

BAS 800 H in Soil/2008-0431/118203/BASF Corporation/241  
PMRA Submission Number: 2008-0431/Company Code: BAZ  
Active Code: SFF/Use Site: 13 and 14  
**ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT**

( Revision 2)

**Data Requirement:** PMRA Data Code: 8.2.2.1  
PMRA Document No.:  
EPA DP Barcode: 350054  
OECD Data Point: IIA.4.4.  
EPA Guideline: ECM Method Review

**Test material:**

Common name: Saflufenacil (BAS 800 H)  
CAS name: 2-chloro-5-[3,6-dihydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)-1(2H)-pyrimidinyl]-4-fluoro-N-[[methyl(1-methylethyl)amino]sulfonyl]-benzamide  
IUPAC name: N'-[2-chloro-4-fluoro-5-[1,2,3,6-tetrahydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)pyrimidin-1-yl]benzoyl]-N-isopropyl-N-methylsulfamide

EPA Primary Evaluator: Elizabeth Flynt Date: 6/30/09  
Elizabeth Flynt, Chemist

EPA Peer Reviewer: Charles Kennedy Date: 6/30/2009  
Charles Kennedy, Chemist

QA Officer: Christian Byrne Date: 6/30/09  
Dr. Christian Byrne, QA Officer

PMRA Global Reviewer: Katherine Keppel-Jones Date: 6/30/09  
Katherine Keppel-Jones, HC-PMRA-CES

APVMA Global Reviewer: D. Murphy Date: 6/30/09  
Dr. Daryl Murphy, DEWHA/APVMA

**ANALYTICAL METHOD:** MRID No. 476999-02 / PMRA No.1731026, Saha, M., March 16, 2009, "Method Validation of BASF Analytical Method D0503 entitled: The Determination of Residues of BAS 800H and Its Metabolites, in Soil Using LC/MS/MS",

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*Metabolites, in Soil Using LC/MS/MS* (MRID No. 471278-32 / PMRA No. 15469-45) was conducted by Adpen Laboratories at Jacksonville, FL. Pages 1-203. The BASF Study No. is 132662.

### **EXECUTIVE SUMMARY**

The method is applicable for the quantitative determination of residues of BAS 800 H and its metabolites in soil. The method was created in accordance with EPA's Good Laboratory Practice Standards, Title 40 Code of Federal Regulations Part 160. Although, the ECB found that this Environmental Chemistry Method (ECM) and its associated independent laboratory validation (ILV) met all criteria for a scientifically valid method, it is considered supplemental because the LOQ validated (0.01 mg/kg) is significantly higher than the lowest phytotoxic endpoint in soil (0.0005 mg/kg).

### **Method Summary**

Soil samples are extracted by vortexing with acetonitrile twice, and then acetonitrile:water twice, followed by centrifugation, and removal of the supernatant for analysis. The residues are determined using LC/MS/MS.

The reported limit of quantification was found to be 0.01 mg/kg for all analytes.

### **METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS**

Although the method was well documented and meets all the requirements for a scientifically valid method there are several inconsistencies/corrections which should be noted.

The LOQ used in this validation was 0.01 mg/kg (ppm), whereas in order to analyze for the lowest phytotoxicity endpoint in soil, the analytical method would need an LOQ of approximately 0.0005 mg/kg (0.0005 ppm). Therefore, although the method is valid, it is considered supplemental.

On page 101 of the amended registrant method, the LOQ is correctly stated as 0.01 ppm, but the example calculation, "Therefore, at the LOQ, if the amount of analyte is 10 pg on column, the LOD is 2 pg on column." uses an arbitrary final value that is different than the value in the method. The reviewers find this unnecessarily confusing.

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### COMPLIANCE

Signed and dated statements that this method was conducted in accordance with the requirements for Good Laboratory Practice Standards, 40 CFR 160 were present in the method. A statement of non-confidentiality on the basis of the method falling within the scope of FIFRA Section 10 (d)(1)(A), (B), or (C) was present.

### A. BACKGROUND INFORMATION

BAS 800 H is a herbicide that will be used for the treatment of cotton, cereal and other crops in the U.S.

Compound	Chemical Structure *See Appendix A for the chemical structure information
Common name	Saflufenacil (BAS 800 H)
Company experimental name	BAS 800 H
IUPAC name	N'-[2-chloro-4-fluoro-5-[1,2,3,6-tetrahydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)pyrimidin-1-yl]benzoyl]-N-isopropyl-N-methylsulfamide
CAS Name	2-chloro-5-[3,6-dihydro-3-methyl-2,6-dioxo-4-(trifluoromethyl)-1(2H)-pyrimidinyl]-4-fluoro-N-[[methyl(1-methylethyl)amino]sulfonyl]-benzamide
CAS #	372137-35-4

Parameter	Value
Melting point/range	Not available
pH	Not available
Density	Not available
Water solubility (25 °C)	Not available
Solvent solubility (mg/ml at 20 °C)	Not available
Vapour pressure	Not available
Dissociation constant (pK <sub>a</sub> )	Not available
Octanol/water partition coefficient	Not available
UV/visible absorption spectrum	Not available

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**B.1. Principle of Method**

A 0.1 gram aliquot of soil sample is placed into a well plate tube and fortified with an appropriate volume of spiking solution. A volume of 0.4 ml of acetonitrile is added and the well tube capped and vortexed twice (once up side down, then right side up) at 2400 rpm for 1 minute each. Then an additional 0.4 ml of acetonitrile:water (40:60 v/v) is added and the well tube capped and vortexed twice (once up side down, then right side up) at 2400 rpm for 1 minute each. The vortexing is followed by centrifugation at 3000 rpm for 5 minutes. The supernatant is removed and transferred into a Matrix Alpha Numeric Tube. Sample and controls are analyzed via LC/MS/MS.

<b>TABLE B.1.1.</b>	<b>Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied</b>
Method ID	ECM0242S1-S7
Analyte(s)	BAS 800 H, M800H01, M800H02, M800H07, M800H08, M800H15, M800H22
Extraction solvent/technique	Soil samples are extracted using acetonitrile and acetonitrile:water
Cleanup strategies	Centrifugation
Instrument/Detector	PE Series 200 Micro Pump System with Series 200 Autosampler / PE Sciex API 3000 Biomolecular Mass Analyzer

**C. RESULTS AND DISCUSSION**

**C.1.Recovery Results Summary**

<b>TABLE C.1.1. Recovery Results from Method Validation of Soil</b>			
Matrix	Spiking Level (conc. units)	Mean% Recoveries	Relative Standard Deviation
*See Appendix B			

**C.1.2. Method Characteristics**

<b>TABLE C.1.2. Method Characteristics</b>	
Analyte	BAS 800 H, M800H01, M800H02, M800H07, M800H08, M800H15, M800H22
Limit of Quantitation	0.01 ppm
Limit of Detection (LOD)	0.002 ppm
Accuracy/Precision at LOQ	*See Appendix B
Reliability of the Method/ [ILV]	An ILV was performed for this method. MRID No. 471278-32
Linearity	Linear curves were prepared for each analyte. The correlation coefficient was > 0.9940 for all compounds.

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<b>TABLE C.1.2. Method Characteristics</b>	
Specificity	The method is very specific due to the use of MS-MS which is the most highly specific method for detection of residues at low concentration.

**C.2. Independent Laboratory Validation (ILV)**

<b>TABLE C.2.1. Recovery Results Obtained by an Independent Laboratory Validation of the Method for the Determination of Saflufenacil and its Metabolites in Water</b>		
Matrix	Spiking Level (conc. units)	Recoveries
See Appendix C		

**D. CONCLUSION**

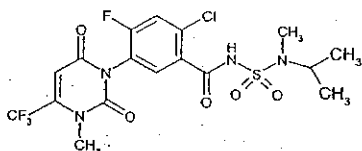
Although this is a well documented method which was confirmed by an independent laboratory validation, ECB finds the method supplemental, because the validated LOQ (0.01 mg/kg) is significantly higher than the lowest phytotoxic endpoint in soil (0.0005 mg/kg).

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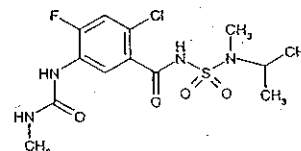
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**Appendix A: Chemical Structures of BAS 800 H and Its Metabolites**

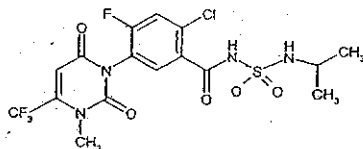
BASF Code Name: BAS 800 H  
 BASF Registry Number: 4054449  
 CAS Number: 372137-35-4  
 Molecular Formula:  $C_{17}H_{17}ClF_4N_4O_5S$   
 Molecular Weight: 500.9  
 Lot No.: L67-140  
 Purity: 99.9%  
 Expiration date: July 01, 2008  
 Structural Formula:



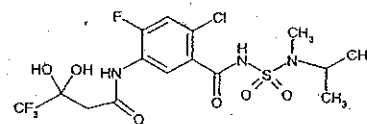
BASF Code Name: M800H07  
 BASF Registry Number: 4775453  
 Molecular Formula:  $C_{15}H_{16}ClF_4N_4O_5S$   
 Molecular Weight: 380.8  
 Lot No.: L67-196  
 Purity: 95.4%  
 Expiration date: March 1, 2009  
 Structural Formula:



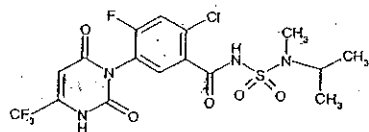
BASF Code Name: M800H01  
 BASF Registry Number: 4118561  
 Molecular Formula:  $C_{16}H_{15}ClF_4N_4O_5S$   
 Molecular Weight: 486.8  
 Lot No.: L74-62  
 Purity: 98.8%  
 Expiration date: February 1, 2008  
 Structural Formula:



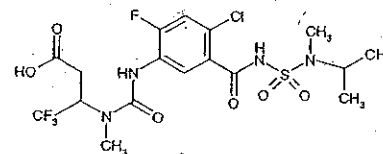
BASF Code Name: M800H015  
 BASF Registry Number: 5264357  
 Molecular Formula:  $C_{15}H_{16}ClF_4N_5O_5S$   
 Molecular Weight: 479.9  
 Lot No.: L74-80  
 Purity: 94.5%  
 Expiration date: June 1, 2008  
 Structural Formula:



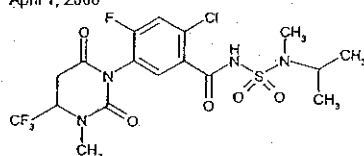
BASF Code Name: M800H02  
 BASF Registry Number: 4118416  
 Molecular Formula:  $C_{16}H_{15}ClF_4N_4O_5S$   
 Molecular Weight: 486.8  
 Lot No.: L67-186  
 Purity: 99.2%  
 Expiration date: March 1, 2009  
 Structural Formula:



BASF Code Name: M800H022  
 BASF Registry Number: 5216337  
 Molecular Formula:  $C_{17}H_{21}ClF_4N_4O_5S$   
 Molecular Weight: 520.9  
 Lot No.: L74-56  
 Purity: 94.1%  
 Expiration date: March 1, 2008  
 Structural Formula:



BASF Code Name: M800H08  
 BASF Registry Number: 4773881  
 Molecular Formula:  $C_{17}H_{19}ClF_4N_4O_5S$   
 Molecular Weight: 502.9  
 Lot No.: L74-66  
 Purity: 97.2%  
 Expiration date: April 1, 2008  
 Structural Formula:



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**Appendix B: Summary of Percent Recoveries, Average and RSDs for BAS 800 H and Its Metabolites**

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fortification Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD <sup>1</sup> (%)
Loamy Sand/0-1" (WA)	BAS 800 H	0.01	101,111,88 113,114	107	7.3	6.8
		0.1	112,97,115 95,109	106	6.7	6.2
		Overall (N=10)		107	7.6	7.1
	M800H01	0.01	84,83,85 92,83	89	5.8	6.4
		0.1	96,92,90 97,95	94	7.9	3.1
		Overall (N=10)		92	5.0	5.4
	M800H02	0.01	80,115,101 112,102	104	9.8	9.4
		0.1	103,76,104 103,102	88	11.3	11.5
		Overall (N=10)		101	10.5	10.3
	M800H07	0.01	92,102,99 85,95	95	5.5	5.8
		0.1	107,95,91 97,102	98	6.2	6.3
		Overall (N=10)		97	5.8	6.0
M800H08	0.01	97,94,102 110,125	106	12.3	11.6	
	0.1	101,112,84 78,105	95	14.4	15.0	
	Overall (N=10)		101	13.6	13.5	
M800H15	0.01	115,80,91 115,105	103	12.5	12.2	
	0.1	118,98,108 99,96	104	9.3	9.0	
	Overall (N=10)		104	10.4	10.1	
M800H22	0.01	113,107,105 101,113	108	5.2	4.8	
	0.1	89,92,101 98,104	97	6.1	6.3	
	Overall (N=10)		102	7.8	7.5	

<sup>1</sup>Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fortification Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD <sup>1</sup> (%)
Sandy Loam/0-2" (German 2.2)	BAS 800 H	0.01	79,83,89 91,95	88	5.8	6.6
		0.1	99,102,110 102,111	105	5.4	5.1
		Overall (N=10)		95	10.0	10.4
	M800H01	0.01	79,84,93 83,81	87	5.8	6.7
		0.1	89,93,95 93,103	95	5.3	5.8
		Overall (N=10)		91	6.7	7.4
	M800H02	0.01	92,85,100 83,80	90	6.5	7.2
		0.1	96,103,98 91,94	97	4.5	4.7
		Overall (N=10)		93	6.3	6.7
	M800H07	0.01	75,83,79 95,82	97	5.3	5.5
		0.1	102,96,100 100,93	104	4.5	4.4
		Overall (N=10)		100	6.1	6.1
M800H08	0.01	89,75,88 106,81	75	14.9	20.1	
	0.1	102,92,105 107,97	101	5.8	5.8	
	Overall (N=10)		88	17.5	20.0	
M800H15	0.01	91,76,83 87,83	80	8.8	11.1	
	0.1	84,83,84 97,83	89	5.6	6.2	
	Overall (N=10)		85	6.5	10.1	
M800H22	0.01	85,114,118 97,97	100	11.4	11.4	
	0.1	93,96,87 94,95	93	3.5	3.7	
	Overall (N=10)		97	8.5	9.2	

<sup>1</sup>Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fortification Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD <sup>1</sup> (%)
Loam/0-1" (CA)	BAS 800 H	0.01	91,102,112,109,95, 110,119,108,105,103	104	7.0	6.7
		0.1	93,104,104,103,107, 98,97,109,100,117	103	6.8	6.5
		Overall (N=20)		103	6.7	6.5
	M800H01	0.01	112,91,98,85,90, 96,94,88,83	95	7.6	8.1
		0.1	100,106,100,100,103, 91,93,100,91,88	98	5.1	5.2
		Overall (N=20)		96	6.6	6.8
	M800H02	0.01	109,95,109,105,97, 100,107,108,95,109	102	5.7	5.5
		0.1	97,99,109,104,112, 88,90,97,98,93	98	7.6	6.0
		Overall (N=20)		100	7.0	7.0
	M800H07	0.01	94,92,106,95,97, 90,109,101,104,100	98	5.4	5.5
		0.1	107,102,97,101,107, 92,103,95,105,103	102	5.5	5.4
		Overall (N=20)		100	5.6	5.6
M800H08	0.01	99,86,119,73,99, 102,106,112,88,103	98	12.1	12.4	
	0.1	106,88,119,77,104, 96,75,99,119,111	99	15.7	15.8	
	Overall (N=20)		99	13.6	13.6	
M800H15	0.01	96,83,83,98,106, 112,93,79,74,72	90	13.5	15.1	
	0.1	106,109,105,91,85, 78,105,111,111,83	99	11.7	11.7	
	Overall (N=20)		95	13.3	14.1	
M800H22	0.01	92,97,85,84,87,91, 108,93,108,100	95	8.7	8.1	
	0.1	85,83,99,114,93,93, 100,101,98,99	95	9.1	9.6	
	Overall (N=20)		95	8.7	9.1	

<sup>1</sup>Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

Summary of Percent Recoveries, Average and Standard Deviation for Each Analyte in Different Types of Soil (Method D0503) Obtained from Primary Quantitation Ion

Soil Type/ Soil Depth/ Location	Analytes	Fortification Level (ppm)	Recovery (%)	Average Recovery (%)	Standard Deviation	RSD <sup>1</sup> (%)
Clay Loam/24-30" (CA)	BAS 800 H	0.01	101,112,100, 109,100	103	5.3	5.2
		0.1	101,105,96, 102,86	101	3.7	3.7
		Overall (N=20)		102	4.4	4.3
	M800H01	0.01	104,96,85, 110,104	100	9.1	9.0
		0.1	89,91,102, 98,112	98	9.4	9.6
		Overall (N=20)		99	8.8	8.8
	M800H02	0.01	92,89,103, 109,82	96	10.6	11.1
		0.1	84,95,96, 89,91	91	5.9	5.4
		Overall (N=20)		93	8.2	8.7
	M800H07	0.01	102,105,102, 93,91	99	6.0	6.1
		0.1	94,98,95, 93,100	96	3.9	3.1
		Overall (N=20)		97	4.7	4.6
M800H08	0.01	123,106,80, 88,97	101	14.5	14.4	
	0.1	111,100,83, 77,103	95	14.3	15.1	
	Overall (N=20)		98	14.0	14.3	
M800H15	0.01	94,88,79, 96,95	89	6.9	7.8	
	0.1	82,88,96, 87,109	92	10.5	11.3	
	Overall (N=20)		91	8.5	9.5	
M800H22	0.01	99,99,95, 82,86	92	6.4	9.2	
	0.1	86,42,80, 92,85	86	3.0	3.4	
	Overall (N=20)		89	6.5	7.4	

<sup>1</sup>Relative Standard Deviation (RSD) = (Standard Deviation/Average Recovery) x 100

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### Appendix C – Summary of ILV Percent Recoveries and RSDs for BAS 800H and Its Metabolites

Analytes	Fortification Level (ppm)	Average Recovery (%)	RSD
BAS 800 H	0.01	117	7.8
	0.1	102	6.1
M800H01	0.01	108	14.1
	0.1	102	8.3
M800H02	0.01	107	12.7
	0.1	93.0	13.2
M800H07	0.01	106	10.4
	0.1	99.5	9.6
M800H08	0.01	111	12.4
	0.1	98.2	9.0
M800H15	0.01	91.6	17.6
	0.1	83.5	10.0
M800H22	0.01	92.8	22.1
	0.1	103	11.9