

**Analytical method for aldicarb and its metabolites, aldicarb-sulfone and aldicarb-sulfoxide, in soil**

**Reports:** ECM: EPA MRID No.: 49793901. DeVellis, S. R. 2015. Aldicarb, Aldicarb-Sulfone and Aldicarb-Sulfoxide – Validation of the Analytical Method for the Determination of Test Substances in Soil. Smithers Viscient Study No.: 14070.6103. Report prepared by Smithers Viscient, Wareham, Massachusetts, and sponsored and submitted by AgLogic, LLC, Chapel Hill, North Carolina; 48 pages. Final report issued October 27, 2015.

ILV: EPA MRID No. 49793902. Wu, X. 2015. Independent Laboratory Validation (ILV) of the Analytical Method: Aldicarb and Metabolites: Aldicarb, Aldicarb-Sulfone and Aldicarb-Sulfoxide – Validation of the Analytical Method for the Determination of Test Substances in Soil. Smithers Viscient Study No.: 14070.6102. Report prepared by Smithers Viscient, Wareham, Massachusetts, and sponsored and submitted by AgLogic, LLC, Chapel Hill, North Carolina; 52 pages. Final report issued December 10, 2015.

**Document No.:** MRIDs 49793901 & 49793902

**Guideline:** 850.6100

**Statements:** ECM: The study was conducted in accordance with USEPA FIFRA and OECD Good Laboratory Practice (GLP) standards (p. 3 of MRID 49793901). Signed and dated No Data Confidentiality, GLP, and Quality Assurance statements were provided (pp. 2-4). A statement of the authenticity of the study report was included with the quality assurance statement (p. 4).

ILV: The study was conducted in accordance with USEPA FIFRA GLP standards (p. 3 of MRID 49793902). Signed and dated No Data Confidentiality, GLP, and Quality Assurance statements were provided (pp. 2-4). A statement of the authenticity of the study report was included with the quality assurance statement (p. 4).

**Classification:** This analytical method is classified as supplemental. Insufficient information was provided to verify that the ECM laboratory was distinct from the ILV laboratory. The method LOQs of the ECM and ILV differed. In the ECM and ILV, no performance data was provided at the ECM LOQ to validate the method. An insufficient number of samples was prepared for all fortification levels in the ECM. In the ILV, chromatograms for aldicarb-sulfoxide did not support the specificity of the method. The correlation coefficient ( $r^2$ ) was 0.99001 for the ECM calibration curve of aldicarb-sulfone.

**PC Code:** 098301**Reviewer:**James Lin  
Environmental Engineer  
**Signature:**  
**Date:** February 23, 2017**Executive Summary**

This analytical method, Smithers Viscient Study No.: 14070.6103, is designed for the quantitative determination of aldicarb and its metabolites, aldicarb-sulfone and aldicarb-sulfoxide, in soil using LC/MS/MS. The method LOQs of the ECM and ILV differed: the method LOQ for all three analytes was calculated as 5.00 µg/L in the ECM and reported as 10.0 µg/kg in the ILV. The ECM LOQ is lower than the lowest toxicological level of concern in soil. The ILV LOQ is lower than the lowest toxicological level of concern in soil. Insufficient information, specifically communication log, was provided to verify that the ECM laboratory was distinct from the ILV laboratory. The soil matrices were not characterized in the ECM or ILV; the soil matrix was the same in the ECM and ILV. It could not be determined if the ILV was provided with the most difficult matrix with which to validate the method. No performance data was provided in the ECM or ILV to validate the ECM LOQ for the method. The specificity of the method was not supported by the ILV chromatograms for aldicarb-sulfoxide due to major baseline noise and a minor nearby contaminant which greatly interfered with peak integration. In the ECM, insufficient samples were prepared at each fortification level. The reviewer assumed that the method was validated by the ILV in the first trial, with insignificant modifications to the analytical parameters, at the ILV LOQ.

**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						
Aldicarb	49793901 <sup>1</sup>	49793902 <sup>2</sup>		Soil	27/10/2015	AgLogic, LLC	LC/MS/MS	5.00 µg/L (ECM) <sup>3</sup>
Aldicarb- sulfone								10.0 µg/kg (ILV) <sup>4</sup>
Aldicarb- sulfoxide								

1 In the ECM, uncharacterized sandy loam soil matrix (SMV Lot No. 060315, Sample ID 2014 100 ROCH LOAM) from Rochester, Massachusetts, was used; soil moisture was determined prior to testing (10.79%; p. 11 of MRID 49793901).

2 In the ILV, uncharacterized sandy loam soil matrix (SMV Lot No. 021814, Sample ID 2014 100 ROCH LOAM) from Rochester, Massachusetts, was used; soil moisture was determined prior to testing (13.61%; p. 11 of MRID 49793902).

3 ECM LOQ (calculated) which was not validated by the ECM or ILV. See Reviewer Comments #2 & 3.

4 ILV LOQ (reported) which was validated by the ILV. See Reviewer Comments #2 & 3.

## I. Principle of the Method

Samples (5.00 g dry weight) of soil in 50.0-mL Nalgene<sup>®</sup> centrifuge tubes were fortified, as necessary, then extracted twice with 0.1% formic acid in acetonitrile (2 x 20.0 mL) by shaking via shaker table for 30 minutes at 150 rpm (pp. 10, 13-14 of MRID 49793901). Soil and extract were separated by centrifugation (10 minutes at 3000 rpm) and decanting into 50.0-mL volumetric flasks. The volume of the combined extracts was adjusted to 50 mL using 0.1% formic acid in acetonitrile. Samples were further diluted (100-200 dilution factor) into calibration range with acetonitrile:purified reagent water (20:80, v:v) prior to analysis.

Samples were analyzed for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide by HPLC/MS/MS (MDS Sciex API 5000 Mass Spectrometer equipped with an ESI Turbo V Ion Source; Serial No. L20354655487) employing a XBridge C18, 2.1 mm x 50 mm, 2.5  $\mu$ m column (column temperature, 30°C) and mobile phase of (A) purified reagent water with 0.1% formic acid and (B) acetonitrile with 0.1% formic acid [percent A:B at 0.50 min. 95:5, 1.50-2.50 min. 0:100, 2.60-4.10 min. 95:5] with an AB Sciex Turbo V ESI Ion Spray source (positive ionization mode and multiple reaction monitoring, MRM; 500°C; pp. 10, 14-16 of MRID 49793901). Injection volume was 100  $\mu$ L. Analytes were identified using one ion transition: 213.10 $\rightarrow$ 116.10 amu for aldicarb; 223.10 $\rightarrow$ 148.00 amu for aldicarb-sulfone; and 207.10 $\rightarrow$ 132.10 amu for aldicarb-sulfoxide. The retention times were *ca.* 3.2 minutes for aldicarb and *ca.* 3.1 minutes for aldicarb-sulfone and aldicarb-sulfoxide. The ECM study author noted that instrument optimization may be required if other instruments were used.

In the ILV, the method was performed exactly as written in the ECM (pp. 10, 13-14, 16 of MRID 49793902). For the analytical method, an AB Sciex API Mass Spectrometer equipped with an ESI Turbo V Ion Source was used (Serial No. not reported). Two noted differences in the analytical conditions were the mobile phase gradient percent [A:B at 0.00-1.20 min. 98.0:2.0, 2.00-3.00 min. 0.0:100.0, 3.20-4.50 min. 98.0:2.0] and the injection volume was decreased to 50  $\mu$ L. The same ion transition was monitored as the ECM. The retention times were *ca.* 2.02, 1.83 and 1.77 minutes for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide, respectively.

In the ECM, the Limit of Quantification (LOQ) was reported as 5.00  $\mu$ g/L for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide (See Reviewer Comment #2; pp. 17-18 of MRID 49793901). The Limit of Detection (LOD) was reported as 1.00, 0.600 and 0.300  $\mu$ g/L for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide, respectively. In the ILV, the LOQ was reported as 10.0  $\mu$ g/kg for all three analytes (See Reviewer Comment #2; pp. 8, 18-20 of MRID 49793902). The LOD was reported as 5, 1 and 2  $\mu$ g/kg for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide, respectively.

## II. Recovery Findings

ECM (MRID 49793901): Mean recoveries and relative standard deviations (RSDs) were within guideline requirements (mean 70-120%; RSD  $\leq$ 20%) for analysis of aldicarb, aldicarb-sulfone and aldicarb-sulfoxide in sandy loam soil at fortification levels of 10.0  $\mu$ g/kg (2 $\times$ LOQ) and 100  $\mu$ g/kg (20 $\times$ LOQ; Tables 1-3, pp. 22-24). The number of samples was insufficient for all analyses (n = 3). Only one ion transition was monitored for each analyte; a confirmatory method is not usually required when LC/MS and GC/MS is the primary method. No samples were fortified at

the ECM LOQ (5.00 µg/L). The soil was not characterized (Rochester Sandy Loam soil; SMV Lot No. 060315, Sample ID 2014 100 ROCH LOAM); soil moisture was determined prior to testing (10.79%; soil from Rochester, Massachusetts; p. 11).

ILV (MRID 49793902): Mean recoveries and relative standard deviations (RSDs) were within guideline requirements for analysis of aldicarb, aldicarb-sulfone and aldicarb-sulfoxide in sandy loam soil at fortification levels of 10.0 µg/kg (LOQ) and 100 µg/kg (10×LOQ; Tables 1-3, pp. 23-25). Only one ion transition was monitored for each analyte. The soil was not characterized (Rochester Sandy Loam soil, SMV Lot No. 021814, Sample ID 2014 100 ROCH LOAM); soil moisture was determined prior to testing (13.61%; p. 11). The number of trials was not specifically reported; however, the reviewer assumed that the method was validated in the first trial with insignificant modifications to the analytical parameters (pp. 8, 10, 13-14, 16, 19-20).

**Table 2. Initial Validation Method Recoveries for Aldicarb, Aldicarb-Sulfone and Aldicarb-Sulfoxide in Soil<sup>1,2</sup>**

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Sandy Loam Soil</b>						
Aldicarb	10.0	3	92.0-111	104	10.4	10.0
	100	3	103-119	111	8.11	7.34
Aldicarb-sulfone	10.0	3	70.9-78.3	73.7	4.02	5.46
	100	3	86.4-88.5	87.5	1.08	1.23
Aldicarb-sulfoxide	10.0	3	73.4-79.9	75.7	3.67	4.85
	100	3 <sup>3</sup>	64.6-75.9	71.5	6.0	8.4

Data (uncorrected recovery results; pp. 16-17) were obtained from Tables 1-3, pp. 22-24 of MRID 49793901 and DER Attachment 2. ECM LOQ = 5.00 µg/L.

1 The sandy loam soil matrix (SMV Lot No. 060315, Sample ID 2014 100 ROCH LOAM) from Rochester, Massachusetts, was not characterized; soil moisture was determined prior to testing (10.79%; p. 11).

2 Analytes were identified using one ion transition: 213.10→116.10 amu for aldicarb; 223.10→148.00 amu for aldicarb-sulfone; and 207.10→132.10 amu for aldicarb-sulfoxide.

3 The study author excluded one sample from the statistical analysis, n = 2 (74.9% mean, 1.38% s.d., 1.84% RSD; Table 3, p. 24). The results provided were reviewer-calculated using all samples, n = 3 (see DER Attachment 2).

**Table 3. Independent Validation Method Recoveries for Aldicarb, Aldicarb-Sulfone and Aldicarb-Sulfoxide in Soil<sup>1,2</sup>**

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Sandy Loam Soil</b>						
Aldicarb	10.0 (LOQ)	5	91.7-113	104	7.91	7.61
	100	5	95.4-111	103	6.90	6.69
Aldicarb-sulfone	10.0 (LOQ)	5	104-110	108	2.22	2.06
	100	5	111-116	115	2.17	1.89
Aldicarb-sulfoxide	10.0 (LOQ)	5	71.5-76.7	74.1	2.14	2.89
	100	5	72.0-86.6	77.5	5.96	7.68

Data (uncorrected recovery results; pp. 17-18) were obtained from Tables 1-3, pp. 23-25 of MRID 49793902.

1 The sandy loam soil matrix (SMV Lot No. 021814, Sample ID 2014 100 ROCH LOAM) from Rochester, Massachusetts, was not characterized; soil moisture was determined prior to testing (13.61%; p. 11).

2 Analytes were identified using one ion transition: 213.10→116.10 amu for aldicarb; 223.10→148.00 amu for aldicarb-sulfone; and 207.10→132.10 amu for aldicarb-sulfoxide.

### III. Method Characteristics

In the ECM, the LOQ was reported as 5.00 µg/L for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide (See Reviewer Comment #2; pp. 17-18 of MRID 49793901). The LOD was reported as 1.00, 0.600 and 0.300 µg/L for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide, respectively. The LOQ was calculated from the LOQ of the instrument (using the lowest concentration calibration standard) and the dilution factor of the control samples using the following equation:

$$LOQ_{INST} = [-b + \sqrt{(b^2 - 4aC)}] / 2a$$

$$LOQ = LOQ_{INST} \times DF_{CTRL}$$

Where,  $LOQ_{INST}$  is the limit of quantitation on the instrument, a, b and c are regression constants, C equals  $(c - Area_{LS})$ , where  $Area_{LS}$  is the mean detector response (peak area) of the low concentration calibration standard (two injections), LOQ is the limit of quantitation reported for the analysis and  $DF_{CTRL}$  is the dilution factor of the control samples (smallest dilution factor used).

The LOD was calculated in the ECM using the following equation:

$$LOD = (3 \times (SN_{ctl}) / (Resp_{LS}) \times Conc_{LS}$$

Where, LOD is the limit of detection of the analysis,  $SN_{ctl}$  is the mean signal to noise in height of the control samples (or Blanks),  $Resp_{LS}$  is the mean response in height of the two low calibration standards, and  $Conc_{LS}$  is the concentration of the low calibration standard.

In the ILV, the LOQ was reported as 10.0 µg/kg for all three analytes (See Reviewer Comment #2; pp. 8, 18-20 of MRID 49793902). The LOD was reported as 5, 1 and 2 µg/kg for aldicarb, aldicarb-sulfone and aldicarb-sulfoxide, respectively. No calculations for the LOQ were reported.

The LOD was calculated in the ILV using the following equation:

$$\text{LOD} = \text{LOD}_{\text{LCAL}} \times \text{DF}_{\text{CTRL}}$$

Where, LOD is the limit of detection of the analysis,  $\text{LOD}_{\text{LCAL}}$  is the lowest concentration calibration standard (0.0500  $\mu\text{g/L}$ ) and  $\text{DF}_{\text{CTRL}}$  is the dilution factor of the control samples (smallest dilution factor used, 100).

**Table 4. Method Characteristics**

		Aldicarb	Aldicarb-sulfone	Aldicarb-sulfoxide
Limit of Quantitation (LOQ) <sup>1</sup>	ECM	5.00 $\mu\text{g/L}$ (calculated)		
	ILV	10.0 $\mu\text{g/kg}$ (reported)		
Limit of Detection (LOD)	ECM	1.00 $\mu\text{g/L}$	0.600 $\mu\text{g/L}$	0.300 $\mu\text{g/L}$
	ILV	5 $\mu\text{g/kg}$	1 $\mu\text{g/kg}$	2 $\mu\text{g/kg}$
Linearity (Least squares calibration curve r and concentration range)	ECM	$r^2 = 0.99595$ (0.0500-1.25 $\mu\text{g/L}$ )	$r^2 = \mathbf{0.99001}$ (0.0500-1.75 $\mu\text{g/L}$ )	$r^2 = 0.99555$ (0.0500-1.75 $\mu\text{g/L}$ )
	ILV	$r^2 = 0.99829$ (0.0500-1.75 $\mu\text{g/L}$ )	$r^2 = 0.99503$ (0.0500-1.25 $\mu\text{g/L}$ )	$r^2 = 0.99689$ (0.0500-1.75 $\mu\text{g/L}$ )
Repeatable	ECM <sup>2</sup>	Yes at 2 $\times$ LOQ and 20 $\times$ LOQ, but n = 3. (Based on ECM LOQ)		
	ILV <sup>3</sup>	Yes at LOQ and 10 $\times$ LOQ. (Based on ILV LOQ)		
Reproducible	Yes at LOQ and 10 $\times$ LOQ. (Based on ILV LOQ) No samples were dosed at ECM LOQ in the ECM or ILV.			
Specific	ECM	Yes, matrix interferences were <5% of the 2 $\times$ LOQ. Minor baseline noise and a minor nearby contaminant interfered slightly with peak integration.	Yes, matrix interferences were <5% of the 2 $\times$ LOQ. Minor baseline noise interfered slightly with peak integration.	
	ILV	Yes, matrix interferences were <15% of the LOQ. Major baseline noise interfered with peak distinction at the LOQ and peak integration at 10 $\times$ LOQ. <sup>4</sup>	Yes, matrix interferences were <5% of the LOQ. Minor baseline noise interfered slightly with peak integration. Peak tailing was observed.	<b>No</b> , matrix interferences were <5% of the LOQ; however, major baseline noise and a minor nearby contaminant greatly interfered with peak integration. <sup>5</sup>

Data were obtained from pp. 17-18; Tables 1-3, pp. 22-24; Figures 1-15, pp. 25-39 of MRID 49793901; pp. 8, 18-20; Tables 1-3, pp. 23-25; Figures 1-18, pp. 26-43 of MRID 49793902; DER Attachment 2.

1 See Reviewer's Comment #2.

2 In the ECM, uncharacterized sandy loam soil matrix (SMV Lot No. 060315, Sample ID 2014 100 ROCH LOAM) from Rochester, Massachusetts, was used; soil moisture was determined prior to testing (10.79%; p. 11 of MRID 49793901).

3 In the ILV, uncharacterized sandy loam soil matrix (SMV Lot No. 021814, Sample ID 2014 100 ROCH LOAM) from Rochester, Massachusetts, was used; soil moisture was determined prior to testing (13.61%; p. 11 of MRID 49793902).

4 Based on Figures 1-2, pp. 26-27 and Figures 4-5, pp. 29-30 of MRID 49793901.

5 Based on Figures 13-14, pp. 38-39 and Figures 16-17, pp. 41-42 of MRID 49793901.

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

Linearity is satisfactory when  $r^2 \geq 0.995$ .

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

1. It could not be determined if the ILV was independent of the ECM due to lack of communication information. According to the OCSPP guidelines, if the laboratory that conducted the validation belonged to the same organization as the originating laboratory, the analysts, study director, equipment, instruments, and supplies of the two laboratories must have been distinct and operated separately and without collusion, and the analysts and study director of the ILV must have been unfamiliar with the method both in its development and subsequent use in field studies. The laboratory which performed the ECM, Smithers Viscient, was the same laboratory which performed the ILV (pp. 1, 5 of MRID 49793901; pp. 1, 5 of MRID 49793902). Based on the information reported in the ECM and ILV, the reviewer concluded that insufficient information was provided to verify that the ECM laboratory was distinct from the ILV laboratory.

Regarding analysts and study director: In the lists of Key Study Personnel for the ECM and ILV, the study directors were different (p. 5 of MRID 49793901; p. 5 of MRID 49793902). In addition, the ECM listed two chemistry technicians while the ILV listed none. However, the Director of Chemistry was identical between the ECM and ILV. No statement was provided in the ILV which stated that the ILV laboratory personnel had no prior knowledge of the method or communication with the method development laboratory.

Regarding equipment, instruments and supplies: In Section 2.0 Materials and Methods of the ECM and ILV, the test and reference substances, reagents and laboratory equipment were the same or similar (pp. 8-10, 14 of MRID 49793901; pp. 9-10, 14-15 of MRID 49793902). However, the analytical instruments differed. The ECM analytical instrument was a MDS Sciex API 5000 Mass Spectrometer equipped with an ESI Turbo V Ion Source (Serial No. L20354655487) with Shimadzu auxiliary equipment. The ILV analytical instrument was an AB Sciex API Mass Spectrometer equipped with an ESI Turbo V Ion Source with Acquity auxiliary equipment (Serial No. was not reported).

Regarding communication: The ILV report stated that communication included protocol and method clarification, acquisition of the test materials and test matrix, and pre-validation evaluation and method establishment (p. 17 of MRID 49793902). A log of the communications was not provided in the ILV; it was reportedly contained in the study raw data.

Additionally, the soil matrix was the same in the ECM and ILV (p. 11 of MRID 49793901; p. 11 of MRID 49793902). The Sample ID was the same, but the Lot No. and soil moisture content differed between the two studies.

2. The Method LOQs of the ECM and ILV differed: the method LOQ for all three analytes was calculated as 5.00 µg/L in the ECM and reported as 10.0 µg/kg in the ILV (pp. 17-18; Appendix 1, pp. 44, 47 of MRID 49793901; pp. 8, 18-20 of MRID 49793902). In the ECM, the 10.0 µg/kg fortification level was referred to as the "low concentration" level, not the LOQ fortification. The reviewer specified the LOQs as either the ECM LOQ or

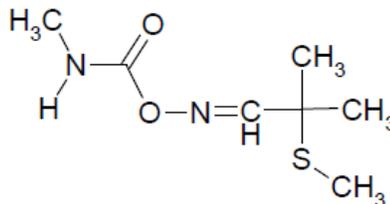
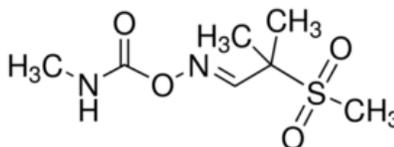
- ILV LOQ, unless the sentence or section already specified that data from the ECM or ILV was being reported.
3. In the ECM and ILV, no performance data was provided for the ECM LOQ to validate the method at 5.00 µg/L. A validation sample set should consist of, at a minimum, a reagent blank, two unspiked 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. In the ECM and ILV, samples were only prepared at 10.0 µg/kg and 100 µg/kg (Tables 1-3, pp. 22-24 of MRID 49793901; Tables 1-3, pp. 23-25 of MRID 49793902).
  4. In the ECM, only three samples were prepared at each fortification level (2×LOQ and 20×LOQ; Tables 1-3, pp. 22-24 of MRID 49793901). The OCSPP guidelines specify that five matrix control samples are prepared at each fortification level.
  5. The specificity of the method was not supported by the ILV chromatograms for aldicarb-sulfoxide due to major baseline noise and a minor nearby contaminant which greatly interfered with peak integration (Figures 13-14, pp. 38-39; Figures 16-17, pp. 41-42 of MRID 49793902).
  6. ECM correlation coefficient ( $r^2$ ) was 0.99001 for the calibration curve of aldicarb-sulfone (Figure 10, p. 34 of MRID 49798601). Linearity is satisfactory when  $r^2 \geq 0.995$ .
  7. The soil matrices of the ECM and ILV were not characterized, and the soil description was not reported as USDA soil texture classification (p. 11 of MRID 49793901; p. 11 of MRID 49793902). It could not be determined if the ILV was provided with the most difficult matrix with which to validate the method.
  8. In the ECM, only one calibration standard produced an instrumental response which was below the instrumental response of the “low” fortification level (Figures 1-15, pp. 25-39 of MRID 49793901). It is preferable to have a minimum of two calibration standards which generate an instrument response below and above the instrument response of the LOQ.
  9. In the ECM and ILV, chromatograms of each analyte were only provided for a control sample, one calibration standard and one for each fortification level (Figures 1-15, pp. 25-39 of MRID 49793901; Figures 1-18, pp. 26-43 of MRID 49793902). A reagent blank was not included in the ECM. In the ECM and ILV, no chromatograms were provided for samples dosed at the ECM LOQ since they were not included in the studies.
  10. The estimations of the LOQ in the ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (pp. 17-18; Appendix 1, pp. 44, 47 of MRID 49793901; pp. 8, 18-20 of MRID 49793902). In the ECM, the LOQ was calculated based on the instrument response of the lowest calibration standard and the dilution factor of the control samples. No calculations for the LOQ were reported in the ILV.

However, the lowest toxicological level of concern in soil was not reported. An LOQ above toxicological level of concern results in an unacceptable method classification.

11. It was reported for the ILV that one set of 22 samples (10 fortified and 5 unfortified samples, 1 reagent blank and 6 solvent standards) required 8 hours (one working day) to complete the preparation and extraction (p. 17 of MRID 49793902). Subsequent LC/MS analysis was performed overnight.

## 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****Aldicarb (Aldicarb PESTANAL®)****IUPAC Name:** (EZ)-2-Methyl-2-(methylthio)propionaldehyde *O*-methylcarbamoyloxime**CAS Name:** 2-Methyl-2-(methylthio)propanal *O*-[(methylamino)carbonyl]oxime**CAS Number:** 116-06-3**SMILES String:** O=C(O\N=C\C(SC)(C)C)NC**Aldicarb-Sulfone (Aldicarb-sulfone PESTANAL®)****IUPAC Name:** Not reported**CAS Name:** Not reported**CAS Number:** 1646-88-4**SMILES String:** Not found**Aldicarb-Sulfoxide (Aldicarb-sulfoxide PESTANAL®)****IUPAC Name:** Not reported**CAS Name:** Not reported**CAS Number:** 1646-87-3**SMILES String:** Not found