

Dimethyl disulfide; EPA PC Code 029088

Cerexagri, Inc.; EPA Company Code

ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

Test Material: Dimethyl disulfide

MRID: 48535303

Title: Arndt, T. 2005. Independent laboratory validation of the analytical method for the determination of dimethyl disulfide from sorbent charcoal tubes.

MRID: 48535303 - Appendix A


Title: Analytical method for the determination of dimethyl disulfide from sorbent charcoal tubes.

EPA PC Code: 029088

OCSPP Guideline: 850.6100

For Cambridge Environmental

Primary Reviewer: Lynne Binari

Signature: 

Date: 8/28/12

Secondary Reviewer: Kathleen Ferguson

Signature: 

Date: 8/28/12

QC/QA Manager: Joan Gaidos

Signature: 

Date: 8/28/12

EPA MRID Number 48535303 (ECM/ILV)

Dimethyl disulfide; EPA PC Code 029088
Cerexagri, Inc.; EPA Company Code
ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

The pages cited below appear in the uppermost right corner of each page of the MRID. Figures in Appendix D (pp. 84-89) mistakenly labeled "Appendix C" in the MRID.

Data Requirement: EPA Guideline:
OECD Data Point: IIA 4.7

Test material:
Common name: Dimethyl disulfide (DMDS).
Chemical name: Dimethyl disulfide.
IUPAC: Dimethyl disulfide.

Primary Reviewer: Gabe Rothman, Environmental Scientist, USEPA
Date: November 22, 2013
[Evaluator, title, and affiliation]

Secondary Reviewer: _____ **Date:** _____
[Additional reviewer(s), title, and affiliation]

ANALYTICAL METHOD: EPA MRID No. 48535303 - Appendix A. Nissanga, J., R. Tauber and R. Bennett. 2005. Analytical method for the determination of dimethyl disulfide from sorbent charcoal tubes. Report prepared by Enviro-Test Laboratories/Ottawa Division, Ontario, Canada, sponsored and submitted by Cerexagri, Inc., King of Prussia, Pennsylvania; 21 pages (p. 3; Appendix A, pp. 53-73). Final report issued June 27, 2005.

INDEPENDENT LABORATORY VALIDATION: EPA MRID No. 48535303. Arndt, T. 2005. Independent laboratory validation of the analytical method for the determination of dimethyl disulfide from sorbent charcoal tubes. Report prepared by PTRL West, Inc., Hercules, California, sponsored and submitted by Cerexagri, Inc., King of Prussia, Pennsylvania; 90 pages. Final report issued October 21, 2005.

EXECUTIVE SUMMARY

This method is designed for the quantitative determination of residues of dimethyl disulfide (DMDS) in sorbent charcoal tubes using an external standardization method. The method was developed by Enviro-Test Laboratories (analytical procedure KP-227R1 [ETL/XAM-84]) for Cerexagri, Inc.; no regulatory guidelines were cited in the ECM (Appendix A, pp. 53-73). An independent laboratory validation (ILV), performed by PTRL West, Inc., was submitted with the method. *The Agency finds that this method meets the criteria for a scientifically valid method and is **acceptable** for (applicable residues).*

Method Summary: Dimethyl disulfide (DMDS) is extracted from charcoal with methyl acetate and the extract analyzed directly by GC/MS (Appendix A, p. 56). The ECM defined a limit of quantitation (LOQ) of 0.10 µg for DMDS on a sorbent charcoal tube; a limit of detection (LOD) was not reported (Appendix A, p. 56). The LOQ defined by the ECM was supported by the ILV, and the ILV estimated a LOD of 0.025 µg DMDS (p. 23).

METHOD ACCEPTABILITY/DEFICIENCIES/CLARIFICATIONS

For the ECM, performance data, LOD, justification for selection of the LOQ concentration, and chromatograms of method blank samples were not provided. Additionally, recovery results could not be calculated from the provided chromatograms.

For the ILV, acceptance criteria were met (mean matrix spike recoveries between 70% and 120% and relative standard deviations of ≤20%) at the LOQ and 10 x LOQ (DER Attachment 2), and a LOD was estimated. However, actual time required per sample set and number of sample sets performed to complete a successful trial were not reported.

The ILV recommended that the GC/MS four ion quantitation (m/z 45, 61, 79, 94) used in the ECM be modified to three ion quantitation (m/z 64, 79, 94) to improve linearity and sensitivity of the analysis (p. 22).

COMPLIANCE

No regulatory guidelines were cited in the ECM (Appendix A, pp. 53-73). For the ILV, signed and dated statements of Data Confidentiality, GLP and Quality Assurance were provided (pp. 2-4). A statement of the authenticity of the study report is included in the Quality Assurance statement (p. 4).

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ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT

A. BACKGROUND INFORMATION

Dimethyl disulfide (DMDS) is a widely used industrial chemical and is regulated as a food additive (Appendix A, p. 56). DMDS has been shown effective in control of soil-born fungi, nematodes and nutsedge, and is a proposed soil fumigant product manufactured by Cerexagri, Inc. (p. 12; Appendix A, p. 56).

| TABLE A.1. Test Compound Nomenclature | |
|--|--------------------------------------|
| Parameter | Value |
| Common name | Dimethyl disulfide (DMDS) |
| Company experimental name | None reported. |
| IUPAC name | Dimethyl disulfide |
| CAS Name | Dimethyl disulfide |
| CAS # | 624-92-0 |
| Structure | H ₃ C-S-S-CH ₃ |

Information obtained from Appendix A, p. 56 of the study report.

| TABLE A.2. Physicochemical Properties of the Technical Grade Test Compound | |
|---|---------------|
| Parameter | Value |
| Melting point/range (°C) | Not reported. |
| pH | Not reported. |
| Density (g/cm ³) | Not reported. |
| Water solubility at 20°C (mg/L) | Not reported. |
| Solvent solubility at 20°C (mg/L) | Not reported. |
| Vapor pressure at ___°C (torr) | Not reported. |
| Dissociation constant (pK _a) | Not reported. |
| Octanol/water partition coefficient | Not reported. |
| UV/visible absorption spectrum (nm) | Not reported. |

ENVIRONMENTAL CHEMISTRY METHOD REVIEW REPORT**B. MATERIALS AND METHODS****B.1. Principle of Method**

Dimethyl disulfide (DMDS) is extracted from charcoal with methyl acetate and the extract analyzed directly by GC/MS using a SPB-1-sulfur column (Appendix A, pp. 58-59).

| TABLE B.1. Summary Parameters for the Analytical Method Used for the Quantitation of Chemical Residues in Matrices Studied | |
|---|---|
| Parameter | Value |
| Method ID | Analytical method for the determination of dimethyl disulfide from sorbent charcoal tubes (Appendix A, p. 53). Also described as analytical procedure KP-227R1 [ETL/XAM-84] (Appendix A, p. 56). |
| Analyte(s) | Dimethyl disulfide. |
| Extraction solvent/technique | Charcoal transferred to culture tube (15 mL) and extracted with methyl acetate by vortexing (30 seconds), sonication (2 minutes), then shaking (platform shaker) for 30 minutes; extraction solvent volumes 5 mL for charcoal from front (<i>ca.</i> 400 mg) or back (<i>ca.</i> 200 mg) of sorbent tube, 10 mL for charcoal from front and back of tube combined (p. 16; Appendix A, p. 58). |
| Cleanup strategies | After shaking, charcoal and extract sonicated (2 minutes), then separated by centrifugation (2,500 rpm, at least 5 minutes). |
| Instrument/Detector | <u>ECM</u> : Varian 3400CX GC using a Supelco SPB-1-sulfur column (0.32 mm x 30 m, 4.0 μ m film thickness) and a Saturn 2000 MS equipped with electron impact with automatic gain control (EI-AGC) ionization and Selected Ion Monitoring (SIM; Appendix A, p. 59). GC retention time <i>ca.</i> 15 minutes (Appendix A, pp. 63-69, 71-73). Total area of ions at <i>m/z</i> 45, 61, 79 and 94 used to quantify DMDS (Appendix A, pp. 56, 59). <u>ILV</u> : same as ECM, except with Hewlett Packard (HP) 5890 GC and HP5971A Mass Selective Detector (p. 16). |

Information obtained from p. 16; Appendix A, pp. 53, 56, 58-59, 63-69, 71-73 of the study report.

C. RESULTS AND DISCUSSION

C.1. Recovery Results Summary

TABLE C.1. Recovery Results from Method Validation for the Determination of Dimethyl Disulfide in Charcoal from Sorbent Tubes¹

| Analyte | Spiking Level (µg a.i.) | Mean Recoveries Obtained (%) | Relative Standard Deviation |
|--------------------|-------------------------|------------------------------|-----------------------------|
| Dimethyl disulfide | 0.1 (LOQ) | -- | -- |
| | 1.0 | -- | -- |
| | 5,000 | -- | -- |

-- = Not reported.

¹ Spiking level from Appendix A, pp. 71-73. Performance data were not reported. Sufficient information, such as dilution factor, was not provided to allow the reviewer to calculate recoveries from the provided chromatograms (Appendix A, pp. 63-73).

C.1.1. Method Characteristics

TABLE C.2. Method Characteristics

| Parameter | Value |
|---------------------------------|--|
| Analyte(s) | Dimethyl disulfide. |
| Limit of Quantitation (LOQ) | 0.10 µg (Appendix A, p. 60). |
| Limit of Detection (LOD) | 0.025 µg (p. 23). |
| Accuracy/Precision at LOQ | ECM: Performance data were not reported. |
| Reliability of the Method/[ILV] | Acceptance criteria were met at the LOQ with mean matrix spike recoveries between 70% and 120% and relative standard deviations of ≤20% (DER Attachment 2). Time required per validation (thirteen) sample set and number of sample sets performed to complete a successful trial were not reported. |
| Linearity | ECM: Linear regression; $r^2 = 0.998$ (DER Attachment 2). ILV: Linear regression; $r^2 = 0.99890$ (Figure 3, p. 30). |
| Specificity | Comparison of chromatograms produced for standards and control and fortified samples demonstrates that the method, based on GC/MS, is highly specific for the analysis of dimethyl disulfide (Figures 4-12, pp. 31-39; Figures 14-19, pp. 41-46; Appendix A, pp. 63-73). Method and matrix blank controls showed no significant interferences at the retention time of the analyte (ca. 14.6 minutes in Figure 4, p. 31; Figures 14-15, pp. 41-42; ca. 15 minutes in Appendix A, p. 70). The ILV did detect minor impurity peak responses near the retention time of DMDS during analysis of reagent blank on individual ion chromatograms (p. 22; Appendix D, p. 86). ILV recommended that the GC/MS four ion quantitation (m/z 45, 61, 79, 94) be modified to |

| TABLE C.2. Method Characteristics | |
|--|--|
| Parameter | Value |
| | three ion quantitation (m/z 64, 79, 94) to improve linearity and sensitivity of the analysis (p. 22; Appendix D, pp. 87-89). |

Information obtained from p. 23; Figures 3-12, pp. 30-39; Figures 14-19, pp. 41-46; Appendix A, pp. 60, 63-73 of the study report and DER Attachment 2.

C.2. Independent Laboratory Validation (ILV)

The ILV was conducted in compliance with USEPA GLP Standards 40 CFR, Part 160, OPPTS 860.1340 guidelines, EU Commission Directive 96/46/EC and SANCO/825/00 rev. 6 (pp. 3, 12).

| TABLE C.3. Recovery Results of the Method Obtained by an Independent Laboratory Validation for the Determination of Residues in Sorbent Charcoal Tubes (n = 5) | | | |
|---|--------------------------------|-------------------------------------|------------------------------------|
| Analyte | Spiking Level (µg a.i.) | Mean Recoveries Obtained (%) | Relative Standard Deviation |
| Dimethyl disulfide (DMDS) | 0.10 (LOQ) | 87 | 5.5 |
| | 1.0 | 90 | 2.3 |

Calculated by reviewer using reported results from Figure 13, p. 40 (DER Attachment 2). Reported results generated using four ion quantitation method (p. 23).

D. CONCLUSION

This method is designed for the quantitative determination of residues of dimethyl disulfide in sorbent charcoal tubes. *The Agency finds that this method meets the criteria for a scientifically valid method and is **acceptable** for (applicable residues).* The ILV recommends that the GC/MS four ion quantitation (m/z 45, 61, 79, 94) used in the ECM be modified to three ion quantitation (m/z 64, 79, 94) to improve linearity and sensitivity of the analysis.

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

**ENVIRONMENTAL CHEMISTRY METHOD (ECM)
STANDARD EVALUATION PROCEDURE (SEP) CHECKLIST:
BACKGROUND AND INITIAL REVIEW INFORMATION**

The pages cited below appear in the uppermost right corner of each page of the MRID. Figures in Appendix D (pp. 84-89) mistakenly labeled "Appendix C" in the MRID.

I. Background Information

| | | | |
|-------------------------------|----------------------------|--|--|
| A. | Title of Method | Analytical method for the determination of dimethyl disulfide from sorbent charcoal tubes (Appendix A, p. 53). Also described as analytical procedure KP-227R1 [ETL/XAM-84] (Appendix A, p. 56). | |
| B. | ECM No. | [Leave blank. This is for BEAD ECB's use.] | |
| C. | MRID No. | 48535303 | |
| D. | Matrix | Sorbent charcoal (Appendix A, p. 56). | |
| E. | Analyte(s) detected | Compound: | |
| | | Common name: | Dimethyl disulfide (Appendix A, p. 56) |
| | | IUPAC name: | Dimethyl disulfide |
| | | CAS name: | Dimethyl disulfide |
| | | CAS No: | 624-92-0 |
| | | Synonyms: | DMDS |
| $\text{H}_3\text{C-S-S-CH}_3$ | | | |

Information obtained from Appendix A, pp. 53, 56 of the study report.

II. Information about the Laboratory

| | | |
|-----------|-----------------------------------|--|
| A. | Name | Enviro-Test Laboratories (Appendix A, p. 53). |
| B. | Address | Enviro-Test Laboratories/Ottawa Division, 210 Colonnade Road, Unit 13, Nepean, Ontario, K2E 7L5, Canada. |
| C. | Telephone No. | (613) 731-1005 |
| D. | Name of the Study Director | Not reported. |
| E. | Name of the Lead Chemist | Not reported. |
| F. | Laboratory Validation: | No. Analytical method appended to independent laboratory validation report (Appendix A, pp. 53-73). |

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

Information obtained from Appendix A, pp. 53-54 of the study report.

III. Method Summary Information for Analyte(s): Dimethyl disulfide.

| | | |
|-----------|--|---|
| A. | Statement of Data Confidentiality | Yes (p. 2). |
| 1. | Is the Method Classified or Confidential? | No claim of confidentiality is made for any information contained in the study on the basis of its falling within the scope of FIFRA §10 (d)(1)(A),(B) or (C). |
| 2. | Submitted Prior to 2008 with a Non-Standard Claim of Confidentiality? | No. |
| B. | Sample Preparation | Anasorb charcoal tubes (SKC Inc., Cat. No. 226-09) were fortified by vapor-phase trapping with 0.10, 1.0 and 5,000 µg of dimethyl disulfide, in methyl acetate, via injection into a gas chamber connected to an air pump with an intake flow rate of 2 L/minute for 15 minutes (Figure 2, p. 29; Appendix A, pp. 57-58, 71-73). |
| C. | Sample Extraction | Charcoal was extracted with methyl acetate by vortexing (30 seconds), sonication (2 minutes), then shaking (platform shaker) for 30 minutes; extraction solvent volumes 5.0 mL for charcoal from front (<i>ca.</i> 400 mg) or back (<i>ca.</i> 200 mg) of sorbent tube, 10.0 mL for charcoal from front and back of tube combined (p. 16; Appendix A, p. 58). |
| D. | Sample Cleanup | After shaking, charcoal and extract sonicated (2 minutes), then separated by centrifugation (2,500 rpm, at least 5 minutes). |
| E. | Sample Derivatization (if applicable) | None. |
| F. | Sample Analysis | GC/MS (Appendix A, p. 59). |
| 1. | Instrumentation | ECM: Varian 3400CX GC with Saturn 2000 MS using electron impact with automatic gain control (EI-AGC) ionization (Appendix A, p. 59). ILV: Hewlett Packard (HP) 5890 GC with HP5971A Mass Selective Detector (p. 16). |
| 2. | Primary Column | Supelco SPB-1-sulfur column (0.32 mm x 30 m, 4.0 µm film thickness; p. 17; Appendix A, pp. 56, 59). |

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

| | | | | |
|-----------|---|--|---------------------------|---|
| 3. | Confirmatory Column (if any) | None reported. | | |
| 4. | Detector | Selected Ion Monitoring (SIM, p. 17). | | |
| 5. | Other Confirmatory Techniques (if any) | None reported. | | |
| 6. | Other Relevant Information | GC retention time <i>ca.</i> 15 minutes (Appendix A, pp. 63-69, 71-73). Total area of ions at m/z 45, 61, 79 and 94 used to quantify DMDS (Appendix A, pp. 56, 59). | | |
| G. | Detection and Quantitation Limits | | | |
| 1. | Limit of Quantitation (LOQ) | | | |
| | Claimed in Method | 0.10 µg a.i. (Appendix A, p. 60). | Estimated | No justification for selected LOQ was provided. |
| 2. | Limit of Detection (LOD) | | | |
| | Claimed in Method | ECM: Not reported. ILV: 0.025 µg a.i. (p. 23). | Estimated | Based on 2 µl injection of lowest calibrant at 0.005 µg/mL (p. 23). |
| H. | Recovery (Accuracy)/Precision Data; expressed as percentage of applied¹ | | | |
| | Spiking Level (µg a.i.) | Parameter | Dimethyl disulfide | |
| | 0.10 (LOQ) | Range | -- | |
| | | Mean | -- | |
| | | SD | -- | |
| | | RSD | -- | |
| | 1.0 | Range | -- | |
| | | Mean | -- | |
| | | SD | -- | |
| | | RSD | -- | |
| | 5,000 | Range | -- | |
| | | Mean | -- | |
| | | SD | -- | |
| | | RSD | -- | |

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Information obtained from pp. 2, 16-17, 23; Figure 2, p. 29; Appendix A, pp. 56-60, 63-69, 71-73 of the study report.

-- = Not reported.

1 Spiking level from Appendix A, pp. 71-73. Performance data were not reported. Sufficient information, such as dilution factor, was not provided to allow the reviewer to calculate recoveries from the provided chromatograms (Appendix A, pp. 63-73).

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

IV. Detailed Information about the Method

| | | YES | NO | REVIEW FURTHER |
|----|--|-----|----|-------------------------|
| A. | Does the method require spiking with the analytes(s) of interest? | x | | Appendix A, p. 58. |
| B. | If the method requires explosive or carcinogenic reagents, are proper precautions explained? | | | Not applicable. |
| C. | Is the following information supplied? | | | |
| 1. | Detailed stepwise description of: | | | |
| a. | The sample preparation procedure? | x | | Appendix A, p. 58. |
| b. | The sample spiking procedure? | x | | Appendix A, pp. 57-58. |
| c. | The extraction procedure? | x | | Appendix A, p. 58. |
| d. | The derivatization procedure? | | | Not applicable. |
| e. | The clean-up procedure? | | | None. |
| f. | The analysis procedure? | x | | Appendix A, pp. 58-59. |
| 2. | Procedures for: | | | |
| a. | Preparation of standards? | x | | Appendix A, pp. 57-58. |
| b. | Calibration of instrument? | x | | Appendix A, pp. 59-60. |
| 3. | List of glassware and chemicals | x | | Appendix A, pp. 56-57. |
| a. | Are sources recommended? | x | | |
| b. | Are they commercially available? | x | | |
| 4. | Name, model, etc., of the instrument, column, detector, etc., used? | x | | Appendix A, pp. 56, 59. |
| a. | Are sources recommended? | x | | |
| b. | Are they commercially available? | x | | |
| 5. | LOD | | | |

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

| | | YES | NO | REVIEW FURTHER |
|--|---|-------------|---------------------|--|
| a. | Is there an explanation of how it was calculated? | ILV | ECM | Based on lowest calibrant used (p. 23). |
| b. | Is it a scientifically accepted procedure? | x | | |
| c. | Is the matrix blank free of interference(s) at the retention time, wavelength, etc., of the analyte(s) of interest? | x | | ECM: Appendix A, p. 70. ILV: Figures 14-15, pp. 41-42. |
| 6. LOQ | | | | |
| a. | Is there an explanation of how it was calculated? | | x | |
| b. | Is it a scientifically accepted procedure? | | | |
| 7. Precision and accuracy data | | | | |
| a. | Were there an adequate number of spiked samples analyzed? | ILV | ECM | ECM: No performance data reported. ILV: Figure 13, p. 40. |
| b. | Are the mean recoveries between 70-120%? | ILV | | |
| c. | Are the RSDs of the replicates 20% or less at or above the LOQ? | ILV | | |
| 8. Description and/or explanation of: | | | | |
| a. | Areas where problems may be encountered? | x | | Appendix A, p. 61. |
| b. | Steps that are critical? | | x | |
| c. | Interferences that may be encountered? | ILV: Minor. | ECM: None reported. | See section IX. <i>Recommendations</i> below. |
| 9. | Characterization of the Matrix(ces)? | x | | Appendix A, p. 57. |

Information obtained from pp. 22-23; Figures 13-15, pp. 40-42; Appendix A, pp. 56-61, 70; Appendix D, p. 86 of the study report.

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V. Representative Chromatograms

| | | YES | NO | REVIEW FURTHER |
|-----------|--|-----|-----|--|
| A. | Are there representative chromatograms for: | | | |
| 1. | Analyte(s) in each matrix at the LOQ and 10 x LOQ? | x | | EMC: Appendix A, pp. 71-72. ILV: Figure 16, p. 43; Figure 18, p. 45. |
| 2. | Method blanks? | ILV | ECM | ILV: Figure 4, p. 31. |
| 3. | Matrix blanks? | x | | ECM: Appendix A, p. 70. ILV: Figures 14-15, pp. 41-42. |
| 4. | Standard curves? | ILV | ECM | ECM: Reviewer generated standard curve using provided calibration standard chromatograms (Appendix A, pp. 63-69; DER Attachment 2). ILV: Figure 3, p. 30. |
| a. | Do the standard curves have acceptable linearity? | x | | ECM: $r^2 = 0.998$ (DER Attachment 2). ILV: $r^2 = 0.99890$ (Figure 3, p. 30). |

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

| | | | | |
|-----------|--|-----|-----|---|
| 5. | Standards that can be used to recalculate some of the values for analyte(s) in the sample chromatograms? | ILV | ECM | ECM: Sufficient information, such as dilution factor, was not provided. ILV: DER Attachment 2. |
| B. | Can the responses of the analytes(s) in the chromatograms of the lowest spiking level be accurately measured? | x | | ECM: Appendix A, p. 63. ILV: Figure 5, p. 32. |

Information obtained from Figures 3-5, pp. 30-32; Figures 14-16, pp. 41-43; Figure 18, p. 45; Appendix A, pp. 63-72 of the study report and DER Attachment 2.

VI. Good Laboratory Practice (GLP) Standards

| | | YES | NO | REVIEW FURTHER |
|-----------|--|-----|-----|----------------|
| A. | Is there a statement of adherence to the FIFRA GLP standards? | ILV | ECM | |

Information obtained from p. 3 of the study report.

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

VII. Independent Lab Validation (ILV)

| | | YES | NO | REVIEW FURTHER |
|------------|--|------------------|--|---|
| A. | Was an ILV performed? | x | | |
| B. | Was the validation independent? | x | | pp. 12, 21 |
| C. | Did the ILV's precision/accuracy data meet the criteria established in OPPTS Guideline 850.6100? | x | | DER Attachment 2. |
| D. | Were recommendations of major or minor modifications to the method made by the independent lab performing the ILV? If major modifications were suggested, what were they? | x | | See section <i>IX. Recommendations</i> below. |
| E. | Recovery (Accuracy)/Precision Data; expressed as percentage of applied (n = 5)¹ | | | |
| | Spiking Level (µg a.i.) | Parameter | Dimethyl disulfide | |
| | | | Vapor-phase Fortification | |
| | 0.1 (LOQ) | Range | 81-93 | |
| | | Mean | 87 | |
| | | SD | 4.8 | |
| | | RSD | 5.5 | |
| | 1.0 | Range | 87-92 | |
| | | Mean | 90 | |
| | | SD | 2.1 | |
| | | RSD | 2.3 | |
| | | | Direct ("Extraction") Fortification ² | |
| 0.1 | n = 1 | 97 | | |
| 1.0 | n = 1 | 87 | | |

Information obtained from pp. 12, 18, 21-22; Figures 1-2, pp. 28-29; Figure 22, p. 49; Appendix D, pp. 87-89 of the study report.

1 Calculated by reviewer using reported results from Figure 13, p. 40 (DER Attachment 2). Reported results generated using four ion quantitation method (p. 23).

2 Dimethyl disulfide, in methyl acetate, added directly to front portion of charcoal (400 mg) from sorbent tube and extracted immediately (p. 15; Figure 13, p. 40; Figures 20-21, pp. 47-48). Similarity in recoveries supports efficient trapping of DMDS from the vapor phase during ILV (p. 21).

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

VIII. Completeness

| | | YES | NO | REVIEW FURTHER |
|----|--|-----|----|--|
| A. | Has enough information been supplied to do a proper review? | | x | ECM: No performance data. |
| B. | Has enough information been supplied to do a laboratory evaluation, if requested? [This may be left blank, as it is a determination made by BEAD ECB.] | | | |
| C. | Are all steps in the method scientifically sound? | x | | |
| D. | Is a confirmatory method or technique provided? | | | GC/MS is used as primary method. |
| E. | Check the category below which best describes this ECM. | | | |
| 1. | Satisfactory [Acceptable] | | | |
| 2. | Major Deficiencies | x | | ECM: No performance data. |
| 3. | Minor Deficiencies | x | | See section IX. Recommendations below. |


IX. Recommendations

1. For the ECM:
 - a) No performance data were provided.
 - b) Sufficient data were not provided to calculate recovery results from the chromatograms (Figures 9-11, pp. 71-73).
 - c) Reagent blank samples were not analyzed.
 - d) No justification for selection of the limit of quantitation concentration was provided.
 - e) The limit of detection concentration was not reported.

ENVIRONMENTAL CHEMISTRY METHOD REVIEW CHECKLIST

- f) No regulatory guidelines were cited (Appendix A, pp. 53-73).
2. A validation sample set should consist of, at a minimum, a reagent blank, two unspiked matrix control samples, five matrix control samples spiked at the LOQ and an additional five matrix control samples spiked at ten times the LOQ (10 x LOQ) for each matrix.
3. For the ILV:
- a) Time required per twelve sample set (two extraction fortification samples, eight standards and 1 solvent blank) was estimated, but actual time required to perform a validation (thirteen sample set) trial was not reported (p. 20).
- b) Number of sample sets performed to complete a successful trial was not reported. The ILV may conduct up to three trials to generate acceptable performance data.
- c) Minor method modifications:
- i. Air sampling apparatus slightly modified, but shown to have recoveries comparable to ECM apparatus (pp. 18, 21-22; Figures 1-2, pp. 28-29; Figure 22, p. 49).
- ii. Different instrumentation reduced injection volume from 5 μ L to 2 μ L (p. 18).
- iii. Minor impurity peak responses were detected near the retention time of DMDS during analysis of reagent blank with individual ion chromatograms (p. 22; Appendix D, p. 86). ILV recommended that the GC/MS four ion quantitation (m/z 45, 61, 79, 94) used in the ECM be modified to three ion quantitation (m/z 64, 79, 94) to improve linearity and sensitivity of the analysis (p. 22; Appendix D, pp. 87-89).

Name and Dated Signature of Primary Reviewer


Gabe Rothman, Environmental Scientist
November 22, 2013

Name(s) and Dated Signature(s) of Secondary Reviewer(s)