

**ANALYTICAL REPORT**

**STUDY TITLE**

Determination of Glufosinate Ammonium Active Ingredient  
Content in Rely (11.3% w/w Glufosinate Ammonium)

**AUTHOR**

David A. Winkler

**EN-CAS STUDY NUMBER**

97-0043

**PESTICIDE ASSESSMENT GUIDELINE**

OPPTS 830.1550

**ANALYTICAL TESTING FACILITY**

David A. Winkler, B.S.  
EN-CAS Analytical Laboratories  
2359 Farrington Point Drive  
Winston-Salem, NC 27107  
(910)785-3252

**STUDY DIRECTOR/SPONSOR AND TESTING FACILITY**

Lance Fritz  
AgrEvo USA Co.  
1740 Whitehall Road  
N. Muskegon, MI 49445  
(616)744-4711

Total Number of Pages = 15

GLP STATEMENT

We, the undersigned, hereby certify that the work described herein was conducted by EN-CAS Laboratories in compliance with EPA Good Laboratory Practices (GLP) as defined in 40 CFR, Part 160.

Principal  
Analytical  
Investigator:

David A. Winkler  
David A. Winkler  
Principal Analytical Investigator  
EN-CAS Analytical Laboratories

5/14/97  
Date

EN-CAS  
Management:

Bert Clayton  
Bert Clayton  
Manager, Analytical and  
Residue Studies  
EN-CAS Analytical Laboratories

5/14/97  
Date

Study Director/  
Sponsor  
Representative:

Lance M. Fritz  
Lance Fritz  
Study Director/Sponsor Representative  
AgrEvo USA, Inc.

5/18/97  
Date

## QUALITY ASSURANCE STATEMENT

The study, entitled Determination of Glufosinate Ammonium Active Ingredient Content in Rely (11.3% Glufosinate Ammonium), under EN-CAS Project # 97-0043, was inspected, and the inspection results were reported to Principal Analytical Investigator, EN-CAS Management and the Study Director on the following dates:

<u>Inspection Phase</u>	<u>Inspection Dates</u>	<u>Date Reported To:</u>		
		<u>Principal Analytical Investigator</u>	<u>EN-CAS Management</u>	<u>Study Director*</u>
Protocol	4/21/97	4/21/97	4/25/97	5/1/97
In-Lab Audit	4/23/97	4/23/97	4/25/97	5/1/97
Data	4/29/97	4/29/97	5/2/97	5/1/97
Final Report	5/12/97	5/12/97	5/14/97	5/14/97

\* And Study Director's Management

Signed: Kathleen H. Faltynski 5/14/97  
Kathleen H. Faltynski, M.S. Date  
Quality Assurance Officer

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## I. OBJECTIVE

The objective of this study was to analyze and determine the active ingredient (a.i.) content in sample(s) from a selected lot of Rely (11.3% Glufosinate Ammonium).

## II. TEST AND REFERENCE SUBSTANCE

### A. Test Substance

The test substance was the glufosinate ammonium content contained in the Rely formulation. Additional characterization of the test substance (i.e., other than determination of the active ingredient content) is the responsibility of the Sponsor.

The test substance was stored under typical room temperature conditions.

<u>Test Substance</u>	<u>Grade</u>	<u>Source</u>	<u>Date Received</u>	<u>EN-CAS E #</u>	<u>Nominal Conc</u>	<u>Batch#</u>	<u>Physical Appearance</u>
Glufosinate Ammonium in Rely	Formulation	AgrEvo	4/4/97	EO7125	11.3%	RLREJE05-01	Blue Liquid

### B. Reference Substance

The reference substance was stored under freezer storage conditions at approximately  $< -15^{\circ}\text{C}$ . The reference substance used in this study was provided by the Sponsor along with the information tabulated below. Characterization of the glufosinate ammonium reference substance is the responsibility of the Sponsor.

<u>Reference Substance</u>	<u>Grade</u>	<u>Source</u>	<u>Date Received</u>	<u>EN-CAS E #</u>	<u>% Purity</u>	<u>Batch #</u>	<u>Exp. Date</u>	<u>Physical Appearance</u>
Glufosinate Ammonium	Analytical	AgrEvo	3/7/97	EO6574	99.2	26880-125-M29	9-4-98	White Powder

### III. EXPERIMENTAL

#### A. Sample Receipt, Logging and Storage

After receipt, the formulation sample was logged with a unique identification number (E#) and the condition recorded on a logging form by EN-CAS personnel. The initials of the logger and the date received were also recorded. The sample was secured under room temperature storage conditions. The storage temperatures were monitored by EN-CAS Laboratory personnel in accordance with EN-CAS SOPs.

#### B. Summary of Analytical Method

The NOR-AM Chemical Co. Method Code No. NA-A120 entitled, The Determination of Glufosinate Ammonium in Glufosinate Ammonium 200 g/L by HPLC, issued May 6, 1994, was used as the reference method.

Briefly, the method involved dilution of an appropriate formulation sample in 85% water/15% 0.1 M potassium dihydrogen orthophosphate (potassium phosphate monobasic) and quantitation was done by HPLC utilizing UV detection at a wavelength of 193 nm. A single point glufosinate ammonium analytical reference substance was used for the quantitation of the a.i. content in the formulated product. Duplicate subsamples from the lot of formulation were analyzed and each replicate was injected twice. The single point glufosinate ammonium reference standard was injected before and after each sample injection and the average of all the standard injections was used for the calculation of each replicate injection.

## C. Summary of Method Modifications

### 1. AgrEvo Suggested Modifications

- a. Section 5, PREPARATION OF BUFFER SOLUTION, 0.1 M Potassium Dihydrogen Orthophosphate Solution

In-lab deionized water was used to prepare the buffer solution, instead of HPLC grade water.

- b. Section 6, PREPARATION OF CALIBRATION STANDARD, Single Point Calibration

A single glufosinate analytical reference standard was weighed instead of duplicate standards and diluted to volume using 85% water/15% 0.1 M potassium dihydrogen orthophosphate instead of 0.1 M potassium dihydrogen orthophosphate.

- c. Section 7, PREPARATION OF SAMPLE SOLUTION

The sample was diluted to volume using 85% water/15% 0.1 M potassium dihydrogen orthophosphate instead of 100% 0.1 M potassium dihydrogen orthophosphate.

### 2. EN-CAS Modifications

- a. Apparatus

The detector used was a UV programmable absorbance detector instead of a diode array detector.

### III. EXPERIMENTAL (continued)

#### D. Calculations

##### 1. Single Point Calibration

$$\begin{array}{l} \text{\% content} \\ \text{of sample} \end{array} = \frac{C_1}{A} \times \frac{W_1}{W_2} \times P$$

A = Average area of active ingredient in calibration standard.

C<sub>1</sub> = Area of active ingredient in sample.

W<sub>1</sub> = Weight of calibration standard.

W<sub>2</sub> = Weight of sample.

P = % purity of glufosinate in calibration standard.

##### 2. Example Calculation for a Formulation Sample

Glufosinate Ammonium Sample EO7125-F1A, set # 1-01-AN, run # 65314.

Where:

A	=	20754637 counts
C <sub>1</sub>	=	12190232 counts
W <sub>1</sub>	=	0.05055 g
W <sub>2</sub>	=	0.25150 g
P	=	99.2%

% w/w glufosinate	12190232 counts	0.05055 g		
content of sample =	-----	x	-----	x 99.2% = 11.7%
	20754637 counts		0.25150 g	

#### E. Statistical Analyses

Statistical analyses were done according to NOR-AM Chemical Co. Method Code No. NA-A120.

### III. EXPERIMENTAL (continued)

#### F. Results

Duplicate subsamples of the Rely formulation were analyzed and each replicate was injected twice. The mean percent active ingredient result is shown below.

<u>Product</u>	<u>Batch #</u>	<u>Date Received</u>	<u>Mean Percent<sup>a</sup> Active Ingredient (Glufosinate Ammonium)</u>
Rely	RLREJE05-01	4/4/97	11.7
<sup>a</sup> 11.7% + 11.7% + 11.7% + 11.7% = 46.8 ÷ 4 = 11.7%			

### IV. DISPOSITION OF SAMPLES AND RAW DATA

Sample solutions will be appropriately disposed of after review and acceptance of raw data by the Study Director. The formulation samples will be retained at ambient storage at EN-CAS until directions are received from the Sponsor for shipment to the appropriate Sponsor designated facility.

All analytical raw data will be temporarily archived at EN-CAS Analytical Laboratories, 2359 Farrington Point Drive, Winston-Salem, NC 27107. Upon request, the raw data will be transferred to the Sponsor, with a copy retained at EN-CAS Analytical Laboratories.

### V. STUDY PARTICIPANTS LIST

**Analyst:** Richard L. Parkes  
Project Coordinator

**Group Leader:** David A. Winkler, B.S.

**Report Prepared By:** Cheryl M. Cortez, B.A.  
Technical Writing Coordinator

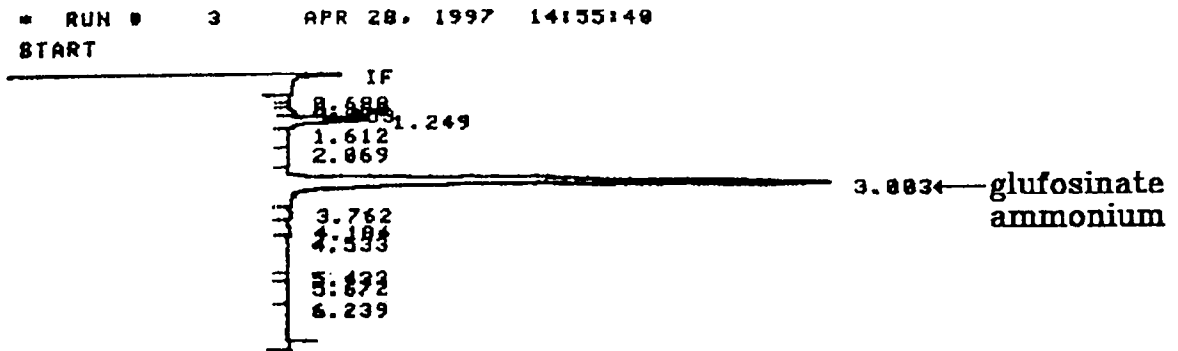
Melinda D. Smith, A.A.S.  
Technical Writer

Donna L. Everhart  
Technical Writing Assistant

FIGURE 1

Typical Chromatography for Determination of Glufosinate  
Ammonium in Rely Formulated Product

HPLC Calibration Standard

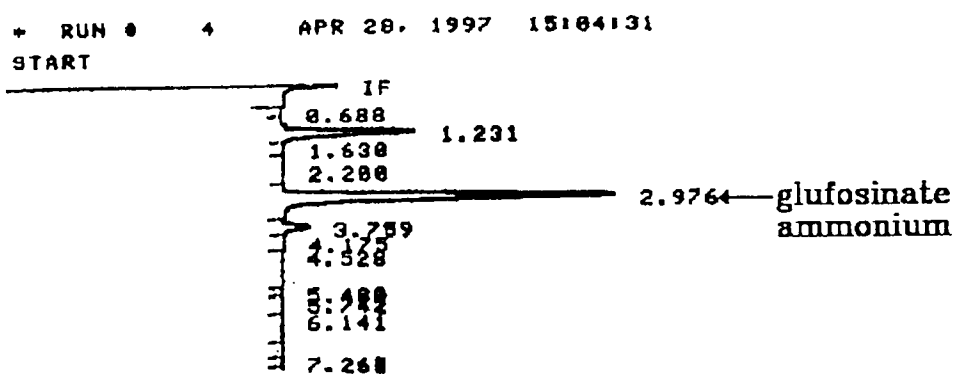


HPLC Run # 65314, dated 4/28/97, set # 1-01-AN  
Peak Area Glufosinate Ammonium = 20745888

FIGURE 2

Typical Chromatography for Determination of Glufosinate Ammonium in Rely Formulated Product

Rely Formulation Sample



EN-CAS Sample # EO7125-F1A  
HPLC Run # 65314, dated 4/28/97, set # 1-01-AN  
Peak Area Glufosinate Ammonium = 12190232

# **APPENDIX I**

## **Certificate of Analysis**

**CERTIFICATE OF ANALYSIS**

**GENERAL**

This certificate of analysis fulfills the requirements for the characterization of a test substance prior to a study according to Good Laboratory Practice (GLP) regulations. It documents the identity, purity/content of the test substance. Stability of the test substance is the responsibility of the Sponsor.

**DESIGNATION OF THE CERTIFIED MATERIAL**

Intended Use: Residue Field Trials  
Material: Rely  
Inv. No.: L004609  
Batch No.: RLREJE05-01

**ANALYTICAL DATA**

Analysis of active ingredient(s)

<u>Name</u>	<u>Content</u>	<u>Method</u>
Rely	11.7% w/w	NA-A120

The purity of the material was determined by: HPLC

Physicochemical Properties

Appearance: Blue Liquid

**STORAGE CONDITIONS**

Date of Analysis: 28-March-97  
Storage Conditions: Room Temperature  
Expiration date: 28-March-98

Whenever the container is opened for removal of aliquot portions of the substance, the person handling the substance must ensure that the integrity of the substance is maintained. Special care has to be taken to avoid any contamination or adulteration of the test substance and appropriate records of its use must be retained.

**ORIGIN OF THE CERTIFIED MATERIAL**

Biological Inventory  
AgrEvo USA Company  
703 NOR-AM Road  
Pikeville, North Carolina 27863  
USA

**ANALYTICAL TESTING FACILITY**

EN-CAS Analytical Laboratories  
2359 Farrington Point Drive  
Winston-Salem, NC 27107

Principal Analytical Investigator: David A. Winkler

Manager, Analytical  
and Residue Studies: Bert Clayton

Starting Date: 23-April-97

Date of Analysis: 28-April-97

Completion Date: 18-May-97 JF

Report and raw data are archived at the testing facility.

Principal Analytical Investigator: David A. Winkler 5/14/97  
David A. Winkler, B.S. Date  
Group Leader

EN-CAS Management Approval: Bert Clayton 5/14/97  
Bert Clayton, B.S. Date  
Manager, Analytical  
and Residue Studies

**TESTING FACILITY**

Sponsor Representative: Lance M. Fritz 5-18-97  
Lance Fritz Date  
Study Director