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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
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March 31, 2003

MEMORANDUM

DP Barcode: D284692

SUBJECT: Fluroxypyr Method Review  
Report No. ECM0205S1-S3

FROM: Aubry E. Dupuy, Jr., Chief *Aubry E. Dupuy, Jr.*  
BEAD/Environmental Chemistry Lab

TO: Dana Spatz  
EFED/Environmental Risk Branch 2 (7507-C)

THRU: Hardip Singh  
EFED/Environmental Risk Branch (7507-C)

As requested ECL has completed an Environmental Chemistry Method Review for fluroxypyr and its major metabolites in soil, MRID #44896703, using a method submitted by Dow AgroScience LLC, entitled "Determination of Residues of Fluroxypyr and its Major Metabolites in Soil by Capillary Gas Chromatography with Mass Selective Detection".

The attached method lab review report includes three parts:

Part I: Summary and Conclusions

ECL's opinion of the acceptability of the method is presented.

Part II: Problems Found During Method Review

A discussion of minor deficiencies discovered during review or any modifications made by the independent lab.



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Part III: Summary of Performance Data

A summary of the registrant's method performance data and the ILV's method performance data. A completed SEP check-list is attached.

If you have any questions concerning this report, please contact Henry Shoemaker at (228) 688-1222 or Aubry Dupuy at (228) 688-3212.

Attachments

cc: Christian Byrne, QA Officer  
BEAD/Environmental Chemistry Lab

Henry Shoemaker, Chemist  
BEAD/Environmental Chemistry Lab

ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT  
NUMBER ECM0205S1-S3

Determination of Residues of Fluroxypyr,  
Fluroxypyr-DCP, and Fluroxypyr-MP in Soil

ENVIRONMENTAL CHEMISTRY LABORATORY  
BIOLOGICAL AND ECONOMIC ANALYSIS DIVISION

Prepared by: Henry Shoemaker Date: 2/18/03  
Henry Shoemaker ECL Chemist

Reviewed by: Christian Byrns Date: 02/18/03  
Christian Byrns, ECL QA Officer

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PART I

SUMMARY AND CONCLUSIONS

The Environmental Chemistry Laboratory (ECL) has completed an Environmental Chemistry Method Review of fluroxypyr and its major metabolites in soil. This method, MRID# 448967-03, submitted by Dow AgroSciences LLC, is entitled, "Determination of Residues of Fluroxypyr and its Major Metabolites in Soil by Capillary Gas Chromatography with Mass Selective Detection".

From the review of the registrant method and the independent laboratory validation data, ECL concludes that this method appears to be sound and capable of being used to determine Fluroxypyr and its major metabolites in soil with acceptable precision ( $\leq 20\%$ ) and accuracy (70-120%). The precision/accuracy data at the LOQ (0.010 ppm) and other levels for both the registrant and independent laboratory are displayed in Part III- Summary of Performance Data, on page 4 of this report.

Part II

Problems Found During Method Review

No problems were encountered in the registrant's or independent lab's reports.

PART III

SUMMARY OF PERFORMANCE DATA OF REGISTRANT AND ILV

Registrant Data - Dow AgroScience LLC

Fortification Conc. 0.010 ppm = LOQ

Compound	N	Recovery	RSD
Fluroxypyr	8	79.0	4%
Fluroxypyr-DCP	8	83.8	10%
Fluroxypyr-MP	8	84.6%	11%

Fortification Conc. 0.25 ppm

Fluroxypyr	4	83.5%	3%
Fluroxypyr-DCP	4	83.3%	3%
Fluroxypyr-MP	4	76.5%	5%

Fortification Conc. 1.00 ppm

Fluroxypyr	3	75.0%	3%
Fluroxypyr-DCP	3	79.0%	3%
Fluroxypyr-MP	3	70.0%	8%

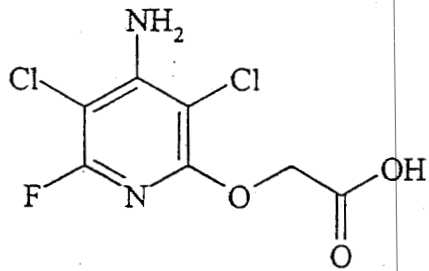
Independent Laboratory Data - Enviro-Bio-Tech, Ltd.

Fortification Conc. 0.010 ppm = LOQ

Compound	N	Recovery	RSD
Fluroxypyr	5	102.2%	4%
Fluroxypyr-DCP	5	81.4%	6%
Fluroxypyr-MP	5	83.2%	4%

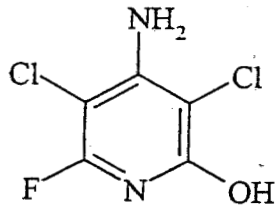
Fortification Conc. 0.10 ppm = 10xLOQ

Fluroxypyr	5	94.4%	7%
Fluroxypyr-DCP	5	85.8%	2%
Fluroxypyr-MP	5	84.2%	12%



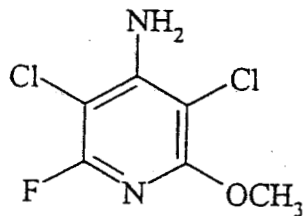
Fluroxypyr  
CAS No. 69377-81-7

((4-amino-3,5-dichloro-6-fluoro-2-pyridinyl)oxy)acetic acid)



Fluroxypyr-DCP  
CAS No. 94133-62-7

(4-amino-3,5-dichloro-6-fluoro-2-pyridinol)



Fluroxypyr-MP  
CAS No. 35622-80-1

(4-amino-3,5-dichloro-6-fluoro-2-methoxypyridine)

**APPENDIX B**

**ENVIRONMENTAL CHEMISTRY METHODS (ECMs) PROGRAM  
STANDARD EVALUATION PROCEDURE (SEP) CHECKLIST  
BACKGROUND AND INITIAL REVIEW INFORMATION**

*I. Background Information*

<b>A.</b>	<b>Title of Method</b>	Determination of Residues of Fluroxypyr and its Major Metabolites in Soil by Capillary Gas Chromatography with Mass Selective Detection.
<b>B.</b>	<b>ECM No.</b>	ECM0205S1-S3
<b>C.</b>	<b>MRID No.</b>	44896703
<b>D.</b>	<b>Matrix</b>	Soil
<b>E.</b>	<b>Analyte(s) detected</b>	Fluroxypyr, Fluroxypyr-DCP, Fluroxypyr-MP

*II. Information About the Laboratory*

<b>A.</b>	<b>Name</b>	Global Environmental Chemistry Laboratory, Dow AgroScience LLC
<b>B.</b>	<b>Address</b>	9330 Zionsville Road Indianapolis, Indiana 46268-1054
<b>C.</b>	<b>Telephone No.</b>	(317) 337-3638
<b>D.</b>	<b>Name of the Study Director</b>	D. D. Shackelford, Dow AgroSciences LLC
<b>E.</b>	<b>Name of the Lead Chemist</b>	J. J. Jachetta
<b>F.</b>	<b>Laboratory Validation:</b>	Primary



III. Method Summary Information for Analyte(s):

A.	<b>Is the Method CLASSIFIED or CONFIDENTIAL</b>	No
B.	<b>Sample Preparation</b>	Crush with hammer, blend with dry ice, and grind using a hammer mill with a 3/16-inch size screen.
C.	<b>Sample Extraction</b>	Sonicate in a capped vial with a solution of 90% acetone/ 10% 0.1 N hydrochloric acid.
D.	<b>Sample Cleanup</b>	Purified using C <sub>18</sub> solid phase extraction (SPE)
E.	<b>Sample Derivatization (if applicable)</b>	Extract is hydrolyzed with 1.0 N sodium hydroxide
F.	<b>Sample Analysis</b>	
1.	<b>Instrumentation</b>	H-P Model 5890 Series II gas chromatograph H-P Model 7673 autoinjector H-P Model 5971A mass selective detector H-P Model G1034C data system
2.	<b>Primary Column</b>	J&W fused silica capillary, DB-1701 10m x 0.18 mm i.d., 0.4- $\mu$ m film thickness
3.	<b>Confirmatory Column (If Any)</b>	
4.	<b>Detector</b>	See F-1 above
5.	<b>Other Confirmatory Techniques (If Any)</b>	
6.	<b>Other Relevant Information</b>	

<b>G.</b>	<b>Detection and Quantitation Limits</b>			
<b>1.</b>	<b>Limit of Quantitation (LOQ)</b>			
	<b>Claimed in Method</b>	<b>0.010 µg/g</b>	<b>Estimated</b>	
<b>2.</b>	<b>Method Detection Limit (MDL)</b>			
	<b>Claimed in Method</b>	<b>calculated at 0.001, 0.002, 0.003 µg/g, respectively, for the three analytes</b>	<b>Estimated</b>	
<b>H.</b>	<b>Recovery /Precision Data</b>		<b>0.010 µg/g</b>	<b>0.25 µg/g</b> <b>1.00 µg/g</b>
		<b>Fluroxypyr</b>	<b>N = 8</b> <b><math>\bar{x}</math> = 79.0%</b> <b>RSD = 4%</b>	<b>N = 4</b> <b><math>\bar{x}</math> = 83.5%</b> <b>RSD = 3%</b>
		<b>Fluroxypyr-DCP</b>	<b>N = 8</b> <b><math>\bar{x}</math> = 83.8%</b> <b>RSD = 10%</b>	<b>N = 3</b> <b><math>\bar{x}</math> = 75.0%</b> <b>RSD = 3%</b>
		<b>Fluroxypyr-MP</b>	<b>N = 4</b> <b><math>\bar{x}</math> = 84.6%</b> <b>RSD = 11%</b>	<b>N = 3</b> <b><math>\bar{x}</math> = 70.0%</b> <b>RSD = 8%</b>

IV. Detailed Information about the Method

		YES	NO	REVIEW FURTHER
<b>A.</b>	<b>Is the method marked CONFIDENTIAL?</b>		X	
<b>B.</b>	<b>Is it the most up-to-date method?</b>	X		
<b>C.</b>	<b>Does the method require spiking with the analytes(s) of interest?</b>	X		
<b>D.</b>	<b>If the method requires explosive or carcinogenic reagents, are proper precautions explained?</b>	X		
<b>E.</b>	<b>Is the following information supplied?</b>			
<b>1.</b>	<b>Detailed stepwise description of</b>			
<b>a.</b>	<b>The sample preparation procedure</b>	X		
<b>b.</b>	<b>The sample spiking procedure</b>	X		
<b>c.</b>	<b>The extraction procedure</b>	X		
<b>d.</b>	<b>The derivatization procedure</b>	X		
<b>e.</b>	<b>The cleanup procedure</b>	X		
<b>f.</b>	<b>The analysis procedure</b>	X		
<b>2.</b>	<b>Procedures for</b>			
<b>a.</b>	<b>Preparation of standards</b>	X		
<b>b.</b>	<b>Calibration of instrument</b>	X		
<b>3.</b>	<b>List of glassware and chemicals</b>	X		
<b>a.</b>	<b>Are sources recommended?</b>	X		
<b>b.</b>	<b>Are they commercially available?</b>	X		

		YES	NO	REVIEW FURTHER
4.	Name, model, etc., of the instrument, column, detector, etc., used	X		
a.	Are sources recommended?	X		
b.	Are they commercially available?	X		
5.	MDL			
a.	Is there an explanation of how it was calculated?	X		
b.	Is it a scientifically accepted procedure?	X		
c.	Is the matrix blank free of interferences(s) at the retention time, wavelength, etc., of the analyte(s) of interest?	X		
6.	LOQ			
a.	Is there an explanation of how it was calculated?	X		
b.	Is it a scientifically accepted procedure?	X		
7.	Precision and accuracy data			
a.	Were there an adequate number of spiked samples analyzed?	X		
b.	Are the mean recoveries between 70-120%?	X		
c.	Are the RSDs of the replicates 20% or less at the LOQ, or above?	X		
8.	Description and/or explanation of			
a.	Areas where problems may be encountered?	X		
b.	Steps that are critical?	X		
c.	Interferences that may be encountered?	X		

		YES	NO	REVIEW FURTHER
9.	Characterization of the Matrix(es)		X	

V. Representative Chromatograms

		YES	NO	REVIEW FURTHER
A.	Are there representative chromatograms for			
1.	Analyte(s) in each matrix at the MDL, LOQ, and 10 x LOQ?	X Not MDL		
2.	Method blanks?		X	
3.	Matrix blanks?	X		
4.	Standard curves?	X		
5.	Standards that can be used to recalculate some of the values for analyte(s) in the sample chromatograms?	X		
B.	Can the responses of the analyte(s) in the chromatograms of the lowest spiking level be accurately measured?	X		

VI. Good Laboratory Practice Standards (GLP)

		YES	NO	REVIEW FURTHER
A.	Is there a statement of adherence to the FIFRA.GLP?	X		

*VII. Independent Lab Validation (ILV)*

		YES	NO	REVIEW FURTHER
A.	Was an ILV performed?	X		
B.	Did the ILV's precision/accuracy data meet the criteria established on page 3 of the Data Reporting Guidelines (OPP-00405;FRL-4943-5)?	X		
C.	Were recommendations of major or minor modifications to the method made by the independent lab performing the ILV? If major modifications were suggested, what were they?		X	

*VIII. Completeness*

		YES	NO	REVIEW FURTHER
A.	Has enough information been supplied to do a proper review?	X		
B.	Has enough information been supplied to do a laboratory evaluation, if requested?	X		
C.	Are all steps in the method scientifically sound?	X		
D.	Is a confirmatory method or technique provided?		X	
E.	Check the category below which best describes this ECM>			
1.	Satisfactory	X		
2.	Major Deficiencies			
3.	Minor Deficiencies			

*XI. Recommendations*

ECL concludes that this method appears to be sound and capable of being used to determine residues of fluroxypyr and its major metabolites in soil with acceptable precision and accuracy.

Name and Signature of Reviewer: *HENRY SHOEMAKER, Henry Shoemaker*

Date Initial Review was Assigned: *10-16-02*

Date Initial Review was Completed: *12-10-02*

Date Final Review was Completed: *03/28/03*

Signature of Laboratory Chief: *Audrey E Dupuy, h.c.*

Name(s) and Signature(s) of Other Reviewers: *CHUCK KENNEDY, Charles Kennedy 01/21/03*  
*CHRISTIAN BYRNE Christian Byrne*