



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF PESTICIDE PROGRAMS  
ENVIRONMENTAL CHEMISTRY LABORATORY  
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March 07, 2002

MEMORANDUM

DP Barcode: D263960

SUBJECT: IM-1-4 ( Metabolite of Acetamiprid ) Method Review  
Report No. ECM0176S3

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

TO: Dana Spatz  
EFED/Environmental Risk Branch 4 (7507-C)

As requested ECL has completed an Environmental Chemistry Method Review for IM-1-4 in soil, MRID # 449885-16 using a method submitted by Aventis CropScience, formerly Rhône-Poulenc Ag. Co., entitled "NI-25, Method of Analysis for IM-1-4, a Metabolite of NI-25, in Soil Using LC/MS/MS".

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.

### 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 ECM0176S3

NI-25, Method of Analysis for IM-1-4, a Metabolite  
of NI-25, in Soil Using LC/MS/MS.

ENVIRONMENTAL CHEMISTRY LABORATORY  
BIOLOGICAL AND ECONOMIC ANALYSIS DIVISION

January 17, 2002

Prepared by:  Date: 3/04/02  
Henry Shoemaker, ECL Chemist

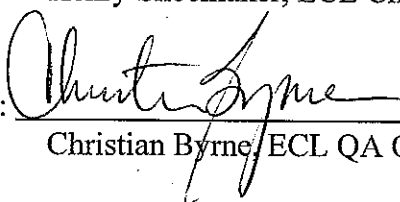
Reviewed by:  Date: 03/05/02  
Christian Byrne, ECL QA Officer

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## PART I

## SUMMARY AND CONCLUSIONS

The Environmental Chemistry Laboratory (ECL) has completed an Environmental Chemistry Method Review of IM-1-4, a metabolite of Acetamiprid (NI-25), in soil. This method, MRID# 449885-16, submitted by Aventis Cropscience, formerly Rhône-Poulenc Ag. Co., is entitled, "NI-25, Method of Analysis for IM-1-4, a Metabolite of NI-25, in Soil Using LC/MS/MS". Centre Analytical Laboratories performed the independent laboratory validation (ILV).

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 IM-1-4 in soil with acceptable precision and accuracy. The precision/accuracy data at the LOQ (10.0 ppb) 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

The registrant's report was well written with clear data and I found no problems during the method review.

## PART III

## SUMMARY OF PERFORMANCE DATA OF REGISTRANT AND ILV

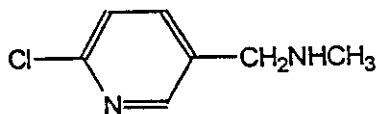
## Registrant Data - Aventis Cropscience

Fort. Conc.	N	Recovery	Range	RSD
10.0 ppb (LOQ)	5	88.8%	83.2%-93.5%	4.6%
20.0 ppb	6	89.3%	79.5%-112.6%	13.9%
300 ppb	11	90.2%	76.5%-108.0%	9.4%

## Independent Laboratory Data - Centre Analytical Laboratories

Extraction Date	Analysis Date	CAL Sample ID	Fortification Level (ppb)	Recovery (%) IM-1-4
01/08-09/99	01/12/99	9815298 Spk A2	10	75.6
01/08-09/99	01/12/99	9815298 Spk B2	10	75.0
01/08-09/99	01/12/99	9815298 Spk C2	10	67.0
01/08-09/99	01/12/99	9815298 Spk D2	10	61.7
01/08-09/99	01/12/99	9815298 Spk E2	10	65.4
Average:				68.9
Standard Deviation:				6.1
Relative Standard Deviation:				8.9
01/08-09/99	01/12/99	9815298 Spk F2	100	78.7
01/08-09/99	01/12/99	9815298 Spk G2	100	80.1
01/08-09/99	01/12/99	9815298 Spk H2	100	53.3
01/08-09/99	01/12/99	9815298 Spk I2	100	86.2
01/08-09/99	01/12/99	9815298 Spk J2	100	85.5
Average:				76.8
Standard Deviation:				13.5
Relative Standard Deviation:				17.6

Name or Code: IM-1-4  
Chemical Name : N-methyl(6-chloro-3-pyridyl)methylamine  
CAS No.: none  
Molecular Weight : 155.5



APPENDIX B

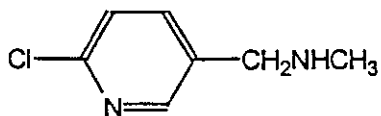
ATTACHMENT 1

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

I. Background Information

- A. Title of Method NI-25, Method of Analysis for IM-1-4, A Metabolite of NI-25, Using LC/MS/MS.
- B. ECM No. ECM0176S3
- C. MRID No. 449885-16
- D. Matrix(es) Soil
- E. Analyte(s) detected IM-1-4  
N-methyl(6-chloro-3-pyridyl)methylamine

Name or Code: IM-1-4  
Chemical Name : N-methyl(6-chloro-3-pyridyl)methylamine  
CAS No.: none  
Molecular Weight : 155.5





**II. Information About the Laboratory**

- A. Name Aventis Cropscience (formerly Rhône-Poulinc Ag.Co.)
- B. Address  
2 T.W. Alexander Drive, Research Triangle Park, North Carolina
- C. Telephone No. 919-549-2634
- D. Name of the Study Director Ju Yang, Ph.D.
- E. Name of the Lead Chemist Kirk Blevins
- F. Laboratory Validation: Primary x Secondary

**III. Method Summary Information for Analyte(s):**

- A. Is the Method CLASSIFIED or CONFIDENTIAL no
- B. Sample Preparation None
- C. Sample Extraction Extracted with 0.4 N NH<sub>4</sub>CL in water and methanol mixture (40:60) using Dionex accelerated solvent extractor (ASE).
- D. Sample Cleanup Extract is loaded onto a Extrelut cartridge and eluted with dichloromethane.  
The dichloromethane is dried and reconstituted using acetonitrile/water.
- E. Sample Derivatization (If Applicable) None
- F. Sample Analysis
1. Instrumentation Sciex API III+ LC/MS/MS
  2.
  3. Primary Column YMC ODS-AQ, 3.0 x 150 mm, 5µm particle size
  4. Confirmatory Column (If Any) None
  5. Detector LC/MS/MS
  6. Other Confirmatory Techniques (If Any) None
  7. Other Relevant Information

G. Detection and Quantitation Limits

1. Limit of Quantitation (LOQ)

Claimed in Method 10 ppb Estimated \_\_\_\_\_

Method Detection Limit (MDL)

Claimed in Method ppm Estimated 3.33 ppb

H. Recovery (Accuracy) Data

Compound	ppb	N	% Rec	RSD
IM-1-4	10.0	5	88.8%	15.4%
	20.0	6	89.3%	13.9%
	300	11	90.2%	9.4%

I. Precision

IV. Detailed Information About the Method

	<u>Yes</u>	<u>No</u>	<u>Further</u>
A. Is the method marked CONFIDENTIAL?	_____	<u>X</u>	_____
	<u>Yes</u>	<u>No</u>	<u>Review Further</u>
B. Is it the most up-to-date method?	<u>X</u>	_____	_____
C. Does the method require spiking with the analyte(s) of interest?	<u>X</u>	_____	_____
D. If the method requires explosive or carcinogenic reagents, are proper precautions explained?	<u>X</u>	_____	_____

E. Is the following information supplied?

		<u>Yes</u>	<u>No</u>	<u>Review Further</u>
1.	Detailed stepwise description of			
	a. The sample preparation procedure	<u>X</u>	_____	_____
	b. The sample spiking procedure	<u>X</u>	_____	_____
	c. The extraction procedure	<u>X</u>	_____	_____
	d. The derivatization procedure	<u>N/A</u>	_____	_____
	e. The cleanup procedure	<u>X</u>	_____	_____
	f. The analysis procedure	<u>X</u>	_____	_____
2.	Procedures for			
	a. Preparation of standards	<u>X</u>	_____	_____
	b. Calibration of instrument	<u>X</u>	_____	_____
3.	List of glassware and chemicals			
	a. Are sources recommended?	<u>X</u>	_____	_____
	b. Are they commercially available?	<u>X</u>	_____	_____
4.	Name, model, etc., of the instrument, Column, detector, etc., used			
	a. Are sources recommended?	<u>X</u>	_____	_____
	b. Are they commercially available?	<u>X</u>	_____	_____
5.	MDL	<u>Yes</u>	<u>No</u>	<u>Review Further</u>
	a. Is there an explanation of how it was calculated?	<u>X</u>	_____	_____
	b. Is it a scientifically accepted procedure?	<u>X</u>	_____	_____
	c. Is the matrix blank free of interference(s) at the retention time, wavelength, etc., of the analyte(s) of interest	<u>X</u>	_____	_____

- |    |                                    |                                                                  |          |       |
|----|------------------------------------|------------------------------------------------------------------|----------|-------|
| 6. | LOQ                                |                                                                  |          |       |
|    | a.                                 | Is there an explanation of how it was calculated?                | <u>X</u> | _____ |
|    | b.                                 | Is it scientifically accepted procedure?                         | <u>X</u> | _____ |
| 7. | Precision and accuracy data        |                                                                  |          |       |
|    | a.                                 | Were there an adequate number of spiked samples analyzed?        | <u>X</u> | _____ |
|    | b.                                 | Are the mean recoveries between 70-120%?                         | <u>X</u> | _____ |
|    | c.                                 | Are the RSDs of the replicates 20% or less at the LOQ, or above? | <u>X</u> | _____ |
| 8. | Description and/or explanation of  |                                                                  |          |       |
|    | a.                                 | Areas where problems may be encountered?                         | <u>X</u> | _____ |
|    | b.                                 | Steps that are critical?                                         | <u>X</u> | _____ |
|    | c.                                 | Interferences that may be encountered?                           | <u>X</u> | _____ |
| 9. | Characterization of the matrix(es) |                                                                  | <u>X</u> | _____ |

**V. Representative Chromatograms**

- |    | <u>Yes</u>                                                                                                   | <u>No</u>                                                                                                | <u>Review Further</u>       |
|----|--------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------------|-----------------------------|
| A. | Are there representative chromatograms for                                                                   |                                                                                                          |                             |
|    | 1.                                                                                                           | Analyte(s) in each matrix at the MDL, LOQ, and 10 x LOQ?                                                 | <u>X</u> ( <u>Not MDL</u> ) |
|    | 2.                                                                                                           | Method blanks?                                                                                           | <u>X</u>                    |
|    | 3.                                                                                                           | Matrix blanks?                                                                                           | <u>X</u>                    |
|    | 4.                                                                                                           | Standard curves?                                                                                         | <u>X</u>                    |
|    | 5.                                                                                                           | Standards that can be used to recalculate Some of the values for analyte(s) in the Sample chromatograms? | <u>X</u>                    |
| B. | Can the responses of the analyte(s) in the chromatograms of the lowest spiking level be accurately measured? |                                                                                                          |                             |
|    |                                                                                                              |                                                                                                          | <u>X</u>                    |

**VI. Good Laboratory Practice Standards (GLP)**

	<u>Yes</u>	<u>No</u>	<u>Review Further</u>
A. Is there a statement of adherence to the FIFRA/GLP?	<u>X</u>	_____	_____

**VII. Independent Lab Validation (ILV)**

A. Was an ILV performed?	<u>X</u>	_____	_____
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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)?	<u>X</u>	_____	_____
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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?	_____	<u>X</u>	_____
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**VIII. Completeness**

	<u>Yes</u>	<u>No</u>	<u>Review Further</u>
A. Has enough information been supplied to do a proper review?	<u>X</u>	_____	_____

B. Has enough information been supplied to do a laboratory evaluation, if requested?	<u>X</u>	_____	_____
--------------------------------------------------------------------------------------	----------	-------	-------

C. Are all steps in the method scientifically sound?	<u>X</u>	_____	_____
------------------------------------------------------	----------	-------	-------

D. Is a confirmatory method or technique provided?	_____	<u>X</u>	_____
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E. Check the category below which best describes this ECM.	_____	_____	_____
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1. Satisfactory	<u>X</u>
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2. Major Deficiencies	_____
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3. Minor Deficiencies	_____
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IX. Recommendations

Acceptable

Name (print) and Signature of Reviewer:

HENRY SHOEMAKER, Henry Shoemaker

Date Initial Review was Assigned:

9-18-00

Date Initial Review was Completed:

9-04-01

Date Final Review was Completed:

2-17-02

Signature of Laboratory Chief:

Name(s) (print) and Signature(s) of Other Reviewers:

CHARLES D. KENNEDY / Charles Kennedy

CHRISTIAN BYRNE Christian Byrne