

Test Material: XDE-848 Benzyl Ester

MRID: 49677722

Title: Aquatic Dissipation of XDE-848 Benzyl Ester (SX-1552) in Pond Systems

MRID: 49677777

Title: Independent Laboratory Validation of EPL Bio Analytical Services Method 477G696A-1 for the Determination of XDE-848 Benzyl Ester (SX-1552) and Five Metabolites (1552-Acid, 1552-OHBE, 1552-OHA, 1552-DBE and 1552-DA) in Sediment

EPA PC Code: 030093

OCSPP Guideline: 850.6100

For CDM Smith

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Date: 2/26/16

Analytical method for XDE-848 Benzyl Ester and its transformation products, 1552-Acid, 1552-OHBE, 1552-OHA, 1552-DBE and 1552-DA, in soil

Reports: ECM: EPA MRID No.: 49677722 (Appendix A, pp. 123-447). Lester, L. 2015. Aquatic Dissipation of SX-1552 in Pond Systems. Laboratory Study ID: 477G696. Report prepared by EPL BAS Bio Analytical Services (EPL BAS), Niantic, Illinois, and sponsored and submitted by SePRO Corporation, Carmel, Indiana; 325 pages (MRID 49677722, 710 pages). Final report issued May 27, 2015.

ILV: EPA MRID No. 49677777. Austin, R. 2015. Independent Laboratory Validation of EPL Bio Analytical Services Method 477G696C for the Determination of XDE-848 Benzyl Ester (SX-1552) and Five Metabolites (1552-Acid, 1552-OHBE, 1552-OHA, 1552-DBE and 1552-DA) in Sediment. Battelle Study No.: YR/15/011. Report prepared by Battelle UK Ltd., Essex, United Kingdom, and sponsored and submitted by SePRO Corporation, Carmel, Indiana; 117 pages. Final report issued August 5, 2015.

Document No.: MRIDs 49677722 & 49677777

Guideline: 850.6100

Statements: ECM: The study was conducted in accordance with USEPA FIFRA Good Laboratory Practices (GLP; Appendix A, p. 124 of MRID 49677722). Signed and dated GLP and Quality Assurance statements were provided (Appendix A, pp. 124-125). The No Data Confidentiality and Authenticity statements were not included.

ILV: The study was conducted in accordance with USEPA (1989) and OECD GLP standards (1998), as well as the UK Department of Health (Directive 2004/9/EC; p. 3; Appendix 3, p. 117 of MRID 49677777). Signed and dated No Data Confidentiality, GLP, Quality Assurance and Authenticity statements were provided (pp. 2-4; Appendix 3, p. 117). A statement of the authenticity of the study report was included with the quality assurance statement (p. 4).

Classification: This analytical method is considered supplemental. In the ECM, representative chromatograms did not support the specificity of the method for all analytes in both matrices. In the ILV, representative chromatograms were not provided for all fortification levels. The determinations of the LOQ and LOD were not based on scientifically acceptable procedures. Clarification on these issues and/or submission of additional representative chromatograms are required.

PC Code: 030093

Reviewer: José Meléndez, U.S. EPA

Signature:

Date: November 10, 2016

All cited page numbers for MRID 49677722 refer to those written in the bottom right-hand corner of the document pages.

Executive Summary

The analytical method, EPL Bio Analytical Services Method 477G696C, is designed for the quantitative determination of XDE-848 (SX-1552) and the five metabolites, 1552-Acid, 1552-OHBE, 1552-OHA, 1552-DBE and 1552-DA, in sediment matrices at the LOQ of 0.003 µg/g using LC/MS/MS. The LOQs are less than the lowest toxicological level of concern in sediment for XDE-848 (SX-1552) and its five metabolites¹. The original ECM, EPL Bio Analytical Services Method 477G696C, was not submitted for review; however, the submitted ECM was performed using EPL Bio Analytical Services Method 477G696C. Characterized pond sediments from two sites were used in the ECM; the Florida (FL) and North Carolina (NC) ponds were sourced by a well and a source reservoir pond, respectively. The ECM was validated by the ILV in the first trial for all six analytes with insignificant modifications to the analytical parameters using the same NC sediment which was used in the ECM. In the ILV, representative chromatograms were not provided for the reagent blank and fortifications at the LOD or 10×LOQ, only calibrants, controls and LOQ. Additionally, due to baseline noise and missing chromatograms, representative ECM chromatograms did not support the specificity of the method for SX-1552 and 1552-DBE in both sediments and for 1552-OHA in the NC sediment.

¹ The lowest toxicological level of concern in sediment is based on the chronic NOAEC for *Chironomus dilutus* of <5.25 mg/kg. This method's LOQ is lower, at 0.003 mg/kg. Although the NOAEC is non-definitive, it is noted that the difference between the endpoint and the LOQ is over three orders of magnitude difference. The NOAEC is based on study MRID 49677750.

Table 1. Analytical Method Summary^{1,2,3}

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						
XDE-848 (SX-1552)	49677722 Appendix A	49677777		Sediment	27/05/2015 ⁴	SePRO Corporation	LC/MS/MS	0.003 µg/g
1552-OHA								
1552-DBE								
1552-DA								
1552-OHBE								
1552-Acid								

1 XDE-848 = [Florpyrauxifen-benzyl, XDE-848 BE; XDE-848 benzyl ester; TSN301734; X11959130; SX-1552; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-OHA = [XDE-848 hydroxy acid; TSN305649; X11966341; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-DBE = [Dechlorinated XDE-848 benzyl ester; TSN305649; X12131932; benzyl 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-DA = [Dechlorinated XDE-848 acid; TSN304479; X12393505; 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-OHBE = [XDE-848 hydroxy benzyl ester; TSN305650; X12300837; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylate]; and 1552-Acid = [XDE-848 acid; TSN301691; X11438848; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid].

2 For the ECM, sediment matrices were well-characterized pond sediments (USDA soil texture classification; pp. 24-25, 28, 36; Tables 9-10, pp. 60-61 of MRID 49677722). The Florida pond sediment (FL) was classified as sand (Stations A-C; 96% sand, 3% silt, 1% clay; pH 7.3-8.0; 0.96-3.3% organic matter). The North Carolina pond sediment (NC) was classified as sandy loam (Stations A and B; 67-71% sand, 23-25% silt, 6-8% clay; pH 6.1-6.2; 1.5-2.9% organic matter) and loam (Station C; 45% sand, 41% silt, 14% clay; pH 6.2; 4.3% organic matter). The Florida pond was sourced by a well; the North Carolina pond was sourced by a source reservoir pond.

3 For the ILV, the sediment was the same North Carolina pond sediment which was used in the ECM, based on the sediment characterization data (p. 15; Appendix 2, pp. 114-116 of MRID 49677777).

4 Date based on EPL Bio Analytical Services Method 477G696 since the original report of EPL Bio Analytical Services Method 477G696C was not provided (See Reviewer's Comment #1).

I. Principle of the Method

During the entire procedure, only glass laboratory equipment was used (Appendix A, pp. 149-151 of MRID 49677722). Samples (5.000-5.100 g) of sediment in 50-mL centrifuge tubes were fortified, as necessary, then extracted four times with 20 mL of acetonitrile:0.1N HCl (90:10, v:v) via vortex and shaking on a platform shaker (180 rpm for ca. 30 minutes). After centrifugation (2000 rpm for 5 minutes), the supernatant was decanted into a 100 mL volumetric flask. The volume of the combined extracts was brought to 100 mL using additional extraction solvent. An aliquot (200 μ L) of the sample was transferred to a glass LC/MS/MS vial with 750 μ L of methanol:DI water:formic acid (50:50:0.1, v:v:v). 50 μ L of the 10 ng/mL mixed internal standard was added to the vial prior to analysis.

Samples were analyzed for XDE-848 (SX-1552) and its metabolites using an Agilent 1290 LC system coupled to an AB Sciex QTRAP 6500 LC/MS/MS (Appendix A, pp. 151-152 of MRID 49677722). The instrumental conditions consisted of a Phenomenex Kinetex PFP column (100 x 2.10 mm, 1.7- μ m; column temperature, 35°C), a gradient mobile phase of (A) DI water containing 0.1% formic acid and (B) methanol containing 0.1% formic acid [percent A:B (v:v) at 0.0 min. 90:10, 7.00-8.50 min. 0:100, 8.60-11.00 min. 90:10], MS/MS detection in positive electrospray mode MS (MRM; temperature, 650°C), and injection volume 15 μ L. Two parent-daughter ion transitions were monitored per analyte (quantification and confirmation, respectively): m/z 441 \rightarrow 65 and m/z 441 \rightarrow 91 for XDE-848 (SX-1552); m/z 335 \rightarrow 254 and m/z 337 \rightarrow 256 for 1552-OHA; m/z 405 \rightarrow 65 and m/z 407 \rightarrow 91 for 1552-DBE; m/z 315 \rightarrow 234 and m/z 315 \rightarrow 124 for 1552-DA; m/z 425 \rightarrow 91 and m/z 4267 \rightarrow 91 for 1552-OHBE; and m/z 349 \rightarrow 268 and m/z 349 \rightarrow 225 for 1552-Acid. Retention times were observed at *ca.* 7.0, 4.6-4.65, 6.8-7.0, 4.85-5.0, 6.6, and 5.5-5.7 min. for XDE-848 (SX-1552), 1552-OHA, 1552-DBE, 1552-DA, 1552-OHBE, and 1552-Acid, respectively (retention times were reviewer-assigned based observed and expected; Appendix A, Figures 7-12, pp. 318-320).

In the ILV, the sample processing of the ECM was performed exactly as written (pp. 14, 16, 20; Appendix 1, pp. 111-113 of MRID 49677777). Samples were analyzed for XDE-848 (SX-1552) and its metabolites using an Agilent 1290 Binary Pump LC system coupled to an AB Sciex QTRAP 6500 LC/MS/MS. All instrumental parameters were the same, except for the following: MS/MS detection in positive Turbo Ion Spray mode MS (MRM; temperature, 650°C), and injection volume 40 μ L. Two parent-daughter ion transitions were monitored per analyte (quantification and confirmation, respectively): m/z 441.1 \rightarrow 65.1 and m/z 441.1 \rightarrow 91.0 for XDE-848 (SX-1552); m/z 334.9 \rightarrow 254.0 and m/z 336.9 \rightarrow 256.0 for 1552-OHA; m/z 404.8 \rightarrow 65.1 and m/z 407.0 \rightarrow 91.0 for 1552-DBE; m/z 315.0 \rightarrow 234.0 and m/z 315.0 \rightarrow 124.0 for 1552-DA; m/z 425.0 \rightarrow 91.0 and m/z 427.0 \rightarrow 91.0 for 1552-OHBE; and m/z 349.0 \rightarrow 268.0 and m/z 349.0 \rightarrow 225.0 for 1552-Acid (a majority of the ions differed from those reported for the ECM by +0.0-0.2 m/z). Retention times were observed at *ca.* 7.36, 4.95, 7.28, 5.33, 6.95, and 5.95 min. for XDE-848 (SX-1552), 1552-OHA, 1552-DBE, 1552-DA, 1552-OHBE, and 1552-Acid, respectively (retention times were reviewer-assigned based observed; Figures 30-53, pp. 82-105). The ILV study author noted that the increase in the injection volume was due to poor sensitivity at the lower injection volume (p. 20). Additionally, the standards in methanol were stored in the freezer, instead of the refrigerator as specified in the method. None of the minor ILV modifications to the instrumental parameters had an effect on the outcome of the study.

LOQ/LOD

The LOQ and LOD in the ECM and ILV were 0.003 µg/g and 0.0009 µg/g, respectively, for XDE-848 (SX-1552) and the five metabolites in sediment (p. 34; Appendix A, pp. 142, 162 of MRID 49677722; pp. 17, 20 of MRID 49677777).

II. Recovery Findings

ECM [49677722 (Appendix A, pp. 123-447)]: Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD ≤20%) for analysis of XDE-848 (SX-1552) and its five metabolites, 1552-OHA, 1552-DBE, 1552-DA, 1552-OHBE, and 1552-Acid, in the two pond sediment matrices at the fortification levels of 0.003 µg/g (LOQ) and 0.03 µg/g (10×LOQ; Appendix A, pp. 162-163; Appendix A, Tables 11-16, pp. 179-190). For all analytes, two ion transitions were monitored using LC/MS/MS; however, performance data (recovery results) were only evaluated and reported for the quantitative ion (see Reviewer's Comment #6). The ECM calculations allowed for recovery data to be corrected for residues found in the control samples; however, no residues were quantified in any of the controls (Appendix A, pp. 158-160; Appendix A, Figures 13-18, pp. 321-323; Appendix A, Figures 55-60, pp. 342-344). Both sediment matrices were well-characterized pond sediments (USDA soil texture classification; pp. 24-25, 28, 36; Tables 9-10, pp. 60-61). The Florida pond was located in Seminole County, north of the town of Oviedo, and sourced by a well. The North Carolina pond was located in Nash County, northwest of the town of Whitakers; the pond was a constructed pond which was sourced by a source reservoir pond. Neither pond had a history of prior pesticide use for 3 years. The sediment samples which were used for the method validation study were untreated and collected from three stations from a three-mile radius of the established sampling location of the ponds (prior to field study initiation). The Florida pond sediment (FL) was classified as sand (Stations A-C; 96% sand, 3% silt, 1% clay; pH 7.3-8.0; 0.96-3.3% organic matter). The North Carolina pond sediment (NC) was classified as sandy loam (Stations A and B; 67-71% sand, 23-25% silt, 6-8% clay; pH 6.1-6.2; 1.5-2.9% organic matter) and loam (Station C; 45% sand, 41% silt, 14% clay; pH 6.2; 4.3% organic matter).

ILV (MRID 49677777): Mean recoveries and RSDs were within guidelines (mean 70-120%; RSD ≤20%) for analysis of XDE-848 (SX-1552) and its five metabolites, 1552-OHA, 1552-DBE, 1552-DA, 1552-OHBE, and 1552-Acid, in the one sediment matrix at the fortification levels of 0.003 µg/g (LOQ) and 0.03 µg/g (10×LOQ; uncorrected recovery results; Tables 26-37, pp. 44-46; Figure 23, p. 75). For all analytes, two ion transitions were monitored using LC/MS/MS; performance data (recovery results) of the quantitative and confirmatory results were comparable. Recoveries from samples fortified at 0.0009 µg/g (LOD) ranged (ions/matrices combined) from 69-116% for all analytes (n = 1 for each matrix/analyte; Tables 14-25, pp. 32-43; DER Attachment 2). The pond sediment matrix was obtained from a pond (number 18) located on the SePRO Research and Development Campus, near the town of Whitakers (Nash County), North Carolina (p. 15). The pond sediment was obtained as three batches and well characterized by Agvise Laboratories, Northwood, North Dakota (p. 15; Appendix 2, pp. 114-116). The sediment was the same North Carolina pond sediment which was

used in the ECM, based on the sediment characterization data. The method was validated in the first trial for all analytes in one pond sediment matrix with insignificant modifications to the analytical parameters (p. 20).

Table 2. Initial Validation Method Recoveries for XDE-848 (XDE-848 BE; SX-1552) and Its Five Metabolites, 1552-OHA, 1552-DBE, 1552-DA, 1552-OHBE, and 1552-Acid, in Sediments from Two Sites^{1,2,3}

Analyte	Fortification Level (µg/g)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Florida (FL) Pond Sediment						
Quantitation ion transition						
XDE-848 (XDE-848 BE; SX-1552)	0.003 (LOQ)	7	77.067- 91.000	81.724	5.967	7.302
	0.03	7	71.033- 98.433	90.534	8.543	9.437
1552-OHA	0.003 (LOQ)	7	67.687- 95.918	85.374	9.577	11.218
	0.03	7	72.415- 103.639	85.860	9.634	11.221
1552-DBE	0.003 (LOQ)	7	63.574- 92.440	79.676	9.556	11.993
	0.03	7	79.210- 100.069	90.054	7.630	8.473
1552-DA	0.003 (LOQ)	7	68.027- 100.340	84.451	9.717	11.507
	0.03	7	75.544- 100.476	87.143	7.689	8.823
1552-OHBE	0.003 (LOQ)	7	62.333- 100.333	85.238	11.821	13.868
	0.03	7	68.633- 95.567	87.186	8.667	9.941
1552-Acid	0.003 (LOQ)	7	69.000- 95.000	83.878	9.487	11.310
	0.03	7	71.800- 93.100	83.939	7.702	9.175
North Carolina (NC) Pond Sediment						
Quantitation ion transition						
XDE-848 (XDE-848 BE; SX-1552)	0.003 (LOQ)	7	63.667- 92.000	76.476	9.441	12.345
	0.03	7	80.833- 105.467	91.967	8.518	9.262
1552-OHA	0.003 (LOQ)	7	72.449- 120.408	84.885	16.146	19.021
	0.03	7	76.769- 107.007	89.392	9.833	10.999
1552-DBE	0.003 (LOQ)	7	69.759- 92.096	78.873	7.130	9.040
	0.03	7	73.265- 114.353	93.126	11.420	12.263

Analyte	Fortification Level (µg/g)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
1552-DA	0.003 (LOQ)	7	67.347-108.163	81.244	13.904	17.114
	0.03	7	79.558-93.367	87.337	4.909	5.621
1552-OHBE	0.003 (LOQ)	7	58.485-90.333	72.831	9.087	12.477
	0.03	7	79.475-104.100	89.963	9.136	10.155
1552-Acid	0.003 (LOQ)	7	68.333-82.000	75.571	6.065	8.026
	0.03	7	71.167-92.067	84.324	6.389	7.576

Data (uncorrected recovery results; Appendix A, pp. 158-160; Appendix A, Figures 13-18, pp. 321-323; Appendix A, Figures 55-60, pp. 342-344) were obtained from Appendix A, pp. 162-163; Appendix A, Tables 11-16, pp. 179-190 of MRID 49677722. Only results from the quantitation ion were reported (see Reviewer's Comment #6).

- XDE-848 = [Florpyrauxifen-benzyl; XDE-848 BE; XDE-848 benzyl ester; TSN301734; X11959130; SX-1552; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-OHA = [XDE-848 hydroxy acid; TSN305649; X11966341; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-DBE = [Dechlorinated XDE-848 benzyl ester; TSN305649; X12131932; benzyl 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-DA = [Dechlorinated XDE-848 acid; TSN304479; X12393505; 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-OHBE = [XDE-848 hydroxy benzyl ester; TSN305650; X12300837; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylate]; and 1552-Acid = [XDE-848 acid; TSN301691; X11438848; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid].
- Both sediment matrices were well-characterized pond sediments (USDA soil texture classification; pp. 24-25, 28, 36; Tables 9-10, pp. 60-61). The Florida pond was located in Seminole County, north of the town of Oviedo, and sourced by a well. The North Carolina pond was located in Nash County, northwest of the town of Whitakers; the pond was a constructed pond which was sourced by a source reservoir pond. Neither pond had a history of prior pesticide use for 3 years. The sediment samples which were used for the method validation study were untreated and collected from three stations from a three-mile radius of the established sampling location of the ponds (prior to field study initiation). The Florida pond sediment (FL) was classified as sand (Stations A-C; 96% sand, 3% silt, 1% clay; pH 7.3-8.0; 0.96-3.3% organic matter). The North Carolina pond sediment (NC) was classified as sandy loam (Stations A and B; 67-71% sand, 23-25% silt, 6-8% clay; pH 6.1-6.2; 1.5-2.9% organic matter) and loam (Station C; 45% sand, 41% silt, 14% clay; pH 6.2; 4.3% organic matter).
- Two parent-daughter ion transitions were monitored per analyte (quantification and confirmation, respectively): m/z 441 \rightarrow 65 and m/z 441 \rightarrow 91 for XDE-848 (SX-1552); m/z 335 \rightarrow 254 and m/z 337 \rightarrow 256 for 1552-OHA; m/z 405 \rightarrow 65 and m/z 407 \rightarrow 91 for 1552-DBE; m/z 315 \rightarrow 234 and m/z 315 \rightarrow 124 for 1552-DA; m/z 425 \rightarrow 91 and m/z 4267 \rightarrow 91 for 1552-OHBE; and m/z 349 \rightarrow 268 and m/z 349 \rightarrow 225 for 1552-Acid. However, only the quantification ion was evaluated for residue recovery.
- One of the recovery values was not accepted by the study author; no justification or calculation was provided for the omission. The reviewer calculated the recovery based on the amount of analyte found without correction (recovery calculations included corrections for residues found in controls). The reported mean, s.d. and RSD were reviewer-calculated (see DER Attachment 2).

Table 3. Independent Validation Method Recoveries for XDE-848 (XDE-848 BE; SX-1552) and Its Five Metabolites, 1552-OHA, 1552-DBE, 1552-DA, 1552-OHBE, and 1552-Acid, in Sediment^{1,2}

Analyte	Fortification Level (µg/g)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
North Carolina (NC) Pond Sediment						
Quantitation ion transition						
XDE-848 (XDE-848 BE; SX-1552)	0.0009 (LOD)	1	69	--	--	--
	0.003 (LOQ)	5	87-105	96	6.6	6.9
	0.03	5	82-99	93	7.1	7.6
1552-OHA	0.0009 (LOD)	1	85	--	--	--
	0.003 (LOQ)	5	90-97	93	2.8	3.0
	0.03	5	82-98	92	6.1	6.6
1552-DBE	0.0009 (LOD)	1	108	--	--	--
	0.003 (LOQ)	5	104-110	108	2.7	2.5
	0.03	5	91-111	103	8.1	7.8
1552-DA	0.0009 (LOD)	1	110	--	--	--
	0.003 (LOQ)	5	96-116	106	7.6	7.2
	0.03	5	83-101	93	7.0	7.5
1552-OHBE	0.0009 (LOD)	1	110	--	--	--
	0.003 (LOQ)	5	94-119	105	9.8	9.3
	0.03	5	82-102	95	7.7	8.1
1552-Acid	0.0009 (LOD)	1	116	--	--	--
	0.003 (LOQ)	5	94-104	98	4.2	4.2
	0.03	5	75-91	87	6.9	8.0
Confirmation ion transition						
XDE-848 (XDE-848 BE; SX-1552)	0.0009 (LOD)	1	77	--	--	--
	0.003 (LOQ)	5	86-100	94	7.1	7.6
	0.03	5	83-99	87	6.9	8.0
1552-OHA	0.0009 (LOD)	1	89	--	--	--
	0.003 (LOQ)	5	86-99	93	5.5	6.0
	0.03	5	81-98	91	6.6	7.2
1552-DBE	0.0009 (LOD)	1	69	--	--	--
	0.003 (LOQ)	5	87-105	94	7.0	7.4
	0.03	5	87-110	99	9.6	9.7
1552-DA	0.0009 (LOD)	1	92	--	--	--
	0.003 (LOQ)	5	96-108	103	4.8	4.7
	0.03	5	80-97	91	6.9	7.5

Analyte	Fortification Level (µg/g)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
1552-OHBE	0.0009 (LOD)	1	108	--	--	--
	0.003 (LOQ)	5	87-122	103	12.6	12.3
	0.03	5	83-103	96	8.0	8.4
1552-Acid	0.0009 (LOD)	1	99	--	--	--
	0.003 (LOQ)	5	92-103	97	4.4	4.5
	0.03	5	78-94	89	6.5	7.3

Data (uncorrected recovery results; Figure 23, p. 75) were obtained from Tables 14-25, pp. 32-43 (LOD results) and Tables 26-37, pp. 44-46 of MRID 49677777 and DER Attachment 2 (LOD calculations).

- XDE-848 = [Florpyrauxifen-benzyl; XDE-848 BE; XDE-848 benzyl ester; TSN301734; X11959130; SX-1552; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-OHA = [XDE-848 hydroxy acid; TSN305649; X11966341; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-DBE = [Dechlorinated XDE-848 benzyl ester; TSN305649; X12131932; benzyl 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-DA = [Dechlorinated XDE-848 acid; TSN304479; X12393505; 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-OHBE = [XDE-848 hydroxy benzyl ester; TSN305650; X12300837; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylate]; and 1552-Acid = [XDE-848 acid; TSN301691; X11438848; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid].
- The pond sediment matrix was obtained from a pond (number 18) located on the SePRO Research and Development Campus, near the town of Whitakers (Nash County), North Carolina (p. 15). The pond sediment was obtained as three batches and well characterized by Agvise Laboratories, Northwood, North Dakota (p. 15; Appendix 2, pp. 114-116). The sediment was the same North Carolina pond sediment which was used in the ECM, based on the sediment characterization data.
- Two parent-daughter ion transitions were monitored per analyte (quantification and confirmation, respectively): m/z 441.1 \rightarrow 65.1 and m/z 441.1 \rightarrow 91.0 for XDE-848 (SX-1552); m/z 334.9 \rightarrow 254.0 and m/z 336.9 \rightarrow 256.0 for 1552-OHA; m/z 404.8 \rightarrow 65.1 and m/z 407.0 \rightarrow 91.0 for 1552-DBE; m/z 315.0 \rightarrow 234.0 and m/z 315.0 \rightarrow 124.0 for 1552-DA; m/z 425.0 \rightarrow 91.0 and m/z 427.0 \rightarrow 91.0 for 1552-OHBE; and m/z 349.0 \rightarrow 268.0 and m/z 349.0 \rightarrow 225.0 for 1552-Acid (a majority of the ions differed from those reported for the ECM by +0.0-0.2 m/z).

III. Method Characteristics

In the ECM and ILV, the established LOQ and LOD in sediment were 0.003 µg/g and 0.0009 µg/g, respectively, for XDE-848 (SX-1552) and its five metabolites (p. 34; Appendix A, pp. 142, 162 of MRID 49677722; pp. 17, 20 of MRID 49677777). In the ECM, no justification or calculation was provided to support the LOQ; the LOD was defined as the concentration which was *ca.* 30% of the LOQ. In the ILV, the LOQ and LOD were reported from the ECM without justification or calculation.

Table 4. Method Characteristics

		XDE-848 (SX-1552)	1552-OHA	1552-DBE	1552-DA	1552-OHBE	1552-Acid	
Limit of Quantitation (LOQ)		0.003 µg/g						
Limit of Detection (LOD)		0.0009 µg/g						
Linearity (Least squares calibration curve r and concentration range)	ECM ¹	r ² = 0.9999 (Q)	r ² = 0.9999 (Q)	r ² = 1.0000 (Q)	r ² = 1.0000 (Q)	r ² = 1.0000 (Q)	r ² = 1.0000 (Q)	
		0.005-50 ng/mL	0.0049-49 ng/mL	0.0049-48.5 ng/mL	0.0049-49 ng/mL	0.005-50 ng/mL		
	ILV ²	r ² = 0.9996 (Q & C)	r ² = 0.9998 (Q & C)	r ² = 0.9968 (Q) r ² = 0.9972 (C)	r ² = 0.9996 (Q) r ² = 0.9994 (C)	r ² = 1.0000 (Q) r ² = 0.9998 (C)	r ² = 0.9994 (Q) r ² = 0.9996 (C)	
		0.005-10 ng/mL	0.005-50 ng/mL	0.005-10 ng/mL		0.005-50 ng/mL		
Repeatable	ECM ^{3,4}	Yes at LOQ and 10×LOQ (n = 7 each for FL and NC). Only the quantification ion was evaluated for recovery.						
	ILV ⁵	Yes at LOQ and 10×LOQ (n = 5; quantification and confirmation ions).						
Reproducible		Yes at LOQ and 10×LOQ (n = 5).						
Specific	ECM	Yes, only minor interferences (<10% of the LOQ) at the retention time of the analytes were observed in the matrix controls. Residues in the matrix controls were quantified as <LOD. Only chromatograms of the quantification ion were provided.						
		FL	Significant baseline noise was observed which disrupted peak attenuation at the LOQ.	Peaks were well defined and distinct from the baseline at LOQ and 10×LOQ fortifications.	Some baseline noise was observed at the LOQ.	Peaks were well defined and distinct from the baseline at LOQ and 10×LOQ fortifications.		
		NC	No LOQ chromatogram was provided. Some baseline noise was observed at the 10×LOQ.	No LOQ chromatogram was provided.	Some baseline noise was observed which disrupted peak attenuation at the LOQ. No 10×LOQ chromatogram was provided.	Peaks were well defined and distinct from the baseline at LOQ and 10×LOQ fortifications.		

		XDE-848 (SX-1552)	1552-OHA	1552-DBE	1552-DA	1552-OHBE	1552-Acid
	ILV	<p>Yes, only minor interferences (<10% of the LOQ) at the retention time of the analytes were observed in the matrix controls.</p> <p>Residues in the matrix controls were quantified as <LOD.</p> <p>No representative chromatograms were provided for the fortifications at the LOD or 10×LOQ, only calibrants, controls and LOQ.</p> <p>Minor baseline noise was observed which disrupted peak attenuation for a few of the analytes, most notable for SX-1552 and 1552-DBE.</p>					

Data were obtained from p. 34; Appendix A, pp. 142, 162; Appendix A, Tables (Recovery Results); Appendix A, Figures 1-6, pp. 312-317 (Linear Regressions); Appendix A, Figures 13-30, pp. 321-329 (FL Chromatograms); Appendix A, Figures 55-72, pp. 342-350 (NC Chromatograms) of MRID 49677722 ; pp. 17, 20; Tables 2-13, pp. 26-31 (Correlation Coefficients); Tables 14-25, pp. 32-43 (Control residues and LOD results); Tables 26-37, pp. 44-46 (Summary Recovery Results); Figures 11-22, pp. 63-74 (Linear regressions); Figures 30-53, pp. 82-105 (Chromatograms) of MRID 49677777 and DER Attachment 2. Q = Quantitative HPLC analysis; C = Confirmatory HPLC analysis. FL = Florida pond sediment matrix; NC = North Carolina pond sediment matrix.

* XDE-848 = [Florpyrauxifen-benzyl; XDE-848 BE; XDE-848 benzyl ester; TSN301734; X11959130; SX-1552; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-OHA = [XDE-848 hydroxy acid; TSN305649; X11966341; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-DBE = [Dechlorinated XDE-848 benzyl ester; TSN305649; X12131932; benzyl 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate]; 1552-DA = [Dechlorinated XDE-848 acid; TSN304479; X12393505; 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid]; 1552-OHBE = [XDE-848 hydroxy benzyl ester; TSN305650; X12300837; benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylate]; and 1552-Acid = [XDE-848 acid; TSN301691; X11438848; 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid].

1 ECM standard curves were reviewer-calculated based on data provided in Appendix A, Figures 1-6, pp. 312-317 of MRID 49677722 (see DER Attachment 2).

2 ILV standard curves were weighted 1/x for all analytes. ILV r^2 values are reviewer-generated for the analytes from reported r values of 0.9984-1.0000 (Q) and 0.9986-0.9999 (C; analytes/ions combined; calculated from data in Tables 2-13, pp. 26-31 and Figures 11-22, pp. 63-74 of MRID 49677777; see DER Attachment 2). Some calibrant results were excluded by the study author due to lack of linearity.

3 For the ECM, sediment matrices were well-characterized pond sediments (USDA soil texture classification; pp. 24-25, 28, 36; Tables 9-10, pp. 60-61 of MRID 49677722). The Florida pond sediment (FL) was classified as sand (Stations A-C; 96% sand, 3% silt, 1% clay; pH 7.3-8.0; 0.96-3.3% organic matter). The North Carolina pond sediment (NC) was classified as sandy loam (Stations A and B; 67-71% sand, 23-25% silt, 6-8% clay; pH 6.1-6.2; 1.5-2.9% organic matter) and loam (Station C; 45% sand, 41% silt, 14% clay; pH 6.2; 4.3% organic matter). The Florida pond was sourced by a well; the North Carolina pond was sourced by a source reservoir pond.

4 For the ILV, the sediment was the same North Carolina pond sediment which was used in the ECM, based on the sediment characterization data (p. 15; Appendix 2, pp. 114-116 of MRID 49677777).

IV. Method Deficiencies and/or Reviewer's Comments

1. The submitted ECM which was contained in Appendix A of MRID 49677722 (pp. 123-447) was not the original ECM for the submitted ILV MRID 49677777 (pp. 14, 22 of MRID 49677777). ILV MRID 49677777 was performed to validate EPL Bio Analytical Services Method 477G696C "Determination of XDE-848 Benzyl Ester (SX-1552) and Five Metabolites (1552-Acid, 1552-OHBE, 1552-OHA, 1552-DBE and 1552-DA) in Sediment". EPL Bio Analytical Services Method 477G696C was authored by F. Claussen in 2014 (incomplete reference detail; Appendix A, p. 166 (Ref. 5) of MRID 49677722). However, the submitted ECM in Appendix A of MRID 49677722 was performed using EPL Bio Analytical Services Method 477G696C and referenced this original ECM [pp. 34-35; p. 48 (Ref. 12); Appendix A, pp. 149, 166 (Ref. 5) of MRID 49677722]. No deviations to the original ECM were reported in the submitted ECM; however, it is preferred that the original method document is provided for method validation assessment.

Note from EPA reviewer: The reviewer noted that Appendix B, p. 448 of MRID 49677722, appears to be the original ECM; however, it was not fully reviewed by the primary reviewer. The following is a brief report of Appendix B: In this part of the study, the FL and NC waters were tested at LOD (n = 1), LOQ (n = 7 for all six chemicals), 10xLOQ (n = 2), and 50 ng/mL (n = 2). Mean values and relative standard deviations were within guideline criteria, but note that the number of samples tested at 10xLOQ was <7.

For sediments from FL and NC, samples were tested at LOD (n = 1), LOQ (n = 7 for all six chemicals), 10xLOQ (n = 2), and 0.15 µg/g (n = 2). Mean values and relative standard deviations were within guideline criteria, with one exception, but note that the number of samples tested at 10xLOQ was <7. The only exception is that the mean value at 10xLOQ was 122% for des-chloro XDE-848 benzyl ester for the FL water samples (n=2).

In the provided data set of the study, example chromatograms at 10xLOQ were provided. Only two samples were tested at 10xLOQ.

2. The estimations of the LOQ and LOD in the ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136. In the ECM, no justification or calculation was provided to support the LOQ; the LOD was defined as the concentration which was *ca.* 30% of the LOQ (p. 34; Appendix A, pp. 142, 162 of MRID 49677722; pp. 17, 20 of MRID 49677777). In the ILV, the LOQ and LOD were reported from the ECM without justification or calculation. Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples. Additionally, the lowest toxicological levels of concern in soil/sediment were not reported. An LOQ above toxicological level of concern results in an unacceptable method classification.
3. In the ECM, due to baseline noise and missing chromatograms, representative ECM chromatograms did not support the specificity of the method for SX-1552 and 1552-DBE

in both sediments and for 1552-OHA in the NC sediment (Appendix A, Figures 13-30, pp. 321-329; Appendix A, Figures 55-72, pp. 342-350 of MRID 49677722). For SX-1552, significant baseline noise which disrupted peak attenuation at the LOQ was observed in the FL sediment. The results in the NC sediment were inconclusive since the LOQ chromatogram was missing, but baseline noise was observed in the 10×LOQ chromatogram of the NC sediment. For 1552-DBE, some baseline noise at the LOQ was observed in both sediment, although peak attenuation disruption was only observed in the NC sediment. The 10×LOQ chromatogram of the NC sediment was missing. For 1552-OHA, the LOQ chromatogram was missing for the NC sediment.

4. In the ILV, representative chromatograms were not complete. Representative chromatograms were not provided for the reagent blank and fortifications at the LOD or 10×LOQ, only calibrants, controls and LOQ (Figures 30-53, pp. 82-105 of MRID 49677777). A reagent blank was included in the validation (p. 17).

In the ECM, representative chromatograms were not complete, only chromatograms of the quantification ion were included. Several NC chromatograms were missing: SX-1552 at LOQ; 1552-DBE at 10×LOQ; and 1552-OHA at LOQ [Appendix A, Figures 13-30, pp. 321-329 (FL Chromatograms); Appendix A, Figures 55-70, pp. 324-350 (NC Chromatograms) of MRID 49677722]. Additionally, representative chromatograms were not provided for the reagent blank. It could not be determined if a reagent blank was included in the validation (Appendix A, pp. 150-151).

5. The ECM calculations allowed for recovery data to be corrected for residues found in the control however, no residues were quantified in any of the controls (Appendix A, pp. 158-160; Appendix A, Figures 13-18, pp. 321-323; Appendix A, Figures 55-60, pp. 342-344). The residues were quantified as <LOD.
6. In the ECM, recovery results and representative chromatograms were only provided for the quantitation ion (Appendix A, Tables 11-16, pp. 179-190; Appendix A, Figures 13-30, pp. 321-329; Appendix A, Figures 55-72, pp. 342-350 of MRID 49677722). In the tables, the ion transition was not reported, but the recovery values matched those reported in the chromatograms, where the ion transition was noted (in the raw chromatogram). A confirmatory method is not usually required when LC/MS and GC/MS is the primary method.
7. The results from the sediment travel spikes and sediment field dissipation studies were included in the ECM, but not addressed in this method validation review (Appendix A, pp. 150, 162-163 of MRID 49677722). Tank mix analyses were also studied for the Florida and North Carolina sites (Appendix A, p. 161).
8. Isotope internal standards or dechlorinated standards were used facilitate analysis (Appendix A, pp. 150-152 of MRID 49677722; p. 19; Appendix 1, pp. 110-111, 113 of MRID 49677777).

9. The ILV reported that no communications occurred between the ILV laboratory and the study director (p. 19 of MRID 49677777).
10. In the ILV, matrix effects were studied (p. 19; Tables 38-43, pp. 47-52 of MRID 49677777). In the ILV, matrix effects were determined to be insignificant in the matrices (<20%) for all analytes in the sediment. Solvent standards were used in the ILV.
11. It was reported for the ILV that the analytical procedure for one set of 19 samples (five calibration standards, two controls, one LOD sample, five LOQ samples, five 10×LOQ samples and one reagent blank) required approximately 6 hours for laboratory preparation (p. 17 of MRID 49677777). The LC/MS/MS was conducted unattended (*ca.* 8 hours or overnight). The interpretation of data required approximately 4 hours. The overall time to complete a set of samples (14 samples, not including calibration standards) was *ca.* 1.5 calendar days.

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.

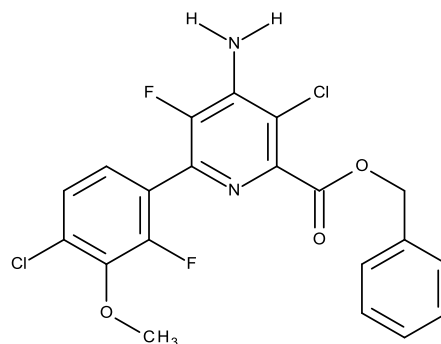
Attachment 1: Chemical Names and Structures**XDE-848 Benzyl Ester (Rinskor, XR-848-BE, XR-848 Benzyl, X11959130, TSN301734)**

IUPAC Name: Benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate

CAS Name: Phenylmethyl ester 3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoro-2-pyridinecarboxylic acid

CAS Number: 1390661-72-9

SMILES String: [H]N([H])c1c(c(nc1Cl)C(=O)OCc2ccccc2)c3ccc(c(c3F)OC)Cl)F

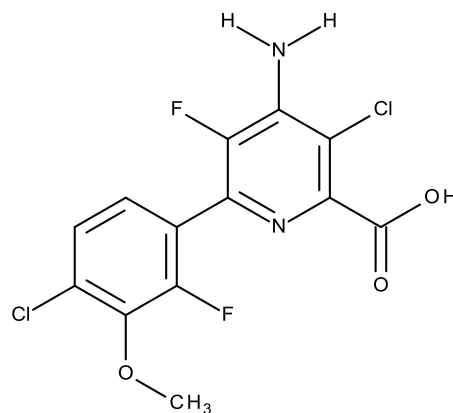
**XDE-848 acid (X11433848, TSN304667)**

IUPAC Name: 4-Amino-3-chloro-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid

CAS Name: --

CAS Number: --

SMILES String: [H]N([H])c1c(c(nc1Cl)C(=O)O)c2ccc(c(c2F)OC)Cl)F



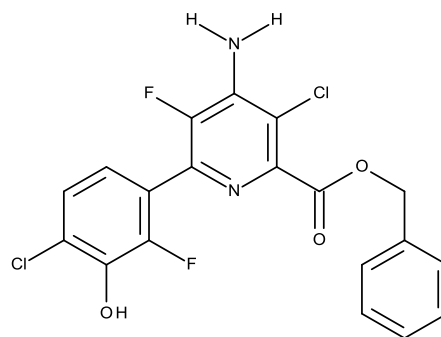
XDE-848 Hydroxy Benzyl Ester (X12300837; TSN305650; XDE-848 BH; Benzyl hydroxyl; 1552-OHBE; OHBE)

IUPAC Name: Benzyl 4-amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylate

CAS Name: --

CAS Number: --

SMILES String: [H]N([H])c1c(c(nc(c1Cl)C(=O)OCc2ccccc2)c3ccc(c(c3F)O)Cl)F

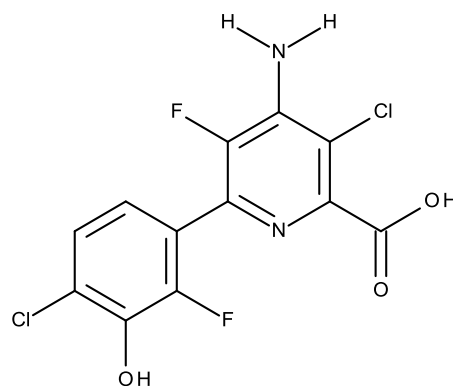
**XDE-848 Hydroxy Acid (X11966341; TSN305649; XDE-848 HA; Hydroxy acid; 1552-OHA; OHA)**

IUPAC Name: 4-Amino-3-chloro-6-(4-chloro-2-fluoro-3-hydroxyphenyl)-5-fluoropyridine-2-carboxylic acid

CAS Name: --

CAS Number: --

SMILES String: [H]N([H])c1c(c(nc(c1Cl)C(=O)O)c2ccc(c(c2F)O)Cl)F



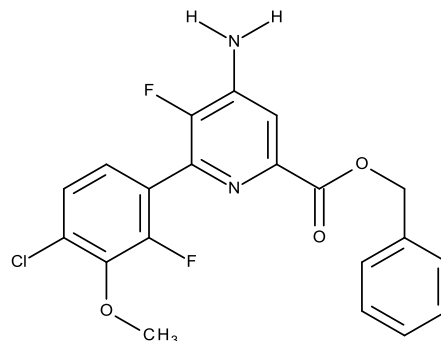
Dechlorinated XDE-848 Benzyl Ester (X12131932; TSN304497; De-chloro BE; Dechlorinated 848 BE; 1552-DBE; DBE)

IUPAC Name: Benzyl 4-amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylate

CAS Name: --

CAS Number: --

SMILES String: [H]N([H])c1cc(nc(c1F)c2ccc(c(c2F)OC)Cl)C(=O)OCc3ccccc3

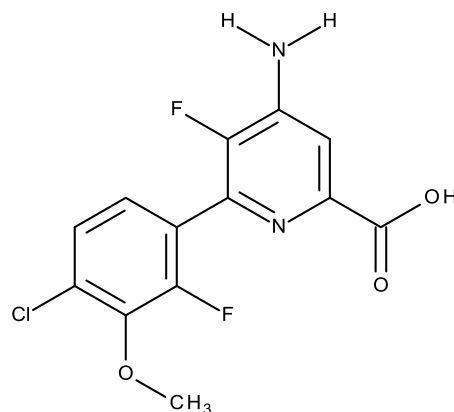
**Dechlorinated XDE-848 Acid (X12393505; TSN304479; De-chloro acid; Dechlorinated 848 BE; 1552-DA; DA)**

IUPAC Name: 4-Amino-6-(4-chloro-2-fluoro-3-methoxyphenyl)-5-fluoropyridine-2-carboxylic acid

CAS Name: --

CAS Number: --

SMILES String: [H]N([H])c1cc(nc(c1F)c2ccc(c(c2F)OC)Cl)C(=O)O



Attachment 2: Calculations

Chemical: XDE-848
 PC: 030093
 MRIDs: 49677722/49677777
 Guideline: 850.6100

ECM Recoveries at LOD of XDE-848 (SX-1552) and its Products

SX-1552		
Fortified (µg a.i./g)	Found (µg/L)	Recovery (%)
0.0009	Quantiation ion	
	0.000620	69
	Confirmation ion	
	0.000697	77

Fortified (µg a.i./g)	1552-OHA		1552-DBE		1552-DA		1552-OHBE		1552-Acid	
	Found (µg/L)	Recovery (%)	Found (µg/L)	Recovery (%)	Found (µg/L)	Recovery (%)	Found (µg/L)	Recovery (%)	Found (µg/L)	Recovery (%)
0.0009	Quantiation ion									
	0.00076	85	0.00097	108	0.00099	110	0.00099	110	0.00104	116
	Confirmation ion									
	0.00080	89	0.00063	69	0.00083	92	0.00098	108	0.00090	99

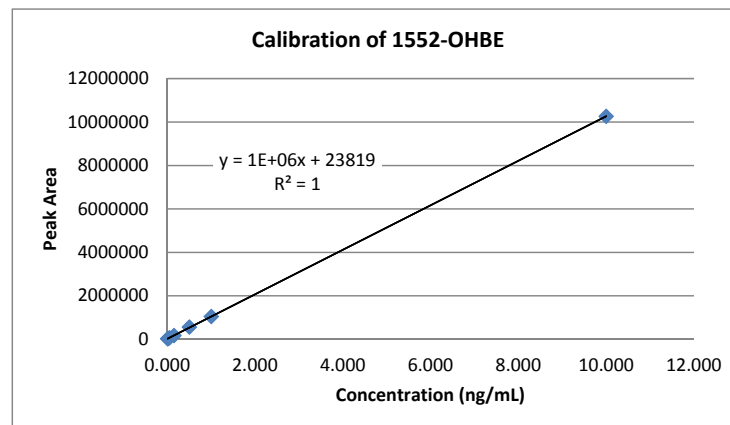
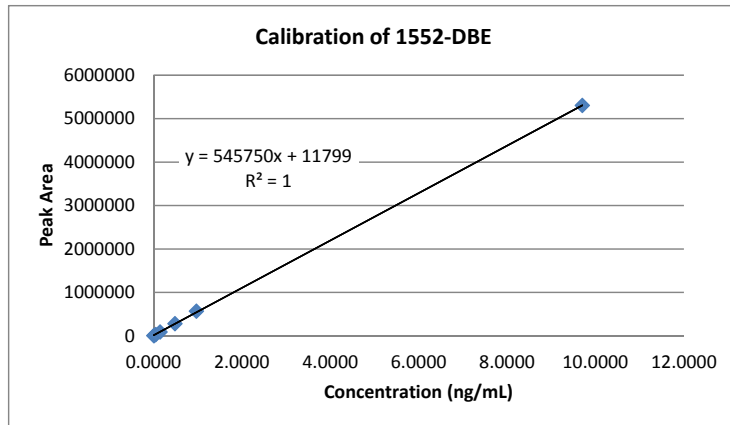
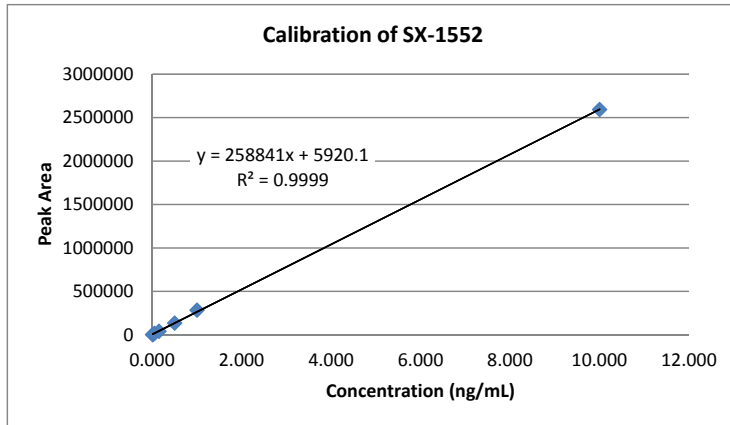
Results from Tables 14-25, pp. 32-43 of MRID 49677777.

Chemical: XDE-848
 PC: 030093
 MRIDs: 49677722/49677777
 Guideline: 850.6100

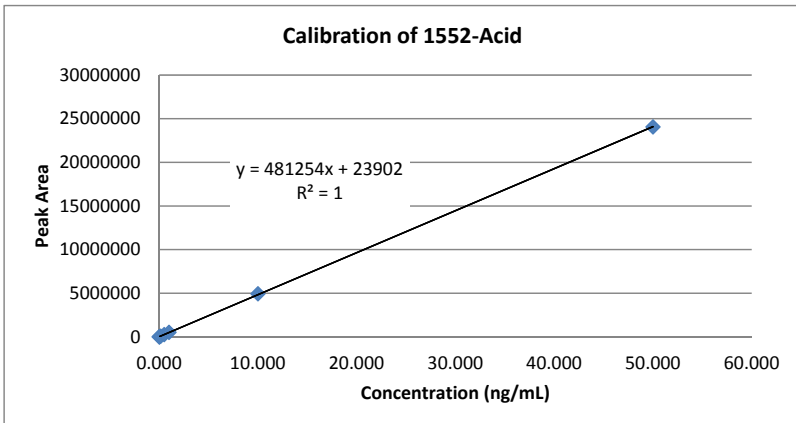
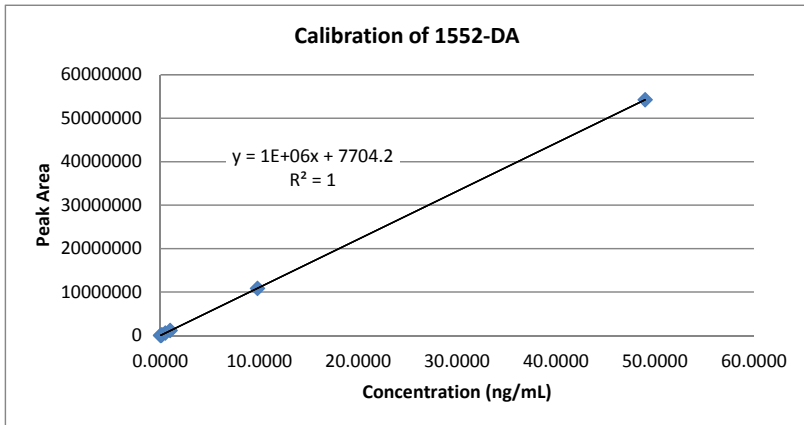
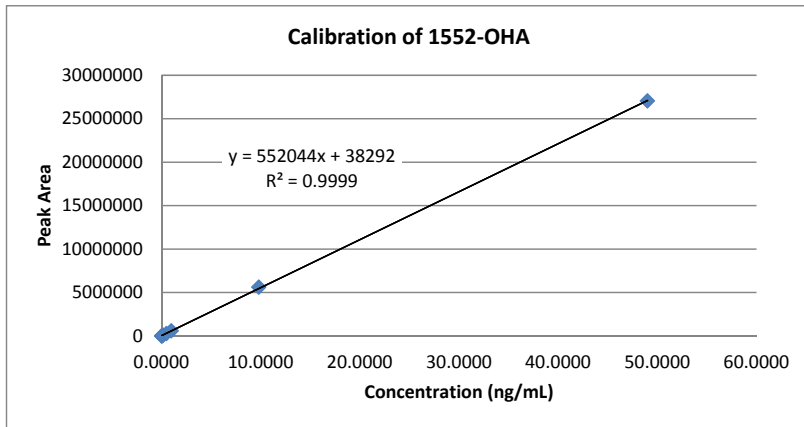
ECM Calibration Curves

Calibration Curves	SX-1552		1552-OHA		1552-DBE		1552-DA		1552-OHBE		1552-Acid	
	Amount ng/mL	Peak Area counts	Amount ng/mL	Peak Area counts	Amount ng/mL	Peak Area counts	Amount ng/mL	Peak Area counts	Amount ng/mL	Peak Area counts	Amount ng/mL	Peak Area counts
	0.005	1888	0.0049	3723	0.0049	5259	0.0049	9599	0.005	21563	0.005	4374
	0.015	4454	0.0147	9370	0.0146	9562	0.049	20234	0.015	30904	0.015	8107
	0.050	16956	0.049	29855	0.0485	32186	0.049	60950	0.050	70348	0.050	27822
	0.150	40993	0.147	89346	0.146	88108	0.147	169493	0.150	173125	0.150	78148
	0.500	134195	0.490	285967	0.485	281127	0.490	561715	0.500	564392	0.500	254172
	1.00	284034	0.98	603018	0.97	568246	0.98	1147775	1.00	1045252	1.00	515973
	10.00	2592532	9.80	5622205	9.70	5302739	9.80	10831740	10.00	10264814	10.00	4939688
	50.00	12635313	49.00	27053590	48.50	26828749	49.00	54247950	50.00	49216136	50.00	24065906

Results (Peak Areas) from Appendix A, Figures 1-6, pp. 312-317 of MRID 49677722.



Chemical: XDE-848
PC: 030093
MRIDs: 49677722/49677777
Guideline: 850.6100



Chemical: XDE-848
 PC: 030093
 MRIDs: 49677722/49677777
 Guideline: 850.6100

ILV Calibration Curve Correlation Coefficients r (1/x weighting) converted to r2

Analyte	Water			
	First Ion Transition (Q)		Second Ion Transition (C)	
	Reported r	Calculated r2	Reported r	Calculated r2
SX-1552	0.9998	0.9996	0.9998	0.9996
1552-OHA	0.9999	0.9998	0.9999	0.9998
1552-DBE	0.9984	0.9968	0.9986	0.9972
1552-DA	0.9998	0.9996	0.9997	0.9994
1552-OHBE	1.0000	1.0000	0.9999	0.9998
1552-Acid	0.9997	0.9994	0.9998	0.9996

Results (r values) from Tables 2-13, pp. 26-31 and Figures 11-22, pp.63-74 of MRID 49677777.