ANALYTICAL REPORT

STUDY TITLE

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

AUTHOR

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EN-CAS STUDY NUMBER

97-0043

PESTICIDE ASSESSMENT GUIDELINE

OPPTS 830.1550

ANALYTICAL TESTING FACILITY

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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
Principal Analytical Investigator
EN-CAS Analytical Laboratories

5/14/97  Date

EN-CAS Management:

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

5/14/97  Date

Study Director/
Sponsor Representative:

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:

<table>
<thead>
<tr>
<th>Inspection Phase</th>
<th>Inspection Dates</th>
<th>Principal Analytical Investigator</th>
<th>EN-CAS Management</th>
<th>Study Director*</th>
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</thead>
<tbody>
<tr>
<td>Data</td>
<td>4/29/97</td>
<td>4/29/97</td>
<td>5/2/97</td>
<td>5/1/97</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
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<tr>
<td>Final Report</td>
<td>5/12/97</td>
<td>5/12/97</td>
<td>5/14/97</td>
<td>5/14/97</td>
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</tbody>
</table>

* And Study Director’s Management

Signed: [Signature]
Kathleen H. Faltynski, M.S.
Quality Assurance Officer

EN-CAS Project # 97-0043
Page 3
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>SUBJECT</th>
<th>PAGE</th>
</tr>
</thead>
<tbody>
<tr>
<td>TITLE PAGE</td>
<td>1</td>
</tr>
<tr>
<td>GLP STATEMENT</td>
<td>2</td>
</tr>
<tr>
<td>QUALITY ASSURANCE STATEMENT</td>
<td>3</td>
</tr>
<tr>
<td>TABLE OF CONTENTS</td>
<td>4</td>
</tr>
<tr>
<td>I. OBJECTIVE</td>
<td>5</td>
</tr>
<tr>
<td>II. TEST AND REFERENCE SUBSTANCE</td>
<td>5</td>
</tr>
<tr>
<td>A. Test Substance</td>
<td>5</td>
</tr>
<tr>
<td>B. Reference Substance</td>
<td>5</td>
</tr>
<tr>
<td>III. EXPERIMENTARY</td>
<td>6</td>
</tr>
<tr>
<td>A. Sample Receipt, Logging and Storage</td>
<td>6</td>
</tr>
<tr>
<td>B. Summary of Analytical Method</td>
<td>6</td>
</tr>
<tr>
<td>C. Summary of Method Modifications</td>
<td>7</td>
</tr>
<tr>
<td>D. Calculations</td>
<td>8</td>
</tr>
<tr>
<td>E. Statistical Analyses</td>
<td>8</td>
</tr>
<tr>
<td>F. Results</td>
<td>9</td>
</tr>
<tr>
<td>IV. DISPOSITION OF SAMPLES AND RAW DATA</td>
<td>9</td>
</tr>
<tr>
<td>V. STUDY PARTICIPANTS LIST</td>
<td>9</td>
</tr>
<tr>
<td>FIGURES</td>
<td></td>
</tr>
<tr>
<td>Figure 1 HPLC Calibration Standard</td>
<td>10</td>
</tr>
<tr>
<td>Figure 2 Rely Formulation Sample, EO7125-F1A</td>
<td>11</td>
</tr>
<tr>
<td>APPENDICES</td>
<td></td>
</tr>
<tr>
<td>APPENDIX I Certificate of Analysis</td>
<td>12</td>
</tr>
</tbody>
</table>

EN-CAS Project # 97-0043
Page 4
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.

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

B. Reference Substance

The reference substance was stored under freezer storage conditions at approximately <-15°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.

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

EN-CAS Project # 97-0043
Page 5
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

\[
\text{% content of sample} = \frac{C_1 \cdot W_1}{A \cdot W_2} \times P
\]

\(A\) = Average area of active ingredient in calibration standard.

\(C_1\) = Area of active ingredient in sample.

\(W_1\) = Weight of calibration standard.

\(W_2\) = 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

\(C_1\) = 12190232 counts

\(W_1\) = 0.05055 g

\(W_2\) = 0.25150 g

\(P\) = 99.2%

\[
\text{% w/w glufosinate content of sample} = \frac{12190232 \text{ counts}}{20754637 \text{ counts}} \times \frac{0.05055 \text{ g}}{0.25150 \text{ g}} \times 99.2\% = 11.7\%
\]

E. Statistical Analyses

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

<table>
<thead>
<tr>
<th>Product</th>
<th>Batch #</th>
<th>Date Received</th>
<th>Mean Percent $^a$ Active Ingredient (Glufosinate Ammonium)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rely</td>
<td>RLREJE05-01</td>
<td>4/4/97</td>
<td>11.7</td>
</tr>
</tbody>
</table>

$^a$ \(11.7\% + 11.7\% + 11.7\% + 11.7\% = 46.8 \div 4 = 11.7\%$

IV. \textbf{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. \textbf{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

EN-CAS Project # 97-0043  
Page 9
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

EN-CAS Project # 97-0043
Page 11
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)

<table>
<thead>
<tr>
<th>Name</th>
<th>Content</th>
<th>Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>Rely</td>
<td>11.7% w/w</td>
<td>NA-A120</td>
</tr>
</tbody>
</table>

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

Report and raw data are archived at the testing facility.

Principal Analytical Investigator:  David A. Winkler, B.S.
Group Leader

EN-CAS Management Approval:  Bert Clayton, B.S.
Manager, Analytical and Residue Studies

TESTING FACILITY

Sponsor Representative:  Lance Fritz
Study Director

EN-CAS Project # 97-0043
Page 15