March 07, 2002

<u>MEMORANDUM</u>   DP Barcode: D263960

SUBJECT: IM-1-2 (Metabolite of Acetamiprid) Method Review
Report No. ECM0176S4

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-1-2 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-1-2, 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 ECM0176S4

NI-25, Method of Analysis for IM-1-2, 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-1-2, 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-1-2, 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-1-2 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)	33	80.0%	57.1%-104%	15.4%
100 ppb	6	82.9%	70.8%-93.8%	11.0%
300 ppb	6	80.5%	69.0%-92.8%	12.6%

Independent Laboratory Data - Centre Analytical Laboratories

Extraction Date	Analysis Date	CAL Sample ID	Fortification Level (ppb)	Recovery (%) IM-1-2
01/28-29/99	01/29/99	9815298 Spk A4	10	88.5
01/28-29/99	01/29/99	9815298 Spk B4	10	89.1
01/28-29/99	01/29/99	9815298 Spk C4	10	85.1
01/28-29/99	01/29/99	9815298 Spk D4	10	88.2
01/28-29/99	01/29/99	9815298 Spk E4	10	83.1
			Average:	86.8
			Standard Deviation:	2.6
			Relative Standard Deviation:	3.0
01/28-29/99	01/29/99	9815298 Spk F4	100	77.4
01/28-29/99	01/29/99	9815298 Spk G4	100	75.6
01/28-29/99	01/29/99	9815298 Spk H4	100	74.9
01/28-29/99	01/29/99	9815298 Spk I4	100	75.0
01/28-29/99	01/29/99	9815298 Spk J4	100	75.2
			Average:	75.6
			Standard Deviation:	1.0
			Relative Standard Deviation:	1.4

APPENDIX A

Name or Code: IM-1-2

Chemical Name: (E)-N^2-carbamoyl-N^1-(6-chloro-3-pyridyl)methyl-N^2-cyano-N^1-methylacetamidine

CAS No.: none

Molecular Weight: 240.69
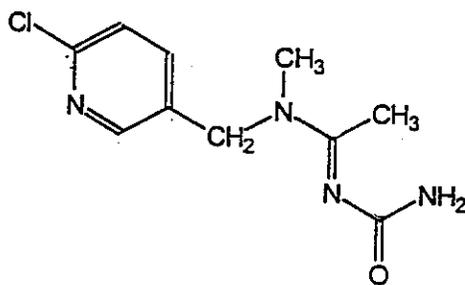
ATTACHMENT 1

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-1-2, A Metabolite of NI-25, Using LC/MS/MS.
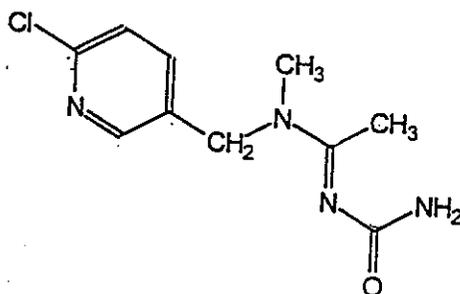
    B.   ECM No.   ECM0176S4

    C.   MRID No.   449885-16

    D.   Matrix(es)   Soil

    E.   Analyte(s) detected   IM-1-2

    (E)-N^2-carbamoyl-N^1-(6-chloro-3-pyridyl)methyl-N^2-cyano-N^1-methylacetamidine

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 50% acetonitrile/water using Dionex accelerated solvent extractor (ASE).

D.   Sample Cleanup   None

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-1-2	10.0	33	80.0%	15.4%
	100	6	82.9%	11.0%
	300	6	80.5%	12.6%

I.　~~Pricision~~

IV.　Detailed Information About the Method

		Yes	No	Further
A.	Is the method marked CONFIDENTIAL?		X	

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

E.   Is the following information supplied?

1.   Detailed stepwise description of

		Yes	No	Review Further
a.	The sample preparation procedure	X		
b.	The sample spiking procedure	X		
c.	The extraction procedure	X		
d.	The derivatization procedure	N/A		
e.	The cleanup procedure	N/A		
f.	The analysis procedure	X		

2.   Procedures for

		Yes	No	Review Further
a.	Preparation of standards	X		
b.	Calibration of instrument	X		

3.   List of glassware and chemicals

		Yes	No	Review Further
a.	Are sources recommended?	X		
b.	Are they commercially available?	X		

4.   Name, model, etc., of the instrument, Column, detector, etc., used

		Yes	No	Review Further
a.	Are sources recommended?	X		
b.	Are they commercially available?	X		

5.   MDL

		Yes	No	Review Further
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 interference(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 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 ___ ___

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 MOL)		
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)**

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 ____

IX.   Recommendations   _Acceptable_

Name (*print*) and Signature of Reviewer: _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 KENNEDY

CHRISTIAN BYRNE