Test Material: Flumioxazin

MRID: 44295046

Title: Field Dissipation Study with V-53482 WDG Herbicide in Iowa

MRID: 44295042

Title: Independent Laboratory Validation of the Method for Quantitation of Flumioxazin in Soil

EPA PC Code: 129034

OCSPP Guideline: 850.6100

For CDM Smith

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

Date: 5/19/15

Secondary Reviewer: Lisa Muto **Signature:** Lisa Muto

Date: 5/19/15

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

Date: 5/19/15

Analytical method for flumioxazin in soil

Reports: ECM: EPA MRID No.: 44295046 (Appendix II, pp. 76-89). Pensyl, J. (pp. 80, 87). 1991. DETERMINATION OF V-53482 RESIDUES IN SOIL, RESIDUE METHOD RM-30D. ECM report prepared by Chevron Chemical Company, Richmond, California; 7 pages (Appendix II, pp. 76, 83). Chevron File No.: 740.01/V-53482. Method dated January 21, 1991, revised March 6, 1991 and August 5, 1991. Chevron Chemical Company became part of Valent U.S.A. Corporation in November, 1991 (p. 8). Study report (MRID 44295046) prepared by Valent U.S.A. Corporation, Dublin, California, sponsored by Sumitomo Chemical Company, Ltd., Osaka, Japan, and submitted by Valent U.S.A. Corporation, Dublin, California; 464 pages (pp. 2-3; Appendix I, pp. 66-67). Valent Project No.: VP-1011.
ILV: EPA MRID No. 44295042. Kruplak, J. 1995. Independent Laboratory Validation of the Method for Quantitation of Flumioxazin in Soil. Report prepared by Analytical Development Corporation (ADC), Colorado Springs, Colorado, sponsored and submitted by Valent U.S.A. Corporation, Walnut Creek, California; 67 pages. ADC Project and Report No.: 1550-1. Final report issued December 29, 1995.

Document No.: MRIDs 44295046 & 44295042

Guideline: 850.6100

Statements: ECM: The study was conducted in compliance with USEPA Good Laboratory Practice (GLP) standards, with minor exceptions (p. 3 of MRID 44295046). Signed and dated Data Confidentiality, GLP, and Quality Assurance statements were provided (pp. 2-3, 6-7). The Authenticity Certification statement provided does not specify that the study report provides a true and accurate record of the results obtained (p. 5).
ILV: The study was conducted in compliance with USEPA GLP standards, with minor exceptions (p. 3 of MRID 44295042). Signed and dated Data Confidentiality, GLP, Quality Assurance, and Authenticity Certification statements were provided (pp. 2-5).

Classification: This analytical method is classified as supplemental. The experimental design of the ILV was inadequate to produce a successful validation of the ECM, with insufficient performance data at the LOQ (n = 2) and no performance data at 10x LOQ. The ECM uses GC-NPD, with no confirmatory method. For the ECM, performance data at 10x LOQ were not reported. The determinations of the LOQ and LOD were not based on scientifically acceptable procedures. For the ILV, the linearity of the quantitative method standard curve was not satisfactory. An updated ECM complete with all procedures and an internal validation of the confirmatory method developed by the independent laboratory was not provided.

PC Code: 129034

Reviewer: Larry Liu
2/16/18

Liu

For both MRIDs, page citations in this review refer to the uppermost set of page numbers located in the top right corner of each page of the MRID.

Executive Summary

This analytical method, Residue Method RM-30D, is designed for the quantitative determination of flumioxazin in soil using GC-NPD. The method is quantitative for the analyte at the stated LOQ of 0.01 mg/kg. The independent laboratory failed to validate the method for analysis of flumioxazin in a sandy clay loam soil because insufficient performance data were reported at the LOQ (n = 2) and no performance data were reported at 10x LOQ. The ECM uses GC-NPD analysis, with no confirmatory method. The independent laboratory developed a confirmatory method, but failed to analyze a sufficient number of samples to validate the method. For the primary quantitative method, the LOD was 0.005 mg/kg for the ECM validation and 0.004 mg/kg for the ILV. A LOD was not reported for the confirmatory method. The provided ECM was incomplete, lacking procedures described by the independent laboratory. The ECM validation did not provide performance data at 10x LOQ for the primary quantitative method and did not provide an internal validation of the confirmatory method developed by the independent laboratory.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Flumioxazin	44295046[1]	44295042[2]		Soil[3]	Initial: 21/01/1991[4] Revision: 05/08/1991	Valent U.S.A. Corporation	GC-NPD	0.01 mg/kg

1 No performance data at 10x LOQ.

2 Only two recoveries at the LOQ and no performance data at 10x LOQ.

3 For the ECM, untreated control soil segments of 0-30, 30-60, and 60-90 cm depths were characterized as sandy clay loam, loam, and sandy clay loam, respectively. For the ILV, the soil matrix was untreated, control, sandy clay loam soil (0-7.5 cm) from the same terrestrial field dissipation study used to support the ECM validation.

4 This ECM is a modification of a method developed by Sumitomo Chemical Co., Ltd. (Report No. ER-MT-8939, November 24, 1989; Appendix II, pp. 76, 80 of MRID 44295046).

I. Principle of the Method

Soil (10 g) was fortified with flumioxazin (V-53482) in acetone for procedural recoveries (Appendix II, pp. 77-78 of MRID 44295046). Control sandy clay loam (0-30 cm and 60-90 cm) and loam (30-60 cm) soil samples from a terrestrial field dissipation study conducted in Dallas Center, Iowa, were used for validation (p. 14; Appendix VIII, pp. 422-423, 425, 427). Florisil columns (19 mm x 300 mm) are prepared using Florisil (two parts 100-200 mesh:three parts 60-100 mesh, activated overnight at 130°C) and hexane:ethyl acetate (2:1, v:v; Appendix II, pp. 76, 79). Soil samples (10 g) are extracted twice with 50 mL of acetone:0.1N HCl (5:1, v:v; Appendix II, p. 78). For each extraction, soil and extraction solvent are shaken (reciprocating shaker, speed not specified) for 10 minutes, allowed to sit overnight at room temperature, shaken for 10 minutes, then filtered (Whatman No. 1 filter paper). The final filter cake is rinsed twice with extraction solvent (20 mL x 2). Extracts and rinsates are combined, mixed with 5% aqueous sodium chloride solution (150 mL), then partitioned twice with methylene chloride; initial partition 80 mL (added in two portions) and second partition 60 mL (Appendix II, pp. 78-79). Following each partition, the organic phase is filtered through sodium sulfate. Organic phases are combined and taken to dryness by rotary evaporation (≤40°C). Resulting residues are reconstituted in ethyl acetate (1 mL), diluted with hexane (2 mL), sonicated, and applied to a prepared Florisil column; three 3-mL rinses of the sample flask are also applied to the column. The loaded column is rinsed with hexane:ethyl acetate (2:1, v:v, 28 mL; total volume applied to column 40 mL). Flumioxazin (V-53482) is then eluted with hexane:ethyl acetate (2:1, v:v, 70 mL). The eluate is taken to dryness (rotary evaporation, ≤40°C), residues are reconstituted in acetone (5 mL x 3), again taken to dryness, and finally reconstituted in 0.5 mL acetone. The sample is immediately transferred to an autosampler vial and sealed to minimize evaporation losses.

Samples are analyzed using an Hewlett-Packard 5890A GC system, or equivalent, equipped with a nitrogen-phosphorus flame ionization detector (GC-NPD; Appendix II, pp. 77-78 of MRID 44295046). The following GC conditions were used: J & W Scientific DB-17 (50% phenyl-methyl silicone megabore) column (0.53 mm x 15 m, 1.0-µm film, injector temperature 275°C), temperature program [250°C (hold for 1 minute), 20°C/min. to 280°C (hold for 8 minutes)], and injection volume of 1 µL. Expected retention time for flumioxazin (V-53482) is *ca*. 7.7 minutes. A confirmatory method was not used.

ILV: The test compound and sandy clay loam soil matrix were supplied by Valent (p. 9 of MRID 44295042). The soil matrix was untreated, control soil (V1011-2U1, 0-7.5 cm) from the same terrestrial field dissipation study described above (Appendix I, pp. 59-60 of MRID 44295046). In addition to some labware substitutions, the independent laboratory performed the method as written with the following exceptions (pp. 9-12 of MRID 44295042). Following preparation of the Florisil column, the elution profile of flumioxazin was determined according to "Method note #4", a procedure not described in the provided ECM (Appendix II, p. 80 of MRID 44295046). A 1-mL aliquot of 1 µg/mL flumioxazin standard in acetone was taken to dryness, reconstituted in hexane:ethyl acetate (2:1, v:v, 3 mL), and applied to the Florisil column (pp. 11-12 of MRID 44295042). Three 3-mL aliquots of hexane:ethyl acetate (2:1, v:v) were applied to the column and the entire 12-mL application volume collected in one flask. An additional 28 mL of hexane:ethyl acetate (2:1, v:v) was applied and collected as 20-mL and 8-mL volumes in separate flasks. Flumioxazin was eluted with 70 mL of hexane:ethyl acetate (2:1, v:v). Then three additional 10-mL aliquots of hexane:ethyl acetate (2:1, v:v) were applied to the column and collected separately. All fractions were taken to dryness, reconstituted in acetone, and analyzed by GC-NPD for flumioxazin (p. 13). The elution profile procedure indicated that, for this ILV, the initial 12 mL + 28 mL of eluate would be discarded and the following 90 mL collected to ensure total collection of flumioxazin. The only modification to the ECM GC-NPD conditions was increasing the hydrogen flow rate from 3.5 to 4 mL/minute (pp. 13-14 of MRID 44295042; Appendix II, pp. 77-78 of MRID 44295046). The retention time of flumioxazin was 7.9 minutes (Appendix 1, pp. 25-28, 36, 38, 40, 42-50 of MRID 44295042).

As a confirmatory method, the independent laboratory also analyzed samples by GC-NPD using a J & W DB-5 column (0.53 mm x 15 m, 1.5-µm film), with all other GC conditions the same as for the DB-17 column (pp. 13-14 of MRID 44295042). The retention time of flumioxazin was 4.7 minutes (Appendix 1, pp. 30-33, 53, 55, 57, 59-67).

LOQ and LOD: In the ECM and ILV, the LOQ was not specified; the lowest fortification level was 0.01 mg/kg (µg/g, ppm; Table III, pp. 35-37 of MRID 44295046; Table 2, p. 18 of MRID 44295042). As part of the terrestrial field dissipation study report, the LOQ of the ECM was reported as 0.01 mg/kg (p. 22 of MRID 44295046). In the ECM, the LOD for flumioxazin was reported as 0.005 mg/kg for a 10 g soil sample (ppm; Appendix II, p. 80 of MRID 44295046). In the ILV, the LOD using the DB-17 column was calculated as 0.004 mg/kg (µg/g, ppm; Table 1, p. 17 of MRID 44295042). A LOD using the DB-5 column was not reported.

II. Recovery Findings

ECM (MRID 44295046): Procedural recoveries for sandy clay loam/loam soil from a terrestrial field dissipation study conducted in Dallas Center, Iowa, were reported to support validation of the ECM (p. 14; Appendix II, pp. 75-89; Appendix III, pp. 90-163; Appendix V, pp. 191-194). Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of flumioxazin in the soil at fortification levels of 0.01 mg/kg (ppm, LOQ, n =75) and 0.05 mg/kg (5x LOQ, n =4; p. 22; DER Attachment 2). Performance data at 10x LOQ were not reported. Flumioxazin was identified by retention time, a confirmatory method was not used. The soil matrix (0-90 cm) was characterized by A & L Mid West Laboratories, Inc., Omaha, Nebraska, according to depth at 0-30, 30-60, and 60-90 cm as sandy clay loam, loam, and sandy clay loam, respectively (Appendix VIII, pp. 422-423, 425, 427).

ILV (MRID 44295042): Only two recoveries were reported for each fortification level; therefore, meaningful statistics (mean, standard deviation, RSD) for the recovery results per fortification level and soil matrix could not be generated. Individual recovery results were within 70-120% for the primary quantitative method (DB-17 column) and the confirmatory method (DB-5 column) in sandy clay loam soil at fortification levels of 0.01 mg/kg (ppm, LOQ) and 0.05 mg/kg (5x LOQ; Table 2, p. 18). Performance data at 10x LOQ were not reported. One validation run using the DB-17 column was conducted and considered successful (p. 8). Extracts from the initial validation (DB-17 column) were then re-injected using the DB-5 column and the results considered successful. The sandy clay loam soil matrix, supplied by Valent, was untreated, control soil (V1011-2U1, 0-7.5 cm) from the same terrestrial field dissipation study used to generate the ECM validation data (p. 9).

Table 2. Initial Validation Method Recoveries for Flumioxazin in Sandy Clay Loam/Loam Soil[1]

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Flumioxazin (V-53482)	0.01 (LOQ)	75	63-130	94	14.9	15.8
	0.05	4	64-77	70	5.56	7.91

Data (recovery results, corrected for any residues detected in matrix control samples) were obtained from Table III, pp. 35-38; Appendix III, pp. 91-95 of MRID 44295046 and DER Attachment 2 (mean, SD, and RSD for 0.01 mg/kg; SD for 0.05 mg/kg).

1 USDA classifications (Appendix VIII, pp. 422-423, 425, 427 of MRID 44295046). Untreated control soil segments of 0-30, 30-60, and 60-90 cm depths were characterized as sandy clay loam, loam, and sandy clay loam, respectively.

Table 3. Independent Validation Method Recoveries for Flumioxazin in Sandy Clay Loam Soil[1]

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Flumioxazin (V-53482)	Quantitative Method (DB-17 column)					
	0.01 (LOQ)	2	87, 90	--[2]	--	--
	0.05	2	85, 87	--	--	--
	Confirmatory Method (DB-5 column)					
	0.01 (LOQ)	2	92, 89	--	--	--
	0.05	2	85, 84	--	--	--

Data (recovery results, corrected for any residues detected in matrix control samples) were obtained from Table 2, p. 18 of MRID 44295042.

1 USDA classification; soil matrix (V1011-2U1) was untreated, control soil (0-7.5 cm) from the same terrestrial field dissipation study used to support the ECM validation (p. 9 of MRID 44295042; Appendix I, pp. 59-60 of MRID 44295046).

2 Not applicable; meaningful statistics could not be generated because n = 2.

III. Method Characteristics

In the ECM and ILV, the LOQ was not specified; the lowest fortification level was 0.01 mg/kg (µg/g, ppm), with no justification (Table III, pp. 35-37 of MRID 44295046; Table 2, p. 18 of MRID 44295042). As part of the terrestrial field dissipation study report, the LOQ of the ECM was reported as 0.01 mg/kg (p. 22 of MRID 44295046). In the ECM, the LOD for flumioxazin was reported as 0.005 mg/kg (ppm), with no justification (Appendix II, p. 80 of MRID 44295046). In the ILV, the LOD using the DB-17 column (quantitative method) was calculated as 0.004 mg/kg (µg/g, ppm; Table 1, p. 17 of MRID 44295042). A LOD for flumioxazin using the DB-5 column (confirmatory method) was not reported. In the ILV, the LOD was defined as the calculated concentration equivalent to a peak height response of 5x the baseline noise.

Table 4. Method Characteristics for Flumioxazin (V-53482) in Soil

			Flumioxazin
Limit of Quantitation (LOQ)			0.01 mg/kg (ppm)
Limit of Detection (LOD)	ECM:		0.005 mg/kg
	ILV:	DB-17:	0.004 mg/kg
		DB-5:	Not reported.
Linearity (calibration curve r^2 and concentration range)	ECM:		Coefficient of determination (r^2) not reported. Coefficients of variation (CV) of calibration standard peak response factors normalized to 1.0 µg/mL were ± 13.0%.[1]
	ILV:[2]	DB-17:	r^2 = **0.9891**
		DB-5:	r^2 = 0.9999
	Range:		0.1-2.0 µg/mL
Repeatable	ECM:		Yes at LOQ and 5x LOQ in sandy clay loam/loam soil. No performance data at 10x LOQ.
	ILV:		Undetermined. Fortifications at LOQ and 5x LOQ performed at n = 2. No performance data at 10x LOQ.
Reproducible			Undetermined. ILV performed at n = 2 at LOQ and 5x LOQ.
Specific	ECM:		Undetermined. A confirmatory method was not used; flumioxazin was identified by retention time using GC-NPD (DB-17 column). Residues were occasionally detected in the matrix blank control samples at 40-80% of the LOD (Appendix III, pp. 98-100, 118, 120-123 of MRID 44295046).
	ILV:		Yes. Primary quantitative method (DB-17 column): interferences in the matrix control blank samples detected at 5% of the LOD (Tables 1-2, pp. 17-18 of MRID 44295042). Confirmatory method (DB-5 column): interferences in the reagent blank sample detected at 2% of LOQ; LOD using DB-5 column was not reported.

Data were obtained from p. 22; Appendix II, pp. 77-78, 80-82; Appendix III, pp. 90-163; Appendix V, pp. 191-196 of MRID 44295046; Tables 1-2, pp. 17-18; Figure 2, p. 20; Appendix 1, pp. 37, 39, 41, 54, 56, 58 of MRID 44295042; and DER Attachment 2.

Linearity is satisfactory when $r^2 \geq 0.995$.

1 CV was required to be ≤10% unless authorized by the Study Director (p. 22; Appendix III, pp. 90-163; Appendix V, pp. 191-196).

2 Standard curve plots and coefficients of determination (r^2) were generated by the reviewer using provided individual calibration standard data (Table 1, p. 17 of MRID 44295042; DER Attachment 2).

IV. Method Deficiencies and Reviewer's Comments

1. The ECM study author reported that this method is a modification of a method developed by Sumitomo Chemical Co., Ltd. (Report No. ER-MT-8939, November 24, 1989; Appendix II, pp. 76, 80 of MRID 44295046).

2. The soil matrices were from a terrestrial field dissipation study conducted in Dallas Center, Iowa (p. 14 of MRID 44295046). The 0-30, 30-60, and 60-90 cm depths were characterized as sandy clay loam, loam, and sandy clay loam soils, respectively (Appendix VIII, pp. 422-428). Percentages of clay were similar for the three soil depths at 26-28%, but percentages of organic matter were 3.8%, 2.3%, and 0.5% at the 0-30, 30-60, and 69-90 cm depths, respectively. For the terrestrial field dissipation study, procedural recoveries were performed using soil samples from 90-cm cores divided into 12 segments; 0-7.5, 7.5-15, 15-22.5, 22.5-

30, 30-37.5, 37.5-45, 45-52.5, 52.5-60, 60-67.5, 67.5-75, 75-82.5, and 82.5-90 cm; however, procedural recoveries obtained during site qualification analyses were not included with the definitive study results (Table III, p. 35; Appendix I, p. 62). Except for fortifications performed during the site qualification analyses, depths for the fortified soil samples were assigned by the reviewer using the study report labeling scheme (Appendix I, pp. 59-60; Appendix III, pp. 90-163; Appendix V, pp. 190-196). It was unclear as to what soil depths were used for the fortifications performed during the site qualification analyses (Appendix III, pp. 91-95). There were no significant differences in the procedural recoveries at the 0-30, 30-60, and 60-90 cm depths; therefore, for this DER, recoveries were combined according to fortification level (0.01 and 0.05 ppm) so as to include the fortifications performed during the site qualification analyses in the statistics (mean, SD, RSD) for the ECM validation data (DER Attachment 2).

Residue Method RM-30D was reportedly validated prior to the terrestrial field dissipation study using uncharacterized soil (pp. 20-21 of MRID 44295046). Mean recoveries and RSDs (coefficients of variation) were 101 ± 2.29% at 0.01 m/kg (ppm, n =3) and 100 ± 16.0% at 0.05 mg/kg (n = 6). No additional information or supporting data were provided.

3. The experimental design of the ILV was inadequate to produce a successful validation of the ECM. For the ILV, only two recoveries were reported for both the LOQ and 5x LOQ fortification levels, with no performance data at 10x LOQ (Table 2, p. 18 of MRID 44295042).

4. The ILV study report includes the description of "Method note #4", a testing procedure to determine the elution profile of flumioxazin for each batch of prepared Florisil (see **I. Principle of the Method**, ILV: above), that was not included in the provided ECM (Appendix II, p. 80 of MRID 44295046; pp. 12-13 of MRID 44295042).

5. The ECM used GC-NPD/DB-17 column for analysis of flumioxazin (Appendix II, pp. 77-78 of MRID 44295046). Flumioxazin was identified only by retention time; a confirmatory method was not used. The independent laboratory developed a confirmatory method using GC-NPD/DB-5 column, but Valent did not internally validate the confirmatory method.

6. For the ECM using GC-NPD/DB-17 column, performance data at 10x LOQ were not reported.

7. The determination of the LOQ and LOD were not based on scientifically acceptable procedures as defined in 40 CFR Part 136, Appendix B. In the ECM and ILV, the LOQ was not specified; the lowest fortification level was 0.01 mg/kg (µg/g, ppm), with no justification (Table III, pp. 35-37 of MRID 44295046; Table 2, p. 18 of MRID 44295042). As part of the terrestrial field dissipation study report, the LOQ of the ECM was reported as 0.01 mg/kg (p. 22 of MRID 44295046). In the ECM, the LOD for flumioxazin was reported as 0.005 mg/kg (ppm), with no justification (Appendix II, p. 80 of MRID 44295046). In the ILV, the LOD using the DB-17 column was calculated as 0.004 mg/kg (µg/g, ppm; Table 1, p. 17 of MRID 44295042). A LOD for flumioxazin using the DB-5 column was not reported. In the ILV, the LOD was defined as the calculated concentration equivalent to a peak height response of 5x the baseline noise.

Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples. Additionally, the lowest toxicological level of concern in soil was not reported. An LOQ above toxicological levels of concern results in an unacceptable method classification.

8. For the ILV, standard curve plots with regression curve analysis and coefficients of determination (r^2) were not provided. Linearity was determined using coefficients of variation (CV) of calibration standard peak response factors normalized to 1.0 µg/mL and were ± 8.9% (Table 1, p. 17; Appendix 1, pp. 35, 52 of MRID 44295042). Standard curve plots and coefficients of determination (r^2) were generated by the reviewer using provided individual calibration standard data (Table 1, p. 17; DER Attachment 2).

   The linearity of the quantitative method (GC-NPD/DB-17 column) standard curve was not satisfactory; the coefficient of determination (r^2) was not ≥0.995.

9. For the ECM, reagent blank and chromatograms of soil fortified at 0.05 ppm (5× LOQ) were not provided; 10× LOQ fortifications were not performed (Appendix II, pp. 75-89; Appendix VI, pp. 199-200, 208-209 of MRID 44295046). For the calibration standards, only chromatograms of a 1.0 µg/mL standard were provided (calibration standard range 0.1-2.0 µg/mL; Appendix II, p. 81; Appendix III, pp. 90-163; Appendix V, pp. 190-196; Appendix VI, pp. 198, 207). Standard curve plots with regression curve analysis and coefficients of determination (r^2) were not provided. Linearity was determined using coefficients of variation (CV) of calibration standard peak response factors normalized to 1.0 µg/mL and were ± 13.0%. The CV was required to be ≤10% unless authorized by the Study Director (p. 22; Appendix III, pp. 90-163; Appendix V, pp. 191-196). Standard curve plots were not generated by the reviewer as only normalized peak response factors were reported.

10. For both the ECM and ILV, sample recoveries were corrected when residues were found in the matrix controls. For the ECM, residues were occasionally detected in the matrix blank control samples at 40-80% of the LOD (Appendix III, pp. 98-100, 118, 120-123 of MRID 44295046). For the ILV using the primary quantitative method (DB-17 column), interferences in the matrix control blank samples were detected at 5% of the LOD (Tables 1-2, pp. 17-18 of MRID 44295042). For the ILV using the confirmatory method (DB-5 column), interferences in the reagent blank sample were detected at 2% of the LOQ; the LOD using the DB-5 column was not reported.

11. The ECM was used in a terrestrial dissipation study (pp. 14, 20; Appendix II, pp. 75-89 of MRID 44295046). However, insufficient information was provided to determine if the LOQ is less than 10% of the expected or actual peak concentration of the test compound in the field. Following a single application at 42.5 g a.i./A, the maximum concentration of flumioxazin detected in the 0-7.5 cm soil depth was 0.074 mg/kg (ppm; p. 23; Table II, p. 33).

12. It was reported for the ILV that a set of seven samples (one reagent blank, two matrix controls, and four fortified samples) required *ca.* 10.5 hours over a 3-day period to complete, assuming the Florisil elution profile for flumioxazin was already characterized/optimized (p. 16; Table 2, p. 18 of MRID 44295042). This time consisted of 2 hours for setup (Day 1), 7 hours for extraction and cleanup procedures (Day 2), and 1.5 hours for calculations (Day 3).

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.

Attachment 1: Chemical Names and Structures

Flumioxazin (V-53482, S-53482)

IUPAC Name:	N-(7-fluoro-3,4-dihydro-3-oxo-4-prop-2-ynyl-2H-1,4-benzoxazin-6-yl)cyclohex-1-ene-1,2-dicarboxamide. 7-Fluoro-6-[(3,4,5,6-tetrahydro)phthalimido]-4-(2-propynyl)-1,4-benzoxazin-3(2H)-one.
CAS Name:	2-[7-Fluoro-3,4-dihydro-3-oxo-4-(2-propynyl)-2H-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1H-isoindole-1,3(2H)-dione. [2-[7-Fluoro-3,4-dihydro-3-oxo-4-(2-propynyl)-2H-1,4-benzoxazin-6-yl]-4,5,6,7-tetrahydro-1H-isoindole-1,3(2HO-dione)].
CAS Number:	103361-09-7
SMILES String:	Fc1cc2OCC(=O)N(CC#C)c2cc1N3C(=O)C(CCCC4)=C4C3=O