Fenpyrazamine; EPA PC Code 090109
Valent U.S.A. Corporation; EPA Company Code
ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

Test Material:	Fenpyrazamine
MRID	48400027
Title:	Bonarenko, S. 2011. V-1-135: independent laboratory validation (ILV) of the analytical method for water.
MRID	48400034
Title:	Dix, M.E. 2006. S-2188 - validation of the analytical method for the determination of a test substance in aqueous solutions following OPPTS 860.1340, SANCO/3029/99 rev.4 and SANCO/825/00 rev.7.
EPA PC Code:	090109
OCSPP Guideline:	835.6200

For Cambridge Environmental

Primary Reviewer: Lynne Binari **Signature:** Lynne Binari

Date: 2/14/12

Secondary Reviewer: Kathleen Ferguson **Signature:** Kathleen P. Ferguson

Date: 2/14/12

QC/QA Manager: Joan Gaidos **Signature:**

Date: 2/14/12

Data Requirement: EPA Guideline: 835.6200
OECD Data Point: IIA 4.5

Test material:
Common name: Fenpyrazamine
Chemical name: 1H-Pyrazole-1-carbothioic acid, 5-amino-2,3-dihydro-2-(1-methylethyl)-4-(2-methylphenyl)-3-oxo-, S-2-propenyl ester (p. 10 of MRID 48400027)
IUPAC: Allyl 5-amino-2-isopropyl-4-(2-methylphenyl)-3-oxo-2,3-dihydro-1H-1-pyrazolecarbothioate

Primary Reviewer: **Gabe Rothman, Environmental Scientist**
US EPA Office of Pesticide Programs, Environmental Fate and Effects Division

Date: 12/7/11

ANALYTICAL METHOD: EPA MRID No. 48400034. Dix, M.E. 2006. S-2188 - validation of the analytical method for the determination of a test substance in aqueous solutions following OPPTS 860.1340, SANCO/3029/99 rev.4 and SANCO/825/00 rev.7. Report prepared by Springborn Smithers Laboratories, Wareham, Massachusetts, sponsored by Sumitomo Chemical Company, Ltd., Tokyo, Japan, and submitted by Valent U.S.A. Corporation, Walnut Creek, California; 39 pages. Final report issued March 2, 2006.

INDEPENDENT LABORATORY VALIDATION: EPA MRID No. 48400027. Bonarenko, S. 2011. V-10135: independent laboratory validation (ILV) of the analytical method for water. Report prepared by Valent U.S.A. Corporation, Dublin, California, sponsored and submitted by Valent U.S.A. Corporation, Walnut Creek, California; 116 pages. Final report issued January 13, 2011.

EXECUTIVE SUMMARY

This method is designed for the quantitative determination of residues of fenpyrazamine in freshwater using an external standardization method. The method was validated by Springborn Smithers Laboratories, for Sumitomo Chemical Company, Ltd., in accordance with USEPA GLP requirements (pp. 2, 8, Appendix 1, p. 33 of MRID

48400034). An independent laboratory validation (ILV), performed by Valent U.S.A. Corporation, a subsidiary of Sumitomo Chemical, was submitted with the method. *The Agency finds that this method meets the criteria for a scientifically valid method and is* ***acceptable*** *for (applicable residues).*

Method Summary: Fenpyrazamine is extracted from water using solid phase extraction (SEP) and quantified by LC/MS/MS (p. 11 of MRID 48400034). The ECM defined limits of quantitation (LOQ) and detection (LOD) of 1.00 µg/L and 0.10 µg/L, respectively, for fenpyrazamine, which were supported by the ILV (pp. 12-14 of MRID 48400034; pp. 20-21 or MRID 48400027).

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

The ECM did not conduct fortifications at 10 x LOQ. The ILV found elution of fenpyrazamine with acetonitrile during SEP to be incomplete, consequently, the SEP elution step of the method was modified to methanol followed by acetonitrile (p. 19 of MRID 48400027).

COMPLIANCE

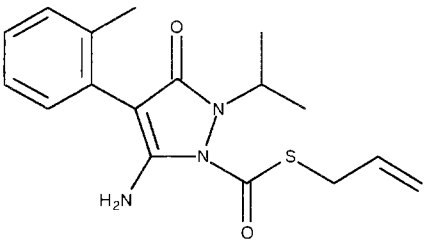
This method was conducted in compliance with USEPA GLP Standards 40 CFR, Part 160 (p. 2 of MRID 48400034). Signed and dated statements of No Data Confidentiality, GLP and Quality Assurance were provided (pp. 1i-3 of MRID 48400034).

A. BACKGROUND INFORMATION

Fenpyrazamine (S-2188) is a new fungicide being developed by Sumitomo Chemical for the control of gray mold and *Sclerotinia* rot in grape, oilseed rape and a variety of vegetables (p. 20 of MRID 48399929).

TABLE A.1. Test Compound Nomenclature

Parameter	Value
Common name	Fenpyrazamine
Company experimental name	S-2188, V-10135
IUPAC name	Allyl 5-amino-2-isopropyl-4-(2-methylphenyl)-3-oxo-2,3-dihydro-1H-1-pyrazolecarbothioate
CAS Name	1H-Pyrazole-1-carbothioic acid, 5-amino-2,3-dihydro-2-(1-methylethyl)-4-(2-methylphenyl)-3-oxo-, S-2-propenyl ester
CAS #	473798-59-3

TABLE A.1. Test Compound Nomenclature	
Parameter	**Value**
Structure	

Information obtained from p. 10 of MRID 48400027 (IUPAC name obtained from MRID 48399929, p. 21).

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound	
Parameter	**Value**
Physical state	Very pale yellow solid at 25°C
Odor	Characteristic of garlic
Melting point/range (°C)	116.4
pH	Not reported.
Relative Density at 20°C (g/mL)	1.250
Water solubility at 20°C (mg/L)	20.4 (OECD #105 - Flask Method)
Solvent solubility at 20 °C (g/L)	Hexane 0.811 Methylene dichloride >250 Acetone >250 Methanol >250
Vapor pressure at 20°C (Pa)	2.89×10^{-8}
Dissociation constant (pK_a)	No dissociation activity at pH 1-13
Octanol/water partition coefficient at 25°C (Log P_{ow})	3.52
UV/visible absorption spectrum at pH 7.8-8.1 (wavelength max, ε)	243 nm (16,700 $M^{-1}cm^{-1}$) 274 nm (13.900 $M^{-1}cm^{-1}$)

Information obtained from p. 24 of MRID 48399928.

B. MATERIALS AND METHODS

B.1. Principle of Method

Fenpyrazamine is extracted from water using SEP (Waters Oasis HLB, 6.0 mL, 500 mg column) and elution with methanol followed by acetonitrile (p. 11 of MRID 48400034; p. 9, 15-16, 19 of MRID 48400027). The analyte is quantified by LC/MS/MS using a YMC ODS-AQ S-3 column, positive electrospray ionization (ESI^+) and multiple reaction monitoring (MRM). Two transition ions of the analyte, 332.1 m/z > 272.0 m/z and 332.1 m/z > 230.3 m/z, are monitored for quantitation and confirmation, respectively.

TABLE B.1. Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied	
Parameter	**Value**
Method ID	S-2188 - validation of the analytical method for the determination of a test substance in aqueous solutions following OPPTS 860.1340, SANCO/3029/99 rev.4 and SANCO/825/00 rev.7.
Analyte(s)	Fenpyrazamine
Extraction solvent/technique	Water (100 mL) is loaded onto a SEP column (Waters Oasis HBL, 6 mL, 500 mg) preconditioned with methanol and water (p. 11 of MRID 48400034). The loaded column is dried (20 psi vacuum, 10 minutes), then the analyte is eluted with methanol (5 mL) followed by acetonitrile (5 mL; pp. 16, 19 of MRID 48400027). The eluate is brought to volume (10.0 mL) with water:acetic acid (1,000:0.5, v:v). If needed, additional dilutions are made with acetonitrile:water:acetic acid (1,000:1,000:0.5, v:v:v).
Cleanup strategies	Not required.
Instrument/Detector	ECM - Waters 2695 HPLC system, with a YMC ODS-AQ S-3 column (2.0 x 50 mm, 3-µm, 120 Å), and MicroMass Quattro Micro MS system equipped with MicroMass Electro Spray in positive ionization mode (ESI^+; pp. 9, 12 of MRID 48400034). ILV - Agilent 1200 HPLC system, with same column as above, and Applied Bioscience API 4000 triple quadrupole MS system with TurboSpray (ESI^+) source and MRM (pp. 13, 16 of MRID 48400027).

Information obtained from pp. 13, 16, 19 of MRID 48400027; and pp. 9, 11-12 of MRID 48400034.

C. RESULTS AND DISCUSSION

C.1. Recovery Results Summary

TABLE C.1. Recovery Results from Method Validation for the Determination of Fenpyrazamine in Well Water (n = 5)			
Analyte	**Spiking Level (µg a.i./L)**	**Mean Recoveries Obtained (%)**	**Relative Standard Deviation**
Fenpyrazamine	1.00 (LOQ)	102	16.2
	500	93.8	3.53
	20,000	81.2	18.8

Results from Table 1, p. 18 of MRID 48400034.

C.1.1. Method Characteristics

TABLE C.2. Method Characteristics	
Parameter	**Value**
Analyte(s)	Fenpyrazamine

TABLE C.2. Method Characteristics	
Limit of Quantitation (LOQ)	1.00 µg a.i./L (p. 12 of MRID 48400034).
Limit of Detection (LOD)	0.10 µg a.i./L (p. 14 of MRID 48400034)
Accuracy/Precision at LOQ	Acceptance criteria (EFED-ECM 2, Version 1, December 2010, p. 5) were met at the LOQ with matrix spike recoveries ranging between 70% to 120% and relative standard deviations of ≤20% (Table 1, p. 18 of MRID 48400034).
Reliability of the Method/[ILV]	Results were repeatable upon modification of SEP elution step from acetonitrile (5 mL) to methanol (5 mL) followed by acetonitrile (5 mL; p. 11 of MRID 48400034; pp, 16, 19 of MRID 48400027).
Linearity	Linear regression - ECM r^2 = 0.99905 (Figure 7, p. 25 of MRID 48400034), ILV r^2 = 0.9983-0.9984 (Appendix 3, Figure 19, p. 95, Figure 36, p. 112 of MRID 48400027) .
Specificity	Comparison of chromatograms produced for standards and control and fortified samples demonstrates that the method, based on LC/MS/MS, is highly specific for the analysis of fenpyrazamine (Figures 1-6, pp. 19-24 of MRID 48400034; Appendix 3, Figures 1-18, pp. 77-94, Figures 20-35, pp. 96-111 of MRID 48400027). Any detections in reagent blank and matrix blank controls were ≤22% of the LOQ (DER Attachment 2). Reported recoveries for fortified samples were not corrected for mean residues detected in the control samples.

Information obtained from pp. 16, 19, Appendix 3, Figure 1-36, pp. 77-112 of MRID 48400027; and pp. 12, 14, Table 1, p. 18, Figures 1-7, pp. 19-25 of MRID 48400034.

C.2. Independent Laboratory Validation (ILV)

TABLE C.3. Recovery Results of the Method Obtained by an Independent Laboratory Validation for the Determination of Fenpyrazamine in Tap water

Analyte	Spiking Level (µg a.i./L)	Mean Recoveries Obtained (%)	Relative Standard Deviation
Fenpyrazamine	1.00 (LOQ)	103	2.62
	10.0	94.6	2.34

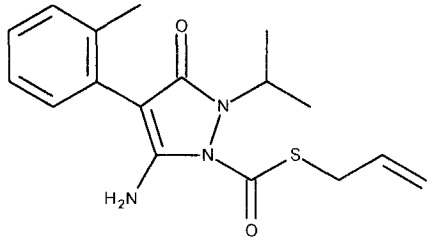
Results from Table 3, p. 27 of MRID 48400027.

D. CONCLUSION

This method is designed for the quantitative determination of residues of fenpyrazamine in freshwater (well, tap). *The Agency finds that this method meets the criteria for a scientifically valid method and is* ***acceptable*** *for (applicable residues).* The ECM did not conduct fortifications at 10 x LOQ and did not use a confirmatory analysis method. For the ILV, fenpyrazamine peak area results were provided for the confirmation ion, but not calculated residue levels.

An additional ECM (MRID 48399918), but no supporting ILV, for the determination of residues of fenpyrazamine in drinking (tap) and surface (pond) water was submitted with this data package. LOQs and LODs were 0.10 µg/L and 1.0 µg/L, respectively, for fenpyrazamine in drinking water, and 1.0 µg/L and 10 µg/L, respectively, in surface water.

DER ATTACHMENT 1. Fenpyrazamine and Its Environmental Transformation Products. [A]

Code Name/ Synonym	Chemical Name	Chemical Structure	Study Type	MRID	Maximum %AR (day)	Final %AR (study length)
PARENT						
Fenpyrazamine S-2188	**IUPAC:** S-allyl 5-amino-2,3-dihydro-2-isopropyl-3-oxo-4-(o-tolyl)pyrazole-1-carbothiaote **CAS:** S-2-propen-1-yl 5-amino-2,3-dihydro-2-(1-methylethyl)-4-(2-methylphenyl)-3-oxo-1H-pyrazole-1-carbothioate **CAS No.:** 473798-59-3 **Formula:** $C_{17}H_{21}N_3O_2S$ **MW:** 331.4 g/mol **SMILES:** Cc1ccccc1c2c(n(n(c2=O)C(C)C)C(=O)SCC=C)N		NG Method validation	48400027 48400034	NA	NA
MAJOR (>10%) TRANSFORMATION PRODUCTS						
No major transformation products were identified.						
MINOR (<10%) TRANSFORMATION PRODUCTS						
No minor transformation products were identified.						
REFERENCE COMPOUNDS NOT IDENTIFIED						
All compounds used as reference compounds were identified.						

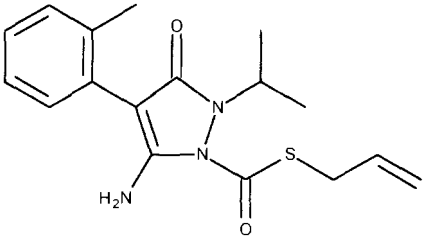
[A] AR means "applied radioactivity". MW means "molecular weight".

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

ENVIRONMENTAL CHEMISTRY METHOD (ECM) STANDARD EVALUATION PROCEDURE (SEP) CHECKLIST: BACKGROUND AND INITIAL REVIEW INFORMATION

Referenced page numbers appear in the uppermost right corner of each page of both MRIDs.

I. Background Information

A.	**Title of Method**	S-2188 - validation of the analytical method for the determination of a test substance in aqueous solutions following OPPTS 860.1340, SANCO/3029/99 rev.4 and SANCO/825/00 rev.7.	
B.	**ECM No.**	[*Leave blank. This is for BEAD ECB's use.*]	
C.	**MRID No.**	48400034	
D.	**Matrix**	Water	
E.	**Analyte(s) detected**	**Compound:**	
		Common name:	Fenpyrazamine
		IUPAC name:	Allyl 5-amino-2-isopropyl-4-(2-methylphenyl)-3-oxo-2,3-dihydro-1H-1-pyrazolecarbothioate
		CAS name:	1H-Pyrazole-1-carbothioic acid, 5-amino-2,3-dihydro-2-(1-methylethyl)-4-(2-methylphenyl)-3-oxo-, S-2-propenyl ester (p. 10 of MRID 48400027).
		CAS No:	473798-59-3 (p. 10 of MRID 48400027).
		Synonyms:	S-2188, V-10135, pyrazoline fungicide (p. 10 of MRID 4840027; p. 8 of MRID 48400034).
			

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Information obtained from p. 10 of MRID 48400027; p. 8 of MRID 48400034 (IUPAC name obtained from MRID 48399929, p. 21).

II. Information about the Laboratory

A.	**Name**	Springborn, Smithers Laboratories.
B.	**Address**	790 Main Street, Wareham, Massachusetts 02571-1037.
C.	**Telephone No.**	Not reported.
D.	**Name of the Study Director**	Marjorie E. Dix (p. 4 of MRID 48400034).
E.	**Name of the Lead Chemist**	Not reported; Assistant Chemist: Donald Gries.
F.	**Laboratory Validation:**	A statement of report validity was included as part of the Quality Assurance statement (p. 3 of MRID 48400034).

Information obtained from pp. 3-4 of MRID 48400034.

III. Method Summary Information for Analyte(s): **Fenpyrazamine (S-2188)**

A.	**Statement of Data Confidentiality**	Yes (p. 1i of MRID 48400034).
1.	**Is the Method Classified or Confidential?**	No.
2.	**Submitted Prior to 2008 with a Non-Standard Claim of Confidentiality?**	No.
B.	**Sample Preparation**	Well water was fortified with $NaHCO_3$ (147 mg/L), $CaSO_4 \cdot H_2O$ (92 mg/L), $MgSO_4$ (92 mg/L) and KCl (6.0 mg/L) to yield a pH of 8.1, hardness and alkalinity (as $CaCO_3$) of 160 mg/L and 110 mg/L, respectively and specific conductance of 500 µmhos/cm (p. 9 of MRID 48400034). The TOC was 1.5 mg/L. Water (100 mL) was fortified with fenpyrazamine, in acetonitrile, at 1.00, 500 and 20,000 µg a.i./L (pp. 11-12 of MRID 484000.4). Co-solvent concentrations were 0.05-0.1% by volume. The 20,000 x LOQ test solutions were sonicated for *ca.* 30 minutes to produce homogenous solutions.

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C.	**Sample Extraction**	Load sample (100 mL, draw rate 7 psi vacuum) onto a solid phase extraction column (Waters Oasis HBL, 6.0 mL, 500 mg) preconditioned with methanol and water (p. 11 of MRID 48400034). The loaded column is dried (20 psi vacuum, 10 minutes), then the analyte is eluted with acetonitrile (5.0 mL). The eluate is brought to volume (10.0 mL) with water:acetic acid (1,000:0.5, v:v). If needed, additional dilutions are made with acetonitrile:water:acetic acid (1,000:1,000:0.5, v:v:v).
D.	**Sample Cleanup**	None reported.
E.	**Sample Derivatization (if applicable)**	None reported.
F.	**Sample Analysis**	LC/MS/MS (p. 12 of MRID 48400034).
1.	**Instrumentation**	Waters 2695 HPLC system and MicroMass Quattro Micro MS system equipped with MicroMass Electro Spray in positive ionization mode (pp. 9, 12 of MRID 48400034).
2.	**Primary Column**	YMC ODS-AQ, S-3 column (2.0 x 50 mm, 3-µm, 120 Å; p. 12 of MRID 48400034).
3.	**Confirmatory Column (if any)**	None reported.
4.	**Detector**	Multiple Reaction Monitoring (MRM; Figures 1-6, pp. 19-24 of MRID 48400034).
5.	**Other Confirmatory Techniques (if any)**	The ECM monitored only one ion transition: 332.1 m/z > 231.0 m/z (p. 12 of MRID 48400034). The ILV monitored two ion transitions, 332.1 m/z > 272.0 m/z and 332.1 m/z > 230.3 m/z, for quantitation and confirmation, respectively; (p. 9; Appendix 3, Figures 1-18, pp. 77-94, Figures 20-35, pp. 96-111 of MRID 48400027).
6.	**Other Relevant Information**	LC conditions: isocratic mobile phase of 0.05% acetic acid in acetonitrile:water (50:50, v:v), column temperature 20°C, injection volume 10 µL, flow rate 0.2 mL/minute (p. 12 of MRID 48400034). Retention time of fenpyrazamine was *ca.* 4.3 minutes.
G.	**Detection and Quantitation Limits**	
1.	**Limit of Quantitation (LOQ)**	

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	Claimed in Method	1.00 µg/L (p. 12 of MRID 48400034).	**Estimated**	No. LOQ defined as the lowest fortification level yielding acceptable mean recovery and relative standard deviation.
2.	**Limit of Detection (LOD)**			
	Claimed in Method	0.10 µg/L (p. 14 of MRID 48400034).	**Estimated**	No. Calculated as follows: $LOD_{INST} = C_{LS}$ and $LOD = LOD_{INST} \times DF_{CNTL}$ C_{LS} = Concentration of lowest calibration standard at which peak detected with a signal/noise greater than three. LOD_{INST} = Limit of detection at the instrument where adequate peak was obtained (1.00 µg a.i./L). DF_{CNTL} = Dilution factor of the control samples (lowest dilution factor used = 0.100).

H. Recovery (Accuracy)/Precision Data; expressed as percentage of applied (n = 5)[1]

Spiking Level (µg a.i./L)	Parameter	Fenpyrazamine in Well Water
1.00 (LOQ)	Range	74.8-116
	Mean	102
	SD	16.4
	RSD	16.2
500	Range	90.2-98.0
	Mean	93.8
	SD	3.32
	RSD	3.53
20,000	Range	55.4-93.9
	Mean	81.2
	SD	15.2
	RSD	18.8

Information obtained from p. 9, Appendix 3, Figures 1-18, pp. 77-94, Figures 20-35, pp. 96-111 of MRID 48400027; and pp. 1i, 9, 11-12, 14, Figures 1-6, pp. 19-24 of MRID 48400034.
1 Results from Table 1, p. 18 of MRID 48400034; verified by reviewer (DER Attachment 2).

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IV. Detailed Information about the Method

		YES	NO	REVIEW FURTHER
A.	**Does the method require spiking with the analytes(s) of interest?**	x		p. 11 of MRID 48400034.
B.	**If the method requires explosive or carcinogenic reagents, are proper precautions explained?**			Not applicable.
C.	**Is the following information supplied?**			
1.	**Detailed stepwise description of:**			
a.	**The sample preparation procedure?**	x		p. 9 of MRID 48400034.
b.	**The sample spiking procedure?**	x		p. 11 of MRID 48400034.
c.	**The extraction procedure?**	x		p. 11 of MRID 48400034.
d.	**The derivatization procedure?**			Not applicable.
e.	**The clean-up procedure?**			Not required.
f.	**The analysis procedure?**	x		p. 12 of MRID 48400034.
2.	**Procedures for:**			
a.	**Preparation of standards?**	x		p. 10 of MRID 48400034.
b.	**Calibration of instrument?**	x		p. 13 of MRID 48400034.
3.	**List of glassware and chemicals**	x		p. 9 of MRID 48400034.
a.	**Are sources recommended?**	Chemicals	Glassware	
b.	**Are they commercially available?**	x		
4.	**Name, model, *etc.*, of the instrument, column, detector, *etc.*, used?**	x		
a.	**Are sources recommended?**	x		
b.	**Are they commercially available?**	x		
5.	**LOD**			

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		YES	NO	REVIEW FURTHER
a.	**Is there an explanation of how it was calculated?**	x		p. 14 of MRID 48400034.
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 (Table 1, p. 18; Figure 6, p. 24 of MRID 48400034)	In three instances (n = 5) peaks were detected at the retention time of fenpyrazamine, but were only 3-5% of LOQ (DER Attachment 2). Reported recoveries for fortified samples were not corrected for mean residues detected in control samples.
6.	**LOQ**			
a.	**Is there an explanation of how it was calculated?**	x		p. 12 of MRID 48400034.
b.	**Is it a scientifically accepted procedure?**	x		
7.	**Precision and accuracy data**			
a.	**Were there an adequate number of spiked samples analyzed?**	x		Five replicates at each spiking level (p. 11 of MRID 48400034).
b.	**Are the mean recoveries between 70-120%?**	x		Table 1, p. 18 of MRID 48400034.
c.	**Are the RSDs of the replicates 20% or less at or above the LOQ?**	x		

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		YES	NO	REVIEW FURTHER
8.	**Description and/or explanation of:**			
a.	**Areas where problems may be encountered?**	x		The SEP elution step was modified to methanol (5 mL) followed by acetonitrile (5 mL, p. 19 of MRID 48400027).
b.	**Steps that are critical?**	x		Flow rate when loading the SPE cartridge should be *ca.* 10-15 mL/minute (pp. 19, 21 of MRID 48400027).
c.	**Interferences that may be encountered?**		x	
9.	**Characterization of the Matrix(ces)?**	x		p. 9 of MRID 48400034.

Information obtained from pp. 19, 21 of MRID 48400027; and pp. 9-14, Table 1, p. 18, Figure 6, p. 24 of MRID 48400034.

V. Representative Chromatograms

		YES	NO	REVIEW FURTHER
A.	**Are there representative chromatograms for:**			
1.	**Analyte(s) in each matrix at the LOQ and 10 x LOQ?**	LOQ (ECM, ILV) 10 x LOQ (ILV)		Figure 3, p. 21 of MRID 48400034; Appendix 3, Figures 9-13, pp. 85-89, Figures 26-30, pp. 102-106 of MRID 48400027.

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2.	**Method blanks?**	ILV	ECM	Reagent blanks were analyzed with no significant levels of residues detected at the retention time of fenpyrazamine (Appendix 3, Figure 6, p. 82, Figure 25, p. 101, Figure 39, p. 116 of MRID 48400027).
3.	**Matrix blanks?**	ECM, ILV		Figure 6, p. 24 of MRID 48400034; Appendix 3, Figures 7-8, pp. 83-84 of MRID 48400027.
4.	**Standard curves?**	ECM, ILV		Figure 7, p. 25 of MRID 48400034; Appendix 3, Figure 19, p. 95, Figure 36, p. 112 of MRID 48400027.
a.	**Do the standard curves have acceptable linearity?**	x		r^2 = 0.9983-0.99905.
5.	**Standards that can be used to recalculate some of the values for analyte(s) in the sample chromatograms?**	ECM		DER Attachment 2.
B.	**Can the responses of the analytes(s) in the chromatograms of the lowest spiking level be accurately measured?**	x		

Information obtained from Appendix 3, Figures 7-13, pp. 83-89, Figure 19, p. 95, Figures 25-30, pp. 101-106, Figure 36, p. 112, Figure 39, p. 116 of MRID 48400027; and Figure 3, p. 21, Figures 6-7, pp. 24-25 of MRID 48400034.

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VI. Good Laboratory Practice (GLP) Standards

		YES	NO	REVIEW FURTHER
A.	**Is there a statement of adherence to the FIFRA GLP standards?**	x		p. 2 of MRID 48400034.

Information obtained from p. 2 of MRID 48400034.

VII. Independent Lab Validation (ILV)

		YES	NO	REVIEW FURTHER
A.	**Was an ILV performed?**	x		
B.	**Was the validation independent?**	x		
C.	**Did the ILV's precision/accuracy data meet the criteria established in OPPTS Guideline 850.7100?**	x		p. 9 of MRID 48400027
D.	**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		pp. 21-22 of MRID 48400027. Elution of analyte from the SPE cartridge with methanol (5 mL) followed by acetonitrile (5 mL, p. 16 of MRID 48400027).[1]

E.	**Recovery (Accuracy)/Precision Data; expressed as percentage of applied (n = 5)[2]**		
	Spiking Level (µg a.i./L)	Parameter	Fenpyrazamine in Tap Water
	1.00 (LOQ)	Range	98.1-105
		Mean	103
		SD	2.68
		RSD	2.62
	10.0	Range	91.2-97.0
		Mean	94.6

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		SD	2.22
		RSD	2.34

Information obtained from pp. 9, 16, 21-22 of MRID 48400027.
1 Recoveries and RSDs from trials using only acetonitrile elution of SPE cartridges were not within OPPTS 850.7100 criteria (pp. 15, 19, Tables 1-2, pp. 25-26 of MRID 48400027).
2 Results from p. 9, Appendix 4, Figure 39, p. 116 of MRID 48400027; verified by reviewer (DER Attachment 2).

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** [*Acceptable*]			
2.	**Major Deficiencies**	x		ECM did not use a confirmatory method.
3.	**Minor Deficiencies**	x		ECM did not conduct fortifications at 10 x LOQ. ILV peak area results were provided for the confirmation ion, but not calculated residue levels.

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

IX. Recommendations

1. For the ECM, fortifications at 10 x LOQ should have been performed.

2. The ECM did not employ a confirmatory analysis method.

3. For the ILV, chromatographic results were provided for the confirmatory ion, but calculated residue levels should also have been provided.

4. An additional ECM (MRID 48399918), but no supporting ILV, for the determination of fenpyrazamine in drinking (tap) and surface (pond) water was submitted with this data package. LOQs and LODs were 0.10 µg/L and 1.0 µg/L, respectively, for fenpyrazamine in drinking water, and 1.0 µg/L and 10 µg/L, respectively, in surface water.

Name and Dated Signature of Primary Reviewer

12/7/12

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