

Saflufenacil in Water/118203/ BASF Corporation/7969
PMRA Submission Number: 2008-0431/Company Code: BAZ/
Active Code: SFF/Use Site: 13 & 14
ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

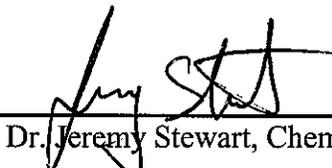
(Revision 1)

Data Requirement: PMRA Data Code: 8.2.2.3
EPA DP Barcode: 356670
OECD Data Point: II A4.5
EPA Guideline: ECM Method Review

Test material:

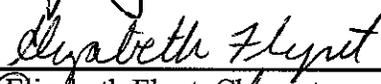
Common name: Saflufenacil
CAS name: 2-chloro-5-[3, 6-dihydro-3-methyl-2, 6-dioxo-4-(trifluoromethyl)-1(2H)-pyrimidinyl]-4-fluoro-N-[[methyl (1-methylethyl) amino] sulfonyl] benzamide
IUPAC name: N'-[2-chloro-4-fluoro-5-[1, 2, 3, 6-tetrahydro-3-methyl-2, 6-dioxo-4-(trifluoromethyl) pyrimidin-1-yl] benzoyl]-N-isopropyl-N-methylsulfamide

EPA Primary Evaluator:


Dr. Jeremy Stewart, Chemist

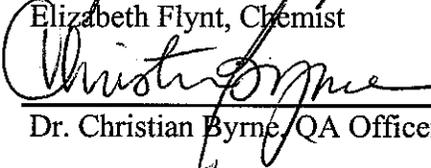
Date: 6/30/09

EPA Peer Reviewer:


Elizabeth Flynt, Chemist

Date: 6/30/09

EPA QA Officer:


Dr. Christian Byrne, QA Officer

Date: 6/30/09

PMRA Global Reviewer:


Katherine Keppel-Jones, HC-PMRA-CES

Date: 6/30/09

APVMA Global Reviewer:


Dr. Daryl Murphy, DEWHA/APVMA

Date: 6/30/09

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ANALYTICAL METHOD: MRID #476999-03/PMRA #1731027, Saha, M., March 13, 2009, "Method Validation of BASF Analytical Method D0502 entitled: *The Determination of Residues of BAS 800 H and Its Metabolites In Water Using LC-MS/MS*", Amended Final Report #2 of unpublished method was developed by BASF Corporation, Research Triangle Park, NC. Pages 1-197. The study number is 132692. ILV MRID #47523803/PMRA #1634457, Smith, M., Perez, R., August 5, 2008, "*Independent Laboratory Validation of BASF Analytical Method D0502*", ADPEN Laboratory, Jacksonville, FL, Pages 1-223. The BASF study number is 351551; the ADPEN study number is ADPEN-2K8-903-351551.

EXECUTIVE SUMMARY

The method is applicable for the quantitative determination of residues of saflufenacil and its metabolites in de-ionized, well, and pond waters. The method was created by BASF Corporation in accordance with EPA's Good Laboratory Practice Standards, Title 40 Code of Federal Regulations Part 160. After a thorough review, ECB together with PMRA and APVMA found that this method met the criteria for a scientifically valid method and is acceptable for use.

Method Summary

A 1mL water (de-ionized, well, or pond) sample aliquot is diluted with 0.1 mL methanol and vortexed. It then undergoes ESI-LC/MS/MS analysis. The reported limit of quantification (LOQ) was found to be 1 µg/kg for all analytes.

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

The original method and final report contain a number of minor deficiencies which were addressed and corrected in the Amended Final Report #2. The amended version meets the essential requirements for a scientifically valid method and is judged acceptable.

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. 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.

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A. BACKGROUND INFORMATION

Saflufenacil is a new herbicide that will be used in tree orchards, vineyards, and numerous field and row crops in the US, Australia, and Canada.

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

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound	
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 _a)	Not available
Octanol/water partition coefficient	Not available
UV/visible absorption spectrum	Not available

MATERIALS AND METHODS

B.1. Principle of Method

A 1mL water (de-ionized, well, or pond) sample aliquot is diluted with 0.1 mL methanol and vortexed. It then undergoes ESI-LC/MS/MS analysis.

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TABLE B.1.1.	Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied
Method ID	ECM0244W1-W7
Analyte(s)	Saflufenacil (BAS800H), M800H01, M800H02, M800H07, M800H08, M800H15, M800H22
Extraction solvent/technique	Water (de-ionized, well, or pond) sample aliquot is diluted with 0.1 mL methanol and vortexed.
Cleanup strategies	n/a
Instrument/Detector	LC/MS/MS – Perkin Elmer HPLC + PE Sciex API 3000 Mass Analyzer

C. RESULTS AND DISCUSSION

C.1.Recovery Results Summary

TABLE C.1.1. Recovery Results from Method Validation of Water Samples			
Matrix	Spiking Level (conc. units)	Mean% Recoveries	Relative Standard Deviation
*See Appendix A			

C.1.2. Method Characteristics

TABLE C.1.2. Method Characteristics	
Analyte	Saflufenacil, M800H01, M800H02, M800H07, M800H08, M800H15, M800H22
Limit of Quantitation	1 µg/kg
Limit of Detection (LOD)	Not Determined , estimated at 20% LOQ
Accuracy/Precision at LOQ	*See Appendix A
Reliability of the Method/ [ILV]	An ILV was performed for this method. MRID No. 47523803/ PMRA No. 1634457
Linearity	Linear curves were prepared for each analyte. The correlation of coefficient for all analytes ranged from r = 0.994 to 1.000
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 conc. Two transitional ions were also monitored during analysis for additional peak confirmation.

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C.2. Independent Laboratory Validation (ILV)

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

Matrix	Spiking Level (conc. units)	Recoveries
* See Appendix C		

D. CONCLUSION

This is a well documented method that was confirmed by an independent laboratory validation. The amended version of the method and final report are acceptable.

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Appendix A

Summary of Recoveries of BASF Method D0502 in Water

Analytes	Fortification Levels (ppm)	De-ionized Water		Well Water		Pond Water	
		Mean Recovery (%) \pm S.D	Relative Standard Deviation (%)	Mean Recovery (%) \pm S.D	Relative Standard Deviation (%)	Mean Recovery (%) \pm S.D	Relative Standard Deviation (%)
BAS 800 H	0.001	106 \pm 8.8	8.3	101 \pm 5.6	5.6	104 \pm 4.0	3.9
	0.01	107 \pm 24.2	22.5	116 \pm 7.6	6.5	102 \pm 4.3	4.2
M800H01	0.001	103 \pm 6.7	6.5	101 \pm 6.1	6.0	110 \pm 6.7	6.1
	0.01	110 \pm 23.6	21.5	116 \pm 10.4	8.9	106 \pm 3.2	3.1
M800H02	0.001	104 \pm 6.0	5.8	101 \pm 7.5	7.4	105 \pm 8.5	8.0
	0.01	113 \pm 10.3	9.1	120 \pm 6.0	5.0	100 \pm 4.1	4.1
M800H07	0.001	100 \pm 4.1	4.1	100 \pm 1.8	1.8	117 \pm 6.3	5.4
	0.01	115 \pm 6.7	5.9	112 \pm 6.1	5.5	98 \pm 2.7	2.8
M800H08	0.001	105 \pm 6.0	5.7	100 \pm 3.6	3.6	102 \pm 6.2	6.1
	0.01	111 \pm 22.0	19.8	115 \pm 3.2	2.7	97 \pm 3.2	3.3
M800H15	0.001	95 \pm 5.4	5.7	96 \pm 8.7	9.1	100 \pm 16.9	16.9
	0.01	117 \pm 11.5	9.8	109 \pm 8.7	8.0	95 \pm 6.2	6.5
M800H22	0.001	99 \pm 2.8	2.9	95 \pm 4.8	5.0	102 \pm 3.8	3.7
	0.01	123 \pm 16.9	13.7	118 \pm 6.0	5.1	100 \pm 8.0	8.0

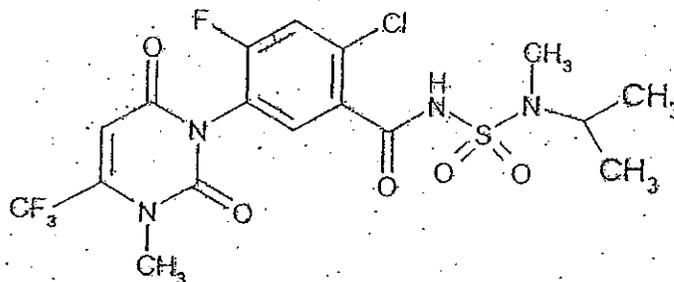
Number of fortifications (n) is five for each fortification level, except for deionized water, for which seven recovery results were obtained.

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Appendix B

Saflufenacil:



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Appendix C

Summary of ILV Recoveries of BASF Method D0502 in Well Water

Analyte	Average Recovery for water fortified at 0.001 ppm	Average Recovery for water fortified at 0.01 ppm
BAS 800 H	93.4 ± 3.2% (n=5)	110 ± 6.9% (n=5)
M800H01	83.1 ± 19.0% (n=5)	102 ± 18.2% (n=5)
M800H02	107 ± 8.9% (n=5)	112 ± 13.0% (n=5)
M800H07	109 ± 10.5% (n=5)	108 ± 8.8% (n=5)
M800H08	103 ± 7.9% (n=5)	113 ± 3.1% (n=5)
M800H15	112 ± 15.9% (n=5)	102 ± 17.4% (n=5)
M800H22	103 ± 7.7% (n=5)	112 ± 16.7% (n=5)
Secondary Ions		
BAS 800 H	117 ± 11.6% (n=5)	115 ± 19.5% (n=5)
M800H07	104 ± 18.0% (n=5)	113 ± 10.4% (n=5)
M800H08	88 ± 15.8% (n=5)	114 ± 27.1% (n=5)
M800H15	107 ± 19.9% (n=5)	117 ± 11.3% (n=5)
M800H22	106 ± 6.2% (n=5)	104 ± 6.6% (n=5)

[% Recovery ± RSD (number of replicates)]