

**Analytical method for oxyfluorfen in water**

**Reports:** ECM: EPA MRID No. 50488803. Clark, S 2018. Enforcement Method Validation of the Determination of Residues of Oxyfluorfen in Water Using LC-MS/MS. Dow AgroSciences Protocol No.: 170887. EAG Study No.: 86610. Report prepared by Analytical Bio-Chemistry Laboratories, Inc., a wholly owned subsidiary of EAG, Inc., Columbia, Missouri, and sponsored and submitted by Dow AgroSciences LLC, Indianapolis, Indiana; 67 pages. Final report issued January 8, 2018.

ILV: EPA MRID No. 50488804. Turner, R., J. Hardy. 2018. Independent Laboratory Validation of the Determination of Oxyfluorfen in Water. Dow AgroSciences Study No.: 170888. Battelle Study No.: YR/17/019. Report prepared by Battelle UK Ltd., Essex, United Kingdom, and sponsored and submitted by Dow AgroSciences LLC, Indianapolis, Indiana; 49 pages. Final report issued January 5, 2018.

**Document No.:** MRIDs 50488803 & 50488804

**Guideline:** 850.6100

**Statements:** ECM: The study was conducted in accordance with USEPA FIFRA Good Laboratory Practice (GLP) standards (40 CFR Part 160; p. 3 of MRID 50488803). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4). A statement of the authenticity of the study report was not included.

ILV: The study was conducted in accordance with USEPA, UK, and OECD Good Laboratory Practice (GLP) standards (p. 3; Appendix 3, p. 49 of MRID 50488804). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4; Appendix 3, p. 49). A statement of the authenticity of the study report was included with the quality assurance statement (p. 4).

**Classification:** This analytical method is classified as **Supplemental**. Uncorrected ILV performance data may or may not be satisfactory for the quantitation LOQ analysis depending on the transition ion analyzed.


**PC Code:** 111601

**EFED Final Reviewer:** A'ja Duncan, Ph.D.  
Chemist

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*This Data Evaluation Record may have been altered by the Environmental Fate and Effects Division subsequent to signing by CDM/CSS-Dynamac JV personnel. The CDM/CSS-Dynamac Joint Venture role does not include establishing Agency policies.*

## Executive Summary

The analytical method, Dow AgroSciences Protocol No. 170887, is designed for the quantitative determination of oxyfluorfen in water at the LOQ of 0.1 µg/L using LC/MS/MS. The LOQ is less than the lowest toxicological level of concern (0.29µg/L; USEPA. 2014) in water for oxyfluorfen. The ECM was performed using characterized drinking, ground, and surface water matrices; the ILV was performed using characterized drinking and surface water. Analytes were identified using two ion transitions. The ILV validated the method for oxyfluorfen in the first trial with insignificant modifications of the use of a different LC/MS/MS system and the increase of the injection volume to 50 µL due to sensitivity. ILV performance data was reported as corrected and not corrected for the mean residues found in the two control samples. Uncorrected ILV performance data was not satisfactory for the quantitation LOQ analysis in drinking water, but all other ILV and ECM data regarding repeatability, accuracy, precision, linearity, and specificity were satisfactory.

**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						
Oxyfluorfen	50488803	50488804		Water <sup>1,2</sup>	08/01/2018	Dow AgroSciences LLC	LC/MS/MS	0.1 µg/L

1 In the ECM, ground water (pH 7.9, hardness 293 mg equiv. CaCO<sub>3</sub>, 2.6 ppm dissolved organic carbon) obtained from a monitoring well, surface water (pH 8.1, hardness 129 mg equiv. CaCO<sub>3</sub>, 3.0 ppm dissolved organic carbon) obtained from a pond, and drinking water (pH 7.9, hardness 283 mg equiv. CaCO<sub>3</sub>, 29.7 ppm dissolved organic carbon) obtained from a drinking fountain were used in the study (p. 11; Table 2, p. 18 of MRID 50488803). All water matrices were sourced from the EAG Laboratories' site.

2 In the ILV, drinking water (16/083 RESIDUES TAP H<sub>2</sub>O; pH 8.2, hardness 291 mg equiv. CaCO<sub>3</sub>, 2.4 ppm dissolved organic carbon) and surface water (16/068 CARS WATER RES; pH 8.0, hardness 127 mg equiv. CaCO<sub>3</sub>, 3.4 ppm dissolved organic carbon) were obtained from Battelle UK and characterized by Agvise Laboratories, Northwood, North Dakota (p. 11; Appendix 2, pp. 47-48 of MRID 50488804).

## I. Principle of the Method

Water samples (4.95 mL) were fortified with 0.05 mL of fortification solution in 8 dram amber glass vials; control samples were prepared by transferring 5 mL of the water sample into the 8 dram amber glass vials (p. 11; Appendix 1, pp. 57-58, 61 of MRID 50488803). The internal standard [oxyfluorfen IS; oxyfluorfen  $d_5$  (M+5) stable isotope] was added (0.020 mL of the 0.500  $\mu\text{g/mL}$  solution). The water was mixed with acetonitrile (5.00 mL) via vortex mixing. After the sample returned to room temperature, a 2-mL aliquot was transferred to an autosampler vial for analysis by LC/MS/MS.

Water samples were analyzed for oxyfluorfen using a Shimadzu LC30AD system coupled to an AB Sciex QTrap 6000 mass spectrometer with electrospray ionization (Appendix 1, pp. 63-64 of MRID 50488803). The LC/MS conditions consisted of a Waters Acquity HSS T3 column (2.1 x 50 mm, 1.8- $\mu\text{m}$ ; column temperature 40°C), Water In-line frit guard column, a gradient mobile phase of (A) 0.01% formic acid in water and (B) 0.01% formic acid in methanol [percent A:B (v:v) at 0.50 min. 40:60, 5.00-7.00 min. 5:95, 7.01-8.00 min. 40:60] and MS/MS detection in positive ion mode (ionization temperature 600°C). Injection volume was 20  $\mu\text{L}$ . Four ion transitions were monitored (quantitation, confirmatory 1, confirmatory 2, and confirmatory 3, respectively) as follows:  $m/z$  362.1 $\rightarrow$ 315.9,  $m/z$  362.1 $\rightarrow$ 236.9,  $m/z$  362.1 $\rightarrow$ 298.0, and  $m/z$  362.1 $\rightarrow$ 274.0 for oxyfluorfen and  $m/z$  367.1 $\rightarrow$ 320.9,  $m/z$  367.1 $\rightarrow$ 236.9,  $m/z$  367.1 $\rightarrow$ 298.0, and  $m/z$  367.1 $\rightarrow$ 274.0 for oxyfluorfen IS; however, recovery results were only reported for the quantitation and confirmatory 1 transitions. Retention time was *ca.* 3.5 minutes for oxyfluorfen and oxyfluorfen IS.

In the ILV, the ECM was performed as written, except for the use of a different LC/MS/MS system and the increase of the injection volume to 50  $\mu\text{L}$  due to sensitivity (pp. 10, 15; Appendix 1, pp. 44-46 of MRID 50488804). The LC/MS/MS system was an Agilent 1290 LC coupled to an AB Sciex QTrap 6500 mass spectrometer; other parameters were the same as the ECM. Similar ion transitions were monitored as in the ECM. Two ion transitions were monitored for oxyfluorfen:  $m/z$  362.1 $\rightarrow$ 316.9 and  $m/z$  362.1 $\rightarrow$ 236.9 (quantitation and confirmatory, respectively); only one ion transition was monitored for oxyfluorfen IS:  $m/z$  367.1 $\rightarrow$ 236.9. Retention time was *ca.* 3.5 minutes for oxyfluorfen. No other modifications to the ECM were reported.

The Limit of Quantification (LOQ) for oxyfluorfen in water was 0.10  $\mu\text{g/L}$  in the ECM and ILV (p. 13; Table 11, p. 26 of MRID 50488803; pp. 12, 15 of MRID 50488804). The Limit of Detection (LOD) for oxyfluorfen in water was reported as 0.03  $\mu\text{g/L}$  in the ECM and ILV. In the ECM, LOD and LOQ for determination of oxyfluorfen in water were calculated using the standard deviation from the 0.10  $\mu\text{g/L}$  recovery results. The calculated values support the LOQ and LOD established for the study.

## II. Recovery Findings

ECM (MRID 50488803): Mean recoveries and relative standard deviations (RSDs) were within guideline requirements (mean 70-120%; RSD  $\leq$ 20%) for analysis of oxyfluorfen in water matrices at the fortification level of 0.10  $\mu\text{g/L}$  (LOQ) and 1.00  $\mu\text{g/L}$  (10 $\times$ LOQ; Tables 3-8, pp. 19-24 and Tables 9-10, p. 25; DER Attachment 2). Two ion transitions were quantified; performance data (recovery results) from primary and confirmatory analyses were comparable. One sample per matrix was prepared at the LOD, 0.03  $\mu\text{g/L}$ ; sample recoveries ranged 76-108% (ions/matrices combined). The ground water (pH 7.9, hardness 293 mg equiv.  $\text{CaCO}_3$ , 2.6 ppm dissolved organic carbon) obtained from a monitoring well, surface water (pH 8.1, hardness 129 mg equiv.  $\text{CaCO}_3$ , 3.0 ppm dissolved organic carbon) obtained from a pond, and drinking water (pH 7.9, hardness 283 mg equiv.  $\text{CaCO}_3$ , 29.7 ppm dissolved organic carbon) obtained from a drinking fountain were used in the study (p. 11; Table 2, p. 18). All water matrices were sourced from the EAG Laboratories' site.

ILV (MRID 50488804): **Corrected** mean recoveries and RSDs were within guideline requirements for analysis of oxyfluorfen in water matrices at the fortification level of 0.10  $\mu\text{g/L}$  (LOQ) and 1.00  $\mu\text{g/L}$  (10 $\times$ LOQ; Tables 6-9, pp. 21-24 and Tables 10-11, p. 25; DER Attachment 2). According to OCSPP guidelines, recovery data should not be corrected for residues quantified in the controls. Mean recoveries and RSDs which were **not corrected** for the mean residues found in the two control samples were within guideline requirements for analysis of oxyfluorfen in water matrices at the fortification level of 0.10  $\mu\text{g/L}$  (LOQ) and 1.00  $\mu\text{g/L}$  (10 $\times$ LOQ), except for the quantitation LOQ analysis in drinking water (mean 122%) and the confirmation LOQ analysis in surface water (mean 124%). The reviewer noted that a confirmatory method is not usually required when LC/MS or GC/MS is the primary method(s) to generate study data. Two ion transitions were quantified; performance data (recovery results) from primary and confirmatory analyses were comparable. One sample per matrix was prepared at the LOD, 0.03  $\mu\text{g/L}$ ; sample recoveries ranged 98-129% (ions/matrices combined). The drinking water (16/083 RESIDUES TAP H<sub>2</sub>O; pH 8.2, hardness 291 mg equiv.  $\text{CaCO}_3$ , 2.4 ppm dissolved organic carbon) and surface water (16/068 CARS WATER RES; pH 8.0, hardness 127 mg equiv.  $\text{CaCO}_3$ , 3.4 ppm dissolved organic carbon) were obtained from Battelle UK and characterized by Agvise Laboratories, Northwood, North Dakota (p. 11; Appendix 2, pp. 47-48). The method for oxyfluorfen was validated in the first trial with insignificant modifications of the use of a different LC/MS/MS system and the increase of the injection volume to 50  $\mu\text{L}$  due to sensitivity (pp. 10, 15).

**Table 2. Initial Validation Method Recoveries for Oxyfluorfen in Water<sup>1,2,3</sup>**

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Ground (Well) Water</b>						
Quantitation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	78	--	--	--
	0.10 (LOQ)	5	93-101	97	2.9	3.0
	1.00	5	90-105	98	6.1	6.2
Confirmation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	92	--	--	--
	0.10 (LOQ)	5	90-115	102	9.0	8.8
	1.00	5	97-106	100	3.5	3.5
<b>Surface (Pond) Water</b>						
Quantitation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	76	--	--	--
	0.10 (LOQ)	5	88-101	97	5.7	5.8
	1.00	5	101-105	102	1.7	1.7
Confirmation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	83	--	--	--
	0.10 (LOQ)	5	96-101	98	1.9	1.9
	1.00	5	98-108	103	3.6	3.5
<b>Drinking Water</b>						
Quantitation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	108	--	--	--
	0.10 (LOQ)	5	83-101	96	7.6	7.9
	1.00	5	97-107	102	4.	4.3
Confirmation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	100	--	--	--
	0.10 (LOQ)	5	99-114	106	6.1	5.8
	1.00	5	97-108	103	4.1	4.0

Data (uncorrected recovery results, Figure 4, p. 37) were obtained from Tables 3-8, pp. 19-24 and Tables 9-10, p. 25 of MRID 50488803 and DER Attachment 2.

1 Four ion transitions were monitored (quantitation, confirmatory 1, confirmatory 2, and confirmatory 3, respectively) as follows:  $m/z$  362.1→315.9,  $m/z$  362.1→236.9,  $m/z$  362.1→298.0, and  $m/z$  362.1→274.0 for oxyfluorfen; however, recovery results were only reported for the quantitation and confirmatory 1 transitions.

2 Percent recovery for the 0.03 µg/L samples was reviewer-calculated based on data from Tables 3-8, pp. 19-24 (DER Attachment 2).

3 The ground water (pH 7.9, hardness 293 mg equiv. CaCO<sub>3</sub>, 2.6 ppm dissolved organic carbon) obtained from a monitoring well, surface water (pH 8.1, hardness 129 mg equiv. CaCO<sub>3</sub>, 3.0 ppm dissolved organic carbon) obtained from a pond, and drinking water (pH 7.9, hardness 283 mg equiv. CaCO<sub>3</sub>, 29.7 ppm dissolved organic carbon) obtained from a drinking fountain were used in the study (p. 11; Table 2, p. 18). All water matrices were sourced from the EAG Laboratories' site.

**Table 3. Independent Validation Method Recoveries for Oxyfluorfen in Water<sup>1,2,3</sup>**

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Uncorrected Recoveries</b>						
<b>Drinking Water</b>						
Quantitation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	<b>129</b>	--	--	--
	0.10 (LOQ)	5	116-126	<b>122</b>	4.3	3.5
	1.00	5	98-103	101	1.9	1.9
Confirmation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	98	--	--	--
	0.10 (LOQ)	5	118-121	119	1.1	1.0
	1.00	5	95-104	99	3.5	3.5
<b>Surface Water</b>						
Quantitation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	<b>129</b>	--	--	--
	0.10 (LOQ)	5	114-120	117	2.4	2.0
	1.00	5	99-101	100	0.9	0.9
Confirmation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	<b>123</b>	--	--	--
	0.10 (LOQ)	5	118-128	<b>124</b>	3.9	3.2
	1.00	5	98-102	100	1.8	1.8
<b>Corrected Recoveries</b>						
<b>Drinking Water</b>						
Quantitation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	<b>129</b>	--	--	--
	0.10 (LOQ)	5	100-111	107	4.8	4.5
	1.00	5	97-101	99	1.6	1.7
Confirmation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	98	--	--	--
	0.10 (LOQ)	5	97-100	98	1.1	1.1
	1.00	5	93-102	97	3.5	3.6
<b>Surface Water</b>						
Quantitation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	<b>129</b>	--	--	--
	0.10 (LOQ)	5	98-103	100	2.1	2.1
	1.00	5	98-100	99	1.0	1.0
Confirmation Ion Transition						
Oxyfluorfen	0.03	<b>1</b>	<b>123</b>	--	--	--
	0.10 (LOQ)	5	97-107	103	4.0	3.8
	1.00	5	96-100	98	1.8	1.8

Data (results were reported as uncorrected and corrected recovery results, Tables 10-11, p. 25) were obtained from Tables 6-9, pp. 21-24 and Tables 10-11, p. 25 of MRID 50488804 and DER Attachment 2.

1 Two ion transitions were monitored for oxyfluorfen:  $m/z$  362.1→316.9 and  $m/z$  362.1→236.9 (quantitation and confirmatory, respectively).

2 Percent recovery for the 0.03 µg/L samples was reviewer-calculated based on data from Tables 6-9, pp. 21-24 (DER Attachment 2).

3 The drinking water (16/083 RESIDUES TAP H2O; pH 8.2, hardness 291 mg equiv. CaCO<sub>3</sub>, 2.4 ppm dissolved organic carbon) and surface water (16/068 CARS WATER RES; pH 8.0, hardness 127 mg equiv. CaCO<sub>3</sub>, 3.4 ppm

dissolved organic carbon) were obtained from Battelle UK and characterized by Agvise Laboratories, Northwood, North Dakota (p. 11; Appendix 2, pp. 47-48).

### III. Method Characteristics

The LOQ for oxyfluorfen in water was 0.10 µg/L in the ECM and ILV (p. 13; Table 11, p. 26 of MRID 50488803; pp. 12, 15 of MRID 50488804). The LOD for oxyfluorfen in water was reported as 0.03 µg/L in the ECM and ILV. In the ECM, LOD and LOQ for determination of oxyfluorfen in water were calculated using the standard deviation from the 0.10 µg/L recovery results. The LOD was calculated as the standard deviation multiplied by the t-statistic (3.747) and the LOQ was calculated as three times the LOD result. In the ECM, the LOD and LOQ were calculated as 0.0114-0.0254 µg/L and 0.0341-0.0761 µg/L, respectively, for oxyfluorfen (quantitation ion transition only; matrices combined). The calculated values support the LOQ and LOD established for the study. No justifications or calculations were provided to support the LOD and LOQ in the ILV.

**Table 4. Method Characteristics**

Analyte			Oxyfluorfen
Limit of Quantitation (LOQ)	ECM	Method	0.10 µg/L
		Calculated <sup>1</sup>	0.0341-0.0761 µg/L
	ILV	Method	0.10 µg/L
		Calculated	Not calculated
Limit of Detection (LOD)	ECM	Method	0.03 µg/L
		Calculated <sup>1</sup>	0.0114-0.0254 µg/L
	ILV	Method	0.03 µg/L
		Calculated	Not calculated
Linearity (calibration curve r <sup>2</sup> and concentration range) <sup>2</sup>	ECM	Ground	r <sup>2</sup> = 0.9990 (Q) r <sup>2</sup> = 0.9992 (C)
			ILV
	Concentration Range	Surface r <sup>2</sup> = 0.9998 (Q) r <sup>2</sup> = 0.9996 (C)	
Repeatable	ECM <sup>3</sup>		Drinking, Surface, and Ground Yes at LOQ and 10×LOQ.
	ILV <sup>4,5</sup>	Uncorrected	Drinking Yes at 10×LOQ. <b>No</b> at LOQ (Q mean <b>122%</b> ).
			Surface Yes at LOQ and 10×LOQ, but LOQ C mean <b>124%</b> . <sup>6</sup>
		Corrected <sup>7</sup>	Drinking and Surface Yes at LOQ and 10×LOQ.
Reproducible			Yes at LOQ and 10×LOQ in surface water.
Specific	ECM		Yes, no matrix interferences were observed. Some minor baseline noise was observed.
	ILV		Yes, matrix interferences were 12-19% (Q) and 20-23% (C) of the LOQ (based on quantified residues). No 10×LOQ chromatograms were provided.

Data were obtained from p. 13; Tables 3-8, pp. 19-24 and Tables 9-10, p. 25 (recovery data); Table 11, p. 26 (calculated LOD/LOQ); Figures 2-3, pp. 35-36 (calibration curves); Figures 7-21, pp. 40-54 (chromatograms) of MRID 50488803; pp. 12, 15; Tables 2-5, pp. 19-20 (correlation coefficients); Tables 6-9, pp. 21-24 and Tables 10-11, p. 25 (recovery data); Figures 3-4, pp. 29-30 (calibration curves); Figures 8-13, pp. 34-39 (chromatograms) of MRID 50488804. Q = Quantitation ion transition; C = Confirmatory ion 1 transition.

1 Only quantitation ion reported; matrices combined.

2 Reported correlation coefficients ( $r^2$ ) were reviewer-calculated based on the reported  $r$  values in the study report (Figures 2-3, pp. 35-36 of MRID 50488803; Tables 2-5, pp. 19-20 of MRID 50488804). Solvent calibration standards were used. Only those for the ground water set were reported in the ECM.

3 In the ECM, ground water (pH 7.9, hardness 293 mg equiv.  $\text{CaCO}_3$ , 2.6 ppm dissolved organic carbon) obtained from a monitoring well, surface water (pH 8.1, hardness 129 mg equiv.  $\text{CaCO}_3$ , 3.0 ppm dissolved organic carbon) obtained from a pond, and drinking water (pH 7.9, hardness 283 mg equiv.  $\text{CaCO}_3$ , 29.7 ppm dissolved organic carbon) obtained from a drinking fountain were used in the study (p. 11; Table 2, p. 18 of MRID 50488803). All water matrices were sourced from the EAG Laboratories' site.

4 In the ILV, drinking water (16/083 RESIDUES TAP H<sub>2</sub>O; pH 8.2, hardness 291 mg equiv.  $\text{CaCO}_3$ , 2.4 ppm dissolved organic carbon) and surface water (16/068 CARS WATER RES; pH 8.0, hardness 127 mg equiv.  $\text{CaCO}_3$ , 3.4 ppm dissolved organic carbon) were obtained from Battelle UK and characterized by Agvise Laboratories, Northwood, North Dakota (p. 11; Appendix 2, pp. 47-48 of MRID 50488804).

5 In the ILV, the method for oxyfluorfen and its isomers was validated in the first trial with insignificant modifications of the use of a different LC/MS/MS system and the increase of the injection volume to 50  $\mu\text{L}$  due to sensitivity (pp. 10, 15 of MRID 50488804).

6 A confirmatory method is not usually required when LC/MS or GC/MS is the primary method(s) to generate study data.

7 According to OCSPP guidelines, recovery data should not be corrected for residues quantified in the controls.

#### IV. Method Deficiencies and Reviewer's Comments

1. ILV performance data was reported as corrected and not corrected for the mean residues found in the two control samples. Uncorrected performance data was not satisfactory for the ILV quantitation LOQ analysis in drinking water (mean 122%) and the confirmation LOQ analysis in surface water (mean 124%; Tables 10-11, p. 25 of MRID 50488804). OCSPP guideline requirements state that the mean recovery is 70-120% and the RSD is  $\leq 20\%$ . According to OCSPP guidelines, recovery data should not be corrected for residues quantified in the controls.
2. In the ILV, no  $10\times\text{LOQ}$  chromatograms were provided. Representative chromatograms from all matrices/ions/fortifications should be provided for review in order to fully assess the specificity of the method. Also, the reviewer noted that matrix interferences were 12-19% (Q) and 20-23% (C) of the LOQ (based on quantified residues; Tables 6-9, pp. 21-24 of MRID 50488804).
3. The matrix effects were found to be insignificant ( $<20\%$ ) for oxyfluorfen in the ECM and ILV (pp. 14-15; Tables 14-15, p. 33 of MRID 50488803; p. 14; Table 12, p. 26 of MRID 50488804). Solvent standards were used in the ECM and ILV.
4. The determinations of LOD and LOQ in the ECM and ILV were not based on procedures as defined in 40 CFR Part 136 (p. 13; Table 11, p. 26 of MRID 50488803; pp. 12, 15 of MRID 50488804). In the ECM, the LOD and LOQ for determination of oxyfluorfen in water were calculated using the standard deviation from the 0.10  $\mu\text{g/L}$  recovery results; the LOD was calculated as the standard deviation multiplied by the t-statistic (3.747) and



the LOQ was calculated as three times the LOD result. No justifications or calculations were provided to support the LOD and LOQ in the ILV.

5. It was reported for the ILV that one sample set (5 calibration samples, 1 reagent blank, 2 controls, 10 fortified controls) required *ca.* 3 hours (*ca.* 1 working day) with LC/MS/MS performed unattended overnight (p. 12 of MRID 50488804). Evaluation of the LC/MS/MS results was performed the next day.
6. The ILV study author reported that communications between the ILV and ECM did not occur (p. 15 of MRID 50488804).
7. In the ECM, the final sample extracts were found to be stable after up to 12-13 days of frozen storage (pp. 13-14; Tables 12-13, pp. 27-32 of MRID 50488803). Fortification, calibration, and stock solutions were found to be stable after up to 32, 19, and 45 days of frozen storage (*ca.* 2-8 °C), respectively.

## V. References

- U.S. Environmental Protection Agency (USEPA). 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.
- USEPA. 2014. Registration Review Problem Formulation for Oxyfluorfen. DP barcode 419419. U.S. Environmental Protection Agency, Office of Chemical Safety and Pollution Prevention, Environmental Fate and Effects Division. Memorandum to the Pesticide Re-Evaluation Division. Nov. 3, 2014.
- 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****Oxyfluorfen (XDE-795)**

**IUPAC Name:** 2-Chloro- $\alpha,\alpha,\alpha$ -trifluoro-p-tolyl 3-ethoxy-4-nitrophenyl ether  
**CAS Name:** 2-Chloro-1-(3-ethoxy-4-nitrophenoxy)-4-(trifluoromethyl)benzene  
**CAS Number:** 42874-03-3  
**SMILES String:** Clc1cc(C(F)(F)F)ccc1Oc2cc(OCC)c(N(=O)=O)cc2

