Test Material:	Flumioxazin
MRID:	48402402
Title:	Terrestrial Field Soil Dissipation of Chateau Herbicide (Flumioxazin) on Bare Ground in Illinois
MRID:	49657401
Title:	Independent Laboratory Validation of Valent U.S.A. Corporation's Residue Analytical Method for the Determination of Flumioxazin, THPA, and HPA in Soil (Method Number: RM-30S-1-1)
EPA PC Code:	129034
OCSPP Guideline:	850.6100

For CDM Smith

Primary Reviewer: Lynne Binari **Signature:** 

Date: 8/18/15

Secondary Reviewer: Lisa Muto **Signature:** 

Date: 8/18/15

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

Date: 8/18/15

Analytical methods for flumioxazin and its transformation products THPA, HPA, SAT-482-HA, APF, and DAPF in soil

Reports: ECM: EPA MRID No.: 48402402. Green, C.A. 2010. Terrestrial Field Soil Dissipation of Chateau Herbicide (Flumioxazin) on Bare Ground in Illinois. ECM-1 title: DETERMINATION OF FLUMIOXAZIN, THPA, HPA, AND SAT-482-HA IN SOIL, Method: RM-30S-1/RM-30S-1-1 (Appendix II, pp. 125, 150). ECM-1 report prepared by Valent U.S.A. Corporation, Dublin, California; 32 pages (24 pages for RM-30S-1, plus excerpt pages from RM-30S-1-1). ECM-1 dated November 21, 2002. ECM-2 title: DETERMINATION OF APF AND DAPF IN SOIL, Method: RM-30S-2-1 (Appendix II, p. 157). ECM-2 report prepared by Valent U.S.A. Corporation, Dublin, California; 13 pages. ECM-2 report dated May 19, 2003. Study report (MRID 48402402) prepared, sponsored, and submitted by Valent U.S.A. Corporation, Dublin, California/Walnut Creek, California/Seymour, Illinois; 467 pages (p. 1; Appendix I, pp. 92-93). Valent Project No.: V-25144. Final report issued December 14, 2010.

ILV: EPA MRID No. 49657401. Schoenau, E. 2015. Independent Laboratory Validation of Valent U.S.A. Corporation's Residue Analytical Method for the Determination of Flumioxazin, THPA, and HPA in Soil (Method Number: RM-30S-1-1). Report prepared by Golden Pacific Laboratories, Fresno, California, sponsored and submitted by Valent U.S.A. Corporation, Dublin, California; 98 pages (pp. 1, 7). GPL Study No.: 150597. Final report issued June 15, 2015.

Document No.: MRIDs 48402402 & 49657401

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 48402402). Signed and dated Data Confidentiality, GLP, and Quality Assurance statements were provided (pp. 2-3, 5-6). 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 49657401). Signed and dated Data Confidentiality, GLP, and Quality Assurance statements were provided (pp. 2-4). The certification of the authenticity of the study report is included in the Quality Assurance Statement (p. 4).

Classification: Analytical method RM-30S-1/RM-30S-1-1 is classified as supplemental. There was no ILV for analyte SAT-482-HA (Method: RM-30S-1). The determinations of the LOQ and LOD were not based on scientifically acceptable procedures. The ILV was not conducted with a soil matrix of the most difficult analytical sample condition. For the ECM, there were no performance data at 10x LOQ for any analyte/soil matrix, except for two recoveries for flumioxazin in a loam soil. For the ECM, analysis of THPA in a silty clay loam soil did not meet OCSPP guidelines at the LOQ (mean 128%) and 5x LOQ (mean 133%).

Analytical method RM-30S-2-1 is classified as supplemental. There was no ILV. The determinations of the LOQ and LOD were not based on scientifically acceptable procedures. There were no performance data at 10x LOQ.

PC Code: 129034

Reviewer: Larry Liu
8/27/18

For MRIDs 48402402 and 49657401, page citations in this review refer to the page numbers located in the uppermost right and bottom right corners, respectively, of each page of the MRID.

Executive Summary

As part of a terrestrial field dissipation study, two ECMs were appended to the study report, plus information regarding revision of one of the ECMs. The first analytical method, Valent Method RM-30S-1, is designed for the quantitative determination of flumioxazin in soil using GC/MS and the quantitative determination of flumioxazin products THPA, HPA, and SAT-482-HA in soil using LC/MS/MS. The method is quantitative for the analytes at the stated LOQ of 0.02 mg/kg (ppm). The independent laboratory validated Method RM-30S-1-1 for analysis of flumioxazin, THPA, and HPA at the LOQ and 10x LOQ in a loamy sand soil matrix after one trial. No major modifications were made by the independent laboratory. The ILV was not conducted with a soil matrix of the most difficult analytical sample condition. An ILV for analysis of SAT-482-HA in soil was not performed.

The second analytical method, Valent Method RM-30S-2-1, is designed for the determination of flumioxazin products APF and DAPF in soil using GC/MS. An ILV for analysis of APF and DAPF in soil was not performed. There were no performance data at 10x LOQ.

Table 1. Analytical Method Summary

<table>
<tr><th rowspan="2">Analyte(s) by Pesticide</th><th colspan="2">MRID</th><th rowspan="2">EPA Review</th><th rowspan="2">Matrix</th><th rowspan="2">Method Date (dd/mm/yyyy)</th><th rowspan="2">Registrant</th><th rowspan="2">Analysis</th><th rowspan="2">Limit of Quantitation (LOQ)</th></tr>
<tr><th>Environmental Chemistry Method</th><th>Independent Laboratory Validation</th></tr>
<tr><td>Flumioxazin</td><td rowspan="6">48402402</td><td rowspan="3">49657401</td><td rowspan="6"></td><td rowspan="6">Soil[1]</td><td rowspan="3">RM-30S-1-1[2] No date (Original method RM-30S-1 21/11/2002)</td><td rowspan="6">Valent</td><td>GC/MS & GC/NPD[3]</td><td rowspan="6">0.02 mg/kg (ppm)</td></tr>
<tr><td>THPA</td><td rowspan="3">LC/MS/MS</td></tr>
<tr><td>HPA</td></tr>
<tr><td>SAT-482-HA</td><td rowspan="3">No ILV</td><td>RM-30S-1 21/11/2002</td></tr>
<tr><td>APF</td><td rowspan="2">RM-30S-2-1 19/05/2003</td><td rowspan="2">GC/MS</td></tr>
<tr><td>DAPF</td></tr>
</table>

1 Valent provided the independent laboratory with a loamy sand (7% clay, 0.45% organic matter) soil from California (pp. 19, 30; Appendix A, p. 52; Appendix B, p. 55 of MRID 49657401). The ECM validations were performed with an uncharacterized California soil, while terrestrial field dissipation procedural recoveries were performed using loam (27% clay, 3.5% organic matter), silty clay loam (39% clay, 1.0% organic matter), and clay loam (29% clay, 0.5% organic matter) soil matrices (Appendix II, pp. 138, 165; Appendix V, p. 204 of MRID 48402402).

2 Method version which was validated by the ILV.

3 GC/NPD analysis was not validated by the ILV.

I. Principle of the Method

The soil used for the ECM validations was only described as California soil (Appendix II, pp. 138, 165). Soil from an untreated plot, as part of a terrestrial field dissipation study conducted in Champaign County, Illinois, and used for procedural recoveries, was characterized as loam, silty clay loam, and clay loam at the 0-30, 30-60, and 60-90 cm soil depths, respectively (p. 12; Appendix V, p. 204).

Flumioxazin, THPA, HPA, and SAT-482-HA (Method: RM-30S-1/RM-30S-1-1): Soil (20 ± 0.1 g) was fortified with a standard solution of flumioxazin in acetone and a mixed standard solution of THPA/THPA 2-Na, HPA, and SAT-482-HA in methanol for procedural recoveries (Appendix II, pp. 127, 133 of MRID 48402402). Soil (20 ± 0.1 g) is extracted once with acetone:1% HCl (9:1, v:v, 80 mL) followed by once with acetonitrile:1% HCl (8:2, v:v, 80 mL) by shaking on a linear (platform) shaker (Erbach) for 30 minutes for each extraction (Appendix II, pp. 129-130, 133). Soil and extract are separated by vacuum filtration (Büchner funnel, Whatman GF/A filter). Extracts are combined and brought to volume (160 mL, but may be adjusted to 180 mL) with acetone.

For flumioxazin, 80 mL of the combined extract (50% of total extract volume, Fraction A) is transferred to a 250-mL round-bottom flask and concentrated by rotary evaporation (water bath, <40°C) to remove the solvent (Appendix II, p. 133). The resulting aqueous residue (*ca*. 15 mL) is transferred to a separatory funnel using deionized water (50 mL; Appendix II, p. 134). The sample is partitioned once with hexane (75 mL) and vigorous shaking for 1 minute. The organic phase is filtered through sodium sulfate (*ca*. 30 g) into a 250-mL round-bottom flask, with the sodium sulfate rinsed with hexane (25 mL) which is combined with the sample. The sample is concentrated by rotary evaporation (water bath, <40°C) to *ca.* 30-40 mL, then transferred to a 100-mL round-bottom flask using ethyl acetate (10-15 mL). Rotary evaporation is continued to just dryness, with the residues reconstituted in toluene (1.0 mL) and transferred to an autosampler vial for GC/MS analysis.

For Method: RM-30S-1, samples are analyzed using an Hewlett-Packard (HP) 6890 GC system equipped with an HP 5973 mass selective detector (MSD; Appendix II, pp. 131, 136). The following GC conditions are used: J & W Scientific DB-1 column (0.32 mm x 30 m, 0.25 µm film thickness), column oven temperature program [200°C (hold for 1.0 min.), 15°C/min. to 320°C, final time 5.0 min., total run time 14.0 min.], injector temperature 280°C, and injection volume 0.5 µL. The following MSD conditions are used: detector temperature 300°C and selected ion monitoring (SIM) at *m/z* 354 for flumioxazin. Retention time was *ca*. 7.9 for flumioxazin (Appendix II, pp. 131, 140-141).

A complete version of Method: RM-30S-1-1 was not provided by Valent, only excerpt pages (p. 19; Appendix II, pp. 149-156). The following are alternate GC/MSD conditions: Restek Rtx-200 column (0.32 mm x 30 m, 0.5 µm film thickness), column oven temperature program [200°C (hold for 1.0 min.), 20°C/min. to 300°C, final time 8.0 min., total run time 14.0 min.]. Retention time was *ca*. 9.6 for flumioxazin (chromatograms not provided).

For Method: RM-30S-1-1, samples can also be analyzed using an HP 6890 GC system with a nitrogen-phosphorous detector (GC-NPD) and an on-column injector (Appendix II, pp. 149-156). The following GC conditions are used: Restek Rtx-200 column (0.53 mm x 15 m, 0.5 µm film thickness), on-column temperature program [130°C (hold for 0.1 min.), 1,250°C/min. to 300°C, final time 10 min.] or column oven temperature program [125°C, 30°C/min. to 280°C, final time 13

min., total run time 18.2 min.], detector temperature 280°C, and on-column injection volume 1.5 µL. Retention time was *ca*. 12.7 for flumioxazin (chromatograms not provided).

For THPA, HPA, and SAT-482-HA, 40 mL of the combined extract (25% of total extract volume, Fraction B) is transferred to a 100-mL round-bottom flask and concentrated by rotary evaporation (water bath, <40°C) to remove all solvent (Appendix II, pp. 133-134). The resulting aqueous residue (*ca*. 7-8 mL) is transferred to a centrifuge tube using deionized water (2 x 1 mL). The sample is centrifuged (10-12 minutes, speed not specified), then the supernatant is applied, under gravity flow, to a C_{18} solid-phase extraction (SEP) cartridge (Bakerbond Octadecyl, 6 mL, 1,000 mg) pre-conditioned with methanol and deionized water (Appendix II, pp. 130, 134-135). The loaded SPE cartridge is rinsed sequentially with deionized water (2 mL) and methanol:water (10:90, v:v, 1.5 mL), then THPA and HPA are eluted with methanol:water (30:70, v:v, 6.0 mL) containing 0.005M ammonium formate (NH_4OOCH). An aliquot of the eluate is transferred to an autosampler vial for LC/MS/MS analysis.

The 100-mL round-bottom flask used for Fraction B is rinsed with methanol:water (30:70, v:v, 5 mL) and the rinsate transferred to the same centrifuge tube also previously used for Fraction B (Appendix II, pp. 134-135). The tube is shaken to dislodge the pellet, vortexed (*ca*. 1 minute), then centrifuged (10-12 minutes, speed not specified). The supernatant is applied, under gravity flow, to the SPE cartridge previously used for isolation of THPA and HPA. The remaining pellet is resuspended in methanol:water (1:1, v:v, 5 mL) with vortexing (*ca*. 1 minute), centrifuged, and the supernatant also applied to the SPE cartridge (Appendix II, pp. 132, 135-136). SAT-482-HA is eluted with methanol:water (1:1, v:v, 2 x 5 mL) containing 0.01M formate buffer. A two-fold dilution of the sample is recommended; combine 0.75 mL of methanol:water (1:1, v:v) with formate buffer and 0.75 mL of the sample in an autosampler vial for LC/MS/MS analysis.

For Method: RM-30S-1, samples are analyzed using an HP 1100 HPLC system and an Applied Biosystems API 2000 MS with electrospray ionization (ESI; Appendix II, pp. 129, 132-133). The following LC conditions were used: Luna C_{18} column (3 mm x 50 mm, 3 µm, column temperature 35°C), mobile phase of (A) aqueous formate buffer and (B) formate buffer in methanol [percent A:B (v:v) at 0.0-1.0 min. 70:30, 7.0-9.0 min. 20:80, 10-15 min. 70:30 for THPA and HPA; percent A:B (v:v) at 0.0-1.0 min. 60:40, 9.0-12.0 min. 10:90, 13-18 min. 60:40 for SAT-482-HA], and injection volume of 15 µL. The following MS/MS conditions were used: negative ion mode for THPA and HPA, positive ion mode for SAT-482-HA, and multiple reaction monitoring (MRM). Analytes are identified using single ion pair transitions as follows: *m/z* 169.0→125.3 for THPA, *m/z* 170.8→127.4 for HPA, and *m/z* 375.0→221.0 for SAT-482-HA. Retention times were *ca*. 3.3, 6.2, and 8.2 minutes for THPA, HPA, and SAT-482-HA, respectively (Appendix II, pp. 142-145). The same LC/MS/MS conditions are used for Method: RM-30S-1-1; however, analysis of SAT-482-HA is not included.

APF and DAPF (Method: RM-30S-2-1): This is a modification of Method: RM-30S-2 which reduces the soil sample size extracted and volume of extraction solvent used in the initial extraction (Appendix II, p. 157). Soil (10 ± 0.1 g) was fortified with a mixed standard solution of APF and DAPF in acetone for procedural recoveries (Appendix II, pp. 158, 162). Soil (10 ± 0.1 g) is extracted twice with 0.2M phosphate buffer (10 mL) plus methanol (40 mL) by shaking on a linear shaker (Erbach) for 15 minutes for each extraction (Appendix II, pp. 159-160, 162). Soil and extract are separated by vacuum filtration (Büchner funnel, Whatman GF/A filter). Extracts are combined and transferred to a 500-mL round-bottom flask with 0.2M phosphate buffer (10 mL), then concentrated by rotary evaporation (water bath, <30°C) to remove the solvent. The resulting

aqueous residue (*ca*. 25 mL) is transferred to a separatory funnel using deionized water (10 mL). The sample is partitioned three times with methylene chloride (100 mL per extraction) and vigorous shaking for 1 minute per extraction. Organic phases are filtered (Whatman No. 1 filter paper) and combined in a 500-mL round-bottom flask. The sample is concentrated by rotary evaporation (water bath, <30°C) to *ca.* 30-40 mL, octanol (3 drops) is added as stabilizer, then the sample is transferred to a 100-mL round-bottom flask using ethyl acetate (10-15 mL). Rotary evaporation is continued until only the octanol remains, with the residues reconstituted in toluene (1.0 mL) and transferred to an autosampler vial for GC/MS analysis.

Samples are analyzed using an HP 6890 GC system equipped with an HP 5973 MSD (Appendix II, pp. 161, 163). The following GC conditions are used: J & W Scientific DB-1 column (0.32 mm x 30 m, 0.25 µm film thickness), column oven temperature program [160°C (hold for 1.0 min.), 15°C/min. to 220°C, 25°C/min. to 320°C, final time 6.0 min., total run time 15.0 min.], injector temperature 300°C, and injection volume 0.5 µL. The following MSD conditions are used: detector temperature 300°C and selected ion monitoring (SIM) at *m/z* 220 for APF and *m/z* 222 for DAPF. Retention times were *ca*. 4.6 and 4.5 minutes for APF and DAPF, respectively (Appendix II, p. 161, 166-167).

ILV of Method: RM-30S-1-1: Test compounds and characterized loamy sand soil from California were supplied by Valent (pp. 12, 15; Appendix A, p. 52; Appendix B, p. 55 of MRID 49657401). The independent laboratory performed the method as written with the following modifications: deionized (DI) water was replaced with HPLC-grade water; the following differing GC/MS conditions were used for flumioxazin analysis: HP 5890 Series II GC, equivalent Supelco SPB-1 column, injection volume of 1 µL, and ionization source/polarity was specified as electron impact/positive; for the THPA/HPA (Fraction B) work up, samples were concentrated at <30°C with the aqueous residue transferred to a 15-mL centrifuge tube and adjusted to 10-mL volume with HPLC-grade water; at the end of THPA/HPA SPE clean-up, vacuum was applied to pull off all liquid from the cartridge and samples were brought to 7 mL with methanol:water (30:70, v:v) containing 0.005M ammonium formate; the following differing LC/MS/MS conditions were used for THPA and HPA analysis: Applied Biosystems Sciex API 4000 LC/MS/MS, addition of Phenomenex Security Guard Cartridge C_{18} (2.0 mm x 4 mm), column temperature 50°C, injection volume 10 µL, and monitoring of the following ion pair transitions: *m/z* 168.9→124.8 for THPA and *m/z* 170.9→126.9 for HPA (pp. 20-24, 26). Retention times were *ca*. 8.5, 3.0, and 4.3 minutes for flumioxazin, THPA, and HPA, respectively.

LOQ and LOD: In the ECMs, the LOQ and LOD were 0.02 mg/kg (ppm) and 0.01 mg/kg, respectively, for flumioxazin, THPA, HPA, SAT-482-HA, APF, and DAPF (Appendix II, p. 138, 156, 164 of MRID 48402402). In the ILV, the LOQ and LOD were also 0.02 mg/kg and 0.01 mg/kg, respectively, for flumioxazin, THPA, and HPA; SAT-482-HA, APF, and DAPF were not included in the ILV (p. 30 of MRID 49657401).

II. Recovery Findings

ECM RM-30S-1/RM-30S-1-1 (Appendix II, pp. 125-156 of MRID 48402402): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of flumioxazin and its transformation products THPA, HPA, and SAT-482-HA at fortification levels of 0.02 mg/kg (LOQ) and 0.10 mg/kg (5x LOQ) in an uncharacterized California soil and in loam, silty clay loam, and clay loam soils, except for THPA at both fortification levels in

the silty clay loam soil (means of 128% at 0.02 mg/kg and 133% at 0.10 mg/kg; Tables XIII-XV, pp. 80-86; Appendix II, pp. 138, 146-148; and DER Attachment 2). Except for two recoveries for flumioxazin at 0.20 mg/kg (10x LOQ; 87.9%, 88.8%) in the loam soil, no performance data were provided at 10x LOQ for any analyte/soil matrix. The number of samples was insufficient for all analytes at 0.02 mg/kg (LOQ, n = 3) in the uncharacterized soil, for flumioxazin at 0.04 mg/kg (2x LOQ, n = 1) and 0.20 mg/kg (10x LOQ, n = 2) in the loam soil, and for all analytes at both 0.02 mg/kg (LOQ, n = 3) and 0.10 mg/kg (5x LOQ, n = 3) in the clay loam soil. Flumioxazin was identified and quantified using GC/MS (single ion monitored), with only one recovery (116%) reported for the alternate GC-NPD method. THPA, HPA, and SAT-482-HA were identified and quantified using LC/MS/MS (single ion pair transition), with no additional confirmatory method. Soil used for the ECM validation was only described as California soil (Appendix II, p. 138). Soil matrices used for procedural recoveries, as part of a terrestrial field dissipation study conducted in Champaign County, Illinois, were fully characterized as loam (27% clay, 3.5% organic matter), silty clay loam (39% clay, 1.0% organic matter), and clay loam (29% clay, 0.5% organic matter) at the 0-30, 30-60, and 60-90 cm soil depths, respectively, by Agvise Laboratories, Northwood, North Dakota (p. 12; Appendix V, p. 204).

ECM RM-30S-2-1 (Appendix II, pp. 157-169 of MRID 48402402): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of flumioxazin transformation products APF and DAPF at a fortification level of 0.02 mg/kg (LOQ) in an uncharacterized California soil and at 0.02 mg/kg (LOQ) and 0.10 mg/kg (5x LOQ) in loam, silty clay loam, and clay loam soils (Table XVI, pp. 87-88; Appendix II, pp. 165, 169; and DER Attachment 2). For both analytes, the number of samples was insufficient at 0.02 mg/kg (LOQ, n = 3) in the uncharacterized soil, and at both 0.02 mg/kg (LOQ, n = 3) and 0.10 mg/kg (5x LOQ, n = 3) in the clay loam soil. No performance data were provided at 10x LOQ for either analyte. Individual recoveries from uncharacterized soil fortified at 0.01 mg/kg (LOD, n = 2) were 89.9-90.9% for APF and 84.6-88.6% for DAPF. APF and DAPF were identified and quantified using GC/MS (single ion monitored), with no additional confirmatory method. Soil used for the ECM validation was only described as California soil (Appendix II, pp. 165). The loam, silty clay loam, and clay loam soil matrices used for procedural recoveries are the same as those discussed above. Method: RM-30S-2-1 is a modification of Method: RM-30S-2 which reduces the soil sample size extracted and volume of extraction solvent used in the initial extraction (Appendix II, p. 157). Mean recoveries for APF and DAPF at 0.02 mg/kg (LOQ) were slightly higher using Method: RM-30S-2-1, as compared to Method: RM-30S-2 (means of 75.8% for APF, 78.7% for DAPF), and the study author also reported improved GC/MS performance (Appendix II, pp. 157, 168-169).

ILV of Method: RM-30S-1-1 (MRID 49657401): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of flumioxazin and its transformation products THPA and HPA at fortification levels of 0.02 mg/kg (LOQ) and 0.20 mg/kg (10x LOQ) in a loamy sand soil (p. 29; Table I-III, pp. 36-38). Flumioxazin was identified and quantified using GC/MS (single ion monitored), while LC/MS/MS (single ion pair transition) was used for THPA and HPA. The method was validated for all analytes at both fortification levels in the soil matrix after one trial, with minor method modifications and equivalent labware, equipment and instrument substitutions (pp. 18, 26). The California loamy sand soil (7% clay, 0.45% organic matter) matrix was fully characterized by Agvise Laboratories, Northwood, North Dakota (Appendix A, p. 52; Appendix B, p. 55).

Table 2a. Initial Validation Method and Procedural Recoveries for Flumioxazin and Its Transformation Products THPA, HPA, and SAT-482-HA in Soil Using Method: RM-30S-1/RM-30S-1-1[1]

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
	Method: RM-30S-1					
	Uncharacterized California Soil					
Flumioxazin	0.02 (LOQ)	**3**	88.2-98.0	92.1	5.2	5.6
	0.10	6	89.3-104.9	96.6	6.2	6.4
THPA (as THPA 2-Na)[2]	0.02 (LOQ)	**3**	76.6-80.2	78.3	1.8	2.3
	0.10	6	74.9-78.3	76.9	1.4	1.8
HPA	0.02 (LOQ)	**3**	100.4-103.7	102.1	1.7	1.6
	0.10	6	95.7-107.5	102.7	4.0	3.9
SAT-482-HA	0.02 (LOQ)	**3**	68.2-74.3	71.5	3.1	4.4
	0.10	6	69.9-75.7	74.0	2.2	2.9
	Loam Soil					
Flumioxazin	0.02 (LOQ)	19	71.2-109	92.4	10.9	11.8
	0.04	**1**	89.8	--[3]	--	--
	0.10	19	74.9-110	92.2	6.9	7.5
	0.20	**2**	87.9, 88.8	--	--	--
THPA (as THPA 2-Na)	0.02 (LOQ)	17	57.6-109	89.1	13.4	15.0
	0.10	17	64.4-110	88.7	11.7	13.1
HPA	0.02 (LOQ)	17	83.6-106	96.3	6.5	6.8
	0.10	17	80.2-103	94.1	6.4	6.8
SAT-482-HA	0.02 (LOQ)	17	70.6-101	81.1	9.0	11.1
	0.10	17	68.6-104	84.3	11.6	13.8
	Silty Clay Loam Soil					
Flumioxazin	0.02 (LOQ)	6	75.5-108	92.0	12.1	13.1
	0.10	6	85.2-104	94.5	9.1	9.6
THPA (as THPA 2-Na)	0.02 (LOQ)	7	113-142	**128**	11.0	8.6
	0.10	7	119-152	**133**	11.1	8.3
HPA	0.02 (LOQ)	7	99.3-128	110	9.9	9.0
	0.10	7	97.8-129	109	10.2	9.3
SAT-482-HA	0.02 (LOQ)	7	84.8-112	97.1	11.4	11.7
	0.10	7	100-147	112	17.0	15.1
	Clay Loam Soil					
Flumioxazin	0.02 (LOQ)	**3**	82.6-109	99.2	14.5	14.6
	0.10	**3**	91.2-105	98.7	7.0	7.1
THPA (as THPA 2-Na)	0.02 (LOQ)	**3**	88.0-99.2	94.1	5.7	6.0
	0.10	**3**	85.2-104	97.1	10.3	10.6
HPA	0.02 (LOQ)	**3**	86.1-106	99.0	11.2	11.3
	0.10	**3**	86.3-107	99.1	11.2	11.3
SAT-482-HA	0.02 (LOQ)	**3**	93.9-100	96.5	3.2	3.3
	0.10	**3**	103-117	110	7.0	6.4
	Method: RM-30S-1-1 (alternate GC-NPD)					
	Loam Soil					
Flumioxazin	0.02 (LOQ)	--	--	--	--	--
	0.10	**1**	116[4]	--	--	--

Data (corrected recovery results; Appendix VII, pp. 228-335) were obtained from p. 20; Tables XIII-XV, pp. 80-86; Appendix II, pp. 138, 146-148 of MRID 48402402 and DER Attachment 2 (means, SDs, and RSDs, as required).

1 Soil used for the ECM validation was only described as California soil (Appendix II, p. 138). Soil matrices used for procedural recoveries, as part of a terrestrial field dissipation study conducted in Champaign County, Illinois, were characterized as loam, silty clay loam, and clay loam at the 0-30, 30-60, and 60-90 cm soil depths, respectively (p. 12; Appendix V, p. 204).
2 Original Method RM-30S-1/RM-30S-1-1 validations did not specify THPA 2-Na (Appendix II, pp. 125-128).
3 Insufficient number of samples to yield meaningful statistics.
4 As part of the terrestrial field dissipation study, the final storage stability set for flumioxazin (extraction date 11/2/2004) was analyzed using the alternate GC-NPD method for flumioxazin, which was part of revised Method: RM-30S-1-1 (p. 19; Table IX, p. 76; Table XIII, p. 81; Appendix II, pp. 149-151; Appendix IX, p. 455). The primary methods of analysis for Method RM: RM-30S-1/RM-30S-1-1 were GC/MS for flumioxazin and LC/MS/MS for THPA, HPA, and SAT-482-HA.

Table 2b. Initial Validation Method Recoveries for Flumioxazin Transformation Products APF and DAPF in Soil Using Method: RM-30S-2-1[1]

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
	Uncharacterized California Soil					
APF	0.01 (LOD)	2	90.9, 89.9	--[2]	--	--
	0.02 (LOQ)	3	76.7-100.4	87.1	12.1	13.9
DAPF	0.01 (LOD)	2	88.6, 84.6	--	--	--
	0.02 (LOQ)	3	72.9-87.7	80.5	7.4	9.2
	Loam Soil					
APF	0.02 (LOQ)	17	71.5-107	90.0	10.5	11.6
	0.10	17	72.3-106	84.5	9.3	11.0
DAPF	0.02 (LOQ)	17	70.1-114	88.6	13.0	14.7
	0.10	17	70.5-113	82.9	11.1	13.4
	Silty Clay Loam Soil					
APF	0.02 (LOQ)	6	96.5-107	102	3.7	3.7
	0.10	6	82.8-117	98.7	11.0	11.2
DAPF	0.02 (LOQ)	6	92.8-118	106	8.2	7.7
	0.10	6	82.5-115	97.5	10.6	10.8
	Clay Loam Soil					
APF	0.02 (LOQ)	3	88.8-117	99.9	15.0	15.0
	0.10	3	83.0-108	99.0	13.9	14.0
DAPF	0.02 (LOQ)	3	93.9-117	104	11.9	11.5
	0.10	3	86.6-117	104	15.8	15.1

Data (corrected recovery results; Appendix VII, pp. 228-335) were obtained from Table XVI, pp. 87-88; Appendix II, pp. 165, 169 of MRID 48402402 and DER Attachment 2 (means, SDs, and RSDs, as required).
1 Soil used for ECM validation was only described as California soil (Appendix II, p. 165). Soil used for procedural recoveries, as part of a terrestrial field dissipation study, was characterized as loam, silty clay loam, and clay loam at the 0-30, 30-60, and 60-90 cm soil depths (p. 12; Appendix V, p. 204).
2 Insufficient number of samples to yield meaningful statistics.

Table 3. Independent Validation Method Recoveries for Flumioxazin and Its Transformation Products THPA and HPA (Method: RM-30S-1-1) in Loamy Sand Soil[1]

Analyte	Fortification Level (mg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Flumioxazin	0.02 (LOQ)	5	90.0-110	96.7	7.91	8.18
	0.20	5	99.0-123	107	10.4	9.72
THPA (as THPA 2-Na)	0.02 (LOQ)	5	91.5-99.0	95.7	2.89	3.02
	0.20	5	101-112	107	3.97	3.71
HPA	0.02 (LOQ)	5	102-114	106	4.51	4.25
	0.20	5	104-109	106	2.59	2.44

Data (uncorrected recovery results; Appendix C, pp. 57-62) were obtained from Tables I-III, pp. 36-38 of MRID 49657401.

1 The soil matrix, supplied by Valent, was characterized and the source location reported as California (p. 12; Appendix A, p. 52; Appendix B, p. 55).

III. Method Characteristics

In the ECMs, the LOQ and LOD were 0.02 mg/kg (ppm) and 0.01 mg/kg, respectively, for flumioxazin, THPA, HPA, SAT-482-HA, APF, and DAPF (Appendix II, p. 138, 156, 164 of MRID 48402402). In the ECMs, the LOQ was justified by the validation recovery results (method recoveries within 70-120%); however, there was no justification for selection of the LOD. In the ILV, the LOQ and LOD were also 0.02 mg/kg and 0.01 mg/kg for flumioxazin, THPA, and HPA; SAT-482-HA, APF, and DAPF were not included in the ILV (p. 30 of MRID 49657401).

Table 4a. Method Characteristics for Flumioxazin and Its Transformation Products THPA, HPA, and SAT-482-HA in Soil Using Method: RM-30S-1/RM-30S-1-1

		Flumioxazin	THPA	HPA	SAT-482-HA
Limit of Quantitation (LOQ)		0.02 mg/kg (ppm)			
Limit of Detection (LOD)		0.01 mg/kg			
Linearity (calibration curve r^2 and concentration range)[1]	ECM:[1]	r^2 = 0.99538-0.99998	r^2 = 0.9969-0.9999	r^2 = **0.9933**-0.9996	r^2 = **0.9926**-0.99992
	Range:	0.10-2.0 µg/mL	0.008-0.20 µg/mL	0.008-0.20 µg/mL	0.005-0.100 µg/mL
	ECM-Rev.:[2]	r^2 = **0.9844**	NA	NA	NA
	Range:	0.05-1.00 µg/mL			
	ILV:[3]	r^2 = **0.9871**-0.9985	r^2 = 0.9992-0.9997	r^2 = 0.9996-0.9998	Not performed.
	Range:	0.10-2.0 µg/mL	0.008-0.20 µg/mL	0.008-0.20 µg/mL	
Repeatable	ECM:[1]	Yes at LOQ and 5x LOQ, except for THPA in the silty clay loam soil (means 128-133% at both fortification levels). Insufficient number of samples (n = 3) at LOQ in uncharacterized California soil. Insufficient number of samples for flumioxazin at 2x LOQ (n = 1) and 10x LOQ (n = 2) in loam soil. Insufficient number of samples (n = 3) at LOQ and 5x LOQ in clay loam soil. No performance data at 10x LOQ, except for two recoveries for flumioxazin in loam soil. [uncharacterized California soil, loam (27% clay, 3.5% organic matter), silty clay loam (39% clay, 1.0% organic matter), and clay loam (29% clay, 0.5% organic matter) soil matrices][4]			
	ECM-Rev.:[2]	Only one GC-NPD recovery at 5x LOQ reported for loam soil.	NA	NA	NA
	ILV:[3]	Yes at LOQ and 10x LOQ. [loamy sand (7% clay, 0.45% organic matter) soil matrix][4]			Not performed.
Reproducible		Yes. However, the ILV was not conducted with a soil of the most difficult analytical sample condition.			Not performed.
Specific	ECM:[1]	Interferences (based on ppm found) were <10% of LOD at analyte retention times in matrix controls (Appendix VII, pp. 228-335; Appendix IX, pp. 452-454).			
	ECM-Rev.:[2]	No interferences at analyte retention time in matrix control (Appendix IX, p. 455)	NA	NA	NA
	ILV:[3]	No interferences at analyte retention times in reagent blank and matrix control samples.			Not performed.

Data were obtained from of Appendix II, pp. 138, 140-148, 156, 164; Appendix V, p. 204; Appendix VII, pp. 228-335; Appendix VIII, pp. 345, 352, 360, 373, 386, 399, 412, 425; Appendix IX, pp. 452-455 of MRID 48402402; p. 30; Tables I-III, pp. 36-38; Appendix A, p. 52; Appendix B, p. 55; Appendix C, pp. 57-62; Appendix D, pp. 66-97 of MRID 49657401; and DER Attachment 2.

NA = Not applicable.

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

1 Method: RM-30S-1; GC/MS analysis for flumioxazin, and LC/MS/MS analysis for THPA, HPA, and SAT-482-HA (p. 19; Appendix II, pp. 125-148 of MRID 49402402). Calibration curves were not provided with the ECM validation; all coefficient of determination (r^2) values were obtained from procedural recovery analyses (Appendix VII, pp. 228-335; Appendix IX, pp. 452-454 of MRID 48402402).

2 Revised Method: RM-30S-1-1 alternate GC-NPD analysis for flumioxazin (p. 19; Table IX, p. 76; Appendix II, pp. 149-156; Appendix IX, p. 455 of MRID 48402402).

3 Validation of Revised Method: RM-30S-1-1; GC/MS analysis for flumioxazin, and LC/MS/MS analysis for THPA and HPA (pp. 18-24 of MRID 49657401). RM-30S-1-1 alternate GC-NPD analysis for flumioxazin was not performed.

4 Soil used for ECM validation was only described as California soil (Appendix II, p. 138 of MRID 48402402). Soils used for ECM procedural recoveries (from Illinois terrestrial field dissipation study) and the ILV (from California, supplied by Valent) were characterized (p. 12; Appendix V, p. 204 of MRID 48402402; p. 12; Appendix A, p. 52; Appendix B, p. 55 of MRID 49657401).

Table 4b. Method Characteristics for Flumioxazin Transformation Products APF and DAPF in Soil Using Method: RM-30S-2-1

		APF	DAPF
Limit of Quantitation (LOQ)		0.02 mg/kg (ppm)	
Limit of Detection (LOD)		0.01 mg/kg	
Linearity (calibration curve r^2 and concentration range)[1]	ECM:[1]	r^2 = **0.9875**-0.9996	r^2 = **0.9878**-0.9997
	Range:	0.10-2.0 µg/mL	
	ILV:	Not performed.	
Repeatable	ECM:	Yes at LOQ in uncharacterized California soil and at LOQ and 5x LOQ in loam, silty clay loam, and clay loam soils. Insufficient number of samples (n = 3) at LOQ in uncharacterized California soil. Insufficient number of samples (n = 3) at LOQ and 5x LOQ in clay loam soil. No performance data at 10x LOQ. [uncharacterized California soil, loam (27% clay, 3.5% organic matter), silty clay loam (39% clay, 1.0% organic matter), and clay loam (29% clay, 0.5% organic matter) soil matrices][2]	
	ILV:	Not performed.	
Reproducible		ILV was not performed.	
Specific	ECM:	No interferences were detected at analyte retention times in matrix controls (Appendix VII, pp. 228-335).	
	ILV:	Not performed.	

Data were obtained from of Appendix II, pp. 164-167, 169; Appendix V, p. 204; Appendix VII, pp. 228-335; Appendix VIII, pp. 437, 444 of MRID 48402402; and DER Attachment 2.
NA = Not applicable.
Linearity is satisfactory when $r^2 \geq 0.995$.
1 Calibration curves were not provided with the ECM validation; all coefficient of determination (r^2) values were obtained from procedural recovery analyses (Appendix VII, pp. 228-335 of MRID 48402402).
2 Soil used for ECM validation was only described as California soil (Appendix II, p. 165 of MRID 48402402). Soils used for ECM procedural recoveries (from Illinois terrestrial field dissipation study) were characterized (p. 12; Appendix V, p. 204 of MRID 48402402).

IV. Method Deficiencies and Reviewer's Comments

1. As part of a terrestrial field dissipation study, two ECMs were appended to the study report, plus information regarding revision of one of the ECMs. The first ECM, Method: RM-30S-1, is designed for the analysis of flumioxazin in soil using GC/MS and for analysis of flumioxazin products THPA, HPA, and SAT-482-HA in soil using LC/MS/MS (Appendix II, pp. 125-148 of MRID 48402402). Method: RM-30S-1 was modified (Method: RM-30S-1-1) for flumioxazin, THPA, and HPA analysis only, and also included analysis of flumioxazin by GC/MS or GC-NPD (with on-column injection option; p. 19). A complete version of Method: RM-30S-1-1 was not provided in the study report, only excerpt pages from the method (Appendix II, pp. 149-156). The second ECM, Method: RM-30S-2-1, is designed for the analysis of flumioxazin products APF and DAPF using GC/MS (Appendix II, pp. 157-169).

   The ILV was performed for Method: RM-30S-1-1 for analysis of flumioxazin, THPA, and HPA, but did not include the alternate GC-NPD analysis for flumioxazin. However, confirmatory methods are typically not necessary where GC/MS and LC/MS methods are used as the primary method(s) to generate study data.

   There was no ILV for analysis of SAT-482-HA (Method: RM-30S-1), or APF/DAPF (Method: RM-30S-2-1).

2. 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 ECMs, the LOQ and LOD were 0.02 mg/kg (ppm) and 0.01 mg/kg, respectively, for flumioxazin, THPA, HPA, SAT-482-HA, APF, and DAPF (Appendix II, p. 138, 156, 164 of MRID 48402402). In the ECMs, the LOQ was justified by the validation recovery results (method recoveries within 70-120%); however, there was no justification for selection of the LOD. In the ILV, the LOQ and LOD were also 0.02 mg/kg and 0.01 mg/kg, respectively, for flumioxazin, THPA, and HPA; SAT-482-HA, APF, and DAPF were not included in the ILV (p. 30 of MRID 49657401).

   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. A LOQ above toxicological levels of concern results in an unacceptable method classification.

3. Valent did not select the most difficult analytical sample condition for the independent laboratory to analyze to demonstrate how well the method performs. Valent provided the independent laboratory with a loamy sand (7% clay, 0.45% organic matter) soil from California (pp. 19, 30; Appendix A, p. 52; Appendix B, p. 55 of MRID 49657401). The ECM validations were performed with an uncharacterized California soil, while the terrestrial field dissipation procedural recoveries were performed using loam (27% clay, 3.5% organic matter), silty clay loam (39% clay, 1.0% organic matter), and clay loam (29% clay, 0.5% organic matter) soil matrices (Appendix II, pp. 138, 165; Appendix V, p. 204 of MRID 48402402). Based on the ECM validation/procedural recovery results, the silty clay loam soil should have been selected for the ILV.

4. For the ECM validations/procedural recoveries, there were no performance data at 10x LOQ (0.20 mg/kg) for any analyte/matrix, except for two recoveries for flumioxazin in the loam soil. For all analytes, there were an insufficient number of samples (n = 3) at the LOQ (0.02 mg/kg) in the uncharacterized California soil and at the LOQ and 5x LOQ (0.10 mg/kg) in the clay loam soil. For flumioxazin, there was only one recovery reported for GC/MS analysis of a 2x LOQ (0.04 mg/kg) fortified loam sample and for GC-NPD analysis of a 5x LOQ (0.10 mg/kg) fortified loam sample.

5. For the ECM validations/procedural recoveries, analysis of THPA at the LOQ (mean 128%) and 5x LOQ (mean 133%) in the silty clay loam soil did not meet OCSPP Guideline 850.6100 criteria for precision and accuracy [mean recoveries for replicates at each spiking level between 70% and 120% and relative standard deviations (RSD) ≤20%].

   For the terrestrial field dissipation study, the soil from the untreated control plot used for the procedural recoveries was characterized as loam, silty clay loam, and clay loam at the 0-30, 30-60, and 60-90 cm depths, respectively (Appendix V, p. 204 of MRID 48402402). For THPA, HPA, and SAT-482-HA, the study author noted some differences between recoveries from fortified top-layer samples (loam) and fortified lower-layer samples (silty clay loam + clay loam) for the terrestrial field dissipation procedural recoveries (p. 22; Tables XIII-XVI, pp. 80-88). Due to this, the reviewer combined procedural recoveries according to soil type for statistical analyses using the study labeling format (Appendix I, p. 107; DER Attachment 2).

6. For the ILV, flumioxazin calibration standard curve linearity was not always satisfactory (r^2 ≥0.995; Appendix D, p. 72 of MRID 49657401).

7. For the ECM validations/procedural recoveries, GC/MS and LC/MS/MS chromatograms were not provided for reagent blanks, or the silty clay loam and clay loam soil matrices. For the ECM validations, calibration standard curves were not provided, and only a single calibration standard chromatogram was provide for each analyte (1.0 µg/mL for flumioxazin, APF & DAPF; 0.1 µg/mL for THPA & HPA; and 0.025 µg/mL for SAT-482-HA; Appendix II, pp. 140-145, 166-167 of MRID 48402402). For the procedural recoveries, regression curve analyses and individual calibration standard data were provided, but standard curve plots were not provided, and only a single calibration standard chromatogram was provided for each analyte [1.0 µg/mL for flumioxazin, APF & DAPF (range 0.10-2.0 µg/mL); 0.1 µg/mL for THPA & HPA (range 0.008-0.20 µg/mL); and 0.05 µg/mL for SAT-482-HA (range 0.005-0.100 µg/mL; Appendix VII, pp. 228-335; Appendix VIII, pp. 345, 352, 359, 372, 385, 398, 411, 424, 437, 444; Appendix IX, pp. 452-454). For HPA, SAT-482-HA, APF, and DAPF, calibration standard curve linearity was not always satisfactory (r^2 ≥0.995).

   For the GC-NPD analysis, no chromatograms were provided. For the one procedural recovery analyzed by GC-NPD, regression curve analysis and individual calibration standard data were provided, but a standard curve plot was not provided (p. 19; Appendix IX, p. 455). Standard curve linearity was not satisfactory (r^2 ≥0.995).

8. For the ECM validations/procedural recoveries, Method: RM-30S-1 example calculations did not include correction for residues detected in the matrix controls and no residues were detected in the matrix control sample chromatograms (Appendix II, pp. 138, 140, 142, 144 of MRID 48402402). However, calculations for Method: RM-30S-1-1 and Method: RM-30S-2-1 did include correction for residues detected in the matrix control samples (Appendix II, pp. 156, 164). No residues were detected in the matrix control sample chromatograms for the Method: RM-30S-2-1 validation, but the terrestrial field dissipation procedural recoveries were corrected (Appendix II, p. 166; Appendix VII, pp. 228-335; Appendix IX, pp. 452-455).

   For the ILV, recoveries were not corrected as no residues were detected in the matrix control samples (p. 28 of MRID 49657401).

9. The ECMs were used in a terrestrial dissipation study (p. 19; Appendix II, pp. 125-169 of MRID 48402402). 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 two applications of Chateau Herbicide at 169 g g a.i./A (0.37 lb a.i./A), maximum concentrations (dry wt. basis) detected in the 0-7.5 cm soil depth were 0.37 ppm for flumioxazin, 0.011 ppm for THPA, and 0.02 ppm for APF, with HPA, SAT-482-HA, and DAPF each detected at <0.01 ppm (p. 15; Tables I-III, pp. 29-36).

   For the terrestrial field dissipation study, the LOD for THPA was reported as 0.008 ppm for the field treated samples to account for the molecular weight difference between THPA and the THPA 2-Na prepared standards (p. 20; Table I, pp. 29-31; Table IV, pp. 37-43).

10. For the ILV, the first sample run of the 10x LOQ samples were mistakenly fortified at 100x LOQ (p. 31 of MRID 49657401). Valent specified that the 100x LOQ samples could not be used, and 10x LOQ sample would have to be extracted. References to the 100x LOQ fortification are present in the ILV study report as the following typographical errors:

    a. In the third paragraph of section **I. SUMMARY, Independent Laboratory Validation** (p. 12), the sentence "The method was validated at 0.02 and **2** ppm (µg/g)..." should read "The method was validated at 0.02 and **0.2** ppm (µg/g)...".

    b. In the third paragraph of section **III. METHODS, A. Principle of Analytical Method** (p. 18), the sentences "...and five **100x** LOQ laboratory fortification samples. The five **100x** LOQ samples..." should read "...and five **10x** LOQ laboratory fortification samples. The five **10x** LOQ samples...".

11. The minor method modifications and equivalent labware, equipment and instrument substitutions implemented by the independent laboratory (see section **I. Principle of the Method**, ILV of Method: RM-30S-1-1: above for details) are not considered substantial changes to the ECM.

12. It was reported for the ILV that it took *ca*. 3 calendar days to prepare, analyze and tabulate an eight sample analytical set (one reagent blank, two matrix controls, and five fortified samples; p. 25 of MRID 49657401). The initial thirteen sample validation set (one reagent blank, two matrix controls, and ten fortified samples) was divided into two eight sample subsets for efficient handling (Appendix A, pp. 46, 52-53). For the 10x LOQ samples, one

8-hour days was required to extract eight samples and prepare Fraction B for the analysis of THPA and HPA by LC/MS/MS. An additional 6 hours were required the following day to prepare Fraction A for analysis of flumioxazin by GC/MS. The LC/MS/MS and GC/MS analysis sets were run overnight with *ca*. 1 hour of data processing the following day for each analytical run.

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

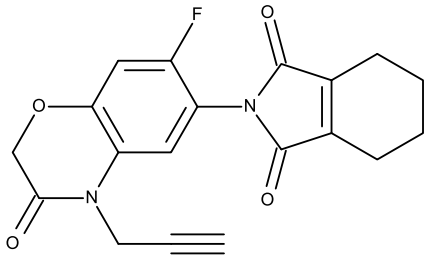
Flumioxazin (V-53482; S-53482)

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



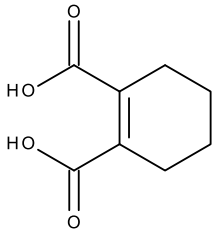
THPA [THPA-2Na]

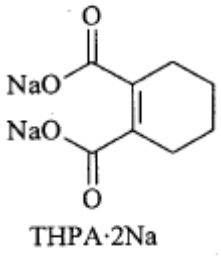
IUPAC Name: 3,4,5,6-Tetrahydrophthalic acid.
Cyclohexene-1,2-dicarboxylic acid.

CAS Name: Not available.

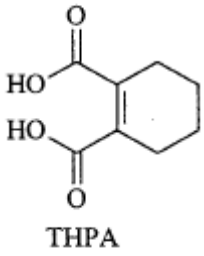
CAS Number: Not available.

SMILES String: C1CCC(=C(C1)C(=O)O)C(=O)O





THPA·2Na



THPA

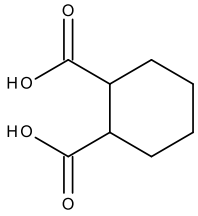
HPA

IUPAC Name: Cyclohexane-1,2-dicarboxylic acid.
trans-1,2-Cyclohexanedicarboxylic acid. (for HPA+)

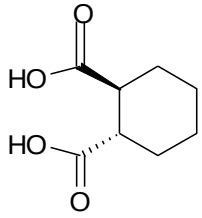
CAS Name: Not available.

CAS Number: Not available.

SMILES String: C1CCC(C(C1)C(=O)O)C(=O)O



HPA



HPA+

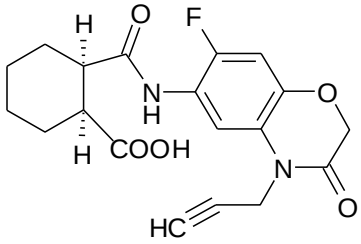
SAT-482-HA

IUPAC Name: N-[7-fluoro-3-oxo-4-(2-propynyl)-2H-1,4-benzoxazin-6-yl]-2-cis-carbamoylcyclohexanecarboxylic acid.
N-[7-fluoro-3-oxo-4-(2-propynyl)-2H-1,4-benzoxazin-6-yl]-2-cis-cyclohexanecarboxylic acid.

CAS Name: Not available.

CAS Number: Not available.

SMILES String: [H]N(c1cc2c(cc1F)OCC(=O)N2CC#C)C(=O)C3CCCCC3C(=O)O



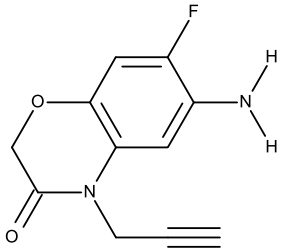
APF

IUPAC Name: 6-Amino-7-fluoro-4-(2-propynyl)-2H-1,4-benzoxazin-3(4H)-one.
6-Amino-7-fluoro-4-prop-2-ynyl-1,4-benzoxazin-3-one

CAS Name: Not available.

CAS Number: Not available.

SMILES String: [H]N([H])c1cc2c(cc1F)OCC(=O)N2CC#C



DAPF (Dihydro-APF)

IUPAC Name: 6-Amino-7-fluoro-4-(2-propenyl)-2H-1,4-benzoxazin-3(4H)-one.
4-Allyl-6-amino-7-fluoro-1,4-benzoxazin-3-one

CAS Name: Not available.

CAS Number: Not available.

SMILES String: [H]N([H])c1cc2c(cc1F)OCC(=O)N2CC=C