

**Analytical method for triadimefon in water**

**Reports:** ECM: EPA MRID No.: 51029803. DeVellis, S.R. 2019. Validation of an Environmental Chemistry Method for the Determination of Triadimefon in Groundwater and Surface Water by LC-MS/MS. Report prepared by Smithers (formerly Smithers Viscient), Wareham, Massachusetts, and sponsored and submitted by ChemStarr, LLC, Irvine, California; 66 pages. Smithers Viscient Study No.: 14181.6108. Final report issued October 7, 2019.

ILV: EPA MRID No.: 51198005. Cashmore, A., and O. Idialu. 2020. Independent Laboratory Validation of Analytical Method 14181.6108 for the Determination of Triadimefon in Water. Report prepared by Smithers ERS Limited, North Yorkshire, United Kingdom, monitored by Pyxis Regulatory Consulting, Inc., Gig Harbor, Washington, and sponsored and submitted by ChemStarr, LLC, Irvine, California; 67 pages. Study No.: 3202454. Final report issued July 9, 2020.

**Document No.:** MRIDs 51029803 & 51198005

**Guideline:** 850.6100


**Statements:** ECM: The study was conducted in compliance with USEPA FIFRA GLP standards (40 CFR Part 160), as accepted by OECD GLP (1998; p. 3 of MRID 51029803). Signed and dated Data Confidentiality, GLP, and Quality Assurance statements were provided (pp. 2-4). An Authenticity statement was included with the Quality Assurance statement.

ILV: The study was conducted in compliance with United Kingdom (1999) GLP standards, as amended by GLP (2004) and OECD GLP (1998), as well as the United Kingdom Department of Health (p. 3; Appendix 6, p. 67 of MRID 51198005). The study was suitable for submission to US FDA, USEPA, and Japanese regulatory authorities. Signed and dated Data Confidentiality, GLP, Quality Assurance, and Authenticity statements were provided (pp. 2-5). An Authenticity statement was also included with the GLP and Quality Assurance statements (pp. 3-4).


**Classification:** This analytical method is classified as **acceptable**. Since the reported method LOQ was not based on scientifically acceptable procedures defined in 40 CFR Part 136, the reported LOQ is the lowest level of method validation (LLMV) rather than LOQ. The ECM adjustment for test material purity caused the ECM LOQ and 10×LOQ fortifications to differ slightly from the ILV LOQ and 10×LOQ fortifications.

**PC Code:** 109901

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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. The CDM/CSS-Dynamac Joint Venture role does not include establishing Agency policies.*

## Executive Summary

The analytical method, Smithers Viscient Study No. 14181.6108, is designed for the quantitative determination of triadimefon in water at the stated LOQ of 0.106 µg/L using LC/MS/MS. The LOQ is less than the lowest toxicological level of concern in water for triadimefon. The ECM Protocol originally stated that the LOQ was 0.10 µg/L, and the ILV was based on the original ECM Protocol. In a Protocol Amendment, the ECM test material purity was corrected which caused the adjusted ECM LOQ. Since the ILV LOQ was slightly less than the ECM LOQ, the ECM LOQ and 10×LOQ was considered to be supported by the ILV performance data at the ILV LOQ and 10×LOQ. Based on the performance data submitted by the ILV and ECM, the LLMV was equivalent to the ECM reported method LOQ for triadimefon in the tested water matrices (0.106 µg/L).

The ECM and ILV validated the method using different groundwater and surface water matrices; only the ECM groundwater matrix was not characterized. The ILV validated the method for triadimefon in water in the first trial with minor modifications to the analytical parameters to improve peak detection. The specificity of the method was supported by the ECM representative chromatograms, so the LC concerns noted in the ILV appeared to be due to the instrument differences. The ILV modifications did not warrant an updated ECM. All ILV and ECM data regarding repeatability, accuracy, precision, linearity, and specificity were satisfactory for triadimefon in test water matrices.

**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						
Triadimefon	51029803 <sup>1</sup>	51198005 <sup>2</sup>		Water	07/10/2019	ChemStarr, LLC	LC/MS/MS	0.106 µg/L (ECM)  0.1 µg/L (ILV) <sup>3</sup>

1 In the ECM, groundwater (uncharacterized, unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-A; pH 6.57, dissolved oxygen concentration 10.22 mg/L) were used in the study (p. 13 of MRID 51029803). The surface water was collected from Taunton River, Bridgewater, Massachusetts, and was characterized by Smithers Viscient.

2 In the ILV, groundwater (CS 13/18 Borehole; pH 8.0, conductivity 436 µS/cm, dissolved organic carbon 0.00 mg/L; hardness 349 mg/L CaCO<sub>3</sub>) and surface water (CS 01/20 Fountains Abbey; pH 7.51, conductivity 140 µS/cm, dissolved organic carbon 8.53 mg/L; hardness 132 mg/L CaCO<sub>3</sub>) were collected by Smithers Viscient ERS (p. 12; Appendix 2, pp. 52-53 of MRID 51198005). The surface water was collected from The Lake, Studley Royal, Ripon, United Kingdom. Water characterization was performed at the ILV.

3 The reported ILV LOQ differed slightly from the reported ECM LOQ (p. 10 of MRID 51029803; p. 10 of MRID 51198005). The difference was due to an ECM adjustment for test material purity (Appendix 1, p. 61 of MRID 51029803; see Reviewer's Comment #1).

## I. Principle of the Method

Water samples (5 mL final volume) were fortified (0.05 mL of 0.0106 or 0.106 mg/L fortification solution) and diluted to 50 mL with acetonitrile:purified reagent water (20:80, v:v; pp. 16-18 of MRID 51029803). The high fortification samples were further diluted by taking 3.00 mL of the sample and diluting to a final volume of 10.0 mL with acetonitrile:test water matrix:purified reagent water (18:10:72, v:v:v). An aliquot was taken for LC/MS/MS analysis.

Samples were analyzed for triadimefon using a Shimadzu LC-20AD HPLC coupled with an AB MDS Sciex 5000 MS with an ESI Turbo V ion source operated in the positive ion mode with multiple reaction monitoring (MRM; pp. 12, 18-19 of MRID 51029803). The following LC conditions were used: Waters XBridge BEH C18 column (2.1 mm x 50 mm, 2.5  $\mu$ m; column temperature 40°C), mobile phase of (A) 0.1% formic acid in water and (B) 0.1% formic acid in acetonitrile [mobile gradient phase of percent A:B (v:v) at 0.50 min. 80.0:20.0, 4.00-5.00 min. 0.00:100.0, 5.10-6.00 min. 80.0:20.0] and injection volume of 100.0  $\mu$ L. MS source temperature was 650°C. Two ion pair transitions were monitored for triadimefon (quantitation and confirmation, respectively):  $m/z$  294.3 $\rightarrow$ 197.1 and  $m/z$  294.3 $\rightarrow$ 69.1. Reported retention time was *ca.* 3.6 minutes for triadimefon.

The ILV performed the ECM methods as written, except for minor modifications to the analytical parameters (pp. 13-17; Appendix 3, pp. 58-60 of MRID 51198005). The LOQ and 10 $\times$ LOQ fortification concentrations differed slightly from those of the ECM, due to an ECM adjustment for test material purity (Appendix 1, p. 61 of MRID 51029803; see Reviewer's Comment #1). Samples were analyzed for triadimefon using Nexera series HPLC coupled with an AB Sciex API 5000 Triple Quadrupole LC/MS/MS. The LC/MS/MS parameters were similar to those of the ECM. The following LC conditions were used: Waters XBridge BEH C18 column (2.1 mm x 50 mm, 2.5  $\mu$ m; column temperature 40°C), mobile phase of (A) 0.1% formic acid in water and (B) 0.1% formic acid in acetonitrile [mobile gradient phase of percent A:B (v:v) at 0.0-0.5 min. 80:20, 3.0-4.0 min. 0:100, 4.1-5.5 min. 80:20] and injection volume of 50  $\mu$ L. MS source temperature was 550°C. Two ion pair transitions were monitored for triadimefon (quantitation and confirmation, respectively):  $m/z$  294.1 $\rightarrow$ 197.6 and  $m/z$  294.1 $\rightarrow$ 69.1. These ion transitions were similar to those of the ECM. Reported retention time was *ca.* 2.4 minutes for triadimefon. The ILV noted that the ramp of the LC gradient was shortened to reduce peak broadening, the flow rate was increased from 0.4 to 0.5 mL to improve peak sensitivity, and the LC column could not be modified. The ILV modifications did not warrant an updated ECM.

The Limit of Quantification (LOQ) for triadimefon in water was 0.106  $\mu$ g/L in the ECM and 0.10  $\mu$ g/L in the ILV (pp. 10, 20-25 of MRID 51029803; pp. 10, 18-23 of MRID 51198005). In the ECM, the Limit of Detection (LOD) for triadimefon was calculated as 0.01  $\mu$ g/L for ground water and surface water. In the ILV, the LOD for triadimefon was calculated as 0.0200-0.0275  $\mu$ g/L for ground water and 0.0074-0.0137  $\mu$ g/L for surface water. Since the LOQ was not based on scientifically acceptable procedures defined in 40 CFR Part 136, the reported LOQ is the lowest level of method validation (LLMV) rather than an LOQ.

## II. Recovery Findings

ECM (MRID 51029803): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD  $\leq$ 20%) for analysis of triadimefon at fortification levels of 0.106  $\mu\text{g/L}$  (LOQ) and 1.060  $\mu\text{g/L}$  (10 $\times$ LOQ) in two water matrices (Tables 1-4, pp. 29-32). Two ion pair transitions were monitored; performance data was comparable between the quantitation and confirmation analyses. The groundwater (uncharacterized, unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-A; pH 6.57, dissolved oxygen concentration 10.22 mg/L) were used in the study (p. 13). The surface water was collected from Taunton River, Bridgewater, Massachusetts, and was characterized by Smithers Viscient.

ILV (MRID 51198005): Mean recoveries and RSDs were within guidelines for analysis of triadimefon at fortification levels of 0.1  $\mu\text{g/L}$  (LOQ) and 1.0  $\mu\text{g/L}$  (10 $\times$ LOQ) in two water matrices (Tables 1-4, pp. 27-30). Two ion pair transitions were monitored; performance data was comparable between the quantitation and confirmation analyses. The groundwater (CS 13/18 Borehole; pH 8.0, conductivity 436  $\mu\text{S/cm}$ , dissolved organic carbon 0.00 mg/L; hardness 349 mg/L  $\text{CaCO}_3$ ) and surface water (CS 01/20 Fountains Abbey; pH 7.51, conductivity 140  $\mu\text{S/cm}$ , dissolved organic carbon 8.53 mg/L; hardness 132 mg/L  $\text{CaCO}_3$ ) were collected by Smithers Viscient ERS (p. 12; Appendix 2, pp. 52-53). The surface water was collected from The Lake, Studley Royal, Ripon, United Kingdom. Water characterization was performed at the ILV. The method for triadimefon in water was validated in the first trial with minor modifications to the analytical parameters to improve peak detection (pp. 10, 21-23; Appendix 4, p. 65). The ILV modifications did not warrant an updated ECM. The LOQ and 10 $\times$ LOQ fortification concentrations differed slightly from those of the ECM, due to an ECM adjustment for test material purity (Appendix 1, p. 61 of MRID 51029803; see Reviewer's Comment #1).

**Table 2. Initial Validation Method Recoveries for Triadimefon in Water<sup>1,2</sup>**

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%) <sup>3</sup>	Relative Standard Deviation (%)
<b>Groundwater</b>						
Quantitation ion transition						
Triadimefon	0.106 (LOQ)	5	103-109	106	2.24	2.11
	1.060	5	104-107	105	1.07	1.02
Confirmation ion transition						
Triadimefon	0.106 (LOQ)	5	102-110	106	2.71	2.55
	1.060	5	103-111	106	2.93	2.75
<b>Surface Water</b>						
Quantitation ion transition						
Triadimefon	0.106 (LOQ)	5	99.6-106	103	2.95	2.87
	1.060	5	95.4-97.2	96.3	0.806	0.837
Confirmation ion transition						
Triadimefon	0.106 (LOQ)	5	104-106	105	0.846	0.808
	1.060	5	96.7-100	98.4	1.26	1.28

Data (uncorrected recovery results; pp. 20-21) were obtained from Tables 1-4, pp. 29-32 of MRID 51029803.

1 The groundwater (uncharacterized, unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-A; pH 6.57, dissolved oxygen concentration 10.22 mg/L) were used in the study (p. 13). The surface water was collected from Taunton River, Bridgewater, Massachusetts, and was characterized by Smithers Visient.

2 Two ion pair transitions were monitored for triadimefon (quantitation and confirmation, respectively):  $m/z$  294.3→197.1 and  $m/z$  294.3→69.1.

**Table 3. Independent Validation Method Recoveries for Triadimefon in Water<sup>1,2,3</sup>**

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
<b>Borehole Groundwater</b>						
Quantitation ion transition						
Triadimefon	0.1 (LOQ)	5	94.0-110	103	7.13	6.90
	1.0	5	88.2-98.1	94.3	3.83	4.06
Confirmation ion transition						
Triadimefon	0.1 (LOQ)	5	98.9-108	104	3.87	3.72
	1.0	5	94.9-99.1	97.5	1.57	1.61
<b>Fountains Abbey Surface Water</b>						
Quantitation ion transition						
Triadimefon	0.1 (LOQ)	5	78.3-97.4	88.9	7.10	7.99
	1.0	5	101-109	104	3.42	3.28
Confirmation ion transition						
Triadimefon	0.1 (LOQ)	5	86.7-93.4	90.7	2.68	2.95
	1.0	5	91.8-109	99.5	6.90	6.93

Data (uncorrected recovery results; p. 18) were obtained from Tables 1-4, pp. 27-30 of MRID 51198005.

1 The groundwater (CS 13/18 Borehole; pH 8.0, conductivity 436 µS/cm, dissolved organic carbon 0.00 mg/L; hardness 349 mg/L CaCO<sub>3</sub>) and surface water (CS 01/20 Fountains Abbey; pH 7.51, conductivity 140 µS/cm, dissolved organic carbon 8.53 mg/L; hardness 132 mg/L CaCO<sub>3</sub>) were collected by Smithers Viscient ERS (p. 12; Appendix 2, pp. 52-53). The surface water was collected from The Lake, Studley Royal, Ripon, United Kingdom. Water characterization was performed at the ILV.

2 Two ion pair transitions were monitored for triadimefon (quantitation and confirmation, respectively): *m/z* 294.1→197.6 and *m/z* 294.1→69.1. These ion transitions were similar to those of the ECM.

### III. Method Characteristics

The LOQ for triadimefon in water was 0.106 µg/L in the ECM and 0.10 µg/L in the ILV (pp. 10, 20-25 of MRID 51029803; pp. 10, 18-23 of MRID 51198005). In the ECM and ILV, the LOQ was defined as the lowest fortification level validated. Also, in the ECM, the LOQ was defined as the level which the blank values did not exceed 30% of the LOQ. In the ECM, the LOD was calculated as 0.01 µg/L for ground water and surface water from the signal-to-noise response of each analyte in matrix at the LOQ level using the following equation:

$$\text{LOD} = (3 \times N_{\text{ctl}}) / (\text{Resp}_{\text{LS}}) \times \text{Conc}_{\text{LS}} \times \text{DF}_{\text{CNTL}}$$

Where, LOD is the limit of detection of the analysis,  $N_{\text{ctl}}$  is the mean signal to noise in height of the control samples (or blanks),  $\text{Resp}_{\text{LS}}$  is the mean response in height of the two low calibration standards,  $\text{Conc}_{\text{LS}}$  is the concentration of the low calibration standard, and  $\text{DF}_{\text{CNTL}}$  is the dilution factor of the control samples (10.0 mL/g).

The LOD for triadimefon in water was estimated in the ILV as 0.0200-0.0275 µg/L for ground water and 0.0074-0.0137 µg/L for surface water at 3 x baseline noise for the primary and confirmatory transitions.

Since the LOQ was not based on scientifically acceptable procedures defined in 40 CFR Part 136, the reported LOQ is the lowest level of method validation (LLMV) rather than an LOQ.



**Table 4. Method Characteristics in Water**

		Triadimefon
Limit of Quantitation (LOQ)*	ECM	0.106 µg/L
	ILV	0.1 µg/L <sup>1</sup>
Limit of Detection (LOD)	ECM (calc)	0.01 µg/L (GW & SW)
	ILV (calc)	0.0200-0.0275 µg/L (GW) 0.0074-0.0137 µg/L (SW)
Linearity (calibration curve r and concentration range)	ECM <sup>2</sup>	r = 0.9995 (Q & C, GW) r = 0.9960 (Q, SW) r = 0.9955 (C, SW)
		0.00530-0.0530 ng/mL
	ILV	r = 0.9972 (Q, GW) r = 0.9976 (C, GW) r = 0.9974 (Q, SW) r = 0.9988 (C, SW)
		0.005-0.05 ng/mL
Repeatable	ECM <sup>3</sup>	Yes at LOQ (0.106 µg/L) and 10×LOQ (1.060 µg/L) (one uncharacterized groundwater and one characterized surface water)
	ILV <sup>4,5</sup>	Yes at LOQ (0.1 µg/L) and 10×LOQ (1.0 µg/L) (one characterized groundwater and one characterized surface water)
Reproducible <sup>6</sup>		Yes for 0.106 µg/L (LLMV)* and 1.060 µg/L in water matrices
Specific	ECM	Yes, matrix interferences were <10% of the LOQ (based on peak area). Minor peak tailing was observed.
	ILV	Yes, no matrix interferences were observed. Minor peak tailing and minor nearby contaminants were observed.

Data were obtained from pp. 10, 20-25 (LOQ/LOD); Tables 1-4, pp. 29-32 (recovery results); p. 23; Figures 11-14, pp. 47-50 (calibration curves); Figures 1-10, pp. 37-46 (chromatograms) of MRID 51029803; pp. 10, 18-23 (LOQ/LOD); Tables 1-4, pp. 27-30 (recovery results); Figures 1-2, pp. 34-35; Figures 15-16, pp. 42-43 (calibration curves); Figures 3-28, pp. 36-49 (chromatograms) of MRID 51198005. Q = quantitation ion transition; C = confirmation ion transition; GW = Groundwater; SW = Surface Water.

\* Since the LOQ was not based on scientifically acceptable procedures defined in 40 CFR Part 136, the reported LOQ is the lowest level of method validation (LLMV) rather than an LOQ. The lowest concentration tested with sufficiently accurate and precise recoveries is the LLMV.

1 The reported ILV LOQ differed slightly from the reported ECM LOQ (p. 10 of MRID 51029803; p. 10 of MRID 51198005). The difference was due to an ECM adjustment for test material purity (Appendix 1, p. 61 of MRID 51029803; see Reviewer's Comment #1).

2 ECM correlation coefficients (r) were reviewer-calculated based on r<sup>2</sup> values reported in the study report (p. 23; Figures 11-14, pp. 47-50 of MRID 51029803; DER Attachment 2). Matrix-matched calibration standards were used in the ECM and ILV (p. 23 of MRID 51029803; p. 24 of MRID 51198005).

3 In the ECM, groundwater (uncharacterized, unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-A; pH 6.57, dissolved oxygen concentration 10.22 mg/L) were used in the study (p. 13 of MRID 51029803). The surface water was collected from Taunton River, Bridgewater, Massachusetts, and was characterized by Smithers Viscient.

4 In the ILV, groundwater (CS 13/18 Borehole; pH 8.0, conductivity 436 µS/cm, dissolved organic carbon 0.00 mg/L; hardness 349 mg/L CaCO<sub>3</sub>) and surface water (CS 01/20 Fountains Abbey; pH 7.51, conductivity 140 µS/cm, dissolved organic carbon 8.53 mg/L; hardness 132 mg/L CaCO<sub>3</sub>) were collected by Smithers Viscient ERS (p. 12; Appendix 2, pp. 52-53 of MRID 51198005). The surface water was collected from The Lake, Studley Royal, Ripon, United Kingdom. Water characterization was performed at the ILV.

5 The ILV validated the method for triadimefon in water in the first trial with minor modifications to the analytical parameters to improve peak detection (pp. 10, 21-23; Appendix 4, p. 65 of MRID 51198005). The ILV modifications did not warrant an updated ECM. The LOQ and 10×LOQ fortification concentrations differed slightly from those of the ECM.

6 Since the ILV LOQ was slightly less than the ECM LOQ, the ECM LOQ and 10×LOQ was considered to be supported by the ILV performance data at the ILV LOQ and 10×LOQ.

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

1. The ECM Protocol originally stated that the LOQ and 10×LOQ validation fortifications were to be 0.100 µg/L and 1.00 µg/L and the test material purity was 93.0% (Appendix 1, pp. 60-61 of MRID 51029803). In order to prepare the stock solutions for the LOQ and 10×LOQ validation fortifications, 0.0539 g was weighed-out which, based on the known purity, would have been equivalent to 0.05 g a.i. yielding a 1000 mg a.i./L primary stock solution concentration (p. 14). However, since the test material purity was subsequently corrected to be 98.4%, the 0.0539 g of test material was corrected as equivalent to 0.0530 g a.i. yielding a 1060 mg a.i./L primary stock solution concentration (p. 14; Appendix 1, pp. 60-61). This ECM adjustment for test material purity caused the ECM LOQ and 10×LOQ fortifications to differ slightly from the ILV LOQ and 10×LOQ fortifications.
2. Since the ILV LOQ was slightly less than the ECM LOQ, the ECM LOQ and 10×LOQ was considered to be supported by the ILV performance data at the ILV LOQ and 10×LOQ.
3. Since the reported method LOQ was not based on scientifically acceptable procedures defined in 40 CFR Part 136, the reported LOQ is the lowest level of method validation (LLMV) rather than an LOQ (pp. 10, 20-25 of MRID 51029803; pp. 10, 18-23 of MRID 51198005). The lowest concentration tested with sufficiently accurate and precise recoveries is the LLMV. Based on the performance data submitted by the ILV and ECM, the LLMV was equivalent to the ECM reported method LOQ for triadimefon in the tested water matrices (0.106 µg/L).
4. The ILV performed the ECM methods as written, except for minor modifications to the analytical parameters (pp. 13-17; Appendix 3, pp. 58-60 of MRID 51198005). The ILV noted that the ramp of the LC gradient was shortened to reduce peak broadening, the flow rate was increased from 0.4 to 0.5 mL to improve peak sensitivity, and the LC column could not be modified. The ILV modifications did not warrant an updated ECM; analytical instrument optimization is expected (p. 19 of MRID 51029803). The specificity of the method was supported by the ECM representative chromatograms, so the LC concerns noted in the ILV appeared to be due to the instrument differences (Figures 1-10, pp. 37-46 of MRID 51029803).
5. The ECM groundwater matrix was not characterized (p. 13 of MRID 51029803).
6. The communications between the ILV study authors (Angela Cashmore and Ofure Idialu, Smithers ERS Limited) and ILV Study Monitor (Janelle Kay, Pyxis Regulatory Consulting, Inc.) were summarized (pp. 1, 10, 24; Appendix 5, p. 66 of MRID 51198005). Reported communications included: protocol issue and the results of the first attempt of the ILV.
7. The reviewer noted that the ECM and ILV laboratories were part of the same company, Smithers (formerly Smithers Viscient) and Smithers ERS Limited, respectively (pp. 1, 5-6 of MRID 51029803; pp. 1, 6 of MRID 51198005). The laboratory location, personnel and equipment differed between the two laboratories. The only exchange of information was the ECM Method/Protocol (by the ECM study authors) provided to the ILV via the Sponsor Representative (Appendix 3, pp. 54-64 of MRID 51198005).

8. The determinations of the LOD and LOQ in the ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (pp. 10, 20-25 of MRID 51029803; pp. 10, 18-23 of MRID 51198005). In the ECM and ILV, the LOQ was defined as the lowest fortification level validated. Also, in the ECM, the LOQ was defined as the level which the blank values did not exceed 30% of the LOQ. No further justification of the LOQ was reported in the ECM or ILV. The LOD was calculated in the ECM using the following equation:  $LOD = (3 \times N_{ctl}) / (Res_{PLS}) \times Conc_{CLS} \times DF_{CNTL}$ , where, LOD is the limit of detection of the analysis,  $N_{ctl}$  is the mean noise in height of the control samples (or blanks),  $Res_{PLS}$  is the mean response in height of the two low calibration standards,  $Conc_{CLS}$  is the concentration of the low calibration standard, and  $DF_{CNTL}$  is the dilution factor of the control samples (10.0 mL/g). The LOD was estimated in the ILV using the following equation: 3x baseline noise for the primary and confirmatory transitions. Detection limits should not be based on the arbitrarily selected lowest concentration in the spiked samples.

Since the LOQ was not based on scientifically acceptable procedures defined in 40 CFR Part 136, the reported LOQ is the lowest level of method validation (LLMV) rather than an LOQ.

The method detection limit (MDL) was calculated in the ECM and ILV as dependent upon the lowest concentration calibration standard and the dilution factor of the controls (p. 21 of MRID 51029803; p. 21 of MRID 51198005). In the ECM, the MDL was equivalent to  $0.0053 \mu\text{g/L} \times 10.0 \text{ mL/g} = 0.0530 \mu\text{g/L}$ ; in the ILV, the MDL was equivalent to  $0.05 \mu\text{g/L}$  for triadimefon ( $0.005 \mu\text{g/L} \times 10.0 \text{ mL/g}$ ). This MDL calculation was not in accordance with the EPA Definition and Procedure for the Determination of the Method Detection Limit, Revision 2 (2016).

9. The matrix interferences were determined to be insignificant (<20%) in the ECM and ILV; however, matrix-matched calibration standards were used in the ECM and ILV (pp. 22-25; Tables 5-8, pp. 33-36 of MRID 51029803; pp. 21-24 of MRID 51198005).
10. The total time required to complete one set of samples was not reported in the ECM or ILV.

## 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, and Revision 2; 1994 and 2016.

**Attachment 1: Chemical Names and Structures****Triadimefon**

**IUPAC Name:** (RS)-1-(4-chlorophenoxy)-3,3-dimethyl-1-(1H-1,2,4-triazol-1-yl)butan-2-one

**CAS Name:** 1-(4-Chlorophenoxy)-3,3-dimethyl-1-(1H-1,2,4-triazol-1-yl)-2-butanone

**CAS Number:** 43121-43-3

**SMILES String:** Clc1ccc(OCC(C)(C)C(=O)C2=CN=CN=C2)cc1

