



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
WASHINGTON, D.C. 20460

SEP 14 1989

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM:

SUBJECT: PP#8F3607: Glufosinate-Ammonium (IGNITE) in or on Soybean Seed, Apples, Grapes, Field Corn (Grain, Forage, Fodder, and Silage), Nuts, and Almond Hulls. Report of Method Validation of Revised Proposed Enforcement Methodology by Analytical Chemistry Section.

FROM: Joel Garbus, PhD., Chemist
Permanent Tolerance Section III
Dietary Exposure Branch (H7509c)

Joel Garbus

THRU: R. D. Schmitt, PhD., Chief
Dietary Exposure Branch
Health Effects Division (H7509C)

Edward J. J. J. J.

TO: A. Heyward, PM-15
Registration Division (H7505C)

Introduction

Hoechst-Celanese has petitioned for permanent tolerances for the herbicide glufosinate-ammonium, (Ignite), and its metabolite, 3-methylphosphinopropionic acid, in or on soybean seed, apples, grapes, field corn (grain, forage, fodder, and silage), and nuts at 0.05 ppm and in or on almond hulls at 0.50 ppm.

DEB requested that a method validation be initiated for glufosinate-ammonium and its metabolite on the above r.a.c's using the method submitted with the petition. (DEB memo of 3/21/88)

The Analytical Chemistry Section responded to this request by stating that before ACS could initiate laboratory work, deficiencies in the description of the proposed enforcement methodology would need to be addressed or resolved. (Warren Bontoyan, memo, 6/23/88)

Hoechst-Celanese submitted a revised version of the proposed enforcement methodology that, according to the petitioner addressed all of the deficiencies noted in ACS's memo.

DEB requested that method validation be conducted for glufosinate-ammonium and its metabolite, 3-methylphosphinopropionic acid on the selected r.a.c.'s corn grain, corn forage, corn fodder, and soybeans. (J. Garbus, memo, 7/31/89)

The Analytical Chemistry Branch has transmitted its report to DEB in a memo by D. M. Swineford and C.J. Stafford dated 8/29/89.

Method Summary

Glufosinate-ammonium and its metabolite are extracted from homogenized samples using water as solvent. The filtered extract is subjected to ion exchange chromatography and the recovered analytes are acetylated, cleaned up, and quantified by GC.

MTO REPORT

Method: Enforcement Procedure: Analytical Method - HRAV 5A
 Chromatographic Determination of HOE-039866, [Ammonium-DL-Homoalanine-4-yl (Methyl)phosphinate] and its Metabolite, HOE-061517, 3-Methylphosphinico-Propionic Acid] as a Residue in Apples, Grapes, Soybeans, Corn, and Tree

Nuts

<u>Commodity</u>	<u>Chemical Added</u>	<u>PPM Added</u>	<u>PPM Found % Recovery</u>	
Soybean Seed	control	0.0	N.D.	
	control	0.0	N.D.	
Soybean Seed	glufosinate	0.05	0.0557	120
	glufosinate	0.05	0.0615	130
	glufosinate	0.10	0.1049	100
	glufosinate	0.10	0.1059	110
Soybean Seed	Metabolite ¹	0.05	0.0495	100
	Metabolite ¹	0.05	0.0495	100
	Metabolite ¹	0.10	0.1295	120
	Metabolite ¹	0.10	0.1047	110
Corn Grain	control	0.0	N.D.	
	control	0.0	N.D.	
Corn Grain	glufosinate	0.05	0.0382	77
	glufosinate	0.05	0.0504	100
	glufosinate	0.10	0.0635	64
	glufosinate	0.10	0.0635	64
Corn Grain	Metabolite ¹	0.05	0.0504	100
	Metabolite ¹	0.05	0.0592	120
	Metabolite ¹	0.10	0.0758	76
	Metabolite ¹	0.10	0.0849	85
Corn Forage	control	0.0	N.D.	
	control	0.0	N.D.	
Corn Forage	glufosinate	0.05	0.0466	93
	glufosinate	0.05	0.0456	90
	glufosinate	0.10	0.0880	88
	glufosinate	0.10	0.0697	70
Corn Forage	Metabolite ¹	0.05	0.0620	120
	Metabolite ¹	0.05	0.0662	130
	Metabolite ¹	0.10	0.1049	100
	Metabolite ¹	0.10	0.1059	110

<u>Commodity</u>	<u>Chemical Added</u>	<u>PPM Added</u>	<u>PPM Found & Recovery</u>	
Corn Fodder	control	0.0	N.D.	
	control	0.0	N.D.	
Corn Fodder	glufosinate	0.05	0.0414	83
	glufosinate	0.05	0.0440	83
	glufosinate	0.10	0.1015	100
	glufosinate	0.10	0.0930	93
Corn Fodder	Metabolite ¹	0.05	0.0575	120
	Metabolite ¹	0.05	0.0569	110
	Metabolite ¹	0.10	0.1307	130
	Metabolite ¹	0.10	0.1262	130

¹ Metabolite = 3-methylphosphinicopropionic acid

Modifications to method (major or minor):

1. The recommended glass microfiber filter (Whatman GF/C 1.2 micron) clogged readily. A Whatman 934-AH (1.5 micron) gave better results.
2. The 25 gram sample of dried corn fodder completely absorbed the 200 ml of water recommended for extraction. The method was modified by using 1000 ml of water for extraction. A 100 ml aliquot was used in the ion-exchange step, maintaining the sample/aliquot ratio.

Special precautions to be taken:

none

Source of analytical reference standards:

Hoechst-Rousell Agri-Vet Company

If derivitized standard is used, give source:

Same

Instrumentation for quantitation:

HP 5880 with phosphorus mode FPD

Instrumentation for confirmation:

none

If instrument parameter differ from those given in method, list parameters used:

Initial column temperature was 120°C; final column temperature was 235°C. Other parameters as specified in method.

Comments

1. The submitted method gave satisfactory results at the 0.1 and 0.05 ppm levels.
2. Six samples can be analyzed in 2 eight hour days.
3. The limits of detection of this method are estimated to be 0.02 ppm.

DEB's Comments and Recommendation:

Recoveries of glufosinate-ammonium (Ignite) and its metabolite from soybeans, corn grain, corn forage, and corn fodder at both levels of fortification (0.05 and 0.10 ppm) are satisfactory.

The petitioner should submit a revised method incorporating the modifications suggested by the ACS report.

Contingent upon the submission of a modified method as suggested above, we conclude that HRAV Method 5A is adequate for the enforcement of the tolerances requested in PP#8F3607 and that HRAV Method 5A be included in PAM II when the requested permanent tolerances are established.

The petitioner should insure that standards of the parent and the metabolite and of their derivitized forms are available in the Standards Repository.

The report of the ACS should be forwarded to the petitioner. A copy is attached for that purpose.

cc with attachment: PM-15

cc without attachment: PP#8F3607, R.F., S.F., Circ., Reviewer, M. Bradley, D. Marlow, Thompson FDA, PMSD/ISB
RDI:PE:9/13/89:RAL:9/13/89
TS-769:JG:jg:803c:557-1405:9/13/89