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OFFICE OF PESTICIDE PROGRAMS
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November 15, 2001

MEMORANDUM

SUBJECT: Pyraclostrobin Method Review - ECM0191S1-6;
DP Barcode D271309

FROM: Aubry E. Dupuy, Jr., Branch Chief Aubry E Dupuy, Jr
BEAD/Environmental Chemistry Laboratory

TO: Sid Able
Environmental Fate and Effects Division
Environmental Risk Branch I

THRU: Hardip Singh, Senior
Gatekeeper Team/IO
Environmental Fate and Effects Division

The EFED/Environmental Fate and Effects Division has requested an Environmental Chemistry Method Review (ECMR) on the determination of Pyraclostrobin and its metabolites in soil using the BASF Corporation Agricultural Products Group method, "The Determination of BAS 500 F and Its Metabolites, BF-500-3, BF 500-4, BF 550-5, BF 500-6, and BF 500-7 in Soil Using LC-MS [9812/1]".

The attached method review report includes three parts:

Part I: Summary and Conclusions

In this section any problems encountered with the method and how they were handled are discussed. ECL's opinion of how well the method performed is also performed.

Part II: Discussion of Problems Encountered During Method Review

In this section the problems encountered in the registrant's method are discussed and their difficulties are assessed as to the overall effect on the validity of the method. The parameters reviewed include instrumental parameters, spiking levels, explanation of instrument calibration, representative sample and standard chromatograms and standard curves.

Part III: Summary of Performance Data of Registrant and ILVs

In this section the analytical recovery results for the minimum detection limit (MDL) [if present], the limit of quantitation (LOQ), and 10 x limit of quantitation (10xLOQ) of registrant and the independent laboratory validation (ILV) representatives.

If there are any questions regarding this report, please contact Christian Byrne at (228)-688-3213 or me at (228)-688-3212.

ATTACHMENTS

cc: Dr. Christian Byrne, QA Officer
BEAD/Environmental Chemistry Laboratory

Environmental Chemistry Method Review Report
ECM 0191S1-6

Validation of BASF Method No. D9812/1: BAS 500 F and Its Metabolites, BF 500-3, BF 500-4, BF 500-5, BF 500-6, & BF 500-7 in Soil Using LC-MS

Environmental Chemistry Laboratory
Biological and Economic Analysis Division

October 19, 2001

Prepared by: Christian Byrne, _____ Date: 10/26/01
ECL Chemist Signature

Reviewed by: Elizabeth Flynt, _____ Date: 11/5/01
ECL QA Coordinator Signature

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PART I

Summary and Conclusions

The Environmental Chemistry Branch (ECB) has completed the Environmental Chemistry Method Review (ECMR) for BAS 500 F (Pyraclostrobin) and its five metabolites: BF 500-3, BF 500-4, BF 500-5, BF 500-6, & BF 500-7 in soil. The method appears to be suitable for the detection of BAS 500 F (Pyraclostrobin) and BF 500-3, BF 500-4, BF 500-5, BF 500-6, & BF 500-7 in soil at levels at or greater than 0.010 µg/g [10.0 ppb (parts-per-billion)]. The performing laboratory was BASF Corporation Agricultural Products Group, Research Triangle Park, North Carolina. The independent laboratory validation (ILV) was performed by Battelle Laboratory, Columbus, Ohio. The MRID is # 451187-7 and the method used for the ECMR is entitled **- Validation of BASF Method No. D9812/1: The Determination of BAS 500 F and Its Metabolites, BF 500-3, BF 500-4, BF 500-5, BF 500-6, and BF 500-7 in Soil Using LC-MS.**

The analytical method involved the separation of BAS 500 F (Pyraclostrobin) and its metabolites from soil by repetitive extractions with acetonitrile and followed by 0.1 N sodium hydroxide solution and collected separately. The alkaline extract was acidified to approximately pH 2 and re-extracted with ethyl acetate. This extract was reduced to dryness. A small amount of triethylamine was added to the acetonitrile extract and it was reduced to approximately 40-50 ml and combined with the dried alkaline/ethyl acetate extract. The two extracts were then combined and reduced to approximately 10 ml and re-diluted with a 30:70 (acetonitrile:water) with 0.01% formic acid and 10 mM ammonium formate solution. This solution underwent HPLC-MS analysis.

ECB estimated that the limits of detection (LODs) for BAS 500 F (Pyraclostrobin) and its metabolites in soil were estimated at 5.0 ppb from the data provided by the registrant. The registrant determined the limit of quantitation (LOQ) to be 10.0 ppb [0.010 ppm]. The accuracy and precision results between the registrant and ILV (Battelle) at various spiking concentrations were comparable. The BASF Corporation laboratory demonstrated average percent recoveries for BAS 500 F @ 10.0 ppb (LOQ) and 100 ppb (10 x LOQ) of 98 and 93%, respectively; for BF 500-3 of 102 and 96%, respectively; for BF 500-4 of 86 and 91%, respectively; for BF 500-5 of 88 and 79% respectively; for BF 500-6 of 101and 90%, respectively; and for BF 500-7 of 97 and 89%, respectively. The Battelle laboratory demonstrated average percent recoveries for BAS 500 F @ 10.0 ppb (LOQ) and 100 ppb (10 x LOQ) of 97 and 85%, respectively; for BF 500-3 of 92 and 85%, respectively; for BF 500-4 of 72 and 77%, respectively; for BF 500-5 of 85 and 92% respectively; for BF 500-6 of 87and 81%, respectively; and for BF 500-7 of 89 and 81%, respectively.

The BASF Corporation laboratory demonstrated relative standard deviations (RSDs) @ 10.0 ppb (LOQ) and 100 ppb (10 x LOQ) for BAS 500 F of 8.2 and 6.5, respectively, for BF 500-3 of 9.8 and 10.4, respectively, for BF 500-4 of 13.9 and 6.6, respectively, for BF 500-5 of 4.5 and 7.6, respectively, for BF 500-6 of 12.9 and 8.9, respectively, and for BF 500-7 of 8.2 and 18.0, respectively. The Battelle laboratory demonstrated relative standard deviations (RSDs) @ 10.0 ppb (LOQ) and 100 ppb (10 x LOQ) for BAS 500 F of 11.0 and 5.1, respectively, for BF 500-3 of 9.4 and 5.2, respectively, for BF 500-4 of 9.6 and 5.2, respectively, for BF 500-5 of 10.2 and 11.8, respectively, for BF 500-6 of 10.9and 4.9, respectively, and for BF 500-7 of 15.3 and 4.2, respectively. The respective laboratories met the targeted recovery range of 70% to 120% and a RSD of ≤20.

The registrant estimates that it takes approximately twelve (12) working hours or 1.5 calendar days to extract and analyze one set of eight (8) samples with appropriate blanks and standards, provided that no special problems arise, such as matrix interference.

This environmental chemistry method review (ECMR) verifies that the registrant has provided satisfactory documentation of the validation of this method; and, therefore, the method does not need any further evaluation.

PART II

Discussion of Problems Encountered During Method Review

There were no major problems with the method and the registrant should be commended for the revision of the method to resolve low recoveries (< 70%) for BF 500-5. This was demonstrated in Method D9812/1. As for minor problems, there was no effort to evaluate the recovery of the compounds at the MDL. The registrant did analyze calibration standards for the analytes, the lowest calibration standards at the MDL for the analyte, and demonstrated that good detect ability (S/N > 3) was possible. The mass spectroscopist suggested that the MS scan and tune could have been performed better. There are textual mistakes in the tables on pages 23, 24, 25, 27, 28, 29, 30, 31, 33, and 34; on those pages the injection volume of the Mg Injected Column [Footnote 2] indicate 10 µl as the injection volume. On page 22, the recovery calculation equation is based on an injection volume of 20 µl. On pages 26 and 33, the table, column, and footnote use 20 µl in the calculation. Discussions with the study director revealed textual error which will be corrected and resubmitted to EFED as a revised final copy of the method.

PART III

Analytical Results

Method: BASF Corporation Agricultural Products Group, Registration Document 1999/5087, **"The Determination of BAS 500 F and Its Metabolites: BF 500-3, BF 500-4, BF 500-5, BF 500-6, and BF 500-7 in Soil Using LC-MS "**

TABLE 1. Recovery of BAS 500 F (Pyraclostrobin) in Soil

BASF Corporation Agricultural Products Group
BAS 500 F - **LOQ (0.010 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0103	103
Recovery #2	0.0109	109
Recovery #3	0.0092	92
Recovery #4	0.0089	89
Recovery #5	0.0104	104
Recovery #6	0.0092	92
Average		98
Standard Deviation		8
RSD		8.2

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Recovery #1	0.0092	93
Recovery #2	0.0103	86
Recovery #3	0.0109	93
Recovery #4	0.0092	114
Recovery #5	0.0089	98
Average		97
Standard Deviation		10.6
RSD		11.0

BASF Corporation Agricultural Products Group
BAS 500 F - **10 x LOQ (0.100 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0970	97
Recovery #2	0.1021	102
Recovery #3	0.0928	93
Recovery #4	0.0874	87
Recovery #5	0.0942	94
Recovery #6	0.0869	87
Average		93
Standard Deviation		6
RSD		6.5

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.000	n/a
Control #2	0.000	n/a
Recovery #1	0.091	91
Recovery #2	0.084	84
Recovery #3	0.084	84
Recovery #4	0.079	79
Recovery #5	0.085	85
Average		85
Standard Deviation		4.3
RSD		5.1

TABLE 2. Recovery of BF 500-3 (Metabolite) in Soil

BASF Corporation Agricultural Products Group
BF 500-3 - **LOQ (0.010 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0103	103
Recovery #2	0.0115	115
Recovery #3	0.0092	92
Recovery #4	0.0090	90
Recovery #5	0.0112	112
Recovery #6	0.0101	101
Average		102
Standard Deviation		10
RSD		9.8

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Recovery #1	0.0088	88
Recovery #2	0.0089	89
Recovery #3	0.0083	83
Recovery #4	0.0105	105
Recovery #5	0.0096	96
Average		92
Standard Deviation		8.7
RSD		9.4

BASF Corporation Agricultural Products Group
BF500-3 - **10 x LOQ (0.100 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.1052	105
Recovery #2	0.1100	110
Recovery #3	0.0868	87
Recovery #4	0.0843	84
Recovery #5	0.0944	94
Recovery #6	0.0928	93
Average		96
Standard Deviation		10
RSD		10.4

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.000	n/a
Control #2	0.000	n/a
Recovery #1	0.092	92
Recovery #2	0.081	81
Recovery #3	0.083	83
Recovery #4	0.085	85
Recovery #5	0.082	82
Average		85
Standard Deviation		4.4
RSD		5.2

TABLE 3. Recovery of BF 500-4 (Metabolite) in Soil

BASF Corporation Agricultural Products Group
BF 500-4 - **LOQ (0.010 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0093	93
Recovery #2	0.0094	94
Recovery #3	0.0064	64
Recovery #4	0.0084	84
Recovery #5	0.0091	91
Recovery #6	0.0092	92
Average		86
Standard Deviation		12
RSD		13.9

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Recovery #1	0.0069	69
Recovery #2	0.0068	68
Recovery #3	0.0080	80
Recovery #4	0.0065	65
Recovery #5	0.0080	80
Average		72
Standard Deviation		6.9
RSD		9.6

BASF Corporation Agricultural Products Group
BF500-4 - **10 x LOQ (0.100 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.000	n/a
Control #2	0.000	n/a
Control #3	0.000	n/a
Recovery #1	0.0992	99
Recovery #2	0.0975	97
Recovery #3	0.0836	84
Recovery #4	0.0840	84
Recovery #5	0.0933	93
Recovery #6	0.0878	88
Average		91
Standard Deviation		6
RSD		6.6

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.000	n/a
Control #2	0.000	n/a
Recovery #1	0.074	74
Recovery #2	0.080	80
Recovery #3	0.072	72
Recovery #4	0.078	78
Recovery #5	0.081	81
Average		77
Standard Deviation		4.0
RSD		5.2

TABLE 4. Recovery of BF 500-5 (Metabolite) in Soil

BASF Corporation Agricultural Products Group
BF 500-5 - **LOQ (0.010 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0090	90
Recovery #2	0.0080	80
Recovery #3	0.0089	89
Recovery #4	0.0090	90
Recovery #5	0.0086	86
Recovery #6	0.0091	91
Average		88
Standard Deviation		4
RSD		4.5

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Recovery #1	0.0075	75
Recovery #2	0.0080	80
Recovery #3	*	*
Recovery #4	0.0090	90
Recovery #5	0.0094	94
Average		85
Standard Deviation		8.6
RSD		10.2

* Sample Compromised

BASF Corporation Agricultural Products Group
BF500-5 - **10 x LOQ (0.100 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0851	85
Recovery #2	0.0869	87
Recovery #3	0.0770	77
Recovery #4	0.0721	72
Recovery #5	0.0817	82
Recovery #6	0.0734	73
Average		79
Standard Deviation		6
RSD		7.6

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.000	n/a
Control #2	0.000	n/a
Recovery #1	0.0093	93
Recovery #2	0.0105	105
Recovery #3	0.0098	98
Recovery #4	0.0082	82
Recovery #5	0.0080	80
Average		92
Standard Deviation		10.9
RSD		11.8

TABLE 5. Recovery of BF 500-6 (Metabolite) in Soil

BASF Corporation Agricultural Products Group
BF 500-6 - **LOQ (0.010 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0113	113
Recovery #2	0.0111	111
Recovery #3	0.0111	111
Recovery #4	0.0094	94
Recovery #5	0.0082	82
Recovery #6	0.0092	92
Average		101
Standard Deviation		13
RSD		12.9

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Recovery #1	0.0083	83
Recovery #2	0.0087	87
Recovery #3	0.0079	79
Recovery #4	0.0103	103
Recovery #5	0.0082	82
Average		87
Standard Deviation		9.4
RSD		10.9

BASF Corporation Agricultural Products Group
BF500-6 - **10 x LOQ (0.100 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.1018	102
Recovery #2	0.0970	97
Recovery #3	0.0883	88
Recovery #4	0.0803	80
Recovery #5	0.0895	89
Recovery #6	0.0841	84
Average		90
Standard Deviation		8
RSD		8.9

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.000	n/a
Control #2	0.000	n/a
Recovery #1	0.084	84
Recovery #2	0.080	80
Recovery #3	0.075	75
Recovery #4	0.083	83
Recovery #5	0.084	84
Average		81
Standard Deviation		4.0
RSD		4.9

TABLE 6. Recovery of BF 500-7 (Metabolite) in Soil

BASF Corporation Agricultural Products Group
BF 500-7 - **LOQ (0.010 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.0100	100
Recovery #2	0.0101	101
Recovery #3	0.0107	107
Recovery #4	0.0086	86
Recovery #5	0.0100	100
Recovery #6	0.0089	89
Average		97
Standard Deviation		8
RSD		8.2

ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Recovery #1	0.0079	79
Recovery #2	0.0085	85
Recovery #3	0.0087	87
Recovery #4	0.0113	113
Recovery #5	0.0083	83
Average		89
Standard Deviation		13.7
RSD		15.3

BASF Corporation Agricultural Products Group
BF500-7 - **10 x LOQ (0.100 ppm)**

Sample #	Detected µg/g	Recovery (%)
Control #1	0.0000	n/a
Control #2	0.0000	n/a
Control #3	0.0000	n/a
Recovery #1	0.1156	115
Recovery #2	0.0987	99
Recovery #3	0.0820	82
Recovery #4	0.0698	70
Recovery #5	0.0807	81
Recovery #6	0.0850	85
Average		89
Standard Deviation		16
RSD		18.0

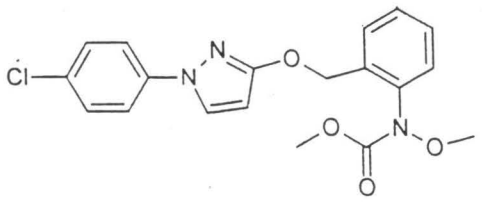
ILV - Battelle Columbus Laboratory

Sample #	Detected µg/g	Recovery (%)
Control #1	0.000	n/a
Control #2	0.000	n/a
Recovery #1	0.081	81
Recovery #2	0.080	80
Recovery #3	0.086	86
Recovery #4	0.078	78
Recovery #5	0.078	78
Average		81
Standard Deviation		3.4
RSD		4.2

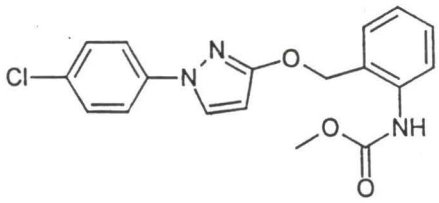
Appendix 1

Chemical Structures of BAS 500 F (Pyraclostrobin) and Its Metabolites: BF 500-3, BF 500-4, BF 500-5, BF 500-6, & BF 500-7

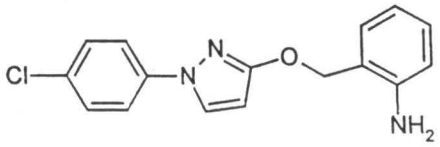
BASF Code Name:	BAS 500 F
BASF Registry Number:	304 428
Chemical Name:	Methy-N-[[[1-(4-chlorophenyl)pyrazol-3-yl]-oxy]-o-tolyl]-N-methoxycarbamate
Molecular Formula:	$C_{19}H_{18}ClN_3O_4$
Molecular Weight:	387.83
Appearance:	White powder
Water Solubility:	1.9 mg/L (at pH 9, 2.3 mg/L)
Lot No.:	00937-128
Purity:	99.8
Stability:	Expected to be stable at least 2 years
Structural Formula:	



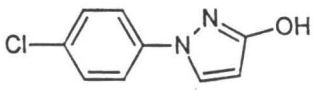
BASF Code Name:	BF 500-3
BASF Registry Number:	340 266
Molecular Formula:	$C_{18}H_{16}ClN_3O_3$
Molecular Weight:	357.8
Lot No.:	00937-272
Purity:	99.0
Stability:	Expected to be stable at least 2 years
Structural Formula:	



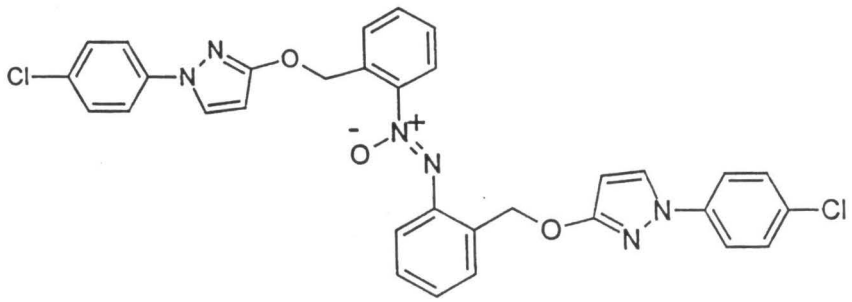
BASF Code Name:	BF 500-4
BASF Registry Number:	358 672
Molecular Formula:	$C_{16}H_{14}ClN_3O$
Molecular Weight:	299.76
Lot No.:	01183-26
Purity:	99.3
Stability:	Expected to be stable at least 2 years
Structural Formula:	

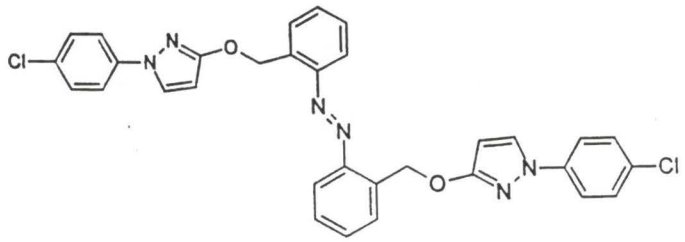


BASF Code Name:	BF 500-5
BASF Registry Number:	298 327
Molecular Formula:	$C_9H_7ClN_2O$
Molecular Weight:	194.6
Lot No.:	00937-275
Purity:	99.9
Stability:	Expected to be stable at least 2 years
Structural Formula:	



BASF Code Name:	BF 500-6
BASF Registry Number:	364 380
Molecular Formula:	$C_{32}H_{24}N_6Cl_2O_3$
Molecular Weight:	611.5
Lot No.:	01185-025
Purity:	99.8
Stability:	Expected to be stable at least 2 years
Structural Formula:	



BASF Code Name:	BF 500-7
BASF Registry Number:	369 315
Molecular Formula:	$C_{32}H_{24}N_6Cl_2O_2$
Molecular Weight:	595.5
Lot No.:	01185-022
Purity:	99.9
Stability:	Expected to be stable at least 2 years
Structural Formula:	

Appendix 2

Standard Evaluation Procedure (SEP) for ECM 0191 S1-6
BAS 500 F (Pyraclostrobin) and Its Metabolites:
BF 500-3, BF 500-4, BF 500-5, BF 500-6, & BF 500-7 in Soil

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

I. Background Information

A. Title of Method The Determination of BAS 500 F and Its Metabolites, BF 500-3, BF 500-4, BF 500-5, BF 500-6, and BF 500-7 in Soil Using LC-MS [D9812/1]

B. ECM No. 0191S1-6

C. MRID No. 451187-07

D. Matrix(es) Soil

E. Analyte(s) detected BAS 500 F (Pyraclostrobin); Metabolites: BF 500-3, BF 500-4, BF 500-5, BF 500-6, BF 500-7

II. Information About the Laboratory

A. Name BASF Corporation Agricultural Products Group

B. Address 26 Davis Drive, P.O. Box 13528

Research Triangle Park, North Carolina 27709

C. Telephone No. (919) 547-2000

D. Name of the Study Director Manasi Saha

E. Name of the Lead Chemists Leonard Colins, Robert Gooding, Manasi Saha

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 Fifty grams of soil was extracted twice with acetonitrile, and the cake was re-extracted once with 0.1 N NaOH.

D. Sample Cleanup The alkaline extract was extracted twice with ethyl acetate, dried, triethylamine added to the acetonitrile extract, reduced, combined, brought to volume and diluted w/ buffer.

E. Sample Derivatization (If Applicable) n/a

F. Sample Analysis

1. Instrumentation HP LC-MSD w/ 1100 Series HPLC System w/ Quarternary Pump
2. Primary Column Inertsil C4, 5µ, 150 x 3.0 mm [MetaChem Technologies, Inc.]
3. Confirmatory Column (If Any) n/a
4. Detector MSD (Ion monitoring - SIM) [low resolution]
5. Other Confirmatory Techniques (If Any) n/a
6. Other Relevant Information

G. Detection and Quantitation Limits

1. Limit of Quantitation (LOQ)

Claimed in Method 0.01 ppm Estimated _____

2. Method Detection Limit (MDL)

Claimed in Method _____ Estimated 0.003 ppm

H. Recovery (Accuracy) Data

BAS 500 F	1.0 ppm: 89%	0.10 ppm: 93%	0.01 ppm: 98%
BF 500-3	1.0 ppm: 89%	0.10 ppm: 96%	0.01 ppm: 102%
BF 500-4	1.0 ppm: 85%	0.10 ppm: 91%	0.01 ppm: 86%
BF 500-5	1.0 ppm: 89%	0.10 ppm: 79%	0.01 ppm: 88%
BF 500-6	1.0 ppm: 84%	0.10 ppm: 90%	0.01 ppm: 101%
BF 500-7	1.0 ppm: 84%	0.10 ppm: 89%	0.01 ppm: 97%

I. Precision Data

BF 500 F	1.0 ppm: 4.5%	0.10 ppm: 6.5%	0.01 ppm: 8.2%
BF 500-3	1.0 ppm: 9.8%	0.10 ppm: 10.4%	0.01 ppm: 9.8%
BF 500-4	1.0 ppm: 13.9%	0.10 ppm: 6.6%	0.01 ppm: 13.9%
BF 500-5	1.0 ppm: 10.1%	0.10 ppm: 7.6%	0.01 ppm: 4.5%
BF 500-6	1.0 ppm: 8.3%	0.10 ppm: 8.9%	0.01 ppm: 12.9%
BF 500-7	1.0 ppm: 7.1%	0.10 ppm: 18.0%	0.01 ppm: 8.2%

REVIEW

IV. **Detailed Information About the Method**		Yes	No	Further Review
A.	Is the method marked CONFIDENTIAL?	___	x	___
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			
a.	The sample preparation procedure	x	___	___
b.	The sample spiking procedure	x	___	___

		Yes	No	Further Review
c.	The extraction procedure	x	___	___
d.	The derivatization procedure	___	___	x
e.	The cleanup procedure	x	___	___
f.	The analysis procedure	x	___	___
2.	Procedures for			
a.	Preparation of standards	x	___	___
b.	Calibration of instrument	x	___	___
3.	List of glassware and chemicals			
a.	Are sources recommended?	x	___	___
b.	Are they commercially available?	x	___	___
4.	Name, model, etc., of the instrument, Column, detector, etc., used			
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 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	___	___

		Yes	No	Further Review
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

A.	Are there representative chromatograms for			
1.	Analyte(s) in each matrix at the MDL, LOQ, and 10 x LOQ?	x	x (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)

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	___	___

		Yes	No	Review Further
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

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

BASF Corporation originally had validated the method D9812, but had encountered some low recoveries for BF 500-5. Documented adjustments were made to the method which was renamed D9812/1. The ECB MS chemist noted that the MSD tune could have been better. The ILV made minor changes in dilution factors and adjustments for differing instrumentation.

Name (*print*) and Signature of Reviewer: Christian Byrne Christian Byrne

Date Initial Review was Assigned: 10/10/01

Date Initial Review was Completed: 10/11/01

Date Final Review was Completed: _____

Signature of Laboratory Chief: _____

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

Charles Kennedy Charles D. Kennedy

Elizabeth Flynt Elizabeth Flynt