



National Institute of Standards & Technology

# Certificate of Analysis

Standard Reference Material® 1944

New York/New Jersey Waterway Sediment

Standard Reference Material (SRM) 1944 is a mixture of marine sediment collected near urban areas in New York and New Jersey. SRM 1944 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and trace elements in marine sediment and similar matrices. Reference values are also provided for selected polybrominated diphenyl ether (PBDE) congeners, selected dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle size characteristics. Information values are provided for selected polychlorinated naphthalenes (PCNs) and hexabromocyclododecanes (HBCDs). All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment.

**Certified Mass Fraction Values:** Certified values for mass fractions of PAHs, PCB congeners, chlorinated pesticides, and trace elements are provided in Tables 1 through 4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST using two or more chemically independent analytical techniques. The certified values for the trace elements are based on NIST measurements by one technique and additional results from several collaborating laboratories.

**Reference Mass Fraction Values:** Reference values are provided for mass fractions of additional PAHs (some in combination) in Tables 5 and 6, additional PCB congeners and chlorinated pesticides in Table 7, PBDE congeners in Table 8, and additional inorganic constituents in Tables 9 and 10. Reference values are provided in Table 11 for the 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and total tetra-, penta-, hexa-, and hepta-congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for particle size characteristics are provided in Table 12 and 13. Reference values for total organic carbon and percent extractable mass are provided in Table 14. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

**Information Mass Fraction Values:** Information values are provided in Table 15 for mass fractions of additional trace elements, in Table 16 for PCN congeners (some in combination), and in Table 17 for HBCD isomers. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1]. Information values cannot be used to establish metrological traceability.

**Expiration of Certification:** The certification of **SRM 1944** is valid, within the measurement uncertainty specified, until **31 March 2027**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see “Instructions for Handling, Storage, and Use”). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Carlos A. Gonzalez, Chief  
Chemical Sciences Division

Gaithersburg, MD 20899  
Certificate Issue Date: 17 February 2017  
*Certificate Revision History on Page 20*

Steven J. Choquette, Director  
Office of Reference Materials

**Maintenance of SRM Certification:** NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification was performed by M.M. Schantz and S.A. Wise of the NIST Chemical Sciences Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh, M.G. Vangel, and M.S. Levenson of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

The sediment was collected with the assistance of the New York District of the U.S. Army Corp of Engineers (ACENYD), who provided the expertise in the site selection, the ship, sampling equipment, and personnel. L. Rosman of ACENYD and R. Parris (NIST) coordinated the collection of this sediment. Collection and preparation of SRM 1944 were performed by R. Parris, M. Cronise, and C. Fales (NIST); L. Rosman and P. Higgins (ACENYD), and the crew of the *Gelberman* from the ACE Caven Point facility in Caven Point, NJ.

Analytical measurements for the certification of SRM 1944 were performed at NIST by E.S. Beary, D.A. Becker, R.R. Greenberg, J.M. Keller, J.R. Kucklick, M. Lopez de Alda, K.E. Murphy, R. Olfaz, B.J. Porter, D.L. Poster, L.C. Sander, P. Schubert, M.M. Schantz, S.S. Vander Pol, and L. Walton of the Analytical Chemistry Division. Measurements for percent total organic carbon measurements were provided by three commercial laboratories and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX, USA). The particle-size distribution data were provided by Honeywell, Inc. (Clearwater, FL, USA). Additional results for PBDE congeners were used from ten laboratories (see Appendix A) that participated in an interlaboratory study specifically for PBDEs in Marine Sediment coordinated by H.M. Stapleton of the NIST Analytical Chemistry Division. M. LaGuardia of Virginia Institute of Marine Science (Gloucester Point, VA, USA) provided one set of measurements for the HBCDs.

Values for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among fourteen laboratories (see Appendix B) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turlle and C. Chiu of Environment Canada Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, ON, Canada). Analytical measurements for selected trace elements were provided by the International Atomic Energy Agency (IAEA, Seibersdorf, Austria) by M. Makarewicz and R. Zeisler. Results were also used from seven laboratories (see Appendix C) that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, National Research Council Canada (NRCC; Ottawa, ON, Canada).

## **INSTRUCTIONS FOR HANDLING, STORAGE, AND USE**

**Handling:** This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

**Storage:** SRM 1944 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

**Use:** Prior to removal of test portions for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1944 are reported on a dry-mass basis. The SRM, as received, contains a mass fraction of approximately 1.3 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis or, if the constituents of interest are volatile, a separate test portion of the sediment should be removed from the bottle at the time of analysis and dried to determine the mass fraction on a dry-mass basis.

## PREPARATION AND ANALYSIS<sup>(1)</sup>

**Sample Collection and Preparation:** The sediment used to prepare this SRM was collected from six sites in the vicinity of New York Bay and Newark Bay in October 1994. Site selection was based on contaminant levels measured in previous samples from these sites and was intended to provide relatively high concentrations for a variety of chemical classes of contaminants. The sediment was collected using an epoxy-coated modified Van Veen-type grab sampler designed to sample the sediment to a depth of 10 cm. A total of approximately 2100 kg of wet sediment was collected from the six sites. The sediment was freeze-dried, sieved (nominally 250  $\mu\text{m}$  to 61  $\mu\text{m}$ ), homogenized in a cone blender, radiation sterilized at an estimated minimum dose of 32 kilograys ( $^{60}\text{Co}$ ), and then packaged in screw-capped amber glass bottles.

**Conversion to Dry-Mass Basis:** The results for the constituents in SRM 1944 are reported on a dry-mass basis; however, the material as received contains residual moisture. The amount of moisture in SRM 1944 was determined by measuring the mass loss after freeze drying test portions of 1.6 g to 2.5 g for five days at 1 Pa with a  $-10\text{ }^\circ\text{C}$  shelf temperature and a  $-50\text{ }^\circ\text{C}$  condenser temperature. The mass fraction of moisture in SRM 1944 at the time of the certification analyses was  $1.25\% \pm 0.03\%$  (95 % confidence level).

**Polycyclic Aromatic Hydrocarbons:** The general approach used for the value assignment of the PAHs in SRM 1944 consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques [2]. Techniques and solvents involved were Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, clean up of the extracts using solid-phase extraction (SPE), or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on four stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, a proprietary non-polar polysiloxane phase, and a smectic liquid crystalline stationary phase.

Seven sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), GC/MS (V), GC/MS (VI), and GC/MS (Sm), were obtained using four columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate test portions of 1 g from eight bottles of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane. (All extraction and LC solvent compositions are expressed as volume fractions unless otherwise noted.) The processed extract was then analyzed by GC/MS using a 0.25 mm i.d.  $\times$  60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25  $\mu\text{m}$  film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 1 g to 2 g test portions from three bottles of SRM 1944 and 2 g to 3 g test portions from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment). These test portions were Soxhlet extracted with DCM and processed through the silica SPE as described above; however, the extract was further fractionated using normal-phase LC on a semi-preparative aminopropylsilane column to isolate the PAH fraction. The PAH fraction was then analyzed using the same column as described above for GC/MS (I); however, the test portions were extracted, processed, and analyzed as part of three different sample sets at different times using different calibrations for each set. For the GC/MS (III), 1 g to 2 g test portions from six bottles of SRM 1944 were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone. The extracts were then processed and analyzed as described for GC/MS (II). For GC/MS (IV) analyses, 1 g to 2 g test portions from six bottles of SRM 1944 were extracted using PFE with a mixture of 50 % hexane/50 % acetone, and the extracts were processed as described above for GC/MS (II). The GC/MS (V) results were obtained by analyzing three of the same PAH fractions that were analyzed in GC/MS (III) and three of the PAH fractions that were analyzed in GC/MS (IV) using a 50 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d.  $\times$  60 m, 0.25  $\mu\text{m}$  film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (VI) analyses, three test portions of 0.7 g from one bottle of SRM 1944 were Soxhlet extracted for 24 h with DCM. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 20 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d.  $\times$  60 m fused silica capillary column with a proprietary non-polar polysiloxane phase (0.25  $\mu\text{m}$  film thickness) (DB-XLB, J&W Scientific). For GC/MS (Sm) 1 g to 2 g test portions from six bottles of SRM 1944 were Soxhlet extracted for 24 h

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<sup>(1)</sup>Certain commercial equipment, instruments, or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

with 250 mL of DCM. The extracts were processed as described above for GC/MS (I) using an aminopropylsilane SPE cartridge followed by GC/MS analysis using 0.2 mm i.d. × 25 m (0.15 µm film thickness) smectic liquid crystalline phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT).

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Test portions of approximately 1 g from six bottles of SRM 1944 were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone. The extracts were concentrated and then processed through two aminopropylsilane SPE cartridges connected in series to obtain the total PAH fraction. A second 1 g test portion from the six bottles was Soxhlet extracted and processed as described above; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH<sub>2</sub>, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5-µm particle-size polymeric octadecylsilane (C<sub>18</sub>) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength-programmed fluorescence detection. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

**Homogeneity Assessment for PAHs:** The homogeneity of SRM 1944 was assessed by analyzing duplicate test portions of 1 g from eight bottles selected by stratified random sampling. Test portions were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at the 1 g test portion size.

**PAH Isomers of Molecular Mass 300 and 302:** For the determination of the molecular mass 300 and 302 PAH isomers, three test portions of approximately 5 g each were extracted using PFE with DCM. The extracts were then concentrated with a solvent change to hexane and passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-17MS, J&W Scientific, Folsom, CA). Perdeuterated dibenzo[*a,i*]pyrene was added to the sediment prior to extraction for use as an internal standard.

**PCBs and Chlorinated Pesticides:** The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1944 consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques [2]. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity.

Eight sets of results were obtained designated as GC-ECD (I) A and B, GC-ECD (II) A and B, GC/MS (I), GC/MS (II), GC/MS (III), and QA Exercise. For the GC-ECD (I) analyses, 1 g test portions from four bottles of SRM 1944 were Soxhlet extracted with DCM for 18 h. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides and, (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm × 100 m fused silica capillary column with a 50 % (mole fraction) octadecyl (C<sub>18</sub>) methylpolysiloxane phase (0.1 µm film thickness) (CPSil 5 C<sub>18</sub> CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C<sub>18</sub> phase are designated as GC-ECD (IB). A second set of samples was also analyzed by GC-ECD (i.e., GC-ECD IIA and IIB). Test portions of 1 g to 2 g from three bottles of SRM 1944 and 2 g to 3 g test portions from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment) were extracted, processed, and analyzed as described above for GC-ECD (I); however, the test portions were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

Three sets of results were obtained by GC/MS. For GC/MS (I), 1 g to 2 g test portions from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The extract was then analyzed by GC/MS using a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness). The GC/MS (II) results were obtained in the same manner as the GC/MS (I) analyses except that the six test portions were extracted using PFE. The GC/MS (III) analyses were performed on the same extract fractions analyzed in GC-ECD (II) using the 5 % phenyl-substituted methylpolysiloxane phase describe above for GC/MS (I). For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [3]), and 4,4'-DDT-*d*<sub>8</sub> were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1944 was used in an interlaboratory comparison exercise in 1995 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [4]. Results from nineteen laboratories that participated in this exercise were used as the eighth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1944. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

**Polybrominated Diphenyl Ethers:** Value assignment of the concentrations of eight PBDE congeners was based on the means of results from two interlaboratory studies [5,6] and two sets of data from NIST. The laboratories participating in the interlaboratory exercises (see Appendix A) employed the analytical procedures routinely used in their laboratories to measure PBDEs. For the two methods used at NIST, six test portions (between 1 g and 2 g) were extracted using PFE at 100 °C with DCM. The extracts were cleaned up using an alumina column (5 % deactivated) SPE column. Size exclusion chromatography (SEC) on a divinylbenzene-polystyrene column (10 µm particle size, 10 nm (100 angstrom) pore size, 7.5 mm i.d. × 300 mm, PL-Gel, Polymer Labs, Inc.) was then used to remove the sulfur. The PBDEs, as well as PCBs and pesticides, were quantified using GC/MS in the electron impact mode on a 0.18 mm i.d. × 30 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.18 µm film thickness; DB-5MS, Agilent Technologies). The PBDEs were also quantified using GC/MS in the negative chemical ionization mode on a 0.18 mm i.d. × 10 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.18 µm film thickness; DB-5MS, Agilent Technologies). Selected Carbon-13 labeled PBDE and PCB congeners were added to the sediment prior to extraction for use as internal standards for quantification purposes.

**Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans:** Value assignment of the concentrations of the polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra- through hepta- substituted polychlorinated dibenzo-*p*-dioxins and dibenzofurans was accomplished by combining results from the analysis of SRM 1944 by fourteen laboratories that participated in an interlaboratory comparison study (see Appendix B). Each laboratory analyzed three test portions (typically 1 g) of SRM 1944 using their routine analytical procedures and high resolution gas chromatography with high resolution mass spectrometry detection (GC-HRMS). The analytical procedures used by all of the laboratories included spiking with <sup>13</sup>C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and carbon columns; and finally analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % cyanopropylphenyl-substituted methylpolysiloxane (DB-225, J&W Scientific, Folsom, CA) capillary column.

**Analytical Approach for Inorganic Constituents:** Value assignment for the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 1944 from NIST, NRCC, IAEA, and seven laboratories that participated in an interlaboratory comparison exercise coordinated by NRCC [7] (see Appendix C). The analytical methods used for the determination of each element are summarized in Table 18. For the certified concentration values listed in Table 4, results were combined from: (1) analyses at NIST using isotope dilution inductively coupled plasma mass spectrometry (ID-ICPMS) or instrumental neutron activation analysis (INAA), (2) analyses at NRCC using ID-ICPMS, graphite furnace atomic absorption spectrometry (GFAAS), and/or inductively coupled plasma optical emission spectroscopy (ICPOES), (3) analyses at IAEA using INAA, and (4) the mean of the results from seven laboratories that participated in the NRCC interlaboratory comparison exercise. The reference mass fraction values in Table 9 were determined by combining results from (1) analyses performed at NIST using INAA; (2) analyses at NRCC using ID-ICPMS, GFAAS, ICPOES, and/or cold vapor atomic absorption spectroscopy (CVAAS); (3) analyses at IAEA using INAA; and (4) the mean of the results from five to seven laboratories that participated in the NRCC interlaboratory comparison exercise. The information concentration values in Table 15 were determined by INAA at NIST and IAEA.

**NIST Analyses using ID-ICPMS:** Lead, cadmium, and nickel were determined by ID-ICPMS [8]. Test portions (0.4 g to 0.5 g) from six bottles of the SRM were spiked with  $^{206}\text{Pb}$ ,  $^{111}\text{Cd}$ , and  $^{62}\text{Ni}$  and wet ashed using a combination of nitric, hydrochloric, hydrofluoric, and perchloric acids. Lead and cadmium were determined in the same test portions; nickel was determined in a second sample set. A small amount of crystalline material remained after the acid dissolution. Lithium metaborate fusion was performed on this residue to confirm that the residue contained insignificant amounts of the analytes. Cadmium and nickel were separated from the matrix material to eliminate the possibility of spectral interferences, and concentrations were determined from the measurement of the  $^{112}\text{Cd}/^{111}\text{Cd}$  and  $^{62}\text{Ni}/^{60}\text{Ni}$  ratios, respectively. The  $^{208}\text{Pb}/^{206}\text{Pb}$  ratios were measured directly because interferences at these masses are negligible.

**NIST Analyses using INAA:** Analyses were performed in two steps [9]. Elements with short-lived irradiation products (Al, Ca, Cl, K, Mg, Mn, Na, Ti, and V) were determined by measuring duplicate 300 mg test portions from each of ten bottles of SRM 1944. The samples, standards, and controls were packaged in clean polyethylene bags and were individually irradiated for 15 s in the NIST Reactor Pneumatic Facility RT-4. Reactor power was 20 MW, which corresponds to a neutron fluence rate of about  $8 \times 10^{13} \text{ cm}^{-2}\text{s}^{-1}$ . After irradiation, the samples, controls, and standards were repackaged in clean polyethylene bags and counted (gamma-ray spectrometry) three times at different decay intervals. A sample-to-detector distance (counting geometry) of 20 cm was used. Elements with long-lived irradiation products (Ag, As, Br, Co, Cr, Cs, Fe, Rb, Sb, Sc, Se, Th, and Zn) were determined by measuring one 300 mg test portion from each of nine bottles of SRM 1944. The samples, standards, controls, and blank polyethylene bags were irradiated together for a total of 1 h at a reactor power of 20 MW. Approximately four days after irradiation, the polyethylene bags were removed, and each sample, standard, control, and blank was counted at 20 cm from the detector. The samples were then recounted at 10 cm from another detector. After an additional decay time of about one month, the samples, standards, controls, and blanks were counted a third time (at 10 cm) from the second detector.

**Homogeneity Assessment for Inorganic Constituents:** For some of the trace elements, most notably Cd, Fe, Pb, Rb, Sb, Sc, and Th, the variations among the test portions measured at NIST (between 0.3 g and 0.5 g) were larger than expected from the measurement process. Based on experience, it was concluded that there is some material inhomogeneity for trace elements in the test portions used. Sample variations among the NIST measurements are used as slightly conservative estimates of the sample inhomogeneities.

**Particle Size Information:** Dry particle-size distribution measurements for SRM 1944 were obtained as part of a collaborative effort with Honeywell's Particle and Components Measurements Laboratory (Clearwater, FL). A Microtrac particle analyzer, which makes use of light-scattering techniques, was used to measure the particle-size distribution of SRM 1944. Briefly, a reference beam is used to penetrate a field of particles and the light that scatters in the forward direction from the field is measured and the particle-size as a volume distribution is derived via a computer-assisted analysis. From these data, the total volume, average size, and a characteristic width of the particle size distribution are calculated. The system has a working range from 0.7  $\mu\text{m}$  to 700  $\mu\text{m}$ .

**Total Organic Carbon and Percent Extractable Mass:** Four laboratories provided results for total organic carbon (TOC) using similar procedures. Briefly, test portions of approximately 200 mg were reacted with 6 mol/L hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed test portions from six bottles of SRM 1944. For the determination of percent extractable mass, six test portions of approximately 1 g to 2 g of SRM 1944 were extracted using Soxhlet extraction for 18 h with DCM. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in the mass before and after extraction was determined.

**Polychlorinated Naphthalenes:** Value assignment of PCN congener concentrations was accomplished by combining results from the analysis of SRM 1944 by six laboratories that participated in an interlaboratory comparison study (see Appendix D). Each laboratory analyzed three test portions (typically 1 g to 2 g) of SRM 1944 using their routine analytical procedures that included high-resolution gas chromatography with either high-resolution mass spectrometry detection (GC-HRMS) or low-resolution MS in the negative chemical ionization mode. Calibration mixtures included either Halowax mixtures with known volume fractions of individual congeners or individual PCN congeners.

**HBCDs:** Value assignment of the concentrations of three HBCD isomers was accomplished by combining results from the analysis of SRM 1944 in two sets from NIST and one set from Virginia Institute of Marine Science. For the two sets analyzed at NIST, the second fraction from an acidified silica SPE clean-up was analyzed by LC/MS/MS for the HBCDs using both electrospray ionization (ESI) and atmospheric pressurized photoionization (APPI). A C18 column (3.0 mm × 150 mm × 3.5 μm column, Eclipse Plus, Agilent Technologies) and YMC Carotenoid S5 C30 column (4.6 mm × 250 mm × 5 μm column) were used with a solvent gradient using 2.5 mmol/L ammonium acetate in 12.5 % water in methanol and acetonitrile at a flow rate of 0.3 mL/min. Carbon-13 labeled HBCDs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

The measurands for certified values are the total concentrations of the analytes listed in Tables 1-4. Metrological traceability is to the SI derived units for mass fraction, expressed as milligrams per kilogram, micrograms per kilogram or percentage, respectively.

Table 1. Certified Mass Fraction Values for Selected PAHs in SRM 1944 (Dry-Mass Basis)

	Mass Fraction <sup>(a,b)</sup> (mg/kg)		
Phenanthrene <sup>(c,d,e,f,g)</sup>	5.27	±	0.22
Fluoranthene <sup>(c,d,e,f,g)</sup>	8.92	±	0.32
Pyrene <sup>(c,d,e,f,g)</sup>	9.70	±	0.42
Benzo[ <i>c</i> ]phenanthrene <sup>(c,d,e,f,h)</sup>	0.76	±	0.10
Benz[ <i>a</i> ]anthracene <sup>(c,d,e,f,g,h)</sup>	4.72	±	0.11
Chrysene <sup>(h,k)</sup>	4.86	±	0.10 <sup>(i)</sup>
Triphenylene <sup>(h,k)</sup>	1.04	±	0.27
Benzo[ <i>b</i> ]fluoranthene <sup>(g,h,j)</sup>	3.87	±	0.42
Benzo[ <i>j</i> ]fluoranthene <sup>(h,j)</sup>	2.09	±	0.44
Benzo[ <i>k</i> ]fluoranthene <sup>(c,d,e,f,g,h,j)</sup>	2.30	±	0.20
Benzo[ <i>a</i> ]fluoranthene <sup>(c,d,e,f,h,j)</sup>	0.78	±	0.12
Benzo[ <i>e</i> ]pyrene <sup>(c,d,e,f,h,j)</sup>	3.28	±	0.11
Benzo[ <i>a</i> ]pyrene <sup>(c,d,e,f,g,h,j)</sup>	4.30	±	0.13
Perylene <sup>(c,d,e,f,g,h,j)</sup>	1.17	±	0.24
Benzo[ <i>ghi</i> ]perylene <sup>(c,d,e,f,j,k)</sup>	2.84	±	0.10
Indeno[1,2,3- <i>cd</i> ]pyrene <sup>(c,d,e,f,j,k)</sup>	2.78	±	0.10
Dibenz[ <i>a,j</i> ]anthracene <sup>(c,d,e,f,j,k)</sup>	0.500	±	0.044
Dibenz[ <i>a,c</i> ]anthracene <sup>(j,k)</sup>	0.335	±	0.013
Dibenz[ <i>a,h</i> ]anthracene <sup>(j,k)</sup>	0.424	±	0.069
Pentaphene <sup>(c,d,e,f,j,k)</sup>	0.288	±	0.026
Benzo[ <i>b</i> ]chrysene <sup>(c,d,e,f,j,k,h)</sup>	0.63	±	0.10
Picene <sup>(c,d,e,f,j,k)</sup>	0.518	±	0.093

<sup>(a)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(b)</sup> Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10].

Each uncertainty, computed according to the Comité International des Poids et Mesures (CIPM) approach as described in the ISO/JCGM Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

<sup>(c)</sup> Gas chromatography/mass spectrometry (GC/MS) (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(d)</sup> GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(e)</sup> GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(f)</sup> GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone mixture.

<sup>(g)</sup> LC-FL of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(h)</sup> GC/MS (Sm) using a smectic liquid crystalline phase after Soxhlet extraction with DCM.

<sup>(i)</sup> The uncertainty interval for chrysene was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

<sup>(j)</sup> GC/MS (V) on 50 % phenyl-substituted methylpolysiloxane phase of extracts from GC/MS (III) and GC/MS (IV).

<sup>(k)</sup> LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 2. Certified Mass Fraction Values for Selected PCB Congeners<sup>(a)</sup> in SRM 1944 (Dry-Mass Basis)

		Mass Fraction <sup>(b,c)</sup> (µg/kg)		
PCB 8	(2,4'-Dichlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	22.3	±	2.3
PCB 18	(2,2',5-Trichlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	51.0	±	2.6
PCB 28	(2,4,4'-Trichlorobiphenyl) <sup>(d,e,f,g,j,k)</sup>	80.8	±	2.7
PCB 31	(2,4',5-Trichlorobiphenyl) <sup>(d,e,f,g,j)</sup>	78.7	±	1.6 <sup>(l)</sup>
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	60.2	±	2.0
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	53.0	±	1.7
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	79.4	±	2.0
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl) <sup>(e,g,h,i,j)</sup>	71.9	±	4.3
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl) <sup>(e,g,h,i,j)</sup>	65.0	±	8.9
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl) <sup>(d,e,f,g,h,i,j)</sup>	29.9	±	4.3
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	37.5	±	2.4
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	73.4	±	2.5
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl) <sup>(e,f,g,h,i,j,k)</sup>	24.5	±	1.1
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl) <sup>(g,h,i,j)</sup>	63.5	±	4.7
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	58.0	±	4.3
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	8.47	±	0.28
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	62.1	±	3.0
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	49.7	±	1.2
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	16.93	±	0.36
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	74.0	±	2.9
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl) <sup>(d,e,f,g,h,i,j)</sup>	6.52	±	0.66
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	22.6	±	1.4
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	44.3	±	1.2
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl) <sup>(d,e,f,g,h,i,j)</sup>	12.19	±	0.57
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	25.1	±	1.0
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl) <sup>(d,e,f,g,h,i,j)</sup>	11.2	±	1.4
PCB 195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	3.75	±	0.39
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) <sup>(d,e,f,g,h,i,j,k)</sup>	9.21	±	0.51
PCB 209	Decachlorobiphenyl <sup>(d,e,f,g,h,i,j,k)</sup>	6.81	±	0.33

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmitter and Zell [13] and later revised by Schulte and Malisch [3] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmitter-Zell numbers correspond to those of Schulte and Malisch.

<sup>(b)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(c)</sup> Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10].

Each uncertainty, computed according to the CIPM approach as described in the ISO/JCGM Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

<sup>(d)</sup> GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(e)</sup> GC-ECD (IB) on the 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

<sup>(f)</sup> GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(g)</sup> GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

<sup>(h)</sup> GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(i)</sup> GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

<sup>(j)</sup> GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

<sup>(k)</sup> Results from nineteen laboratories participating in an interlaboratory comparison exercise.

<sup>(l)</sup> The uncertainty interval for PCB 31 was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

Table 3. Certified Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1944 (Dry-Mass Basis)

	Mass Fraction <sup>(a,b)</sup> (µg/kg)		
Hexachlorobenzene <sup>(e,f,g,h,i,j)</sup>	6.03	±	0.35
<i>cis</i> -Chlordane ( $\alpha$ -Chlordane) <sup>(c,d,e,f,g,h,i,j)</sup>	16.51	±	0.83
<i>trans</i> -Nonachlor <sup>(c,d,e,f,g,h,i,j)</sup>	8.20	±	0.51

- (a) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.  
 (b) Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [10].  
 Each uncertainty, computed according to the CIPM approach as described in the ISO/JCGM Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.  
 (c) GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.  
 (d) GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).  
 (e) GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.  
 (f) GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).  
 (g) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.  
 (h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.  
 (i) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).  
 (j) Results from nineteen laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Mass Fraction Values for Selected Elements in SRM 1944 (Dry-Mass Basis)

	Degrees of Freedom	Mass Fractions <sup>(a,b)</sup> (%)		
Aluminum <sup>(c,d,e)</sup>	4	5.33	±	0.49
Iron <sup>(c,d,e)</sup>	6	3.53	±	0.16

  

		Mass Fractions <sup>(a,b)</sup> (mg/kg)		
Arsenic <sup>(c,d,e,f,g)</sup>	10	18.9	±	2.8
Cadmium <sup>(c,f,h,i)</sup>	6	8.8	±	1.4
Chromium <sup>(c,d,f,g,i)</sup>	9	266	±	24
Lead <sup>(c,h,i)</sup>	5	330	±	48
Manganese <sup>(c,d,e)</sup>	8	505	±	25
Nickel <sup>(c,g,h,i)</sup>	6	76.1	±	5.6
Zinc <sup>(c,d,e,g,i)</sup>	9	656	±	75

- (a) The certified value is the mean of four results: (1) the mean of NIST INAA or ID-ICPMS analyses, (2) the mean of two methods performed at NRCC, and (3) the mean of results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) the mean results from INAA analyses at IAEA. The expanded uncertainty in the certified value is equal to  $U = k u_c$  where  $u_c$  is the combined standard uncertainty and  $k$  is the coverage factor, both calculated according to the ISO/JCGM Guide [11,12]. The value of  $u_c$  is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here  $u_c$  accounts for both possible method biases, within-method variation, and material inhomogeneity. The coverage factor,  $k$ , is the Student's  $t$ -value for a 95 % confidence interval with the corresponding degrees of freedom. Because of the material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.  
 (b) Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.  
 (c) Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.  
 (d) Measured at NIST using INAA.  
 (e) Measured at NRCC using ICPOES.  
 (f) Measured at NRCC using GFAAS.  
 (g) Measured at IAEA using INAA.  
 (h) Measured at NIST using ID-ICPMS.  
 (i) Measured at NRCC using ID-ICPMS.

The measurands for reference values are indicated in Tables 5-11, 14, 16-17; as determined by the indicated methods indicated in the text. Metrological traceability is to the SI derived units for mass fraction, expressed as milligrams per kilogram, micrograms per kilogram or percentage, respectively.

Table 5. Reference Mass Fraction Values for Selected PAHs in SRM 1944

	Mass Fractions <sup>(a)</sup> (mg/kg)		
Naphthalene <sup>(b)</sup>	1.28	±	0.04 <sup>(c)</sup>
1-Methylnaphthalene <sup>(b)</sup>	0.47	±	0.02 <sup>(c)</sup>
2-Methylnaphthalene <sup>(b)</sup>	0.74	±	0.06 <sup>(c)</sup>
Biphenyl <sup>(b)</sup>	0.25	±	0.02 <sup>(c)</sup>
Acenaphthene <sup>(b)</sup>	0.39	±	0.03 <sup>(c)</sup>
Fluorene <sup>(b)</sup>	0.48	±	0.04 <sup>(c)</sup>
Dibenzothiophene <sup>(b)</sup>	0.50	±	0.03 <sup>(c)</sup>
Anthracene <sup>(b)</sup>	1.13	±	0.07 <sup>(c)</sup>
1-Methylphenanthrene <sup>(d,e,f,g)</sup>	1.7	±	0.1 <sup>(h)</sup>
2-Methylphenanthrene <sup>(d,e,f,g)</sup>	1.90	±	0.06 <sup>(h)</sup>
3-Methylphenanthrene <sup>(d,e,f,g)</sup>	2.1	±	0.1 <sup>(h)</sup>
4-Methylphenanthrene and 9-Methylphenanthrene <sup>(d,e,f,g)</sup>	1.6	±	0.2 <sup>(h)</sup>
2-Methylantracene <sup>(d,e,f,g)</sup>	0.58	±	0.04 <sup>(h)</sup>
3,5-Dimethylphenanthrene <sup>(d)</sup>	1.31	±	0.04 <sup>(h)</sup>
2,6-Dimethylphenanthrene <sup>(d)</sup>	0.79	±	0.02 <sup>(h,i)</sup>
2,7-Dimethylphenanthrene <sup>(d)</sup>	0.67	±	0.02 <sup>(h,i)</sup>
3,9-Dimethylphenanthrene <sup>(d)</sup>	2.42	±	0.05 <sup>(h,i)</sup>
1,6-, 2,9-, and 2,5-Dimethylphenanthrene <sup>(d)</sup>	1.67	±	0.03 <sup>(h,i)</sup>
1,7-Dimethylphenanthrene <sup>(d)</sup>	0.62	±	0.02 <sup>(h,i)</sup>
1,9- and 4,9-Dimethylphenanthrene <sup>(d)</sup>	1.20	±	0.03 <sup>(h,i)</sup>
1,8-Dimethylphenanthrene <sup>(d)</sup>	0.24	±	0.01 <sup>(h,i)</sup>
1,2-Dimethylphenanthrene <sup>(d)</sup>	0.28	±	0.01 <sup>(h,i)</sup>
8-Methylfluoranthene <sup>(d)</sup>	0.86	±	0.02 <sup>(h,i)</sup>
7-Methylfluoranthene <sup>(d)</sup>	0.69	±	0.02 <sup>(h)</sup>
1-Methylfluoranthene <sup>(b)</sup>	0.39	±	0.01 <sup>(c)</sup>
3-Methylfluoranthene <sup>(b)</sup>	0.56	±	0.02 <sup>(c)</sup>
2-Methylpyrene <sup>(d)</sup>	1.81	±	0.04 <sup>(h,i)</sup>
4-Methylpyrene <sup>(d)</sup>	1.44	±	0.03 <sup>(h,i)</sup>
1-Methylpyrene <sup>(d)</sup>	1.29	±	0.03 <sup>(h)</sup>
Anthanthrene <sup>(i)</sup>	0.9	±	0.1 <sup>(h)</sup>

<sup>(a)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(b)</sup> GC/MS (VI) on proprietary non-polar methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(c)</sup> Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty,  $U$ , is calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the analyte mean, and the coverage factor,  $k$ , is determined from the Student's  $t$ -distribution corresponding to the associated degrees of freedom ( $df = 2$ ) and 95 % confidence level for each analyte.

<sup>(d)</sup> GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(e)</sup> GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(f)</sup> GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(g)</sup> GC/MS (IV) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with 50 % hexane/50 % acetone mixture.

<sup>(h)</sup> The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

<sup>(i)</sup> The uncertainty interval for this compound was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

<sup>(j)</sup> LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 6. Reference Mass Fractions for Selected PAHs of  
Relative Molecular Mass 300 and 302 in SRM 1944 (Dry-Mass Basis)

	Mass Fraction <sup>(a,b,c)</sup> (mg/kg)		
Coronene	0.53	±	0.04
Dibenzo[ <i>b,e</i> ]fluoranthene	0.076	±	0.008
Naphtho[1,2- <i>b</i> ]fluoranthene	0.70	±	0.06
Naphtho[1,2- <i>k</i> ]fluoranthene and Naphtho[2,3- <i>j</i> ]fluoranthene	0.66	±	0.05
Naphtho[2,3- <i>b</i> ]fluoranthene	0.21	±	0.01
Dibenzo[ <i>b,k</i> ]fluoranthene	0.75	±	0.06
Dibenzo[ <i>a,k</i> ]fluoranthene	0.22	±	0.02
Dibenzo[ <i>j,l</i> ]fluoranthene	0.56	±	0.03
Dibenzo[ <i>a,l</i> ]pyrene	0.12	±	0.02
Naphtho[2,3- <i>k</i> ]fluoranthene	0.11	±	0.01
Naphtho[2,3- <i>e</i> ]pyrene	0.33	±	0.02
Dibenzo[ <i>a,e</i> ]pyrene	0.67	±	0.05
Naphtho[2,1- <i>a</i> ]pyrene	0.76	±	0.05
Dibenzo[ <i>e,l</i> ]pyrene	0.28	±	0.02
Naphtho[2,3- <i>a</i> ]pyrene	0.23	±	0.01
Benzo[ <i>b</i> ]perylene	0.43	±	0.04
Dibenzo[ <i>a,i</i> ]pyrene	0.30	±	0.03
Dibenzo[ <i>a,h</i> ]pyrene	0.11	±	0.01

<sup>(a)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(b)</sup> Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty,  $U$ , is calculated as  $U = k u_c$ , where  $u_c$  is one standard deviation of the analyte mean, and the coverage factor,  $k$ , is determined from the Student's  $t$ -distribution corresponding to the associated degrees of freedom ( $df = 2$ ) and 95 % confidence level for each analyte.

<sup>(c)</sup> GC/MS on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

Table 7. Reference Mass Fractions for Selected PCB Congeners<sup>(a)</sup>  
and Chlorinated Pesticides in SRM 1944 (Dry-Mass Basis)

		Mass Fraction <sup>(b)</sup> (µg/kg)
PCB 45	(2,2',3,6-Tetrachlorobiphenyl) <sup>(c)</sup>	10.8 ± 1.4 <sup>(d)</sup>
PCB 146	(2,2',3,4',5,5'-Hexachlorobiphenyl) <sup>(c)</sup>	10.1 ± 1.9 <sup>(d)</sup>
PCB 163	(2,3,3',4',5,6-Hexachlorobiphenyl) <sup>(c)</sup>	14.4 ± 2.0 <sup>(d)</sup>
PCB 174	(2,2',3,3',4,5,6'-Heptachlorobiphenyl) <sup>(c)</sup>	16.0 ± 0.6 <sup>(d)</sup>
$\alpha$ -HCH <sup>(f,g,h,i)</sup>		2.0 ± 0.3 <sup>(e)</sup>
<i>trans</i> -Chlordane ( $\gamma$ -Chlordane) <sup>(c)</sup>		19.0 ± 1.7 <sup>(d)</sup>
<i>cis</i> -Nonachlor <sup>(g,h,i,l,m)</sup>		3.7 ± 0.7 <sup>(e)</sup>
2,4'-DDE <sup>(f,g,h,i,j,k,l,m)</sup>		19 ± 3 <sup>(e)</sup>
2,4'-DDD <sup>(h,j,k,l,m)</sup>		38 ± 8 <sup>(e)</sup>
4,4'-DDE <sup>(f,g,h,ihj,k,l,m)</sup>		86 ± 12 <sup>(e)</sup>
4,4'-DDD <sup>(f,g,h,l,j,k,l,m)</sup>		108 ± 16 <sup>(e)</sup>
4,4'-DDT <sup>(c)</sup>		170 ± 32 <sup>(d)</sup>

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [13] and later revised by Schulte and Malisch [3] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

<sup>(b)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(c)</sup> NIST participation in the 2007 interlaboratory study using GC/MS.

<sup>(d)</sup> Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty,  $U$ , is calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the analyte mean, and the coverage factor,  $k$ , is determined from the Student's  $t$ -distribution corresponding to the associated degrees of freedom ( $df = 2$ ) and 95 % confidence level for each analyte.

<sup>(e)</sup> The reference value for each analyte is the equally-weighted mean of the means from two or more analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study.

<sup>(f)</sup> GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(g)</sup> GC-ECD (IB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IA).

<sup>(h)</sup> GC-ECD (IIA) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

<sup>(i)</sup> GC-ECD (IIB) on the 50 % octadecyl (C-18) methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

<sup>(j)</sup> GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(k)</sup> GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone mixture.

<sup>(l)</sup> GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).

<sup>(m)</sup> Results from nineteen laboratories participating in an interlaboratory comparison exercise.

Table 8. Reference Mass Fraction Values for Selected PBDEs in SRM 1944 (Dry-Mass Basis)

	Mass Fractions <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )
PBDE 47 (2,2',4,4'-Tetrabromodiphenyl ether) <sup>(c,d,e,f)</sup>	1.72 $\pm$ 0.28 <sup>(b)</sup>
PBDE 99 (2,2',4,4',5-Pentabromodiphenyl ether) <sup>(c,d,f)</sup>	1.98 $\pm$ 0.26 <sup>(b)</sup>
PBDE 100 (2,2',4,4',6-Pentabromodiphenyl ether) <sup>(c,d)</sup>	0.447 $\pm$ 0.027 <sup>(b)</sup>
PBDE 153 (2,2',4,4',5,5'-Hexabromodiphenyl ether) <sup>(c,d,e,f)</sup>	6.44 $\pm$ 0.37 <sup>(b)</sup>
PBDE 154 (2,2',4,4',5,6'-Hexabromodiphenyl ether) <sup>(c,d,f)</sup>	1.06 $\pm$ 0.08 <sup>(b)</sup>
PBDE 183 (2,2',3,4,4',5',6-Heptabromodiphenyl ether) <sup>(c,d,e,f)</sup>	31.8 $\pm$ 0.1 <sup>(b)</sup>
PBDE 206 (2,2',3,3',4,4',5,5',6-Nonabromodiphenyl ether) <sup>(d,e)</sup>	6.2 $\pm$ 1.0 <sup>(b)</sup>
PBDE 209 (Decabromodiphenyl ether) <sup>(c,d,e,f)</sup>	93.5 $\pm$ 4.4 <sup>(b)</sup>

<sup>(a)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(b)</sup> Reference values are weighted means of the results from two to four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/JCGM/NIST Guide to the Expression of Uncertainty in Measurements [11,12].

<sup>(c)</sup> Results from ten laboratories participating in an interlaboratory study for PBDEs in sediment [12].

<sup>(d)</sup> Results from four laboratories participating in the 2007 interlaboratory study [13].

<sup>(e)</sup> NIST participation in the 2007 interlaboratory study using GC/MS.

<sup>(f)</sup> Data set from NIST for PBDEs using GC/MS following PFE with alumina SPE and SEC clean-up.

Table 9. Reference Mass Fraction Values for Selected Elements in SRM 1944 (Dry-Mass Basis)

	Degrees of Freedom	Mass Fraction <sup>(a,b)</sup> (%)
Silicon <sup>c,d</sup>	81	31 $\pm$ 3
		Mass Fraction <sup>(a,b)</sup> (mg/kg)
Antimony <sup>(c,e,f,g)</sup>	18	4.6 $\pm$ 0.9
Beryllium <sup>(c,h)</sup>	17	1.6 $\pm$ 0.3
Copper <sup>(c,d,f)</sup>	101	380 $\pm$ 40
Mercury <sup>(c,i)</sup>	18	3.4 $\pm$ 0.5
Selenium <sup>(c,e,f)</sup>	24	1.4 $\pm$ 0.2
Silver <sup>(c,d,e,g)</sup>	8	6.4 $\pm$ 1.7
Thallium <sup>(c,f)</sup>	12	0.59 $\pm$ 0.1
Tin <sup>(c,f)</sup>	22	42 $\pm$ 6

<sup>(a)</sup> The reference value is the equally weighted mean of available results from: (1) NIST INAA analyses, (2) two methods performed at NRCC, (3) results from seven selected laboratories participating in the NRCC intercomparison exercise, and (4) results from INAA analyses at IAEA. The expanded uncertainty in the reference value is equal to  $U = k u_c$  where  $u_c$  is the combined standard uncertainty and  $k$  is the coverage factor, both calculated according to the ISO/JCGM Guide [11,12]. The value of  $u_c$  is intended to represent at the level of one standard deviation the uncertainty in the value. Here  $u_c$  accounts for possible method differences, within-method variation, and material inhomogeneity. The coverage factor,  $k$ , is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple test portions can be expected to be greater than that due to measurement variability alone.

<sup>(b)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(c)</sup> Results from five to seven laboratories participating in the NRCC interlaboratory comparison exercise.

<sup>(d)</sup> Measured at NRCC using GFAAS.

<sup>(e)</sup> Measured at NIST using INAA.

<sup>(f)</sup> Measured at NRCC using ID-ICPMS.

<sup>(g)</sup> Measured at IAEA using INAA.

<sup>(h)</sup> Measured at NRCC using ICPOES.

<sup>(i)</sup> Measured at NRCC using cold vapor atomic absorption spectroscopy (CVAAS).

Table 10. Reference Mass Fraction Values for Elements in SRM 1944 as Determined by INAA (Dry-Mass Basis)

	Effective Degrees of Freedom	Mass Fraction <sup>(a,b)</sup>		
		(% )		
Calcium	21	1.0	±	0.1
Chlorine	21	1.4	±	0.2
Potassium	21	1.6	±	0.2
Sodium	25	1.9	±	0.1
		Mass Fraction <sup>(a,b)</sup>		
		(mg/kg)		
Bromine	10	86	±	10
Cesium	11	3.0	±	0.3
Cobalt	10	14	±	2
Rubidium	14	75	±	2
Scandium	37	10.2	±	0.2
Titanium	21	4300	±	300
Vanadium	21	100	±	9

<sup>(a)</sup> The reference value is based on the results from an INAA study. The associated uncertainty accounts for both random and systematic effects, but because only one method was used, the results should be used with caution. The expanded uncertainty in the reference value is equal to  $U = ku_c$  where  $u_c$  is the combined standard uncertainty and  $k$  is the coverage factor, both calculated according to the ISO/JCGM Guide [11,12]. The value of  $u_c$  is intended to represent at the level of one standard deviation the uncertainty in the value. Here  $u_c$  accounts for possible method differences, within-method variation, and material inhomogeneity. The coverage factor,  $k$ , is the Student's t-value for a 95 % confidence interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple test portions can be expected to be greater than that due to measurement variability alone.

<sup>(b)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 11. Reference Mass Fraction Values for Selected Dibenzo-*p*-Dioxin and Dibenzofuran Congeners in SRM 1944 (Dry-Mass Basis)

	Mass Fraction <sup>(a,b)</sup> (µg/kg)		
2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin	0.133	±	0.009
1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin	0.019	±	0.002
1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	0.026	±	0.003
1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	0.056	±	0.006
1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin	0.053	±	0.007
1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin	0.80	±	0.07
Octachlorodibenzo- <i>p</i> -dioxin	5.8	±	0.7
2,3,7,8-Tetrachlorodibenzofuran <sup>(c)</sup>	0.039	±	0.015 <sup>(d)</sup>
1,2,3,7,8-Pentachlorodibenzofuran	0.045	±	0.007
2,3,4,7,8-Pentachlorodibenzofuran	0.045	±	0.004
1,2,3,4,7,8-Hexachlorodibenzofuran	0.22	±	0.03
1,2,3,6,7,8-Hexachlorodibenzofuran	0.09	±	0.01
2,3,4,6,7,8-Hexachlorodibenzofuran	0.054	±	0.006 <sup>(e)</sup>
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.0	±	0.1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.040	±	0.006 <sup>(e)</sup>
Octachlorodibenzofuran	1.0	±	0.1
Total Toxic Equivalents (TEQ) <sup>(f)</sup>	0.25	±	0.01
Total Tetrachlorodibenzo- <i>p</i> -dioxins	0.25	±	0.05 <sup>(e)</sup>
Total Pentachlorodibenzo- <i>p</i> -dioxins	0.19	±	0.06
Total Hexachlorodibenzo- <i>p</i> -dioxins	0.63	±	0.09
Total Heptachlorodibenzo- <i>p</i> -dioxins	1.8	±	0.2
Total Tetrachlorodibenzofurans	0.7	±	0.2
Total Pentachlorodibenzofurans	0.74	±	0.07
Total Hexachlorodibenzofurans	1.0	±	0.1
Total Heptachlorodibenzofurans	1.5	±	0.1
Total Dibenzo- <i>p</i> -dioxins <sup>(g)</sup>	8.7	±	0.9
Total Dibenzofurans <sup>(g)</sup>	5.0	±	0.5

<sup>(a)</sup> Each reference value is the mean of the results from up to fourteen laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to  $U = k u_c$  where  $u_c$  is the combined standard uncertainty calculated according to the ISO/JCGM Guide [11,12] and  $k$  is the coverage factor. The value of  $u_c$  is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the reference value. Here  $u_c$  is the uncertainty in the mean arising from the variation among the laboratory results. The degrees of freedom is equal to the number of available results minus one (13 unless noted otherwise). The coverage factor,  $k$ , is the value from a Student's *t*-distribution for a 95 % confidence interval.

<sup>(b)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(c)</sup> Confirmation results using a 50 % cyanopropyl phenyl polysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenyl polysiloxane phase columns.

<sup>(d)</sup> Degrees of freedom = 7 for this compound.

<sup>(e)</sup> Degrees of freedom = 12 for this compound.

<sup>(f)</sup> TEQ is the sum of the products of each of the 2,3,7,8-substituted congeners multiplied by their individual toxic equivalency factors (TEFs) recommended by the North Atlantic Treaty Organization (NATO) [15]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

<sup>(g)</sup> Total of tetra- through octachlorinated congeners.

The measurands for reference values are indicated in Tables 12-13; as determined by the methods indicated in the text. Metrological traceability is to the SI unit for length and the SI derived units of area and volume, expressed as micrometers, and square meters per cubic centimeters; respectively.

Table 12. Reference Values for Particle Size Characteristics for SRM 1944

Particle Measurement	Value <sup>(a)</sup>
Mean diameter (volume distribution, MV, $\mu\text{m}$ ) <sup>(b)</sup>	151.2 $\pm$ 0.4
Mean diameter (area distribution, $\mu\text{m}$ ) <sup>(c)</sup>	120.4 $\pm$ 0.1
Mean diameter (number distribution, $\mu\text{m}$ ) <sup>(d)</sup>	75.7 $\pm$ 0.3
Surface Area ( $\text{m}^2/\text{cm}^3$ ) <sup>(e)</sup>	0.050 $\pm$ 0.013

<sup>(a)</sup> The reference value is the mean value of measurements from the analysis of test portions from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO/JCGM Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

<sup>(b)</sup> The mean diameter of the volume distribution represents the center of gravity of the distribution and compensates for scattering efficiency and refractive index. This parameter is strongly influenced by coarse particles.

<sup>(c)</sup> The mean diameter of the area distribution, calculated from the volume distribution with less weighting by the presence of coarse particles than MV.

<sup>(d)</sup> The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

<sup>(e)</sup> Calculated specific surface area assuming solid, spherical particles. This is a computation and should not be interchanged with an adsorption method of surface area determination as this value does not reflect porosity or topographical characteristics.

Table 13. Percentage of the Volume That is Smaller Than the Indicated Size

Percentile	Particle Diameter <sup>(a)</sup> ( $\mu\text{m}$ )
95	296 $\pm$ 5
90	247 $\pm$ 2
80	201 $\pm$ 1
70	174 $\pm$ 1
60	152 $\pm$ 1
50 <sup>(b)</sup>	135 $\pm$ 1
40	120 $\pm$ 1
30	106 $\pm$ 1
20	91 $\pm$ 1
10	74 $\pm$ 1

<sup>(a)</sup> The reference value for particle diameter is the mean value of measurements from the analysis of test portions from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO/JCGM Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

<sup>(b)</sup> Median diameter (50 % of the volume is less than 135  $\mu\text{m}$ ).

Table 14. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1944

	Mass Fraction (%)		
Total Organic Carbon (TOC) <sup>(a,b)</sup>	4.4	±	0.3
Extractable Mass <sup>(c,d)</sup>	1.15	±	0.04

<sup>(a)</sup> Mass fraction is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(b)</sup> The reference value for total organic carbon is an equally weighted mean value from routine measurements made by three laboratories. Each uncertainty, computed according to the CIPM approach as described in the ISO/JCGM Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

<sup>(c)</sup> Extractable mass as determined from Soxhlet extraction using DCM.

<sup>(d)</sup> The reference value for extractable mass is the mean value of six measurements. Each uncertainty, computed according to the CIPM approach as described in the ISO/JCGM Guide [11,12], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. The expanded uncertainty defines a range of values for the reference value within which the true value is believed to lie, at a level of confidence of 95 %.

Table 15. Information Mass Fraction Values for Selected Elements in SRM 1944 as Determined by INAA (Dry-Mass Basis)

	Mass Fraction <sup>(a)</sup> (%)
Magnesium <sup>(b)</sup>	1.0

  

	Mass Fraction <sup>(a)</sup> (mg/kg)
Cerium <sup>(b)</sup>	65
Europium <sup>(b)</sup>	1.3
Gold <sup>(b)</sup>	0.10
Lanthanum <sup>(b)</sup>	39
Thorium <sup>(b)</sup>	13
Uranium <sup>(b)</sup>	3.1

<sup>(a)</sup> Mass fraction is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

<sup>(b)</sup> Measured at IAEA using INAA

Table 16. Information Mass Fraction Values for Selected Polychlorinated Naphthalenes in SRM 1944 (Dry-Mass Basis)

		Mass Fraction <sup>(a)</sup> (µg/kg)
PCN	19 (1,3,5-Trichloronaphthalene)	1.4
PCN	23 (1,4,5-Trichloronaphthalene)	2.4
PCN	42 (1,3,5,7-Tetrachloronaphthalene)	2.7
PCN	47 (1,4,6,7-Tetrachloronaphthalene)	3.5
PCN	52 (1,2,3,5,7-Pentachloronaphthalene)	2.5
	60 (1,2,4,6,7-Pentachloronaphthalene)	
PCN	50 (1,2,3,4,6-Pentachloronaphthalene)	1.0
PCN	66 (1,2,3,4,6,7-Hexachloronaphthalene)	0.63
	67 (1,2,3,5,6,7-Hexachloronaphthalene)	
PCN	69 (1,2,3,5,7,8-Hexachloronaphthalene)	1.6
PCN	73 (1,2,3,4,5,6,7-Heptachloronaphthalene)	0.51
PCN	75 (Octachloronaphthalene)	0.20

<sup>(a)</sup> Mass fractions reported on dry-mass basis; material as received contains approximately 1.3 % moisture. Information values are the median of the results from six laboratories participating in an interlaboratory comparison exercise (Appendix D).

Table 17. Information Mass Fraction Values for Three HBCD Isomers in SRM 1944 (Dry-Mass Basis)

	Mass Fraction <sup>(a,b)</sup> (µg/kg)
<i>alpha</i> -HBCD <sup>(b)</sup>	2.2
<i>beta</i> -HBCD <sup>(b)</sup>	1.0
<i>gamma</i> -HBCD <sup>(b)</sup>	18

<sup>(a)</sup> The information value is the median of the results from three analytical methods.

<sup>(b)</sup> Mass fractions are reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 18. Analytical Methods Used for the Measurement of Elements in SRM 1944

<b>Elements</b>	<b>Analytical Methods</b>
Aluminum	FAAS, ICPOES, INAA, XRF
Antimony	GFAAS, HGAAS, ICP-MS, ID-ICPMS, INAA
Arsenic	GFAAS, HGAAS, ICPMS, INAA, XRF
Beryllium	GFAAS, ICP-AES, ICPMS
Bromine	INAA
Cadmium	FAAS, GFAAS, ICPMS, ID-ICPMS
Calcium	INAA
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	FAAS, GFAAS, ICPMS, ID-ICPMS, INAA, XRF
Cobalt	INAA
Copper	FAAS, GFAAS, ICPOES, ICPMS, ID-ICPMS, XRF
Europium	INAA
Gold	INAA
Iron	FAAS, ICPOES, ICPMS, ID-ICPMS, INAA, XRF
Lanthanum	INAA
Lead	FAAS, GFAAS, ICPMS, ID-ICPMS, XRF
Magnesium	INAA
Manganese	FAAS, ICPOES, ICPMS, INAA, XRF
Mercury	CVAAS, ICPMS
Nickel	GFAAS, ICPOES, ICPMS, ID-ICPMS, INAA, XRF
Potassium	INAA
Rubidium	INAA
Scandium	INAA
Selenium	GFAAS, HGAAS, ICPMS, INAA
Silicon	FAAS, ICPOES, XRF
Silver	FAAS, GFAAS, ICPMS, INAA
Sodium	INAA
Thallium	GFAAS, ICPOES, ICPMS, ID-ICPMS,
Thorium	INAA
Tin	GFAAS, ICPMS, ID-ICPMS
Titanium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICPOES, ICPMS, ID-ICPMS, XRF, INAA
 <b>Methods</b>	
CVAAS	Cold vapor atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICPOES	Inductively coupled plasma optical emission spectrometry
ICPMS	Inductively coupled plasma mass spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
XRF	X-ray fluorescence spectrometry

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*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <http://www.nist.gov/srm>.*

## APPENDIX A

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of PBDEs in SRM 1944 [4].

D. Hoover and C. Hamilton, AXYS Analytical, Sidney, BC, Canada  
S. Klosterhaus and J. Baker, Chesapeake Biological Laboratory, Solomons, MD, USA  
S. Backus, Environment Canada, Ecosystem Health Division, Burlington, ON, Canada  
E. Sverko, Environment Canada, Canada Centre for Inland Waters, Burlington, ON, Canada  
P. Lepom, Federal Environmental Agency, Berlin, Germany  
R. Hites and L. Zhu, Indiana University, Bloomington, IN, USA  
G. Jiang, Research Center for Eco-Environmental Sciences, Beijing, China  
H. Takada, Tokyo University of Agriculture and Technology, Tokyo, Japan  
A. Covaci and S. Vorspoels, University of Antwerp, Antwerp, Belgium  
A. Li, University of Illinois at Chicago, Chicago, IL, USA

## APPENDIX B

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1944.

W.J. Luksemburg, Alta Analytical Laboratory, Inc., El Dorado Hills, CA, USA  
L. Phillips, AXYS Analytical Services Ltd., Sidney, British Columbia, Canada  
M.J. Armbruster, Battelle Columbus Laboratories, Columbus, OH, USA  
G. Reuel, Canviro Analytical Laboratories Ltd., Waterloo, Ontario, Canada  
C. Brochu, Environment Québec, Laval, Québec, Canada  
G. Poole, Environment Canada Environmental Technology Centre, Ottawa, Ontario, Canada  
B. Henkelmann, GSF National Research Center for Environment and Health, Neuherberg, Germany  
R. Anderson, Institute of Environmental Chemistry, Umeå University, Umeå, Sweden  
C. Lastoria, Maxxam Analytics Inc., Mississauga, Ontario, Canada  
E. Reiner, Ontario Ministry of Environment and Energy, Etobicoke, Ontario, Canada  
J. Macaulay, Research and Productivity Council, Fredericton, New Brunswick, Canada  
T.L. Wade, Texas A&M University, College Station, TX, USA  
C. Tashiro, Wellington Laboratories, Guelph, Ontario, Canada  
T.O. Tiernan, Wright State University, Dayton, OH, USA

## APPENDIX C

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of trace elements in SRM 1944.

A. Abby, Applied Marine Research Laboratory, Old Dominion University, Norfolk, VA, USA  
A. Scott, Australian Government Analytical Laboratories, Pymble, Australia  
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## APPENDIX D

The analysts and laboratories listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated naphthalenes in SRM 1944.

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