Analytical method for dicamba in air from air sampling tube and filter paper

Reports:	ECM: EPA MRID No.: 50102120. Mayer, L. 2017. Dicamba: Dicamba – Method GRM022.08A for the Determination of Dicamba from Air Sampling Tube and Filter Paper by LC-MS/MS – Analytical Method. Report No.: GRM022.08A. Task No.: TK0318998. Report prepared, sponsored and submitted by Syngenta Crop Protection, LLC., Greensboro, North Carolina; 48 pages. Final report issued March 6, 2017. ILV: EPA MRID No. 50102121. Xu, A. 2017. Dicamba: Independent Laboratory Validation of Analytical Method (GRM022.08A) for the Determination of Dicamba from Air Sampling Tube and Filter Paper by LC-MS/MS. ILV Final Report. Report No.: PASC-REP-1030. PASC Project No.: 141-1749. Task No.: TK0324474. Report prepared by Primera Analytical Solutions Corp., Princeton, New Jersey, sponsored and submitted by Syngenta Crop Protection, LLC., Greensboro, North Carolina; 146 pages. Final report issued March 3, 2017.
Document No.:	MRIDs 50102120 & 50102121
Guideline:	850.6100
Statements:	ECM: The study was not conducted in compliance with USEPA FIFRA Good Laboratory Practice (GLP) standards (p. 3 of MRID 50102120). 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 summary of revisions to previous method version was included (p. 4). ILV: The study was conducted in accordance with the USEPA FIFRA GLP standards (40 CFR Part 160; p. 3 of MRID 50102121). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4). An authenticity statement was not included.
Classification:	This analytical method is classified as **supplemental**. The specificity of the method for the air sampling tubes was not supported by the representative chromatograms of the ECM and ILV (OVS XAD-2 tubes) and the ILV LOQ (PUF). Updated ILV and ECM reports should be submitted with acceptable chromatographic support for the OVS XAD-2 tube and PUF matrix.
PC Code:	128931

EFED Final Reviewer:	Chuck Peck Senior Fate Scientist	Signature: Date:	2019.04.05 15:19:52 -04'00'
CDM/CSS-Dynamac JV Reviewers:	Lisa Muto, M.S., Environmental Scientist	Signature: Date:	Lisa Muto 10/23/17
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This Data Evaluation Record may have been altered by the Environmental Fate and Effects Division subsequent to signing by CDM/CSS-Dynamac JV personnel.

Executive Summary

This analytical method, Syngenta Method GRM022.08A, is designed for the quantitative determination of dicamba in air from air sampling tubes at the LOQ of 1.0 ng/air sample tube and from filter paper at the LOQ of 20 ng/filter paper using LC/MS/MS. The ECM and ILV test matrices were OVS XAD-2 sorbent air sampling tubes, PUF sorbent air sampling tubes, and Whatman™ Filter Paper. The ILV validated the method in all three matrices after one trial with insignificant modifications to the analytical method. One ion transition was monitored in the ECM; two ion transitions were monitored in the ILV. All ILV and ECM data regarding repeatability, accuracy, and precision were satisfactory for dicamba in all three matrices; however, no samples were prepared at 10×LOQ for the filter paper matrix in the ECM. ILV calibration curve linearity was not satisfactory for dicamba. Representative chromatograms did not support the specificity of the method for OVS XAD-2 (ECM and ILV) and PUF (ILV only) sorbent air sampling tubes. Updated ILV and ECM reports should be submitted with acceptable chromatographic support for all matrices or, if necessary, additional sample clean-up in the sample processing procedure prior to analysis.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Dicamba	50102120	50102121[1]		Air from air sampling tube[2]	06/03/2017	Syngenta Crop Protection, LLC	LC/MS/MS	1.0 ng/air sample tube
				Air from filter paper[3]				20 ng/filter paper

1 The ILV validated the method in all three matrices after one trial with insignificant modifications to the analytical instruments and parameters (pp. 11, 13-14, 17-21, 23 of MRID 50102121).

2 OVS XAD-2 sorbent (140/270 mg) air sampling tube (SKC Inc. Cat No. 226-30-16) and PUF sorbent (76-mm plug) air sampling tube (SKC Inc. Cat No. 226-92) and Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; p. 10 of MRID 50102120; pp. 14, 23 of MRID 50102121).

3 Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; p. 10 of MRID 50102120; pp. 14, 23 of MRID 50102121).

I. Principle of the Method

Air samples are collected from OVS XAD-2 sorbent (140/270 mg) air sampling tube (SKC Inc. Cat No. 226-30-16), PUF sorbent (76-mm plug) air sampling tube (SKC Inc. Cat No. 226-92) and Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; pp. 10, 13-14; Appendix 4, p. 48 of MRID 50102120). After the sample tubes or filter papers are fortified, if necessary, with the appropriate amount of dicamba in methanol, each sample should stand for at least five minutes before extraction.

Air samplers: Remove all contents of the tube into a 15 or 50 mL polypropylene tube (size of tube depends on extraction solvent volume); the glass tube is not included in the extraction (pp. 10, 13-14; Appendix 4, p. 48 of MRID 50102120). Extract sample using 10 to 40 mL of acidified acetone with 1% formic acid (volume of extraction solvent depending on sample type). After shaking samples for 30 minutes on a platform or orbital shaker, the samples are sonicated for 10 minutes, then centrifuged for 5 minutes at 5000 rpm, if necessary. An aliquot of the supernatant (volume depending on analytical instrument sensitivity) is transferred to a polypropylene tube, and the solvent is evaporated to near dryness (≤ 100µL) at 50°C under a gentle stream of nitrogen or air. The residue is mixed with 50 µL of 0.20 µg/mL of $^{13}C_6$ dicamba and reconstituted to 0.5 mL final volume using 0.1% formic acid ultra-pure water:methanol (95:5, v:v). After vortexing, the sample is analyzed by liquid chromatography/mas spectrometry/mass spectrometry (LC/MS/MS) directly or diluted, if necessary.

Filter papers: The filter papers should remain in the original collection tube and be located in the lower half of the tube (pp. 10, 13-14; Appendix 4, p. 48 of MRID 50102120). Extract the sample using 40 mL of acidified methanol with 1% formic acid. After shaking samples for 30 minutes on a platform or orbital shaker, the samples are sonicated for 10 minutes, then centrifuged for 5 minutes at 5000 rpm, if necessary. An aliquot of the supernatant (volume depending on analytical instrument sensitivity) is transferred to a polypropylene tube, and the solvent is evaporated to near dryness (≤ 100µL) at 50°C under a gentle stream of nitrogen or air. The residue is mixed with 50 µL of 0.20 µg/mL of $^{13}C_6$ dicamba and reconstituted to 0.5 mL final volume using 0.1% formic acid ultra-pure water:methanol (95:5, v:v). After vortexing, the sample is analyzed by LC/MS/MS directly or diluted, if necessary.

Samples were analyzed for dicamba by Acquity UPLC (Phenomenex Kinetex Phenyl-Hexyl column, 100 x 2.1 mm, 2.6 µm column; Ace 3 C18 column, 50 x 3.0 mm, 3.0 µm column; column temperature 50°C) using a gradient mobile phase of (A) 0.1% formic acid in Optima water and (B) 0.1% formic acid in Optima methanol [time ratio A:B; 0.0-2.0 min. 90:10, 2.0-5.0 min. 50:50, 5.0-7.1 min. 20:80, 7.1-10.0 min. 90:10] coupled with a Sciex API 5500QTRAP mass spectrometer with HESI-II probe (300°C) in ESI-negative ion mode (Multiple Reaction Monitoring mode, MRM; pp. 15-16 of MRID 50102120). Injection volumes were 10-50 µL. Dicamba was identified with the following two ion transitions (primary and confirmation, respectively): *m/z* 219→175 and *m/z* 221→177. One ion transition was used for the identification of $^{13}C_6$ dicamba: *m/z* 225→181. Retention time was 3.9 minutes for dicamba.

In the ILV, the ECM was performed as written, except for insignificant modifications of the analytical instruments and parameters (pp. 13-14, 17-20, 23 of MRID 50102121). The analytical

instrument was a Shimadzu UPLC system coupled to an AB Sciex 6500 mass spectrometer. The Phenomenex Kinetex Phenyl-Hexyl column was used; injection volume was 50 µL. Retention time was 4.5 minutes for dicamba. Some MS conditions differed, but the same ion transitions were used. No other modifications of the ECM were reported.

The Limit of Quantification (LOQ) for dicamba was reported as 1.0 ng/air sample tube and 20 ng/filter paper in the ECM and the ILV (pp. 10, 20 of MRID 50102120; pp. 11, 23 of MRID 50102121). The Limit of Detection (LOD) for dicamba was 0.10 pg/µL, equivalent to 5 pg injected on column when using a 50 µL injection volume by LC/MS/MS in the ECM. The LOD was not reported in the ILV.

II. Recovery Findings

ECM (MRID 50102120): Mean recoveries and relative standard deviations (RSDs) were within guideline requirements (mean 70-120%; RSD ≤20%) for analysis of dicamba from air sampling tubes at fortification levels of 1.0 ng/tube (LOQ) and 10.0 ng/tube (10×LOQ) and from filter paper at fortification levels of 20.0 ng/sample (LOQ) and 500.0 ng/sample (25×LOQ; Table 1, p. 23; DER Attachment 2). No samples were prepared at 10×LOQ for the filter paper matrix. Two ion transitions were reported in the method, but only the primary ion transition was monitored (*m/z* 219→175). A confirmatory method is not usually required when the primary analytical method is LC/MS/MS or GC/MS/MS. Standard deviations were reviewer-calculated using the individual recovery values reported in the study report since standard deviations were not provided by the study author (DER Attachment 2). The matrices were OVS XAD-2 sorbent (140/270 mg) air sampling tube (SKC Inc. Cat No. 226-30-16), PUF sorbent (76-mm plug) air sampling tube (SKC Inc. Cat No. 226-92) and Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; p. 10).

ILV (MRID 50102121): Mean recoveries and RSDs were within guideline requirements for analysis of dicamba from air sampling tubes at fortification levels of 1.0 ng/tube (LOQ) and 10.0 ng/tube (10×LOQ) and from filter paper at fortification levels of 20.0 ng/sample (LOQ) and 200 ng/sample (10×LOQ; pp. 11-12, 22). All analytes were identified using two ion transitions; performance data (recovery results) from primary and confirmatory analyses were comparable. The matrices were the same as those of the ECM (pp. 14, 23). The method was validated in all three matrices after one trial with insignificant modifications to the analytical instruments and parameters (pp. 11, 13-14, 17-21, 23).

Table 2. Initial Validation Method Recoveries for Dicamba in Air[1,2]

Analyte	Fortification Level (ng/tube or ng/sample)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)[3]	Relative Standard Deviation (%)
	OVS XAD-2 sorbent air sampling tube					
	Primary Transition					
Dicamba	1.0 (LOQ)	5	90-102	97	5	4.8
	10	5	85-96	92	4	4.5
	Confirmatory Transition					
Dicamba	1.0 (LOQ)	5	Not performed			
	10	5				
	PUF sorbent air sampling tube					
	Primary Transition					
Dicamba	1.0 (LOQ)	5	80-109	88	12	13.4
	10	5	95-106	100	4	4.5
	Confirmatory Transition					
Dicamba	1.0 (LOQ)	5	Not performed			
	10	5				
	Filter Paper					
	Primary Transition					
Dicamba	20.0 (LOQ)	5	96-110	102	5	5.1
	500	5	92-99	96	3	3.2
	Confirmatory Transition					
Dicamba	20.0 (LOQ)	5	Not performed			
	500	5				

Data (uncorrected recovery results, pp. 17-18; Table 1, p. 23) were obtained from Table 1, p. 23 of MRID 50102120 and DER Attachment 2.

1 The matrices were OVS XAD-2 sorbent (140/270 mg) air sampling tube (SKC Inc. Cat No. 226-30-16), PUF sorbent (76-mm plug) air sampling tube (SKC Inc. Cat No. 226-92) and Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; p. 10 of MRID 50102120).

2 Two ion transitions were reported in the method, but only the primary ion transition was monitored (*m/z* 219→175). A confirmatory method is not usually required when the primary analytical method is LC/MS/MS or GC/MS/MS.

3 Standard deviations were reviewer-calculated using the data in the study report since the study author did not report these values (see DER Attachment 2). Rules of significant figures were followed.

Table 3. Independent Validation Method Recoveries for Dicamba in Air[1,2]

Analyte	Fortification Level (ng/tube or ng/sample)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
	OVS XAD-2 sorbent air sampling tube					
	Primary Transition					
Dicamba	1.0 (LOQ)	5	81.9-104	90.3	8.6	9.5
	10	5	75.7-83.1	78.8	2.7	3.4
	Confirmatory Transition					
Dicamba	1.0 (LOQ)	5	91.2-105	96.3	5.8	6.1
	10	5	72.7-79.8	75.5	2.6	3.5
	PUF sorbent air sampling tube					
	Primary Transition					
Dicamba	1.0 (LOQ)	5	92.5-105	98.4	5.0	5.1
	10	5	70.6-85.7	79.2	6.7	8.5
	Confirmatory Transition					
Dicamba	1.0 (LOQ)	5	86.7-96.2	91.5	3.4	3.8
	10	5	68.0-82.3	76.9	6.5	8.5
	Filter Paper					
	Primary Transition					
Dicamba	20.0 (LOQ)	5	90.0-93.3	91.3	1.2	1.4
	200	5	81.5-84.4	83.5	1.2	1.5
	Confirmatory Transition					
Dicamba	20.0 (LOQ)	5	93.0-95.7	94.3	1.4	1.4
	200	5	81.9-85.2	84.0	1.3	1.6

Data (uncorrected recovery results,) were obtained from pp. 11-12, 22 of MRID 50102121.

1 The matrices were OVS XAD-2 sorbent (140/270 mg) air sampling tube (SKC Inc. Cat No. 226-30-16), PUF sorbent (76-mm plug) air sampling tube (SKC Inc. Cat No. 226-92) and Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; pp. 14, 23 of MRID 50102121).

2 Dicamba was identified with the following two ion transitions (primary and confirmation, respectively): *m/z* 219→175 and *m/z* 221→177.

III. Method Characteristics

Table 4 provides a summary of the method characteristics for both the eCM and ILV. The LOQ for dicamba was reported as 1.0 ng/air sample tube and 20 ng/filter paper in the ECM and the ILV (pp. 10, 20 of MRID 50102120; pp. 11, 23 of MRID 50102121). In the ECM, the LOQ was defined as the lowest analyte concentration which was demonstrated to have acceptable mean recovery (70 to 120%) and precision (relative standard deviation ≤ 20%). The ECM also stated that the response of the LOQ analyte peak should be no lower than four times the mean amplitude of the background noise in an untreated sample at the corresponding retention time. No justification for the LOQ was provided in the ILV. The LOD for dicamba in the ECM was 0.10 pg/µL, equivalent to 5 pg injected on column when using a 50 µL injection volume by LC/MS/MS. In the ECM, the LOD was defined as the lowest analyte concentration detectable above the mean amplitude of the background noise in an untreated sample at the corresponding retention time. The ECM also stated that an estimate of the LOD can be taken as three times background noise. The LOD was not reported in the ILV.

Table 4. Method Characteristics

Analyte		**Dicamba**		
Matrix		**OVS XAD2 Tube**	**PUF Tube**	**Filter Paper**
Limit of Quantitation (LOQ)		1.0 ng/air sample tube		20 ng/filter paper
Limit of Detection (LOD)	ECM	0.10 pg/µL (5 pg injected on column when using a 50 µL injection volume)		
	ILV	Not reported		
Linearity (calibration curve r^2 and concentration range)	ECM[1]	r^2 = 0.9999 (Q) (0.1 pg/µL to 80 pg/µL)		
	ILV[2]	r^2 = **0.9936** (Q) r^2 = **0.9932** (C) (0.2-20 ng/mL)		
Repeatable	ECM[1,3]	Yes at LOQ and 10×LOQ.		Yes at LOQ and 25×LOQ.
	ILV[4,5]	Yes at LOQ and 10×LOQ.		
Reproducible		Yes at LOQ.		Yes at LOQ.
Specific	ECM	Matrix interferences were 25-30% of the LOQ (based on peak height estimation), and significant contaminant (peak height equal to LOQ peak; peak area 150% of LOQ peak area) very near analyte retention time was observed.[6]	Yes, matrix interferences were <1% of the LOQ (based on peak area).	
	ILV	Matrix interferences which interfered with peak integration were noted in the LOQ Q & C chromatograms.[7] Significant contaminant (peak height 300% of LOQ peak height) near analyte retention time was observed.	A significant matrix contaminant was attached to the LOQ peak which interfered with peak integration in the LOQ Q chromatograms.[8]	Matrix interferences which interfered with peak integration were noted in the LOQ Q chromatograms.

Data were obtained from pp. 10, 12, 20; Table 1, p. 23 (recovery data); Figure 9, p. 33 (calibration curve); Figures 10-19, pp. 34-43 (chromatograms) of MRID 50102120; pp. 11-12, 22-23 (recovery data); Figures 9-95, pp. 35-81 (calibration curves and chromatograms) of MRID 50102121 and DER Attachment 2. Q = primary ion transition; C = confirmatory ion transition.

1 In the ECM, two ion transitions were reported in the method, but only the primary ion transition was monitored. A confirmatory method is not usually required when the primary analytical method is LC/MS/MS or GC/MS/MS.

2 For the ILV, correlation coefficients were reviewer-calculated from r values provided in the study report (Figure 9, p. 35 and Figure 56, p. 60 of MRID 50102121; DER Attachment 2).

3 The ECM matrices were OVS XAD-2 sorbent (140/270 mg) air sampling tube (SKC Inc. Cat No. 226-30-16), PUF sorbent (76-mm plug) air sampling tube (SKC Inc. Cat No. 226-92) and Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; p. 10 of MRID 50102120).

4 The ILV matrices were OVS XAD-2 sorbent (140/270 mg) air sampling tube (SKC Inc. Cat No. 226-30-16), PUF sorbent (76-mm plug) air sampling tube (SKC Inc. Cat No. 226-92) and Filter Paper (Whatman™ Qualitative Grade Plain Circles and Sheets – Grade 3, 15 cm diameter; pp. 14, 23 of MRID 50102121).

5 The ILV validated the method in all three matrices after one trial with insignificant modifications to the analytical instruments and parameters (pp. 11, 13-14, 17-21, 23 of MRID 50102121).

6 Based on Figures 17-18, pp. 41-42 of MRID 50102120. Study author noted that the interference peak was erroneously integrated instead of the matrix interference at the analyte retention time in the control sample.

7 Based on Figures 26-30, pp. 44-46 of MRID 50102121. In Figure 26, a matrix contaminant (peak height 90% of LOQ peak height) appeared to be joined to the integrated analyte peak.
8 Based on Figures 39-43, pp. 51-53 of MRID 50102121.
Linearity is satisfactory when $r^2 \geq 0.995$.

IV. Method Deficiencies and Reviewer's Comments

1. In the ILV, linearity was not satisfactory for dicamba [$r^2 = 0.9936$ (Q) and $r^2 = 0.9932$ (C); Figure 9, p. 35; Figure 56, p. 60 of MRID 50102121; DER Attachment 2]. Linearity is satisfactory when $r^2 \geq 0.995$.

2. In the ECM, no samples were prepared at 10×LOQ for the filter paper matrix (Table 1, p. 23 of MRID 50102120; DER Attachment 2). A validation sample set should consist of, at a minimum, a reagent blank, two unspiked matrix control samples, five matrix control samples spike at the LOQ, and five matrix control samples spiked at 10×LOQ for each analyte and matrix.

3. The ECM and ILV LOQ chromatograms for OVS XAD-2 sorbent air sampling tubes did not support the specificity of the method for that matrix (Figures 17-18, pp. 41-42 of MRID 50102120; Figures 26-30, pp. 44-46 of MRID 50102121). The chromatograms contained matrix interferences which interfered with peak integration. Additionally, a significant contaminant (peak height equal to ≥ LOQ peak) near analyte retention time was observed in the ECM and ILV chromatograms. In Figure 26 of the ILV chromatograms, a matrix contaminant (peak height 90% of LOQ peak height) appeared to be joined to the integrated analyte peak. For the control sample chromatogram in the ECM, the study author noted that the interference peak was erroneously integrated instead of the matrix interference at the analyte retention time; therefore, the matrix interference could not be fully accessed by the reviewer, and the interference of this matrix contaminant was demonstrated by being mistaken for the analyte. Updated ILV and ECM reports should be submitted with acceptable chromatographic support for the OVS XAD-2 tube matrix.

4. The ILV LOQ chromatograms for PUF sorbent air sampling tubes did not support the specificity of the method for that matrix (Figures 39-43, pp. 51-53 of MRID 50102121). A significant matrix contaminant was attached to the LOQ peak which interfered with peak integration in the LOQ Q chromatograms. The reviewer observed that the peak integration of the analyte appeared to cut off a portion of the total LOQ peak. Updated ILV report should be submitted with acceptable chromatographic support for the PUF tube matrix.

5. Overall, the reviewer noted that additional sample clean-up in the sample processing procedure may have been required since the chromatograms of the ECM and ILV contained many matrix contaminants, especially the sample tube matrices (see Reviewer's Comments #3 & 4).

6. The estimations of LOQ and LOD in ECM were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 ILV (pp. 10, 20 of MRID 50102120; pp. 11, 23 of MRID 50102121). In the ECM, the LOQ was defined as the lowest analyte concentration which was demonstrated to have acceptable mean recovery (70 to 120%) and precision (relative standard deviation of 20%). The ECM also stated that the response of the LOQ analyte peak should be no lower than four times the mean amplitude of the background noise in an untreated sample at the corresponding retention time. No justification for the LOQ was provided in the ILV. In the ECM, the LOD was defined as the lowest analyte concentration detectable above the mean amplitude of the background noise in an untreated sample at the corresponding retention time. The ECM also stated that an estimate of the LOD can be taken as three times background noise. The LOD was not reported in the ILV.

7. Communications between the ILV and study monitor were summarized as 1) clarification/approval of the protocol and method, and 2) acquisition of analytical standard and control sample (p. 23 of MRID 50102121). The list of email communications was included in the raw data, but not provided for review.

8. The only deviation reported by the ILV was to address the preparation of the standard solutions in clear volumetric flasks (p. 23 of MRID 50102121).

9. In the ECM, no significant matrix effects have been observed for air sample type tested (p. 20 of MRID 50102120). Non-matrix-matched standards were recommended, but an internal standard was suggested to compensate for any matrix effects or sample-to-sample variability. The internal standard was used in the ILV to combat any matrix effects (p. 23 of MRID 50102121).

10. In the ECM, the final extracts in 0.1% formic acid in ultra-pure water:methanol (95:5, v:v) was found to be acceptable after 7 days of refrigerated storage (4°C; p. 21 of MRID 50102120).

11. The reviewer noted the following typographical errors: 1) in the title of Figure 16 of the ECM, the 500 ng fortification was noted as 20×LOQ instead of 25×LOQ; and 2) in the title of Figure 18 of the ILV, OVS Tube was written instead of Filter Paper (Figure 16, p. 40 of MRID 50102120; Figure 18, p. 40 of MRID 50102121).

12. It was reported for the ILV that one batch of thirteen samples required one working day with LC/MS/MS performed overnight (p. 23 of MRID 50102121).

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

Dicamba (BAS183 22 H)

IUPAC Name: 3,6-Dichloro-o-anisic acid
CAS Name: 3,6-Dichloro-2-methoxybenzoic acid
CAS Number: 1918-00-9
SMILES String: COc1c(Cl)ccc(Cl)c1C(O)=O