Bifenthrin **Test Material:**

MRID: 49024201, 49168201, 49189001

Bifenthrin: Independent Laboratory Validation (ILV) Study in Water, Title:

Soil and Sediment

EPA PC Code: 128825

OCSPP Guideline: 850.6100

For CDM Smith

Primary Reviewer: Lisa Muto

Signature: Leva Muto

Date: 9/2/14

Signature: Signature: Januari

Date: 9/2/14

Signature: Januari

Date: 9/2/14 **Secondary Reviewer:** Lynne Binari

QC/QA Manager: Joan Gaidos

Date: 9/2/14

Indepent laboratory validations of previously submitted analytical methods for bifenthrin in water and soil/sediment

Reports: Previous ECMs: Previously submitted and reviewed EPA MRID Nos.

48638501 (water method; Morse Labs Project No.: ML10-1602-PWG) and 47053001/47053002 (soil method; Morse Project No.: ML06-1286-PWG). Not considered in this DER. Refer to *Background* for additional details. **ECM:** EPA MRIDs **49024201 & 49189001**. Yang, J. 2013. Independent Laboratory Validation of the Method for the Analysis of Bifenthrin in Water and Soil by LC-MS/MS. PASC Project No.: 058-0612A. PASC Report No.: PASC-REP-0202. FMC Study No.: 182ILV11R1. FMC Report No.: PC-0721. Report prepared by Primera Analytical Solutions Corporation, Princeton, NJ; sponsored by FMC Corporation, Ewing, NJ; 45 pages. Final

report issued September 20, 2012 and revised July 15, 2013.

<u>ILV</u>: EPA MRID **49168201**. Habeeb, S. B. 2013. Bifenthrin: Independent Laboratory Validation (ILV) Study in Sediment. Ricerca Document No.: 031001-1. Report prepared by Ricerca Biosciences, LLC, Concord, Ohio; sponsored and submitted by Consumer Specialty Products Association Inc. for Bifenthrin Task Force Steering Committee/Joint Venture, Washington,

D.C.; 94 pages. Final report issued July 2, 2013.

Document No.: MRIDs 49024201/49189001 and 49168201

Guideline: 850.6100

Statements: ECM: The study was conducted in accordance with U.S. EPA GLP (CFR

Title 40, Part 160; p. 3). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Certification of the Authenticity statements were

provided (pp. 2-6).

<u>ILV</u>: The study was conducted in accordance with U.S. EPA GLP (CFR Title 40, Part 160; p. 3). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Certification of the Authenticity statements were

provided (pp. 2-5). An approvals page was also included (p. 6).

Classification: This analytical method is considered acceptable. The method for the

analysis of water described in MRIDs 49024201/49189001 appears to have

no associated ILV. Recoveries were corrected in the ILV; sample

calculations were not provided in the ECM. Some of the supporting data, *i.e.* chromatograms and matrix details, were not provided. The LOQ in the ECM and ILV is less than the lowest toxicological level of concern in soil/sediment; however, the LOQ in the ECM is greater than the lowest toxicological level of concern in water. Refer to sections **Executive**

Summary and Background for additional details.

PC Code: 128825

Reviewer: José L. Meléndez, U.S. EPA **Date:** February 6, 2017

José fais Melende

Signature:

All information is taken from MRID 49168201 (ILV) unless otherwise noted. All page numbers are those corresponding to the lower right side of the pages.

Executive Summary

Two independent laboratory validations (ECM and ILV) were performed. <u>ECM</u>, PASC Project No. 058-0612A (MRIDs 49024201, as superseded by 49189001), is quantitative for bifenthrin at the stated LOQ of 0.500 ppt (pg/g) for water and 0.100 ppb (ng/g) for soil using LC/MS/MS. <u>ILV</u>, Ricerca Document No. 031001-1 (MRID 49168201), is quantitative for bifenthrin at the stated LOQ of 0.102 ppb (ng/g) for sediment using LC/MS/MS. The LOQ in the ECM and ILV is <u>less than</u> the lowest toxicological level of concern in soil/sediment [*i.e.*, the NOAEC in MRID 48593601 for *Hyalella azteca*, 0.25 μg/kg-dw]; however, the LOQ in the ECM is <u>greater than</u> the lowest toxicological level of concern in water [*i.e.*, the NOAEC in MRID 48593601 for *H. azteca*, 0.050 ng/L]¹. The soil/sediment method obtained acceptable validation results after the first trial.

Table 1. Analytical Method Summary¹

Analyte(s) by Pesticide	MRID							Limit of
	Environmental Chemistry Method	Independent Laboratory Validation	EPA Review	Matrix	Method Date	Registrant	Analysis	Quantitation (LOQ)
Bifenthrin	49024201/ 49189001 ²	Not available		Water	09/20/2012, revised July 15, 2013	Inc. for	LC/MS/MS	0.500 pg/g or 0.500 ppt or 0.500 ng/L or 0.000500 μg/L
		49168201		Soil/ Sediment				0.100 ng/g or 0.100 ppb or 0.100 μg/kg

¹ Previously submitted and reviewed ECMs MRIDs 48638501 and 47053001/47053002 were not considered to be validated by the currently reviewed studies due to differences in study design, separation and detection techniques. Refer to *Background* below, for additional details.

² Study 49189001 supersedes 49024201; it differs from the original study by only minor issues described in page 4A of MRID 49189001.

¹ The ECM study author reported LOAEC values of 0.003-0.004 μg a.i./L for freshwater invertebrates and estuarine/marine invertebrates and a NOAEC value of 0.0013 μg a.i./L on reproductive and growth parameters for freshwater invertebrates (based on the EPA EFED Registration Review Problem Formulation for Bifenthrin, 06/09/11).

Background

Three studies were separately received for review as follows:

49024201: This is a method for the analysis of bifenthrin in soil and water using HPLC-MS/MS. It cites three previously reviewed studies as the associated ECMs: (a) EPA MRIDs 48638501 (water method; Morse Labs Project No.: ML10-1602-PWG), and (b) 47053001/47053002 (soil method; Morse Project No.: ML06-1286-PWG). These ECMs had been previously submitted and reviewed. The soil/sediment method was found to be supplemental and to partially fulfill the guideline requirement, provided additional information was submitted, namely an ILV (DP Barcode 337222). The water method (i.e., influent and effluent) was found to be unacceptable (DP Barcode 395988). Both these soil/sediment and water methods, submitted by the Pyrethroid Working Group (PWG), used GC/MS as the separation and detection technique and involve the analysis of multiple pyrethroids, besides bifenthrin.

49189001: This study supersedes 49024201. It differs from the original study in a few details, described in page 4A of the study.

49168201: It is a method for the analysis of bifenthrin in sediment using HPLC-MS/MS. It contains a copy of MRID 49024201 appended to it. An inspection of the method appears to confirm that the study authors used MRID 49024201 as the source method for the sediment analysis.

In this DER, it is assumed that MRID 49024201, as superseded by 49189001, is the ECM, and MRID 49168201 is the ILV, for the analysis of *soil/sediment*. Furthermore, the method for the analysis of *water* described in MRIDs 49024201/49189001 appears to have no associated ILV.

**The ECMs for the analysis of water (48638501) and soil (47053001/47053002), using GC/MS, and which were cited in the ECM (49024201/49189001, reviewed in this DER), are not comparable to the methods described in the DER in that the separation and detection techniques were LC/MS/MS.²

² The following changes were cited in the ECM study: the quantification method was changed from GC/MS to LC/MS/MS; the final reconstitution solvent was changed from acetone to methanol due to the LC/MS/MS analysis; and sodium chloride was added to the water sample prior to hexane extraction.

I. Principle of the Method

Water extraction: ECM only (MRID 49168201; Appendix E, Appendix A, pp. 52-94).

Water samples (500 mL) were mixed with saturated sodium chloride (10 g) then extracted with hexane (100 mL; extraction details unreported; Appendix E, Appendix A, pp. 67-68; Appendix E, Appendix A, Tables 2A and 3A, pp. 67-68). The hexane extract was removed and dried with anhydrous sodium sulphate. The volume was reduced to dryness then the residue was reconstituted in hexane (evaporation details not reported; volume of reconstitution not reported). The hexane extract was purified via silica solid phase extraction (SPE) cartridge (SPE details not reported, including elution solvent). The eluate was reduced to dryness then the residue was reconstituted in methanol (0.5 mL) prior to analysis via LC/MS/MS.

<u>Soil/sediment extraction</u>: ECM and ILV (MRID 49168201; data contained in pages 1-37). Information in **bold** was reported only in ILV.

Soil/sediment samples (10.0 g or ca. 10 g) were mixed with methanol:water (1:1, v:v; 15 mL) then extracted with hexane (10.0 mL or 7.5-9.0 mL) via shaking in a wrist-action shaker for ca. 60 minutes (pp. 12-13, 15-16; Table 3, p. 23; Appendix E, Appendix A, pp. 63, 68; Appendix E, Appendix A, Tables 2B and 3B, pp. 67-68). After centrifugation (4000 rpm for 5 minutes), the top hexane extract was reduced to dryness under nitrogen (evaporation temperature not reported). The residue was reconstituted in 2 mL of hexane using sonication. The hexane extract was purified via solid phase extraction (SPE) using Silica Bond Elut SPE cartridges preconditioned with hexane (3 mL). After charging the column with the extract using hexane (1 mL), the analytes were eluted with 6 mL of hexane:diethyl ether (9:1, v:v). The eluate was reduced to dryness under nitrogen (evaporation temperature not reported). The residue was reconstituted in 2 mL of methanol prior to analysis via LC/MS/MS.

Samples were analyzed for bifenthrin by liquid chromatography with mass spectrometry (LC/MS/MS; pp. 13-14; Appendix E, Appendix A, pp. 64-65). A Varian MonoChrome C18 (30 mm x 2.0 mm i.d., 3.0 μm) column was used with a gradient mobile phase of (A) aqueous 5 mM ammonium acetate and (B) 5 mM ammonium acetate in acetonitrile:water (99:1, v:v; time A:B; 0.0-0.1 min. 80:20, 2-5 min. 5:95, 5.1-6.0 min. 80:20). Bifenthrin ions were monitored using positive ESI MS (Q1, 440.2 m/z; Q3, 181.1 m/z; MRM scan type). Injection volume was 20 μL.

In ECM, the LOQ values for bifenthrin were 0.500 ppt (pg/g) in water and 0.100 ppb (ng/g) in soil (Appendix E, Appendix A, pp. 60, 72-74). The LOD values were 0.2 ppt in water and 0.03 ppb. In the ILV, the LOQ value for bifenthrin in sediment was 0.102 ppb (ng/g); the LOD was not reported (pp. 7, 16-17).

II. Recovery Findings

ECM (MRID 49168201; Appendix E, Appendix A, pp. 52-94): Mean recoveries and RSDs were within guideline requirements for analysis of bifenthrin in surface water and sandy loam soil (Appendix E, Appendix A, pp. 60, 63, 78; Appendix E, Appendix A, Tables 7A-7B, pp. 76-77;

Appendix E, Appendix A, Attachment II, p. 85). The method was validated with the first trial. The water (uncharacterized) was collected from the Delaware River in Yardley, Pennsylvania. The soil was characterized as sandy loam (58% sand, 27% silt, 15% clay) with 5.01% organic matter, 2.91% organic carbon and pH 7.21; the primary soil source was not reported. Bifenthrin was identified by LC/MS/MS using MRM (Appendix E, Appendix A, pp. 64-65).

ILV (MRID 49168201; Data contained in pages 1-37): Mean recoveries and RSDs were within guideline requirements for analysis of bifenthrin in sandy clay loam sediment (p. 7; Table 7, p. 27; Appendix A, p. 28). The method was validated with the first trial. The Wyoming sediment was characterized as sandy clay loam (47% sand, 19% silt, 34% clay) with 3.4% organic matter and pH 8.4 (soil:water ratio, 1:1; p. 12; Appendix A, p. 28). Bifenthrin was identified by LC/MS/MS using MRM (pp. 13-14). Water samples were not tested in the ILV.

Table 2. Initial Validation Method Recoveries for Bifenthrin in Water and Soil

Analyte	Fortification Level (ppt) or (ppb) ¹		Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)	
Surface Water (Delaware River)							
D:f41	0.500 (LOQ)	5	71.7-94.6	85.9	8.60	10.0	
Bifenthrin	5.00	5	83.7-98.7	88.2	6.05	6.86	
Sandy Loam Soil							
Bifenthrin	0.100 (LOQ)	5	70.6-94.5	79.7	9.08	11.4	
Bilenurin	1.00	5	79.0-92.4	86.6	4.87	5.63	

Data were obtained from Appendix E, Appendix A, pp. 60, 63; Appendix E, Appendix A, Tables 7A-7B, pp. 76-77; Appendix E, Appendix A, Attachment II, p. 85 of the study report.

Table 3. Independent Validation Method Recoveries for Bifenthrin in Sediment

Analyte	Fortification Level (ppb)		Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)	
Sandy Clay Loam Sediment							
Bifenthrin	0.102 (LOQ) ¹	5	75.2-106	93.3	13.3	14.2	
Ditentin	1.02^{1}	5	77.2-103	87.3	9.7	11.1	

Data were obtained from Table 7, p. 27; Appendix A, p. 28 of the study report.

III. Method Characteristics

In ECM, the LOQ values for bifenthrin were 0.500 ppt (pg/g) in water and 0.100 ppb (ng/g) in soil (Appendix E, Appendix A, pp. 60, 72-74). At this LOQ, signal-to-noise ratios (S/N) ranged 104 to 308 in water samples and 219 to 377 in soil samples. The LOD were estimated by multiplying the standard deviation of replicate samples fortified at LOQ level with the t-

¹ Fortification units were ppt (pg/g) for water and ppb (ng/g) for soil.

¹ Fortification levels ranged 0.101-0.102 ppb for the nominal LOQ of 0.102 ppb and 0.952-1.02 ppb for the nominal 10×LOQ of 1.02 ppb.

distribution value at 99% confidence level ($t_{0.99} = 3.747$ for 5 replicates). The LOD values for bifenthrin were 0.2 ppt (S/N ca. 85) in water and 0.03 ppb (S/N ca. 119) in soil.

The ECM study author reported the lowest toxicity values for freshwater invertebrates and estuarine/marine invertebrates as LOAEC values of 0.003 µg a.i./L (waterflea) and 0.004 µg a.i./L (mysid shrimp), respectively (based on the EPA EFED Registration Review Problem Formulation for Bifenthrin, 06/09/11). The ECM study author reported the NOAEC (No Observed Adverse Effect Concentration) value of 0.0013 µg a.i./L on reproductive and growth parameters for freshwater invertebrates (based on the EPA EFED Registration Review Problem Formulation for Bifenthrin, 06/09/11). The ECM study author also reported that "bifenthrin has no adverse effect on various soil microorganisms or their soil activity" (Appendix E, Appendix A, p. 74). Based on this data, the ECM study author concluded that the LOQ levels validated by the study report are below levels of ecological concern in soil and water (Appendix E, Appendix A, p. 74).

In ILV, the LOQ value for bifenthrin in sediment was 0.102 ppb (ng/g); the LOD was not reported (pp. 7, 16-17). At this LOQ, signal-to-noise ratios (S/N) ranged 51.6 to 84.7. The ILV study author noted the Method LOQ was changed from 0.100 ppb to 0.102 ppb due to the calculation error (significant figure correction) in the concentration of the stock solution which was prepared (p. 20).

Based upon the most recent toxicity data, the LOQ in the ECM and ILV is <u>less than</u> the lowest toxicological level of concern in soil/sediment (*i.e.*, the NOAEC in MRID 48593601 for *Hyalella azteca*, 0.25 μg/kg-dw). The LOQ in the ECM is <u>greater than</u> the lowest toxicological level of concern in water (*i.e.*, the NOAEC in MRID 48593601 for *H. azteca*, 0.050 ng/L).

Table 4. Method Characteristics*

	Bifenthrin				
	Water Soil/Sediment				
	River water	Sandy loam soil	Sandy clay loam sediment		
Limit of Quantitation (LOQ)	0.500 ppt (pg/g)	0.100 ppb (ng/g)	0.102 ppb (ng/g) ¹		
Limit of Detection (LOD)	0.2 ppt	0.03 ppb	Not reported		
Linearity (calibration curve r ² and concentration range)	$r^2 = 0.9916^{2,3}$ (0.250-50.0 ng/mL)	$r^2 = 0.9989^{2,3}$ $(0.250-50.0$ $ng/mL)$	$r^2 = 0.9985^{3,4}$ (0.255-51.0 ng/mL)		
Repeatable	Yes				
Reproducible	Not available ⁵ Yes ⁶		es ⁶		
Specific	Yes				

^{*} Data were obtained from Tables 5-6, pp. 25-26; Figure B-1, p. 3; Appendix E, Appendix A, Tables 5A-5B, p. 72; Appendix E, Appendix A, Figures 1a-1b, p. 79 of the ILV study report unless noted otherwise.

¹ The ILV study author noted the Method LOQ was changed from 0.100 ppb to 0.102 ppb due to an error during the preparation of the stock solution.

² The ECM study author reported that all correlation coefficients (r), were over 0.996, for linear regression of the curve for peak area versus concentration, by using 1/x², reciprocal of the square of the analyte concentration, citing Figure 1 of the document for reference; however, the reviewer could not validate this information due to the poor resolution of the document copy (Appendix E, Appendix A, p. 71; Appendix E, Appendix A, Figures 1a-1b, p. 79). The linearity values listed in the table above were reviewer-calculated based on data provided in the ECM

document (see DER Attachment 2). The reviewer-calculated linearity for the water did not agree with the ECM study author's reported results; however, the reviewer believed that the water calibration curve shown in Figure 1a did not show excellent linearity and did match the calibration curve generated by the reviewer.

³ Linearity is satisfactory when $r^2 \ge 0.995$.

- ⁴ The reviewer-calculated calibration curve yielded a satisfactory correlation coefficient (r^2 = 0.9985; see DER Attachment 2). The registrant reported correlation coefficient r = 0.9929 (pp. 16, 31), for linear regression of the curve for peak area versus concentration, by using $1/x^2$, reciprocal of the square of the analyte concentration.
- ⁵ The ECM evaluated in this document utilized LC/MS/MS analysis while the original cited ECM utilized GC/MS analysis. The MRID 48638501 was previously submitted and reviewed. The reviewer-estimated LOQ was 1.6 ng/L (ppt) for effluent wastewater in the ECM data (GC/MS analysis; data from Environmental Chemistry Method Review Report of MRID 48638501).
- ⁶ The ECM and ILV evaluated in this document utilized LC/MS/MS analysis while the original method cited utilized GC/MS analysis. The MRIDs 47053001/47053002 were previously submitted and reviewed. The reported LOQ was 0.1 ng/g (ppb) for sediment in the ECM data (GC/MS analysis; data from Environmental Chemistry Method Review Report of MRID 47053001/47053002).

IV. Method Deficiencies and Reviewer's Comments

1. The methods (ECM and ILV) were performed in support of the previously submitted and reviewed EPA MRIDs 48638501 (water method; Morse Labs Project No.: ML10-1602-PWG) and 47053001/47053002 (soil method; Morse Project No.: ML06-1286-PWG); however, ILV, Ricerca Document No. 031001-1, was specifically noted to be in support of ECM, PASC Project No. 058-0612A (p. 7). ECM was noted to be specifically in support of EPA MRIDs 48638501 and 47053001/47053002 (Appendix E, Appendix A, pp. 61-62). Overall, the submitted methods were not suitable for verifying the procedures reported by the ECMs mentioned above, since the quantification method was changed from GC/MS to LC/MS/MS. Additionally, the matrices for these reports differed slightly, in that the ECMs considered wastewater and sediment (not characterized) and the methods reported in this DER considered river water, sandy loam soil and sandy clay loam sediment.

The ECM study author reported the following method deviations/modifications: the quantification method was changed from GC to LC/MS/MS; the final reconstitution solvent was changed from acetone to methanol due to the LC/MS/MS analysis; and sodium chloride was added to the water sample prior to hexane extraction (Appendix E, Appendix A, p. 69). In addition to these modifications, the reviewer also noted the possibility of more modifications based on the summary of the ECM methods reported in the Environmental Chemistry Method Review Report of MRID 48638501, including the elimination of the addition of methanol to the water sample prior to hexane extraction. The ECM study protocol (Appendix E, Appendix A, Attachment III, pp. 86-94) was included in the study report; however, it did not contain any method details. The method of ILV was almost identical to that of ECM, except that it contained more details.

2. The ECM and ILV soil/sediment LOQs were the same or within range of the LOQs of the ECM.

- 3. The recoveries were corrected in ILV (pp. 17-18). Sample recovery calculations were not provided in the ECM.
- 4. In the ECM and ILV, representative chromatograms of calibration standards were only included for the lowest concentration calibration standard (Figures B-2 to B-6, pp. 32-36; Appendix E, Appendix A, Figures 2-6, pp. 80-84)
- 5. In ECM, the source of the surface water was reported (Delaware River), but no other characterization was provided (Appendix E, Appendix A, pp. 60, 63, 78; Appendix E, Appendix A, Tables 7A-7B, pp. 76-77; Appendix E, Appendix A, Attachment II, p. 85). The primary source of the soil was not reported, but all other matrix characterization was provided.
- 6. For ECM, the reviewer noted that some interference was observed at the retention time of bifenthrin in all soil samples (Appendix E, Appendix A, pp. 60, 78; Appendix E, Appendix A, Attachment II, Figures 2-4, pp. 80-82). The ECM study author reported that there was no significant matrix effect during analysis of the sediment samples.
- 7. The reviewer noted one significant typographical error in ILV: the LOQ was incorrectly reported as $0.102 \,\mu\text{g/g}$ (and $10\times\text{LOQ}$ as $1.02 \,\mu\text{g/g}$), instead of $0.102 \,\text{ng/g}$ (and $10\times\text{LOQ}$ as $1.02 \,\text{ng/g}$; p. 7). The correct LOQ was reported later in the document (pp. 16, 18), but it was also reported with units of ng/mL (Table 2, p. 22).
- 8. Communication between the original ECM personnel and the method's study (*i.e.*, 49024201/49189001) author regarding analytical procedure prior to method validation was reportedly recorded, but not provided (Appendix E, Appendix A, p. 78). For the ILV, communication was reported (Appendix C, p. 37).
- 9. The time requirement for sets of 12 samples was reported as *ca.* 8 hours for extraction and *ca.* 15 hours for analysis, data processing and review in the ILV report (p. 19).
- 10. The reviewer noted that the poor quality of the copy of ECM in the Appendix E of ILV made it difficult to report data from ECM with complete accuracy. The ECM was also available separately for review.

V. References

- 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. Final review completed April 20, 2012.
- Meléndez, J. Environmental Chemistry Method Review Report. MRID 48638501. Final review completed October 24, 2011.
- 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.

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(C1)C(F)(F)F)ccc2

Attachment 2: Calculations

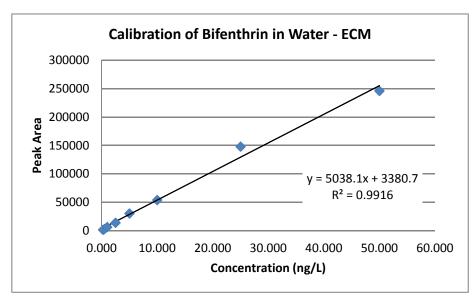
Chemical: Bifenthrin

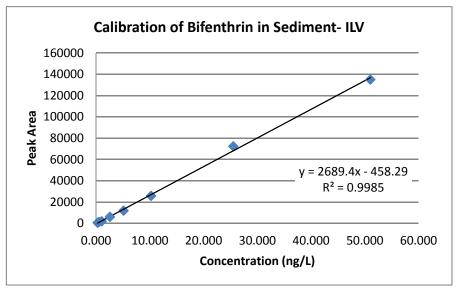
PC: 128825 MRID: 49168201 Guideline: 850.6100

ECM and ILV Calibration Curves

	Bifenthrin							
	Water-	- ECM	Soil -	ECM	Sediment ILV			
Calibration	Conc.	Peak Area	Conc.	Peak Area	Conc.	Peak Area		
Curve Data	(ng/mL)	counts	(ng/mL)	counts	(ng/mL)	counts		
	0.250	1392	0.250	3162	0.255	542		
	0.500	2822	0.500	5524	0.510	1290		
	1.000	6097	1.000	9628	1.020	1930		
	2.500	13767	2.500	25066	2.550	6120		
	5.000	30058	5.000	47982	5.100	11900		
	10.000	53797	10.000	98016	10.200	25800		
	25.000	147908	25.000	259588	25.500	72300		
	50.000	246049	50.000	484692	51.000	135000		

Peak Area from Table 5, p. 25; and Appendix E, Appendix A, Tables 5A-5B, p. 72 of MRID 49168201





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