Designation: D7858 – 13 (Reapproved 2018)

Standard Test Method for Determination of Bisphenol A in Soil, Sludge, and Biosolids by Pressurized Fluid Extraction and Analyzed by Liquid Chromatography/Tandem Mass Spectrometry[1]

This standard is issued under the fixed designation D7858; 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 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 limit (MDL),[2] electrospray ionization (ESI) mode, and reporting range[3] 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:*[4]

D653 Terminology Relating to Soil, Rock, and Contained Fluids
D1193 Specification for Reagent Water
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 SW-846 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods[5]
40 CFR Part 136, Appendix B Definition and Procedure for the Determination of the Method Detection Limit[6]

3. Terminology

3.1 *Definitions:*

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

3.1.2 *Bisphenol A (propane-D_6) (BPA-D_6), n*—deuterium labeled Bisphenol A where the two methyl moieties contain all ^2H and is used as a surrogate in this method.

[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. A 10-g 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-µL 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-g 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.

[5] Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

[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

3.1.3 *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.

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-D_6) 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] Zincke, T., "Mittheilungen aus dem chemischen Laboratorium der Universitat Marburg," *Justus Leibigs Annals Chemie*, Vol 343, 1905, pp. 75–79.

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

7.1.1 *Liquid Chromatography (LC) System*[9]—A complete LC system is required in order to analyze samples. An 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*[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.

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 (CH_3CN, CAS # 75-05-8).

8.5 Ethyl acetate ($CH_3COOC_2H_5$, CAS # 141-78-6).

8.6 2-Propanol (C_3H_8O, CAS # 67-63-0).

8.7 Methanol (CH_3OH, CAS # 67-56-1).

8.8 Ammonium acetate ($CH_3CO_2NH_4$, CAS # 631-61-8).

8.9 Bisphenol A ($C_{15}H_{16}O_2$, 2,2'-Bis(4-hydroxyphenyl) propane, CAS # 80-05-7).

8.10 Bisphenol A (Propane-D_6) represents deuterium labeled Bisphenol A where the two methyl moieties contain all ^2H.

8.10.1 *Discussion*—BPA-D_6 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 (Na_2SO_4, CAS # 7757-82-6).

[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.

[10] A Waters-UPLC® T3, 100 mm x 2.1 mm, 1.8-µm 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 × 5 mm, 1.8-µm 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 system[11] 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 GxF/0.2 µm 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.

[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®, 1 kg (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.

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 Type I of Specification D1193. The glassware is then dried and heated in an oven at 250 °C for 15 to 30 min. All glassware is subsequently cleaned with acetone and methanol, respectively. The cleaned glassware should be protected from contamination by placing in a sealed cabinet or covering with foil to reduce impurities from entering.

10.2 *Sampling*—Grab samples must be collected in pre-cleaned glass jars with polytetrafluoroethylene (PTFE)-lined caps demonstrated to be free of interferences. This test method requires at least a 10-g sample size per analysis. A 100-g sample amount should be collected to allow for quality control samples and re-analysis. Field blanks are needed to follow conventional sampling practices.

10.3 *Preservation*—Store samples between 0 °C and 6 °C from the time of collection until analysis. Extract the samples within 14 days of collection. If the samples are above 6 °C when received or during storage or not extracted within 14 days of collection, the data are qualified and noted in the case narrative that accompanies the data that they were not extracted within the preliminary holding time. The sample extracts are analyzed within 14 days of extraction or the data are qualified and noted in the case narrative that accompanies the data that they were not analyzed within the preliminary holding time.

11. Preparation of LC/MS/MS

11.1 *LC Operating Conditions,* used to develop this test method:[8]

11.1.1 Injection volumes of all calibration standards and samples are 25 µL and are composed of 50 % water/50 % methanol. 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, 35 °C; sample compartment, 20 °C.

11.1.3 *Wash Solvent*—60 % acetonitrile/40 % 2-Propanol, pre- and post-inject wash solvent: 6 s.

11.1.4 *Purge Solvent*—50 % water/50 % acetonitrile.

TABLE 2 Gradient Conditions for Liquid Chromatography

Time (min)	Flow (µL/min)	Percent CH_3CN	Percent 95 % Water: 5 % CH_3CN	Percent 100 mM NH_4OAc in 95 % Water: 5 % CH_3CN
0	300	0	95	5
1	300	0	95	5
3	300	50	45	5
4	300	60	35	5
6	300	70	25	5
7	300	70	25	5
9	300	95	0	5
12	300	95	0	5
13	300	0	95	5
16	300	0	95	5

11.1.5 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:*[10]

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 should have at least ten scans per peak for adequate quantitation. Variable parameters regarding retention times, SRM transitions, and cone and collision energies are shown in Table 3. Mass spectrometer parameters used in the development of this method are listed below:

The instrument is set in the Electrospray 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
Desolvation Temperature: 300 °C
Desolvation Gas Flow: 800 L/hr
Cone Gas Flow: 100 L/hr
Low-Mass Resolution 1: 14.0
High-Mass Resolution 1: 14.0
Ion Energy 1: 0.6 V
Entrance Energy: –1 V
Collision Energy: Variable depending on analyte (Table 3)
Exit Energy: 1 V
Low-Mass Resolution 2: 14
High-Mass Resolution 2: 14
Ion Energy 2: 1.5 V
Multiplier: 650 V
Gas Cell Pirani Gauge: 0.60 Pa
Inter-Channel Delay: 0.02 s
Inter-Scan Delay: 0.010 s
Repeats: 1
Span: 0 Daltons
Dwell: 0.05 to 0.1 s to optimize scans

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

Analyte	ESI Mode	Retention Time (min)	SRM Mass Transition (Parent > Product)	Cone Voltage (Volts)	Collision Energy (eV)
BPA	negative	4.2	227.3 > 212.2	40	18
BPA confirmatory[A]	negative	4.2	227.3 > 133.1	40	25
BPA-D_6 (Surrogate)	negative	4.2	233.3 > 215.3	40	19
BPA-D_6 confirmatory[A] (Surrogate)	negative	4.2	233.3 > 138.2	40	25

[A] Confirmatory transitions are optional but should be included for added qualitative information.

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.

12.2 *Calibration and Standardization*—To calibrate the instrument, analyze seven calibration standards containing the seven concentration levels of BPA and surrogate prior to analysis as shown in Table 4. A calibration stock standard solution is prepared from standard materials or purchased as certified solutions. Stock standard solution A (Level 7) containing BPA and surrogate is prepared at Level 7 concentration, and aliquots of that solution are diluted in 50 % water/50 % methanol to prepare Levels 1 through 6. The following steps will produce standards with the concentration values shown in Table 4. 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 Prepare stock standard solution A (Level 7) by adding to a 25-mL volumetric flask 1.0 mL of target and surrogate spike solutions (12.4 and 12.6) and diluting to 25 mL with 50 % water/50 % methanol solution. The preparation of the Level 7 standard can be accomplished using different volumes and concentrations of stock solutions as is accustomed in the individual laboratory. Depending on stock concentrations prepared, the solubility at that concentration shall be ensured.

12.2.2 Aliquots of Solution A are then diluted with 50 % water/50 % methanol to prepare the desired calibration levels in 2-mL amber glass LC vials at concentrations shown in Table 4. Calibration standards are not filtered. The calibration standard vials shall be used within 24 h to ensure optimum results. Stock calibration standard solutions are replaced every 28 days if not previously discarded for quality control failure.

12.2.3 Inject each calibration standard and obtain its chromatogram. External calibration curves are generated from the calibration standards 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. The use of SRM transitions gives additional confirmation than by the selective ion monitoring technique because the parent ion is isolated and fragmented to the product ion.

12.2.4 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 units. Concentrations may be calculated using the data system software to generate linear regression or quadratic calibration curves. 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 % from the generated curve. Refer to 12.2.4.1 and 12.2.4.2 to determine if linear or quadratic calibration curves may be used.

TABLE 4 Concentrations of Calibration Standards (PPB)

Analyte/ Surrogate	LV1	LV2	LV3	LV4	LV5	LV6	LV7
BPA	20, RL	50	100	200	300	400	500
$BPA-D_6$ (Surrogate)	20, RL	50	100	200	300	400	500

12.2.4.1 Linear calibration may be used if the coefficient of determination, r^2, is >0.98 for the analyte. The point of origin is excluded, and a fit weighting of 1/X is used in order to give more emphasis to the lower concentrations. If one of the calibration standards other than the high or low point causes the r^2 of the curve to be <0.98, this point shall be re-injected or a new calibration curve shall be regenerated. If the low or high point, or both, is excluded, minimally a five-point curve is acceptable, the reporting range shall be modified to reflect this change.

12.2.4.2 Quadratic calibration may be used if the coefficient of determination, r^2, is >0.99 for the analyte. The point of origin is excluded, and a fit weighting of 1/X is used in order to give more emphasis to the lower concentrations. If one of the calibration standards, other than the high or low, causes the curve to be <0.99, this point shall be re-injected or a new calibration curve shall be regenerated. If the low or high point, or both, is excluded, a six-point curve is acceptable using a quadratic fit. An initial seven-point curve over the calibration range is suggested in the event that the low or high point must be excluded to obtain a coefficient of determination >0.99. In this event, the reporting range shall be modified to reflect this change.

12.2.5 The retention time window of the SRM transitions shall be within 5 % of the retention time of the analyte in a midpoint calibration standard. A midpoint calibration standard is defined at or between Levels 3 and 5 in Table 4 in this test method. If this is not the case, re-analyze the calibration curve to determine if there was a shift in retention time during the analysis and re-inject the sample. If the retention time is still incorrect in the sample, refer to the analyte as an unknown.

12.2.6 A midpoint calibration check standard shall be analyzed at the end of each batch of 20 samples or within 24 h after the initial calibration curve was generated. This end calibration check shall be the same calibration standard that was used to generate the initial curve. The results from the end calibration check standard shall have a percent deviation less than 35 % from the calculated concentration for the target analyte and surrogate. If the results are not within these criteria, the problem shall be corrected and either all samples in the batch shall be re-analyzed against a new calibration curve or the affected results shall be qualified with an indication that they do not fall within the performance criteria of the test method. If the analyst inspects the vial containing the end calibration check standard and notices that the sample evaporated affecting the concentration, a new end calibration check standard shall be made and analyzed. If this new end calibration check standard has a percent deviation less than 35 % from the calculated concentration for the target analyte and surrogate, the results shall be reported unqualified if all other quality control parameters are acceptable.

12.3 If a laboratory has not performed the test before, or if there has been a major change in the measurement system (for example, new analyst or new instrument), perform a precision and bias study to demonstrate laboratory capability and verify that all technicians are adequately trained and follow relevant safety procedures.

12.3.1 Analyze at least four replicates of a sample containing the target compound and surrogate at a concentration at or between Levels 3 and Level 5 in Ottawa sand. This test method was tested using a 10-g Ottawa sand sample with the concentration of the surrogate in the sample at 125 ppb (µg/kg) and BPA at 125 ppb. Each replicate shall be taken through the complete analytical test method.

12.3.2 Calculate the mean (average) percent recovery and relative standard deviation (RSD) of the four values and compare to the acceptable ranges of the quality control (QC) acceptance criteria for the initial demonstration of performance in Table 5.

12.3.3 This study shall be repeated until the single-operator precision and mean recovery are within the limits in Table 5.

12.3.4 The QC acceptance criteria for the initial demonstration of performance in Table 5 are preliminary until a collaborative study is conducted. Single-laboratory data are shown in the Precision and Bias section. The analyst shall be aware that the performance data generated from single-laboratory data tend to be significantly tighter than those generated from multi-laboratory data. The laboratory shall generate its own in-house QC acceptance criteria which meet or exceed the criteria in this test method. References on how to generate QC acceptance criteria are Practice E2554 or Method 8000B in EPA SW-846.

12.4 *Surrogate Spiking Solution:*

12.4.1 A surrogate standard solution containing BPA-D_6 is added to each 10-g soil sample. A stock surrogate spiking solution is prepared in methanol at 12.5 ppm for BPA-D_6. The surrogate is added to each sample to achieve a concentration of 125 ppb (that is, 100 µL of a 12.5-ppm methanol solution containing BPA-D_6 is added to a 10-g soil sample). The result obtained for the surrogate recovery shall fall within the limits of Table 5. If the limits are not met, the affected results shall be qualified with an indication that they do not fall within the performance criteria of the test method.

12.5 *Method Blank:*

12.5.1 Analyze a sample blank, Ottawa sand, with each batch of 20 or fewer samples. The surrogate is added to all samples at the concentration listed in 12.4. The concentration of target analyte found in the blank must be three times below the reporting limit. If the concentration of target analyte is found above this level, analysis of samples is halted until the contamination is eliminated, and a blank shows no contamination at or above this level or the results shall be qualified with an indication that there is a blank contamination and report the concentration found in the blank sample.

12.6 *Laboratory Control Sample (LCS):*

12.6.1 To ensure that the test method is in control, analyze an LCS prepared with BPA at a concentration between Levels 3 and Level 5 of the calibration curve. The surrogate is added to all samples at the concentration listed in 12.4. This test method was tested at 125 ppb for BPA. The LCS is prepared following the analytical method and analyzed with each batch of 20 samples or less. Prepare a stock matrix spiking solution in methanol containing BPA at 12.5 ppm in methanol. An Ottawa sand sample is spiked with the matrix spiking solution to achieve a concentration of 125 ppb for BPA (that is, 100 µL of a methanol solution containing BPA at 12.5 ppm is added to a 10-g soil sample). The results obtained for the LCS shall fall within the limits in Table 5.

12.6.2 If the results are not within these limits, analysis of samples is halted until the problem is corrected, and either all samples in the batch shall be re-analyzed or the results shall be qualified with an indication that they do not fall within the performance criteria of the test method.

12.7 *Matrix Spike (MS):*

12.7.1 To check for interferences in the specific matrix being tested, perform an MS on at least one sample from each batch of ten or fewer samples. This is accomplished by spiking the sample with a known concentration of BPA and following the analytical method. The surrogate is added to all samples at the concentration listed in 12.4. A matrix sample is spiked with the matrix spiking solution to achieve a concentration of 125 ppb for BPA (that is, 100 µL of a methanol solution containing BPA at 12.5 ppm is added to a 10-g soil sample).

12.7.2 If the spiked concentration plus the background concentration exceeds that of the Level 7 calibration standard, the sample shall be diluted to a level near the midpoint of the calibration curve. Biosolid and sludge samples may contain high levels of BPA and may require multiple dilutions.

12.7.3 Calculate the percent recovery of the spike (P) using Eq 1:

$$P = 100 \frac{|A(V_s + V) - BV_s|}{CV} \tag{1}$$

where:

A = concentration found in spiked sample,
B = concentration found in unspiked sample,
C = concentration of analyte in spiking solution,
V_s = volume of sample used,
V = volume of spiking solution added, and
P = percent recovery.

12.7.4 The percent recovery of the spike shall fall within the limits in Table 6. If the percent recovery is not within these limits, a matrix interference may be present in the selected sample, a matrix suppression or enhancement of the response or extraction efficiency, or both, of the analyte may be poor in the soil matrix. The results shall be qualified with an indication that they do not fall within the performance criteria of the test method. The recoveries of BPA and surrogate in the matrix spike samples are required.

TABLE 5 Quality Control Acceptance Criteria

Analyte	Initial Demonstration of Performance			Lab Control Sample	
	Recovery (%)		Precision	Recovery (%)	
	Lower Limit	Upper Limit	Maximum % RSD	Lower Limit	Upper Limit
BPA	70	130	30	70	130
BPA-D_6 (Surrogate)	70	130	30	70	130