



DataPack™

Lot No. D053-540

Trace Metals in Soil

Catalog No. 540

Certification

Method 3051 HNO₃

Parameter	Total Concentration ¹ (mg/Kg)	Certified Value ² (mg/Kg)	Performance Acceptance Limits™ ³ (mg/Kg)
aluminum	56600*	8420	4530 - 12300
antimony	250	51.3	D.L. - 129
arsenic	155	133	104 - 162
barium	990	316	262 - 370
beryllium	100	89.4	75.2 - 104
boron	159	110	69.0 - 151
cadmium	78.6	67.1	57.2 - 77.0
calcium	10200*	4160	3480 - 4840
chromium	87.2	71.0	59.0 - 83.0
cobalt	83.6	71.8	58.1 - 85.5
copper	75.3	65.0	54.4 - 75.6
iron	24400*	12600	7520 - 17700
lead	156	132	110 - 154
magnesium	4260	2620	2060 - 3180
manganese	745	438	370 - 506
mercury	8.84	8.28	5.47 - 11.0
molybdenum	59.7	45.4	33.9 - 56.9
nickel	68.2	56.7	46.6 - 66.8
potassium	33800*	3660	2650 - 4670
selenium	184	160	120 - 200
silver	117	44.6	D.L. - 96.0
sodium	14900*	596	426 - 766
strontium	264	53.1	42.9 - 63.3
thallium	154	130	105 - 155
tin	206	42.8	21.8 - 63.8
titanium	3100	156	D.L. - 325
vanadium	127	79.1	62.8 - 95.4
zinc	211	182	152 - 212

Method 3051 HNO₃, HCl

Parameter	Total Concentration ¹ mg/Kg	Certified Value ² mg/Kg	Performance Acceptance Limits™ ³ mg/Kg
aluminum	56600*	8120	4130 - 12100
antimony	250	192	93.0 - 291
arsenic	155	138	108 - 168
barium	990	317	264 - 370
beryllium	100	87.6	75.2 - 100
boron	159	115	78.0 - 152
cadmium	78.6	67.2	56.3 - 78.1
calcium	10200*	4060	3310 - 4810
chromium	87.2	77.7	63.2 - 92.2
cobalt	83.6	73.6	59.7 - 87.5
copper	75.3	66.2	55.3 - 77.1
iron	24400*	16200	9140 - 23300
lead	156	131	111 - 151
magnesium	4260	2640	1990 - 3290
manganese	745	443	355 - 531
mercury	8.84	8.28	5.47 - 11.0
molybdenum	59.7	53.3	43.9 - 62.7
nickel	68.2	59.8	48.1 - 71.5
potassium	33800*	3700	2610 - 4790
selenium	184	164	125 - 203
silver	117	101	66.7 - 145
sodium	14900*	570	358 - 782
strontium	264	53.6	41.1 - 66.1
thallium	154	136	107 - 167
tin	206	140	86.9 - 193
titanium	3100	246	49.1 - 443
vanadium	127	93.7	70.0 - 117
zinc	211	178	148 - 208



Certificate of Analysis

Standard Reference Material[®] 1944

New York/New Jersey Waterway Sediment

This Standard Reference Material (SRM) 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 dibenzo-*p*-dioxin and dibenzofuran congeners, total organic carbon, total extractable material, and particle-size characteristics. All of the constituents for which certified, reference, and information values are provided in SRM 1944 were naturally present in the sediment material before processing. A unit of SRM 1944 consists of a bottle containing 50 g of radiation sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 24 PAHs, 35 PCB congeners (some in combination), four chlorinated pesticides, and nine trace elements are provided in Tables 1-4. 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 accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from 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 Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 32 additional PAHs (some in combination) in Table 5, seven additional chlorinated pesticides in Table 6, and 19 additional inorganic constituents in Tables 7 and 8. Reference values are provided in Table 9 for the 17 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 10. Reference values for total organic carbon and percent extractable mass are provided in Table 11. 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. Explanations in support of each reference value are given as notes in Tables 5-11.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 12 for eight additional trace elements. 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.

Expiration of Certification: The certification of SRM 1944 is valid, within the measurement uncertainty specified, until **31 March 2019**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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) will facilitate notification.

The coordination of the technical measurements leading to the certification was under the leadership of S.A. Wise of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Consultation on the statistical design of the experimental work and evaluation of the data were provided by 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 Measurement Services Division.

The sediment material 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 material. 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. Demiralp, R.R. Greenberg, M. Lopez de Alda, K.E. Murphy, B.J. Porter, D.L. Poster, L.C. Sander, M.M. Schantz, 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). The particle-size distribution data were provided by Honeywell, Inc. (Clearwater, FL).

Analytical measurements for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among 14 laboratories (see Appendix A) coordinated by S.A. Wise of the NIST Analytical Chemistry Division and R. Turle and C. Chiu of Environment Canada, Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, Ontario, 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 B) that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, National Research Council Canada (NRCC, Ottawa, Ontario, Canada).

NOTICE AND WARNING TO USERS

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

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.

INSTRUCTIONS FOR USE

Prior to removal of subsamples 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 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 subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS¹

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 µm to 61 µm), homogenized in a cone blender, radiation sterilized (⁶⁰Co), and then packaged in screw-capped amber glass bottles.

¹Certain commercial equipment, instruments or materials are identified in this certificate 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.

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 subsamples of 1.6 g to 2.5 g for five days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1944 at the time of the certification analyses was 1.25 % ± 0.03 % (95 % confidence level).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [1-5] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup 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) for 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 spectrometric (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase, and a smectic liquid crystalline stationary phase.

Six sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), GC/MS (V), and GC/MS (Sm), were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples 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. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 1 g to 2 g subsamples from three bottles of SRM 1944 and 2 g to 3 g subsamples from three bottles of SRM 1944 that had been mixed with a similar amount of water (i.e., a wetted sediment). These samples 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 [6-9]. The PAH fraction was then analyzed using the same column as described above for GC/MS (I); however, the subsamples 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 subsamples from six bottles of SRM 1944 were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone (volume fractions). The extracts were then processed and analyzed as described for GC/MS (II). For GC/MS (IV) analyses, 1 g to 2 g subsamples from six bottles of SRM 1944 were extracted using PFE with a mixture of 50 % hexane/50 % acetone as described by Schantz et al. [10], 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 % phenyl-substituted methylpolysiloxane stationary phase (0.25 mm i.d. × 60 m, 0.25 µm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For GC/MS (Sm) 1 g to 2 g subsamples from six bottles of SRM 1944 were Soxhlet extracted for 24 h 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. Subsamples of approximately 1 g from six bottles of SRM 1944 were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone (volume fractions). The extracts were concentrated and then processed through two aminopropylsilane solid phase extraction (SPE) cartridges connected in series to obtain the total PAH fraction. A second 1 g subsample 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 NH₂, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [6-9]. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5-µm particle-size polymeric octadecylsilane (C₁₈) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [7,8]. 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 samples of 1 g from eight bottles selected by stratified random sampling. Samples 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 sample size.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1944 was similar to that reported for the recent certification of several environmental matrix SRMs [2,4,11,12,13], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. 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 subsamples 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-18) methylpolysiloxane phase (0.1 µm film thickness) (CPSil 5 C18 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-18 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). Subsamples of 1 g to 2 g from three bottles of SRM 1944 and 2 g to 3 g subsamples 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 subsamples 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 subsamples 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 subsamples were extracted using PFE as described by Schantz et al. [10]. 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 [14,15]), and 4,4'-DDT-*d*₈ 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 [16]. Results from 19 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.

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans: Value assignment of the concentrations of the 17 2,3,7,8-substituted 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 14 laboratories that participated in an interlaboratory comparison study (see Appendix A). Each laboratory analyzed three subsamples (typically 1 g) of SRM 1944 using their routine analytical procedures and gas chromatography with high resolution mass spectrometric detection (GC-HRMS).

The analytical procedures used by all of the laboratories included spiking with ^{13}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 % (mole fraction) cyanopropylphenyl-substituted methylpolysiloxane capillary column (DB-225, J&W Scientific, Folsom, CA).

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 selected laboratories that participated in an interlaboratory comparison exercise coordinated by the NRCC [17] (see Appendix B). A similar approach was recently used to provide certified and reference concentration values for trace elements in two mussel tissue materials [18-20]. The analytical methods used for the determination of each element are summarized in Table 13. 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 atomic emission spectroscopy (ICPAES), (3) analyses at IAEA using INAA, and (4) the mean of the results from seven selected laboratories that participated in the NRC interlaboratory comparison exercise. The reference concentration values in Table 7 were determined by combining results from (1) analyses performed at NIST using INAA; (2) analyses at NRCC using ID-ICPMS, GFAAS, ICPAES, 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 12 were determined by INAA at NIST and IAEA.

NIST Analyses using ID-ICPMS: Lead, cadmium, and nickel were determined by ID-ICPMS [21]. Subsamples (0.4 g to 0.5 g) from six bottles of the SRM were spiked with ^{206}Pb , ^{111}Cd , and ^{62}Ni and wet ashed using a combination of nitric, hydrochloric, hydrofluoric, and perchloric acids. Lead and cadmium were determined in the same sample; 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 [22]. Elements with short-lived irradiation products (Al, Ca, Cl, K, Mg, Mn, Na, Ti, and V) were determined by measuring duplicate 300 mg samples from each of 10 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 megawatts which corresponds to a neutron fluence rate of about $8 \times 10^{13} \text{ cm}^{-2}\cdot\text{s}^{-1}$. 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 sample 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 megawatts. 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.

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 μm to 700 μm .

Total Organic Carbon and Percent Extractable Mass: Four laboratories provided results for Total Organic Carbon (TOC) using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6 N 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 subsamples from six bottles of SRM 1944. For the determination of percent extractable mass, six subsamples 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.

Table 1. Certified Concentrations for Selected PAHs in SRM 1944

PAHs	Mass Fractions in mg/kg (dry-mass basis) ^(a,b)		
Naphthalene ^(c,d,e,f,g)	1.65	±	0.31
Phenanthrene ^(c,d,e,f,g)	5.27	±	0.22
Anthracene ^(c,d,e,f,g)	1.77	±	0.33
Fluoranthene ^(c,d,e,f,g)	8.92	±	0.32
Pyrene ^(c,d,e,f,g)	9.70	±	0.42
Benzo[<i>c</i>]phenanthrene ^(c,d,e,f,h)	0.76	±	0.10
Benzo[<i>a</i>]anthracene ^(c,d,e,f,g,h)	4.72	±	0.11
Chrysene ^(h,k)	4.86	±	0.10 ⁱ
Triphenylene ^(h,k)	1.04	±	0.27
Benzo[<i>b</i>]fluoranthene ^(g,h,j)	3.87	±	0.42
Benzo[<i>j</i>]fluoranthene ^(h,j)	2.09	±	0.44
Benzo[<i>k</i>]fluoranthene ^(c,d,e,f,g,h,j)	2.30	±	0.20
Benzo[<i>a</i>]fluoranthene ^(c,d,e,f,h,j)	0.78	±	0.12
Benzo[<i>e</i>]pyrene ^(c,d,e,f,h,j)	3.28	±	0.11
Benzo[<i>a</i>]pyrene ^(c,d,e,f,g,h,j)	4.30	±	0.13
Perylene ^(c,d,e,f,g,h,j)	1.17	±	0.24
Benzo[<i>ghi</i>]perylene ^(c,d,e,f,j,k)	2.84	±	0.10
Indeno[1,2,3- <i>cd</i>]pyrene ^(c,d,e,f,j,k)	2.78	±	0.10
Dibenz[<i>a,j</i>]anthracene ^(c,d,e,f,j,k)	0.500	±	0.044
Dibenz[<i>a,c</i>]anthracene ^(j,k)	0.335	±	0.013
Dibenz[<i>a,h</i>]anthracene ^(j,k)	0.424	±	0.069
Pentaphene ^(c,d,e,f,j,k)	0.288	±	0.026
Benzo[<i>b</i>]chrysene ^(c,d,e,f,j,k,h)	0.63	±	0.10
Picene ^(c,d,e,f,j,k)	0.518	±	0.093

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^(b) The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], 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/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

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

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

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

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

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

⁽ⁱ⁾ The uncertainty interval for chrysene was widened based on expert consideration of the analytical methods and analysis of the data for all PAHs, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

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

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

Table 2. Certified Concentrations for Selected PCB Congeners^(a) in SRM 1944

PCB Congeners	Mass Fractions in $\mu\text{g}/\text{kg}$ (dry-mass basis) ^(b,c)
PCB 8 (2,4'-Dichlorobiphenyl) ^(d,e,f,g,h,i,j,k)	22.3 \pm 2.3
PCB 18 (2,2',5-Trichlorobiphenyl) ^(d,e,f,g,h,i,j,k)	51.0 \pm 2.6
PCB 28 (2,4,4'-Trichlorobiphenyl) ^(d,e,f,g,j,k)	80.8 \pm 2.7
PCB 31 (2,4',5-Trichlorobiphenyl) ^(d,e,f,g,j)	78.7 \pm 1.6 ^l
PCB 44 (2,2',3,5'-Tetrachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	60.2 \pm 2.0
PCB 49 (2,2',4,5'-Tetrachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	53.0 \pm 1.7
PCB 52 (2,2',5,5'-Tetrachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	79.4 \pm 2.0
PCB 66 (2,3',4,4'-Tetrachlorobiphenyl) ^(e,g,h,i,j)	71.9 \pm 4.3
PCB 87 (2,2',3,4,5'-Pentachlorobiphenyl) ^(d,e,f,g,h,i,j)	29.9 \pm 4.3
PCB 95 (2,2',3,5',6-Pentachlorobiphenyl) ^(e,g,h,i,j)	65.0 \pm 8.9
PCB 99 (2,2',4,4',5-Pentachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	37.5 \pm 2.4
PCB 101 (2,2',4,5,5'-Pentachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	73.4 \pm 2.5
90 (2,2',3,4',5-Pentachlorobiphenyl)	
PCB 105 (2,3,3',4,4'-Pentachlorobiphenyl) ^(e,f,g,h,i,j,k)	24.5 \pm 1.1
PCB 110 (2,3,3',4',6-Pentachlorobiphenyl) ^(g,h,i,j)	63.5 \pm 4.7
PCB 118 (2,3',4,4',5-Pentachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	58.0 \pm 4.3
PCB 128 (2,2',3,3',4,4'-Hexachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	8.47 \pm 0.28
PCB 138 (2,2',3,4,4',5'-Hexachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	62.1 \pm 3.0
163 (2,3,3',4',5,6-Hexachlorobiphenyl)	
164 (2,3,3',4',5',6-Hexachlorobiphenyl)	
PCB 149 (2,2',3,4',5',6-Hexachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	49.7 \pm 1.2
PCB 151 (2,2',3,5,5',6-Hexachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	16.93 \pm 0.36
PCB 153 (2,2',4,4',5,5'-Hexachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	74.0 \pm 2.9
PCB 156 (2,3,3',4,4',5-Hexachlorobiphenyl) ^(d,e,f,g,h,i,j)	6.52 \pm 0.66
PCB 170 (2,2',3,3',4,4',5-Heptachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	22.6 \pm 1.4
190 (2,3,3',4,4',5,5'-Heptachlorobiphenyl)	
PCB 180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	44.3 \pm 1.2
PCB 183 (2,2',3,4,4',5',6-Heptachlorobiphenyl) ^(d,e,f,g,h,i,j)	12.19 \pm 0.57
PCB 187 (2,2',3,4',5,5',6-Heptachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	25.1 \pm 1.0
159 (2,3,3',4,5,5'-Hexachlorobiphenyl)	
182 (2,2',3',4,4',5,6'-Heptachlorobiphenyl)	
PCB 194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl) ^(d,e,f,g,h,i,j)	11.2 \pm 1.4
PCB 195 (2,2',3,3',4,4',5,6-Octachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	3.75 \pm 0.39
PCB 206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) ^(d,e,f,g,h,i,j,k)	9.21 \pm 0.51
PCB 209 Decachlorobiphenyl ^(d,e,f,g,h,i,j,k)	6.81 \pm 0.33

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [14] and later revised by Schulte and Malisch [15] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the conditions used, the congener listed first is the major component; additional congeners may be present as minor components.

^(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^(c) The results are expressed as the certified value \pm the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], 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 %.

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

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

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

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

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

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

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

^(k) Results from 19 laboratories participating in an interlaboratory comparison exercise.

^(l) The uncertainty interval for PCB 31 was widened based on expert consideration of the analytical methods and analysis of the data for all PCB congeners, which suggests that the half-widths of the expanded uncertainties should not be less than 2 %.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1944

Chlorinated Pesticides	Mass Fractions in $\mu\text{g}/\text{kg}$ (dry-mass basis) ^(a,b)	
Hexachlorobenzene ^(e,f,g,h,i,j)	6.03	\pm 0.35
<i>cis</i> -Chlordane (α -Chlordane) ^(c,d,e,f,g,h,i,j)	16.51	\pm 0.83
<i>trans</i> -Nonachlor ^(c,d,e,f,g,h,i,j)	8.20	\pm 0.51
4,4'-DDT ^(c,d,e,f,g,h,i,j)	119	\pm 11

- (a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.
- (b) The results are expressed as the certified value \pm the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [23]. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], 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.
- (h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.
- (i) GC/MS (III) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (IIA).
- (j) Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Concentrations for Selected Inorganic Constituents in SRM 1944

Elements	Degrees of Freedom	Mass Fractions in percent (dry-mass basis) ^(a,b)	
Aluminum ^(c,d,e)	4	5.33	\pm 0.49
Iron ^(c,d,e)	6	3.53	\pm 0.16

		Mass Fractions in mg/kg (dry-mass basis) ^(a,b)	
Arsenic ^(c,d,e,f,g)	10	18.9	\pm 2.8
Cadmium ^(c,f,h,i)	6	8.8	\pm 1.4
Chromium ^(c,d,f,g,i)	9	266	\pm 24
Lead ^(c,h,i)	5	330	\pm 48
Manganese ^(c,d,e)	8	505	\pm 25
Nickel ^(c,g,h,i)	6	76.1	\pm 5.6
Zinc ^(c,d,e,g,i)	9	656	\pm 75

- (a) The results are expressed as the certified value \pm the expanded uncertainty. 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 $U = ku_c$, where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO and NIST Guides [24]. 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 % prediction 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) Concentrations 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 ICPAES.
- (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.

Table 5. Reference Concentrations for Selected PAHs in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

PAHs	Mass Fractions in mg/kg (dry-mass basis) ^(a,b)
1-Methylnaphthalene ^(c,d,e,f)	0.52 ± 0.03
2-Methylnaphthalene ^(c,d,e,f)	0.95 ± 0.05
Biphenyl ^(c,d,e,f)	0.32 ± 0.07
Acenaphthene ^(c,d,e,f)	0.57 ± 0.03
Fluorene ^(c,d,e,f)	0.85 ± 0.03
Dibenzothiophene ^(d,e,f)	0.62 ± 0.01 ^(g)
1-Methylphenanthrene ^(c,d,e,f)	1.7 ± 0.1
2-Methylphenanthrene ^(c,d,e,f)	1.90 ± 0.06
3-Methylphenanthrene ^(c,d,e,f)	2.1 ± 0.1
4-Methylphenanthrene and 9-Methylphenanthrene ^(c,d,e,f)	1.6 ± 0.2
2-Methylanthracene ^(c,d,e,f)	0.58 ± 0.04
3,5-Dimethylphenanthrene ^(c)	1.31 ± 0.04
2,6-Dimethylphenanthrene ^(c)	0.79 ± 0.02 ^(g)
2,7-Dimethylphenanthrene ^(c)	0.67 ± 0.02 ^(g)
3,9-Dimethylphenanthrene ^(c)	2.42 ± 0.05 ^(g)
1,6-, 2,9-, and 2,5-Dimethylphenanthrene ^(c)	1.67 ± 0.03 ^(g)
1,7-Dimethylphenanthrene ^(c)	0.62 ± 0.02 ^(g)
1,9- and 4,9-Dimethylphenanthrene ^(c)	1.20 ± 0.03 ^(g)
1,8-Dimethylphenanthrene ^(c)	0.24 ± 0.01 ^(g)
1,2-Dimethylphenanthrene ^(c)	0.28 ± 0.01 ^(g)
8-Methylfluoranthene ^(c)	0.86 ± 0.02 ^(g)
7-Methylfluoranthene ^(c)	0.69 ± 0.02
1-Methylfluoranthene ^(c)	0.66 ± 0.02 ^(g)
3-Methylfluoranthene ^(c)	2.46 ± 0.07
2-Methylpyrene ^(c)	1.81 ± 0.04 ^(g)
4-Methylpyrene ^(c)	1.44 ± 0.03 ^(g)
1-Methylpyrene ^(c)	1.29 ± 0.03
Anthanthrene ^(h)	0.9 ± 0.1

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^(b) 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.

^(c) GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

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

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

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

^(g) 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 %.

^(h) LC-FL of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone.

Table 6. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1944

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the agreement among results from multiple methods was insufficient for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Chlorinated Pesticides	Mass Fractions in $\mu\text{g}/\text{kg}$ (dry-mass basis) ^(a,b)
α -HCH ^(c,d,e,f)	2.0 \pm 0.3
<i>trans</i> -Chlordane (γ -Chlordane) ^(c,d,e,f,g,h,i,j)	8 \pm 2
<i>cis</i> -Nonachlor ^(d,e,f,i,j)	3.7 \pm 0.7
2,4'-DDE ^(c,d,e,f,g,h,i,j)	19 \pm 3
2,4'-DDD ^(e,f,g,h,i,j)	38 \pm 8
4,4'-DDE ^(c,d,e,f,g,h,i,j)	86 \pm 12
4,4'-DDD ^(c,d,e,f,g,h,i,j)	108 \pm 16

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

^(b) 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.

^(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.

^(h) GC/MS (II) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with 50 % hexane/50 % acetone.

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

^(j) Results from 19 laboratories participating in an interlaboratory comparison exercise.

Table 7. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by Multiple Laboratories

NOTE: These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification, the agreement among results from multiple methods was insufficient for certification, or insufficient analyses have been performed at NIST to confirm the results of the outside laboratories.

Elements	Degrees of Freedom	Mass Fraction in percent (dry-mass basis) ^(a,b)
Silicon ^(c,d)	81	31 \pm 3
Mass Fraction in mg/kg (dry-mass basis) ^(a,b)		
Beryllium ^(c,h)	17	1.6 \pm 0.3
Copper ^(c,d,l)	101	380 \pm 40
Mercury ^(c,i)	18	3.4 \pm 0.5
Selenium ^(c,e,f)	24	1.4 \pm 0.2
Silver ^(c,d,e,g)	8	6.4 \pm 1.7
Thallium ^(c,f)	12	0.59 \pm 0.1
Tin ^(c,l)	22	42 \pm 6

^(a) The results are expressed as the reference value \pm the expanded uncertainty. 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 $U = ku_c$ where u_c is the combined standard uncertainty and k is the coverage factor, both calculated according to the ISO and NIST Guides [24]. 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 both possible method differences, within-method variation, and material inhomogeneity. The coverage factor, k , is the Student's t -value for a 95 % prediction interval with the

corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

- (b) Concentrations 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 NRCC using GFAAS.
- (e) Measured at NIST using INAA.
- (f) Measured at NRCC using ID-ICPMS.
- (g) Measured at IAEA using INAA.
- (h) Measured at NRCC using ICPAES.
- (i) Measured at NRCC using CVAAS.

Table 8. Reference Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification; therefore, unrecognized bias may exist for some analytes in this matrix.

Element	Effective Degrees of Freedom	Mass Fraction in percent (dry-mass basis) ^(a,b)		
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 in mg/kg (dry-mass basis) ^(a,b)		
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

(a) The results are expressed as the reference value ± the expanded uncertainty. 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, unrecognized bias may exist for some analytes in this matrix. 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 and NIST Guides [24]. 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 within-method variation and material inhomogeneity. The coverage factor, k , is the Student's t -value for a 95 % prediction interval with the corresponding degrees of freedom. Because of material inhomogeneity, the variability among the measurements of multiple samples can be expected to be greater than that due to measurement variability alone.

(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

Table 9. Reference Concentrations for Selected Dibenzo-*p*-dioxin and Dibenzofuran Congeners in SRM 1944

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Dibenzo- <i>p</i> -dioxin and Dibenzofuran Congeners	Mass Fraction in µg/kg (dry-mass basis) ^(a,b)		
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 ^(c)	0.039	±	0.015 ^(d)
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 ^(e)
1,2,3,7,8,9-Hexachlorodibenzofuran	0.019	±	0.018 ^(f)
1,2,3,4,6,7,8-Heptachlorodibenzofuran	1.0	±	0.1
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.040	±	0.006 ^(e)
Octachlorodibenzofuran	1.0	±	0.1
Total Toxic Equivalents (TEQ) ^(g)	0.25	±	0.01
Total Tetrachlorodibenzo- <i>p</i> -dioxins	0.25	±	0.05 ^(e)
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 ^(h)	8.7	±	0.9
Total Dibenzofurans ^(h)	5.0	±	0.5

^(a) Each reference value is the mean of the results from up to 14 laboratories participating in an interlaboratory exercise. The expanded uncertainty in the reference value is equal to $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO and NIST Guides [24] 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.

^(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.3 % moisture.

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

^(d) Degrees of freedom = 7 for this compound.

^(e) Degrees of freedom = 12 for this compound.

^(f) Degrees of freedom = 9 for this compound.

^(g) 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) [25]. With regard to 2,3,7,8-tetrachlorodibenzofuran, the results of the confirmation column were used when available to calculate the TEQ.

^(h) Total of tetra- through octachlorinated congeners.

Table 10. Reference Values for Particle-Size Characteristics for SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

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

^(a) The reference value is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], 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 %.

^(b) 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.

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

^(d) The mean diameter of the number distribution, calculated using the volume distribution weighted to small particles.

^(e) 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.

The following data show the percent of the volume that is smaller than the indicated size:

Percentile	Particle Diameter (μm) ^(a)
95	296 \pm 5
90	247 \pm 2
80	201 \pm 1
70	174 \pm 1
60	152 \pm 1
50 ^(b)	135 \pm 1
40	120 \pm 1
30	106 \pm 1
20	91 \pm 1
10	74 \pm 1

^(a) The reference value for particle diameter is the mean value of measurements from the analysis of subsamples from four bottles. Each uncertainty, computed according to the CIPM approach as described in the ISO and NIST Guides [24], 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 %.

^(b) Median diameter (50 % of the volume is less than 135 μm).

Table 11. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1944

NOTE: These results are provided as reference values because the results are method specific as defined by the procedures described in the Preparation and Analysis section. Although bias has not been evaluated for the procedures used, the reference values should be useful for comparison with results obtained using similar procedures.

Total Organic Carbon (TOC)	4.4 % ± 0.3 % mass fraction ^(a,b)
Extractable Mass ^(c)	1.15 % ± 0.04 % mass fraction ^(a,d)

^(a) Concentration is reported on a dry-mass basis; material as received contains approximately 1.3% moisture.

^(b) 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 and NIST Guides [24], 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 %.

^(c) Extractable mass as determined from Soxhlet extraction using DCM.

^(d) 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 and NIST Guides [24], 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 12. Information Values for Concentrations for Selected Inorganic Constituents in SRM 1944 as Determined by INAA

NOTE: These results are provided as information values only because insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed.

Elements	Mass Fractions in percent (dry-mass basis) ^(a)
Magnesium ^(b)	1.0
	Mass Fractions in mg/kg (dry-mass basis) ^(a)
Antimony ^(b,c)	5
Cerium ^(c)	65
Europium ^(c)	1.3
Gold ^(c)	0.10
Lanthanum ^(c)	39
Thorium ^(c)	13
Uranium ^(c)	3.1

^(a) Concentration is reported on a dry-mass basis; material as received contains approximately 1.3 % moisture.

^(b) Measured at NIST using INAA.

^(c) Measured at IAEA using INAA.

Table 13. Analytical Methods Used for the Analysis of SRM 1944 for Inorganic Constituents

Elements	Analytical Methods
Aluminum	FAAS, ICPAES, 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, ICPAES, ICPMS, ID-ICPMS, XRF
Europium	INAA
Gold	INAA
Iron	FAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Lanthanum	INAA
Lead	FAAS, GFAAS, ICPMS, ID-ICPMS, XRF
Magnesium	INAA
Manganese	FAAS, ICPAES, ICPMS, INAA, XRF
Mercury	CVAAS, ICPMS
Nickel	GFAAS, ICPAES, ICPMS, ID-ICPMS, INAA, XRF
Potassium	INAA
Rubidium	INAA
Scandium	INAA
Selenium	GFAAS, HGAAS, ICPMS, INAA
Silicon	FAAS, ICPAES, XRF
Silver	FAAS, GFAAS, ICPMS, INAA
Sodium	INAA
Thallium	GFAAS, ICPAES, ICPMS, ID-ICPMS
Thorium	INAA
Tin	GFAAS, ICPMS, ID-ICPMS
Titanium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICPAES, ICPMS, ID-ICPMS, XRF, INAA

Methods

CVAAS	Cold vapor atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICPAES	Inductively coupled plasma atomic 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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APPENDIX A

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.

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M.J. Armbruster, Battelle Columbus Laboratories, Columbus, OH
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
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J. Macaulay, Research and Productivity Council, Fredericton, New Brunswick, Canada
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C. Tashiro, Wellington Laboratories, Guelph, Ontario, Canada
T.O. Tiernan, Wright State University, Dayton, OH

APPENDIX B

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

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H. Mawhinney, Animal Research Institute, Queensland Department of Primary Industries, Queensland, Australia
E. Crecelius, Battelle Pacific Northwest, Sequim, WA
M. Stephenson, California Department of Fish and Game, Moss Landing, CA
B. Presley, Department of Oceanography, Texas A&M University, College Station, TX
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