Addendum No. 1 for MRIDs 49775206 and 49775207

Study Title: ECM, MRID 49775206: VALIDATION OF DRAFT RESIDUE METHOD CAM-0004/003 FOR THE DETERMINATION OF PHENOXY ACIDS AND THEIR CORRESPONDING 2 ETHYL-HEXYL ESTERS IN SURFACE WATER, SOIL AND AIR

ILV, MRID 49986901 (supersedes 49775207): Phenoxy Herbicides - Independent Laboratory Validation of the Analytical Method CAM-0004/003 for the Determination of Phenoxy Acids and Their Corresponding 2 Ethyl-Hexyl Esters in Drinking Water by LC-MS/MS

PC code: 019201 (MCPB), 030001 (2,4-D), 030501 (MCPA), 030801 (2,4-DB), 031402 (2,4-DP-p), 129046 (Mecoprop-p)

Guideline Number: 850.6100

- This DER is addended based on a review of an amended ILV report (MRID 49986901, which supersedes 49775207) with additional information to satisfy the data requirement for a guideline 850.6100 study. The study classification is upgraded from unacceptable to **Acceptable**.

- Reasons for changes:

    - The amended report (MRID 49986901) provided the following information:
        1) A clarification that no communication regarding the analytical method was made between Eurofins Agroscience Services Ghem Ltd and CEMAS or Nufarm during the conduct of the ILV;
        2) An addition of chromatography results; and
        3) An addition of method parameters used by Eurofins during the conduct of the ILV.

    This information addresses the deficiencies documented in the original DER that were the basis for the original study classification of Unacceptable but upgradeable: "*Any communication between the ILV and ECM staff was not documented, summarized, or discussed. The ECM and ILV reports were incomplete, missing details of the Materials and Methods. Chromatograms were incomplete in the ECM and ILV; in the ILV, no chromatograms were noted as those of the 2-EH analytes.*"

Revised by: He Zhong Zhong, He Digitally signed by Zhong, He Date: 2017.05.18 13:30:06 -04'00' Date: 5-17-17

Secondarily reviewed by: Greg Orrick Greg Orrick 2017.05.18 13:40:17 -04'00' Date: 5-17-17

Analytical method for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, MCPB, Mecoprop-p and their 2-EHs in water

Reports:	ECM: EPA MRID No. 49775206. Allen, L. 2014. VALIDATION OF DRAFT RESIDUE METHOD CAM-0004/003 FOR THE DETERMINATION OF PHENOXY ACIDS AND THEIR CORRESPONDING 2 ETHYL-HEXYL ESTERS IN SURFACE WATER, SOIL AND AIR. Study No. CEMS-6230. Report No. CEMR-6230. Report prepared by CEM Analytical Services Limited (CEMAS), Berkshire, United Kingdom; sponsored by Nufarm UK Limited, Bradford, West Yorkshire, United Kingdom; and submitted by Nufarm Americas, Alsip, Illinois; 364 pages. Final report issued August 5, 2014. ILV: EPA MRID No. 49775207. Weir, A. 2014. Phenoxy Herbicides – Independent Laboratory Validation of the Analytical Method CAM-0004/003 for the Determination of Phenoxy Acids and Their Corresponding 2 Ethyl-Hexyl Esters in Drinking Water by LC-MS/MS – Final Report on Study S14-01199. EAS Study No. S14-01199. Report prepared by Eurofins Agroscience Services Chem Ltd., Derbyshire, United Kingdom; sponsored by Nufarm UK Limited, Bradford, West Yorkshire, United Kingdom; and submitted by Nufarm Americas, Alsip, Illinois; 141 pages. Final report issued October 1, 2014.
Document No.:	MRIDs 49775206 & 49775207
Guideline:	850.6100
Statements:	ECM: The study was conducted compliance with OECD, UK and The Department of Health of the Government of the United Kingdom Principles of Good Laboratory Practice (GLP; p. 3; Appendix 2, pp. 363-364 of MRID 49775206). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4; Appendix 2, pp. 363-364). Authenticity statements were included with the GLP and Quality Assurance statements. ILV: The study was conducted compliance with OECD, UK and The Department of Health of the Government of the United Kingdom Principles of Good Laboratory Practice (GLP; pp. 3-4; Appendix A, p. 95 of MRID 49775207). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-5; Appendix A, p. 95). Authenticity statements were included with the GLP and Quality Assurance statements.
Classification:	This analytical method is classified as Unacceptable but upgradeable. Any communication between the ILV and ECM staff was not documented, summarized, or discussed. The ECM and ILV reports were incomplete, missing details of the Materials and Methods. Chromatograms were incomplete in the ECM and ILV; in the ILV, no chromatograms were noted as those of the 2-EH analytes.

PC Code:	031402 (2,4-DP-p) ; 019201 (MCPB); 030001 (2,4-D) ; 030501 (MCPA) ; 030801 (2,4-DB) ; 129046 (Mecoprop-p)	
Reviewer:	Lewis Ross Brown, III Environmental Biologist	Signature: Date: June 09, 2016

All cited page numbers refer to those written in the bottom, right-hand corner of the pages of the MRIDs.

Executive Summary

This analytical method, CEMAS CAM-0004/003 and CEMAS Study No. CEMS-6230, is designed for the quantitative determination of 2,4-DP-p, 2,4-DP-p 2-EH, 2,4-D, 2,4-D 2-EH, 2,4-DB, 2,4-DB 2-EH, MCPA, MCPA 2-EH, MCPB, and MCPB 2-EH at the LOQ of 0.01 µg/L and of Mecoprop-p and Mecoprop-p 2-EH at the LOQ of 0.02 µg/L in water using LC/MS/MS. The lowest toxicological level of concern in water was not related to the LOQ for the analytes. Analytes were identified using two ion pair transitions, one quantification and one confirmation. The 2 ethyl-hexyl ester analytes (2-EH analytes) were intended to be fully hydrolysed back to the corresponding esters during sample processing, so 2-EH analytes were monitored with the same ion transitions as the corresponding acids. The ECM validated the method using characterized surface water; the ILV validated the method for all analytes in the second trial with no reported modifications using uncharacterized, bottled, drinking water. Tap water was used as the control matrix in the first ILV trial, but residues of some analytes were detected above 30% of the LOQ in the control samples. It could not be determined if the ILV was provided with the most difficult matrix with which to validate the method. In the ECM and ILV, fortifications of Mecoprop-p and Mecoprop-p 2-EH were not performed at 10×LOQ, only 5×LOQ. Representative chromatograms were incomplete in the ECM and ILV; in the ILV, no chromatograms were noted as those of the 2-EH analytes. In the ECM, matrix interferences were >30% of the LOQ in MCPB chromatograms, and the recovery was 121% for the confirmation ion of MCPA 2-EH at 10×LOQ. Due to lack of method details, updates to the ECM and ILV study reports were needed. ECM MRID 49775206 should be updated with the method details of CEMAS CAM-0004/003 in order to be a complete ECM. ILV MRID 49775207 should be updated with the analytical method details which were used by the ILV laboratory.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide[1]	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
2,4-DP-p[2]	49775206	49775207		Water[4,5]	05/08/2014 (ECM Validation)[6] 11/08/2014 (Method date)[7]	Nufarm UK LTD	LC/MS/MS	0.01 µg/L
2,4-DP-p 2-EH								
2,4-D								
2,4-D 2-EH								
2,4-DB								
2,4-DB 2-EH								
MCPA								
MCPA 2-EH								
MCPB								
MCPB 2-EH								
Mecoprop-p[3]								0.02 µg/L
Mecoprop-p 2-EH								

1 2,4-DP-p = Dichloroprop-p; (R+)-2-(2,4-dichlorophenoxy)propionic acid. 2,4-DP-p 2-EH = (R+)-2-(2,4-dichlorophenoxy)propionic acid 2-ethylhexyl ester. 2,4-D = (2,4-Dichlorophenoxy)acetic acid; 2,4-D 2-EH = 2,4-Dichlorophenoxyacetic acid, 2-ethylhexyl ester. 2,4-DB = 4-(2,4-Dichlorophenoxy)butyric acid; 2,4-DB 2-EH = 4-(2,4-Dichlorophenoxy)butyric acid, 2-ethylhexyl ester. MCPA = 4-Chloro-2-methylphenoxyacetic acid; MCPA 2-EH; 4-Chloro-2-methylphenoxyacetic acid, 2-ethylhexyl ester. MCPB = 4-(4-Chloro-2-methylphenoxy)butyric acid. MCPB 2-EH = 4-(4-Chloro-2-methylphenoxy)butyric acid, 2-ethylhexyl ester. Mecoprop-p = CMPP-p; (R+)- 2-(4-chloro-2-methylphenoxy)propionic acid. Mecoprop-p 2-EH = (R+)-2-(4-chloro-2-methylphenoxy)propionic acid, 2-ethylhexyl ester.

2 2,4-DP-p included Dichloroprop and Dichloroprop-p which are isomers which cannot not distinguished by LC/MS/MS (Appendix B, p. 98 of MRID 49775207).

3 Mecoprop-p included Mecoprop and Mecoprop-p which are isomers which cannot not distinguished by LC/MS/MS (Appendix B, p. 98 of MRID 49775207).

4 In the ECM, three surface water specimens were reported: CCON/037/005 (pH 5.8, dissolved organic carbon 2.27 mg/L); CCON/037/007 (pH 6.6, dissolved organic carbon 2.66 mg/L); and CCON/037/008 (pH 7.3, dissolved organic carbon 5.94 mg/L; p. 23; Table 47, p. 72 of MRID 49775206). The study author did not specify if the water specimens were mixed for samples or used individually. The specific sources of the water specimens were not reported.

5 Bottled, still mineral water, purchased from a local supermarket, was used in the ILV; no characterization data was reported (pp. 11, 20 of MRID 49775207).

6 From CEMAS Study No. CEMS-6230 (p. 1 of MRID 49775206).

7 From CEMAS CAM-0004/003 (Appendix C, p. 111 of MRID 49775207).

I. Principle of the Method

Water samples (100 mL, room temperature) were measured into 100-mL glass bottles and fortified, as necessary (0.2 mL of 0.01 µg/mL or 0.1 mL of 0.1 µg/mL for Mecoprop-p; 0.1 mL of 0.01 µg/mL or 0.1 mL of 0.1 µg/mL for all other analytes; p. 24 of MRID 49775206; Appendix C, pp. 112, 120 of MRID 49775207; see Reviewer's Comment #1). The samples were mixed gently by hand with 1 mL of sodium hydroxide hydrolysis solution [47% sodium hydroxide:deionized water (15:85, v:v)]. The samples were placed in an oven set to 85°C overnight to hydrolyze. After cooling, the samples were acidified by mixing gently by hand with 1 mL of 15N sulphuric acid. A Strata X SPE cartridge (30 mg/ 3 mL) was pre-conditioned with 3 mL of methanol then 3 mL of 0.05% hydrochloric acid in water. The entire water sample was loaded onto the pre-conditioned column. The column was washed with 3 mL of methanol:water:hydrochloric acid (40:60:0.5, v:v:v) and 3 mL of deionized water. The analytes were eluted with 2 x 2 mL of 1% ammonium in acetonitrile. The eluate was reduced to dryness (method not specified). The residue was reconstituted in 0.5 mL of injection buffer [0.2% formic acid in water:acetonitrile (60:40, v:v)]. Internal standard [5 µL of 5 µg/mL solution of (2,4,6-triphenoxy)acetic acid (2,4,6-TMAA) or (4-chloro-3,5-dimethylphenoxy)acetic acid (4-CDMAA)] was added with gently mixing to all standards and samples prior to LC/MS/MS (final sample concentration 0.2 L/mL; Appendix C, pp. 116, 121 of MRID 49775207).

Samples are analyzed using an Applied Biosystems Sciex API4000 triple quadrupole mass spectrometer with Symbiosis Pharma liquid chromatograph (Appendix C, pp. 122-123 of MRID 49775207). The following LC conditions were used: Onyx C18 monolithic column (3.0 mm x 100 mm, column temperature ambient), Chromolith RP-18 end capped guard column (5 mm x 3 mm), mobile phase of (A) HPLC grade water + 0.1% formic acid and (B) methanol + 0.1% formic acid [percent A:B (v:v) at 0.01-0.03 min. 55:45, 6.00 min. 25:75, 6.01-7.15 min. 5:95, 7.16-9.00 min. 55:45], split flow 1:4 to the mass spectrometer, and injection volume of 40 µL. The MRM parameters were ESI negative mode for all analytes. Two ion pair transitions were monitored for each analyte (quantitation and confirmation, respectively): *m/z* 232.9→160.8 and *m/z* 234.9→162.8 for 2,4-DP-p and 2,4-DP-p 2-EH, *m/z* 218.8→161.0 and *m/z* 220.8→162.9 for 2,4-D and 2,4-D 2-EH, *m/z* 247.0→161.0 and *m/z* 249.0→163.0 for 2,4-DB and 2,4-DB 2-EH, *m/z* 199.0→140.9 and *m/z* 200.9→142.9 for MCPA and MCPA 2-EH, *m/z* 227.0→140.9 and *m/z* 229.0→142.9 for MCPB and MCPB 2-EH, and *m/z* 212.9→140.9 and *m/z* 215.0→142.9 for Mecoprop-p and Mecoprop-p 2-EH. One ion transition was monitored for each internal standard: *m/z* 193.0→135.0 for 2,4,6-TMAA, and *m/z* 212.9→155.0 for 4-CDMAA. Expected retention times were minutes for 4.32-4.33 min. for 2,4-DP-p and 2,4-DP-p 2-EH, 3.35 min. for 2,4-D, 3.47 min. for 2,4-D 2-EH, 4.99-5.01 min. for 2,4-DB and 2,4-DB 2-EH, 3.54-3.56 min. for MCPA and MCPA 2-EH, 5.10 min. for MCPB and MCPB 2-EH, and 5.00 min. for Mecoprop-p and Mecoprop-p 2-EH (Figures 49-90, pp. 121-162 of MRID 49775206).

In the ILV, no modifications were reported; the ECM was performed as written in CEMAS CAM-0004/003, which was included in Appendix C of the ILV (p. 20; Appendix B, pp. 99, 104-110; Appendix C, pp. 111-129 of MRID 49775207). The analytical instrument, equipment and parameters were not reported. The monitored ion transitions were the same as those in the ECM (Tables 1-6, pp. 25-32 of MRID 49775207). Expected retention times were minutes for 4.20 min. for 2,4-DP-p and 2,4-DP-p 2-EH, 3.20-3.21 min. for 2,4-D and 2,4-D 2-EH, 4.89-4.90 min. for

2,4-DB and 2,4-DB 2-EH, 3.46 min. for MCPA and MCPA 2-EH, 5.04 min. for MCPB and MCPB 2-EH, and 4.32 min. for Mecoprop-p and Mecoprop-p 2-EH (Figures 13-60, pp. 46-93).

In the ECM and ILV, the LOQ was 0.01 µg/L for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, and MCPB, and 0.02 µg/L for Mecoprop-p (pp. 21, 27 of MRID 497758206; p. 11; Appendix C, pp. 112, 127 of MRID 49775207). The calculated LODs were 0.000481-0.001340 µg/L for 2,4-DP-p, 0.000143-0.000528 µg/L for 2,4-D, 0.001006-0.001062 µg/L for 2,4-DB, 0.000195-0.001623 µg/L for MCPA, 0.000129-0.000621 µg/L for MCPB, and 0.000385-0.001420 µg/L for Mecoprop-p in the ECM (Table 37, p. 64 of MRID 497758206). The LOD was reported in the ILV as equivalent to the 0.6 ng/mL standard, which equated to 30% of the LOQ for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, and MCPB, and 15% of the LOQ for Mecoprop-p (pp. 13-14 of MRID 49775207).

II. Recovery Findings

ECM (MRID 49775206): Mean recoveries and relative standard deviations (RSD) were within guideline requirements (mean 70-120%; RSD ≤20%) for analysis for 2,4-DP-p, 2,4-DP-p 2-EH, 2,4-D, 2,4-D 2-EH, 2,4-DB, 2,4-DB 2-EH, MCPA, MCPA 2-EH, MCPB, and MCPB 2-EH at the LOQ (0.01 µg/L) and 10×LOQ (0.1 µg/L) in surface water, except for the mean recovery for the confirmation ion of MCPA 2-EH at 10×LOQ (121%; Tables 1-6, pp. 31-36; DER Attachment 2). Mean recoveries and RSDs were within guideline requirements for analysis for Mecoprop-p and Mecoprop-p 2-EH at the LOQ (0.02 µg/L) and 5×LOQ (0.1 µg/L) in surface water; no samples were prepared at 10×LOQ. Results were comparable or fairly comparable between quantification and confirmation ions. Standard deviation were reviewer-calculated because the study author did not provide these values. Analytes were identified using two ion pair transitions, one quantification and one confirmation. The 2 ethyl-hexyl ester analytes (2-EH analytes) were intended to be fully hydrolysed back to the corresponding esters during sample processing, so 2-EH analytes were monitored with the same ion transitions as the corresponding acids (Table 1-6, pp. 31-36 of MRID 49775206; Appendix C, pp. 115, 123 of MRID 49775207). Sample recoveries were corrected for residues quantified in the controls (ranged <10% to *ca*. 33% of the LOQ for all analytes); residues were quantified in the controls for all analytes (Figures 49-90, pp. 121-162 of MRID 49775206; Appendix C, p. 124 of MRID 49775207). Surface water was characterized; three surface water specimens were reported in the study: CCON/037/005 (pH 5.8, dissolved organic carbon 2.27 mg/L); CCON/037/007 (pH 6.6, dissolved organic carbon 2.66 mg/L); and CCON/037/008 (pH 7.3, dissolved organic carbon 5.94 mg/L; p. 23; Table 47, p. 72 of MRID 49775206). The study author did not specify if the water specimens were mixed for samples or used individually. The specific sources of the water specimens were not reported.

ILV (MRID 49775207): Mean recoveries and relative standard deviations (RSD) were within guideline requirements (mean 70-120%; RSD ≤20%) for analysis for 2,4-DP-p, 2,4-DP-p 2-EH, 2,4-D, 2,4-D 2-EH, 2,4-DB, 2,4-DB 2-EH, MCPA, MCPA 2-EH, MCPB, and MCPB 2-EH at the LOQ (0.01 µg/L) and 10×LOQ (0.1 µg/L) and for analysis for Mecoprop-p and Mecoprop-p 2-EH at the LOQ (0.02 µg/L) and 5×LOQ (0.1 µg/L) in surface water (Tables 1-6, pp. 27-32; DER Attachment 2). No samples of Mecoprop-p and Mecoprop-p 2-EH were prepared at 10×LOQ. Results were comparable or fairly comparable between quantification and

confirmation ions. Standard deviation were reviewer-calculated because the study author did not provide these values. Analytes were identified using the same two ion pair transitions as the ECM. Sample recoveries were corrected for residues quantified in the controls; residues were only quantified in the controls for 2,4-D and MCPA (*ca*. 3% of the LOQ; chromatograms for the 2-EH analytes were not provided; p. 22; Figures 13-60, pp. 46-93). The drinking water was bottled, still mineral water purchased from a local supermarket; no characterization data was reported (pp. 11, 20). The method was validated for all analytes in the second trial, after an alternative source of control matrix was used; tap water was used as the control matrix in the first trial, but residues of some analytes were detected above 30% of the LOQ in the control samples (pp. 11, 20). No modifications of CEMAS CAM-0004/003 were reported by ILV (see Reviewer's Comment #1).

Table 2. Initial Validation Method Recoveries for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, MCPB, Mecoprop-p and their 2-EHs in Water[1]

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Surface Water[2]						
	Quantification ion[3]					
2,4-DP-p	0.01 (LOQ)	5	79-93	89	6	6.5
	0.1	5	96-120	105	11	10.0
2,4-DP-p 2-EH	0.01 (LOQ)	5	84-108	99	10	10.3
	0.1	5	87-101	93	5	5.4
2,4-D	0.01 (LOQ)	5	89-97	93	4	3.8
	0.1	5	98-112	105	5	4.7
2,4-D 2-EH	0.01 (LOQ)	5	101-126	113	11	9.3
	0.1	5	105-115	110	4	3.6
2,4-DB	0.01 (LOQ)	5	75-88	80	5	6.3
	0.1	5	79-113	94	14	14.8
2,4-DB 2-EH	0.01 (LOQ)	5	81-97	91	6	6.6
	0.1	5	91-102	96	4	4.6
MCPA	0.01 (LOQ)	5	98-110	106	5	4.4
	0.1	5	104-134	117	14	11.7
MCPA 2-EH	0.01 (LOQ)	5	108-131	117	9	8.1
	0.1	5	110-128	118	7	5.7
MCPB	0.01 (LOQ)	5	69-89	78	10	12.9
	0.1	5	92-117	102	9	9.0
MCPB 2-EH	0.01 (LOQ)	5	92-113	98	9	8.7
	0.1	5	101-112	106	4	4.0
Mecoprop-p	0.02 (LOQ)	5	109-122	116	5	4.5
	0.1	5	97-116	107	9	8.1
Mecoprop-p 2-EH	0.02 (LOQ)	5	82-101	96	8	8.1
	0.1	5	87-124	109	15	13.3
	Confirmation ion[3]					
2,4-DP-p	0.01 (LOQ)	5	82-90	86	3	3.6
	0.1	5	92-120	105	12	11.4
2,4-DP-p 2-EH	0.01 (LOQ)	5	87-111	99	11	11.1
	0.1	5	84-116	100	12	11.8

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
2,4-D	0.01 (LOQ)	5	87-103	95	7	7.0
	0.1	5	99-115	108	7	6.4
2,4-D 2-EH	0.01 (LOQ)	5	101-125	113	10	8.7
	0.1	5	106-115	110	4	3.8
2,4-DB	0.01 (LOQ)	5	76-92	87	7	7.4
	0.1	5	81-102	89	10	10.8
2,4-DB 2-EH	0.01 (LOQ)	5	89-101	95	4	4.7
	0.1	5	90-97	94	3	2.8
MCPA	0.01 (LOQ)	5	91-104	98	5	5.6
	0.1	5	100-126	114	10	9.2
MCPA 2-EH	0.01 (LOQ)	5	102-128	114	11	9.8
	0.1	5	107-131	121	9	7.3
MCPB	0.01 (LOQ)	5	73-87	80	5	6.8
	0.1	5	99-105	101	2	2.5
MCPB 2-EH	0.01 (LOQ)	5	93-109	100	6	5.9
	0.1	5	105-108	107	1	1.0
Mecoprop-p	0.02 (LOQ)	5	98-111	104	5	4.7
	0.1	5	95-136	111	16	14.5
Mecoprop-p 2-EH	0.02 (LOQ)	5	75-94	88	8	9.0
	0.1	5	91-110	104	8	7.3

1 Data (corrected results, Appendix C, p. 124 of MRID 49775207) were obtained from Tables 1-6, pp. 31-36 of MRID 49775206. Reported values for standard deviation were reviewer-calculated because the study author did not provide these values (see DER Attachment 2).

2 The surface water was characterized; three surface water specimens were reported in the study: CCON/037/005 (pH 5.8, dissolved organic carbon 2.27 mg/L); CCON/037/007 (pH 6.6, dissolved organic carbon 2.66 mg/L); and CCON/037/008 (pH 7.3, dissolved organic carbon 5.94 mg/L; p. 23; Table 47, p. 72 of MRID 49775206). The study author did not specify if the water specimens were mixed for samples or used individually. The specific sources of the water specimens were not reported.

3 Two ion pair transitions were monitored for each analyte (quantitation and confirmation, respectively): *m/z* 232.9→160.8 and *m/z* 234.9→162.8 for 2,4-DP-p and 2,4-DP-p 2-EH, *m/z* 218.8→161.0 and *m/z* 220.8→162.9 for 2,4-D and 2,4-D 2-EH, *m/z* 247.0→161.0 and *m/z* 249.0→163.0 for 2,4-DB and 2,4-DB 2-EH, *m/z* 199.0→140.9 and *m/z* 200.9→142.9 for MCPA and MCPA 2-EH, *m/z* 227.0→140.9 and *m/z* 229.0→142.9 for MCPB and MCPB 2-EH, and *m/z* 212.9→140.9 and *m/z* 215.0→142.9 for Mecoprop-p and Mecoprop-p 2-EH.

Table 3. Independent Validation Method Recoveries for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, MCPB, Mecoprop-p and their 2-EHs in Water[1]

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Drinking Water[2]						
	Quantification ion[3]					
2,4-DP-p	0.01 (LOQ)	5	78-89	84	5	5.5
	0.1	5	86-93	90	3	3.5
2,4-DP-p 2-EH	0.01 (LOQ)	5	80-93	86	5	5.9
	0.1	5	92-109	97	7	7.0
2,4-D	0.01 (LOQ)	5	81-99	90	7	7.9
	0.1	5	92-97	94	2	2.5
2,4-D 2-EH	0.01 (LOQ)	5	93-104	99	4	4.0
	0.1	5	98-120	106	8	8.0
2,4-DB	0.01 (LOQ)	5	73-88	81	6	7.2
	0.1	5	84-93	87	3	3.8
2,4-DB 2-EH	0.01 (LOQ)	5	80-96	87	6	7.1
	0.1	5	88-108	93	8	8.9
MCPA	0.01 (LOQ)	5	82-95	89	5	5.7
	0.1	5	88-96	93	4	3.8
MCPA 2-EH	0.01 (LOQ)	5	86-104	95	7	7.7
	0.1	5	96-116	103	8	7.6
MCPB	0.01 (LOQ)	5	84-92	87	4	4.1
	0.1	5	94-99	96	2	2.1
MCPB 2-EH	0.01 (LOQ)	5	89-105	99	7	6.9
	0.1	5	103-128	111	10	8.8
Mecoprop-p	0.02 (LOQ)	5	79-89	85	5	5.7
	0.1	5	87-94	91	3	3.1
Mecoprop-p 2-EH	0.02 (LOQ)	5	88-106	96	7	7.0
	0.1	5	96-116	102	8	8.0
	Confirmation ion[3]					
2,4-DP-p	0.01 (LOQ)	5	80-92	84	5	6.0
	0.1	5	85-92	89	3	3.0
2,4-DP-p 2-EH	0.01 (LOQ)	5	76-91	84	5	6.4
	0.1	5	90-109	96	8	8.0
2,4-D	0.01 (LOQ)	5	83-95	90	6	6.1
	0.1	5	90-95	93	2	2.5
2,4-D 2-EH	0.01 (LOQ)	5	92-107	99	6	5.6
	0.1	5	99-120	105	8	8.0
2,4-DB	0.01 (LOQ)	5	74-86	80	5	6.8
	0.1	5	85-90	87	2	2.4
2,4-DB 2-EH	0.01 (LOQ)	5	72-97	81	9	11.6
	0.1	5	84-108	92	9	10.1
MCPA	0.01 (LOQ)	5	84-101	93	7	7.0
	0.1	5	85-92	89	3	3.6
MCPA 2-EH	0.01 (LOQ)	5	91-107	97	7	6.8
	0.1	5	95-109	99	6	5.8

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
MCPB	0.01 (LOQ)	5	79-94	85	6	6.6
	0.1	5	95-100	97	2	1.9
MCPB 2-EH	0.01 (LOQ)	5	85-102	97	7	7.1
	0.1	5	103-128	111	10	8.7
Mecoprop-p	0.02 (LOQ)	5	80-90	84	5	5.8
	0.1	5	84-92	89	3	3.6
Mecoprop-p 2-EH	0.02 (LOQ)	5	83-102	93	7	7.4
	0.1	5	92-110	97	7	7.6

1 Data (corrected results, p. 22) were obtained from Tables 1-6, pp. 27-32 of MRID 49775207. Reported values for standard deviation were reviewer-calculated because the study author did not provide these values (see DER Attachment 2).

2 The drinking water was bottled, still mineral water purchased from a local supermarket; no characterization data was reported (pp. 11, 20 of MRID 49775207).

3 Two ion pair transitions were monitored for each analyte (quantitation and confirmation, respectively): *m/z* 232.9→160.8 and *m/z* 234.9→162.8 for 2,4-DP-p and 2,4-DP-p 2-EH, *m/z* 218.8→161.0 and *m/z* 220.8→162.9 for 2,4-D and 2,4-D 2-EH, *m/z* 247.0→161.0 and *m/z* 249.0→163.0 for 2,4-DB and 2,4-DB 2-EH, *m/z* 199.0→140.9 and *m/z* 200.9→142.9 for MCPA and MCPA 2-EH, *m/z* 227.0→140.9 and *m/z* 229.0→142.9 for MCPB and MCPB 2-EH, and *m/z* 212.9→140.9 and *m/z* 215.0→142.9 for Mecoprop-p and Mecoprop-p 2-EH.

III. Method Characteristics

In the ECM and ILV, the LOQ was 0.01 µg/L for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, and MCPB, and 0.02 µg/L for Mecoprop-p (pp. 21, 27 of MRID 497758206; p. 11; Appendix C, pp. 112, 127 of MRID 49775207). The LOQ was defined in the ECM as the lowest fortification level where an acceptable mean recovery is obtained (70-120%). No comparison to baseline noise, other justification or calculation was provided. The ECM study author reported that the LOD was calculated as the estimated "baseline noise in the control sample multiplied by 3 and then "compared to the intensity of the 3 ng/mL standard for surface water" (p. 27 of MRID 497758206). The calculated LODs were 0.000481-0.001340 µg/L for 2,4-DP-p, 0.000143-0.000528 µg/L for 2,4-D, 0.001006-0.001062 µg/L for 2,4-DB, 0.000195-0.001623 µg/L for MCPA, 0.000129-0.000621 µg/L for MCPB, and 0.000385-0.001420 µg/L for Mecoprop-p in the ECM (Table 37, p. 64). In CEMAS CAM-0004/003, which was the ECM provided in Appendix C of the ILV, the LOD was calculated using the following equation:

$$LOD = C_{standard} \times [(3 \times Noise)/h_{peak}]$$

Where $C_{standard}$ is the concentration of the lowest standard, Noise is the estimate of the background noise at the retention time of the peak of interest, and h_{peak} is the peak height (Appendix C, p. 127 of MRID 49775207; see Reviewer's Comment #4). The LOD was reported in the ILV as equivalent to the 0.6 ng/mL standard, which equated to 30% of the LOQ for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, and MCPB, and 15% of the LOQ for Mecoprop-p (pp. 13-14).

Table 4. Method Characteristics

		2,4-DP-p	2,4-D	2,4-DB	MCPA	MCPB	Mecoprop-p
Limit of Quantitation (LOQ)		0.01 µg/L					0.02 µg/L
Limit of Detection (LOD)	ECM (calculated)	0.000481 µg/L (Q) 0.001340 µg/L (C)	0.000143 µg/L (Q) 0.000528 µg/L (C)	0.001062 µg/L (Q) 0.001006 µg/L (C)	0.000195 µg/L (Q) 0.001623 µg/L (C)	0.000129 µg/L (Q) 0.000621 µg/L (C)	0.000385 µg/L (Q) 0.001420 µg/L (C)
	ILV	30% of the LOQ (equivalent to 0.6 ng/mL standard)					15% of the LOQ (equivalent to 0.6 ng/mL standard)
Linearity (calibration curve r^2 and concentration range)	ECM	r^2 = 0.9965 (Q) r^2 = 0.9989 (C)	r^2 = 0.9979 (Q) r^2 = 0.9997 (C)	r^2 = 0.9995 (Q) r^2 = 0.9980 (C)	r^2 = 0.9998 (Q) r^2 = 0.9999 (C)	r^2 = 0.9989 (Q) r^2 = 0.9997 (C)	r^2 = 0.9999 (Q) r^2 = 0.9996 (C)
	ILV[1]	r^2 = 0.9992 (Q) r^2 = 0.9998 (C)	r^2 = 0.9996 (Q) r^2 = 0.9994 (C)	r^2 = 0.9994 (Q) r^2 = 0.9990 (C)	r^2 = 0.9992 (Q) r^2 = 0.9996 (C)	r^2 = 0.9984 (Q) r^2 = 0.9988 (C)	r^2 = 0.9984 (Q) r^2 = 0.9994 (C)
	Concentration range	0.6-200 ng/mL					
Repeatable	ECM[2]	Yes at the LOQ and 10×LOQ.			Yes at LOQ; Yes at 10×LOQ (Q), No at 10×LOQ (C).[3]	Yes at the LOQ and 10×LOQ.	Yes at the LOQ and 5×LOQ; no samples were prepared at 10×LOQ.
	ILV[4]	Yes at the LOQ and 10×LOQ.					
Reproducible		Yes at the LOQ and 10×LOQ.					Yes at the LOQ and 5×LOQ; no samples were prepared at 10×LOQ.
Specific[5]	ECM	Yes, matrix interferences were <20% of the LOQ.		Yes, matrix interferences were <20% of the LOQ (Q); matrix interferences were *ca*. 21-22% of the LOQ (C).	Yes, matrix interferences were <20% of the LOQ (acid) and <10% of the LOQ (2-EH).	No, matrix interferences were ≤20% of the LOQ (2-EH), but *ca*. 33-35% of the LOQ (acid).[6]	Yes, matrix interferences were <15% of the LOQ (acid) and <10% of the LOQ (2-EH).
	ILV[7]	Yes, no matrix interferences were observed.	Yes, matrix interferences were <5% of the LOQ.	Yes, no matrix interferences were observed.	Yes, matrix interferences were <5% of the LOQ.	Yes, no matrix interferences were observed; however, baseline noise at the LOQ was notable.	Yes, no matrix interferences were observed.

Data were obtained from pp. 21, 27; Tables 1-6, pp. 31-36; Table 37, p. 64 (calculated LODs); Figures 1-12, pp. 73-84 (calibration curves); Figures 49-90, pp. 121-162 (acid and 2-EH chromatograms) of MRID 49775206; p. 11; Tables 1-6, pp. 27-32; Figures 1-12, pp. 34-45 (calibration curves); Figures 13-60, pp. 46-93 (acid chromatograms); Appendix C, pp. 112, 127 of MRID 49775207; DER Attachment 2. Q = quantification ion; C = confirmation ion. Acid = 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, MCPB and Mecoprop-p; 2-EH = 2,4-DP-p 2-EH, 2,4-D 2-EH, 2,4-DB 2-EH, MCPA 2-EH, MCPB 2-EH and Mecoprop-p 2-EH.

1 Reviewer-calculated r^2 values from the reported r values of 0.9992-0.9999 (combined analytes and ions; Figures 1-12, pp. 34-45 of MRID 49775207; see DER Attachment 2).

2 In the ECM, three surface water specimens were reported: CCON/037/005 (pH 5.8, dissolved organic carbon 2.27 mg/L); CCON/037/007 (pH 6.6, dissolved organic carbon 2.66 mg/L); and CCON/037/008 (pH 7.3, dissolved organic carbon 5.94 mg/L; p. 23; Table 47, p. 72 of MRID 49775206). The study author did not specify if the water specimens were mixed for samples or used individually. The specific sources of the water specimens were not reported.

3 Mean recovery was 121% for the confirmation ion at 10×LOQ. A confirmatory method is not usually required when LC/MS and GC/MS is the primary method.

4 Bottled, still mineral water, purchased from a local supermarket, was used in the ILV; no characterization data was reported (pp. 11, 20 of MRID 49775207).

5 Results refer to the acid and 2-EH analyte, as well as quantification and confirmation ion, chromatographic data unless specified otherwise. The percent of the LOQ for the matrix interference was reviewer-determined based on the peak areas provided in the representative chromatograms.

6 Based on Figures 71-72, pp. 143-144 of MRID 49775206. The ECM study author reported that "no residues..greater than 30% of the LOQ were observed in any of the control samples" (p. 29 of MRID 49775206).

7 Chromatograms were only noted as those of the acid analytes; no chromatograms were noted as those of the 2-EH analytes.

IV. Method Deficiencies and Reviewer's Comments

1. Any communication between the ILV and ECM staff was not documented, summarized, or discussed. This affects the study classification.

2. Method for ECM MRID 49775206 (CEMAS Study No. CEMS-6230; dated August 5, 2014; p. 1 of MRID 49775206) was contained in Appendix C of ILV MRID 49775207 (CEMAS CAM-0004/003; dated August 11, 2016; Appendix C, p. 111 of MRID 49775207). The only details of the method contained in the ECM MRID 49775206 was the "Principle of the method" (p. 24 of MRID 49775206) which was the reproduction of the fourth paragraph of *Section 1.2 Summary* of CEMAS CAM-0004/003 (Appendix C, p. 112 of MRID 49775207). The ECM MRID 49775206 did not contain any specific information or details about the extraction procedure or analytical method, except the ion transitions for the analytes which was reported in the recovery data tables. ECM MRID 49775206 did contain procedural recovery results and chromatograms, whereas CEMAS CAM-0004/003 did not contain any individual procedural recovery results and chromatograms. The reviewer noted that the ILV-provided CEMAS CAM-0004/003 was an excerpt and not the full report since it contained a detailed results summary and data table references in that summary. Additionally, CEMAS CAM-0004/003 and ECM MRID 49775206 cross-referenced each other (Ref. 5, p. 30 of MRID 49775206; Ref. 5, Appendix C, p. 129 of MRID 49775207). Therefore, the reviewer reported the ECM information for the DER using CEMAS CAM-0004/003 and ECM MRID 49775206 and considered ECM MRID 49775206 as the sub-report of CEMAS CAM-0004/003. ECM MRID 49775206 should be updated with the method details of CEMAS CAM-0004/003 in order to be a complete ECM.

The ILV only cited CEMAS CAM-0004/003 as the ECM; MRID 49775206 was not referenced in the ILV. No modifications of CEMAS CAM-0004/003 were reported by ILV; however, the analytical instrument, equipment and parameters which were used by the ILV were not reported (p. 20 of MRID 49775207). ILV MRID 49775207 should be updated with the analytical method details which were used by the ILV laboratory. Incomplete documentation affects the study classification.

3. Procedural recoveries were corrected for residues found in the controls in the ECM and ILV (p. 22; Appendix C, p. 124 of MRID 49775207). In the ECM, residues were quantified in the controls for all analytes (<10% to *ca*. 33% of the LOQ; Figures 49-90, pp. 121-162 of MRID 49775206). In the ILV, residues were only quantified in the controls for 2,4-D and MCPA (*ca*. 3% of the LOQ; chromatograms for the 2-EH analytes were not provided; p. 22; Figures 13-60, pp. 46-93 of MRID 49775207). While recoveries should not be corrected, the corrections were negligible. Therefore, this does not impact the study classification.

4. In the ILV, chromatograms were only noted as those of the acid analytes; no chromatograms were noted as those of the 2-EH analytes. Also, chromatograms of reagent blank and all calibration standards, except 3 ng/mL, were not provided. Incomplete documentation affects the study classification.

5. In the ECM, matrix interferences were <30% of the LOQ in MCPB chromatograms (Figures 71-72, pp. 143-144 of MRID 49775206). Residues in the controls were 8910 and 3225 counts for the quantification and confirmation ions, respectively; residues at the LOQ were 27128 and 9094 counts for the quantification and confirmation ions, respectively. Chromatograms of reagent blank and all calibration standards, except 3 ng/mL, were not provided.

6. No fortifications of Mecoprop-p and Mecoprop-p 2-EH were performed at 10×LOQ, only 5×LOQ. OCSPP guidelines recommend that minimum of five spiked replicates were analyzed at each concentration (*i.e.*, minimally, the LOQ and 10×LOQ) for each analyte.

7. It could not be determined if the ILV was provided with the most difficult matrix with which to validate the method. Bottled, still mineral water, purchased from a local supermarket, was used in the ILV; no characterization data was reported (pp. 11, 20; Appendix B, Study Plan Amendment No. 2, pp. 106-108 of MRID 49775207). Also, the reviewer noted that, when tap water was used as the control matrix in the first trial, residues of some analytes were detected above 30% of the LOQ in the control samples and the trial failed.

8. The estimations of the LOQ and LOD in the ECM were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (pp. 21, 27; Table 37, p. 64 of MRID 497758206; p. 11; Appendix C, pp. 112, 127 of MRID 49775207). No calculations were reported for the LOQ; no comparison was made to chromatogram background levels. Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples. For the LOD, the explanation of the LOD calculation in the ECM was vague, i.e. the estimated "baseline noise in the control sample multiplied by 3 and then "compared to the intensity of the 3 ng/mL standard for surface water" (p. 27 of MRID 497758206). An exact equation was provided in CEMAS CAM-0004/003 {$LOD = C_{standard} \times [(3 \times Noise)/h_{peak}]$}; however, the reviewer could not determine to what $C_{standard}$ (the concentration of the lowest standard) was equivalent (Appendix C, p. 127 of MRID 49775207). Based on that equation in MRID 49775207 and Table 37 of MRID 49775206, $C_{standard}$ should equal 3 ng/mL; however, based on the LOD results reported in Table 37 of MRID 49775206, $C_{standard}$ equaled 0.015 ng/mL (Table 37, p. 64 of MRID 49755206; Appendix C, p. 127 of MRID 49775207). Also, 3 ng/mL was not the lowest standard; 0.6 ng/mL was the lowest standard (Figure 1, p. 73 of MRID 49775206). Furthermore, the reviewer noted that the values provided in Table 37 for baseline noise and 3 ng/mL peak height were not compatible with the values in the provided chromatograms (see Table 37, p. 64 and Figures 49-50, pp. 121-122 of MRID 49775206). In the ILV, the LOD was reported as equivalent to the 0.6 ng/mL standard, which equated to 30% of the LOQ for 2,4-DP-p, 2,4-D, 2,4-DB, MCPA, and MCPB, and 15% of the LOQ for Mecoprop-p (pp. 13-14 of MRID 49775207).

9. In the ECM, method recoveries did not meet guideline criteria for precision and accuracy (mean 70-120%; RSD ≤20%) for the confirmation ion of MCPA 2-EH at 10×LOQ (121%; Tables 1-6, pp. 31-36 of MRID 49775206). The reviewer noted that a

confirmatory method is not usually required when LC/MS and GC/MS is the primary method.

10. In the Special Requirements of the Sponsor Study Plan for the ILV, the sponsor noted that the 2-EH analytes were included and were to be analyzed as the equivalent acid analytes in order to demonstrate that the alkaline hydrolysis step of the sample processing procedure was valid (Appendix B, p. 98 of MRID 49775207).

11. Matrix interferences were studied in the ECM and ILV (pp. 25, 27; Table 40, pp. 67-68 of MRID 49775206; Table 7, p. 33 of MRID 49775207). No significant suppression or enhancement of detector response was observed for any analyte in the water matrix in either study.

12. Stability of the extracts and standards was studied in the ECM (pp. 25, 27; Tables 31-36, pp. 61-63; Tables 41-46, pp. 69-71 of MRID 49775206). Extracts were determined to be stable for at least 7 days when stored at 2-8°C. Standards were determined to be stable for up to 149 days when stored at 2-8°C.

13. No time requirement for the method was reported in the ECM or ILV, other than the fact that the hydrolysis step was performed overnight (p. 20; Appendix C, p. 120 of MRID 49775207).

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

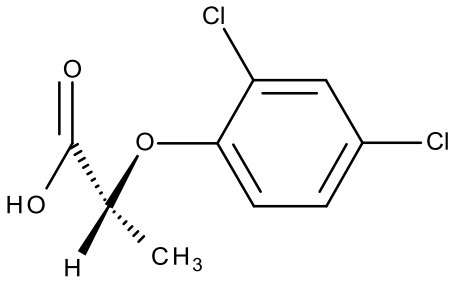
2,4-DP-p; Dichloroprop-p; Dichlorprop-p; 2,4-Dichlorprop-p

IUPAC Name: (2R)-2-(2,4-dichlorophenoxy)propanoic acid
(+)-(R)-2-(2,4-dichlorophenoxy)propionic acid
(R+)-2-(2,4-dichlorophenoxy)propionic acid

CAS Name: Not reported

CAS Number: 15165-67-0

SMILES String: O=C(O)C(Oc(c(cc(c1)Cl)Cl)c1)C



2,4-DP-p 2-EH

IUPAC Name: (R+)-2-(2,4-dichlorophenoxy)propionic acid 2-ethylhexyl ester

CAS Name: Not reported

CAS Number: 865363-39-9

SMILES String: Not found

No Structure Provided

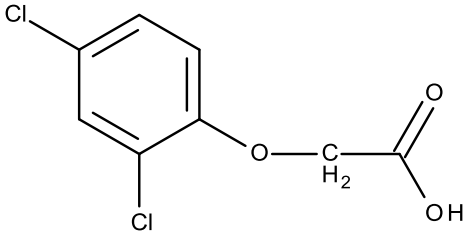
2,4-D

IUPAC Name: 2,4-Dichlorophenoxyacetic acid
(2,4-Dichlorophenoxy)acetic acid

CAS Name: 2-(2,4-Dichlorophenoxy)acetic acid

CAS Number: 94-75-7

SMILES String: O=C(O)COc(c(cc(c1)Cl)Cl)c1



2,4-D 2-EH

IUPAC Name: 2,4-Dichlorophenoxyacetic acid, 2-ethylhexyl ester
CAS Name: Not reported
CAS Number: 1928-43-4
SMILES String: Not found

No Structure Provided

2,4-DB

IUPAC Name: 4-(2,4-Dichlorophenoxy)butyric acid
CAS Name: Not reported
CAS Number: 94-82-6
SMILES String: Not found

No Structure Provided

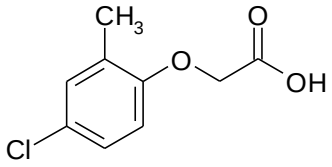
2,4-DB 2-EH

IUPAC Name: 4-(2,4-Dichlorophenoxy)butyric acid, 2-ethylhexyl ester
CAS Name: Not reported
CAS Number: 7720-36-7
SMILES String: Not found

No Structure Provided

MCPA

IUPAC Name: 4-Chloro-2-methylphenoxyacetic acid
CAS Name: Not reported
CAS Number: 94-74-6
SMILES String: Not found



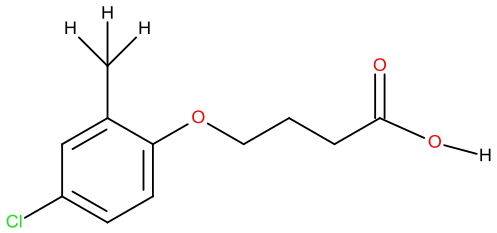
MCPA 2-EH

IUPAC Name: 4-Chloro-2-methylphenoxyacetic acid, 2-ethylhexyl ester
CAS Name: Not reported
CAS Number: 29450-45-1
SMILES String: Not found

No Structure Provided

MCPB

IUPAC Name: 4-(4-Chloro-2-methylphenoxy)butyric acid
CAS Name: Not reported
CAS Number: 94-81-5
SMILES String: C1C(Cl)=CC=C(OCCCC(=O)OH)C=1C(H)(H)H (EPISuite 4.0).



MCPB 2-EH

IUPAC Name: 4-(4-Chloro-2-methylphenoxy)butyric acid, 2-ethylhexyl ester
CAS Name: Not reported
CAS Number: 94232-74-3
SMILES String: Not found

No Structure Provided

Mecoprop-p (CMPP-p)

IUPAC Name: (R+)- 2-(4-chloro-2-methylphenoxy)propionic acid
CAS Name: Not reported
CAS Number: 16484-77-8
SMILES String: Not found

No Structure Provided

Mecoprop-p 2-EH

IUPAC Name: (R+)-2-(4-chloro-2-methylphenoxy)propionic acid, 2-ethylhexyl ester
CAS Name: Not reported
CAS Number: 861229-15-4
SMILES String: Not found

No Structure Provided

Test Material:	2,4-DP-p, 2,4-D, 2,4-DB, MCPA, MCPB, Mecoprop-p and their 2-EHs
MRID:	49775206
Title:	VALIDATION OF DRAFT RESIDUE METHOD CAM-0004/003 FOR THE DETERMINATION OF PHENOXY ACIDS AND THEIR CORRESPONDING 2 ETHYL-HEXYL ESTERS IN SURFACE WATER, SOIL AND AIR
MRID:	49775207
Title:	Phenoxy Herbicides – Independent Laboratory Validation of the Analytical Method CAM-0004/003 for the Determination of Phenoxy Acids and Their Corresponding 2 Ethyl-Hexyl Esters in Drinking Water by LC-MS/MS – Final Report on Study S14-01199
EPA PC Codes:	019201 (MCPB); 030001 (2,4-D) ; 030501 (MCPA) ; 030801 (2,4-DB) ; 031402 (2,4-DP-p) ; 129046 (Mecoprop-p)
OCSPP Guideline:	850.6100

For CDM Smith

Primary Reviewer: Lisa Muto Signature: Lisa Muto

Date: 5/14/16

Secondary Reviewer: Signature:

Date: 5/14/16

QC/QA Manager: Joan Gaidos Signature:

Date: 5/14/16