Test Material:	Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-Cyhalothrin and Permethrin
MRID:	47053001, 47053002, **48935001**
Title:	Independent Laboratory Validation of "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-Cyhalothrin and Permethrin in Sediment".
EPA PC Code:	097805, 109303, 109702, 109701, 127901, 128825, 128831, 128897
OCSPP Guideline:	850.6100

For CDM Smith

Primary Reviewer: Lisa Muto **Signature:** 

Date: 9/2/14

Secondary Reviewer: Lynne Binari **Signature:** 

Date: 9/2/14

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

Date: 9/2/14

Independent laboratory validation of previously submitted analytical method for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in sediment

Reports: **ECM:** Previously submitted and reviewed EPA MRID No. 47053001/ 47053002. Reed II, R. 2006. Laboratory Validation: Validation of the Residue Analytical Method: "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-Cyhalothrin and Permethrin in Sediment", Final Report. Unpublished study performed by Morse Laboratories, Inc., Sacramento, California; sponsored and submitted by Pyrethroid Working Group (Consortium No. 64977), c/o Syngenta Crop Protection, Inc., Greensboro, North Carolina; 418 pages (pp. 1-4). Morse Protocol No.: MLI-06-02 and Project No.: ML06-1286-PWG. Experimental start date April 21, 2006, and completion date May 18, 2006 (p. 7). Final report issued November 29, 2006.
ILV: EPA MRID No. 48935001. Grant, J. 2012. Independent Laboratory Validation of "Residue Analytical Method for the Determination of Residues of Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-Cyhalothrin and Permethrin in Sediment". ABC Study No.: 68768. Report prepared by ABC Laboratories, Inc., Columbia, Missouri; sponsored and submitted by Pyrethroid Working Group c/o Landis International, Inc., Valdosta, Georgia; 571 pages. Final report issued August 30, 2012.

Document No.: MRID 47053001/47053002 (ECM) and **48935001 (ILV)**

Guideline: 850.6100

Statements: **ECM**: Previously reviewed/data not included in this DER, except as needed. The ECM DER is in **Attachment 3**.
ILV: The study was conducted in accordance with U.S. EPA GLP (CFR Title 40, Part 160; p. 3). Signed and dated No Data Confidentiality (for U.S.A.), Data Confidentiality (countries other than U.S.A.), GLP, Quality Assurance and Certification of the Authenticity statements were provided (pp. 2-5).

Classification: This analytical method is considered supplemental. Portions of the study can be used to fulfil the data requirement, while *some issues need to be addressed*. Recovery data did not meet guideline requirements for analysis of bifenthrin at the LOQ, permethrin at the 10×LOQ, and *lambda*-cyhalothrin at either fortification level. The determination of the LOQ and LOD were not based on scientifically acceptable procedures. The LOD of the ECM was not the same than the ILV. The residue calculations specified for the correction of sample recoveries; in the ILV, only recoveries of bifenthrin were actually corrected. The identity of fenpropathrin was not confirmed in the method.

PC Code: 097805, 109303, 109702, 109701; 127901, 128825, 128831, 128897

Reviewer: José Meléndez, USEPA **Date:** January 23, 2017

Signature: José Luis Meléndez

Executive Summary

This independent laboratory validation (ILV) analytical method (ABC Study No.: 68768), of previously submitted and reviewed EPA MRID No. 47053002, Morse Project No.: ML06-1286-PWG, is designed for the quantitative determination of eight pyrethroids (bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin) in sediment by GC/MS using negative ion chemical ionization (see **Attachment 3**). The stated LOQs for the method are 0.10 µg/kg-dw for all of the analytes, except permethrin (1.0 µg/kg-dw; **Table 1**). Assuming a typical range of organic carbon content for majority of sediments, ranging from 2-10%, the organic carbon normalized LOQ would range from 1.0-5.0 µg/kg$_{OC}$ for all the analytes except permethrin, and 10-50 µg/kg$_{OC}$ for permethrin. The LOQs are less than the lowest toxicological level of concern in sediment for all eight tested compounds. The ILV validated the method with the second trial for cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin and permethrin at the stated LOQs; however, the recovery results of bifenthrin and *lambda*-cyhalothrin did not meet guideline requirements at the LOQ. The ILV required some minor modifications of the GC/MS method to achieve desired sensitivity. The identification of all of the analytes, except fenpropathrin, was confirmed in the analytical method. No matrix characterization of the sediment was provided.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, and *Lambda*-cyhalothrin	47053001, 47053002[1]	48935001		Sediment	11/29/2006	Pyrethroid Working Group c/o Landis International, Inc.	GC/MS	0.10 µg/kg[2] (0.10 ppb)
Permethrin								1.0 µg/kg[2] (1.0 ppb)

[1] Previously submitted and reviewed study. Refer to **Attachment 3**.

[2] Assuming a typical range of organic carbon content for majority of sediments of 2-10%, the organic carbon normalized LOQ would range from ~10-50 µg/kg$_{OC}$ for permethrin, and ~1.0-5.0 µg/kg$_{OC}$ for all other pyrethroids.

All page numbers refer to those in the bottom right-hand corner of "Volume 1 of 2" of MRID 48935001 (ILV) unless otherwise noted. "Volume 2 of 2" of MRID is attached after the last page of "Volume 1 of 2". "Volume 2 of 2" is independently paginated.

I. Principle of the Method

Soil samples (50 g) were mixed with 75 mL of methanol:water (1:1, v:v) and 50 mL of hexane for 60 minutes on a platform shaker (pp. 13, 20; Appendix 1, p. 128; Appendix 1, Appendix 1, pp. 308-309; Appendix 1, Appendix 1, Appendices 1-2, pp. 317-318). After centrifugation (*ca*. 4000 rpm for 5 minutes), an aliquot (10 mL) of the hexane extract was reduced to dryness using a heating block set at *ca*. 40°C. The residue was reconstituted in 2 mL of hexane using brief sonication. The hexane extract was purified via solid phase extraction (SPE) using a Silica (Si) SPE column eluted with 6 mL of hexane:diethyl ether (9:1, v:v). The eluate was reduced to dryness using a heating block set at *ca*. 40°C then the residue was reconstituted in 1 mL of 0.1% peanut oil in acetone prior to analysis via GC/MS.

Samples were analyzed for all analytes with a HP Agilent 6890N GC system equipped with a Agilent CP-Sil 8CB-MS column (0.25 mm x 30 m, 0.25 µm film thickness) and a HP 5973 MS (pp. 20-22; Appendix 1, pp. 128-130; Appendix 1, Appendix 1, pp. 309-311). The GC/MS system utilized a quadrupole GC/MS with negative ion chemical ionization (NCI; methane @ 40%) with a splitless injector (50 mL/min., purge on at 2 min.); injection volume was 4 µL. Two qualifier ions (Q1 and 2) were monitored along with the target ion (T) for five of the eight analytes: bifenthrin, 386 m/z (T), 387 m/z (Q1), 241 m/z (Q2); cypermethrin and cyfluthrin, 207 m/z (T), 209 m/z (Q1), 171 m/z (Q2); deltamethrin, 297 m/z (T), 81 m/z (Q1), 296 m/z (Q2); and *lambda*-cyhalothrin, 205 m/z (T), 241 m/z (Q1), 243 m/z (Q2). Two of the analytes were monitored with one qualifier ions along with the target ion: permethrin, 207 m/z (T), 209 m/z (Q1); and esfenvalerate, 211 m/z (T), 213 m/z (Q1). Fenpropathrin was only monitored with a target ion, 141 m/z (T).

In the ILV, the LOQs for the method are 0.10 µg/kg (0.10 ppb) for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and 1.0 µg/kg (1.0 ppb) for permethrin (p. 20). The corresponding LODs were estimated to be 0.03 µg/kg for all analytes, except permethrin (0.3 µg/kg); the LODs were estimated at *ca*. 1/3 of the LOQ.

II. Recovery Findings

ECM (MRID 47053001/2): Previously submitted and reviewed separately (**Attachment 3**). The ECM study report was included in the ILV MRID 48935001 as Appendix 1 (pp. 105-320 of Volume 1 of 2; pp. 1-206 of Volume 2 of 2).

ILV (MRID 48935001): Mean recoveries and RSDs were within guideline requirements for analysis of cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, and fenpropathrin at the LOQ and 10×LOQ (p. 10-12). Mean recoveries and RSDs were within guideline requirements for analysis of bifenthrin at the 10×LOQ only and permethrin at the LOQ only. Mean recoveries of *lambda*-cyhalothrin did not meet guideline requirements for either fortification level. The method was validated with the second trial. The sediment was described as fresh water sediment from Tift County, Georgia; no matrix characterization was provided (p. 18). The sediment was checked for pyrethroid contamination prior to use (p. 25-26). The analytes were identified by

GC/MS with negative ion chemical ionization. Seven of the eight pyrethroids were identified with one or two ion transitions; fenpropathrin was only identified with a target ion.

Table 2. Initial Validation Method Recoveries for Pyrethroids in Sediment

Analyte	Fortification Level (ppb)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-cyhalothrin and Permethrin	ECM data previously reviewed (refer to previous DER in **Attachment 3**).					

Table 3. Independent Validation Method Recoveries for Pyrethroids in Sediment

Analyte	Fortification Level (ppb)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Bifenthrin	0.10 (LOQ)	5	87-144	105	22.51	**21**
	1.0	5	86-109	96	8.58	9
Fenpropathrin	0.10 (LOQ)	5	96-103	100	2.45	2
	1.0	5	98-123	109	9.18	8
Lambda-cyhalothrin	0.10 (LOQ)	5	112-137	**126**	10.62	8
	1.0	5	108-136	**122**	12.25	10
Permethrin[1]	1.00 (LOQ)	5	98-118	110	10.49	8
	10.0	5	115-147	**127**	5.42	4
Cyfluthrin	0.10 (LOQ)	5	100-142	120	18.64	16
	1.0	5	95-125	108	10.72	10
Cypermethrin[1]	0.10 (LOQ)	5	83-131	110	20.08	18
	1.0	5	93-129	108	13.39	12
Esfenvalerate	0.10 (LOQ)	5	98-131	118	13.17	11
	1.0	5	104-134	119	11.62	10
Deltamethrin	0.10 (LOQ)	5	90-110	101	9.09	9
	1.0	5	113-137	120	9.86	8

Data were obtained from p. 12; Table 2, p. 32; Table 4, p. 34; Table 6, p. 36; Table 8; p. 38; Table 10, p. 40; Table 12; p. 42; Table 14, p. 44; Table 16, p. 46 of the study report. Only Trial 2 results shown. Values which did not meet guideline requirements are reported in **bold and red**.

[1] Supplied as separate *cis*- and *trans*-test substances.

III. Method Characteristics

In the ILV, the LOQs for the method are 0.10 µg/kg (0.10 ppb) for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and 1.0 µg/kg (1.0 ppb) for permethrin (p. 20). The corresponding LODs were estimated to be 0.03 µg/kg for all

analytes, except permethrin (0.3 µg/kg); the LODs were estimated at *ca*. 1/3 of the LOQ. The ECM MRID 47053002 was previously submitted and reviewed. "The ECM utilized limits of quantitation (LOQ) and detection (LOD) of 0.1 µg/kg and 0.02 µg/kg, respectively, for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin and *lambda*-cyhalothrin, and 1.0 µg/kg and 0.2 µg/kg, respectively, for permethrin (p. 30 of MRID 47053002)" (data from Environmental Chemistry Method Review Report of MRID 47053002, **Attachment 3**). In the ECM validation, the LOQ was defined as the lowest level of fortification to have demonstrated an acceptable recovery and precision; no calculations were provided (Appendix 1, p. 135). The ECM validation study author noted that the LOQ and LOD "were not statistically determined for this method" (Appendix 1, p. 135). The ILV did not provide any other support for the LOQ or LOD. Additionally, the estimated LODs of the ILV and ECM were not equivalent.

Table 4. Method Characteristics*

	Sediment			
Characteristic\Chemical	**Bifenthrin**	**Fenpropathrin**	***Lambda*-cyhalothrin**	**Permethrin[1]**
Limit of Quantitation (LOQ)	0.10 µg/kg			1.0 µg/kg
Limit of Detection (LOD)	0.02 µg/kg (ECM)[2] 0.03 µg/kg (ILV)			0.2 µg/kg (ECM) 0.3 µg/kg (ILV)
Linearity (calibration curve r^2 and concentration range)[3]	r^2 = 0.9953 (0.5-20 ng/mL)	r^2 = 0.9963 (0.5-20 ng/mL)	r^2 = **0.9872** (0.5-20 ng/mL)	r^2 = 0.9956 (5-200 ng/mL)
Repeatable[2]	Yes			
Reproducible	No[4]	Yes	No[5]	No[6]
Specific	Yes	No[7]	No[8]	Yes
Characteristic\Chemical	**Cyfluthrin**	**Cypermethrin[1]**	**Esfenvalerate**	**Deltamethrin**
Limit of Quantitation (LOQ)	0.10 µg/kg			
Limit of Detection (LOD)	0.02 µg/kg (ECM) 0.03 µg/kg (ILV)			
Linearity (calibration curve r^2 and concentration range)[3]	r^2 = 0.9959 (0.5-20 ng/mL)	r^2 = **0.9944** (0.5-20 ng/mL)	r^2 = 0.9958 (0.5-20 ng/mL)	r^2 = 0.9965 (0.5-20 ng/mL)
Repeatable[2]	Yes			
Reproducible	Yes			
Specific	No[9]	No[9]	Yes	No[8]

***** Data were obtained from p. 20; Figure 2, p. 48; Figure 4, p. 50; Figure 6, p. 52; Figure 8, p. 54; Figure 10, p. 56; Figure 12, p. 58; Figure 14, p. 60; Figure 16, p. 62 of the study report unless noted otherwise.

1 Supplied as separate *cis*- and *trans*-test substances.

2 The ECM (MRID 47053002) was previously submitted and reviewed. Data was taken from Environmental Chemistry Method Review Report of MRID 47053002 (**Attachment 3**). A copy of the ECM was included in the ILV MRID as Appendix 1.

3 Reviewer-calculated calibration curves yielded the same linearity, r^2 values of 0.9872-9963 for the eight analytes (see DER **Attachment 2**). Linearity is satisfactory when $r^2 \geq 0.995$.

4 RSD at LOQ fortification was 21%.

5 Mean recoveries at LOQ and 10×LOQ were >120%.

6 Mean recovery at 10×LOQ was >120%.

7 The method did not include a confirmation of analyte identity.

8 Matrix interferences were significant around the retention time of the analyte peak at the LOQ (Figure 19, p. 82; Figure 22, p. 103).

9 Cyfluthrin and cypermethrin, as well as permethrin, were detected together in the chromatogram of 207 m/z (Figure 20, pp. 89-90). The retention time ranges of cyfluthrin and cypermethrin were extremely close and not easily distinguishable in the example chromatograms at the LOQ and 10×LOQ.

IV. Method Deficiencies and/or Reviewer's Comments

1. The ILV report was submitted in support of the ECM MRID 4703001/47053002 which was previously submitted and reviewed in 2011 (**Attachment 3**). The reviewer used the data obtained from the Environmental Chemistry Method Review Report of MRID 47053001/47053002, as well as that from the original MRID contained in Appendix 1 of this MRID, in order to assess the data from the ILV, as necessary (pp. 105-320 of Volume 1 of 2; pp. 1-206 of Volume 2 of 2). The majority of the results of MRID 47053001/47053002 were not re-reported in this DER, since they are reported elsewhere.

2. Recovery data did not meet guideline requirements for analysis of bifenthrin at the LOQ (RSD 21%) and permethrin at the 10×LOQ (mean 127%; pp. 10-12). Mean recoveries of *lambda*-cyhalothrin did not meet guideline requirements at the LOQ (mean 126%) and 10×LOQ (mean 122%). OCSPP Guideline 850.6100 criteria for precision and accuracy requires mean recoveries for replicates at each spiking level to be between 70% and 120% and relative standard deviations (RSD) ≤20%.

3. The determination of the LOQ and LOD were not based on scientifically acceptable procedures. In the ECM, the LOQ was defined as the lowest level of fortification to have demonstrated an acceptable recovery and precision; no calculations were provided (Appendix 1, p. 135). The ECM validation study author noted that the LOQ and LOD "were not statistically determined for this method" (Appendix 1, p. 135). The ILV did not provide any other support for the LOQ or LOD. Additionally, the estimated LODs of the ILV and ECM were not equivalent. Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples.

4. The lowest toxicological level of concern in sediment was not reported in the study. An LOQ above toxicological levels of concern results in an unacceptable method classification. According to the latest ecological effects information available (refer to the Preliminary Risk Assessment for Synthetic Pyrethroids and Pyrethrins, dated September 30, 2016, DP Barcode 425791+).

5. The ECM and ILV residue calculations specified for the correction of sample recoveries for any residues found in the matrix control samples (pp. 24-25; Appendix 1, p. 131). In the ILV, only recoveries of bifenthrin were corrected since only those samples contained measurable residues in the control samples (p. 26). ECM report (MRID 47053002) was previously submitted and reviewed (**Attachment 3**).

6. There was no confirmation of the identity of fenpropathrin in the method, only the target ion was monitored (p. 22; Appendix 1, p. 129; Appendix 1, Appendix 1, p. 310).

7. The study author reported that matrix interference was ≤30% for the test matrix and each analyte (p. 13); however, the reviewer noted significant interferences around the retention times of *lambda*-cyhalothrin and deltamethrin at the LOQ (Figure 19, p. 82; Figure 22, p. 103). Cyfluthrin and cypermethrin, as well as permethrin, were detected together in the chromatogram of 207 m/z (Figure 20, pp. 89-90). The retention time ranges of cyfluthrin and cypermethrin were very close and not easily distinguishable in the example chromatograms at the LOQ and 10×LOQ.

8. Linearity (r^2) of the calibration standards was not always ≥0.995 (see Table 4 above).

9. The ILV report indicated that GC/MS optimization was necessary to increase sensitivity, especially for the analysis of deltamethrin (p. 20). The injection volume was increased from 2 µL to 4 µL. A splitless injection mode (50 mL/min., purge on at 2 min.) was used in the place of a pulsed splitless injection (30 psi for 1 min., purge flow to split vent 50 psi @ 2 min.). Also, the methane percentage to the detector was increased from 30% to 40%. These changes were not substantial and did not appear to require an internal validation of the ECM.

10. Recovery data and calibration curves from Trial 1 of the ILV were included in the MRID, but not reviewed in this DER. No GC chromatograms from Trial 1 were included.

11. Communication between the ECM personnel and ILV study author was recorded (p. 29; Volume 2 of 2, Appendix 4, pp. 233-250). The communication was limited to chromatography issues (optimization of the GC/MS analysis for deltamethrin) and recovery updates (Trial 2 recovery results of *lambda*-cyhalothrin and bifenthrin).

12. In the ILV, the time requirement for the sets consisting of 13 samples was 1 workday (8 hours) with the GC/MS analysis performed overnight (p. 30).

V. References

U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.

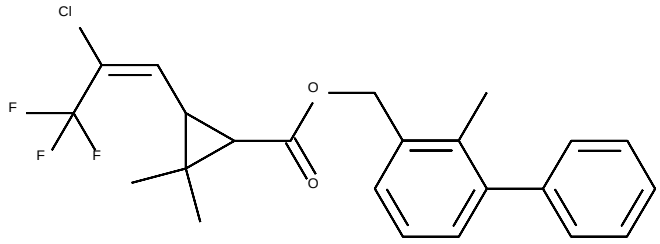
40 CFR Part 136. Appendix B. Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11, pp. 317-319.

Binari, L. Environmental Chemistry Method Review Report. MRIDs 47053001 and 47053002. [Secondary review by J. Meléndez; date reviewed April 20, 2012.]

Attachment 1: Chemical Names and Structures

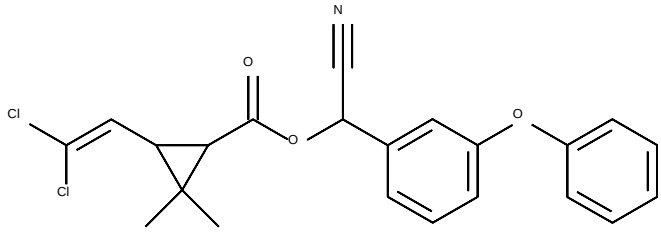
Bifenthrin

IUPAC name	2-Methylbiphenyl-3-ylmethyl (1RS,3RS)-3-[(Z)-2-chloro-3,3,3-trifluoroprop-1-enyl]-2,2-dimethylcyclopropanecarboxylate
CAS Name	(2-Methyl[1,1′-biphenyl]-3-yl)methyl (1R,3R)-rel-3-[(1Z)-2-chloro-3,3,3-trifluoro-1-propen-1-yl]-2,2-dimethylcyclopropanecarboxylate
CAS #	82657-04-3
SMILES	c1ccccc1c2c(C)c(COC(=O)C3C(C)(C)C3C=C(Cl)C(F)(F)F)ccc2



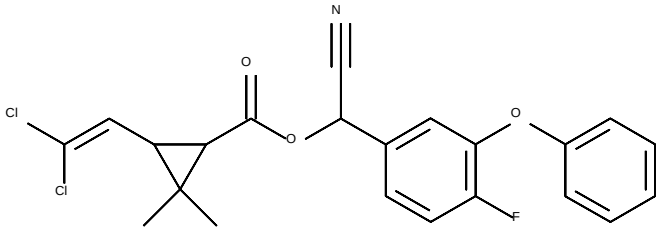
Cypermethrin

IUPAC name	(RS)-α-Cyano-3-phenoxybenzyl (1RS,3RS;1RS,3SR)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Name	Cyano(3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate
CAS #	52315-07-8
SMILES	Not found



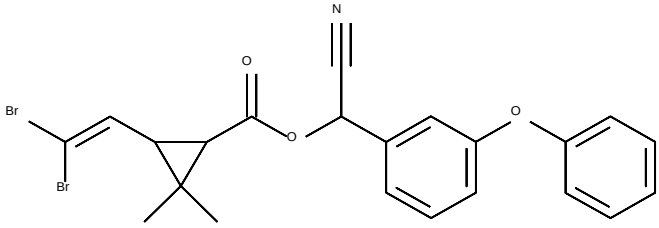
Cyfluthrin

IUPAC name	(RS)-α-Cyano-4-fluoro-3-phenoxybenzyl (1RS,3RS;1RS,3SR)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate
CAS Name	Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropancecarboxylate
CAS #	68359-37-5
SMILES	Not found



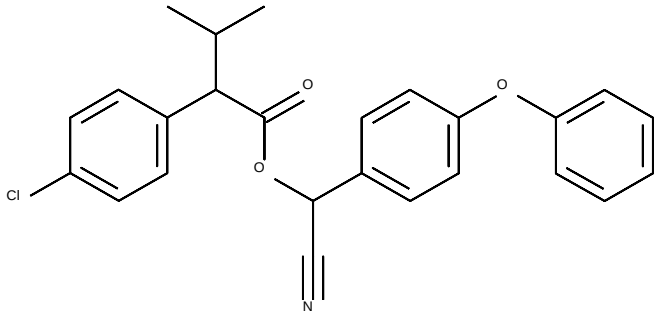
Deltamethrin

IUPAC name	(S)-α-cyano-3-phenoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate
CAS Name	(S)-cyano(3-phenoxyphenyl)methyl (1R,3R)-3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate
CAS #	52918-63-5
SMILES	CC1(C)C(C=C(Br)Br)C1C(=O)OC(C#N)c3cccc(Oc2ccccc2)c3



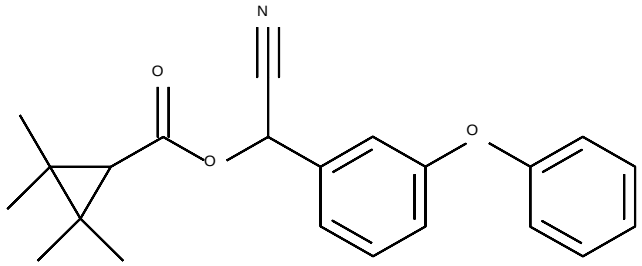
Esfenvalerate

IUPAC name	(S)-α-Cyano-3-phenoxybenzyl (S)-2-(4-chlorophenyl)-3-methylbutyrate
CAS Name	[S-(R*,R*)]-Cyano(3-phenoxyphenyl)methyl 4-chloro-2-(1-methylethyl)benzeneacetate
CAS #	66230-04-4
SMILES	[C@H](C(C)C)(C(=O)O[C@@H](C#N)C1=CC(=CC=C1)OC2=CC=CC=C2)C3=CC=C(C=C3)Cl



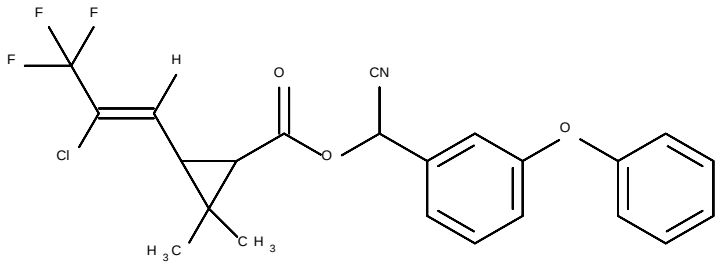
Fenpropathrin; Danitol

IUPAC name	(RS)-α-Cyano-3-phenoxybenzyl 2,2,3,3,-tetramethylcyclopropancecarboxylate
CAS Name	Cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate
CAS #	64257-84-7
SMILES	Not found



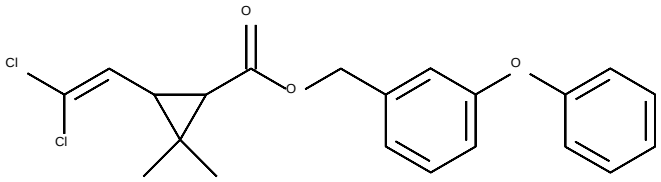
***Lambda*-cyhalothrin**

IUPAC name	Reaction product comprising equal quantities of (R)-α-cyano-3-phenoxybenzyl (1S,3S)-3-[(Z)-2-chloro-3,3,3-trifluoropropenyl]-2,2-dimethylcyclopropanecarboxylate and (S)-α-cyano-3-phenoxybenzyl (1R,3R)-3-[(Z)-2-chloro-3,3,3-trifluoropropenyl]-2,2-dimethylcyclopropanecarboxylate
CAS Name	(R)-cyano(3-phenoxyphenyl)methyl (1S,3S)-rel-3-[(1Z)-2-chloro-3,3,3-trifluoro-1-propen-1-yl]-2,2-dimethylcyclopropanecarboxylate
CAS #	91465-08-6
SMILES	FC(F)(F)C(Cl)=CC1C(C)(C)C1C(=O)OC(C#N)c2cc(Oc3ccccc3)ccc2



Permethrin

IUPAC name	3-Phenoxybenzyl(1RS)-cis-trans-3-(2,2-dichlorovinyl)-2,2-dimethyl-cyclopropanecarboxylate
CAS Name	(3-Phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethyl-cyclopropanecarboxylate
CAS #	52645-53-1
SMILES	CC1(C)C(C=C(Cl)Cl)C1C(=O)OCc3cccc(Oc2ccccc2)c3



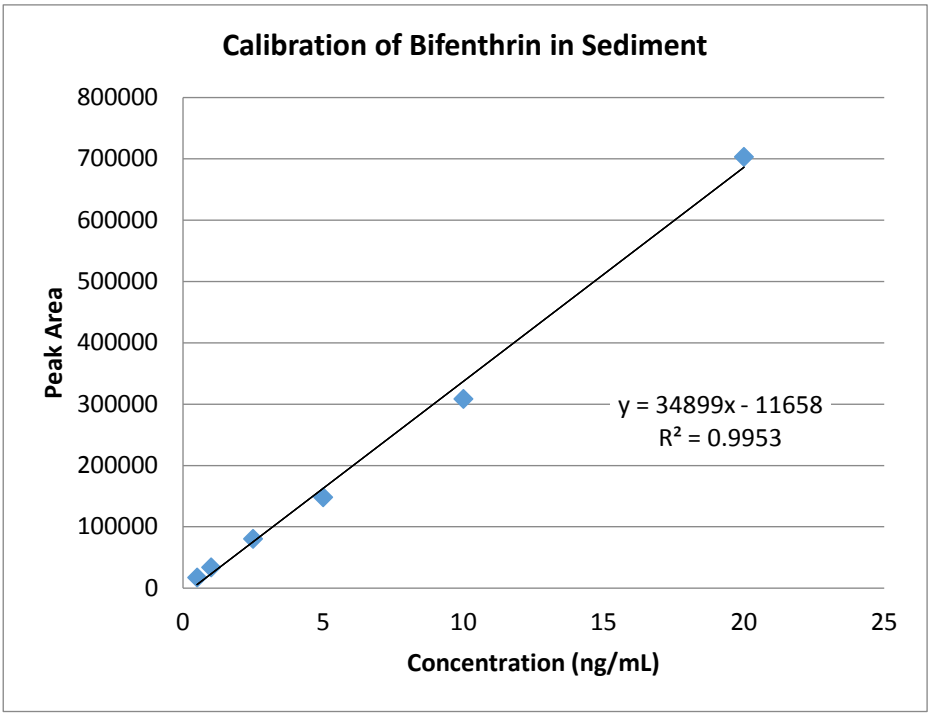
Attachment 2: Calculations

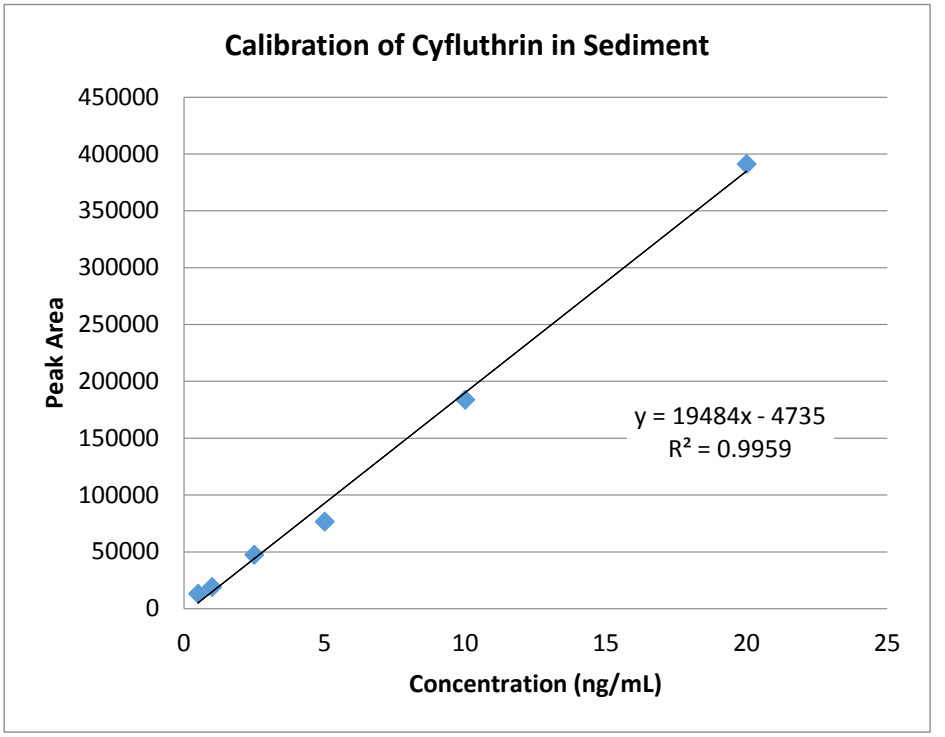
Chemical: Multiple Pyrethroids
MRID: 48935001
PC: 109701 -File
Guideline: 850.6100

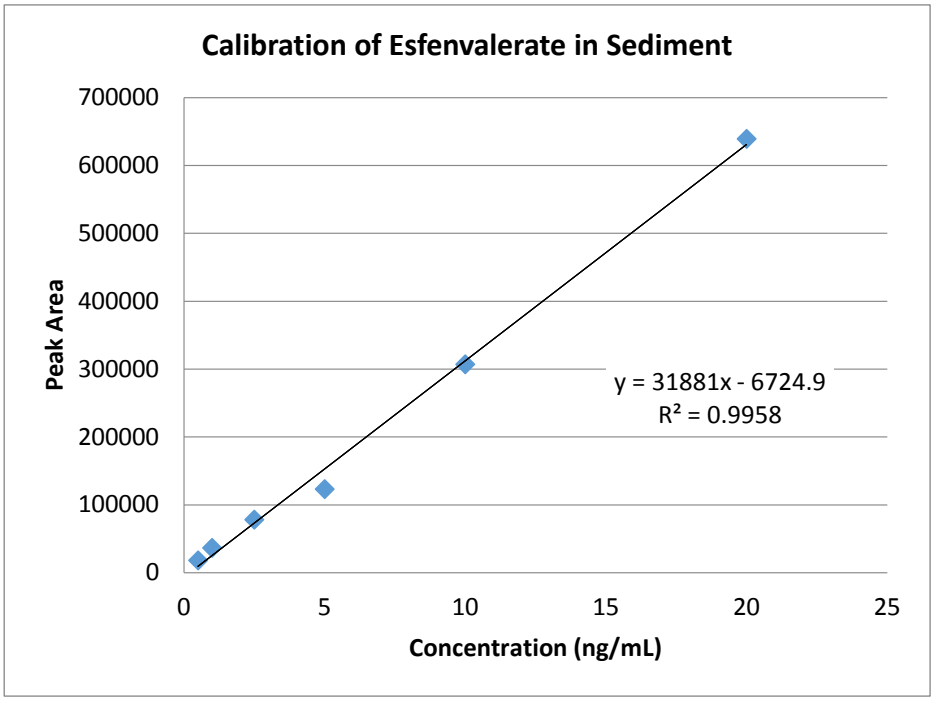
ILV Calibration Curve - Trial 2

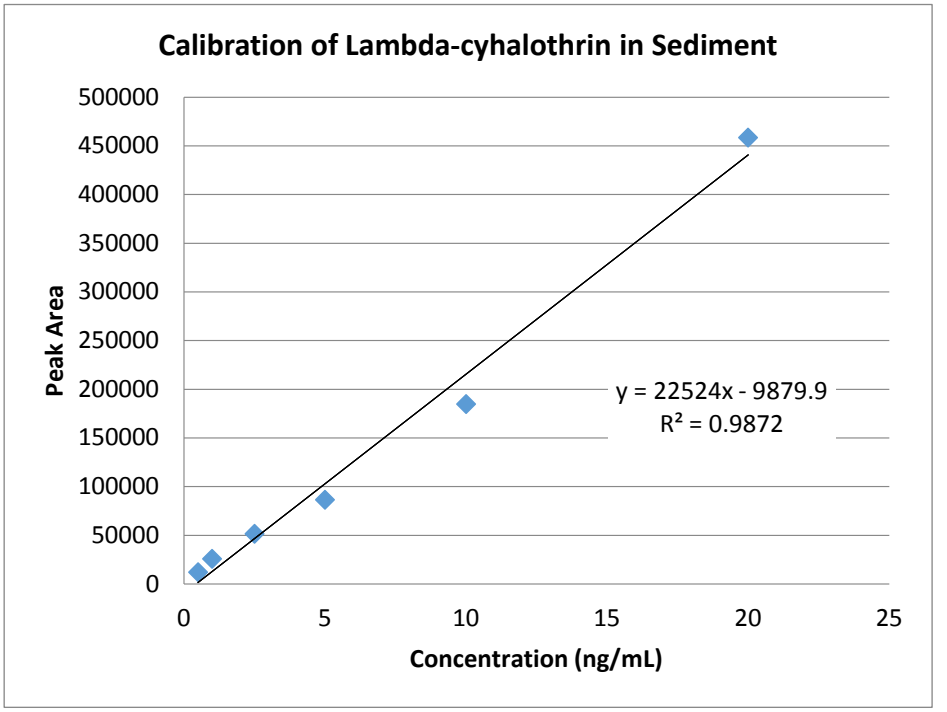
Calibration Curve Data	Bifenthrin		Cypermethrin		Cyfluthrin		Deltamethrin	
	Conc. (ng/mL)	Peak response	Conc. (ng/mL)	Peak response	Conc. (ng/mL)	Peak response	Conc. (ng/mL)	Peak response
	0.5	17392	0.5	25073	0.5	13283	0.5	2879
	1.0	33802	1.0	34141	1.0	19004	1.0	4650
	2.5	80409	2.5	96374	2.5	47578	2.5	12125
	5.0	148018	5.0	139957	5.0	76557	5.0	19814
	10.0	308443	10.0	376976	10.0	183762	10.0	46732
	20.0	703054	20.0	709417	20.0	391271	20.0	99699

Results (Peak Area) from Figure 2, p. 48; Figure 4, p. 50; Figure 6, p. 52; Figure 8, p. 54; Figure 10, p.



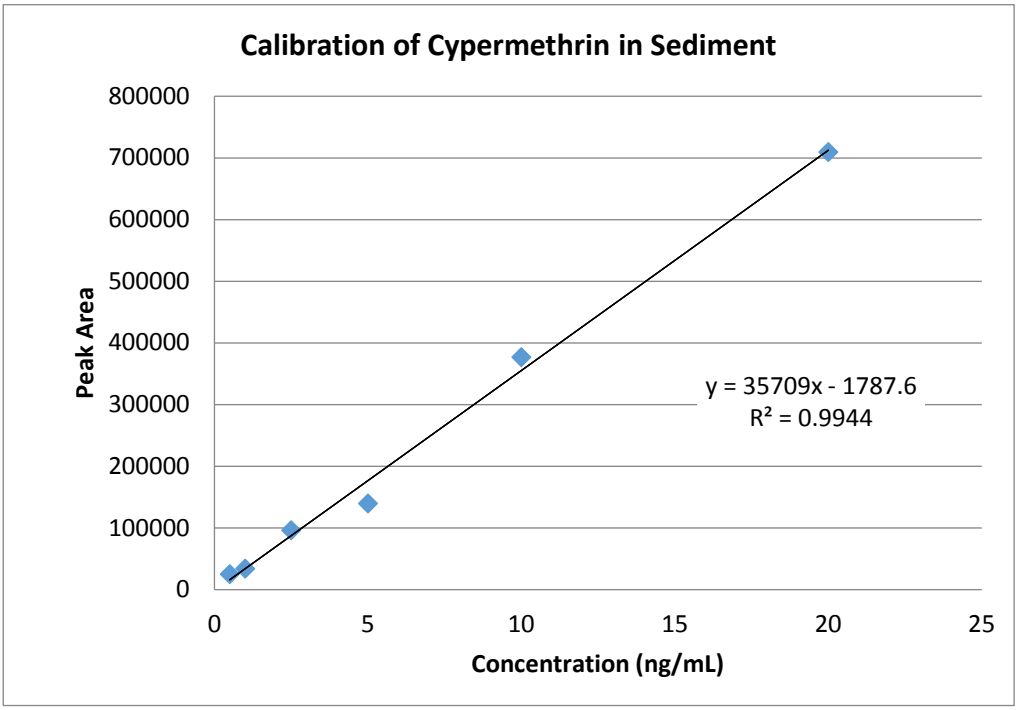

Calibration of Cyfluthrin in Sediment
Peak Area
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
0
5
10
15
20
25
Concentration (ng/mL)
y = 19484x - 4735
R² = 0.9959

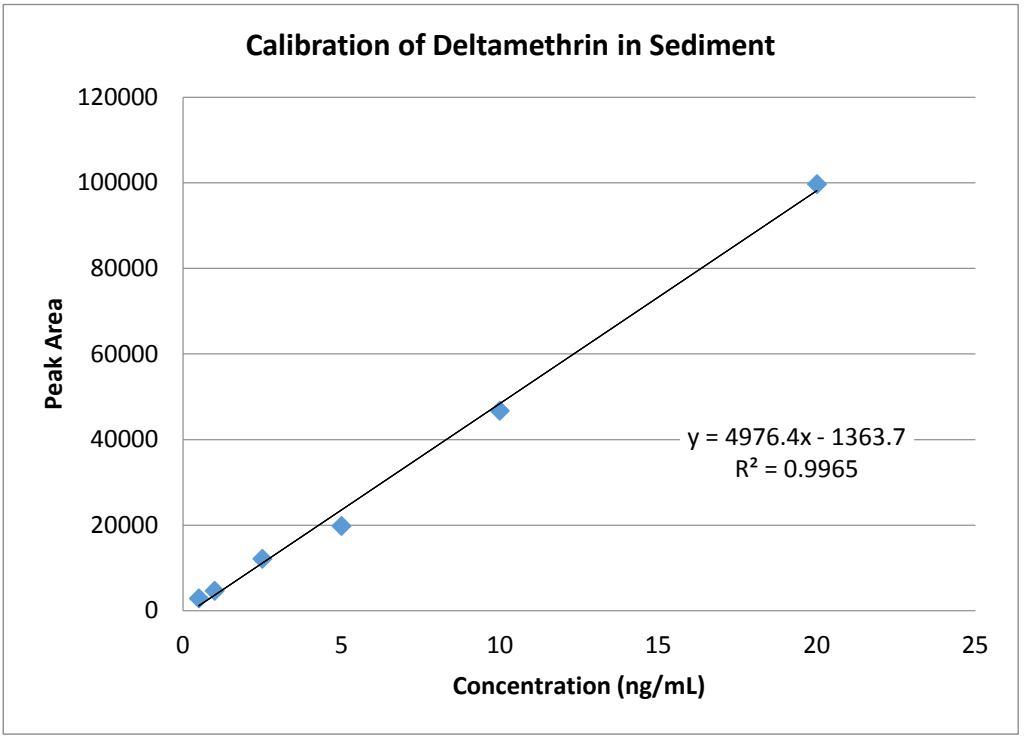

Calibration of Esfenvalerate in Sediment
Peak Area
700000
600000
500000
400000
300000
200000
100000
0
0
5
10
15
20
25
Concentration (ng/mL)
y = 31881x - 6724.9
R² = 0.9958

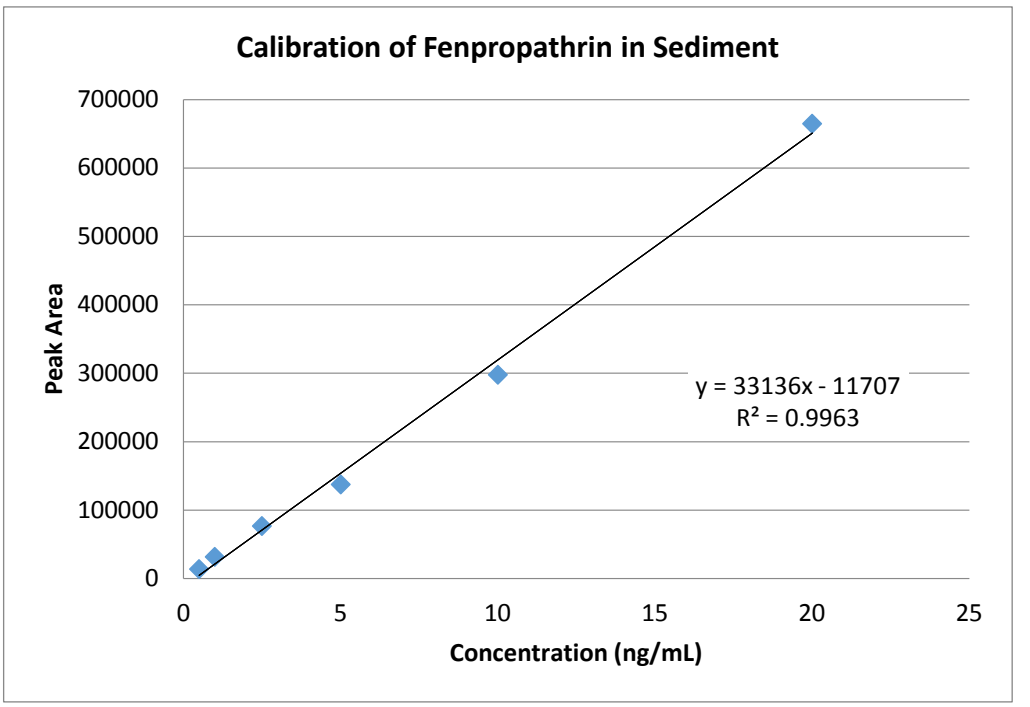

Calibration of Lambda-cyhalothrin in Sediment
500000
450000
400000
350000
300000
250000
200000
150000
100000
50000
0
Peak Area
y = 22524x - 9879.9
R² = 0.9872
0
5
10
15
20
25
Concentration (ng/mL)

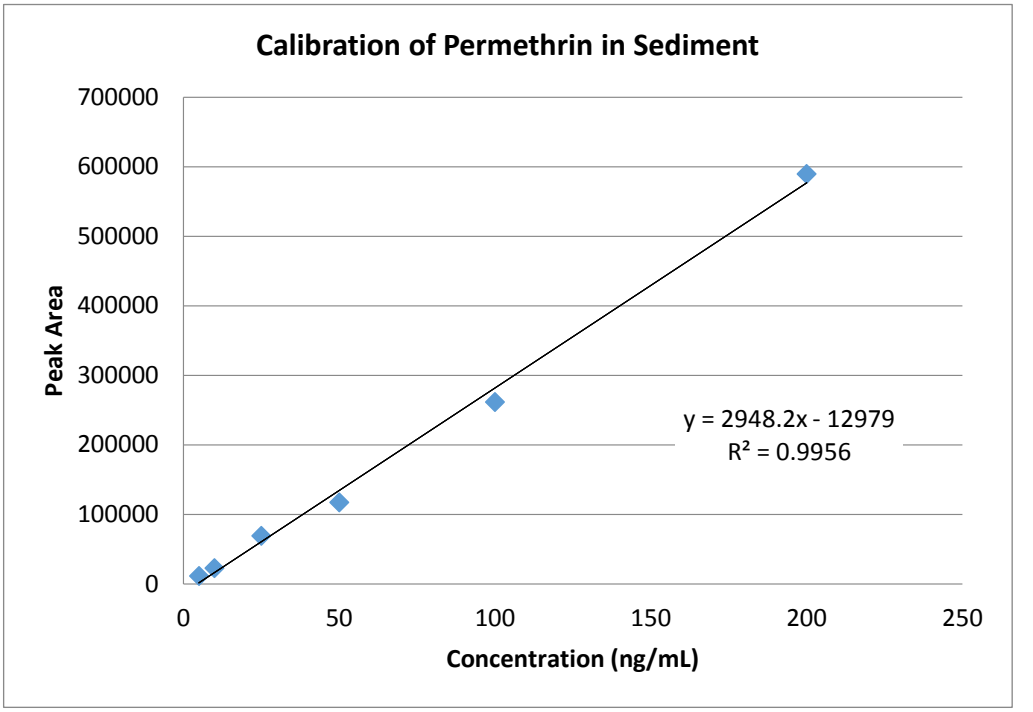
Esfenvalerate		Fenpropathrin		Lambda-cyhalothrin		Permethrin	
Conc. (ng/mL)	Peak response	Conc. (ng/mL)	Peak response	Conc. (ng/mL)	Peak response	Conc. (ng/mL)	Peak response
0.5	18317	0.5	13669	0.5	12073	5	11501
1.0	36819	1.0	31713	1.0	25935	10	22856
2.5	78284	2.5	76819	2.5	51491	25	69237
5.0	123292	5.0	137337	5.0	86567	50	117266
10.0	306969	10.0	297938	10.0	184652	100	261628
20.0	639321	20.0	664580	20.0	458421	200	589427

56; Figure 12, p. 58; Figure 14, p. 60; Figure 16, p. 62 of MRID 48935001.




Calibration of Deltamethrin in Sediment
120000
100000
80000
60000
40000
20000
0
Peak Area
0
5
10
15
20
25
Concentration (ng/mL)
y = 4976.4x - 1363.7
R² = 0.9965


Calibration of Fenpropathrin in Sediment
700000
600000
500000
400000
300000
200000
100000
0
Peak Area
0
5
10
15
20
25
Concentration (ng/mL)
y = 33136x - 11707
R² = 0.9963


Calibration of Permethrin in Sediment
700000
600000
500000
400000
300000
200000
100000
0
Peak Area
0
50
100
150
200
250
Concentration (ng/mL)
y = 2948.2x - 12979
R² = 0.9956

Chemical: Multiple Pyrethroids
MRID: 48935001
PC: 109701 -File
Guideline: 850.6100

ECM Validation for Determination of Multiple Pyrethroids in Sediment at the LOQ, Trial 2

Fortified (ng a.i.)	Recovery (ng)	Recovery (%)	Mean (%)	SD[1] (%)	RSD[2] (%)	Max	Min	n =
Bifenthrin								
0.1	0.0986	99						
	0.1444	144						
	0.0873	87						
	0.0966	97						
	0.0979	98	105	22.51	**21**	144	87	5
Fenpropathrin								
0.1	0.0959	96						
	0.1003	100						
	0.0999	100						
	0.1025	103						
	0.1009	101	100	2.45	2	103	96	5
***Lambda*-cyhalothrin**								
0.1	0.1312	131						
	0.1373	137						
	0.1331	133						
	0.1185	119						
	0.1122	112	**126**	10.62	8	137	112	5
Permethrin								
1.0	1.1843	118						
	0.9825	98						
	1.0335	103						
	1.1452	115						
	1.1498	115	110	10.49	10	118	98	5
Cyfluthrin								
0.1	0.1419	142						
	0.1357	136						
	0.1036	104						
	0.1002	100						
	0.1183	118	120	18.64	16	142	100	5
Cypermethrin								
0.1	0.1291	129						
	0.1306	131						
	0.0825	83						
	0.1062	106						
	0.1027	103	110	20.08	18	131	83	5
Esfenvalerate								
0.1	0.1314	131						
	0.1256	126						
	0.0981	98						
	0.1229	123						
	0.1120	112	118	13.17	11	131	98	5
Deltamethrin								
0.1	0.1100	110						
	0.0896	90						
	0.0939	94						
	0.1080	108						
	0.1053	105	101	9.06	9	110	90	5

Data taken from pp. 32, 34, 36, 38, 40, 42, 44, 46 of MRID 48935001.

1 SD = Standard Deviation; determined using the “unbiased” or “n-1” method.

2 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Multiple Pyrethroids
MRID: 48935001
PC: 109701 -File
Guideline: 850.6100

ECM Validation for Determination of Multiple Pyrethroids in Sediment at 10 x LOQ, Trial 2

Fortified (ng a.i.)	Recovery (ng)	Recovery (%)	Mean (%)	SD[1] (%)	RSD[2] (%)	Max	Min	n =
Bifenthrin								
1.0	0.9814	98						
	0.9108	91						
	0.8612	86						
	1.0894	109						
	0.9541	95	96	8.58	9	109	86	5
Fenpropathrin								
1.0	1.0981	110						
	1.0731	107						
	0.9772	98						
	1.2340	123						
	1.0891	109	109	9.18	8	123	98	5
***Lambda*-cyhalothrin**								
1.0	1.3567	136						
	1.1577	116						
	1.0833	108						
	1.3434	134						
	1.1603	116	**122**	12.25	10	136	108	5
Permethrin								
10.0	11.8430	118						
	12.5378	125						
	11.4691	115						
	14.6549	147						
	12.9759	130	**127**	5.42	4	147	115	5
Cyfluthrin								
1.0	1.0609	106						
	1.0540	105						
	0.9517	95						
	1.2487	125						
	1.0860	109	108	10.72	10	125	95	5
Cypermethrin								
1.0	1.0390	104						
	0.9318	93						
	1.0310	103						
	1.2903	129						
	1.1190	112	108	13.39	12	129	93	5
Esfenvalerate								
1.0	1.2396	124						
	1.0432	104						
	1.1070	111						
	1.3422	134						
	1.1962	120	119	11.62	10	134	104	5
Deltamethrin								
1.0	1.1367	114						
	1.1614	116						
	1.1269	113						
	1.3676	137						
	1.2014	120	120	9.86	8	137	113	5

Data taken from pp. 32, 34, 36, 38, 40, 42, 44, 46 of MRID 48935001.

1 SD = Standard Deviation; determined using the “unbiased” or “n-1” method.

2 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Attachment 3: Review of Environmental Chemistry Method

Pyrethroids; EPA PC Code 109701
Pyrethroid Working Group; EPA Company Code Consortium 64977

ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

Test Material: Multiple pyrethroids
Determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in sediment

EPA PC Code: 109701 (File)

Other PC Codes: 128825, 109702, 128831, 097805, 109303, 127901, 128897

OCSPP Guideline: 835.6200/Aquatic Field Dissipation; 850.7100/Data Reporting for Environmental Chemistry Methods

For Cambridge Environmental

Primary Reviewer: Lynne Binari **Signature:** Lynne Binari
Date: 11/09/2011

Secondary Reviewer: Kathleen Ferguson **Signature:** Kathleen P. Ferguson
Date: 11/09/2011

QC/QA Manager: Joan Gaidos **Signature:** Joan Gaidos
Date: 11/09/2011

USEPA/OPP Environmental Fate and Effects Division/Environmental Risk Branch 5

Final Reviewer: José L. Meléndez **Signature:** José Luis Meléndez
Chemist
Date: April 20, 2012

EPA MRID Numbers 47053001/47053002 (both same ECM)

ANALYTICAL METHOD: EPA MRID No. 47053001. Robinson, N. 2007. Residue analytical method for the determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in sediment. Report prepared by Syngenta Crop Protection, Inc., Jealott's Hill Research Centre, Bracknell, Berkshire, United Kingdom; sponsored and submitted by Pyrethroid Working Group (Consortium No. 64977), c/o Syngenta Crop Protection, Inc., Greensboro, North Carolina; 185 pages (pp. 1-3). Final report issued February 5, 2007.

ANALYTICAL METHOD: EPA MRID No. 47053002. Reed II, R. 2006. Laboratory validation: validation of the residue analytical method: "Residue analytical method for the determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in sediment". Unpublished study performed by Morse Laboratories, Inc., Sacramento, California; sponsored and submitted by Pyrethroid Working Group (Consortium No. 64977), c/o Syngenta Crop Protection, Inc., Greensboro, North Carolina; 418 pages (pp. 1-4). Morse Protocol No.: MLI-06-02 and Project No.: ML06-1286-PWG. Experimental start date April 21, 2006, and completion date May 18, 2006 (p. 7). Final report issued November 29, 2006.

INDEPENDENT LABORATORY VALIDATION: None provided.

EXECUTIVE SUMMARY

This method is designed for the quantitative determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in sediment using an external standardization method (p. 7 of MRID 47053001). The method was developed by Syngenta Crop Protection, Inc., and validated by Morse Laboratories, Inc. (p. 12 of MRID 47053002). An independent laboratory validation (ILV) was not submitted with this method.

The Agency finds that this study is supplemental. It meets the criteria for a scientifically valid method and partially satisfies the requirement for the analysis of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in two sediments. Deficiencies include that an independent laboratory validation of this method was not provided and the test sediments were incompletely characterized.

Method Summary: Analytes are extracted from sediment by shaking with methanol:water (1:1, v:v) and hexane, then the hexane phase, containing the recovered analytes, is cleaned up using silica solid phase extraction (Appendix 1, p. 203 of MRID 47053002). Bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin are quantified by GC/MS. The ECM utilized limits of quantitation (LOQ) and detection (LOD) of 0.1 µg/kg and 0.02 µg/kg, respectively, for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin and *lambda*-cyhalothrin, and 1.0 µg/kg and 0.2 µg/kg, respectively, for permethrin (p. 30 of MRID 47053002).

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

Isomerization of analytes has been found to occur with some GC instrumentation (p. 21 of MRID 47053001). In those instances, addition of 0.1% (v:v) acetic acid to all samples and standards was found to prevent isomerization; however, the reported results did not utilize this technique.

COMPLIANCE

This method was conducted in compliance with USEPA GLP Standards 40 CFR, Part 160 (p. 3 of MRID 47053002). Signed and dated statements of No Data Confidentiality, GLP and Quality Assurance were provided (pp. 2-3, 5 of MRID 47053002).

A. BACKGROUND INFORMATION

The pyrethroids share similar modes of action and are considered axonic poisons. It is now well established that severe neurological symptoms of poisoning with pyrethroids in mammals and insects are the result of modification of Na^+ channel activity (cellular pores through which sodium ions are permitted to enter the axon to cause excitation) (Matsamura, 1985).[1] Advanced electrophysiological experiments using voltage clamp and patch clamp, together with ligand binding and ionic flux experiments, have unveiled unique actions of pyrethroids of keeping the Na^+ channel in the open state for an extremely long period, sometimes as long as several seconds. This modification of Na^+ channel properties leads to hyperactivity of the nervous system. Pyrethroids have also been shown to suppress GABA (*gamma*-aminobutyric acid) and glutamate receptor-channel complexes and voltage-activated Ca^{2+} channels.

Relative to physiological responses, researchers have designated two types of pyrethroids, Type I (*e.g.*, bifenthrin and permethrin) and Type II (*e.g.*, cypermethrin,

[1] Matsumura, F. 1985. Toxicology of insects. 2nd ed. Plenum New York.

deltamethrin and fenvalerate). Structurally, Type I pyrethroids lack the *alpha*-cyano group that characterizes Type II pyrethroids. Physiologically, Type I pyrethroids typically have shorter periods of sodium channel disruption (shorter inactivation time) than that of Type II pyrethroids.

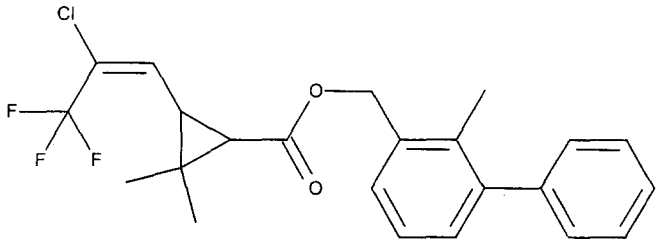
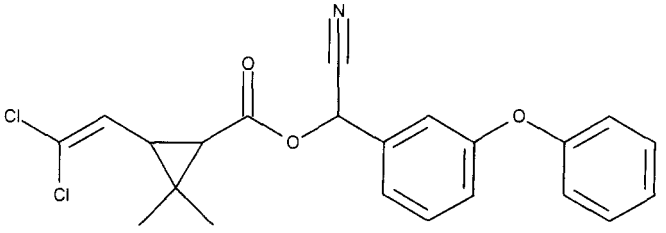
TABLE A.1. Test Compound Nomenclature	
Parameter	**Value**
Common name	**Bifenthrin.**
Company experimental name	Not reported.
IUPAC name	2-Methylbiphenyl-3-ylmethyl (Z)-(1RS,3RS)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate.
CAS Name	(2-Methyl[1,1'-biphenyl]-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate.
CAS #	82657-04-3.
Structure	
Common name	**Cypermethrin.**
Company experimental name	Not reported.
IUPAC name	(RS)-α-Cyano-3-phenoxybenzyl (1RS,3RS;1RS,3SR)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate.
CAS Name	Cyano(3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate.
CAS #	52315-07-8.
Structure	
Common name	**Cyfluthrin.**
Company experimental name	Not reported.
IUPAC name	(RS)-α-Cyano-4-fluoro-3-phenoxybenzyl (1RS,3RS;1RS,3SR)-3-

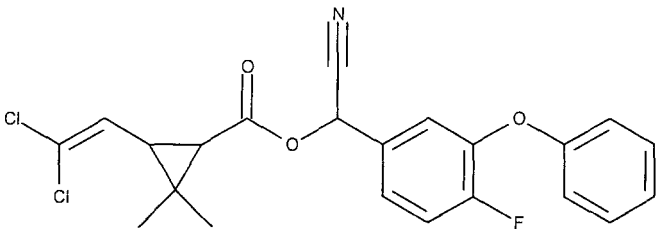
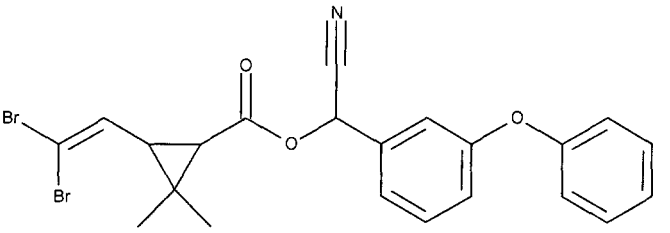
TABLE A.1. Test Compound Nomenclature	
Parameter	**Value**
	(2,2-dichlorovinyl)-2,2-dimethylcyclopropane carboxylate.
CAS Name	Cyano(4-fluoro-3-phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropancecarboxylate.
CAS #	68359-37-5.
Structure	
Common name	**Deltamethrin.**
Company experimental name	Not reported.
IUPAC name	(S)-α-Cyano-3-phenyoxybenzyl (1R,3R)-3-(2,2-dibromovinyl)-2,2-dimethylcyclopropanecarboxylate.
CAS Name	1-[R-[1-α-(S*),3α]]-Cyano(3-phenoxyphenyl)methyl 3-(2,2-dibromoethenyl)-2,2-dimethylcyclopropanecarboxylate.
CAS #	52918-63-5.
Structure	
Common name	**Esfenvalerate.**
Company experimental name	Not reported.
IUPAC name	(S)-α-Cyano-3-phenoxybenzyl (S)-2-(4-chlorophenyl)-3-methylbutyrate.
CAS Name	[S-(R*,R*)]-Cyano(3-phenoxyphenyl)methyl 4-chloro-2-(1-methylethyl)benzeneacetate.
CAS #	66230-04-4.

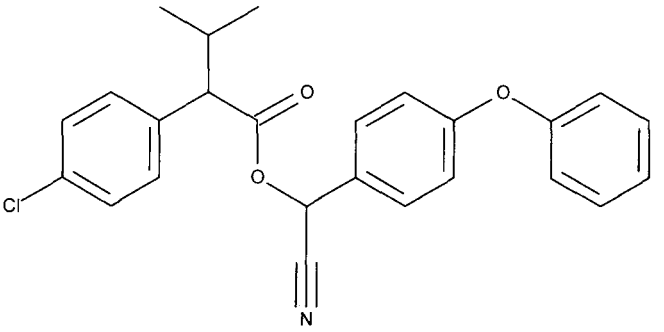
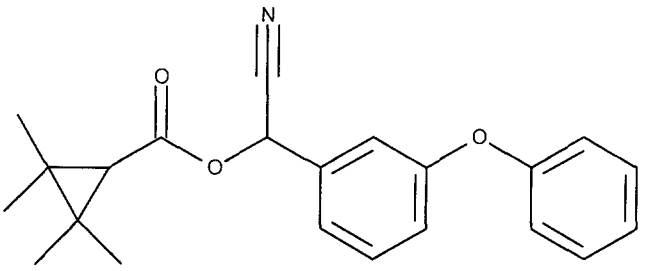
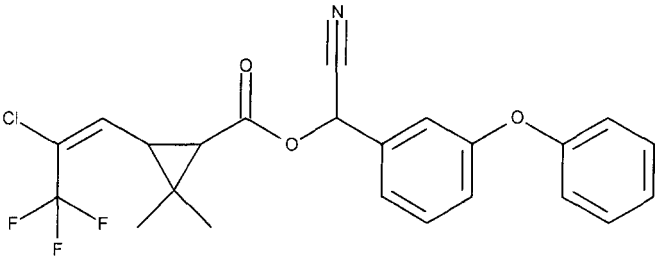
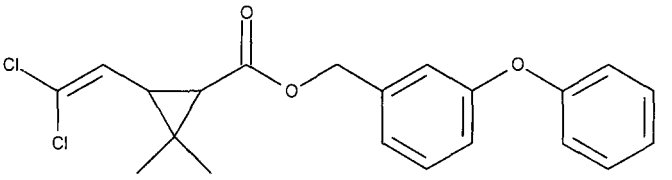
TABLE A.1. Test Compound Nomenclature	
Parameter	**Value**
Structure	
Common name	**Fenpropathrin.**
Company experimental name	Not reported.
IUPAC name	(RS)-α-Cyano-3-phenoxybenzyl 2,2,3,3,-tetramethylcyclopropancecarboxylate.
CAS Name	Cyano(3-phenoxyphenyl)methyl 2,2,3,3-tetramethylcyclopropanecarboxylate.
CAS #	64257-84-7.
Structure	
Common name	***Lambda*-cyhalothrin.**
Company experimental name	Not reported.
IUPAC name	Reaction product of equal quantities of (S)- and (R)- α-cyano-3-phenoxybenzyl (Z)-(1R,3R)-3-(2-chloro-3,3,3-trifluoroprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate.
CAS Name	[1α(S*),3α(Z)]-(±)-Cyano(3-phenoxyphenyl)methyl 3-(2-chloro-3,3,3-trifluoro-1-propenyl)-2,2-dimethylcyclopropanecarboxylate.
CAS #	91465-08-6.

TABLE A.1. Test Compound Nomenclature	
Parameter	**Value**
Structure	
Common name	**Permethrin.**
Company experimental name	Not reported.
IUPAC name	3-Phenoxybenzyl (1RS,3RS;1RS,3SR)-3-(2,2-dichlorovinyl)-2,2-dimethylcyclopropanecarboxylate.
CAS Name	(3-Phenoxyphenyl)methyl 3-(2,2-dichloroethenyl)-2,2-dimethylcyclopropanecarboxylate.
CAS #	52645-53-1.
Structure	

Information obtained from pp. 15-18 of MRID 47053002.

TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound	
Parameter	**Value**
Melting point/range (°C)	Not reported.
pH	Not reported.
Density (g/cm^3)	Not reported.
Water solubility at 20 °C (mg/L)	Not reported.
Solvent solubility at 20 °C (mg/L)	Not reported.
Vapor pressure at 25°C (torr)	Not reported.
Dissociation constant (pK_a)	Not reported.
Octanol/water partition coefficient	Not reported.
UV/visible absorption spectrum (nm)	Not reported.

B. MATERIALS AND METHODS

B.1. Principle of Method

Analytes are extracted from sediment by mechanical shaking with methanol:water (1:1, v:v) and hexane (pp. 23-24; Appendix 1, pp. 203-205 of MRID 47053002). The hexane phase, containing the recovered analytes, is cleaned up using silica solid phase extraction (SPE). Analytes are separated and quantified by GC/MS using a Varian CP-Sil 8CB-MS column, negative ion chemical ionization (NICI) and selected ion monitoring (SIM). A confirmatory method was not utilized.

TABLE B.1. Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied

Parameter	Value
Method ID	Residue analytical method for the determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in sediment (p. 23; Appendix 1, p. 194 of MRID 47053002).
Analyte(s)	Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-cyhalothrin and Permethrin.
Extraction solvent/technique	Sediment (50 g) is extracted with methanol: water (1:1, v:v, 75 mL) and hexane (50 mL) via mechanical shaking for 60 minutes (pp. 22-23; Appendix 1, p. 203 of MRID 47053002). Sample is then centrifuged to disperse emulsions and separate extract phases from sediment.
Cleanup strategies	An aliquot of the hexane phase, equivalent to 10 g sediment, is taken to dryness at 40°C under an air stream (Appendix 1, pp. 203-204 of MRID 47053002). Residues are reconstituted in hexane and applied to a Varian Silica Bond Elut SPE cartridge. Analytes are eluted with hexane: diethyl ether (9:1, v:v), the eluate is taken to dryness as described above and resulting residues reconstituted in acetone containing 0.1% (v:v) peanut oil.
Instrument/Detector	Agilent 6890 GC system equipped with a Varian CP-Sil 8CB-MS column (0.25 mm x 30 m, 0.25 µm film thickness, 95% dimethylpolysiloxane:5% diphenyl) and Agilent 5973N MS system with NICI and SIM (p. 23; Appendix 1, p. 204 of MRID 47053002).

Information obtained from pp. 22-23; Appendix 1, pp. 194, 203-204 of MRID 47053002.

C. RESULTS AND DISCUSSION

C.1. Recovery Results Summary

TABLE C.1. Recovery Results from Method Validation for the Determination of Residues in Two Sediments

Analyte	Spiking Level (µg/kg)	Mean Recoveries Obtained (%)	Relative Standard Deviation
California Fresh Water Sediment (TOC 1.31%)[1,2]			
Bifenthrin	0.1	106	2.1
	1.0	107	5.8
Cypermethrin	0.1	106	2.9
	1.0	118	8.4
Cyfluthrin	0.1	106	2.6
	1.0	117	6.5
Deltamethrin	0.1	88	10
	1.0	108	6.1
Esfenvalerate	0.1	78	5.7
	1.0	112	8.4
Fenpropathrin	0.1	104	5.7
	1.0	113	3.3
Lambda-cyhalothrin	0.1	93	11
	1.0	112	6.6
Permethrin	1.0	100	5.2
	10.0	108	12
California Estuarine Sediment (TOC 0.86%)[1,3]			
Bifenthrin	0.1	91	2.8
	1.0	99	5.2
Cypermethrin	0.1	103	7.8
	1.0	108	4.5
Cyfluthrin	0.1	99	8.7
	1.0	106	5.5
Deltamethrin	0.1	85	5.0
	1.0	85	13
Esfenvalerate	0.1	109	7.0
	1.0	105	6.4
Fenpropathrin	0.1	105	5.9
	1.0	105	6.0
Lambda-cyhalothrin	0.1	101	6.5
	1.0	104	5.4
Permethrin	1.0	106	5.4
	10.0	104	5.7
Sediments Combined[4]			
Bifenthrin	0.1	99	8.7
	1.0	103	6.8
	All data	101	7.9

TABLE C.1. Recovery Results from Method Validation for the Determination of Residues in Two Sediments

Analyte	Spiking Level (µg/kg)	Mean Recoveries Obtained (%)	Relative Standard Deviation
Cypermethrin	0.1	104	5.8
	1.0	113	7.8
	All data	109	8.0
Cyfluthrin	0.1	102	6.8
	1.0	111	7.9
	All data	107	8.4
Deltamethrin	0.1	86	7.8
	1.0	97	15
	All data	91	14
Esfenvalerate	0.1	94	19
	1.0	108	7.9
	All data	101	15
Fenpropathrin	0.1	104	5.6
	1.0	109	5.9
	All data	107	6.1
Lambda-cyhalothrin	0.1	97	9.7
	1.0	108	7.2
	All data	102	9.9
Permethrin	1.0	103	5.9
	10.0	106	9.1
	All data	104	7.7

1 Sediments not further characterized (p. 29; Appendix 5, pp. 417-418 of MRID 47053002). Freshwater sediment obtained from Butte Creek, Butte County, California; estuarine sediment obtained from Paradise Cove, Marin County California (Appendix 2, pp. 387, 392 of MRID 47053002).
2 Results from Table 1, pp. 33-34 of MRID 47053002.
3 Results from Table 2, pp. 35-36 of MRID 47053002.
4 Results determined by primary reviewer using data obtained from Tables 1-2, pp. 33-36 of MRID 47053002 (DER Attachment 2).

C.1.1. Method Characteristics

TABLE C.2. Method Characteristics

Parameter	Value
Analyte(s)	Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-cyhalothrin, Permethrin (p. 12 of MRID 47053002).
Limit of Quantitation (LOQ)	0.1 µg/kg for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin and *lambda*-cyhalothrin (p. 30 of MRID 47053002).

TABLE C.2. Method Characteristics	
	1.0 µg/kg for permethrin.
Limit of Detection (LOD)	0.02 µg/kg for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin and *lambda*-cyhalothrin (p. 30 of MRID 47053002). 0.2 µg/kg for permethrin.
Accuracy/Precision at LOQ	Acceptance criteria (EFED-ECM 2, Version 1, December 2010, p. 5) were met at the LOQ for all analytes with matrix spike recoveries ranging between 70% to 120% and relative standard deviations of ≤20% (Tables 1-2, pp. 33-36 of MRID 47053002).
Reliability of the Method/[ILV]	An ILV of the ECM was not provided.
Linearity	Linear regression; range r = 0.9998-0.9999 (Figure 22, pp. 185-192 of MRID 47053002).
Specificity	Mean residues were detected at >30% of the LOQ in the matrix blank control samples for deltamethrin, esfenvalerate and *lambda*-cyhalothrin in the fresh water sediment, and for bifenthrin and deltamethrin in the estuarine sediment (p. 30 of MRID 47053002). For bifenthrin in the fresh water sediment, for esfenvalerate and *lambda*-cyhalothrin in the estuarine sediment, and for cypermethrin, cyfluthrin, fenpropathrin and permethrin in both sediments, any detections in the matrix blank controls were <30% of the LOQ (Tables 1-2, pp. 33-36 of MRID 47053002). Reported recoveries for fortified samples were corrected for mean residues detected in the control samples.

Information obtained from pp. 12, 30; Tables 1-2, pp. 33-36; Figure 22, pp. 185-192 of MRID 47053002.

C.2. Independent Laboratory Validation (ILV)

An ILV of the ECM was not provided.

TABLE C.3. Recovery Results of the Method Obtained by an Independent Laboratory Validation for the Determination of Residues in [*Matrix*]			
Analyte	**Spiking Level (units)**	**Mean Recoveries Obtained (%)**	**Relative Standard Deviation**
An Independent Laboratory Validation (ILV) was not conducted.			

D. CONCLUSION

This environmental chemistry method (ECM) is designed for the quantitative determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin. The Agency finds that this study is supplemental. The ECM meets the criteria for a scientifically valid method and partially satisfies the data requirement for residues of eight syntethic pyrethroids in two sediments, given the following information is provided and found acceptable.

- **An independent laboratory validation (ILV) report for the ECM, performed by scientists differing from those who developed the original ECM,**
- Test sediments characterization, and
- Further information regarding how the LOD and LOQ were determined.

MRIDs 47053001 and 47053002 were submitted concurrently. Upon initial review, MRID 47053001 appeared to be the ECM, with MRID 47053002 the supporting Independent Laboratory Validation (ILV). However, comparison of data found that the two MRIDs contained the same results. MRID 47053002 is the ECM as performed by Morse Laboratories, Inc. MRID 47053001 presents all the same data generated by Morse Labs, but with all references to Morse Labs, including Morse Protocol and Laboratory Project Numbers, removed. The method presented in MRID 47053001 does include a section addressing the potential problem of analyte isomerization that was not included as part of the method description in MRID 47053002.

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

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

Referenced page numbers are from MRID 47053002, except where noted otherwise. Cited pages appear in the bottom most right corner of each page for both MRIDs.

I. Background Information

A.	**Title of Method**	Residue analytical method for the determination of residues of bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin and permethrin in sediment (p. 12).	
B.	**ECM No.** [BEAD]		
C.	**MRID No.**	47053001 and 47053002: These documents were submitted concurrently. Upon initial review, MRID 47053001 appeared to be the ECM, with MRID 47053002 the supporting ILV. However, comparison of data found that the two MRIDs contained the same results. MRID 47053002 is the ECM as performed by Morse Laboratories, Inc. MRID 47053001 presents all the same data generated by Morse Labs, but with all references to Morse Laboratories, including Morse Protocol and Laboratory Project Numbers, removed. MRID 47053001 lists the study "Performer" as Syngenta Crop Protection, Inc., Jealott's Hill Research Centre. MRID 47053002 lists Syngenta Crop Protection, Inc., Jealott's Hill Research Centre as the study monitor (p. 6; Appendix 2, p. 377). The method presented in MRID 47053001 includes a section addressing the potential problem of analyte isomerization (see section ***IV. Detailed Information about the Method* C. 8. a** below) that was not included as part of the method description in MRID 47053002.	
D.	**Matrix**	Sediment.	
E.	**Analyte(s) detected**	**Compounds:**	Bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, *lambda*-cyhalothrin, permethrin

Information obtained from pp. 12, 15-18 of MRID 47053002. For structures, see the review report.

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II. Information about the Laboratory

A.	**Name**	Morse Laboratories, Inc. (p. 1).
B.	**Address**	1525 Fulton Avenue, Sacramento, California, 95825.
C.	**Telephone No.**	Not reported.
D.	**Name of the Study Director**	Richard L. Reed II (p. 7).
E.	**Name of the Lead Chemist**	Kevin Clark, Chief GC Chemist (p. 7).
F.	**Laboratory Validation:**	Yes, at LOQ and 10 x LOQ (p. 12).

Information obtained from pp. 1, 7, 12 of MRID 47053002.

III. Method Summary Information for Analyte(s): **Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-cyhalothrin and Permethrin.**

A.	**Statement of Data Confidentiality**	Yes (p. 2).
1.	**Is the Method Classified or Confidential?**	No.
2.	**Submitted Prior to 2008 with a Non-Standard Claim of Confidentiality?**	No.
B.	**Sample Preparation**	Rocks and plant debris removed manually (p. 21). Thoroughly mix sediment to homogenize prior to aliquot removal (p. 23; Appendix 1, p. 202). At each fortification level (LOQ, 10 x LOQ), samples were fortified with all eight analytes using a mixed standard solution (p. 22).
C.	**Sample Extraction**	Sediment aliquot (50 g) weighed into centrifuge bottle; add 75 mL methanol: water (1:1, v:v) and 50 mL hexane; shake on mechanical shaker for 60 minutes (p. 23; Appendix 1, p. 203). Centrifuge sample at speed to disperse emulsions and separate extract phases from sediment (*e.g.* 4,000 rpm, 5 minutes).

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D.	**Sample Cleanup**	An aliquot (10 mL) of the hexane phase, equivalent to 10 g sediment, is taken to dryness at 40°C under an air stream (Appendix 1, p. 203). Residues are reconstituted in hexane (2 mL) and applied to a solid phase extraction cartridge (Varian Silica Bond Elut, 500 mg, 3 mL). Analytes are eluted with hexane: diethyl ether (9:1, v:v, 6 mL), the eluate is taken to dryness as described above and resulting residues reconstituted in acetone containing 0.1% (v:v) peanut oil (1 mL).
E.	**Sample Derivatization**	Not applicable.
F.	**Sample Analysis**	GC with mass selective detection using negative ion chemical ionization (GC-MS/NICI, GC-MSD; pp. 23-25; Appendix 1, pp. 204-206).
1.	**Instrumentation**	Agilent 6890 GC system with split/splitless injector and Agilent 5973N MS system using negative chemical ionization mode (p. 23; Appendix 1, p. 204).
2.	**Primary Column**	Varian CP-Sil 8CB-MS column (0.25 mm x 30 m, 0.25 µm film thickness, 95% dimethylpolysiloxane: 5% diphenyl; p. 23; Appendix 1, p. 204).
3.	**Confirmatory Column**	None reported.
4.	**Detector**	Selected Ion Monitoring (SIM; p. 24; Appendix 1, p. 205).
5.	**Other Confirmatory Techniques**	In addition to the retention time and the target ion, one qualifier ion was listed for esfenvalerate and permethrin, and two qualifier ions for bifenthrin, cyfluthrin, cypermethrin, deltamethrin and *lambda*-cyhalothrin; however, no results for the qualifier ions were reported (p. 24). Only the target ion was listed for fenpropathrin.
6.	**Other Relevant Information**	

Compound	Ion monitored (m/z)	Retention time(s) (minutes)[1]
Bifenthrin	386	18.1
Fenpropathrin	141	18.5
Lambda-cyhalothrin	205	19.6 19.9
Permethrin	207	21.5 21.8

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

		Cyfluthrin	207	22.5 22.7 22.8 22.9
		Cypermethrin	207	23.2 23.4 23.5 23.6
		Esfenvalerate	211	24.9 25.3
		Deltamethrin	297	25.9 26.3
G.	**Detection and Quantitation Limits**			
1.	**Limit of Quantitation (LOQ)**			
	Claimed in Method	0.1 µg/kg for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, and *lambda*-cyhalothrin (p. 30). 1.0 µg/kg for permethrin.	**Estimated**	No. LOQ defined as lowest concentration of analyte yielding mean recovery of 70-110% with a relative standard deviation of ≤20% (Appendix 1, p. 210).
2.	**Limit of Detection (LOD)**			
	Claimed in Method	0.02 µg/kg for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin, and *lambda*-cyhalothrin (p. 30). 0.2 µg/kg for permethrin.	**Estimated**	Yes. LOD defined as lowest concentration of analyte detectable above mean amplitude of background noise in an untreated sample at the corresponding retention time; estimated at three times background noise (Appendix 1, p. 210).

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H.	Recovery (Accuracy) /Precision Data; percent recovery (mean, SD, RSD, n = 5)[2]									
	Level[3]	Cmpd[4]	Bifen-	Cyper-	Cyflu-	Delta-	Esfen-	Fenpro-	*Lambda-*	Per-
	California Fresh Water Sediment (TOC 1.31%)[5]									
	LOQ	Range	103-109	102-110	101-108	78-102	73-84	94-108	83-108	94-108
		Mean	106	106	106	88	78	104	93	100
		SD	2.2	3.0	2.8	8.9	4.5	5.9	10	5.2
		RSD	2.1	2.9	2.6	10	5.7	5.7	11	5.2
	10 x LOQ	Range	100-115	105-130	108-127	99-116	99-122	108-116	104-121	93-124
		Mean	107	118	117	108	112	113	112	108
		SD	6.3	9.9	7.6	6.5	9.4	3.7	7.4	13
		RSD	5.8	8.4	6.5	6.1	8.4	3.3	6.6	12
	California Estuarine Sediment (TOC 0.86%)[5]									
	LOQ	Range	87-94	96-114	89-108	78-89	97-118	95-111	90-106	98-111
		Mean	91	103	99	85	109	105	101	106
		SD	2.5	8.0	8.6	4.2	7.7	6.2	6.5	5.7
		RSD	2.8	7.8	8.7	5.0	7.0	5.9	6.5	5.4
	10 x LOQ	Range	93-106	102-114	98-113	74-97	95-112	98-114	97-111	96-111
		Mean	99	108	106	85	105	105	104	104
		SD	5.1	4.8	5.8	11	6.7	6.3	5.6	5.9
		RSD	5.2	4.5	5.5	13	6.4	6.0	5.4	5.7

Information obtained from pp. 2, 21-25, 29-30; Tables 1-2, pp. 33-36; Appendix 1, pp. 202-206, 210; Appendix 5, pp. 417-418 of MRID 47053002.

1 Multiple retention times are for individual isomer peaks of the pyrethroid analyte; peak areas of each isomer were combined to determine total residue value (p. 25; Appendix 1, p. 206 of MRID 47053002).

2 Results from Tables 1-2, pp. 33-36 of MRID 47053002; verified by primary reviewer (DER Attachment 2).

3 LOQ and 10 x LOQ 0.1 and 1.0 µg/kg, respectively, for bifenthrin, cypermethrin, cyfluthrin, deltamethrin, esfenvalerate, fenpropathrin and *lambda*-cyhalothrin, and 1.0 and 10.0 µg/kg, respectively, for permethrin.

4 Bifenthrin, Cypermethrin, Cyfluthrin, Deltamethrin, Esfenvalerate, Fenpropathrin, *Lambda*-cyhalothrin and Permethrin, respectively.

5 Sediments not further characterized (p. 29; Appendix 5, pp. 417-418 of MRID 47053002). Freshwater sediment obtained from Butte Creek, Butte County, California; estuarine sediment obtained from Paradise Cove, Marin County, California (Appendix 2, pp. 387, 392 of MRID 47053002).

IV. Detailed Information about the Method

		YES	NO	REVIEW FURTHER
A.	**Does the method require spiking with the analytes of interest?**		x	pp. 21-22.

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		YES	NO	REVIEW FURTHER
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. 21; Appendix 1, p. 202.
b.	**The sample spiking procedure?**		x	p. 22 fortification of sediments
c.	**The extraction procedure?**	x		p. 23; Appendix 1, p. 203.
d.	**The derivatization procedure?**			Not applicable.
e.	**The clean-up procedure?**	x		p. 23; Appendix 1, p. 203.
f.	**The analysis procedure?**	x		pp. 23-25.
2.	**Procedures for:**			
a.	**Preparation of standards?**	x		pp. 20-21.
b.	**Calibration of instrument?**	x		p. 22.
3.	**List of glassware and chemicals**	x		Appendix 1, pp. 212-213.
a.	**Are sources recommended?**	x		
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**			
a.	**Is there an explanation of how it was calculated?**	x		Appendix 1, p. 210.
b.	**Is it a scientifically accepted procedure?**			Not determined statistically (p. 30).

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

		YES	NO	REVIEW FURTHER
c.	**Is the matrix blank free of interference at the retention time, wavelength, *etc.*, of the analytes of interest?**	Freshwater sediment: bifenthrin, cypermethrin, cyfluthrin, fenpropathrin, permethrin (Table 1, pp. 33-34). Estuarine sediment: fenpropathrin, permethrin (Table 2, pp. 35-36).	Freshwater sediment: deltamethrin, esfenvalerate and *lambda*-cyhalothrin. Estuarine sediment: bifenthrin, cyfluthrin, deltamethrin, esfenvalerate and *lambda*-cyhalothrin.	In five instances mean residues were detected at >30% of the LOQ; 0.0453, 0.0655 and 0.0414 µg/kg for deltamethrin, esfenvalerate and *lambda*-cyhalothrin, respectively, in FWS, and 0.0469 and 0.0720 µg/kg for bifenthrin and deltamethrin, respectively, in ES (p. 30). Reported recoveries for fortified samples were corrected for mean residues detected in control samples.
6.	**LOQ**			
a.	**Is there an explanation of how it was calculated?**	x		Appendix 1, p. 210.
b.	**Is it a scientifically accepted procedure?**			Not determined statistically (p. 30).
7.	**Precision and accuracy data**			
a.	**Were there an adequate number of spiked samples analyzed?**	x		Five replicates each at LOQ and 10 x LOQ (p. 22).
b.	**Are the mean recoveries between 70-120%?**	x		Tables 1-2, pp. 33-36.
c.	**Are the RSDs of the replicates 20% or less at or above the LOQ?**	x		

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

		YES	NO	REVIEW FURTHER
8.	**Description and/or explanation of:**			
a.	**Areas where problems may be encountered?**	x		Addition of 0.1% (v:v) acetic acid to all samples and standards may be required to prevent isomerization (p. 21 of MRID 47053001).
b.	**Critical steps?**	x		
c.	**Interferences that may be encountered?**	x		Disposable labware is used to prevent any cross contamination (Appendix 1, p. 209).
9.	**Characterization of the Matrices?**		Incomplete	Only total organic carbon reported (pp. 19, 29).

Information obtained from pp. 19-25, 29-30; Tables 1-2, pp. 33-36; Appendix 1, pp. 202-203, 209-210, 212-213 of MRID 47053002; and p. 21 of MRID 47053001.

V. Representative Chromatograms

		YES	NO	REVIEW FURTHER
A.	**Are there representative chromatograms for:**			
1.	**Analytes in each matrix at the LOQ and 10 x LOQ?**	x		Figure 4, pp. 59-65; Figure 7, pp. 80-86; Figure 12, pp. 115-121; Figure 15, pp. 136-142.
2.	**Method blanks?**		x	Reagent blanks were analyzed with no residues detected at the retention times of the pyrethroid analytes (Tables 1-2, pp. 33-36).

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

		YES	NO	REVIEW FURTHER
3.	**Matrix blanks?**	x		Figure 2, pp. 45-51; Figure 10, pp. 101-107.
4.	**Standard curves?**	x		Figure 22 (pp. 185-192).
a.	**Do the standard curves have acceptable linearity?**	x		r = 0.9998-0.9999).
5.	**Standards that can be used to recalculate some of the values for analytes in the sample chromatograms?**	x		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 Tables 1-2, pp. 33-36; Figure 2, pp. 45-51; Figure 4, pp. 59-65; Figure 8, pp. 80-86; Figure 10, pp. 101-107; Figure 12, pp. 115-121; Figure 15, pp. 136-142; Figure 22, pp. 185-192 of MRID 47053002.

VI. Good Laboratory Practice (GLP) Standards

		YES	NO	REVIEW FURTHER
A.	**Is there a statement of adherence to the FIFRA GLP standards?**	x		p. 3.

Information obtained p. 3 of MRID 47053002.

VII. Independent Lab Validation (ILV)

		YES	NO	REVIEW FURTHER
A.	**Was an ILV performed?**		x	None provided.
B.	**Was the validation independent?**			None provided.

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

		YES	NO	REVIEW FURTHER
C.	**Did the ILV's precision/accuracy data meet the criteria established in OPPTS Guideline 850.7100?**			None provided.
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?**			None provided.

VIII. Completeness

		YES	NO	REVIEW FURTHER
A.	**Has enough information been supplied to do a proper review?**		x	ILV required.
B.	**Has enough information been supplied to do a laboratory evaluation, if requested?** [BEAD]			
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.** [*Is the data supplied in the method package satisfactory or deficient in any way? If there are deficiencies, are the deficiencies major or minor? Note whether deficiencies are with the method procedure, whether they are with respect to guidelines, and whether they affect the review classification.*]			
1.	**Satisfactory**		x	
2.	**Major Deficiencies**	x		ILV required.

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

		YES	NO	REVIEW FURTHER
3.	**Minor Deficiencies**	x		Confirmatory method not employed; LOD and LOQ were not determined statistically.

IX. Recommendations

- Section ***VIII. Completeness*** **E. 2. Major Deficiencies.** An independent laboratory validation (ILV) report for the ECM, performed by scientists differing from those who developed the original ECM, was not submitted with this data package and should be submitted.

- Section ***VIII. Completeness*** **E. 3. Other Deficiencies.** A confirmatory method was not provided to verify the identities of the pyrethroid analytes, the test sediments were incompletely characterized, and the LOD and LOQ were not statistically determined.

Final Reviewer: José L. Meléndez **Signature:** José Luis Meléndez
Chemist

USEPA/OPP/EFED/ERB5 **Date:** April 20, 2012

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Bifenthrin in two California sediments.[1]

Fortified (µg a.i./kg)	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0.1	0	0.103	0.103	103				0.0469	0.138	0.091	91			
		0.106	0.106	106					0.141	0.094	94			
		0.107	0.107	107					0.137	0.090	90			
		0.107	0.107	107					0.138	0.091	91			
		0.109	0.109	109	106	2.2	2.1		0.134	0.087	87	91	2.5	2.8
1.0		0.997	0.997	100					0.979	0.932	93			
		1.11	1.11	111					1.03	0.983	98			
		1.09	1.09	109					1.11	1.06	106			
		1.02	1.02	102					1.06	1.01	101			
		1.15	1.15	115	107	6.4	5.9		1.00	0.953	95	99	5.2	5.2
Overall mean				107							95			
SD				4.5							5.8			
RSD				4.2							6.1			
Max				115							106			
Min				100							87			
n =				10							10			

Soils combined:	
Overall mean	101
SD	8.0
RSD	7.9
Max	115
Min	87
n =	20

0.1 µg a.i./kg fortifications combined:			
Overall mean			99
SD			8.6
RSD			8.7
Max			109
Min			87
n =			10

1.0 µg a.i./kg fortifications combined:			
Overall mean			103
SD			7.0
RSD			6.8
Max			115
Min			93
n =			10

Results from Table 1, p. 33; Table 2, p. 35 of MRID 47053002.
Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).
1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.
2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.
3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Cypermethrin in two California sediments.[1]

Fortified (µg a.i./kg)	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0.1	0	0.107	0.107	107				0	0.0979	0.098	98			
		0.102	0.102	102					0.114	0.114	114			
		0.105	0.105	105					0.108	0.108	108			
		0.104	0.104	104					0.0955	0.096	96			
		0.110	0.110	110	106	3.0	2.9		0.0970	0.097	97	102	8.1	7.9
1.0		1.11	1.11	111					1.02	1.02	102			
		1.30	1.30	130					1.10	1.10	110			
		1.20	1.20	120					1.14	1.14	114			
		1.05	1.05	105					1.11	1.11	111			
		1.23	1.23	123	118	9.9	8.4		1.05	1.05	105	108	4.8	4.5
Overall mean				112							105			
SD				9.4							7.0			
RSD				8.4							6.7			
Max				130							114			
Min				102							96			
n =				10							10			

Soils combined:	
Overall mean	109
SD	8.7
RSD	8.0
Max	130
Min	96
n =	20

0.1 µg a.i./kg fortifications combined:			
Overall mean			104
SD			6.0
RSD			5.8
Max			114
Min			96
n =			10

1.0 µg a.i./kg fortifications combined:			
Overall mean			113
SD			8.8
RSD			7.8
Max			130
Min			102
n =			10

Results from Table 1, p. 33; Table 2, p. 35 of MRID 47053002.

Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).

1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.

2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.

3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Cyfluthrin in two California sediments.[1]

Fortified (µg a.i./kg)	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0.1	0	0.107	0.107	107				0.0117	0.101	0.089	89			
		0.105	0.105	105					0.120	0.108	108			
		0.107	0.107	107					0.104	0.092	92			
		0.101	0.101	101					0.119	0.107	107			
		0.108	0.108	108	106	2.8	2.6		0.110	0.098	98	99	8.6	8.7
1.0		1.11	1.11	111					0.992	0.980	98			
		1.27	1.27	127					1.08	1.07	107			
		1.19	1.19	119					1.14	1.13	113			
		1.08	1.08	108					1.09	1.08	108			
		1.20	1.20	120	117	7.6	6.5		1.03	1.02	102	105	5.7	5.4
Overall mean				111							102			
SD				8.1							7.6			
RSD				7.3							7.5			
Max				127							113			
Min				101							89			
n =				10							10			

Soils combined:	
Overall mean	107
SD	8.9
RSD	8.4
Max	127
Min	89
n =	20

0.1 µg a.i./kg fortifications combined:			
Overall mean			102
SD			6.9
RSD			6.8
Max			108
Min			89
n =			10

1.0 µg a.i./kg fortifications combined:			
Overall mean			111
SD			8.8
RSD			7.9
Max			127
Min			98
n =			10

Results from Table 1, p. 33; Table 2, p. 35 of MRID 47053002.

Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).

1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.

2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.

3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Deltamethrin in two California sediments.[1]

Fortified (µg a.i./kg)	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0.1	0.0454	0.134	0.089	89				0.0720	0.159	0.087	87			
		0.123	0.078	78					0.150	0.078	78			
		0.129	0.084	84					0.161	0.089	89			
		0.131	0.086	86					0.156	0.084	84			
		0.147	0.102	102	87	8.9	10		0.158	0.086	86	85	4.2	5.0
1.0		1.09	1.04	104					0.812	0.740	74			
		1.21	1.16	116					0.879	0.807	81			
		1.16	1.11	111					1.03	0.958	96			
		1.04	0.99	99					1.04	0.968	97			
		1.14	1.09	109	108	6.5	6.0		0.844	0.772	77	85	11	13
Overall mean				98							85			
SD				13							7.7			
RSD				14							9.0			
Max				116							97			
Min				78							74			
n =				10							10			

Soils combined:	
Overall mean	91
SD	12
RSD	14
Max	116
Min	74
n =	20

0.1 µg a.i./kg fortifications combined:			
Overall mean			86
SD			6.7
RSD			7.8
Max			102
Min			78
n =			10

1.0 µg a.i./kg fortifications combined:			
Overall mean			97
SD			15
RSD			15
Max			116
Min			74
n =			10

Results from Table 1, p. 33; Table 2, p. 35 of MRID 47053002.
Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).
1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.
2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.
3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Esfenvalerate in two California sediments.[1]

Fortified (µg a.i./kg)	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0.1	0.0656	0.141	0.075	75				0.0148	0.112	0.097	97			
		0.142	0.076	76					0.126	0.111	111			
		0.147	0.081	81					0.127	0.112	112			
		0.138	0.072	72					0.124	0.109	109			
		0.149	0.083	83	78	4.5	5.8		0.133	0.118	118	110	7.7	7.0
1.0		1.12	1.05	105					0.967	0.952	95			
		1.29	1.22	122					1.10	1.09	109			
		1.24	1.17	117					1.13	1.12	112			
		1.06	0.994	99					1.08	1.07	107			
		1.22	1.15	115	112	9.4	8.4		1.03	1.02	102	105	6.4	6.1
Overall mean				95							107			
SD				19							7.2			
RSD				20							6.7			
Max				122							118			
Min				72							95			
n =				10							10			

Soils combined:	
Overall mean	101
SD	15
RSD	15
Max	122
Min	72
n =	20

0.1 µg a.i./kg fortifications combined:			
Overall mean			94
SD			18
RSD			19
Max			118
Min			72
n =			10

1.0 µg a.i./kg fortifications combined:			
Overall mean			108
SD			8.5
RSD			7.9
Max			122
Min			95
n =			10

Results from Table 1, p. 34; Table 2, p. 36 of MRID 47053002.

Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).

1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.

2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.

3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Fenpropathrin in two California sediments.[1]

Fortified	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
(µg a.i./kg)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0.1	0	0.102	0.102	102				0	0.0945	0.095	95			
		0.0938	0.094	94					0.109	0.109	109			
		0.107	0.107	107					0.106	0.106	106			
		0.108	0.108	108					0.111	0.111	111			
		0.107	0.107	107	104	5.9	5.7		0.105	0.105	105	105	6.4	6.1
1.0		1.10	1.10	110					0.984	0.984	98			
		1.15	1.15	115					1.05	1.05	105			
		1.16	1.16	116					1.14	1.14	114			
		1.08	1.08	108					1.07	1.07	107			
		1.16	1.16	116	113	3.7	3.3		0.999	0.999	100	105	6.2	5.9
Overall mean				108							105			
SD				6.8							5.9			
RSD				6.3							5.7			
Max				116							114			
Min				94							95			
n =				10							10			

Soils combined:	
Overall mean	107
SD	6.5
RSD	6.1
Max	116
Min	94
n =	20

0.1 µg a.i./kg fortifications combined:			
Overall mean			104
SD			5.9
RSD			5.6
Max			111
Min			94
n =			10

1.0 µg a.i./kg fortifications combined:			
Overall mean			109
SD			6.5
RSD			5.9
Max			116
Min			98
n =			10

Results from Table 1, p. 34; Table 2, p. 36 of MRID 47053002.
Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).
1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.
2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.
3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Lambda-cyhalothrin in two California sediments.[1]

Fortified (µg a.i./kg)	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
0.1	0.0415	0.125	0.084	84				0.0111	0.101	0.090	90			
		0.124	0.083	83					0.117	0.106	106			
		0.137	0.096	96					0.113	0.102	102			
		0.134	0.093	93					0.115	0.104	104			
		0.149	0.108	108	92	10	11		0.116	0.105	105	101	6.5	6.5
1.0		1.10	1.06	106					0.978	0.967	97			
		1.25	1.21	121					1.06	1.05	105			
		1.17	1.13	113					1.12	1.11	111			
		1.08	1.04	104					1.07	1.06	106			
		1.22	1.18	118	112	7.4	6.6		1.00	0.989	99	103	5.7	5.5
Overall mean				102							102			
SD				13							5.9			
RSD				13							5.8			
Max				121							111			
Min				83							90			
n =				10							10			

Soils combined:	
Overall mean	102
SD	10
RSD	9.9
Max	121
Min	83
n =	20

0.1 µg a.i./kg fortifications combined:			
Overall mean			97
SD			9.4
RSD			9.7
Max			108
Min			83
n =			10

1.0 µg a.i./kg fortifications combined:			
Overall mean			108
SD			7.8
RSD			7.2
Max			121
Min			97
n =			10

Results from Table 1, p. 34; Table 2, p. 36 of MRID 47053002.

Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).

1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.

2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.

3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Method validation for determination of Permethrin in two California sediments.[1]

Fortified (µg a.i./kg)	Fresh Water Sediment (BUCGR, TOC 1.31%)							Estuarine Sediment (Paradise Cove, TOC 0.86%)						
	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD[2] (%)	RSD[3] (%)	Control Mean (µg/kg)	Measured (µg/kg)	Corrected (µg/kg)	Recovery (%)	Mean (%)	SD (%)	RSD (%)
1.0	0	0.973	0.973	97				0	0.984	0.984	98			
		0.988	0.988	99					1.11	1.11	111			
		0.939	0.939	94					1.02	1.02	102			
		0.995	0.995	100					1.07	1.07	107			
		1.08	1.08	108	100	5.2	5.2		1.11	1.11	111	106	5.6	5.3
10.0		9.72	9.72	97					9.58	9.58	96			
		12.4	12.4	124					10.6	10.6	106			
		10.7	10.7	107					11.1	11.1	111			
		9.32	9.32	93					10.8	10.8	108			
		11.7	11.7	117	108	13	12		10.1	10.1	101	104	6.0	5.8
Overall mean				104							105			
SD				10							5.5			
RSD				9.9							5.3			
Max				124							111			
Min				93							96			
n =				10							10			

Soils combined:	
Overall mean	104
SD	8.1
RSD	7.7
Max	124
Min	93
n =	20

1.0 µg a.i./kg fortifications combined:	
Overall mean	103
SD	6.1
RSD	5.9
Max	111
Min	94
n =	10

10.0 µg a.i./kg fortifications combined:	
Overall mean	106
SD	9.7
RSD	9.1
Max	124
Min	93
n =	10

Results from Table 1, p. 34; Table 2, p. 36 of MRID 47053002.
Means and standard deviations calculated using Microsoft program functions =AVERAGE(A1:A2) and =STDEV(A1:A2).
1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.
2 SD = Standard Deviation; determined using the "unbiased" or "n-1" method.
3 RSD = Relative Standard Deviation; calculated as (SD/mean) x 100.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Verification of reported results using GC peak area data.[1]

Fortified (µg a.i./kg)	Compound	Fresh Water Sediment (BUCGR, TOC 1.31%)						Estuarine Sediment (Paradise Cove, TOC 0.86%)					
		Bracket Std 1 (peak area)	Bracket Std 2 (peak area)	Mean Std (peak area)	Measured (peak area)	Measured (µg/kg)	Reported[2] (µg/kg)	Bracket Std 1 (peak area)	Bracket Std 2 (peak area)	Mean Std (peak area)	Measured (peak area)	Measured (µg/kg)	Reported[3] (µg/kg)
0.1	Bifenthrin	7454	6804	7129	7355	0.103	0.103	6312	5926	6119	8465	0.138	0.138
	Cypermethrin	7257	6027	6642	7080	0.107	0.107	6869	6254	6562	6423	0.0979	0.0979
	Cyfluthrin	10870	8872	9871	10542	0.107	0.107	10000	9051	9526	9613	0.101	0.101
	Deltamethrin	6022	5406	5714	7633	0.134	0.134	4855	4457	4656	7418	0.159	0.159
	Esfenvalerate	13421	10554	11988	16887	0.141	0.141	12026	10940	11483	12911	0.112	0.112
	Fenpropathrin	11706	9881	10794	11040	0.102	0.102	10783	10414	10599	10014	0.0945	0.0945
	Lambda-cyhalothrin	10784	9119	9952	12470	0.125	0.125	8164	7569	7867	7959	0.101	0.101
1.0	Permethrin	5857	4918	5388	5246	0.974	0.973	5435	5606	5521	5433	0.984	0.984
1.0	Bifenthrin	115423	116412	115918	57760	0.997	0.997	121315	124239	122777	60105	0.979	0.979
	Cypermethrin	90994	91267	91131	50676	1.11	1.11	111862	117395	114629	58299	1.02	1.02
	Cyfluthrin	146068	144415	145242	80574	1.11	1.11	168645	178012	173329	85979	0.992	0.992
	Deltamethrin	90699	88712	89706	48891	1.09	1.09	85238	90585	87912	35683	0.812	0.812
	Esfenvalerate	185356	181974	183665	102955	1.12	1.12	221658	234713	228186	110281	0.967	0.967
	Fenpropathrin	170037	169884	169961	93570	1.10	1.10	216355	224573	220464	108530	0.985	0.984
	Lambda-cyhalothrin	153812	154546	154179	85102	1.10	1.10	154964	162527	158746	77659	0.978	0.978
10.0	Permethrin	80094	80924	80509	39075	9.71	9.72	105832	110099	107966	51714	9.58	9.58

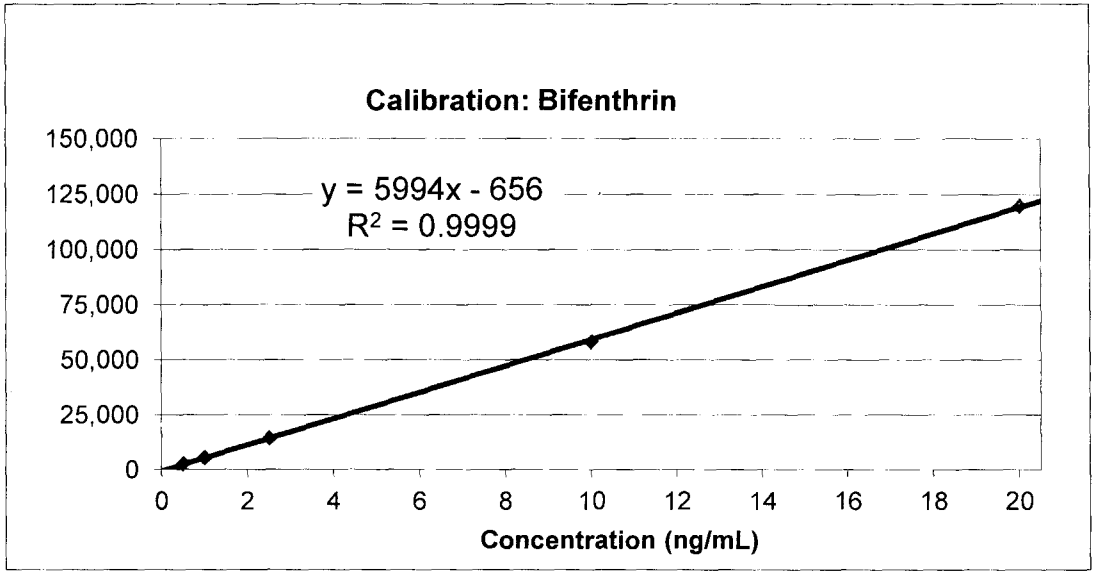
Results from Tables 1-2, pp. 33-36; Figures 3-8, pp. 52-93; Figures 11-16, pp. 108-149 of MRID 47053002.
Measured (µg/kg) determined using reported example calculations (pp. 25-27 of MRID 47053002).
1 Sediment source and TOC obtained from pp. 12, 19, 29 of MRID 47053002.
2 Fortified Control 11 at LOQ and Fortified Control 16 at 10 x LOQ.
3 Fortified Control 21 at LOQ and Fortified Control 26 at 10 x LOQ.

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Bifenthrin.

Concentration (ng/mL)	Peak Area (counts)
0.5	2681
1.0	5493
2.5	14349
10	58285
20	119698

Results from Figure 17, p. 150; Figure 18, p. 157; Figure 19, p. 164; Figure 20, p. 171; Figure 21, p. 178.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999931367
R Square	0.999862738
Adjusted R Square	0.999816984
Standard Error	673.9598784
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	9926113167	9.93E+09	21853.004	6.825E-07
Residual	3	1362665.753	454221.9		
Total	4	9927475833			

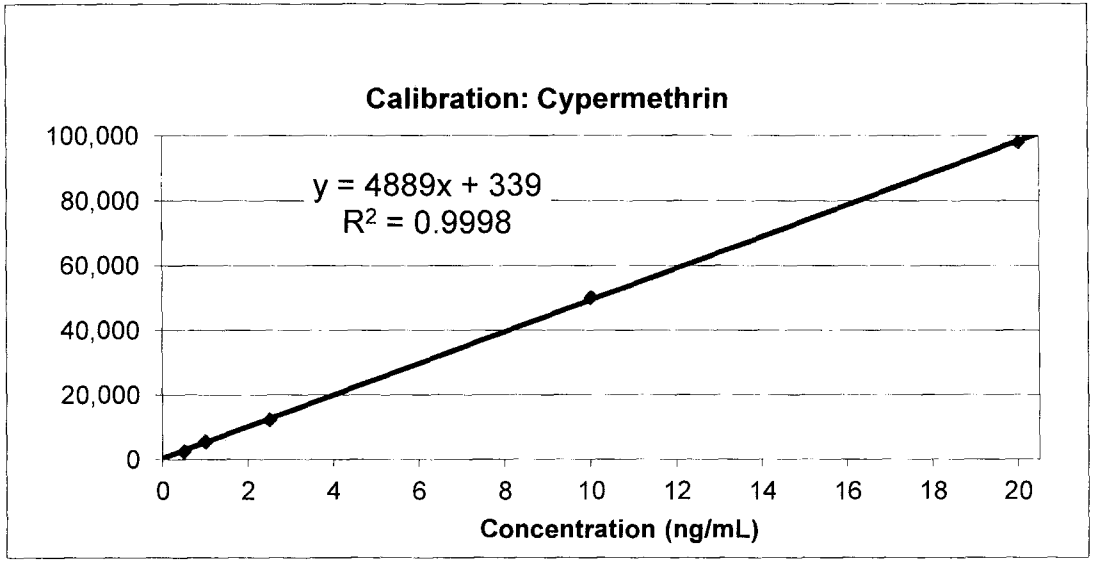
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-656.351068	408.4854783	-1.606792	0.2064569	-1956.3342	643.632033	-1956.33417	643.6320334
X Variable 1	5993.75751	40.54558779	147.8276	6.825E-07	5864.7234	6122.79167	5864.723354	6122.791666

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Cypermethrin.

Concentration (ng/mL)	Peak Area (counts)
0.5	2452
1.0	5474
2.5	12184
10	50030
20	97707

Results from Figure 17, p. 154; Figure 18, p. 161; Figure 19, p. 168; Figure 20, p. 175; Figure 21, p. 182.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999914998
R Square	0.999830003
Adjusted R Square	0.999773338
Standard Error	611.5290007
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	6598440600	6.6E+09	17644.412	9.407E-07
Residual	3	1121903.156	373967.7		
Total	4	6599562503			

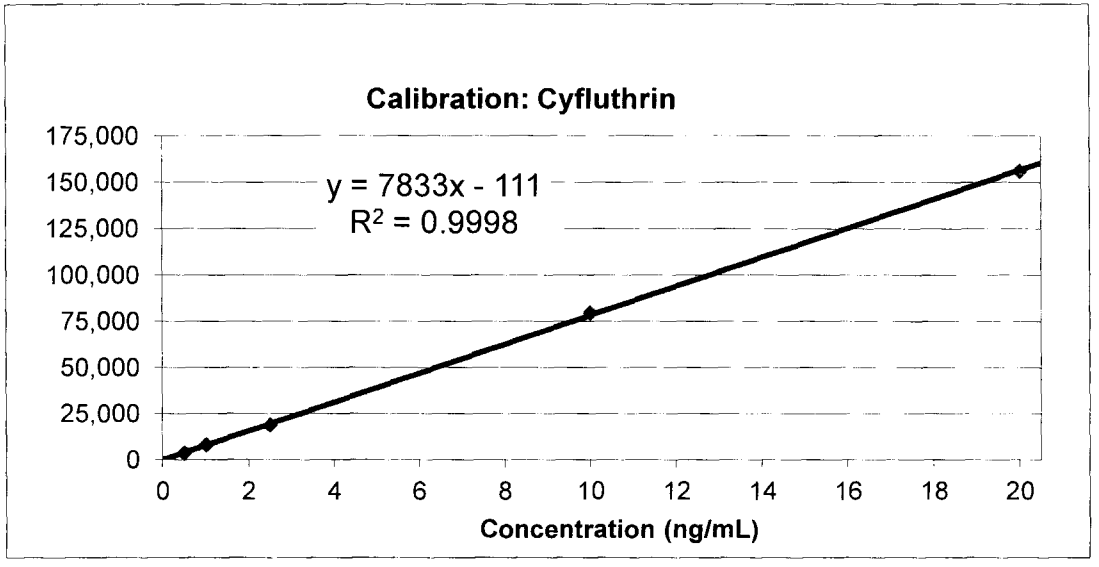
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	338.7278321	370.6462719	0.913884	0.4281645	-840.83403	1518.28969	-840.834026	1518.28969
X Variable 1	4886.863554	36.78973123	132.8323	9.407E-07	4769.7822	5003.9449	4769.78221	5003.944898

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Cyfluthrin.

Concentration (ng/mL)	Peak Area (counts)
0.5	3564
1.0	8056
2.5	18640
10	79513
20	156003

Results from Figure 17, p. 153; Figure 18, p. 160; Figure 19, p. 167; Figure 20, p. 174; Figure 21, p. 181.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999916438
R Square	0.999832884
Adjusted R Square	0.999777179
Standard Error	971.9010229
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	16954093800	1.7E+10	17948.597	9.169E-07
Residual	3	2833774.795	944591.6		
Total	4	16956927575			

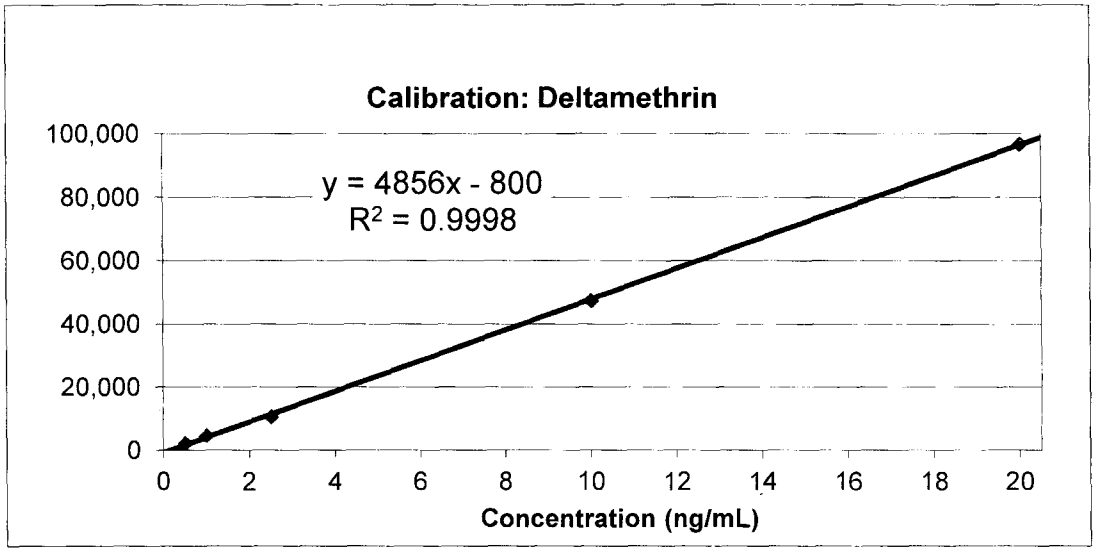
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-111.4962	589.0668969	-0.189276	0.8619582	-1986.17	1763.17757	-1986.16997	1763.177569
X Variable 1	7833.337676	58.46979844	133.9724	9.169E-07	7647.2607	8019.41467	7647.260682	8019.41467

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Deltamethrin.

Concentration (ng/mL)	Peak Area (counts)
0.5	2141
1.0	4572
2.5	10511
10	47234
20	96652

Results from Figure 17, p. 156; Figure 18, p. 163; Figure 19, p. 170; Figure 20, p. 177; Figure 21, p. 184.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.99987697
R Square	0.999753956
Adjusted R Square	0.999671941
Standard Error	731.1072057
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	6515738073	6.52E+09	12189.938	1.638E-06
Residual	3	1603553.239	534517.7		
Total	4	6517341626			

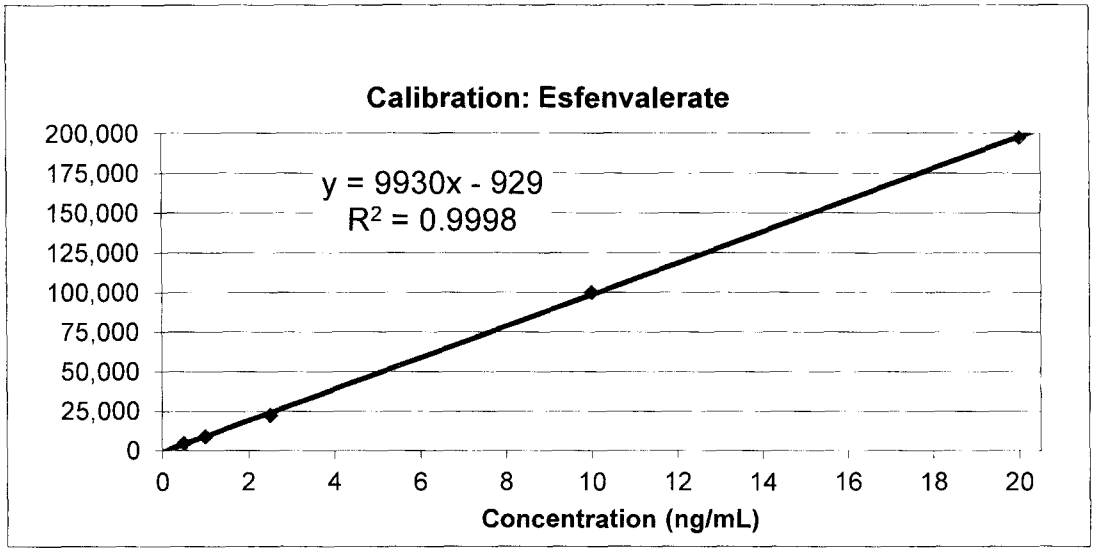
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-799.764748	443.1223373	-1.80484	0.1688599	-2209.9778	610.448297	-2209.97779	610.4482966
X Variable 1	4856.141875	43.9835847	110.4081	1.638E-06	4716.1665	4996.11727	4716.166478	4996.117271

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Esfenvalerate.

Concentration (ng/mL)	Peak Area (counts)
0.5	4521
1.0	8960
2.5	22472
10	99991
20	197023

Results from Figure 17, p. 155; Figure 18, p. 162; Figure 19, p. 169; Figure 20 p. 176; Figure 21, p. 183.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999902578
R Square	0.999805166
Adjusted R Square	0.999740221
Standard Error	1330.282969
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	27243260859	2.72E+10	15394.693	1.154E-06
Residual	3	5308958.329	1769653		
Total	4	27248569817			

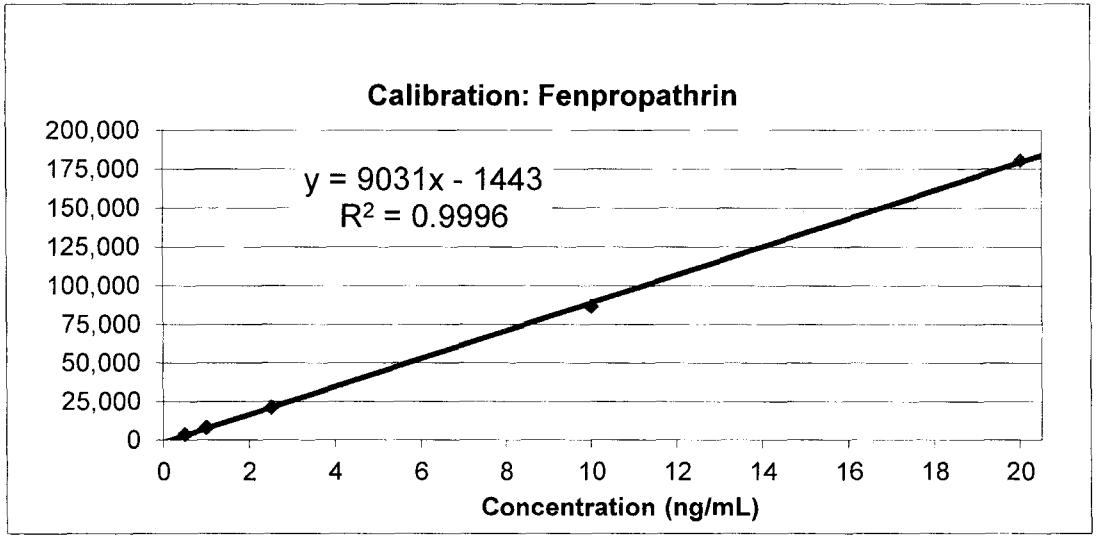
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-929.022439	806.2813412	-1.152231	0.3327126	-3494.9695	1636.92464	-3494.96951	1636.924636
X Variable 1	9929.768006	80.03014218	124.0754	1.154E-06	9675.0764	10184.4596	9675.076376	10184.45964

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Fenpropathrin.

Concentration (ng/mL)	Peak Area (counts)
0.5	3802
1.0	8251
2.5	21056
10	86322
20	180408

Results from Figure 17, p. 150; Figure 18, p. 157; Figure 19, p. 164; Figure 20, p. 171; Figure 21, p. 178.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999801027
R Square	0.999602094
Adjusted R Square	0.999469459
Standard Error	1729.189356
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	22534768017	2.25E+10	7536.4702	3.369E-06
Residual	3	8970287.492	2990096		
Total	4	22543738305			

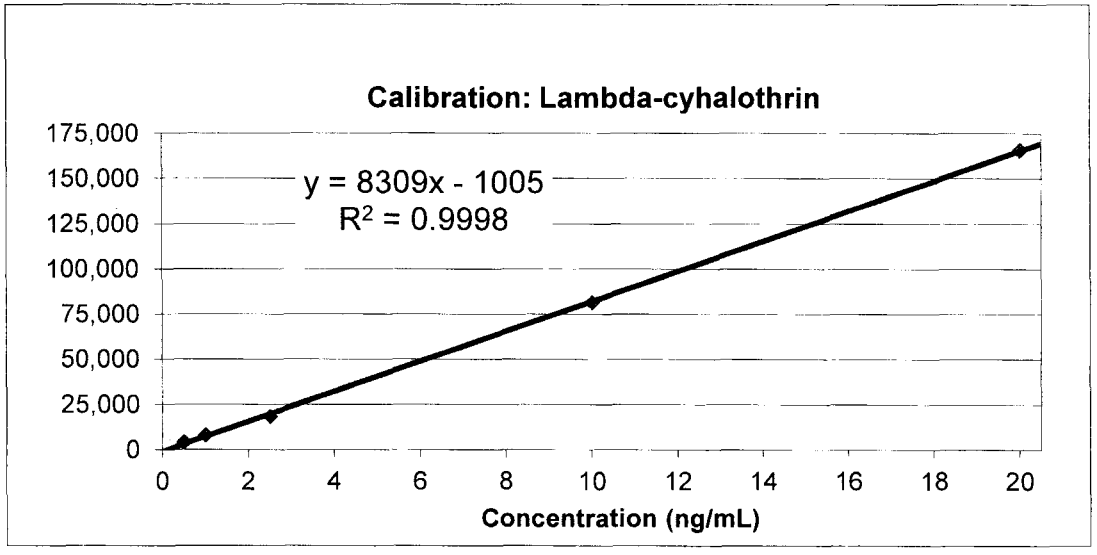
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-1443.03692	1048.057554	-1.376868	0.2623099	-4778.4238	1892.34997	-4778.42381	1892.349972
X Variable 1	9031.005429	104.0284461	86.81285	3.369E-06	8699.9405	9362.07037	8699.940485	9362.070373

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Lambda-cyhalothrin.

Concentration (ng/mL)	Peak Area (counts)
0.5	4057
1.0	8036
2.5	18359
10	81386
20	165637

Results from Figure 17, p. 151; Figure 18, p. 158; Figure 19, p. 165; Figure 20, p. 172; Figure 21, p. 179.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.99989398
R Square	0.999787971
Adjusted R Square	0.999717295
Standard Error	1161.218192
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	19074865783	1.91E+10	14146.006	1.31E-06
Residual	3	4045283.068	1348428		
Total	4	19078911066			

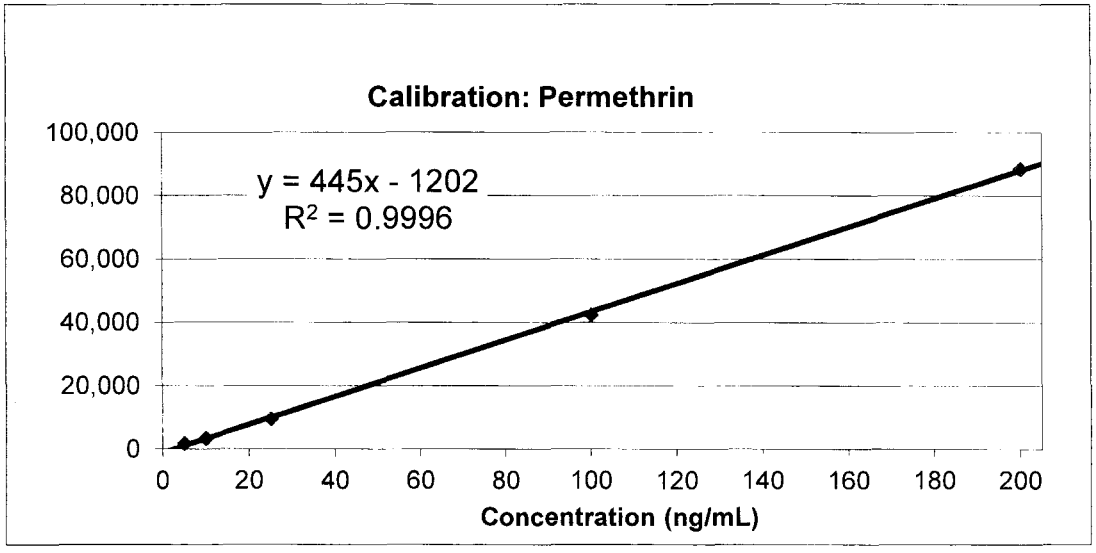
	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-1005.09989	703.8115825	-1.428081	0.2485743	-3244.9425	1234.74268	-3244.94246	1234.742679
X Variable 1	8308.838219	69.85916471	118.937	1.31E-06	8086.5152	8531.16126	8086.515179	8531.16126

Chemical: Pyrethroids
PC: 069007
MRID: 47053002
Guideline: 850.7100 ECM in sediment

Linear regression Permethrin.

Concentration (ng/mL)	Peak Area (counts)
5.0	1788
10	3434
25	9434
100	42339
200	88355

Results from Figure 17, p. 152; Figure 18, p. 159; Figure 19, p. 166; Figure 20, p. 173; Figure 21, p. 180.



SUMMARY OUTPUT

Regression Statistics	
Multiple R	0.999809461
R Square	0.999618959
Adjusted R Square	0.999491945
Standard Error	834.1243105
Observations	5

ANOVA

	df	*SS*	*MS*	*F*	*Sig F*
Regression	1	5475774812	5.48E+09	7870.1683	3.157E-06
Residual	3	2087290.096	695763.4		
Total	4	5477862102			

	Coefficients	*Standard Error*	*t Stat*	*P-value*	*Lower 95%*	*Upper 95%*	*Lower 95.0%*	*Upper 95.0%*
Intercept	-1202.01013	505.5607594	-2.377578	0.0978316	-2810.9301	406.909837	-2810.9301	406.9098368
X Variable 1	445.1766196	5.018111841	88.71397	3.157E-06	429.20675	461.146491	429.2067481	461.1464911