



Standard Test Method for Determination of Thiodiglycol on Wipes by Solvent Extraction Followed by Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS)¹

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1. Scope

1.1 This procedure details the determination of thiodiglycol (TDG), also known as 2,2'-thiobis-ethanol, on wipes with 3,3'-thiodipropanol (TDP) as the surrogate. This method is based upon solvent extraction of wipes by either sonication or a pressurized fluid extraction (PFE) technique as an alternative option. The extract is filtered, concentrated and analyzed by liquid chromatography/tandem mass spectrometry (LC/MS/MS). TDG is qualitatively and quantitatively determined.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 The Method Detection Limit (MDL)² and Reporting Range³ for TDG are listed in [Table 1](#).

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:⁴

[D653 Terminology Relating to Soil, Rock, and Contained Fluids](#)

[D1193 Specification for Reagent Water](#)

¹ This test method is under the jurisdiction of ASTM Committee E54 on Homeland Security Applications and is the direct responsibility of Subcommittee E54.03 on Decontamination.

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² The MDL is determined following the Code of Federal Regulations, 40 CFR Part 136, Appendix B utilizing solvent extraction of wipes by sonication.

³ Reporting range concentrations are calculated from [Table 4](#) concentrations assuming a 10 µL injection of the lowest and highest level calibration standards with a 2 mL final extract volume. Volume variations will change the reporting limit and ranges. The reporting limit (RL), lowest concentration of the reporting range, is calculated from the concentration of the Level 1 calibration standard as shown in [Table 4](#).

⁴ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[D3694 Practices for Preparation of Sample Containers and for Preservation of Organic Constituents](#)

[D3740 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction](#)

[E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method in a Single Laboratory Using a Control Sample Program](#)

2.2 Other Documents:

[EPA Publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods](#)⁵

[The Code of Federal Regulations, 40 CFR Part 136, Appendix B](#)

3. Terminology

3.1 Abbreviations:

3.1.1 *mM*—millimolar, 1×10^{-3} moles/L

3.1.2 *ND*—non-detect

3.1.3 *SRM*—single reaction monitoring

3.1.4 *MRM*—multiple reaction monitoring

3.1.5 *VOA*—volatile organic analysis

4. Summary of Test Method

4.1 For TDG wipe analysis, samples are shipped to the lab between 0°C and 6°C. The samples are to be extracted, concentrated, and analyzed directly by LC/MS/MS within 7 days of collection. The handling, storage, preservation, and LC/MS/MS analysis are consistent between the two extraction procedures described in this test method. Only one extraction procedure is required, documenting which was performed.

4.2 TDG and TDP are identified by retention time and one SRM transition. The target analyte and surrogate are quantitated using the SRM transitions utilizing an external calibration. The final report issued for each sample lists the concentration of TDG and the TDP recovery.

⁵ Available from National Technical Information Service (NTIS), U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA, 22161 or at <http://www.epa.gov/epawaste/hazard/testmethods/index.htm>

TABLE 1 Method Detection Limit and Reporting Range

Analyte	CAS ^A Number	MDL (µg/wipe)	Reporting Range (µg/wipe)
Thiodiglycol	111-48-8	0.085	1-80
3,3'-Thiodipropanol (Surrogate)	10595-09-2	Not done for surrogates	1-80

^A Chemical Abstract Service (CAS), A division of the American Chemical Society, 2540 Olenangy River Road, Columbus, OH, 43202, USA.

5. Significance and Use

5.1 This is a performance based method, and modifications are allowed to improve performance.

5.1.1 Due to the rapid development of newer instrumentation and column chemistries changes to the analysis described in this standard are allowed as long as better or equivalent performance data result. Any modifications shall be documented and performance data generated. The user of the data generated by this Standard shall be made aware of these changes and given the performance data demonstrating better or equivalent performance.

5.2 TDG is a Schedule 2 compound under the Chemical Weapons Convention (CWC).⁶ Schedule 2 chemicals include those that are precursors to chemical weapons, chemical weapons agents or have a number of other non-military commercial uses. Schedule 2 chemicals can also be found in applications unrelated to chemical weapons. These chemicals are used as ingredients to produce insecticides, herbicides, lubricants, and some pharmaceutical products. TDG is a mustard gas precursor and a degradant as well as an ingredient in water-based inks, ballpoint pen inks, dyes, and some pesticides.

5.3 This method has been investigated for use on surface wipes. TDG is also a human metabolite resulting from sulfur mustard exposure but this method has not been investigated for such determinations.

6. Interferences

6.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other apparatus producing discrete artifacts or elevated baselines. All of these materials shall be demonstrated to be free from interferences by analyzing laboratory reagent blanks under the same conditions as samples.

6.2 All reagents and solvents shall be of pesticide residue purity or higher to minimize interference problems.

6.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences can vary considerably from sample source depending on variations of the sample matrix.

⁶ Additional information about CWC and thiodiglycol is available on the Internet at <http://www.opcw.org> (2009).

7. Apparatus

7.1 LC/MS/MS System:

7.1.1 *Liquid Chromatography (LC) System*⁷—A LC system is required in order to analyze samples. A LC system that is capable of performing at the flows, pressures, controlled temperatures, sample volumes, and requirements of the standard shall be used.

7.1.2 *Analytical Column*⁸—A column that achieves adequate resolution shall be used. The retention times and order of elution may change depending on the column used and need to be monitored. A reverse-phase analytical column with strong embedded basic ion-pairing groups was used to develop this test method.

7.1.3 *Tandem Mass Spectrometer (MS/MS) System*⁹—A MS/MS system capable of multiple reaction monitoring (MRM) analysis or a system that is capable of performing at the requirements in this standard shall be used.

7.2 *Pressurized Fluid Extraction (PFE) Device*¹⁰ (optional)—PFE devices with appropriately-sized extraction cells are available that will accommodate the wipe sample sizes used in this test method. Cells shall be made of stainless steel or other material capable of withstanding the pressure requirements (≥ 2000 psi) necessary for this procedure. A pressurized fluid extraction device shall be used that can meet the necessary requirements in this test method.

7.3 Glass Fiber Filters.¹¹

7.4 *Solvent Blowdown Device*, with 24- and 50-vial capacity trays and a water bath maintained at 50 to 60°C for analyte

⁷ A Waters Alliance® High Performance Liquid Chromatography (HPLC) System was used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the apparatus known to the committee at this time is Waters Corporation, Milford, MA 01757. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁸ A SIELC- Primesep SB™ 5 µm, 100 Å particle, 150 by 2.1 mm column was used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the apparatus known to the committee at this time is SIELC Technologies, Prospect Heights, IL 60070. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

⁹ A Waters Quattro micro™ API mass spectrometer was used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the apparatus known to the committee at this time is Waters Corporation, Milford, MA 01757. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹⁰ A Dionex Accelerated Solvent Extraction (ASE® 200) system with appropriately-sized extraction cells was used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the apparatus known to the committee at this time is Dionex Corporation, Sunnyvale, CA 94088. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

concentration from solvent volumes up to 50 mL or similar device shall be used.¹²

7.5 *Sonication Device*, capable of holding 40 mL vials.¹³

7.6 *Nitrogen Evaporation Device*, equipped with a water bath that can be maintained at 50°C for final analyte concentration (<10 mL volume) or similar shall be used.¹⁴

7.7 *Wipes*.¹⁵

7.8 *Filter Paper*.¹⁶

7.9 *Kuderna-Danish Vials (K-D)*, 10 mL.

7.10 *Amber VOA Vials*, 40 mL for sonication, or 60 mL for PFE.

7.11 *Filtration Device*:

7.11.1 *Hypodermic Syringe*—A luer-lock tip glass syringe capable of holding a syringe driven filter unit.

7.11.1.1 A 25 or 50 mL luer-lock tip glass syringe size is recommended in this test method.

7.11.2 *Filter Units*¹⁷—A filter unit of polytetrafluoroethylene (PTFE) 0.20 µm was used for the sonication extraction and

a polyvinylidene fluoride (PVDF) 0.22 µm was used for the PFE process. Either PTFE or PVDF filter units shall be used.

NOTE 1—Any filter unit brand may be used that meets the requirements of the test method.

8. Reagents and Materials

8.1 *Purity of Reagents*—High Performance Liquid Chromatography (HPLC) pesticide residue analysis and spectrophotometry grade chemicals shall be used in all tests. Unless indicated otherwise, it is intended that all reagents shall conform to the Committee on Analytical Reagents of the American Chemical Society.¹⁸ Other reagent grades may be used provided they are first determined to be of sufficiently high purity to permit their use without affecting the accuracy of the measurements.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to ASTM Type I of Specification D653. It shall be demonstrated that this water does not contain contaminants at concentrations sufficient to interfere with the analysis.

8.3 *Gases*—Nitrogen (purity ≥97%) and argon (purity ≥99.999%).

8.4 *Acetic Acid* (CH₃CO₂H, CAS# 64-19-7).

8.5 *Acetone* (CH₃COCH₃, CAS # 67-64-1).

8.6 *Acetonitrile* (CH₃CN, CAS # 75-05-8).

8.7 *Ammonium Formate* (NH₄CO₂H, CAS # 540-69-2).

8.8 *Formic Acid* (HCO₂H, CAS# 64-18-6).

8.9 *Methanol* (CH₃OH, CAS # 67-56-1).

8.10 *Thiodiglycol* (S(CH₂CH₂OH)₂, CAS # 111-48-8).

8.11 *3,3'-Thiodipropanol* (S(CH₂CH₂CH₂OH)₂, CAS # 10595-09-2).

8.12 *Drying Agent*.¹⁹

8.13 *Sand*—Reagent Grade sand, such as Ottawa Sand.

9. Hazards

9.1 Normal laboratory safety applies to this method. Analysts shall wear safety glasses, gloves, and lab coats when working in the lab. Analysts shall review the Material Safety Data Sheets (MSDS) for all reagents used in this method and shall be fully trained to perform the tests.

¹⁸ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulators*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

¹⁹ Varian – Chem Tube – Hydromatrix®, 1 kg (Part # 198003) was used to develop this test method and generate the precision and bias data presented in Section 17 by recommendation of the PFE manufacturer. The sole source of supply of the drying agent known to the committee at this time is Agilent Technologies, United States, 5301 Stevens Creek Blvd, Santa Clara, CA 95051. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend. (Note: Some drying agents have been shown to clog PFE transfer lines.)

¹¹ Whatman Glass Fiber Filters 19.8 mm, Part # 047017, specially designed for the PFE system¹⁰ were used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the apparatus known to the committee at this time is Dionex Corporation, Sunnyvale, CA 94088. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹² The sole source of supply of the apparatus (a TurboVap LV) known to the committee at this time is Caliper Life Sciences, Hopkinton, MA 01748. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹³ The sole source of supply of the apparatus (a Branson® Model 5510 Sonicator) known to the committee at this time is Branson Ultrasonics, Americas Headquarters, 41 Eagle Road, Danbury, CT 06810. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹⁴ The sole source of supply of the apparatus (N-Evap 24-port nitrogen evaporation device) known to the committee at this time is Organomation Associates Inc., West Berlin, MA 01503. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹⁵ Certi-Gauze™ pads, sterile, 3 by 3 in. (Part # 52639), were used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the pads known to the committee at this time is Certified Safety Mfg., Kansas City, MO. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹⁶ Whatman 42 ashless, 125 mm filter paper (Catalog # 1442 125) were used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the filter paper known to the committee at this time is Whatman Inc., Building 1, 800 Centennial Avenue, Piscataway, NJ 08854. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

¹⁷ An IC Millex®-LG Syringe Driven Filter Unit PTFE 0.20 µm (Catalog # SLLGC25NS) and Millex®-GV Syringe Driven Filter Unit PVDF 0.22 µm (Catalog # SLGV033NS) were used to develop this test method and generate the precision and bias data presented in Section 17. The sole source of supply of the filter units known to the committee at this time is Millipore Corporation, 290 Concord Road, Billerica, MA 01821. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.