Data Evaluation Record (DER) Addendum for MRID 49193108 (ECM) + 49193105 (ILV)

Citation:

MRID 49193108. Huang, S.-B. 2012. Fluazifop-P-Butyl: Fluazifop-P-Butyl – Residue Method for the Determination of Fluazifop-P-Butyl (R154875; PP5), Fluazifop-P-Acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in Water: Analytical Method. Report No.: GRM044.04A. Task No.: TK0024911. Report prepared, sponsored and submitted by Syngenta Crop Protection, LLC., Greensboro, North Carolina; 84 pages. Final report issued March 26, 2012.

MRID 49193105. Perez, R., J. L. Schmitt. 2013. Fluazifop-P-Butyl: Independent Laboratory Validation of Residue Method (GRM044.04A) for the Determination of Fluazifop-P-Butyl (R154875; PP5), Fluazifop-P-Acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in Water: Final Report. Report and Task No.: TK0015287. Report prepared by ADPEN Laboratories, Inc., Jacksonville, Florida, sponsored and submitted by Syngenta Crop Protection, LLC., Greensboro, North Carolina; 223 pages. Final report issued February 6, 2013.

MRID 49700401. Perez, R., J. L. Schmitt. 2013. Fluazifop-P-Butyl: Independent Laboratory Validation of Residue Method (GRM044.04A) for the Determination of Fluazifop-P-Butyl (R154875; PP5), Fluazifop-P-Acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in Water: Amended Final Report. Report and Task No.: TK0015287-02. Report prepared by ADPEN Laboratories, Inc., Jacksonville, Florida, sponsored and submitted by Syngenta Crop Protection, LLC., Greensboro, North Carolina; 223 pages. Final report issued February 6, 2013. Amended Final report issued August 5, 2015.

MRIDs: 49193108, 49193105, 49700401

Guideline: OCSPP 850.6100

Chemical (PC): Fluazifop-p-butyl, PC 122809

Date of review: 06-27-2019

RICHARD SHAMBLEN Digitally signed by RICHARD SHAMBLEN Date: 2019.07.01 15:53:13 -04'00'

Primary Reviewer: Richard Shamblen, Biologist
Environmental Fate and Effects Division, ERB2

STEPHEN WENTE Digitally signed by STEPHEN WENTE Date: 2019.07.02 07:38:24 -04'00'

Secondary Reviewer: Stephen Wente, Ph.D., Senior Scientist
Environmental Fate and Effects Division, ERB2

Purpose of Addendum: Provides additional information on residues in matrix blanks.

Summary of Study Findings: This study was revised with recovery data not corrected for residues in the matric blank as per OCSPP 850.6100, Section (5)(i)(B)(1)(a).

Study Limitations: With the revised recovery data not corrected for residues in the matrix blank, the study has no major limitations.

Study Classification: With the additional information provided in MRID 49700401, MRID 49193108 (ECM) / 49193105 (ILV) the classification is upgraded from unacceptable to **acceptable**.

Analytical method for fluazifop-P-butyl, fluazifop-P-acid, Compound IV (CGA181847) and Compound X (CGA142110) in water

Reports: ECM: MRID 49193108. Huang, S.-B. 2012. Fluazifop-P-Butyl: Fluazifop-P-Butyl – Residue Method for the Determination of Fluazifop-P-Butyl (R154875; PP5), Fluazifop-P-Acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in Water: Analytical Method. Report No.: GRM044.04A. Task No.: TK0024911. Report prepared, sponsored and submitted by Syngenta Crop Protection, LLC., Greensboro, North Carolina; 84 pages. Final report issued March 26, 2012.
ILV: MRID 49193105. Perez, R., J. L. Schmitt. 2013. Fluazifop-P-Butyl: Independent Laboratory Validation of Residue Method (GRM044.04A) for the Determination of Fluazifop-P-Butyl (R154875; PP5), Fluazifop-P-Acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in Water: Final Report. Report and Task No.: TK0015287. Report prepared by ADPEN Laboratories, Inc., Jacksonville, Florida, sponsored and submitted by Syngenta Crop Protection, LLC., Greensboro, North Carolina; 218 pages. Final report issued February 6, 2013.

Document No.: MRIDs 49193108 & 49193105

Guideline: 850.6100

Statements: ECM: The study was not conducted in compliance with USEPA FIFRA or OECD Good Laboratory Practice (GLP) standards (p. 3). Signed and dated No Data Confidentiality and GLP statements were provided (pp. 2-3). A certification of authenticity and Quality Assurance statement were not included. A signed authorization of revisions to previous method version was included (p. 4).
ILV: The study was conducted in accordance with the USEPA FIFRA GLP standards (p. 3 of MRID 49193105). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4 of MRID 49193105). An authenticity statement was included with the quality assurance statement.

Classification: This analytical method is classified as ***unacceptable***. The method may be valid. However, matrix blanks had residues up to 50% of the LOD. It was unclear whether the residues in the matrix blanks were due to contamination or to residual carryover in the chromatographic equipment. The independent laboratory used matrices supplied by the registrant rather than independently sourced matrices. Recoveries were corrected in the ECM and ILV when residues were detected. Representative chromatograms were not provided for three of the four water matrices in the ECM report.

PC Code: 122809

Reviewer:

Edmund M. Wong
Environmental Chemist

Signature: 
Date: 08/19/2014

All page citations refer to those in MRID 49193108 (ECM) unless noted otherwise

Executive Summary

This analytical method, Syngenta Crop Method GRM044.04A, is designed for the quantitative determination of fluazifop-P-butyl (R154875; PP5), fluazifop-P-acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in water using LC/MS/MS. The method appears to be quantitative for all four analytes at the stated LOQ of 0.10 µg/L (0.10 ppb). However, the method specifies the correction of procedural recoveries for residues in the controls. The LOQ is less than the lowest toxicological level of concern in water. No major modifications were made by the independent laboratory.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Fluazifop-P-Butyl, Fluazifop-P-Acid, Compound IV and Compound X	49193108	49193105		Water	03/26/2012	Syngenta Crop Protection, LLC	LC/MS/MS	0.10 µg/L

I. Principle of the Method

Water samples were stored chilled then warmed to ambient temperature prior to experiment (p. 17). If water was not clear, water was centrifuged or filtered prior to experimentation. Samples of water (10 mL) were mixed with 1.0 mL of stabilizer (2% acetic acid in acetonitrile; pp. 14, 17; Appendix 3, p. 84). An aliquot (1 mL) was directly injected for LC/MS/MS analysis.

Samples were analyzed for fluazifop-P-butyl, fluazifop-P-acid, Compound IV (CGA181847) and Compound X (CGA142110) by HPLC (Ascentis Express C8, 50 x 3.0 mm, 2.7 µm column) with a column shield (ColumnSaver or UltraShield) using a gradient mobile phase of (A) 0.1% formic acid in Optima LC/MS grade water and (B) 0.1% formic acid in HPLC grade methanol [time ratio A:B; 0.0-0.5 min. 90:10, 2.0-4.0 min. 40:60, 4.5-6.5 min. 10:90, 6.6-7.5 min. 90:10] with mass spectrometry in positive ion or negative ion mode (Multiple Reaction Monitoring mode, MRM; pp. 19-21). Analytes were identified with two transitions, quantification and confirmation ion transitions. Positive mode was employed for fluazifop-P-butyl with transitions of 384.15→328.00 and 384.14→282.00, Compound IV (CGA181847) with transitions of 256.05→93.00 and 256.06→164.00 and Compound X (CGA142110) with transitions of 164.05→146.00 and 164.06→75.00. Negative mode was employed for fluazifop-P-acid with transitions of 326.06→254.00 and 326.07→226.00. Injection volumes were 50 µL. In the ILV, only the quantitative transition was monitored, and injection volume was 10 µL (pp. 15-16 of MIRD 49193105).

The LOQ for fluazifop-P-butyl, fluazifop-P-acid, Compound IV (CGA181847) and Compound X (CGA142110) was reported as 0.10 µg/L in the ECM and the ILV (pp. 24-25 and Figure 16, p. 58; pp. 10-11 and Appendix 6, pp. 201-217 of MRID 49193105). The LOD for all analytes was 0.05 µg/L in the ECM and the ILV.

II. Recovery Findings

ECM (MRID 49193108): Mean recoveries and relative standard deviations (RSD) were within guideline requirements (mean 70-120%; RSD ≤20%) for analysis of fluazifop-P-butyl, fluazifop-P-acid, Compound IV (CGA181847) and Compound X (CGA142110) at the LOQ, 10×LOQ and 100×LOQ in de-ionized and finished water from Syngenta Laboratory, ground water from a Residency Well in Summerfield, North Carolina and surface water from Lake Higgins in Greensboro, North Carolina (Tables 3-6, pp. 32-35). Confirmation ion results were comparable (Tables 8-11, pp. 37-40). All of the procedural recovery values were corrected for the average of the residues found in the controls (based on protocol and data in chromatograms of surface water; no other chromatograms or raw data were provided; p. 22; Figures 5-28, pp. 47-70). Waters were fully characterized (Tables 1A-1B, p. 29).

ILV (MRID 49263805): Mean recoveries and RSDs were within guideline requirements for analysis of fluazifop-P-butyl, fluazifop-P-acid, Compound IV (CGA181847) and Compound X (CGA142110) at the LOQ and 10×LOQ in ground water and surface water supplied by Syngenta Laboratory (sources not specifically reported; pp. 10-11, 14, 17; Tables 3-10, pp. 23-30; Appendix 4, pp. 197-199 of MRID 49193105). Recovery values were corrected for the average of the residues found in the controls; the only recovery values which were not corrected were those of fluazifop-P-acid and Compound IV in surface water due to absence of residues in the controls. The waters were fully characterized in Appendix 4; the sample identification numbers differed from those reported in the report text, most likely due to a typographical error. Only the quantitative ion was monitored. The method was validated with the first trial (p. 10 of MRID 49193105).

Table 2. Initial Validation Method Recoveries for Analytes in Water

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[1]	Relative Standard Deviation (%)
De-ionized water (DI water; RIMV00710-0001) from Syngenta, Greensboro, North Carolina						
Quantitative ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	88.7-103	98.4	5.6	5.7
	1.0	5	95.7-98.5	96.9	1.4	1.4
	10	5	89.4-92.2	90.7	1.2	1.3
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	76.3-94.8	89.6	7.7	8.5
	1.0	5	94.3-96.1	95.0	0.7	0.8
	10	5	88.9-91.5	90.4	1.0	1.1
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	86.9-96.6	90.6	4.7	5.2
	1.0	5	97.4-101	99.5	1.4	1.4
	10	5	94.3-97.0	95.9	1.0	1.1
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	80.8-91.0	84.2	4.8	5.7
	1.0	5	89.9-93.4	91.7	1.3	1.4
	10	5	87.1-91.6	88.9	1.7	2.0
Confirmation ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	89.9-101	94.6	4.3	4.6
	1.0	5	96.9-101	98.3	1.6	1.6
	10	5	89.0-91.2	90.5	0.9	1.0
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	76.3-98.3	88.5	9.0	10
	1.0	5	91.9-99.0	94.9	2.7	2.8
	10	5	88.2-91.6	90.0	1.2	1.4
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	86.2-108	97.1	7.9	8.1
	1.0	5	96.5-105	100	3.3	3.3
	10	5	94.6-98.7	96.9	1.5	1.6
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	85.1-97.7	92.3	5.0	5.5
	1.0	5	97.1-106	101	3.2	3.2
	10	5	96.7-101	98.3	1.6	1.7
Finished water (RIMV00710-0004) from Syngenta, Greensboro, North Carolina						
Quantitative ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	85.0-96.9	91.3	5.1	5.6
	1.0	5	88.3-92.9	91.2	1.8	2.0
	10	5	86.1-91.6	89.3	2.0	2.2
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	89.8-90.4	90.3	0.3	0.3
	1.0	5	93.6-97.0	94.9	1.3	1.4
	10	5	90.8-93.0	92.0	0.8	0.9
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	90.2-106	95.2	6.3	6.6
	1.0	5	92.4-98.0	94.2	2.2	2.3
	10	5	91.8-94.0	93.0	1.0	1.0
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	82.2-98.2	87.8	6.5	7.4
	1.0	5	86.3-88.8	87.7	0.9	1.1
	10	5	85.6-88.2	87.3	1.0	1.2
Confirmation ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	88.2-97.7	92.6	3.7	3.9
	1.0	5	90.6-93.1	92.0	1.1	1.2
	10	5	86.9-91.3	89.8	1.8	2.0

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[1]	Relative Standard Deviation (%)
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	82.9-107	95.4	9.6	10
	1.0	5	93.1-100	95.7	2.7	2.8
	10	5	89.7-92.2	91.2	1.0	1.1
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	81.6-93.9	88.5	4.7	5.3
	1.0	5	93.7-99.1	96.3	2.4	2.5
	10	5	92.1-93.8	92.9	0.7	0.8
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	79.9-100	92.9	8.3	8.9
	1.0	5	96.9-101	98.9	1.5	1.5
	10	5	95.8-98.2	96.9	0.9	0.9
Ground water (RIMV00710-0002) from Residency Well in Summerfield, North Carolina						
Quantitative ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	84.5-101	94.7	6.2	6.6
	1.0	5	91.2-97.3	95.5	2.5	2.6
	10	5	86.9-93.6	90.7	2.5	2.8
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	86.2-92.6	89.3	2.3	2.6
	1.0	5	93.1-95.9	94.7	1.3	1.4
	10	5	93.0-94.3	93.6	0.6	0.6
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	94.2-103	97.6	3.7	3.7
	1.0	5	95.1-97.6	96.7	1.2	1.3
	10	5	94.8-97.8	96.2	1.1	1.2
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	81.2-92.2	89.1	4.9	5.5
	1.0	5	89.1-92.5	91.0	1.3	1.4
	10	5	88.3-91.3	89.4	1.1	1.3
Confirmation ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	92.6-101	95.7	3.2	3.4
	1.0	5	92.3-99.6	97.4	2.9	3.0
	10	5	87.3-94.4	91.0	2.8	3.1
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	79.0-103	90.2	10.1	11
	1.0	5	91.6-97.4	94.0	2.4	2.5
	10	5	91.2-93.1	92.3	0.7	0.8
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	91.1-119	104	10.8	10.4
	1.0	5	96.2-100	97.7	1.6	1.6
	10	5	95.4-97.7	97.0	0.9	1.0
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	97.9-106	101	3.3	3.3
	1.0	5	97.6-103	100	2.0	2.0
	10	5	95.1-98.8	97.3	1.5	1.6
Surface water (RIMV00710-0003) from Lake Higgins in Greensboro, North Carolina						
Quantitative ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	92.2-99.7	95.9	3.0	3.1
	1.0	5	93.1-102	98.9	3.5	3.5
	10	5	93.0-96.0	94.8	1.2	1.3
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	88.2-92.6	90.2	1.7	1.9
	1.0	5	91.4-95.2	93.7	1.5	1.6
	10	5	86.7-91.0	89.5	1.6	1.8
Compound IV (R150397;	0.10 (LOQ)	5	94.4-104	96.8	4.1	4.2
	1.0	5	97.7-101	99.2	1.3	1.3

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[1]	Relative Standard Deviation (%)
CGA181847)	10	5	94.2-96.1	95.4	0.8	0.8
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	78.3-99.9	89.2	7.8	8.7
	1.0	5	94.3-96.2	95.2	0.7	0.7
	10	5	90.7-92.2	91.6	0.7	0.8
Confirmation ion						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	92.2-101	95.7	4.2	4.3
	1.0	5	94.9-101	98.7	2.3	2.3
	10	5	93.5-95.8	94.6	1.0	1.1
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	78.1-86.9	83.8	3.8	4.5
	1.0	5	90.6-94.0	92.1	1.4	1.5
	10	5	86.0-88.8	87.7	1.1	1.2
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	88.7-108	96.5	8.1	8.4
	1.0	5	92.9-102	98.2	3.7	3.7
	10	5	94.1-96.6	95.4	1.1	1.1
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	74.8-95.9	87.4	9.0	10.3
	1.0	5	92.2-97.7	94.7	3.0	3.2
	10	5	88.8-91.2	90.3	0.9	1.0

Data were obtained from Tables 3-6, pp. 32-35 and Tables 8-11, pp. 37-40 in the study report. All recovery values were corrected for the average of the residues found in the controls (based on protocol and data in chromatograms of surface water; no other chromatograms or raw data were provided; p. 22; Figures 5-28, pp. 47-70).

Table 3. Independent Validation Method Recoveries for Analytes in Water

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Surface water (RIMV00512-0001) from Residency Well in Summerfield, North Carolina						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	68-75	71	2.7	3.8
	1.0	5	75-80	77	2.1	2.7
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	84-93	89	3.4	3.8
	1.0	5	95-101	97	3.1	3.2
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	103-107	105	1.8	1.8
	1.0	5	96-100	99	1.8	1.8
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	91-100	96	3.7	3.9
	1.0	5	93-97	95	1.6	1.7
Ground water (RIMV00512-0002) from Lake Higgins in Greensboro, North Carolina						
Fluazifop-P-Butyl (R154875; PP5)	0.10 (LOQ)	5	67-80	73	5.2	7.2
	1.0	5	80-94	87	5.3	6.1
Fluazifop-P-Acid (R156172)	0.10 (LOQ)	5	83-97	89	5.5	6.2
	1.0	5	93-107	100	4.9	4.9
Compound IV (R150397; CGA181847)	0.10 (LOQ)	5	95-111	102	5.9	5.7
	1.0	5	102-112	107	3.8	3.5
Compound X (R154719; CGA142110)	0.10 (LOQ)	5	84-101	89	7.1	7.9
	1.0	5	93-107	101	5.1	5.1

Data were obtained from pp. 10-11, 14, 17; Tables 3-10, pp. 23-30 of MRID 49193105. Recovery values were corrected for the average of the residues found in the controls; the only recovery values which were not corrected were those of Fluazifop-P-acid and Compound IV in surface water due to absence of residues in the controls.

III. Method Characteristics

The LOQ for fluazifop-P-butyl, fluazifop-P-acid, Compound IV (CGA181847) and Compound X (CGA142110) was reported as 0.10 µg/L in the ECM and the ILV (pp. 24-25 and Figure 16, p. 58; pp. 10-11 and Appendix 6, pp. 201-217 of MRID 49193105). In the ECM, the LOQ was defined as the lowest analyte concentration which yielded a mean recovery of 70-120% and relative standard deviation of ≤20%. The ECM study author also noted that the LOQ was a value which was no lower than four times the mean amplitude of the background noise of the untreated sample at the retention time of the analytes. The LOD for all analytes was 0.05 µg/L in the ECM and the ILV. In the ECM, the LOD was defined as the lowest analyte concentration detectable above the mean amplitude of the background noise of an untreated sample, as well as three times the background noise. The ECM study author noted that the LOD was approximately equivalent to half of the theoretical amount for a recovery sample at the method LOQ. The ECM study author also noted that LOD may vary based on the specific laboratory analytical instrument.

Table 4. Method Characteristics

	Fluazifop-P-Butyl (R154875; PP5)	**Fluazifop-P-Acid (R156172)**	**Compound IV (R150397; CGA181847)**	**Compound X (R154719; CGA142110)**
Limit of Quantitation (LOQ)	0.10 µg/L	0.10 µg/L	0.10 µg/L	0.10 µg/L
Limit of Detection (LOD)	0.05 µg/L[1]	0.05 µg/L[1]	0.05 µg/L[1]	0.05 µg/L[1]
Linearity (calibration curve r^2 and concentration range)	r^2 = 0.9997[1] (0.05-10 pg/µL)	r^2 = 0.9996-0.9999[1] (0.05-10 pg/µL)	r^2 = 0.9995-0.9998[1] (0.05-10 pg/µL)	r^2 = 0.9986-0.9992[1] (0.05-10 pg/µL)
Repeatable	Yes	Yes	Yes	Yes
Reproducible	Yes[2]	Yes[2]	Yes[2]	Yes[2]
Specific	Yes	Yes	Yes	Yes

Data were obtained from pp. 15, 25; Figure 16, p. 58; Figures 29-36, pp. 71-78 of the study report.
1. Calibration curves were reported for the quantification and confirmation ion transitions; no water matrix was specified. ILV calibration curves were linear, r^2 = *ca.* 0.9988-0.9998 for all four analytes, for concentration range of 0.5-100 pg (see Figures 61-64, pp. 92-95 and Appendix 6, pp. 201-216 of MRID 49193105). ILV calibration curves were calculated for the quantitative ion transition only. Reviewer-calculated calibration curves verified linearity for the ILV (r^2 = 0.9946-0.9999 for all four analytes in surface and ground water; reviewer-calculated values contain a degree of uncertainty due to poor resolution of the study report; see DER Attachment 2). Individual calibration data was not reported in the ECM.
2. The ECM validated the method using DI, finished, surface and ground water; the ILV only validated the method using surface and ground water.

IV. Method Deficiencies and Reviewer's Comments

1. In the ILV report, analyte residues in the matrix blanks were 0-50% of the LOD (Appendix 6, pp. 206-209, 214-217 of MRID 49193105). In the ECM report, analyte residues were in the matrix blanks, but the amounts were not reported. The ECM study

author stated that these interfering residues were due to residual analyte carryover and minor chromatographic and isobaric interferences (pp. 23). However, it was unclear whether they were due to matrix contamination or to residual carryover in the chromatographic equipment. The independent laboratory used matrices supplied by the registrant rather than independently sourced matrices. Guideline 850.6100 says that the independent laboratory "verifies that matrix control samples are free of interferences at the appropriate retention time, wavelength or detector setting" and that "interferences with peak areas that are less than 50 percent (%) at the MDL or LOD, are considered not significant." Residues in the matrix blanks were mostly less than 50% of the LOD, with one set approximately 50% of the LOD (p. 206 of MRID 49193105). The independent laboratory should have used independently sourced matrices and further limited interferences. The ECM report should have reported the amount of analyte residues in the matrix blanks (if the amounts were insignificant, it is unclear why recoveries were corrected for them).

2. Recovery values were corrected for residues found in the controls in both the ECM and ILV reports. Guideline 850.6100 says that "data from matrix control samples (blanks) are not used to correct values from spiked matrix controls for recoveries." In the ECM, all procedural recovery values were corrected for the average of the residues found in the controls (based on protocol and data in chromatograms of surface water; no other chromatograms or raw data were provided; p. 22; Tables 3-6, pp. 32-35; Tables 8-11, pp. 37-40; Figures 5-28, pp. 47-70). In the ILV, the only recovery values which were not corrected were those of fluazifop-P-acid and Compound IV in surface water due to absence of residues in the controls (pp. 10-11; Tables 3-10, pp. 23-30; Figures 41-48, pp. 72-79; Figures 52-60, pp. 83-91 of MRID 49193105).

3. In the ECM, sample chromatograms are only provided for surface water (Figures 1-28, pp. 47-70). No representative chromatograms were provided for the de-ionized, finished and ground water samples.

4. It was reported for the ILV that a single analyst completed a sample set consisting of 13 samples in less than 2 hours, not including LC/MS/MS (p. 18 of MRID 49193105).

5. The ILV concluded that the method was adequate as written (p. 18 of MRID 49193105).

V. References

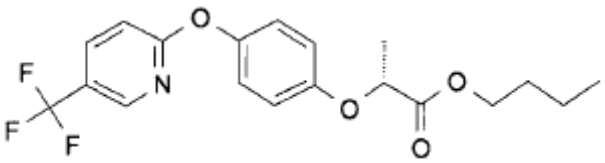
U.S. Environmental Protection Agency. 2012. Ecological Effects Test Guidelines, OCSPP 850.6100, Environmental Chemistry Methods and Associated Independent Laboratory Validation. Office of Chemical Safety and Pollution Prevention, Washington, DC. EPA 712-C-001.

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

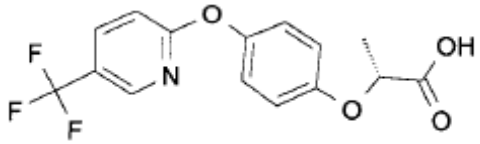
Fluazifop-P-Butyl; R154875; PP5

IUPAC Name:	(R)-2-[4-(5-Trifluoromethyl-pyridin-2-yloxy)-phenoxyl]-propionic acid butyl ester.
CAS Name:	(2R)-2-[4-[[5-(Trifluoromethyl)-2-pyridinyl]oxy]phenoxyl]-propionic acid butyl ester.
CAS Number:	79241-46-6
SMILES String:	Not reported



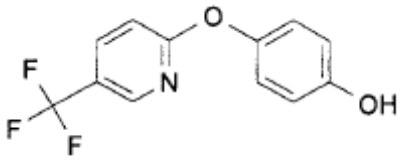
Fluazifop-P-Acid; R156172

IUPAC Name:	(R)-2-[4-(5-Trifluoromethyl-pyridin-2-yloxy)-phenoxyl]-propionic acid.
CAS Name:	(2R)-2-[4-[[5-(Trifluoromethyl)-2-pyridinyl]oxy]phenoxyl]-propionic acid.
CAS Number:	83066-88-0
SMILES String:	Not reported



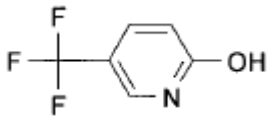
Compound IV; R150397; CGA181847

IUPAC Name:	4-(5-Trifluoromethyl-pyridin-2-yloxy)-phenol.
CAS Name:	4-[[5-(Trifluoromethyl)-2-pyridinyl]oxy]phenol.
CAS Number:	69045-85-8
SMILES String:	Not reported



Compound X; R154719; CGA142110

IUPAC Name: 5-Trifluoromethyl-pyridin-2-ol.
CAS Name: 5-(Trifluoromethyl)-2(1H)-pyridinone.
CAS Number: 33252-63-0
SMILES String: Not reported



Test Material:	Fluazifop-P-Butyl
MRID:	49193108
Title:	Fluazifop-P-Butyl: Fluazifop-P-Butyl – Residue Method for the Determination of Fluazifop-P-Butyl (R154875; PP5), Fluazifop-P-Acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in Water: Analytical Method
MRID:	49193105
Title:	Fluazifop-P-Butyl: Independent Laboratory Validation of Residue Method (GRM044.04A) for the Determination of Fluazifop-P-Butyl (R154875; PP5), Fluazifop-P-Acid (R156172), Compound IV (R150397; CGA181847) and Compound X (R154719; CGA142110) in Water: Final Report
EPA PC Code:	122809
OCSPP Guideline:	850.6100

For CDM Smith

Primary Reviewer: Lisa Muto **Signature:** Lisa Muto

Date: 7/8/14

Secondary Reviewer: Dan Hunt **Signature:** Dan Hunt

Date: 7/8/14

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

Date: 7/8/14