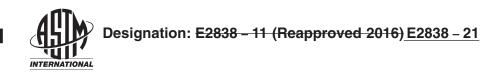
This document is not an ASTM standard and is intended only to provide the user of an ASTM standard an indication of what changes have been made to the previous version. Because it may not be technically possible to adequately depict all changes accurately, ASTM recommends that users consult prior editions as appropriate. In all cases only the current version of the standard as published by ASTM is to be considered the official document.



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

This standard is issued under the fixed designation E2838; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

### 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 Limitmethod detection limit (MDL)<sup>2</sup> and Reporting Rangereporting range<sup>3</sup> 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.

1.5 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

### 2. Referenced Documents

2.1 ASTM Standards:<sup>4</sup>

D653 Terminology Relating to Soil, Rock, and Contained Fluids D1193 Specification for Reagent Water

D3694D2777 PracticesPractice for PreparationDetermination of Sample Containers and for Preservation of Organic ConstituentsPrecision and Bias of Applicable Test Methods of Committee D19 on Water

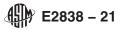
<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

Current edition approved June 1, 2016 May 1, 2021. Published July 2016 May 2021. Originally approved in 2011. Last previous edition approved in  $\frac{2011}{2016}$  as  $\frac{22838 - 11}{2016}$ . DOI:  $\frac{10.1520}{22016}$  DOI:  $\frac{10.1520}{2016}$  DOI: 0.1520 DOI: 0.1520 D

<sup>&</sup>lt;sup>2</sup> The MDL is determined following the Code of Federal Regulations, 40 CFR Part 136, Appendix B utilizing solvent extraction of wipes by sonication.

 $<sup>^{3}</sup>$  Reporting range concentrations are calculated from Table 4 concentrations assuming a 10  $\mu$ L 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.

<sup>&</sup>lt;sup>4</sup> 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.



#### TABLE 1 Method Detection Limit and Reporting Range

		•	0 0
Analyte	CAS <sup>A</sup> Number	MDL (µg/wipe)	Reporting Range (µg/wipe)
Thiodiglycol	<del>111-48-8</del>	<del>0.085</del>	<del>1-80</del>
3,3'-Thiodipropanol	<del>10595-09-2</del>	Not done	<del>1-80</del>
(Surrogate)		for surrogates	
TABLE I MEL	Iou Delection	Limit and Rep	onling hange
Analyte	CAS <sup>A</sup> Number	MDL	Reporting Range
	Number	(µg/wipe)	
			(µg/wipe)
Thiodiglycol	111-48-8	0.085	<u>(µg/wipe)</u>
Thiodiglycol 3,3'-Thiodipropanol	<u>111-48-8</u> 10595-09-2	0.085 Not done	

<sup>A</sup>-Chemical Abstract Service (CAS), A<u>a</u> division of the American Chemical Society, 2540 Olentangy River Road, Columbus, OH, 43202, USA.

 D3740D5681 Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and ConstructionTerminology for Waste and Waste Management
E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques

E2554 Practice for Estimating and Monitoring the Uncertainty of Test Results of a Test Method Using Control Chart Techniques 2.2 *Other Documents:* 

EPA Publication SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods<sup>5</sup> The Code of Federal Regulations, 40 CFR Part 136, <u>Appendix B</u> <u>Appendix B</u> <u>The Code of Federal Regulations</u>

### 3. Terminology

3.1 Definitions—For definitions of terms used in this test method, refer to Terminology D5681.

3.2 Abbreviations:

3.2.1 *mM*—millimolar,  $1 \times 10^{-3}$  moles/L m c s f and a

3.2.2 ND—non-detect

3.2.3 SRM—single reaction monitoring

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3.2.4 MRM—multiple reaction monitoring ndards/sist/49ae9ef3-0242-43a2-947b-dac3cc54a5e4/astm-e2838-21

3.2.5 VOA—volatile organic analysis

### 4. Summary of Test Method

4.1 For TDG wipe analysis, samples are shipped to the lab between  $\theta^{\circ}C\underline{0} \circ C$  and  $\underline{6}^{\circ}C\underline{.6} \circ C$ . The samples are to be extracted, concentrated, and analyzed directly by LC/MS/MS within 7<u>seven</u> 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.

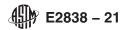
4.2 TDG and TDP are identified by retention time and one SRM transition. The target analyte and surrogate are quantitated using the SRM transitions utilizing an external calibration. The final report issued for each sample lists the concentration of TDG and the TDP recovery.

### 5. Significance and Use

5.1 This is a performance based 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

<sup>&</sup>lt;sup>5</sup> 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



performance data generated. The user of the data generated by this <u>Standardstandard</u> 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).<sup>6</sup> 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.

### 7. Apparatus

7.1 LC/MS/MS System:

7.1.1 *Liquid Chromatography (LC) System*<sup>7</sup>—A<u>An</u> LC system is required in order to analyze samples. A<u>An</u> 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*<sup>8</sup>—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*<sup>9</sup>—A<u>An</u> 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*<sup>10</sup> (optional)—PFE devices with appropriately-sized 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

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

<sup>&</sup>lt;sup>7</sup> 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. Any HPLC system that produces results that meet or exceed the performance criteria of this test method <sup>1</sup>-which you may attend may be used.

<sup>&</sup>lt;sup>8</sup> A SIELC- Primesep <u>SBT<sup>M</sup>SB</u> 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 <u>SIELC</u> Technologies, Prospect Heights, <u>IL 60070</u>. 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 eommittee, Any column that produces results that meet or exceed the performance criteria of this test method <u>-</u>-which you may attend.may be used.

<sup>&</sup>lt;sup>9</sup> A Waters Quattro micro<sup>™</sup>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, Any mass spectrometer that produces results that meet or exceed the performance criteria of this test method <sup>1</sup>/<sub>2</sub> which you may attend.may be used.

<sup>&</sup>lt;sup>10</sup> A Dionex Accelerated Solvent Extraction (ASE@(ASE 200) system with appropriately-sized 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 eonsideration at a meeting of the responsible technical committee, Any extraction system that produces results that meet or exceed the performance criteria of this test method <sup>1</sup> which you may attend.may be used.



other material capable of withstanding the pressure requirements ( $\geq$ 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.<sup>11</sup>

7.4 Solvent Blowdown Device, with 24- and 50-vial capacity trays and a water bath maintained at 50 to  $60^{\circ}C_{0}$  for analyte concentration from solvent volumes up to 50 mL or similar device shall be used.<sup>12</sup>

7.5 Sonication Device, capable of holding 40 mL vials.<sup>13</sup>

7.6 *Nitrogen Evaporation Device*, equipped with a water bath that can be maintained at  $\frac{50^{\circ}C}{50^{\circ}C}$  for final analyte concentration (<10 mL volume) or similar shall be used.<sup>14</sup>

7.7 Wipes.<sup>15</sup>

7.8 Filter Paper.<sup>16</sup>

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

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

7.11 *Filtration Device:* 

# iTeh Standards

7.11.1 Hypodermic Syringe-A luer-lock tip glass syringe capable of holding a syringe driven 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*<sup>17</sup>—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.

### <u>ASTM E2838-2</u>

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<sup>11</sup> Whatman Glass Fiber Filters 19.8 mm, Part #<u>No.</u> 047017, specially designed for the PFE system<sup>10</sup> 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, Any filter that produces results that meet or exceed the performance criteria of this test method <u>which you may attend.may be used</u>.

<sup>12</sup> The sole source of supply of the apparatus (a TurboVap LV) known to the committee at this time is <u>A</u> TurboVap LV by 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: 01748 was used. Any evaporation system that produces results that meet or exceed the performance criteria of which you may attend; this test method may be used.

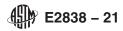
<sup>13</sup> The sole source of supply of the apparatus (a Bransonic® Model 5510 Sonicator) known to the committee at this time is <u>A Bransonic Model 5510 Sonicator</u> by Branson Ultrasonics, Americas Headquarters, 41 Eagle Road, Danbury, CT <del>06810. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, <u>06810</u> was used. Any sonicator that produces results that meet or exceed the performance criteria of <u>which you may attend</u> this test method may be used.</del>

<sup>14</sup> The sole source of supply of the apparatus (N-Evap <u>An N-Evap</u> 24-port nitrogen evaporation device) known to the committee at this time is <u>device by</u> 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.01503 was used. Any evaporation system that produces results that meet or exceed the performance criteria of<sup>1</sup> which you may attend this method may be used.

<sup>15</sup> Certi-Gauze<sup>™</sup>Certi-Gauze pads, sterile, 3 by 3 in. (Part #<u>No.</u> 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;<u>Any</u> gauze pad that produces results that meet or exceed the performance criteria of this test method <sup>1</sup>-which you may attend.<u>may</u> be used.

<sup>16</sup> Whatman 42 ashless, 125 mm filter paper (Catalog #<u>No.</u> 1442 125) werewas 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, Piseataway, 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, <u>Any filter paper that produces results that meet or exceed the performance criteria of this test method</u> <u>-which you may attend.may</u> be used.

<sup>17</sup> An IC Millex®-LG Syringe Driven-Millex-LG Syringe-Driven Filter Unit PTFE 0.20 μm (Catalog #<u>No.</u> SLLGC25NS) and <u>Millex®-GV-Syringe Driven-Millex-GV</u> Syringe-Driven Filter Unit PVDF 0.22 μm (Catalog #<u>No.</u> 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, Any filter that produces results that meet or exceed the performance criteria of this test method <sup>1</sup>-which you may attend.may be used.



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

### 8. Reagents and Materials

8.1 *Purity of Reagents*—High Performance Liquid Chromatographyperformance 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.<sup>18</sup> 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 <u>D653D1193</u>. 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%)≥97%) and argon (purity ≥99.999%).≥99.999%).
- 8.4 Acetic Acid (CH<sub>3</sub>CO<sub>2</sub>H, CAS# CAS No. 64-19-7).
- 8.5 *Acetone* (CH<sub>3</sub>COCH<sub>3</sub>, CAS #<u>No.</u> 67-64-1).
- 8.6 Acetonitrile (CH<sub>3</sub>CN, CAS #<u>No.</u> 75-05-8).
- 8.7 Ammonium Formate (NH<sub>4</sub>CO<sub>2</sub>H, CAS #No. 540-69-2).
- 8.8 Formic Acid (HCO<sub>2</sub>H, CAS# CAS No. 64-18-6).
- 8.9 Methanol (CH<sub>3</sub>OH, CAS #<u>No.</u> 67-56-1).
- 8.10 *Thiodiglycol* (S(CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub>, CAS #<u>No.</u> 111-48-8).
- 8.11 3,3'-Thiodipropanol (S(CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>OH)<sub>2</sub>, CAS #<u>No.</u> 10595-09-2).

8.12 Drying Agent.<sup>19</sup>

8.13 Sand—Reagent Gradegrade sand, such as Ottawa Sand.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.

### 10. Glassware Washing, Sampling, and Preservation

10.1 Glassware Washing—All glassware is washed in hot tap water with a detergent and rinsed in hot water conforming to ASTM

<sup>&</sup>lt;sup>18</sup> 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 <u>AnnualAnalar</u> Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulators, Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

<sup>&</sup>lt;sup>19</sup> Varian – Chem Tube – Hydromatrix, Hydromatrix, 1 kg (Part #<u>No.</u> 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, Any drying agent that produces results that meet or exceed the performance criteria of this test method <sup>1</sup>-which you may attend. (Note: Some drying agents have been shown to clog PFE transfer lines.)may be used.

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Type I of Specification  $\frac{D653D1193}{D1193}$ . The glassware is then dried and heated in an oven at  $\frac{250^{\circ}C250^{\circ}C}{C250^{\circ}C}$  for 15 to 30 minutes.min. All glassware is subsequently cleaned with acetone and methanol, respectively.

10.2 *Sampling*—The wipe sample is folded and placed into a 40 mL pre-cleaned amber glass VOA vial with a PTFE-lined cap in the field. The wipe is shipped to the laboratory between  $\frac{0^{\circ}C_{0} \circ C}{C}$  and  $\frac{6^{\circ}C_{0} \circ C}{C}$ . The required surrogate and matrix spike solutions are added to the wipe in the VOA vial at the laboratory. Field blanks are needed to follow conventional sampling practices.

10.3 *Preservation*—Store samples between  $\theta^{\circ}C\underline{0}^{\circ}C$  and  $\underline{6}^{\circ}C\underline{6}^{\circ}C$  from the time of collection until analysis. Analyze the sample within 7<u>seven</u> days of collection.

### 11. Preparation of LC/MS/MS

11.1 LC Chromatograph Operating Conditionschromatograph operating conditions for the LC used to develop this test method:<sup>7</sup>

11.1.1 Injection volumes of all calibration standards and samples are  $10 \ \mu$ L. The first sample analyzed after the calibration curve is a blank to ensure there is no carry-over. The gradient conditions for the liquid chromatograph are shown in Table 2.

11.1.2 Temperatures—Column, <del>30°C; 30 °C;</del> Sample compartment, <del>15°C.<u>15</u> °C.</del>

11.1.3 Seal Wash-Solvent: 50% Acetonitrile/50% 50% Acetonitrile/50% Water; Time: 5 minutes.min.

11.1.4 *Needle Wash*—Solvent: 50% Acetonitrile/50% 50 % Acetonitrile/50 % Water; Normal Wash, normal wash, approximately a 13 second 13-s wash time.

11.1.5 Autosampler Purge—Three loop volumes. en Standards

11.1.6 Specific instrument manufacturer wash and purge specifications shall be followed in order to eliminate sample carry-over in the analysis.

11.2 Mass Spectrometer Parameters:<sup>9</sup> **Document Preview** 

11.2.1 To acquire the maximum number of data points per SRM channel while maintaining adequate sensitivity, the tune parameters shall be optimized according to the instrument. Each peak requires at least <u>10ten</u> scans per peak for adequate quantitation. This standard contains one target compound and one surrogate which are in different SRM experiment windows in order to optimize the number of scans and sensitivity. Variable parameters regarding retention times, SRM transitions, and cone and collision energies are shown in Table 3 for the mass spectrometer used to develop this test method. Other mass spectrometer parameters used in the development of this method are listed below:

The instrument is set in the Electrospray (+) positive source setting. Capillary Voltage: 3.5 kV Cone: Variable depending on analyte (Table 3) Extractor: 2 V RF Lens: 0.2 V Source Temperature: 120°C Source Temperature: 120 °C Desolvation Temperature: 300°C

### TABLE 2 Gradient Conditions for Liquid Chromatography

Time	Flow	Percent	Percent	Percent 500 mM Ammonium
(min)	(µL/min)	CH₃CN	Water	<del>Formate/</del> <del>2%</del> Formate/2 %
				Formic Acid
0	300	0	95	5
2	300	0	95	5
3	300	50	45	5
6	300	90	5	5
10	300	90	5	5
12	300	0	95	5
16	300	0	95	5

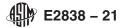


TABLE 3 Retention Times, SRM Transitions, and Analyte-Specific Mass Spectrometer Parameters

Analyte	SRM Mass	Retention	Cone	Collision
	Transition (m/z)	Time	Voltage	Energy
	(Parent > Product)	(min)	(Volts)	(eV)
Thiodiglycol	123.1 > 104.9	2.75	18	5
3,3'-Thiodipropanol	151.2 > 133.1	5.75	19	8

Desolvation Temperature: 300 °C Desolvation Gas Flow: 500 L/hr Desolvation Gas Flow: 500 L/h Cone Gas Flow: 25 L/hr Cone Gas Flow: 25 L/h Low Mass Resolution 1: 14.5 High Mass Resolution 1: 14.5 Ion Energy 1: 0.5 V Entrance Energy: -1 V Entrance Energy: -1 V Collision Energy: Variable depending on analyte (Table 3) Exit Energy: 2 V Low Mass Resolution 2: 14.5 High Mass resolution 2: 14.5 High Mass Resolution 2: 14.5 Ion Energy 2: 0.5 V Multiplier: 650 V Gas Cell Pirani Gauge: 0.33 Pa Inter-Channel Delay: 0.02 s Inter-Scan Delay: 0.1 s Repeats: 1 Span: 0 Daltons Dwell: 0.1 s

### 12. Calibration and Standardization

12.1 The mass spectrometer shall be calibrated per manufacturer specifications before analysis. In order to obtain valid and accurate analytical values within the confidence limits, the following procedures shall be followed when performing the test method.

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12.2 *Calibration and Standardization*—To calibrate the instrument, analyze eight calibration standards containing the eight concentration levels of TDG and TDP in water prior to analysis as shown in Table 4. A calibration stock standard solution is prepared from standard materials or purchased as certified solutions. Aliquots of Level 8 are then diluted with water to prepare the desired calibration levels in 2 mL amber glass LC vials. The calibration vials shall be used within 24 hoursh to ensure optimum results. Stock calibration standards are routinely replaced every six months if not previously discarded for quality control failure. The analyst is responsible for recording initial component weights carefully when working with pure materials and correctly carrying the weights through the dilution calculations. Calibration standards are not filtered.

12.2.1 Inject each standard and obtain its chromatogram. An external calibration is used <u>in</u> monitoring the SRM transition of each analyte. Calibration software is utilized to conduct the quantitation of the target analyte and surrogate. The SRM transition of each analyte is used for quantitation and confirmation. Confirmation occurs by isolating the parent ion, fragmenting it to the product ion, and relating it to the retention time in the calibration standard.

12.2.2 The calibration software manual shall be consulted to use the software correctly. The quantitation method is set as an external calibration using the peak areas in ppb or ppm units as long as the analyst is consistent. Concentrations may be calculated using the data system software to generate linear regression or quadratic calibration curves. The calibration curves may be either linear or quadratic depending on your instrument. Forcing the calibration curve through the origin is not recommended. Each calibration point used to generate the curve shall have a calculated percent deviation less than 30%30 % from the generated curve.

TABLE 4 Concentrations of Calibration Standards ( $\mu$ g/L)								
Analyte/Surrogate	LV 1	LV 2	LV 3	LV 4	LV 5	LV 6	LV 7	LV 8
Thiodiglycol	500	1000	2000	4000	8000	16 000	32 000	40 000
3,3'-Thiodipropanol	500	1000	2000	4000	8000	16 000	32 000	40 000