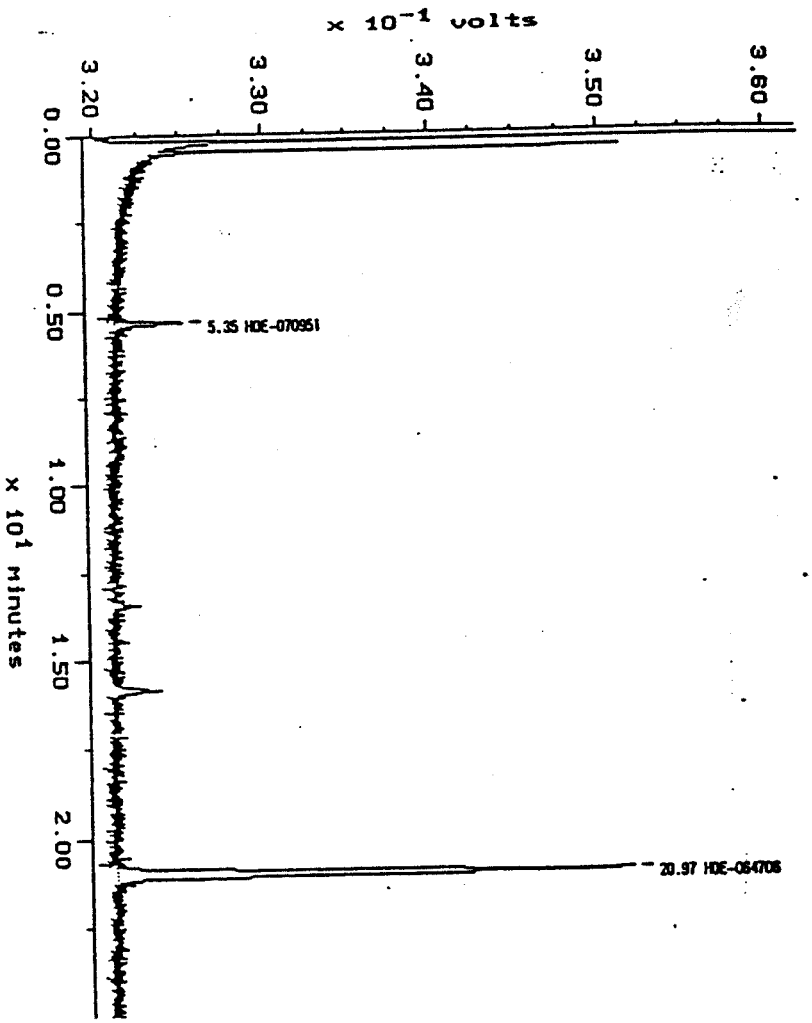


Figure 13 Control Corn Silage
Fortified at 0.05 ppm with HOE-061517
and at 0.05 ppm with HOE-099730
Recovery = 73% for HOE-061517, 89% for HOE-099730

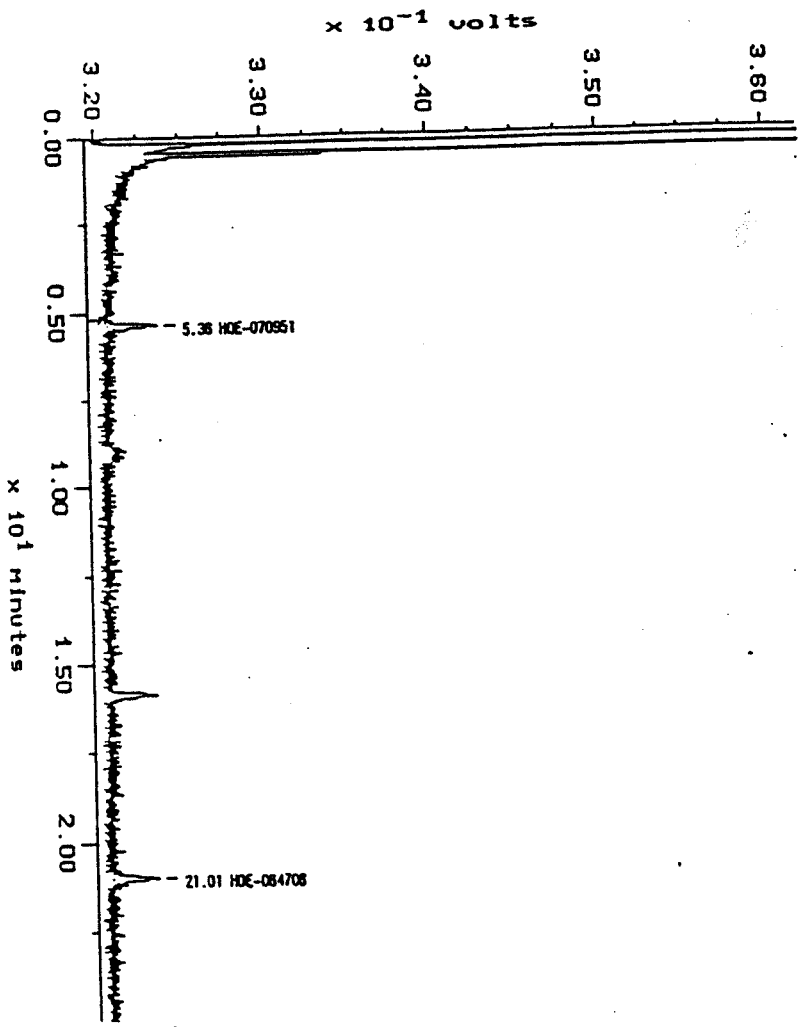


Figure 14 Treated Corn Silage
< 0.05 ppm of HOE-061517
and 0.12 ppm with HOE-039866/HOE-099730 found

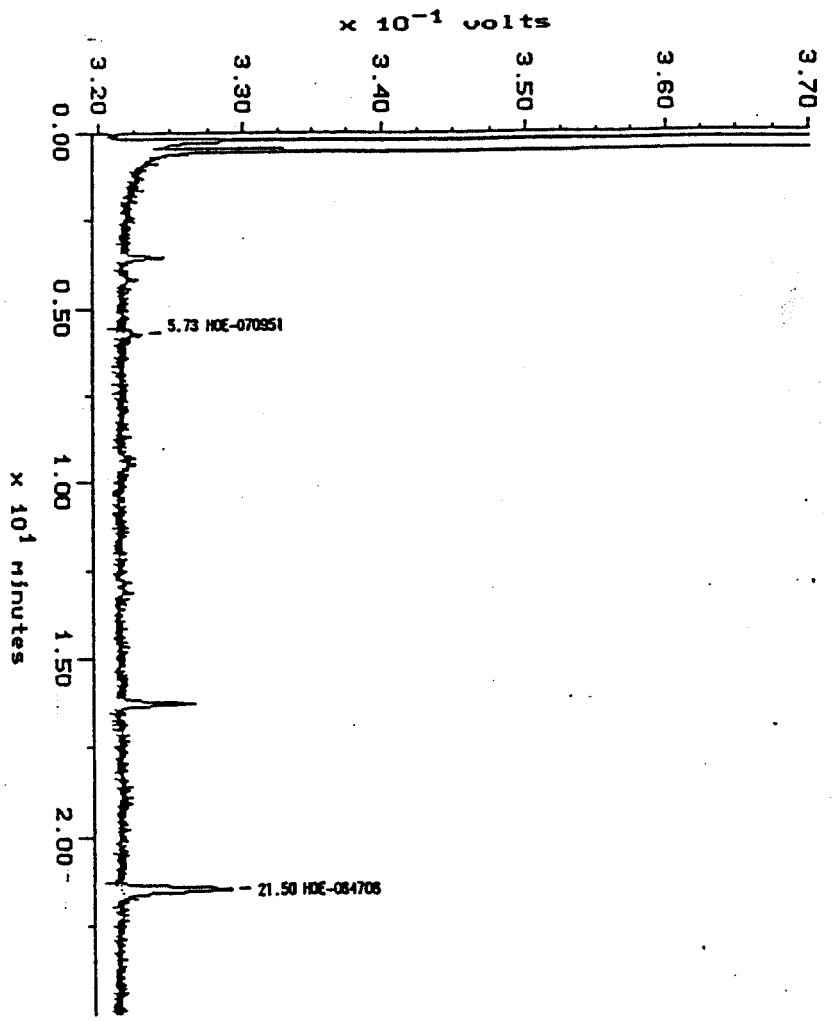


Figure 15 Control Corn Fodder
< 0.05 ppm HOE-061517
or HOE-039866/HOE-099730 found

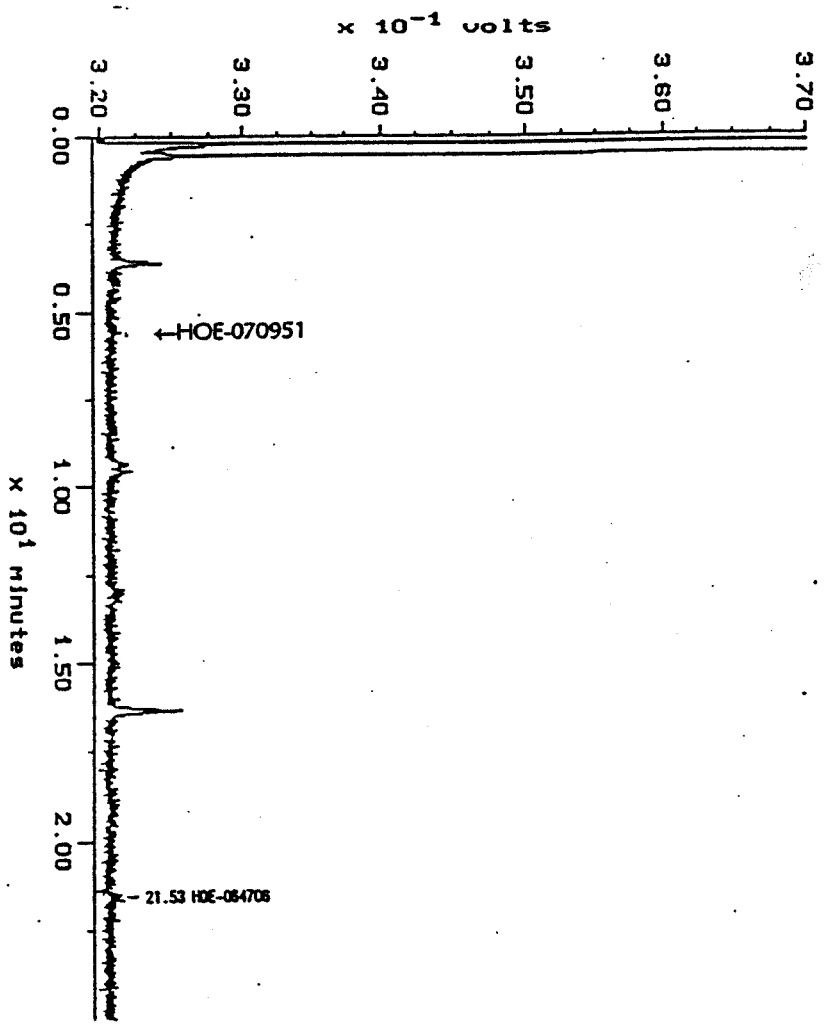


Figure 16 Control Corn Fodder
Fortified at 0.05 ppm with HOE-061517
and at 0.05 ppm with HOE-039866
Recovery = 108% for HOE-061517, 117% for HOE-039866

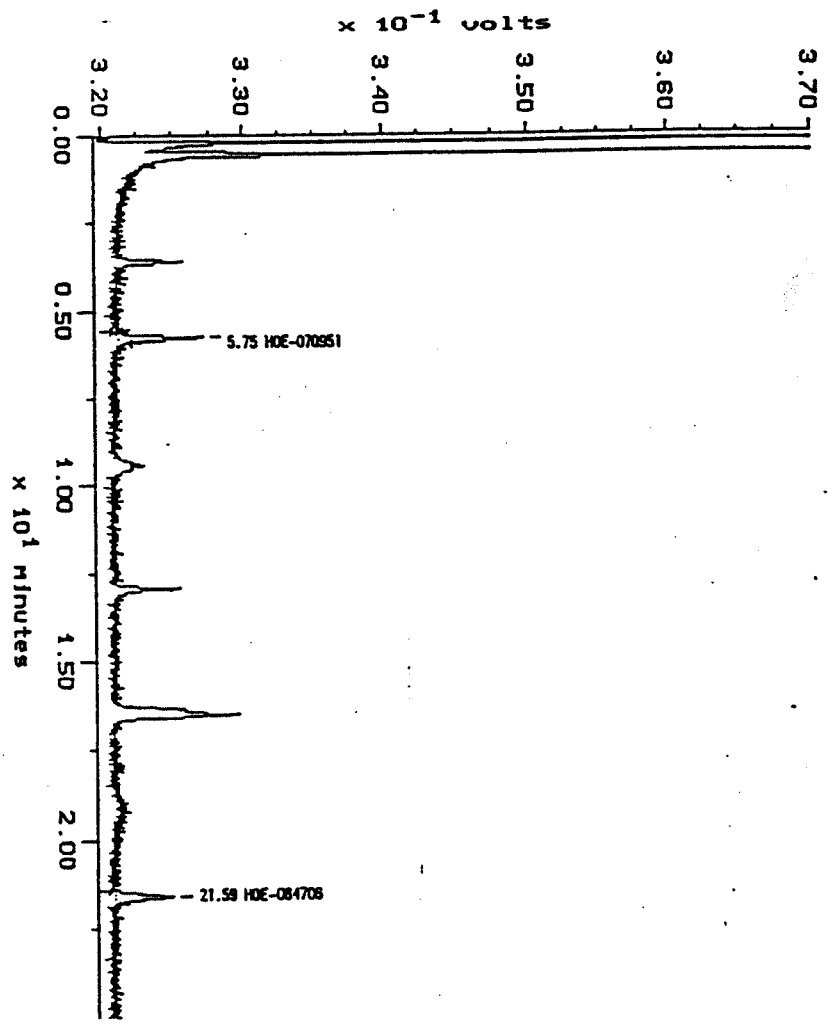


Figure 17 Control Corn Fodder
Fortified at 0.60 ppm with HOE-061517
and at 6.02 ppm with HOE-099730
Recovery = 98% for HOE-061517, 92% for HOE-099730

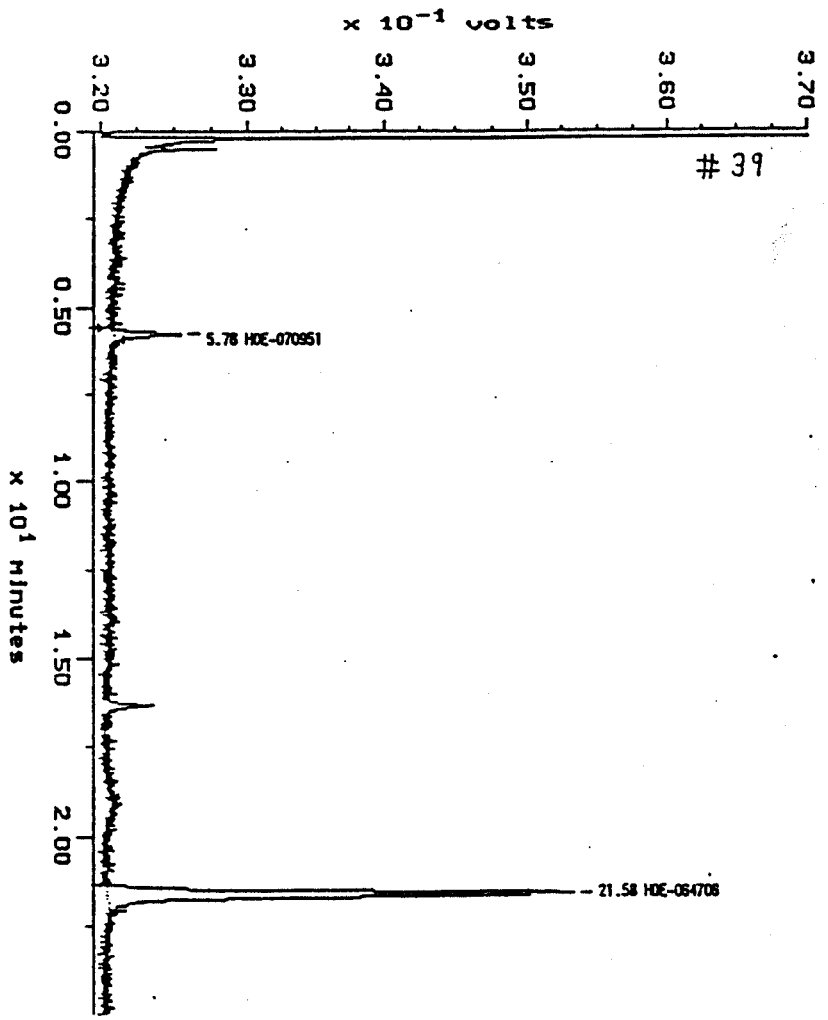


Figure 18 Treated Corn Fodder
0.16 ppm HOE-061517,
and 0.43 ppm HOE-039866/HOE-099730 found

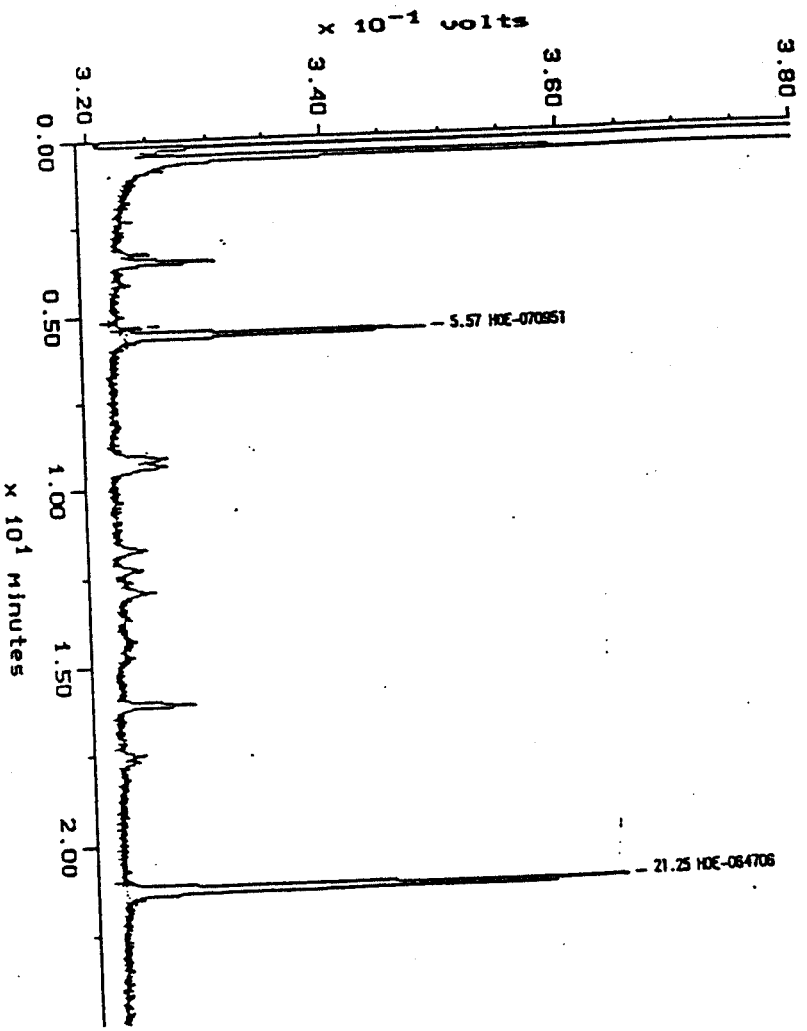


Figure 19 Control Corn Grain
< 0.05 ppm HOE-061517
or HOE-039866/HOE-099730 found

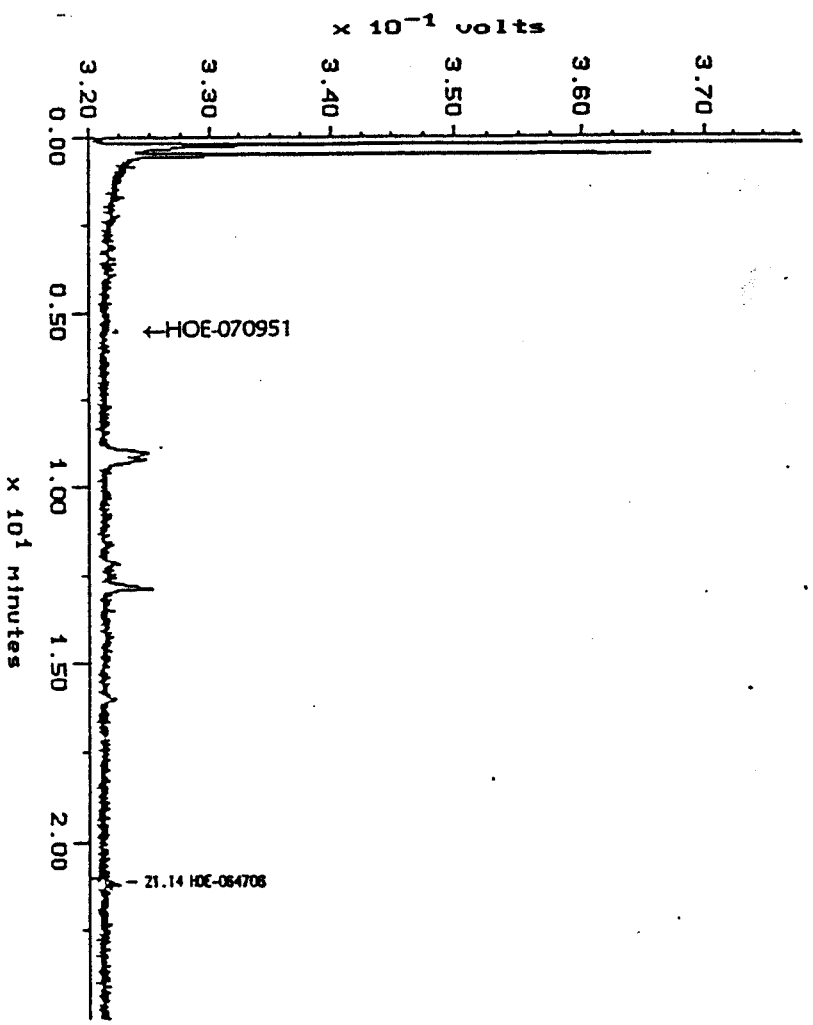


Figure 20 Control Corn Grain
Fortified at 0.10 ppm with HOE-061517
and at 0.10 ppm with HOE-039866
Recovery = 90% for HOE-061517, 83% for HOE-039866

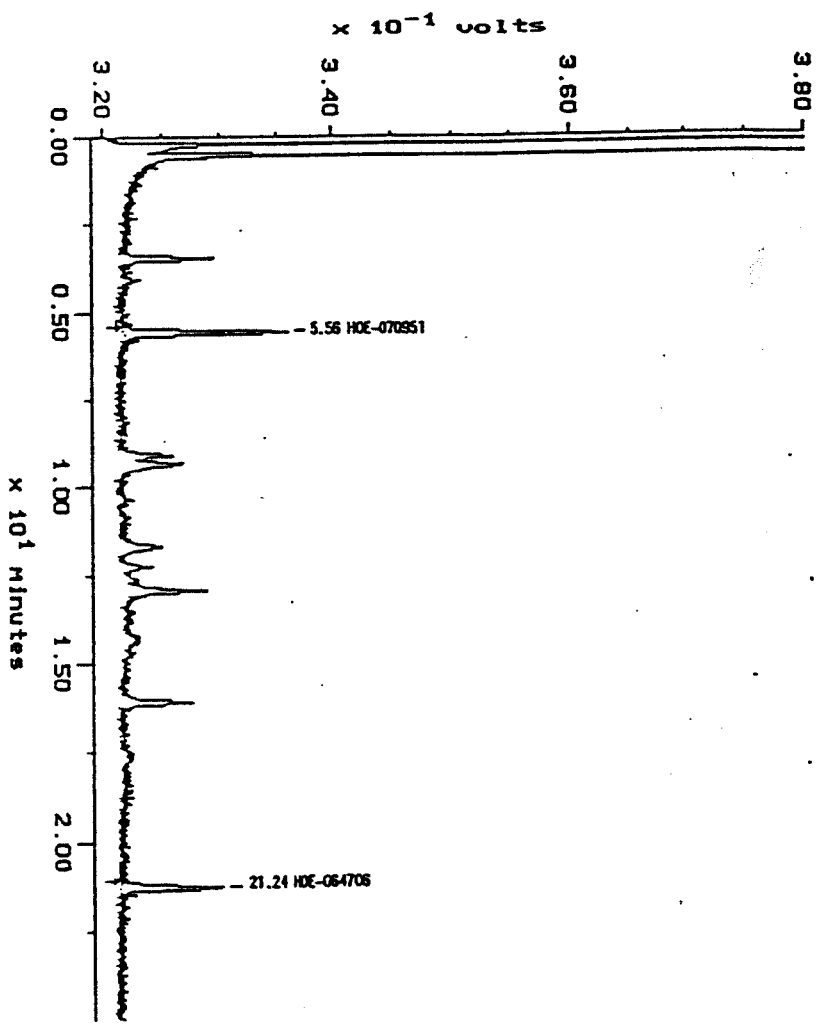


Figure 21 Control Corn Grain
Fortified at 0.05 ppm with HOE-061517
and at 0.05 ppm with HOE-099730
Recovery = 91% for HOE-061517, 100% for HOE-099730

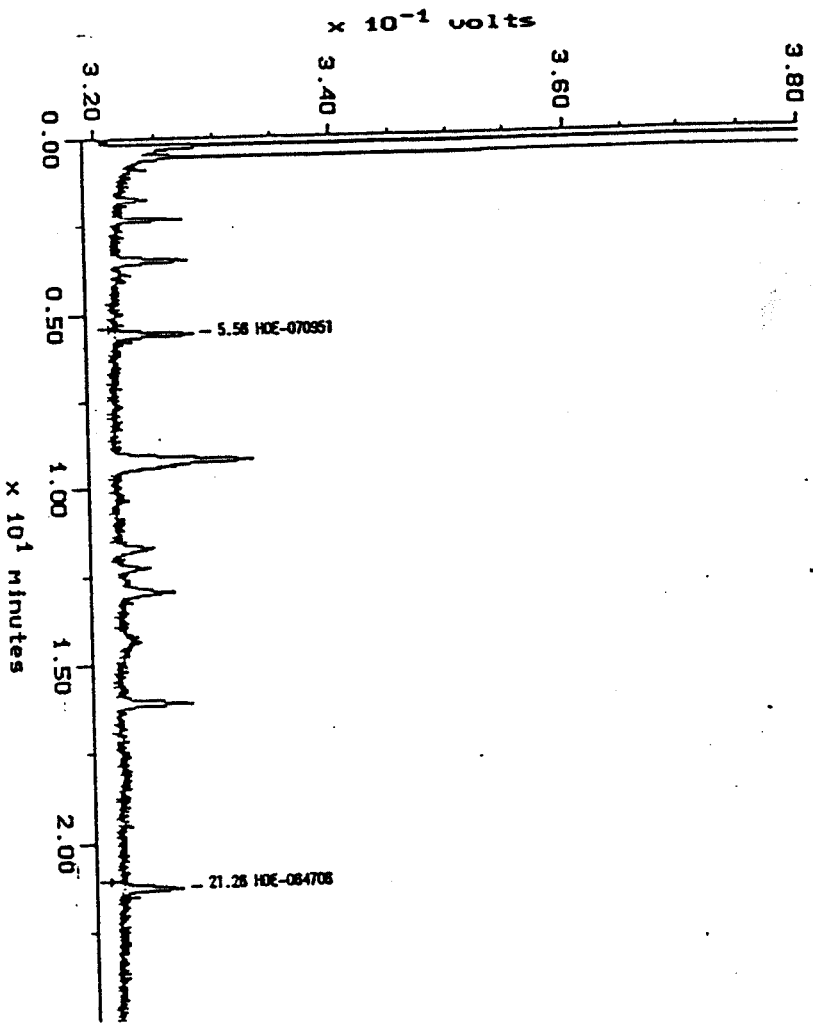


Figure 22 Treated Corn Grain
<0.05 ppm with HOE-061517
and at 0.05 ppm HOE-039866/HOE-099730 found

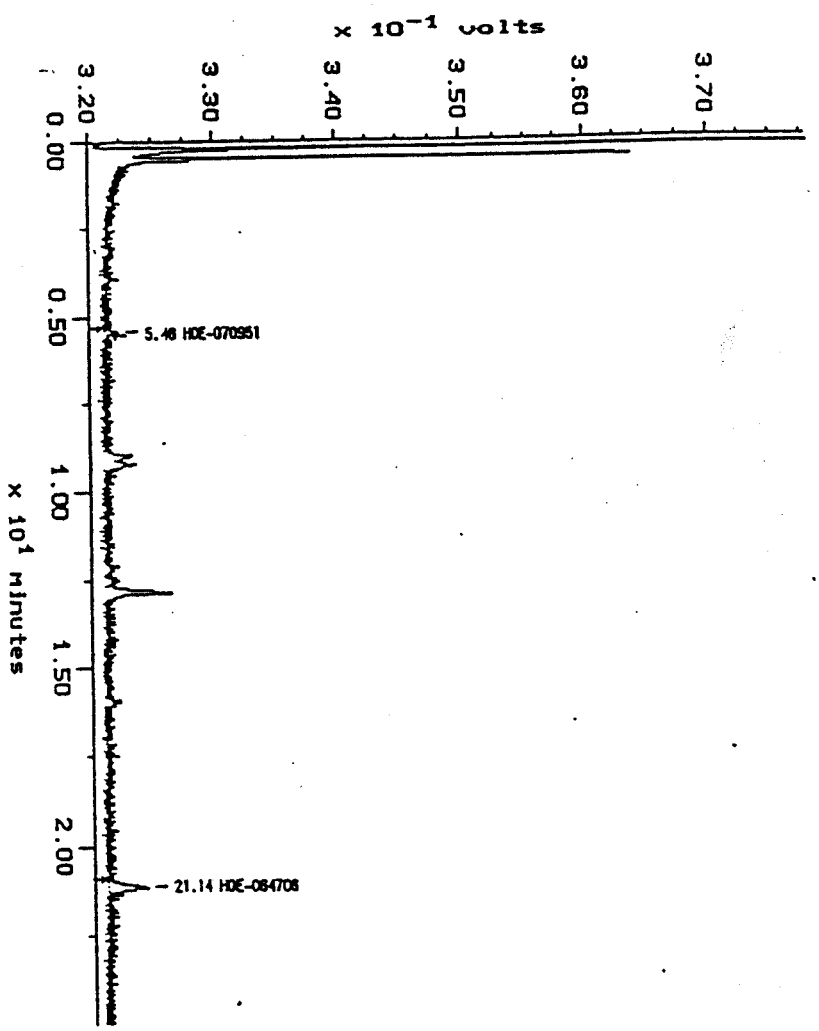


Figure 23 Control Corn Aspirated Grain Fractions
Not fortified
<0.05 ppm HOE-061517 or HOE-039866/HOE-099730 found

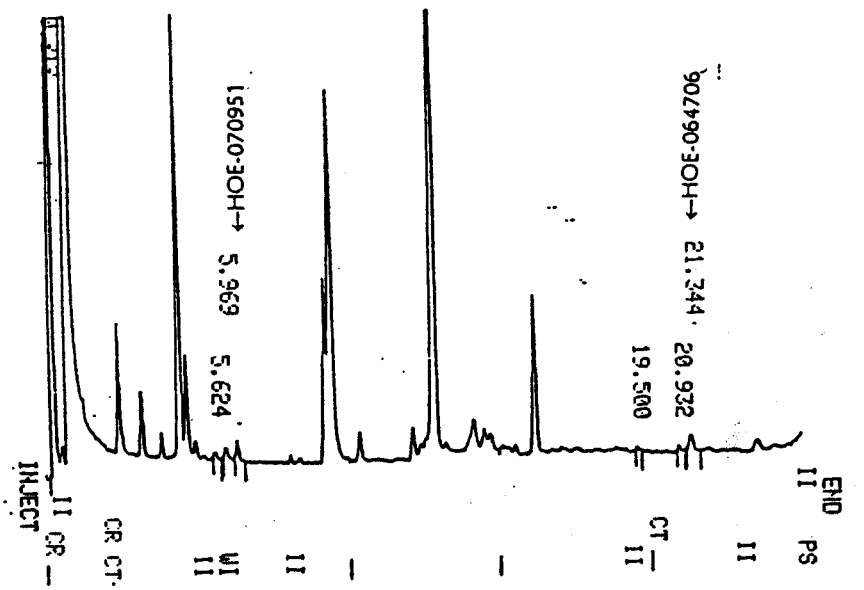


Figure 24 Treated Corn Aspirated Grain Fractions
0.71 pppm HOE-061517 and 3.24 ppm HOE-039866/HOE-099730 found

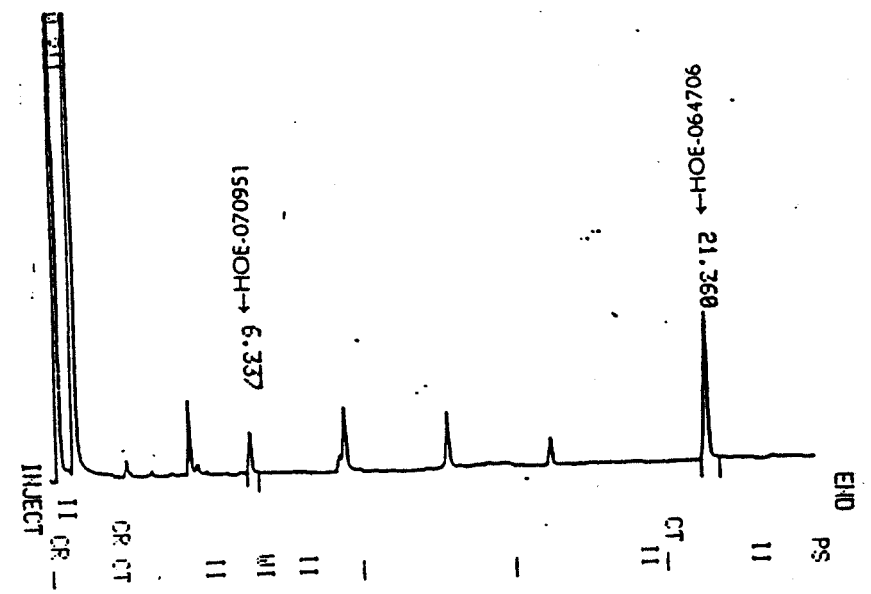


Figure 25 Control Corn Aspirated Grain Fractions
Fortified at 0.05 ppm with HOE-061517 and at 0.05 ppm with HOE-039866
Recovery = 108% for HOE-061517, and 106% for HOE-039866

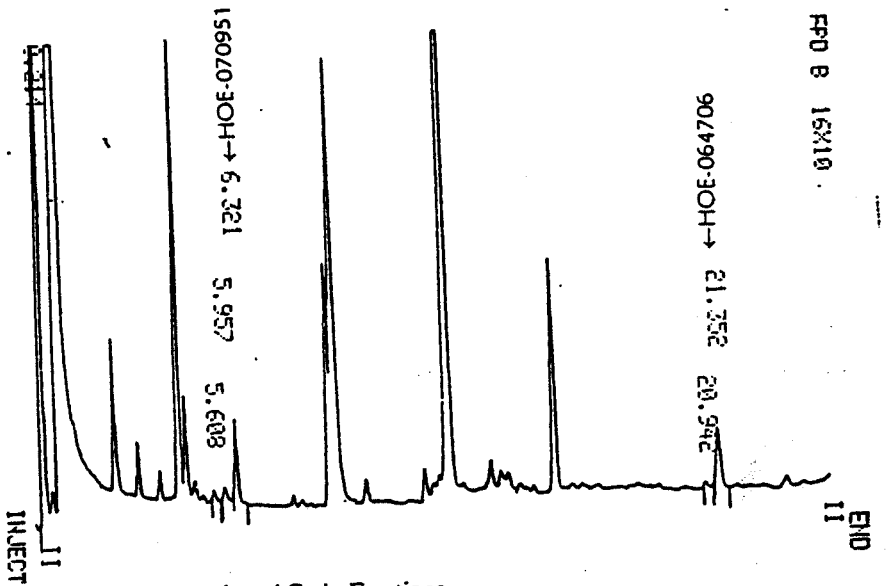


Figure 26 Control Corn Aspirated Grain Fractions
Fortified at 0.50 ppm with HOE-061517 and at 0.50 ppm with HOE-099730
Recovery = 102% for HOE-061517, and 96% for HOE-099730

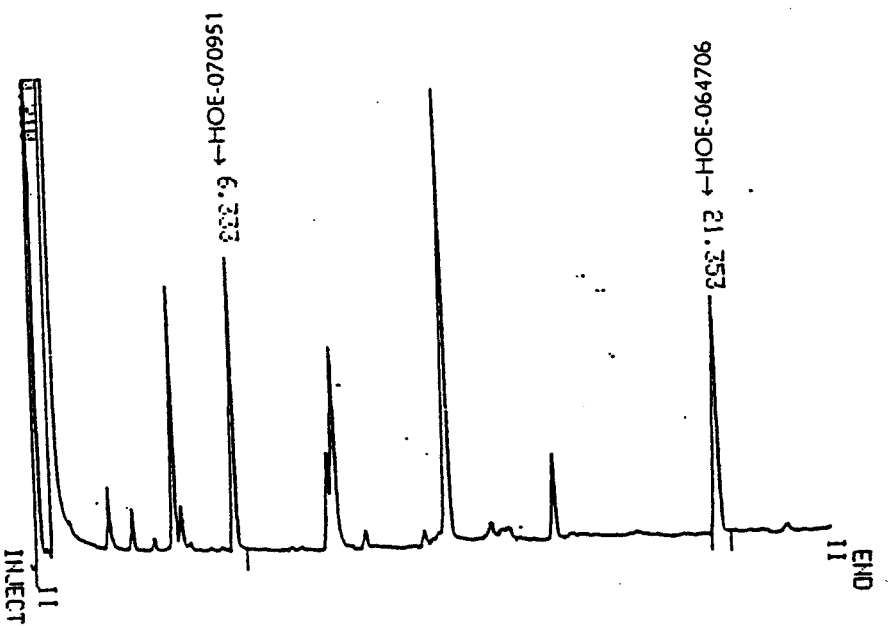


Figure 27 Control Soybean Hay
Not fortified
<0.05 ppm HOE-061517 or HOE-039866/HOE-099730 found

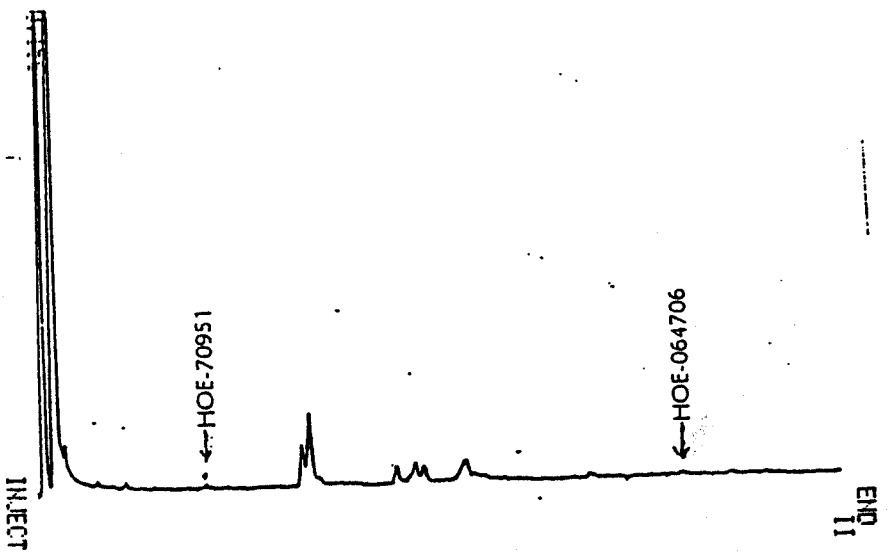


Figure 28 Treated Soybean Hay
0.32 pppm HOE-061517 and 1.00 ppm HOE-039866/HOE-099730 found

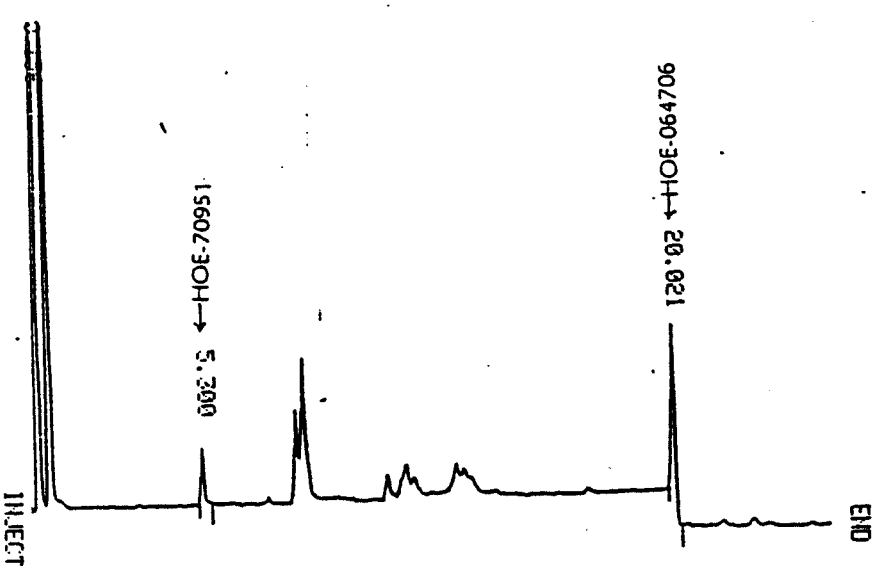


Figure 29 Control Soybean Hay
Fortified at 0.50 ppm with HOE-061517 and at 0.50 ppm with HOE-039866
Recovery = 82% for HOE-061517, and 78% for HOE-039866

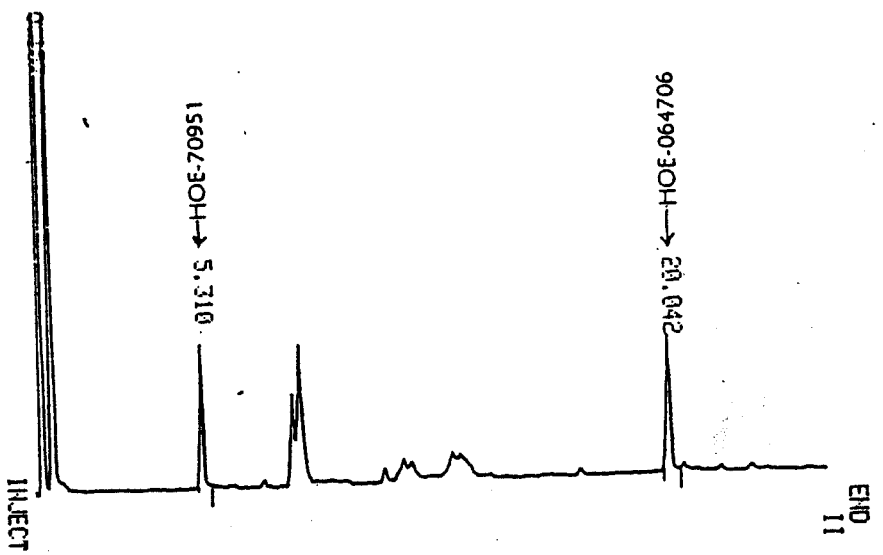


Figure 30 Control Soybean Hay
Fortified at 1.0 ppm with HOE-061517, and at 1.0 ppm with HOE-099730
Recovery = 115% for HOE-061517 and 70% for HOE-099730

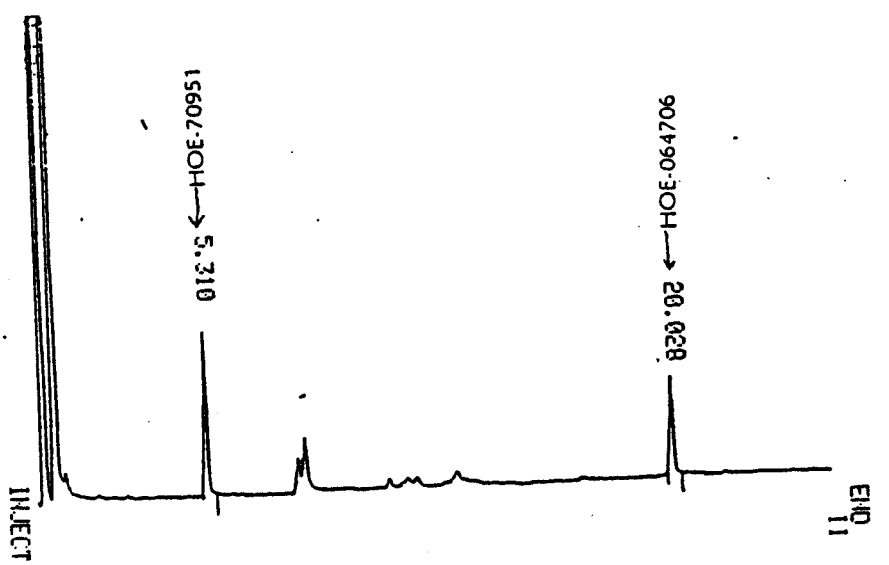


Figure 31 Control Soybean Seed
Not fortified
<0.05 ppm HOE-061517 or HOE-039866/HOE-099730 found

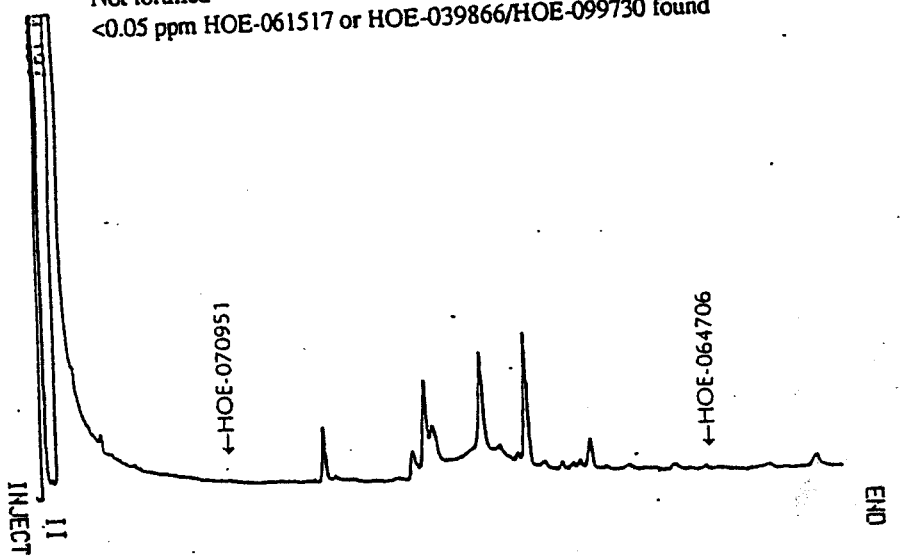


Figure 32 Control Soybean Seed
Fortified at 0:05 ppm with HOE-061517 and at 0.05 ppm with HOE-099730
Recovery = 97% for HOE-061517, and 90% for HOE-099730

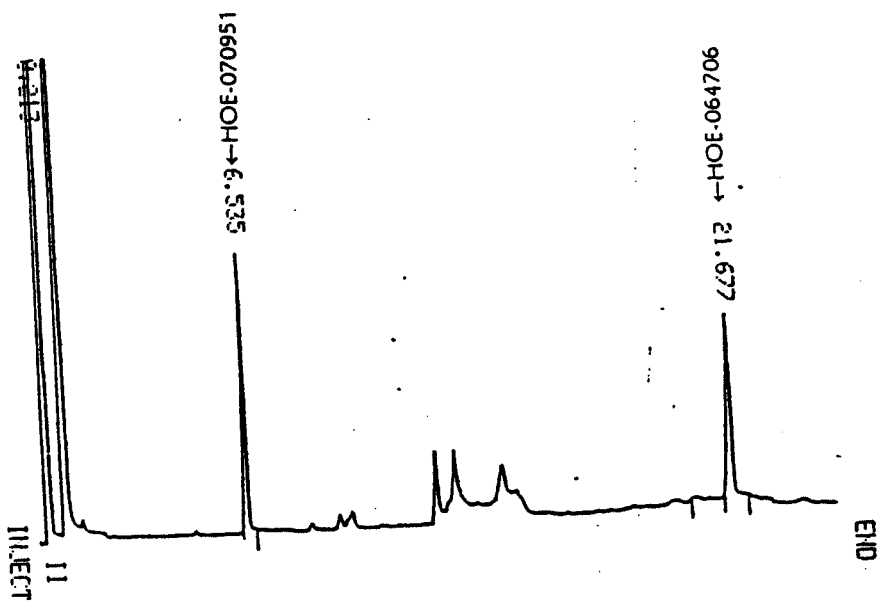


Figure 33 Control Soybean Seed
Fortified at 0.05 ppm with HOE-061517, and at 0.05 ppm with HOE-039866
Recovery = 91% for HOE-061517 and 117% for HOE-039866

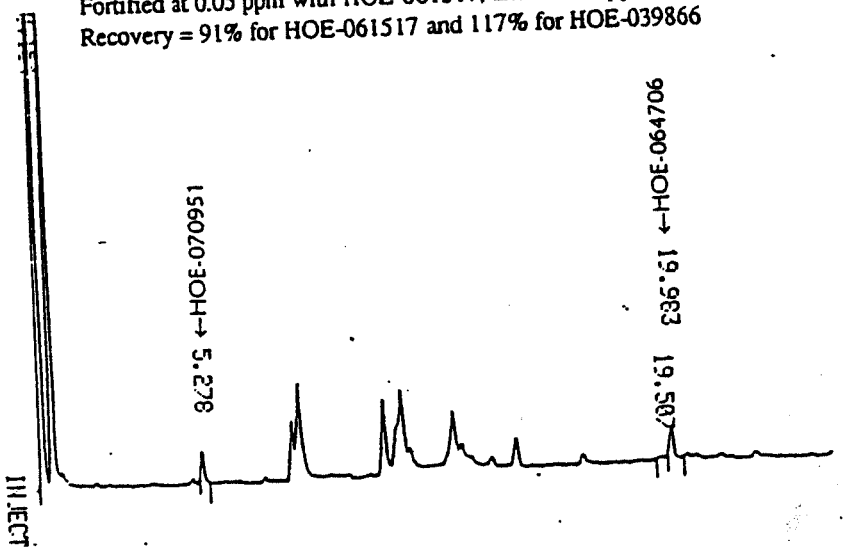


Figure 34 Treated Soybean Seed
0.36 pppm HOE-061517 and 1.43 ppm HOE-039866/HOE-099730 found

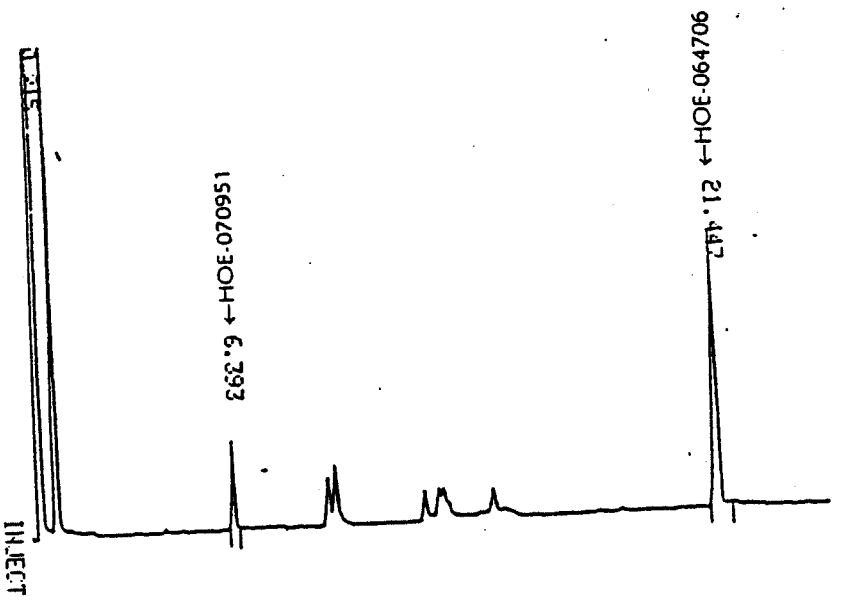


Figure 35 Control Soybean Aspirated Grain Fractions
Not fortified
<0.05 ppm HOE-061517 or HOE-039866/HOE-099730 found

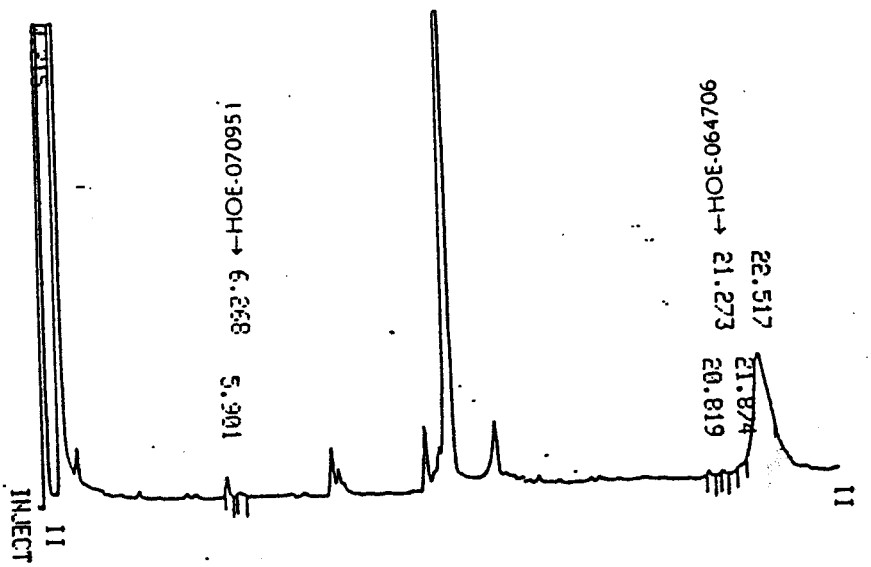


Figure 36 Control Soybean Aspirated Grain Fractions
Fortified at 0.05 ppm with HOE-061517, and at HOE-039866 with HOE-039866
Recovery = 105% for HOE-061517, and 86% for HOE-039866

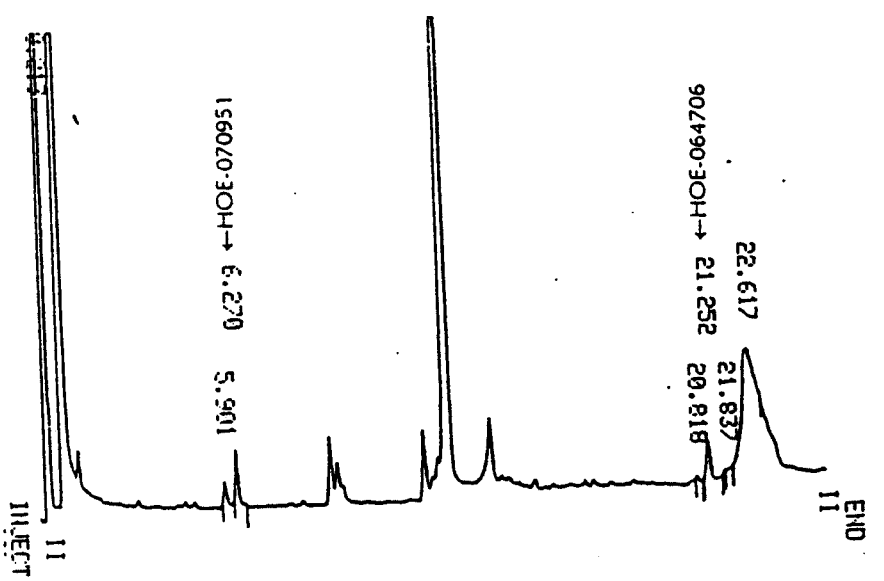


Figure 37 Control Soybean Aspirated Grain Fractions
Not fortified
<0.05 ppm HOE-061517 or HOE-039866/HOE-099730 found

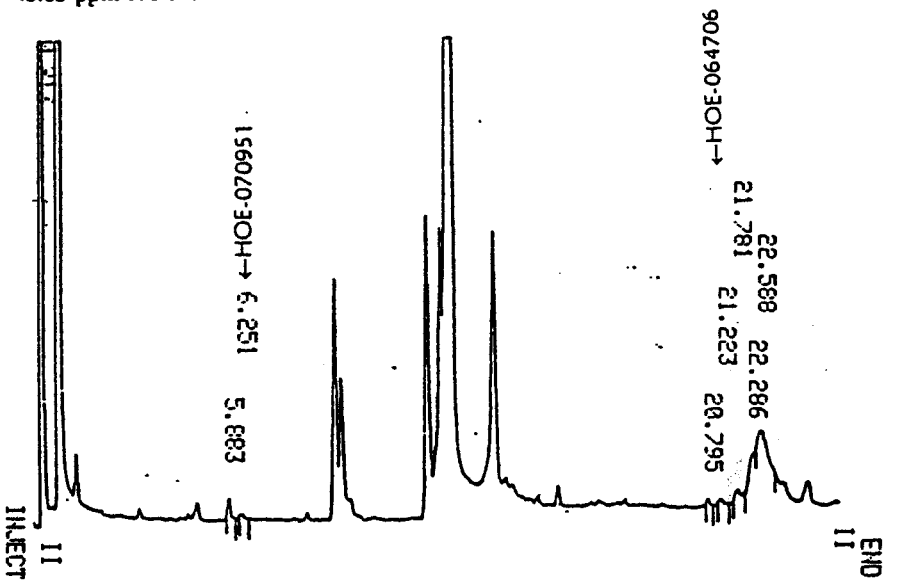


Figure 38 Condtrol Soybean Aspirated Grain Fractions
Fortified at 5.0 ppm with HOE-061517 and at 5.0 ppm with HOE-099730
Recovery = 81% for HOE-061517, and 76% for HOE-099730

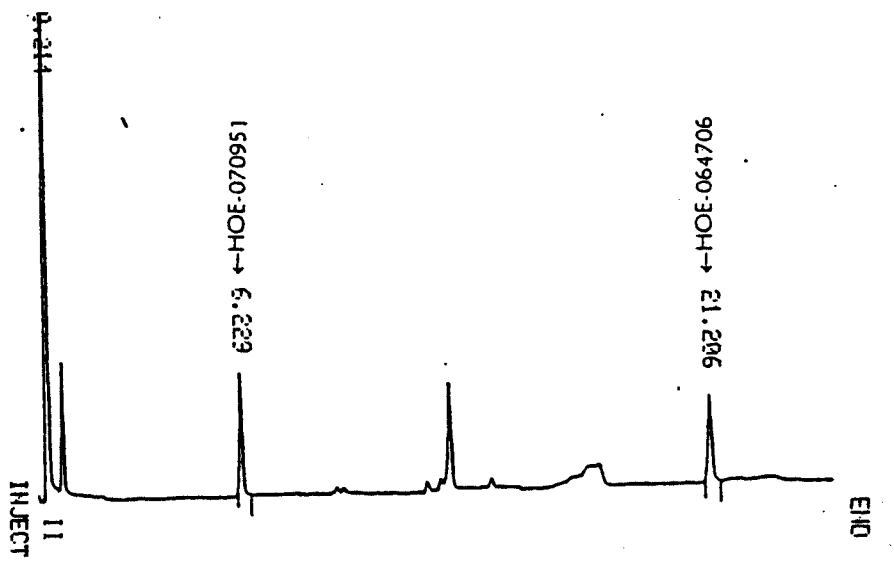


Figure 39 Treated Soybean Aspirated Grain Fractions
4.77 ppm of HOE-061517, and 4.23 ppm of HOE-039866/HOE-099730 found

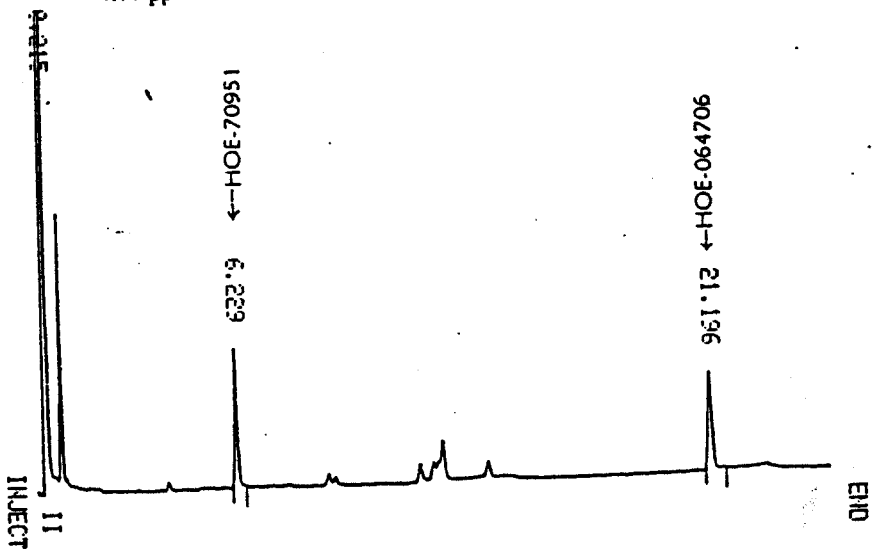
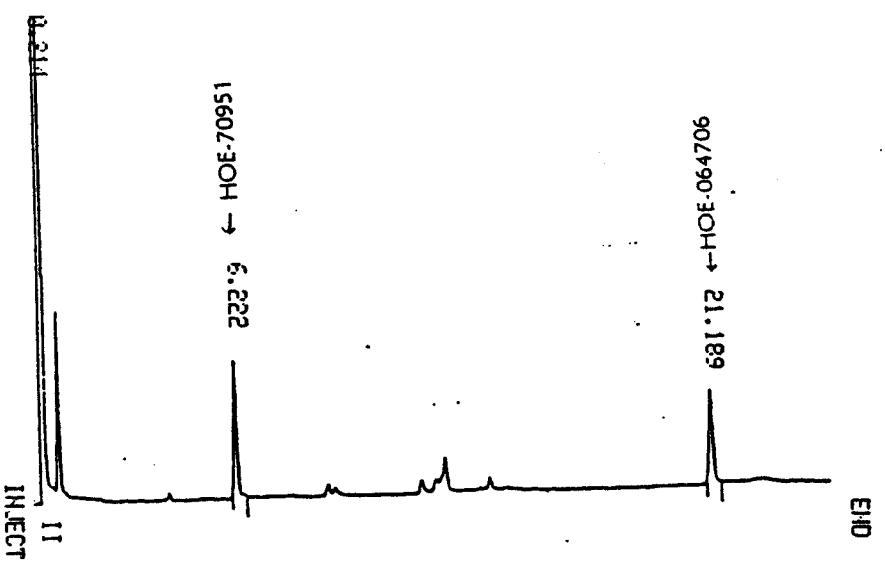


Figure 40 Treated Soybean Aspirated Grain Fractions
4.69 pppm HOE-061517 and 4.06 ppm HOE-039866/HOE-099730 found



METHOD APPENDIX II

Typical Calibration Curves

Reference: ABC/Pan-Ag Division Study No. 94354

Note: Figures are reduced and/or annotated (as necessary) for clarity of presentation.

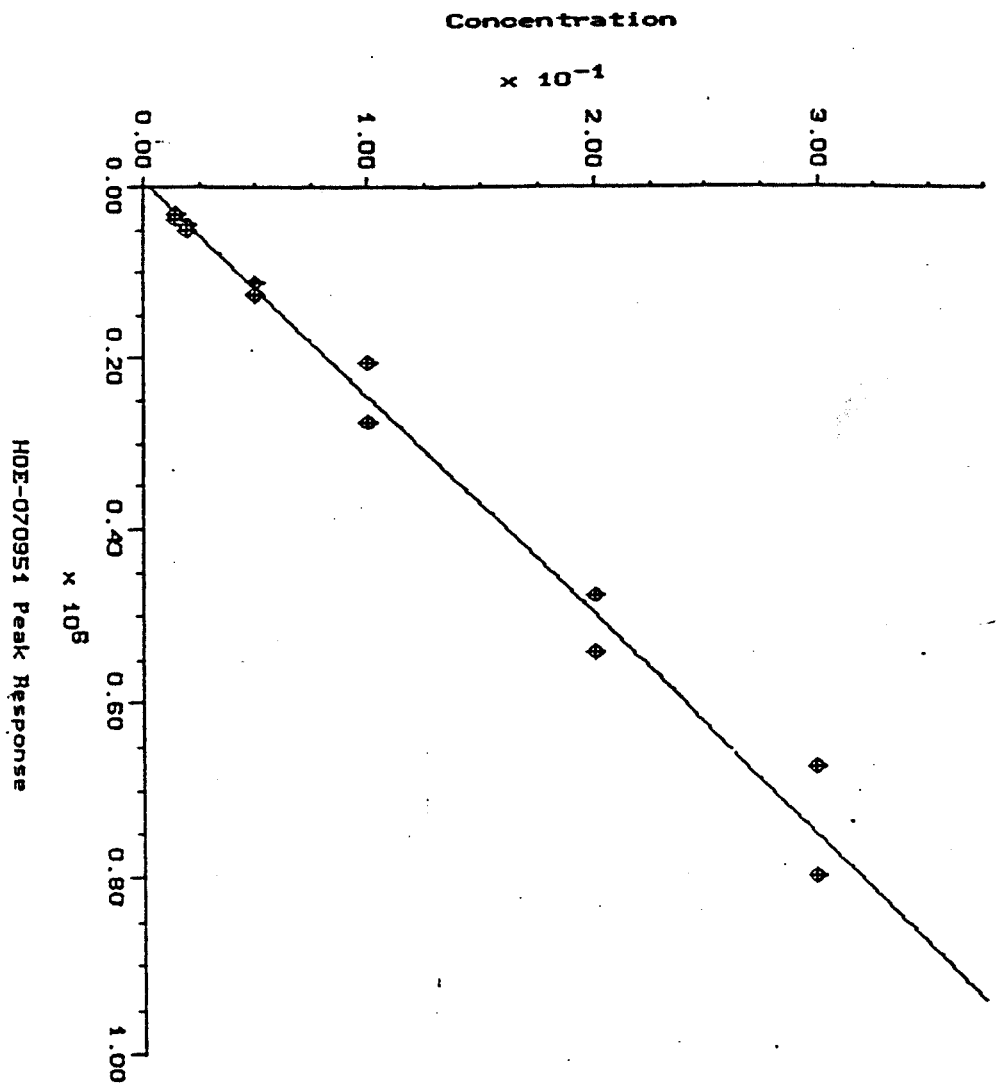
HOE-070951 Calibration Report

Quant Basis: Area | Rejection Tolerance: None | Internal Standard: None
Curve Type: Linear | Weighting: None | Forced Through Origin: No
Y-axis Label: Concentration
Corr. Coef. (r): 0.9920068 Coef. of Determination (r^2): 0.9840815

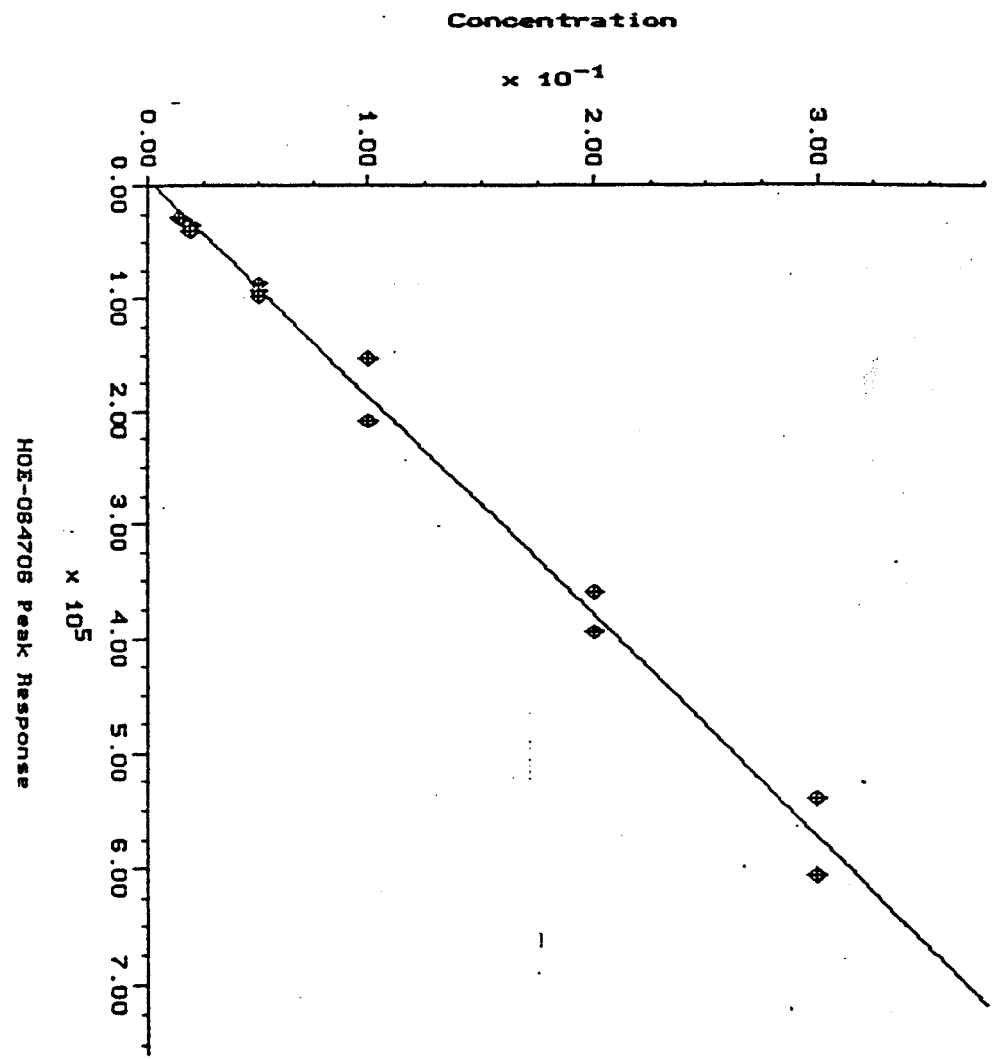
Equation: Conc = 2.784958E-03 + 3.950144E-07 * R

Sample	File Name	Valid	Concentration	Response	Calc'd Concentration	% Deviation	Response Factor
0.0150 ug/ml	F3001	Y	1.500000E-02	3.888607E+04	1.814552E-02	-1.73E+01	3.857422E-07
0.0200 ug/ml	F3002	Y	2.000000E-02	5.187005SE+04	2.327438E-02	-1.41E+01	3.855789E-07
0.0500 ug/ml	F3005	Y	5.000000E-02	1.1142822E+05	4.680071E-02	6.84E+00	4.487194E-07
0.100 ug/ml	F3008	Y	1.000000E-01	2.7437572E+05	1.111673E-01	-1.00E+01	3.644637E-07
0.200 ug/ml	F3011	Y	2.000000E-01	4.7598103E+05	1.907964E-01	4.82E+00	4.202025E-07
0.300 ug/ml	F3014	Y	3.000000E-01	6.7440344E+05	2.691841E-01	1.14E+01	4.448376E-07
0.0150 ug/ml	F3017	Y	1.500000E-02	3.1344002E+04	1.516629E-02	-1.10E+00	4.785604E-07
0.0200 ug/ml	F3020	Y	2.000000E-02	4.3600805E+04	2.000791E-02	-3.95E-02	4.587071E-07
0.0500 ug/ml	F3023	Y	5.000000E-02	1.2449177E+05	5.196101E-02	-3.77E+00	4.016330E-07
0.100 ug/ml	F3026	Y	1.000000E-01	2.0563784E+05	8.401488E-02	1.90E+01	4.862918E-07
0.200 ug/ml	F3029	Y	2.000000E-01	5.4277806E+05	2.171893E-01	-7.91E+00	3.684761E-07
0.300 ug/ml	F3032	Y	3.000000E-01	8.0136594E+05	3.193361E-01	-6.06E+00	3.743608E-07
0.0500 ug/ml	F3033	Y	5.000000E-02	1.2701096E+05	5.295612E-02	-5.58E+00	3.936668E-07

HOE-070951 Typical Standard Curve



HOE-064706 Typical Standard Curve



METHOD APPENDIX III

Example Calculation Sheet

Reference: ABC/Pan-Ag Division Study No. 94354

SUMMARY OF RAW DATA FOR CORN FORAGE SAMPLES

Sponsor Trial No.: WI-01, Pan-Ag Trial No.: 94354

Standard ID	Std. Amt. (ug/ml) HOE-070951	Peak Area	Std. Amt. (ug/ml) HOE-064706	Peak Area
IGC-1A	0.0150	38886	0.0150	28658
IGC-2A	0.0200	51870	0.0200	40569
IGC-3A	0.0500	111428	0.0500	85901
IGC-4A	0.100	274376	0.100	207827
IGC-5A	0.200	475961	0.200	358574
IGC-6A	0.300	674403	0.300	538524
IGC-1A	0.0150	31344	0.0150	27018
IGC-2A	0.0200	43601	0.0200	33631
IGC-3A	0.0500	124492	0.0500	92566
IGC-4A	0.100	205638	0.100	152851
IGC-5A	0.200	342776	0.200	393875
IGC-6A	0.300	801366	0.300	606891
IGC-3A	0.0500	127011	0.0500	98251

Extraction Date: 3-1-95 to 3-2-95
GC Analysis Date: 3-2-95 to 3-3-95
Injection volume: 4ul

Sample Weight = 12.5 g
Total Extraction Volume = 500 ml
Aliquot Volume = 100 ml

Curve Equation HOE-070951
Conc. (ug/ml) = $2.784958 \times 10^{-3} + 3.950144 \times 10^{-7}$ (R)
Correlation Coefficient (r) HOE-070951 = 0.9920088

Retention time range HOE-070951: 5.33 to 5.39 minutes

Curve Equation HOE-064706
Conc. (ug/ml) = $2.760436 \times 10^{-3} + 5.189517 \times 10^{-7}$ (R)
Correlation Coefficient (r) HOE-064706 = 0.9951740

Retention time range HOE-064706: 20.95 to 21.00 minutes

Lab ID	Sample ID	Final Volume (ml)	Peak Area HOE-070951	Peak Area HOE-064706	Amt. Found HOE-061517 (ug/g)	Amt. Added HOE-061517 (ug/g)	Recovery HOE-061517	Amt. Found HOE-099730 + 039866 (ug/g)	Amt. Added HOE-099730 or 039866 (ug/g)	Recovery HOE-099730 or HOE-039866
354F3-1	1c - 30 DAT Control (Regimen A)	5.0	0	12615 [1]	<0.05	N/A	N/A	<0.05	N/A	N/A
354F3-2	1c - Spike - HOE-061517 & 099730	5.0	47255	55035	0.0429	0.0500	85.8%	0.0495	0.0502	98.6%
354F3-3	2c - 30 DAT (Regimen B)	5.0	11652	555023	<0.05	N/A	N/A	0.582	N/A	N/A
354F3-4	3c - 30 DAT (Regimen B)	5.0	8104	444705	<0.05	N/A	N/A	0.467	N/A	N/A
354F3-5	5c - 30 DAT (Regimen C)	5.0	10604	462989	<0.05	N/A	N/A	0.486	N/A	N/A
354F3-6	6c - 30 DAT (Regimen C)	5.0	10430	462540	<0.05	N/A	N/A	0.486	N/A	N/A
354F3-7	13c-75-85 DAT Control (Regimen A)	5.0	0	8729 [1]	<0.05	N/A	N/A	<0.05	N/A	N/A
354F3-8	15c - Spike - HOE-061517 & 039866	10	45659	425630	0.0833	0.100	83.3%	0.884	1.01	87.7%
354F3-9	16c - 75-85 DAT (Regimen B)	5.0	26433	376881	<0.05	N/A	N/A	0.397	N/A	N/A
354F3-10	17c - 75-85 DAT (Regimen B)	5.0	13950	307864	<0.05	N/A	N/A	0.325	N/A	N/A
354F3-11	19c - 75-85 DAT (Regimen C)	5.0	18326	303699	<0.05	N/A	N/A	0.321	N/A	N/A
354F3-12	20c - 75-85 DAT (Regimen C)	5.0	10163	228275	<0.05	N/A	N/A	0.242	N/A	N/A

R = Peak Area

All Concentrations are expressed as HOE-033936 equivalents

Recovery = Amount Found / Amount Added X 100

1 Response >10% below lowest standard. Area of control was subtracted from area of fortified control before calculating ug/g HOE-099730 + HOE-039866 found.