



Standard Test Method for Determination of Bisphenol A in Soil, Sludge and Biosolids by Pressurized Fluid Extraction and Analyzed by Liquid Chromatography/Tandem Mass Spectrometry¹

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1. Scope

1.1 This procedure covers the determination of Bisphenol A (BPA) in soil, sludge, and biosolids. This test method is based upon solvent extraction of a soil matrix by pressurized fluid extraction (PFE). The extract is filtered and analyzed by liquid chromatography/tandem mass spectrometry (LC/MS/MS). BPA is qualitatively and quantitatively determined by this test method.

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 Limit² (MDL), electrospray ionization (ESI) mode, and Reporting Range³ for BPA 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 D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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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 soil by PFE. 10 gram sample of Ottawa Sand was utilized. A detailed process determining the MDL is explained in the reference and is beyond the scope of this test method to be explained here.

³ Reporting range concentration is calculated from Table 4 concentrations assuming a 25 μ L injection of the Level 1 calibration standard for BPA, and the highest level calibration standard with a 5 mL final extract volume of a 10 gram soil sample. Volume variations will change the reporting limit and ranges.

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

D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water

D5681 Terminology for Waste and Waste Management

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 Methods⁵

The Code of Federal Regulations 40 CFR Part 136, Appendix B⁶

3. Terminology

3.1 Definitions:

3.1.1 *Bisphenol A (BPA)*, *n*—2,2-bis(4-hydroxyphenyl) propane.

3.1.2 *Bisphenol A (propane-D₆) (BPA-D₆)*, *n*—deuterium labeled Bisphenol A where the two methyl moieties contain all ²H and is used as a surrogate in this method.

3.1.3 *filter unit*, *n*—in this test method, a filter that is supported with a housing that is inert to the solvents used as described in 7.4 of this test method.

3.1.4 *filtration device*, *n*—a device used to remove particles from the extract that may clog the liquid chromatography system as described in 7.4 of this test method.

3.1.5 *glass fiber filter*, *n*—a porous glass fiber material onto which solid particles present in the extraction fluid, which flows through it, are largely caught and retained, thus removing them from the extract.

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>

⁶ Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, <http://www.access.gpo.gov>.

TABLE 1 Method Detection Limit and Reporting Range

Analyte	ESI Mode	MDL (PPB)	Reporting Range (PPB)
Bisphenol A	Negative	2.8	10-250

3.1.6 *hypodermic syringe, n*—in this test method, a luer-lock-tipped glass syringe capable of holding a syringe-driven filter unit as described in 7.4 of this test method.

3.1.7 *pressurized fluid extraction, n*—the process of transferring the analytes of interest from the solid matrix, a soil, into the extraction solvent using pressure and elevated temperature.

3.1.8 *reporting range, n*—the quantitative concentration range for an analyte in this test method.

3.2 Abbreviations:

3.2.1 *BPA*—Bisphenol A

3.2.2 *LC*—Liquid Chromatography

3.2.3 *LCS/LCSD*—Laboratory Control Spike/Laboratory Control Spike Duplicate

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

3.2.5 *MRM*—Multiple Reaction Monitoring

3.2.6 *MS*—Matrix Spike

3.2.7 *NA*—not available

3.2.8 *ND*—non-detect

3.2.9 *PFE*—Pressurized Fluid Extraction

3.2.10 *PPB*—Parts Per Billion

3.2.11 *QC*—Quality Control

3.2.12 *RL*—Reporting Limit

3.2.13 *SD*—Standard Deviation

3.2.14 *SRM*—Single Reaction Monitoring

3.2.15 *VOA*—Volatile Organic Analysis

4. Summary of Test Method

4.1 For BPA analysis in soil, sludge and biosolid, samples are shipped to the lab between 0°C and 6°C. The samples are to be extracted and filtered within 14 days of collection, and analyzed by LC/MS/MS within 14 days of extraction.

4.2 BPA and the surrogate (BPA-D₆) are identified by retention time and one SRM transition. The target analytes and surrogates are quantitated using the SRM transitions utilizing an external calibration. The final report issued for each sample lists the concentration of BPA and surrogate recovery.

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 test method 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 test method shall be made aware of these changes and given the performance data demonstrating better or equivalent performance.

5.2 The first reported synthesis of BPA was by the reaction of phenol with acetone by Zincke.⁷ BPA has become an important high volume industrial chemical used in the manufacture of polycarbonate plastic and epoxy resins. Polycarbonate plastic and resins are used in numerous products including electrical and electronic equipment, automobiles, sports and safety equipment, reusable food and drink containers, electrical laminates for printed circuit boards, composites, paints, adhesives, dental sealants, protective coatings and many other products.⁸

5.3 The environmental source of BPA is predominantly from the decomposition of polycarbonate plastics and resins. BPA is not classified as bio-accumulative by the U.S. Environmental Protection Agency and will biodegrade. BPA has been reported to have adverse effects in aquatic organisms and may be released into environmental waters directly at trace levels through landfill leachate and sewage treatment plant effluents. This method has been investigated for use with soil, sludge, and biosolids.

5.4 The land application of biosolids has raised concerns over the fate of BPA in the environment and a standard method is needed to monitor concentrations. This method has been investigated for use with various soils.

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 are 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*⁹—A complete 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 Zincke, T., 1905, "Mitteilungen aus dem chemischen Laboratorium der Universitat Marburg," *Justus Leibigs Annals Chemie*, Vol 343, pp. 75–79.

⁸ Additional information about BPA is available on the Internet at <http://www.bisphenol-a.org> (2008).

⁹ A Waters Acquity UPLC® H-Class System was used to develop this test method and generate the precision and bias data presented in Section 16. Waters Corporation, Milford, MA 01757. Instrumentation from other vendors may also be able to generate similar method performance.

7.1.2 *Analytical Column*¹⁰—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 that combines the desirable characteristics of a reversed-phase HPLC column with the ability to separate polar compounds was used to develop this test method.

7.1.3 *Tandem Mass Spectrometer (MS/MS) System*¹¹—A MS/MS system capable of multiple reaction monitoring (MRM) analysis or any system that is capable of performing at the requirements in this test method shall be used.

7.2 *Pressurized Fluid Extraction Device (PFE)*:¹²

7.2.1 A PFE system was used for this test method with appropriately-sized extraction cells. Cells are available that will accommodate the 10 g 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.

7.2.2 Glass Fiber Filters.¹³

7.2.3 Amber VOA vials-60 mL for sample extracts for PFE.

7.3 *Organic Solvent Evaporation Device*.¹⁴

7.4 *Filtration Device*:

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

7.4.1.1 A 10 mL Lock Tip Glass Syringe size is recommended since a 3 mL sample extract results after blow-down.

7.4.2 *Filter Unit*¹⁵—Filter units of polyvinylidene fluoride (PVDF) with a glass fiber prefilter were used to filter the PFE extracts.

7.4.3 *Discussion*—A filter unit shall be used that meets the requirements of the test method.

¹⁰ A Waters-UPLC® T3, 100 mm x 2.1 mm, 1.8 μ m particle size, was used to develop this test method and generate the precision and bias data presented in Section 16. Waters Corporation, Milford, MA 01757. Columns from other vendors that are able to generate similar method performance and that achieve adequate resolution may be used. A guard column was also used, VanGuard™ Pre-Column, 2.1 x 5 mm, 1.8 μ m particle size.

¹¹ A Waters Quattro micro™ API mass spectrometer was used to develop this test method and generate the precision and bias data presented in Section 16. Waters Corporation, Milford, MA 01757. Instrumentation from other vendors may also be able to generate similar method performance.

¹² 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 16. Dionex Corporation, Sunnyvale, CA 94088. Instrumentation from other vendors may also be able to generate similar method performance.

¹³ Whatman Glass Fiber Filters 19.8 mm, Dionex Corporation, Part # 047017 specially designed for the PFE system¹¹ were used to develop this test method and generate the precision and bias data presented in Section 16. Filters from other vendors may also be able to generate similar method performance.

¹⁴ A TurboVap LV was used in this test method from Caliper Life Sciences, Hopkinton, MA 01748 and an N-Evap 24-port nitrogen evaporation device was used in this test method from Organomation Associates Inc., West Berlin, MA 01503. In-house built or devices from other vendors may also be able to generate similar method performance.

¹⁵ Pall®-Acrodisc® Premium 25 mm Syringe Filter with GxP/0.2 μ m PVDF Membrane (Pall Corporation, Catalog # AP-4793, were used to develop this test method and generate the precision and bias data presented in Section 16. Filters from other vendors may also be able to generate similar method performance.

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.¹⁶ 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 mean reagent water conforming to ASTM Type I of Specification D1193. It must 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 Acetonitrile (CH₃CN, CAS # 75-05-8).

8.5 Ethyl Acetate (CH₃COOC₂H₅, CAS # 141-78-6).

8.6 2-Propanol (C₃H₈O, CAS # 67-63-0).

8.7 Methanol (CH₃OH, CAS # 67-56-1).

8.8 Ammonium Acetate (CH₃CO₂NH₄, CAS # 631-61-8).

8.9 Bisphenol A (C₁₅H₁₆O₂, 2,2'-Bis(4-hydroxyphenyl)propane, CAS # 80-05-7).

8.10 Bisphenol A (Propane-D₆) represents deuterium labeled Bisphenol A where the two methyl moieties contain all ²H.

8.10.1 *Discussion*—BPA-D₆ is used as a surrogate in this test method.

8.11 Ottawa Sand (CAS # 14808-60-7) or equivalent.

8.12 Drying Agent.¹⁷

8.13 Sodium sulfate (Na₂SO₄, CAS # 7757-82-6).

8.14 Sodium Chloride (NaCl, CAS # 7647-14-5).

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 test method and shall be fully trained to perform this test method.

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, then ASTM

¹⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the United States *Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

¹⁷ Varian—Chem Tube—Hydromatrix®, 1kg (Part # 198003) was used to develop this test method and generate the precision and bias data presented in Section 16 by recommendation of the PFE manufacturer. Drying agent from other vendors may also be able to generate similar method performance. Note—Some drying agents have been shown to clog PFE transfer lines.