

Analytical method for cumyluron in water

Reports: ECM: EPA MRID No.: 51301803. Smith, R.J. 2020. Environmental Chemistry Method: Validation of the Analytical Method for the Determination of Cumyluron in Ground Water and Surface Water by LC-MS/MS. Report prepared by Smithers, Wareham, Massachusetts, sponsored by Marubeni Corporation, Tokyo, Japan, and submitted by Helena Chemical Company (Agent), Collierville, Tennessee and Marubeni America Corporation, New York, New York; 54 pages. Smithers Viscient Study No.: 14102.6120. Final report issued May 18, 2020.

ILV: EPA MRID No.: 51301804. Cashmore, A., and O. Idialu. 2020. Independent Laboratory Validation of Analytical Method 14102.6120 for the Determination of Cumyluron in Water. Report prepared by Smithers ERS Limited, North Yorkshire, United Kingdom, sponsored by Marubeni Corporation, Tokyo, Japan, monitored by Wagner Regulatory Associates, Hockessin, Delaware, and submitted by Helena Chemical Company (Agent), Collierville, Tennessee and Marubeni America Corporation, New York, New York; 78 pages. Study No.: 3202655. Final report issued September 22, 2020.

Document No.: MRIDs 51301803 & 51301804

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 51301803). 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 (p. 4).

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. 69 of MRID 51301804). 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. 2-4). An Authenticity statement was also included with the GLP and Quality Assurance statements (pp. 3-4).

Classification: This analytical method is classified as **acceptable**. The ECM method should be updated with the ILV study recommendations of the use of disposable glassware, since contamination in the control samples prevented the initial validation of the method in surface water by the ILV laboratory.

PC Code: 027902

EFED Final Reviewer: He Zhong, Ph.D.
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Environmental Scientist

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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. 14102.6120, is designed for the quantitative determination of cumyluron in water at the stated LOQ of 0.100 µg/L using LC-MS/MS. The LOQ is less than 11.6 µg/L, the lowest toxicological level of concern in water for cumyluron.

The ECM and ILV validated the method using different groundwater and surface water matrices; all test matrices were characterized. The ILV validated the method for cumyluron in water with insignificant modifications to the analytical parameters. The validation for groundwater analysis was acceptable in the first trial while the surface water analysis was acceptable in the second trial. The first trial of the surface water failed due to unacceptable interference in the control samples which was suspected to be due to contamination of the dilution solvent. The groundwater samples were reinjected due to poor instrument precision on the first injection. The re-injection is not considered an additional attempt. The ILV modifications did not warrant an updated ECM; however, the ILV recommended the use of disposable glassware due to the contamination seen in the calibration standards of both test soils. Therefore, the ECM method should be updated with the ILV study recommendations of the use of disposable glassware, since the contamination in the control samples prevented the initial validation of the method in surface water by the ILV laboratory.

All ILV and ECM data regarding repeatability, accuracy, precision, linearity, and specificity were satisfactory for cumyluron in all test water matrices.

Table 1. Analytical Method Summary

Analyte(s) by Pesticide	MRID		Matrix	Method Date (dd/mm/yyyy)	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation					
Cumyluron	51301803 ¹	51301804 ²	Water	18/05/2020	Marubeni Corporation	LC-MS/MS	0.100 µg/L

1 In the ECM, groundwater (Sample ID: Groundwater 2019; pH 7.6, hardness 92 mg equivalent to CaCO₃/L, conductivity 0.70 mmhos/cm, total dissolved solids 228 ppm) collected as unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-A; pH 7.3, hardness 33 mg equivalent to CaCO₃/L, conductivity 0.38 mmhos/cm, total dissolved solids 10 ppm) collected from the Taunton River were used in the study (pp. 11-12 of MRID 51301803). The test waters were characterized by Agvise Laboratories, Northwood, North Dakota.

2 In the ILV, groundwater (CS38/20 Borehole; pH 8.4, conductivity 631 µS/cm, dissolved organic carbon 3.68 mg/L; hardness 312 mg/L CaCO₃) and surface water (CS01/20 Fountains Abbey; pH 7.51, conductivity 140 µS/cm, dissolved organic carbon 8.53 mg/L; hardness 140 mg/L CaCO₃) were collected by Smithers Viscient ERS (p. 12; Appendix 2, pp. 54-55 of MRID 51301804). The surface water was collected from The Lake, Studley Royal, Ripon, United Kingdom. Water characterization was performed at the ILV.

I. Principle of the Method

Water samples (5.00 mL final volume) were fortified (0.05 mL or 0.5 mL of 0.01 mg/L fortification solution) and adjusted to pH 2 (measured with pH paper) with 0.0200 mL of phosphoric acid (pp. 14-16 of MRID 51301803). Samples were diluted to a final volume of 50.0 mL with acetonitrile:purified reagent water (20:80, v:v). After dilution, the samples were vortexed for 15 seconds then centrifugation at 13000 rpm for 5 minutes prior to analysis using LC/MS/MS.

Samples were analyzed for cumyluron using a Shimadzu LC-20AD HPLC coupled with an AB MDS Sciex API 4000 QTrap MS with an ESI Turbo V ion source operated in the positive ion mode with multiple reaction monitoring (MRM; pp. 11, 16-17 of MRID 51301803). The following LC conditions were used: Water XBridge BEH C18 column (2.1 x 50 mm, 2.5 μ m; column temperature 40°C), mobile phase of (A) 0.1% formic acid in reagent grade water and (B) 0.1% formic acid in acetonitrile [mobile gradient phase of percent A:B (v:v) at 0.01-0.50 min. 75.0:25.0, 4.00-6.00 min. 0.00:100, 6.10-7.50 min. 75.0:25.0] and injection volume of 50.0 μ L. MS source temperature was 550°C. Two ion pair transitions were monitored for cumyluron (quantitation and confirmation, respectively): m/z 303.0 \rightarrow 184.9 and m/z 303.0 \rightarrow 125.0. Reported retention time was *ca.* 3.6 minutes for cumyluron.

The ILV performed the ECM method (Smithers Viscient Method No. 14102.6120) as written, except for insignificant modifications to the analytical parameters (pp. 13-17; Appendix 3, pp. 56-65 of MRID 51301804). Samples were analyzed for cumyluron using Shimadzu Nexera series HPLC coupled with an AB Sciex API 5000 Triple Quadrupole LC-MS/MS. The LC-MS/MS parameters were the same as those of the ECM, with the exception of some minor MS parameters. Two ion pair transitions were monitored for cumyluron (quantitation and confirmation, respectively): m/z 303.0 \rightarrow 185.3 and m/z 303.0 \rightarrow 125.2. These ion transitions were similar to those of the ECM. Reported retention time was *ca.* 3.0 minutes for cumyluron. The ILV noted that the LC column and mobile phase solvents could not be modified. The ILV modifications did not warrant an updated ECM.

The Limit of Quantification (LOQ) for cumyluron in water was 0.100 μ g/L in the ECM and ILV (pp. 9, 20-24 of MRID 51301803; pp. 10, 18-21, 23-25 of MRID 51301804). In the ECM, the Limit of Detection (LOD) for cumyluron was calculated as 0.002-0.006 μ g/L for groundwater and 0.002-0.003 μ g/L for surface water. In the ILV, the LOD for cumyluron was calculated as 0.00221-0.00283 μ g/L for groundwater and 0.00472-0.00538 μ 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 51301803): Mean recoveries and relative standard deviations (RSDs) were within guidelines (mean 70-120%; RSD \leq 20%) for analysis of cumyluron at fortification levels of 0.100 μ g/L (LOQ) and 1.00 μ 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. Groundwater (Sample ID: Groundwater 2019; pH 7.6, hardness 92 mg equivalent to CaCO₃/L, conductivity 0.70 mmhos/cm, total dissolved solids 228 ppm) collected as unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-

A; pH 7.3, hardness 33 mg equivalent to CaCO₃/L, conductivity 0.38 mmhos/cm, total dissolved solids 10 ppm) collected from the Taunton River were used in the study (pp. 11-12). The test waters were characterized by Agvise Laboratories, Northwood, North Dakota.

ILV (MRID 51301804): Mean recoveries and RSDs were within guidelines for analysis of cumyluron at fortification levels of 0.1 µg/L (LOQ) and 1.0 µg/L (10×LOQ) in two water matrices (Tables 1-4, pp. 28-31). Two ion pair transitions were monitored; performance data was comparable between the quantitation and confirmation analyses. The groundwater (CS38/20 Borehole; pH 8.4, conductivity 631 µS/cm, dissolved organic carbon 3.68 mg/L; hardness 312 mg/L CaCO₃) and surface water (CS01/20 Fountains Abbey; pH 7.51, conductivity 140 µS/cm, dissolved organic carbon 8.53 mg/L; hardness 140 mg/L CaCO₃) were collected by Smithers Viscient ERS (p. 12; Appendix 2, pp. 54-55). The surface water was collected from The Lake, Studley Royal, Ripon, United Kingdom. Water characterization was performed at the ILV. The method for cumyluron in water was validated with insignificant modifications to the analytical parameters (pp. 13-17, 20-22). The ILV modifications did not warrant an updated ECM. The validation for groundwater analysis was acceptable in the first trial while the surface water analysis was acceptable in the second trial (pp. 20-22; Appendix 4, p. 67). The first trial of the surface water failed due to unacceptable interference in the control samples which was suspected to be due to contamination of the dilution solvent. The groundwater samples were re-injected due to poor instrument precision on the first injection. The re-injection is not considered an additional attempt. However, the ILV recommended the use of disposable glassware due to the contamination seen in the control samples of the first validation attempt of surface water (p. 22). Therefore, the ECM method should be updated with the ILV study recommendations of the use of disposable glassware due to the contamination seen in the surface water control samples, since the contamination in the control samples prevented the validation of the method in surface water by the ILV laboratory.

Table 2. Initial Validation Method Recoveries for Cumyluron in Water^{1,2}

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Groundwater						
Quantitation ion transition						
Cumyluron	0.100 (LOQ)	5	94.4-106	102	4.98	4.90
	1.00	5	102-105	104	1.36	1.31
Confirmation ion transition						
Cumyluron	0.100 (LOQ)	5	99.5-106	102	2.69	2.65
	1.00	5	102-106	104	1.42	1.36
Surface Water						
Quantitation ion transition						
Cumyluron	0.100 (LOQ)	4 ³	102-113	107	5.09	4.79
	1.00	5	103-106	104	1.26	1.22
Confirmation ion transition						
Cumyluron	0.100 (LOQ)	4 ³	99.7-112	106	6.65	6.29
	1.00	5	98.2-103	101	1.95	1.93

Data (uncorrected recovery results; pp. 18-19) were obtained from Tables 1-4, pp. 27-30 of MRID 51301803.

¹ In the ECM, groundwater (Sample ID: Groundwater 2019; pH 7.6, hardness 92 mg equivalent to CaCO₃/L, conductivity 0.70 mmhos/cm, total dissolved solids 228 ppm) collected as unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-A; pH 7.3, hardness 33 mg equivalent to CaCO₃/L,

conductivity 0.38 mmhos/cm, total dissolved solids 10 ppm) collected from the Taunton River were used in the study (pp. 11-12 of MRID 51301803). The test waters were characterized by Agvise Laboratories, Northwood, North Dakota.

2 Two ion pair transitions were monitored for cumyluron (quantitation and confirmation, respectively): m/z 303.0→184.9 and m/z 303.0→125.0.

3 The recovery for one replicate was reported as <0.0200 µg/L; therefore, this sample was omitted.

Table 3. Independent Validation Method Recoveries for Cumyluron in Water^{1,2}

Analyte	Fortification Level (µg/L)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Borehole Groundwater						
Quantitation ion transition						
Cumyluron	0.1 (LOQ)	5	91.6-98.4	95.0	3.22	3.39
	1.0	5	95.7-98.0	96.7	0.945	0.978
Confirmation ion transition						
Cumyluron	0.1 (LOQ)	5	92.6-103	96.4	3.97	4.12
	1.0	5	96.1-99.7	98.8	1.50	1.52
Fountains Abbey Surface Water						
Quantitation ion transition						
Cumyluron	0.1 (LOQ)	5	92.5-102	97.4	4.00	4.11
	1.0	5	94.5-107	99.5	4.60	4.63
Confirmation ion transition						
Cumyluron	0.1 (LOQ)	5	94.0-104	98.9	4.08	4.13
	1.0	5	93.5-104	99.4	4.79	4.82

Data (uncorrected recovery results; p. 18) were obtained from Tables 1-4, pp. 28-31 of MRID 51301804.

1 In the ILV, groundwater (CS38/20 Borehole; pH 8.4, conductivity 631 µS/cm, dissolved organic carbon 3.68 mg/L; hardness 312 mg/L CaCO₃) and surface water (CS01/20 Fountains Abbey; pH 7.51, conductivity 140 µS/cm, dissolved organic carbon 8.53 mg/L; hardness 140 mg/L CaCO₃) were collected by Smithers Visient ERS (p. 12; Appendix 2, pp. 54-55 of MRID 51301804). 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 cumyluron (quantitation and confirmation, respectively): m/z 303.0→185.3 and m/z 303.0→125.2. These ion transitions were similar to those of the ECM.

III. Method Characteristics

The LOQ for cumyluron in water was 0.100 µg/L in the ECM and ILV (pp. 9, 20-24 of MRID 51301803; pp. 10, 18-21, 23-25 of MRID 51301804). 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 0.002-0.006 µg/L for groundwater and 0.002-0.003 µg/L for 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_{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, Conc_{LS} is the concentration of the low calibration standard, and DF_{CNTL} is the dilution factor of the control samples (smallest dilution factor used, i.e., 10.0).

The LOD for cumyluron in water was estimated in the ILV as 0.00221-0.00283 µg/L for groundwater and 0.00472-0.00538 µg/L for surface water at 3 x height of control baseline noise x control sample dilution factor x calibration standard concentration (µg/L) / height of calibration standard peak.

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

		Cumyluron
Limit of Quantitation (LOQ)*	ECM	0.100 µg/L
	ILV	
Limit of Detection (LOD)	ECM (calc)	0.002 µg/L (Q, GW) 0.006 µg/L (C, GW) 0.002 µg/L (Q, SW) 0.003 µg/L (C, SW)
	ILV (calc)	0.00283 µg/L (Q, GW) 0.00221 µg/L (C, GW) 0.00538 µg/L (Q, SW) 0.00472 µg/L (C, SW)
Linearity (calibration curve r and concentration range)	ECM ^{1,2}	r = 1.00 (Q & C, GW) r = 1.00 (Q & C, SW)
	ILV ²	r = 0.9993 (Q, GW) r = 0.9996 (C, GW) r = 0.9971 (Q, SW) r = 0.9988 (C, SW)
	Range	0.002-0.2 µg/L
Repeatable	ECM ³	Yes at LOQ (0.100 µg/L) and 10×LOQ (1.00 µg/L) (one characterized groundwater and one characterized surface water)
	ILV ^{4,5}	Yes at LOQ (0.1 µg/L) and 10×LOQ (1.0 µg/L) (one characterized groundwater and one characterized surface water)
Reproducible		Yes for 0.100 µg/L (LLMV)* and 1.00 µg/L in water matrices
Specific	ECM	Yes, matrix interferences were <5% of the LOQ (based on peak area).
	ILV	

Data were obtained from pp. 9, 20-24 (LOQ/LOD); Tables 1-4, pp. 27-30 (recovery results); p. 20; Figures 10-11, pp. 42-43 (calibration curves); Figures 1-9, pp. 33-41 (chromatograms) of MRID 51301803; pp. 10, 18-21, 23-25 (LOQ/LOD); Tables 1-4, pp. 28-31 (recovery results); p. 20; Figures 1-2, pp. 35-36; Figures 15-16, pp. 43-44 (calibration curves); Figures 3-28, pp. 37-50 (chromatograms) of MRID 51301804. 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 ECM correlation coefficients (r) were reviewer-calculated based on r² values reported in the study report (p. 20; Figures 10-11, pp. 42-43 of MRID 51301803; DER Excel Attachment). Rules of significant figures were followed.
- 2 In the ECM and ILV, matrix effects were insignificant (<±20%) for all soils; therefore, solvent-based calibration standards were used for all soils (p. 20; Tables 5-6, pp. 31-32 of MRID 51301803; p. 21; Tables 5-6, pp. 32-33 of MRID 51301804).
- 3 In the ECM, groundwater (Sample ID: Groundwater 2019; pH 7.6, hardness 92 mg equivalent to CaCO₃/L, conductivity 0.70 mmhos/cm, total dissolved solids 228 ppm) collected as unadulterated water from a 100-meter bedrock well) and surface water (SMV Lot No. 05Feb19Wat-A; pH 7.3, hardness 33 mg equivalent to CaCO₃/L, conductivity 0.38 mmhos/cm, total dissolved solids 10 ppm) collected from the Taunton River were used in the study (pp. 11-12 of MRID 51301803). The test waters were characterized by Agvise Laboratories, Northwood, North Dakota.
- 4 In the ILV, groundwater (CS38/20 Borehole; pH 8.4, conductivity 631 µS/cm, dissolved organic carbon 3.68 mg/L; hardness 312 mg/L CaCO₃) and surface water (CS01/20 Fountains Abbey; pH 7.51, conductivity 140 µS/cm, dissolved organic carbon 8.53 mg/L; hardness 140 mg/L CaCO₃) were collected by Smithers Viscient ERS (p. 12; Appendix 2, pp. 54-55 of MRID 51301804). 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 cumyluron in water with insignificant modifications to the analytical parameters (pp. 13-17, 20-22 of MRID 51301804). The validation for groundwater analysis was acceptable in the first trial while the surface water analysis was acceptable in the second trial (pp. 20-22; Appendix 4, p. 67). The first trial of the

surface water failed due to unacceptable interference in the control samples which was suspected to be due to contamination of the dilution solvent. The groundwater samples were reinjected due to poor instrument precision on the first injection. The re-injection is not considered an additional attempt. The ILV modifications did not warrant an updated ECM; however, the ILV recommended the use of disposable glassware due to the contamination seen in the control samples of the first validation attempt of surface water (p. 22). Therefore, the ECM method should be updated with the ILV study recommendations of the use of disposable glassware due to the contamination seen in the surface water control samples, since the contamination in the control samples prevented the validation of the method in surface water by the ILV laboratory.

IV. Method Deficiencies and Reviewer's Comments

1. 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. 9, 20-24 of MRID 51301803; pp. 10, 18-21, 23-25 of MRID 51301804). 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 cumyluron in the tested water matrices (0.100 µg/L).
2. The ECM method should be updated with the ILV study recommendations of the use of disposable glassware due to contamination seen in the surface water control samples, since the contamination in the control samples prevented the validation of the method in surface water by the ILV laboratory (p. 22 of MRID 51301804).
3. The communications between the ILV study authors (Angela Cashmore and Ofure Idialu, Smithers ERS Limited) and ILV Study Monitor (James Wagner, Wagner Regulatory Associates) were not summarized (p. 1; Appendix 5, p. 68 of MRID 51301804). Reported communications included: protocol issue, the relaying of ILV instrument performance issues to the Sponsor, and the results of the ILV validations in surface water and groundwater.
4. 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 51301803; pp. 1, 6 of MRID 51301804). The laboratory location, personnel and equipment differed between the two laboratories. The only exchange of information was the ECM Method/Protocol provided to the ILV via the Sponsor Representative (Appendix 3, pp. 56-65; Appendix 5, p. 68 of MRID 51301804).
5. The determinations of the LOQ in the ECM and ILV were not based on scientifically acceptable procedures as defined in 40 CFR Part 136 (pp. 9, 20-24 of MRID 51301803; pp. 10, 18-21, 23-25 of MRID 51301804). 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: $LOD = (3x(N_{ctl}) / (Res_{PLS}) \times Conc_{LS} \times DF_{CNTL})$, 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, $Conc_{LS}$ is the concentration of the low calibration standard, and DF_{CNTL} is the dilution factor of the control samples (smallest dilution factor used, i.e., 10.0). The LOD was estimated in the ILV using the following equation: 3 x 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 (pp. 19-20 of MRID 51301803; p. 19 of MRID 51301804). In the ECM, the MDL was equivalent to $0.002 \mu\text{g/L} \times 10.0 = 0.0200 \mu\text{g/L}$; in the ILV, the MDL was also equivalent to $0.02 \mu\text{g/L}$ for cumyluron ($0.002 \mu\text{g/L} \times 10$). This MDL calculation was not in accordance with the EPA Definition and Procedure for the Determination of the Method Detection Limit, Revision 2 (2016).

6. The total time required to complete one set of thirteen samples was reported in the ILV as one working day (8 hours; p. 13 of MRID 51301804).

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.
- USEPA. 2012. *Environmental Chemistry Method Guidance*. Memorandum From D. Brady to Environmental Fate and Effects Division. December 20, 2012. Environmental Fate and Effects Division. Office of Pesticide Programs. Office of Chemical Safety and Pollution Prevention. U.S. Environmental Protection Agency. Available at: <https://www.epa.gov/pesticide-science-and-assessing-pesticide-risks/environmental-chemistry-methods-guidance-pesticides>.
- 40 CFR Part 136. Appendix B. Definition and Procedure for the Determination of the Method Detection Limit-Revision 1.11, pp. 344-347, and Revision 2; 2015 and 2016.

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

IUPAC Name: 1-(2-Chlorobenzyl)-3-(1-methyl-1-phenylethyl)urea
CAS Name: Not reported
CAS Number: 99485-76-4
SMILES String: Not found

