



Designation: D7574 – 23

Standard Test Method for Determination of Bisphenol A in Environmental Waters by Liquid Chromatography/Tandem Mass Spectrometry¹

This standard is issued under the fixed designation D7574; 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 (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of bisphenol A (BPA) extracted from water utilizing solid phase extraction (SPE), separated using liquid chromatography (LC) and detected with tandem mass spectrometry (MS/MS). BPA is qualitatively and quantitatively determined by this method. This test method adheres to multiple reaction monitoring (MRM) mass spectrometry.

1.2 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) and reporting limit (RL) 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, 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:*²

[D1129 Terminology Relating to Water](#)

[D1193 Specification for Reagent Water](#)

[D2777 Practice for Determination of Precision and Bias of Applicable Test Methods of Committee D19 on Water](#)

¹ This test method is under the jurisdiction of ASTM Committee [D19](#) on Water and is the direct responsibility of Subcommittee [D19.06](#) on Methods for Analysis for Organic Substances in Water.

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² 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](#)

[D3856 Guide for Management Systems in Laboratories Engaged in Analysis of Water](#)

[D5847 Practice for Writing Quality Control Specifications for Standard Test Methods for Water Analysis](#)

[D5905 Practice for the Preparation of Substitute Wastewater](#)

2.2 *Other Documents:*³

[40 CFR Part 136, Appendix B Definition and Procedure for the Determination of the Method Detection Limit](#)

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology [D1129](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *environmental water, n*—shall refer to water tested using this method. See Section [5](#).

3.2.2 *independent reference material, IRM, n*—a material of known purity and concentration obtained either from the National Institute of Standards and Technology (NIST) or other reputable supplier; the IRM must be obtained from a different lot of material than is used for calibration.

3.3 *Acronyms:*

3.3.1 *CCC, n*—Continuing Calibration Check

3.3.2 *IC, n*—Initial Calibration

3.3.3 *LC, n*—Liquid Chromatography

3.3.4 *LCS/LCSD, n*—Laboratory Control Sample/Laboratory Control Sample Duplicate

3.3.5 *MDL, n*—Method Detection Limit

3.3.6 *MeOH, n*—Methanol

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

3.3.8 *MRM, n*—Multiple Reaction Monitoring

3.3.9 *MS/MSD, n*—Matrix Spike/Matrix Spike Duplicate

3.3.10 *NA, adj*—Not Available

³ 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 MDL and Reporting Range

Analyte	MDL ^A (ng/L)	Reporting Range ^B (ng/L)
Bisphenol A	5	20–600

^A MDL determined following the Code of Federal Regulations, 40 CFR Part 136, Appendix B.

^B Lowest point of the reporting range, reporting limit, is calculated from the LV 1 concentration calibration standard in Table 4. Fig. 1 displays the signal/noise ratio at the reporting limit.

- 3.3.11 *ND*, *n*—non-detect
- 3.3.12 *P&A*, *n*—Precision and Accuracy
- 3.3.13 *PPB*, *n*—parts per billion
- 3.3.14 *PPT*, *n*—parts per trillion
- 3.3.15 *QA*, *adj*—Quality Assurance
- 3.3.16 *QC*, *adj*—Quality Control
- 3.3.17 *RL*, *n*—Reporting Limit
- 3.3.18 *RSD*, *n*—Relative Standard Deviation
- 3.3.19 *RT*, *n*—Retention Time
- 3.3.20 *SDS*, *n*—Safety Data Sheets
- 3.3.21 *SRM*, *n*—Single Reaction Monitoring
- 3.3.22 *SS*, *n*—Surrogate Standard
- 3.3.23 *TC*, *n*—Target Compound
- 3.3.24 μM , *n*—micromolar, 1×10^{-6} moles/L
- 3.3.25 *VOA*, *n*—Volatile Organic Analysis

4. Summary of Test Method

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

4.2 Solid phase extraction is used to extract water samples.

4.2.1 *Solid Phase Extraction*—250 mL volume of sample adjusted to pH 2 is extracted using a solid phase extraction cartridge. The resulting methyl tert-butyl ether (MTBE) extract is solvent exchanged into methanol, concentrated to a volume of 0.2 mL, brought to a final volume of 1 mL with water and then analyzed by LC/MS/MS operated in the multiple reaction monitoring (MRM) mode.

4.3 The target compound, surrogate and internal standards are identified by retention time and two SRM transitions. The target analyte and surrogate are quantitated using the primary SRM transitions utilizing internal standard calibration. The final report issued for each sample lists the concentration of BPA and the bisphenol A (Ring-13C12) surrogate recovery.

5. Significance and Use

5.1 The first reported synthesis of BPA was by the reaction of phenol with acetone by Zincke.⁴ 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.⁵

5.2 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 POTW effluents. This method has been investigated for use with surface water and secondary and tertiary POTW effluent samples therefore, it is applicable to these matrices only. It has not been investigated for use with salt water or solid sample matrices.

6. Interferences

6.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other apparatus producing discrete artifacts or elevated baselines. The use of plastic supplies and equipment must be avoided because they may contain BPA. All of these materials are routinely demonstrated to be free from interferences by analyzing laboratory reagent blanks under the same conditions as the samples.

6.2 All glassware is washed in hot water with a detergent, rinsed in hot water followed by distilled water. The glassware is then dried and heated in an oven at 250 °C for 15 min to 30 min. All glassware is subsequently cleaned with acetone, then methanol. Detergents in plastic containers that contain BPA must not be used.

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

6.4 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 to sample source, depending on variations of the sample matrix.

7. Apparatus

7.1 *LC/MS/MS System*:

7.1.1 *Liquid Chromatography System*—A complete LC system is needed in order to analyze samples. This should include a sample injection system, a solvent pumping system capable of mixing solvents, a sample compartment capable of maintaining required temperature and a temperature controlled column compartment. A system that is capable of performing at the flows, pressures, controlled temperatures, sample volumes and requirements of the standard may be used.

7.1.1.1 *Columns*—Column to separate instrument background (isolator): A short (2.1 mm $\times$ 50 mm) C8 or C18 column stable at higher pH up to 12 and an analytical column.⁶

⁵ Additional information about BPA is available at <http://www.bisphenol-a.org> (2008).

⁶ A Waters ACQUITY UPLC (a trademark of the Waters Corporation, Milford, MA) HSS T3, 1.8 μm , 2.1 mm $\times$ 50 mm column was used, if you are aware of an alternative column that meets the performance of the standard, 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.

⁴ Zincke, T., 1905, "Mittheilungen aus dem chemischen Laboratorium der Universitat Marburg," *Justus Leibigs Annals Chemie*, Vol. 343, pages 75–79.