

Analytical method for etridiazole and its metabolites etridiazole acid and 3-DCMT (DCE) in soil

Reports: ECM: EPA MRID No. 50534504. DeVellis, S.R. 2018. Validation of the Analytical Method for the Determination of Etridiazole and its Metabolites in Soil Matrices by LC-MS/MS and GC-MS. Smithers Viscient Study No.: 14088.6158. Sponsor Study No.: 2017-252. Report prepared by Smithers Viscient, Wareham, Massachusetts; sponsored and submitted by MacDermid Agricultural Solutions, Inc., c/o Arysta LifeScience North America, LLC, Cary, North Carolina; 139 pages. Final report issued January 9, 2018.

ILV: EPA MRID No. 50584602. Cashmore, A. 2018. Etridiazole – Independent Laboratory Validation of Analytical Method 14088.6158 for the Determination of Etridiazole and its metabolites Etridiazole acid and DCE in Soil. Smithers Viscient (ESG) Ltd. Study No.: 3202057 and Document No.: 2017-357. Report prepared by Smithers Viscient (ESG) Ltd., North Yorkshire, United Kingdom; sponsored and submitted by MacDermid Agricultural Solutions, Inc., c/o Arysta LifeScience North America, LLC, Cary, North Carolina; 108 pages. Final report issued May 9, 2018 (pp. 2-5).

Document No.: MRIDs 50534504 & 50584602

Guideline: 850.6100

Statements: ECM: The study was conducted in accordance with the USEPA FIFRA (40 CFR Part 160) and OECD Good Laboratory Practices (GLP; p. 3 of MRID 50534504). Signed and dated No Data Confidentiality, GLP and Quality Assurance statements were provided (pp. 2-4). A certification of the authenticity of the report was included with the QA statement.

ILV: The study was conducted in accordance with the UK GLP and OECD GLP and was in compliance with the GLP regulation and are suitable for submission to the US FDA/EPA/Japanese regulatory authorities (p. 3; Appendix 3, p. 105 of MRID 50584602). Signed and dated No Data Confidentiality, GLP, Authenticity, and Quality Assurance statements were provided (pp. 2-5; Appendix 3, p. 105).

Classification: This analytical method is classified as not acceptable. To upgrade the study classification, an updated ECM should be submitted which includes ILV modifications. Soil extract stability data should be provided for 3-DCMT to support the ECM. ILV analysis was not satisfactory for etridiazole, 3-DCMT or 3-Carb-T (etridiazole acid) analysis. ECM linearity was not satisfactory for 3-DCMT analysis. The specificity of the method was not supported for 3-Carb-T based on ILV representative chromatograms. The suitability of the ILV matrices could not be determined.

PC Code: 084701

EFED Final Reviewer: Cheryl Sutton, Ph.D.
Environmental Scientist

Signature:
Date: October 24, 2018

CDM/CSS-Dynamac JV Reviewers: Teresa Nelis, M.S.,
Environmental Scientist

Signature: 
Date: 9/28/18

Reviewers: Lisa Muto, M.S.,

Signature: 

Environmental Scientist

Date: 9/28/18

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

This analytical method, Smithers Viscient Study No. 14088.6158, is designed for the quantitative determination of etridiazole and its metabolite 3-DCMT (DCE) in soil at the LOQ of 50 µg/kg using GC-MS/EI, and metabolite 3-Carb-T (etridiazole acid) in soil at the LOQ of 50 µg/kg using LC-MS/MS. The ECM validated the method for the three analytes using one characterized clay loam and one characterized silt loam soil matrices, and the ILV validated the method using one characterized sandy loam and one characterized silt loam soil matrices. In the ECM and ILV, three ions were monitored for etridiazole and 3-DCMT, and two ion transitions were monitored for 3-Carb-T analysis. **An updated ECM should be submitted which includes ILV modifications:** the use of glass pipets instead of positive displacement pipets (plastic) for handling samples, the use of fresh soil extracts for 3-DCMT analysis due to stability issues, and the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks. The ILV validated the ECM method for the quantitation and confirmation analyses of etridiazole and 3-DCMT (10×LOQ only) in silt loam soil in the first trial; the ILV validated the ECM method for the quantitation and confirmation analyses of etridiazole and 3-DCMT in sandy loam soil, 3-DCMT at the LOQ in silt loam soil, and 3-Carb-T in sandy loam and silt loam soil matrices in the second trial. The ILV modifications were necessary for all trials to succeed. The ILV also noted that 3-DCMT extracts may be unstable in stored extracts; **soil extract stability data should be provided for 3-DCMT to support the ECM.** All ECM and ILV data was satisfactory regarding accuracy and precision for all analytes. ECM and ILV data was satisfactory regarding specificity for all analytes, except for 3-Carb-T ILV chromatograms, which showed multiple significant contaminants in all Q chromatograms, including the reagent blank, controls, and calibration standards. ILV linearity was not satisfactory for etridiazole, 3-DCMT or 3-Carb-T analysis in at least one matrix. ECM linearity was satisfactory for all analytes, except 3-DCMT analysis in silt loam soil.

Table 1. Analytical Method Summary.

Analyte(s) by Pesticide	MRID		EPA Review	Matrix	Method Date	Registrant	Analysis	Limit of Quantitation (LOQ)
	Environmental Chemistry Method	Independent Laboratory Validation						
Etridiazole	50534504	50584602		Soil ^{1,2}	01/09/2018	MacDermid Agricultural Solutions, Inc. c/o Arysta LifeScience North America, LLC	GC/MS	50.0 µg/kg
3-DCMT (DCE)							LC/MS/MS	
3-Carb-T								

1 In the ECM, clay loam soil (SMV Lot No. 12DEC16SOIL-B; 40/28/32 sand/silt/clay, pH 5.4 in 1:1 soil:water ratio, 5.6% organic matter) obtained from Grand Forks, North Dakota, and silt loam soil (SMV Lot No. 13SEP17SOIL-B; 24/60/16 sand/silt/clay), pH 7.2 in 1:1 soil:water ratio, 3.9% organic matter Walkley-Black) obtained from Jackson, Iowa, were used (USDA soil texture classification not specified; pp. 18-19 of MRID 50534504). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota.

2 In the ILV, sandy loam soil (CS 27/16 Speyer 5M; 59/30/11 sand/silt/clay, pH 8.5 in 1:5 soil:water ratio, 1.0% organic carbon) was obtained from Rheinland-Pfalz, Germany, and supplied by LUFA Speyer, and silt loam soil (CS 30/16 Brierlow; 26/58/16 sand/silt/clay, pH 6.4 in 1:5 soil:water ratio, 2.5% organic carbon) was obtained from Derbyshire, UK, and supplied by LRA Labsoil (USDA soil texture classification; p. 15; Appendix 2, pp. 103-104 of MRID 50584602). Soil was characterized by Smithers Viscient (ESG), North Yorkshire, UK, except some of the analyses on the sandy loam soil was characterized by LUFA Speyer.

I. Principle of the Method

Samples (5.00 g dry weight) were transferred to 45-mL glass vials with teflon-lined caps and fortified with 0.0250 mL or 0.250 mL of the fortification solutions (10.0 mg/L) of etridiazole and 3-DCMT in acetonitrile to prepare LOQ (50 µg/kg) and 10×LOQ (500 µg/kg) fortified samples, respectively; samples (*ca.* 5.00 g dry weight) were transferred to 50-mL Nalgene centrifuge tubes and fortified with 0.250 mL of the fortification solutions (1.00 or 10.0 mg/L) of 3-Carb-T in acetonitrile to prepare LOQ (50 µg/kg) and 10×LOQ, fortified samples, respectively (pp. 20-21, 24-26 of MRID 50534504). The soil samples fortified with etridiazole and 3-DCMT were extracted with 30.0 mL of dichloromethane:acetone (75:25, v:v) and placed on a shaker table (150 rpm for 30 minutes) (pp. 26-27 of MRID 50534504). After centrifugation (1200 rpm for 15 minutes), a portion of each extract was concentrated under a gentle stream of nitrogen to incipient dryness (*ca.* 100 µL) and reconstituted with internal standard diluent to a volume of 10.0 mL for the LOQ and 10×LOQ.

The soil samples (5.00 g dry weight) fortified with 3-CarbT in 50-mL Nalgene centrifuge tubes were extracted with 20.0 mL of acetonitrile:purified reagent water (20:80, v:v) and placed on a shaker table (150 rpm for 30 minutes) (pp. 27-29 of MRID 50534504). After centrifugation (3000 rpm for 10 minutes), extracts were transferred to 50.0-mL volumetric flasks, and the extraction and centrifugation procedure was repeated with an additional 20.0 mL of acetonitrile:purified reagent water (20:80, v:v). The extracts were combined to a total volume of 50.0 mL, and 5.00 mL of the final extract was further processed by solid phase extraction. The 5.00 mL aliquots received 1.0 µL ammonium hydroxide, were loaded onto Oasis Mixed-Mode Strong Anion Exchange (MAX) SPE columns (60 mg, 3 mL, pre-conditioned with two column volumes of methanol and two column

volumes of purified reagent water) and allowed to flow through under vacuum (1 drop/sec. flow) (pp. 27-29 of MRID 50534504). Each sample vessel and column were rinsed with 5.00 mL of purified reagent water, the rinsate loaded onto the column, allowed to flow under vacuum at 1 drop/sec., and then rinsed in a similar manner with 5.00 mL of methanol, and the rinsates discarded. The columns were quickly dried under full vacuum, and the test substance was eluted from the SPE columns with 3.00 mL of 2% trifluoroacetic acid in methanol under vacuum at 1 drop/sec. and collected into glass conical vials. The samples were concentrated to incipient dryness under a gentle stream of nitrogen at 50.0°C. The residue was reconstituted to 5.00 mL with 20:80:0.1 acetonitrile:purified reagent water:trifluoroacetic acid (v:v:v) via mixing and sonication for five minutes before analysis via LC-MS/MS.

Etridiazole and 3-DCMT sample extracts were analyzed via GC-MS/EI using an Agilent 6890 series GC and Agilent DB-5MS column (15 m × 0.250 mm × 0.25 μm) using a column temperature program (initial 50°C and held for 2.00 minutes to post 250°C held for 0.00 minutes) and helium carrier gas coupled with MS detection (300°C transfer line and 230°C source temperature) (pp. 18, 29-30 of MRID 50534504). Three ions were monitored as follows (quantitation, confirmation 1, and confirmation 2, respectively): *m/z* 211.00, 185.00, and 183.00 for etridiazole, and *m/z* 143.00, 184.00, and 186.00 for 3-DCMT. Reported retention times for etridiazole and 3-DCMT were *ca.* 6.1 and 5.8 minutes, respectively. Benzophenone was used as the internal standard (p. 17 of MRID 50534504)

3-Carb-T sample extracts were analyzed via LC-MS/MS using a Phenomenex column and MDS Sciex API 5000 mass spectrometer equipped with a with an ESI Turbo V source (pp. 17-18, 31-32 of MRID 50534504). The LC conditions consisted of a Phenomenex Kinetex 5 μm EVO C18 column (50 μm × 2.1 mm, column temperature 35°C), a mobile phase of (A) 0.1% trifluoroacetic acid in water and (B) 0.1% trifluoroacetic acid in acetonitrile [percent A:B (v:v) at 0.01 min. 98.0:2.0, 0.50 min. 98.0:2.0, 2:00 min. 0:100, 3.00 min. 0:100, 3.10 min. 98.0:2.0, and 4.00 min. 98.0:2.0] and MS/MS detection in positive ion mode (ionization temperature 550°C). Injection volume was 50 μL. Two ion transitions were monitored (quantitation and confirmation, respectively) as follows: *m/z* 175.16→147.10 and *m/z* 175.16→129.00. Retention time was *ca.* 1.5 minutes.

In the ILV, the ECM was performed as written with significant modifications of the use of glass pipets instead of positive displacement pipets (plastic) for handling samples and of the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks (pp. 20-23, 30-32; Appendix 4, pp. 106-107 of MRID 50584602). Initially, the ILV incorporated the substitution of plastic for glass extraction vessels; however, recovery issues due to the analytes adhering to plastic were identified when plastic vessels were used in the place of glass equipment. The ILV also discovered that glass pipets should be used instead of positive displacement pipets (plastic) which were listed in the ECM. During, 3-Carb-T analysis, the ILV suspected that 3-Carb-T adhered to the glass volumetric flask during the serial dilutions of the 10,000 μg/L stock for fortification of the LOQ and 10 × LOQ samples and calibration standards. Recovery issues with 3-DCMT at the LOQ, resulting from storing extracts, was corrected by preparing fresh soil extracts, and it was noted that 3-DCMT extracts may be unstable. The ILV also incorporated insignificant modifications of the analytical instrumentation and parameters, and the use of matrix-matched calibration standards for 3-Carb-T analysis on silt loam soil. The ILV reported that a Thermo Trace 1300 Gas Chromatograph with ISQ LT single quadrupole mass spectrometer detector was used for etridiazole and 3-DCMT (similar GC-MS parameters; retention

times for etridiazole and 3-DCMT were 5.1 and 4.8 min., respectively) and a Shimadzu Nexera HPLC system coupled with an AB Sciex API 5000 MS/MS detector (similar LC-MS/MS parameters; same ionization temperature, 550°C, retention time *ca.* 1.35 min.) was used for 3-Carb-T (pp. 15, 24-25).

In the ECM and ILV, the method LOQs in soil were 50.0 µg/kg for etridiazole, 3-DCMT, and 3-Carb-T (pp. 32-33, 38-43 of MRID 50534504; pp. 12, 30 of MRID 50584602). In the ECM, the method LODs ranged from 5 to 7 µg/kg in clay loam soil and 6 to 11 µg/kg in silt loam soil for etridiazole; 3 to 18 µg/kg in clay loam soil and 2 to 20 µg/kg in silt loam soil for 3-DCMT; and 0.1 to 1 µg/kg in clay loam soil and 0.5 to 1 µg/kg in silt loam soil for 3-Carb-T (pp. 35-36 of MRID 50534504). In the ILV, the method LODs ranged from 1.28 to 2.37 µg/L in sandy loam soil and 3.88 to 24.5 µg/L in silt loam soil for etridiazole; 3.52 to 4.50 µg/L in sandy loam soil and 3.89 to 25.7 µg/L in silt loam soil for 3-DCMT; and 4.15 to 4.49 µg/L in sandy loam soil and 3.46 to 3.68 µg/L in silt loam soil for 3-Carb-T (pp. 28-29 of MRID 50584602).

II. Recovery Findings

ECM (MRID 50534504): Mean recoveries and relative standard deviations (RSD) from GC-MS/EI analysis were within guideline requirements (mean 70-120%; RSD ≤20%) for etridiazole and its metabolite 3-DCMT at 50.0 µg/kg (LOQ) and 500 µg/kg (10×LOQ) in two soil matrices (Tables 1-12, pp. 46-57). Mean recoveries and relative standard deviations from LC-MS/MS analysis were within guideline requirements for 3-Carb-T at 50.0 µg/kg (LOQ) and 500 µg/kg (10×LOQ) in two soil matrices (Tables 13-16, pp. 58-61). Performance data (recovery results) from primary and confirmatory analyses were comparable. Clay loam soil (SMV Lot No. 12DEC16SOIL-B; 40/28/32 sand/silt/clay, pH 5.4 in 1:1 soil:water ratio, 5.6% organic matter) was obtained from Grand Forks, North Dakota, and silt loam soil (SMV Lot No. 13SEP17SOIL-B; 24/60/16 sand/silt/clay), pH 7.2 in 1:1 soil:water ratio, 3.9% organic matter Walkley-Black) was obtained from Jackson, Iowa (USDA soil texture classification not specified; pp. 18-19). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota.

ILV (MRID 50584602): Mean recoveries and RSDs from GC-MS analysis were within guideline requirements for etridiazole and its metabolite 3-DCMT at 50.0 µg/kg (LOQ) and 500 µg/kg (10×LOQ) in two soil matrices (Tables 1-6, pp. 35-40, Tables 9-14, pp. 43-48 of MRID 50584602). Mean recoveries and RSDs from LC-MS/MS analysis were within guideline requirements for 3-Carb-T at 50.0 µg/kg (LOQ) and 500 µg/kg (10×LOQ) in two soil matrices (Tables 7-8, pp. 41-42, Tables 15-16, pp. 49-50 of MRID 50584602). Performance data (recovery results) from primary and confirmatory analyses were comparable. Sandy loam soil (CS 27/16 Speyer 5M; 59/30/11 sand/silt/clay, pH 8.5 in 1:5 soil:water ratio, 1.0% organic carbon) was obtained from Rheinland-Pfalz, Germany, and supplied by LUFA Speyer, and silt loam soil (CS 30/16 Brierlow; 26/58/16 sand/silt/clay, pH 6.4 in 1:5 soil:water ratio, 2.5% organic carbon) was obtained from Derbyshire, UK, and supplied by LRA Labsoil (USDA soil texture classification; p. 15; Appendix 2, pp. 103-104). Soil was characterized by Smithers Viscient (ESG), North Yorkshire, UK, except some of the analyses on the sandy loam soil was characterized by LUFA Speyer. The ILV validated the ECM method for the quantitation and confirmation analyses of etridiazole and 3-DCMT (10×LOQ only) in silt loam soil in the first trial and of etridiazole and 3-DCMT in sandy loam soil in the second trial with significant modifications of the use of glass pipets instead of positive displacement pipets (plastic) for handling samples (pp. 20-23, 30-32; Appendix 4, pp. 106-107). The ILV validated the

ECM method for the quantitation and confirmation analyses of 3-DCMT at the LOQ in silt loam soil in the second trial with significant modifications of the use of glass pipets instead of positive displacement pipets (plastic) for handling samples and the use of fresh soil extracts; it was noted that 3-DCMT extracts may be unstable in stored extracts. The ILV validated the ECM method for the quantitation and confirmation analyses of 3-Carb-T in sandy loam and silt loam soil matrices in the second trial with significant modifications of the use of glass pipets instead of positive displacement pipets (plastic) for handling samples and of the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks. Insignificant modifications to the analytical instruments were also employed. **An updated ECM should be submitted** which specifies the use of glass pipets instead of positive displacement pipets (plastic) for handling samples, the use of fresh soil extracts for 3-DCMT analysis due to stability issues, and the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks.

Table 2. Initial Validation Method Recoveries for Etridiazole and Metabolites 3-DCMT and 3-Carb-T in Soil.^{1,2}

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Clay Loam Soil						
Quantitation ion						
Etridiazole	50.0 (LOQ)	5	78.2-94.0	85.9	5.74	6.68
	500	5	91.2-102	94.8	4.15	4.37
3-DCMT	50.0 (LOQ)	5	80.3-104	89.2	8.68	9.74
	500	5	88.5-104	97.7	5.61	5.74
3-Carb-T	50.0 (LOQ)	5	82.2-92.4	87.2	4.28	4.91
	500	5	87.5-92.0	88.8	2.04	2.30
Confirmation ion 1						
Etridiazole	50.0 (LOQ)	5	77.4-98.8	86.8	8.37	9.65
	500	5	87.2-104	95.0	6.74	7.09
3-DCMT	50.0 (LOQ)	5	71.4-86.3	77.1	5.83	7.57
	500	5	87.4-95.6	91.5	3.20	3.49
3-Carb-T	50.0 (LOQ)	5	84.0-88.9	85.5	1.94	2.27
	500	5	82.7-87.1	85.7	1.83	2.14
Confirmation ion 2						
Etridiazole	50.0 (LOQ)	5	80.6-90.9	85.4	4.07	4.77
	500	5	88.9-101	94.2	5.20	5.52
3-DCMT	50.0 (LOQ)	5	75.4-86.4	79.2	4.28	5.40
	500	5	89.0-102	93.9	5.06	5.39
Silt Loam Soil						
Quantitation ion						
Etridiazole	50.0 (LOQ)	5	86.3-95.1	91.6	3.74	4.08
	500	5	88.3-96.1	92.6	2.95	3.19
3-DCMT	50.0 (LOQ)	5	88.7-93.6	90.7	1.88	2.07
	500	5	78.6-90.5	85.8	4.56	5.32
3-Carb-T	50.0 (LOQ)	5	88.5-92.0	89.5	1.43	1.59
	500	5	80.1-100.0	93.0	8.19	8.81
Confirmation ion 1						
Etridiazole	50.0 (LOQ)	5	85.2-94.5	90.2	4.13	4.58
	500	5	80.8-93.1	89.7	5.09	5.68
3-DCMT	50.0 (LOQ)	5	78.8-85.8	83.3	2.89	3.47
	500	5	83.2-95.6	90.5	4.94	5.46
3-Carb-T	50.0 (LOQ)	5	76.9-90.5	84.6	6.08	7.19
	500	5	86.6-98.8	92.6	4.67	5.04
Confirmation ion 2						
Etridiazole	50.0 (LOQ)	5	86.9-93.9	89.8	3.46	3.85
	500	5	86.3-94.6	90.6	2.99	3.30
3-DCMT	50.0 (LOQ)	5	83.3-90.8	86.1	2.95	3.43
	500	5	88.0-100	94.5	4.38	4.63

Data (uncorrected recovery results) were obtained from Tables 1-16, pp. 46-61, of MRID 50534504.

¹ The clay loam soil (SMV Lot No. 12DEC16SOIL-B; 40/28/32 sand/silt/clay, pH 5.4 in 1:1 soil:water ratio, 5.6% organic matter) was obtained from Grand Forks, North Dakota, and the silt loam soil (SMV Lot No. 13SEP17SOIL-B; 24/60/16 sand/silt/clay, pH 7.2 in 1:1 soil:water ratio, 3.9% organic matter Walkley-Black) was obtained from Jackson, Iowa (USDA soil texture classification not specified; pp. 18-19). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota.

2 For GC-MS/EI: three ions were monitored as follows (quantitation, confirmation 1, and confirmation 2, respectively): m/z 211.00, 185.00, and 183.00 for etridiazole, and m/z 143.00, 184.00, and 186.00 for 3-DCMT. For LC-MS/MS: two ion transitions were monitored (quantitation and confirmatory, respectively) as follows: m/z 175.16→147.10 and m/z 175.16→129.00 for 3-Carb-T.

Table 3. Independent Validation Method Recoveries for Etridiazole and Metabolites 3-DCMT and 3-Carb-T in Soil.^{1,2,3}

Analyte	Fortification Level (µg/kg)	Number of Tests	Recovery Range (%)	Mean Recovery (%)	Standard Deviation (%)	Relative Standard Deviation (%)
Sandy Loam Soil						
Quantitation ion						
Etridiazole	50.0 (LOQ)	5	84-93	89	4.1	4.6
	500	5	88-111	97	8.7	9.0
3-DCMT	50.0 (LOQ)	5	73-105	86	12.2	14.2
	500	5	84-118	96	13.2	13.7
3-Carb-T	50.0 (LOQ)	5	81-95	88	5.1	5.7
	500	5	101-121	110	7.7	7.0
Confirmation ion 1						
Etridiazole	50.0 (LOQ)	5	80-94	86	5.8	6.7
	500	5	91-101	96	3.9	4.1
3-DCMT	50.0 (LOQ)	5	76-98	85	8.3	9.8
	500	5	91-115	99	9.2	9.3
3-Carb-T	50.0 (LOQ)	5	89-93	92	1.9	2.1
	500	5	106-118	109	5.2	4.8
Confirmation ion 2						
Etridiazole	50.0 (LOQ)	5	78-91	87	5.0	5.8
	500	5	87-108	96	7.9	8.2
3-DCMT	50.0 (LOQ)	5	78-99	85	9.0	10.6
	500	5	82-107	96	9.1	9.5
Silt Loam Soil						
Quantitation ion						
Etridiazole	50.0 (LOQ)	5	56-84	71	11.3	15.9
	500	5	71-98	82	12.1	14.7
3-DCMT	50.0 (LOQ)	5	86-102	94	7.4	7.9
	500	5	60-93	81	12.6	15.6
3-Carb-T	50.0 (LOQ)	5	83-96	92	5.5	6.0
	500	5	101-108	105	2.9	2.7
Confirmation ion 1						
Etridiazole	50.0 (LOQ)	5	53-87	70	13.6	19.3
	500	5	72-92	83	8.9	10.7
3-DCMT	50.0 (LOQ)	5	81-97	87	6.1	7.0
	500	5	65-92	78	10.8	13.9
3-Carb-T	50.0 (LOQ)	5	82-96	89	5.1	5.8
	500	5	90-113	101	8.2	8.1
Confirmation ion 2						
Etridiazole	50.0 (LOQ)	5	78-119	94	15.8	16.9
	500	5	79-107	91	11.1	12.2
3-DCMT	50.0 (LOQ)	5	78-103	88	9.3	10.7
	500	5	65-86	74	9.3	12.7

Data (uncorrected recovery results) were obtained from Tables 1-16, pp. 35-50, of MRID 50584602.

1 3-DCMT synonym DCE and 3-Carb-T synonym etridiazole acid used in ILV.

2 Sandy loam soil (CS 27/16 Speyer 5M; 59/30/11 sand/silt/clay, pH 8.5 in 1:5 soil:water ratio, 1.0% organic carbon) was obtained from Rheinland-Pfalz, Germany, and supplied by LUFA Speyer, and silt loam soil (CS 30/16 Brierlow; 26/58/16 sand/silt/clay, pH 6.4 in 1:5 soil:water ratio, 2.5% organic carbon) was obtained from Derbyshire, UK, and supplied by LRA Labsoil (USDA soil texture classification; p. 15; Appendix 2, pp. 103-104). Soil was characterized

by Smithers Viscient (ESG), North Yorkshire, UK, except some of the analyses on the sandy loam soil was characterized by LUFA Speyer.

3 For GC-MS: three ions were monitored as follows (quantitation, confirmation 1, and confirmation 2, respectively): m/z 211, 185, and 183 for etridiazole, and m/z 143, 184, and 186 for 3-DCMT (DCE). For LC-MS/MS: two ion transitions were monitored (quantitation and confirmatory, respectively) as follows: m/z 174.9→146.9 and m/z 174.9→129.0 for 3-Carb-T (etridiazole acid).

III. Method Characteristics

In the ECM and ILV, the method LOQs in soil were 50.0 $\mu\text{g}/\text{kg}$ for etridiazole, 3-DCMT, and 3-Carb-T (pp. 32-33, 38-43 of MRID 50534504; pp. 12, 30 of MRID 50584602). In the ECM and ILV, the LOQ was defined as the lowest fortification level. In the ECM, it was noted that blank values should not exceed 30% of the LOQ. No calculations or comparisons to background levels were reported to justify the LOQ for the method in the ECM. In the ECM, the method LODs ranged from 5 to 7 $\mu\text{g}/\text{kg}$ in clay loam soil and 6 to 11 $\mu\text{g}/\text{kg}$ in silt loam soil for etridiazole; 3 to 18 $\mu\text{g}/\text{kg}$ in clay loam soil and 2 to 20 $\mu\text{g}/\text{kg}$ in silt loam soil for 3-DCMT; and 0.1 to 1 $\mu\text{g}/\text{kg}$ in clay loam soil and 0.5 to 1 $\mu\text{g}/\text{kg}$ in silt loam soil for 3-Carb-T (pp. 35-36 of MRID 50534504).

In the ILV, the method LODs ranged from 1.28 to 2.37 $\mu\text{g}/\text{L}$ in sandy loam soil and 3.88 to 24.5 $\mu\text{g}/\text{L}$ in silt loam soil for etridiazole; 3.52 to 4.50 $\mu\text{g}/\text{L}$ in sandy loam soil and 3.89 to 25.7 $\mu\text{g}/\text{L}$ in silt loam soil for 3-DCMT; and 4.15 to 4.49 $\mu\text{g}/\text{L}$ in sandy loam soil and 3.46 to 3.68 $\mu\text{g}/\text{L}$ in silt loam soil for 3-Carb-T (pp. 28-29 of MRID 50584602). The ILV calculated LOD generally supported the LOD of the ECM, with the only exception being the LOD for 3-Carb-T in the ECM was much lower than the ILV.

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

$$\text{LOD} = (3 \times \text{SN}_{\text{ctl}}) / (\text{Res}_{\text{PLS}}) \times \text{Con}_{\text{CLS}}$$

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), Res_{PLS} is the mean response in height of the two low calibration standards, and Con_{CLS} is the concentration of the low calibration standard (p. 34 of MRID 50534504). The ILV, LOD is calculated from $3 \times$ height of control baseline noise \times control dilution factor \times calibration standard concentration ($\mu\text{g}/\text{mL}$) / height of calibration standard peak (p. 26 of MRID 50584602).

Table 4. Method Characteristics for Etridiazole, 3-DCMT, and 3-Carb-T in Soil.

Analyte ¹		Etridiazole	3-DCMT	3-Carb-T	
Analysis		GC/MS		LC/MS/MS	
Limit of Quantitation (LOQ)	ECM	50 µg/kg	50 µg/kg	50 µg/kg	
	ILV				
Limit of Detection (LOD)	ECM (calculated)	Clay loam	5 µg/kg (Q) 6 µg/kg (C1) 7 µg/kg (C2)	18 µg/kg (Q) 3 µg/kg (C1) 4 µg/kg (C2)	0.1 µg/kg (Q) 1 µg/kg (C)
		Silt loam	6 µg/kg (Q) 11 µg/kg (C1) 6 µg/kg (C2)	30 µg/kg (Q) 2 µg/kg (C1) 4 µg/kg (C2)	0.5 µg/kg (Q) 1 µg/kg (C)
	ILV (calculated)	Sandy loam	1.28 µg/L (Q) 2.35 µg/L (C1) 2.37 µg/L (C2)	4.5 µg/L (Q) 4.02 µg/L (C1) 3.52 µg/L (C2)	4.15 µg/L (Q) 4.49 µg/L (C)
		Silt loam	7.76 µg/L (Q) 3.88 µg/L (C1) 24.5 µg/L (C2)	25.7 µg/L (Q) 3.91 µg/L (C1) 3.89 µg/L (C2)	3.46 µg/L (Q) 3.68 µg/L (C)
Linearity (calibration curve r ² and concentration range)	ECM	Clay loam	r ² = 0.99708 (Q) r ² = 0.99618 (C1) r ² = 0.99739 (C2)	r ² = 0.99589 (Q) r ² = 0.99694 (C1) r ² = 0.99542 (C2)	r ² = 0.99604 (Q) r ² = 0.99339 (C)
		Silt loam	r ² = 0.99773 (Q) r ² = 0.99377 (C1) r ² = 0.99792 (C2)	r ² = 0.99462 (Q) r ² = 0.99350 (C1) r ² = 0.99553 (C2)	r ² = 0.99643 (Q) r ² = 0.99049 (C)
	ILV ²	Sandy loam	r ² = 0.9951 (Q) r ² = 0.9951 (C1) r ² = 0.9927 (C2)	r ² = 0.9935 (Q) r ² = 0.9977 (C1) r ² = 0.9987 (C2)	r ² = 0.9888 (Q- LOQ) r ² = 0.9819 (Q- 10×LOQ) r ² = 0.9922 (C- LOQ) r ² = 0.9920 (C- 10×LOQ)
		Silt loam	r ² = 0.9951 (Q- LOQ) r ² = 0.9871 (Q- 10×LOQ) r ² = 0.9951 (C1- LOQ) r ² = 0.9890 (C1- 10×LOQ) r ² = 0.9927 (C2- LOQ) r ² = 0.9885 (C2- 10×LOQ)	r ² = 0.9903 (Q) r ² = 0.9884 (C1) r ² = 0.9836 (C2)	r ² = 0.9968 (Q- LOQ) r ² = 0.9976 (Q- 10×LOQ) r ² = 0.9950 (C- LOQ) r ² = 0.9946 (C- 10×LOQ)
	Range		0.750-7.50 µg/L	0.750-7.50 µg/L	1.00-10.0 µg/L
	Repeatable	ECM ³	Yes at LOQ and 10×LOQ (two characterized soils; USDA soil texture classification not specified)		
ILV ^{4,5}		Yes at LOQ and 10×LOQ (two characterized soils; USDA soil texture classification)			
Reproducible		Yes at LOQ and 10×LOQ			
Specific	ECM ⁶	Yes, no peaks were detected in the controls.	Yes, matrix interferences were <10% (clay loam) and <i>ca.</i> 25% (silt loam) ⁷ of the LOQ (Q; based on peak area).	Yes, matrix interferences were <1% of the LOQ (based on peak area). Peak fronting was observed.	
	ILV ⁸	Yes, matrix interferences were <10% of the LOQ (based on peak area)..	Yes, no matrix interferences were observed; however, Q chromatograms for silt loam soil showed significant baseline noise ⁹	No , multiple significant contaminants were present in all Q chromatograms, including the reagent blank, controls, and calibration standards. ¹⁰	

Data were obtained from pp. 35-36, 38-43 (LOQ/LOD); Tables 1-16, pp. 46-61 (recovery results); p. 22, Figures 1-66, pp. 62-127 (chromatograms and calibration curves) of MRID 50534504; pp. 12, 28-29 (LOQ/LOD); Tables 1-16, pp. 35-50 (recovery results); pp. 28-29, Figures 1-79, pp. 58-97 (chromatograms and calibration curves); Tables 17-22, pp. 51-56 (matrix effects) of MRID 50584602; DER Attachment 2. Analytes were identified using one quantitation ion and two confirmation ions for etridiazole and 3-DCMT and one quantitation and one confirmatory ion transition for 3-Carb-T. Q = Quantitation ion/ion transition; C1 = Confirmation ion 1; C2 = Confirmation ion 2; C = Confirmation ion transition

1 3-DCMT synonym DCE and 3-Carb-T synonym etridiazole acid used in ILV.

2 In the ILV, two coefficients of determination (r^2) values were provided for etridiazole, 3-DCMT, and 3-Carb-T when LOQ and 10×LOQ were run as separate batches as a result of failed initial validation attempts for one of the levels (pp. 28-29, Appendix 4, pp. 106-108 of MRID 50584602). The calibration curves for the later validation attempts were not provided. 3-Carb-T correlation coefficients (r^2) values were reviewer-calculated from r values provided in the study report (pp. 28-29; DER Attachment 2).

3 In the ECM, the clay loam soil (SMV Lot No. 12DEC16SOIL-B; 40/28/32 sand/silt/clay, pH 5.4 in 1:1 soil:water ratio, 5.6% organic matter) was obtained from Grand Forks, North Dakota, and the silt loam soil (SMV Lot No. 13SEP17SOIL-B; 24/60/16 sand/silt/clay), pH 7.2 in 1:1 soil:water ratio, 3.9% organic matter Walkley-Black) was obtained from Jackson, Iowa (USDA soil texture classification not specified; pp. 18-19 of MRID 50534504). Soil characterization was performed by Agvise Laboratories, Northwood, North Dakota.

4 In the ILV, sandy loam soil (CS 27/16 Speyer 5M; 59/30/11 sand/silt/clay, pH 8.5 in 1:5 soil:water ratio, 1.0% organic carbon) was obtained from Rheinland-Pfalz, Germany, and supplied by LUFA Speyer, and silt loam soil (CS 30/16 Brierlow; 26/58/16 sand/silt/clay, pH 6.4 in 1:5 soil:water ratio, 2.5% organic carbon) was obtained from Derbyshire, UK, and supplied by LRA Labsoil (USDA soil texture classification; p. 15; Appendix 2, pp. 103-104). Soil was characterized by Smithers Viscient (ESG), North Yorkshire, UK, except some of the analyses on the sandy loam soil was characterized by LUFA Speyer.

5 The ILV validated the ECM method for the quantitation and confirmation analyses of etridiazole and 3-DCMT (10×LOQ only) in silt loam soil in the first trial and of etridiazole and 3-DCMT in sandy loam soil in the second trial with significant modifications of the use of glass pipets instead of positive displacement pipets (plastic) for handling samples (pp. 20-23, 30-32; Appendix 4, pp. 106-107 of MRID 50584602). The ILV validated the ECM method for the quantitation and confirmation analyses of 3-DCMT at the LOQ in silt loam soil in the second trial with significant modifications of the use of glass pipets instead of positive displacement pipets (plastic) for handling samples and the use of fresh soil extracts; it was noted that 3-DCMT extracts may be unstable in stored extracts. The ILV validated the ECM method for the quantitation and confirmation analyses of 3-Carb-T in sandy loam and silt loam soil matrices in the second trial with significant modifications of the use of glass pipets instead of positive displacement pipets (plastic) for handling samples and of the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks. Insignificant modifications to the analytical instruments were also employed. **An updated ECM should be submitted** which specifies the use of glass pipets instead of positive displacement pipets (plastic) for handling samples, the use of fresh soil extracts for 3-DCMT analysis due to stability issues, and the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks.

6 In the ECM, matrix interferences based on representative peak areas of the control and the LOQ were 0%, <10%, and <1% for etridiazole, 3-DCMT, and 3-Carbon-T, respectively. Matrix effects assessment performed by the study author determined matrix effects were minimal for etridiazole, 3-DCMT, and 3-Carb-T in the clay loam and the silt loam (pp. 38-43 of MRID 50534503); results of the matrix assessment were not provided.

7 Based on Figures 42-43, pp. 103-104 of MRID 50534504. This matrix interference was <50% of the LOD since the calculated ECM LOD was 60% of the LOQ for the Q analysis (143.00). The reviewer noted that the study author noted that no peak was present at the RT of 3-DCMT (*ca.* 5.8 min.) in the controls, but a peak at RT 5.806 min. was quantified.

8 In the ILV, matrix interference was >20% in matrix-matched standards compared to non-matrix standards for 3-Carb-T in silt loam soil (Table 22, p. 56 or MRID 50584602); as a result, matrix-matched standards were used for 3-Carb-T analysis on silt loam soil. Matrix interference was <20% for all other analytes and soils (p. 30, Table 17-22, pp. 51-56). The ILV indicated etridiazole, 3-DCMT, and 3-Carb-T were found in the controls at ≤50% of the LOD and ≤30% of the LOQ (p. 29 of MRID 50584602).

9 Based on Figure 35, p. 75 of MRID 50584602.

10 Based on Figures 59-68, pp. 87-91 of MRID 50584602.

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

IV. Method Deficiencies and Reviewer's Comments

1. An updated ECM should be submitted which specifies the use of glass pipets instead of positive displacement pipets (plastic) for handling samples, the use of fresh soil extracts for 3-DCMT analysis due to stability issues, and the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks. Initially, the ILV incorporated the substitution of plastic for glass extraction vessels; however, recovery issues due to the analytes adhering to plastic were identified when plastic vessels were used in the place of glass equipment. The ILV also discovered that glass pipets should be used instead of positive displacement pipets (plastic) which were listed in the ECM. During, 3-Carb-T analysis, the ILV suspected that 3-Carb-T adhered to the glass volumetric flask during the serial dilutions of the 10,000 µg/L stock for fortification of the LOQ and 10×LOQ samples and calibration standards. Recovery issues with 3-DCMT at the LOQ, resulting from storing extracts, was corrected by preparing fresh soil extracts, and it was noted that 3-DCMT extracts may be unstable. The ILV included a modification of the stock dilution preparation of 3-Carb-T by preparing directly into disposable glass vials instead of glass volumetric flasks. Without these modifications, ILV recoveries and linearity were unacceptable, and the method could not be validated. Additionally, soil extract stability data should be provided for 3-DCMT to support the ECM.
2. For quantitation analysis, ILV linearity was not satisfactory for etridiazole analysis in silt loam, $r^2 = 0.9871$ ($10 \times \text{LOQ}$) (p. 29, Figure 1, p. 58); 3-DCMT in sandy loam and silt loam, $r^2 = 0.9935$ and 0.9903 , respectively (pp. 28-29, Figure 28, p. 71); 3-Carb-T in sandy loam, $r^2 = 0.9888$ (LOQ) and $r^2 = 0.9819$ ($10 \times \text{LOQ}$) (p. 28, Figure 58, p. 86 of MRID 50586402; DER Attachment 2)

For quantitation analysis, ECM linearity was not satisfactory for 3-DCMT analysis in silt loam, $r^2 = 0.99462$ (p. 41, Figure 46, p. 107 of MRID 50534504).

For the confirmation analysis, ILV linearity was not satisfactory for etridiazole in sandy loam soil, $r^2 = 0.9927$ (C2), or in silt loam soil, $r^2 = 0.9890$ (C1 – $10 \times \text{LOQ}$), 0.9927 (C2 – LOQ), and 0.9885 (C2 – $10 \times \text{LOQ}$); for 3-DCMT in silt loam soil, $r^2 = 0.9884$ (C1) and 0.9836 (C2); and for 3-Carb-T in sandy loam soil, $r^2 = 0.9922$ (LOQ) and 0.9920 ($10 \times \text{LOQ}$), or in silt loam soil, $r^2 = 0.9946$ ($10 \times \text{LOQ}$; pp. 28-29 of MRID 50584602). For the confirmation analysis, ECM linearity was not satisfactory for etridiazole in silt loam soil, $r^2 = 0.99377$ (C1); for 3-DCMT in silt loam soil, $r^2 = 0.99350$ (C1); and for 3-Carb-T in clay loam soil, $r^2 = 0.99339$, and silt loam soil, $r^2 = 0.99049$ (pp. 38-43 of MRID 50534504; DER Attachment 2). In the case of the confirmation analyses, the reviewer did not consider this guideline deviation to be significant since a confirmatory method is not typically required where GC/MS and/or LC/MS methods are used as the primary method(s) to generate study data

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

3. The specificity of the method was not supported for 3-Carb-T based on ILV representative chromatograms. Multiple significant contaminants were present in all Q chromatograms, including the reagent blank, controls, and calibration standards (Figures 59-68, pp. 87-91 of MRID 50584602). Minor baseline noise was observed in the confirmation ion chromatograms (Figures 70-79, pp. 92-97).

4. It could not be determined whether the ILV was provided with the most difficult matrices with which to validate the method since the percentage of soil organic carbon was 1.0-2.5% for the two ILV soils (p. 15; Appendix 2, pp. 103-104 of MRID 505084602). Also, it could not be confirmed if the ILV soil matrices covered the range of soils used in any submitted terrestrial field dissipation (TFD) studies.

In the ECM, USDA soil texture characterization was not specified (pp. 18-19 of MRID 50534504).

5. For the ILV, the first validation attempts for etridiazole and 3-DCMT in sandy loam soil were unsuccessful and required replacement of plastic extraction vessels with glass extraction vessels. The first validation attempts for etridiazole and 3-DCMT in silt loam soils were unsuccessful and required re-aliquoting extracts with glass pipettes instead of plastic. In addition, the first validation attempts for 3-Carb-T in sandy loam and silt loam soils were unsuccessful at $10 \times$ LOQ and were attributed to analyte adhering to the volumetric flasks used to prepare serial dilutions; as a result, serial dilutions were prepared directly into glass vials. Re-analysis was completed successfully, however in most cases numerous re-injections and re-aliquoting were required, indicating that the ILV method as specified may not be reproducible (Appendix 4, pp. 106-107 of MRID 50584602).
6. The ILV reported >20% matrix effects for 3-Carb-T analysis on silt loam soil, but not for the other analytes or soils (pp. 30, 32; Table 17-22, pp. 51-56 of MRID 50584602); matrix-matched calibration standards were used for 3-Carb-T analysis on silt loam soil. The ILV matrix assessment differed from the ECM in concentration of matrix matched standards [2.0 $\mu\text{g/L}$ etridiazole and 3-DCMT and 5.0 $\mu\text{g/L}$ for 3-Carb-T in the ILV (p. 18 of MRID 50584602); and 2.50 $\mu\text{g/L}$ for etridiazole and 3-DCMT and 5.00 $\mu\text{g/L}$ for 3-Carb-T in the ECM (p. 23 of MRID 50534504)], and the ILV reported the matrix effect as the difference between the (mean measured concentration with solvent – mean measured concentration with matrix)/measured concentration with solvent $\times 100$ (Tables 17-22, pp. 51-56).

In the ECM matrix-matched standards were not used for calibration. The ECM reported matrix effects were minimal for etridiazole, 3-DCMT and 3-Carb-T in clay loam and silt loam soil but did not provide matrix effects data (pp. 38-43 of MRID 50534504).

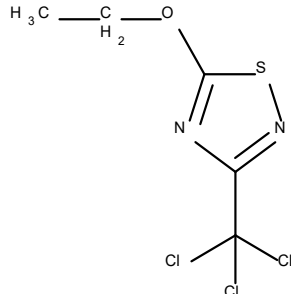
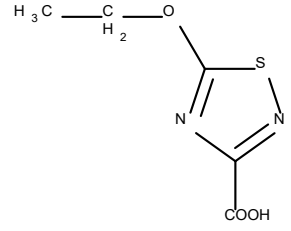
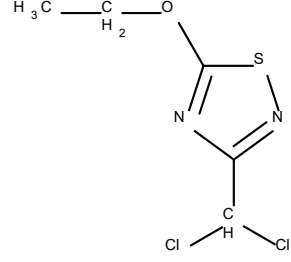
7. In the ILV, the coefficient of determination (r^2) data for all of the linear calibration curves required to validate 3-DCMT (DCE) in the Brierlow silt loam soil did not appear to be provided in the study report (p. 29; Appendix 4, p. 106-107 of MRID 50584602). The $10 \times$ LOQ analysis passed in Sequences Val 2R and 2Ri; the LOQ analysis passed in Sequence Val 6 (Figures 35-36, p. 75; Figures 44-45, pp. 79-80; Figures 53-54, p. 84). However, in the reported r^2 data on p. 29, only one r^2 value was provided for each ion analysis, and only calibration curves from Sequence Val 5 were provided in the study report (Figures 28, 38, and 46, pp. 71, 76, 80). The reviewer noted that linearity was unsatisfactory for all provided calibration data for 3-DCMT in the Brierlow silt loam soil.
8. The reviewer noted that the calculated ECM LOD for 3-DCMT in silt loam was 60% of the LOQ for the Q analysis (143.00; p. 41 of MRID 50534504).

9. In the ILV, one of each of the 1.5, 2.0, and 7.5 µg/L calibration standards for etridiazole were deleted from the linear calibration curve for the quantitation ion and both confirmation ions because these data points were outliers for the corresponding 3-DCMT curves; a result of the laboratory software used in calculating linear calibration curves for mixed standards (Figures 1, 10, 19, pp. 58, 62, and 67 of MRID 50584602). One of each of the 1.5, 2.0, and 7.5 µg/L calibration standards were deleted from the 3-DCMT linear calibration curves because they deviated from the line (Figure 28, 37, 46, pp. 71, 76, 80 of MRID 50584602).
10. The ECM laboratory is Smithers Viscient Laboratory in Wareham, Massachusetts, and the ILV laboratory is also a Smithers Viscient Laboratory, but located in Harrogate, North Yorkshire, United Kingdom. One communication was documented between the two laboratories, and that was to confirm the SPE cartridge particle size. The other documented communications were between the Harrogate laboratory and the sponsor MacDermid Agricultural Solutions, Inc., concerning schedule and approval to repeat validation of etridiazole and 3-DCMT using glass extraction vessels and re-aliquoting extracts with a glass pipette, and to repeat preparation of 3-Carb-T intermediate stock, calibration standards and analysis at the LOQ and 10×LOQ (Appendix 5, p. 108 of MRID 50584602). The reviewer determined that no collusion occurred.
11. The time required to complete the method for a sample set 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.

DER ATTACHMENT 1. Etridiazole and Its Environmental Transformation Products. ^A

Code Name/ Synonym	Chemical Name	Chemical Structure	Study Type	MRID	Maximum %AR (day)	Final %AR (study length)
PARENT						
Etridiazole	IUPAC: Ethyl 3-trichloromethyl-1,2,4-thiadiazol-5-yl ether CAS: 5-Ethoxy-3-(trichloromethyl)-1,2,4-thiadiazole CAS No.: 2593-15-9 Formula: C ₅ H ₅ Cl ₃ N ₂ OS MW: 247.5 g/mol SMILES: CCOc1nc(ns1)C(Cl)(Cl)Cl		850.6100 ECM soil	50534504	NA	NA
			850.6100 ILV soil	50584602		
Etridiazole acid (3-Carb-T)	IUPAC: 5-Ethoxy-1,2,4-thiadiazole-3-carboxylic acid CAS No.: 67472-43-9 Formula: C ₅ H ₆ N ₂ O ₃ S MW: 174.17 g/mol SMILES: CCOc1nc(ns1)C(=O)O		850.6100 ECM soil	50534504	NA	NA
			850.6100 ILV soil	50584602		
DCE (3-DCMT; T-03)	IUPAC: 5-Ethoxy-3-dichloromethyl-1,2,4-thiadiazole Formula: C ₅ H ₆ Cl ₂ N ₂ OS MW: 213.08 g/mol SMILES: CCOc1nc(ns1)C(Cl)Cl		850.6100 ECM soil	50534504	NA	NA
			850.6100 ILV soil	50584602		

^A AR means "applied radioactivity". MW means "molecular weight". NA means "not applicable". ECM means "environmental chemical methods". ILV means "independent laboratory validation".