National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1649a

Urban Dust

Standard Reference Material (SRM) 1649a is an atmospheric particulate material collected in an urban area, and is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, and chlorinated pesticides in atmospheric particulate material and similar matrices. Reference values are also provided for selected inorganic constituents, total organic carbon, total extractable material, mutagenic activity, particle size characteristics, and carbon composition. All of the constituents for which certified and reference values are provided in SRM 1649a are naturally present in the particulate material. SRM 1649a is the same particulate material that was issued previously in 1982 as SRM 1649 [1]; this material has been rebottled and reanalyzed to provide updated certified values as well as certified and reference values for additional constituents. A unit of SRM 1649a consists of a bottle containing 5 g of particulate material.

Certified Concentration Values: Certified values for the concentrations, expressed as mass fractions, for 22 PAHs, 35 PCB congeners (some in combination), and 8 chlorinated pesticides are provided in Tables 1-3. 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 from two or more chemically independent analytical techniques performed at NIST.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 22 additional PAHs in Tables 4 and 5 and for one additional chlorinated pesticide in Table 6. Reference values for 32 selected inorganic constituents are provided in Table 7. Reference values for mutagenic activity are provided in Table 8. Reference values for particle size characteristics are provided in Table 9. Reference values are provided for total organic carbon and total extractable material in Table 10 and for the carbon composition 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 a note in Tables 4-11.

Expiration of Certification: The certification of this SRM lot is valid until **30 June 2007**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or 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. Return of the attached registration card will facilitate notification.

The support aspects involved in the preparation, recertification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by B.S. MacDonald and T.E. Gills.

Gaithersburg, MD 20899
Certificate Issue Date: 19 November 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

Certain commercial equipment, instruments, or materials are identified in this report to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

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

Analytical measurements for the certification of SRM 1649a were performed by B.A. Benner, Jr., A. Deissler, R.R. Greenberg, M.J. Hays, B.J. Porter, D.L. Poster, L.C. Sander, M.M. Schantz, and R.L. Watters, Jr. of the NIST Analytical Chemistry Division. Carbon composition measurements were performed by D.B. Klinedinst, G. Klouda, J.L. Marolf, and A.E. Sheffield of the NIST Surface and Microanalysis Science Division, R.A. Cary at Sunset Laboratory Inc. (Forest Grove, OR), D.J. Donahue and A.J.T. Jull at the University of Arizona, J.S. Vogel at Lawrence Livermore National Laboratory, and C. Masiello at the University of California, Irvine, CA. Measurements for percent total organic carbon measurements were provided by two 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).

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.B. Schiller and M.G. Vangel of the NIST Statistical Engineering Division.

The collection and preparation of the material for SRM 1649a were supported in part by the U.S. Environmental Protection Agency.

NOTICE AND WARNING TO USERS

Storage: SRM 1649a is provided in amber glass bottles and should be stored away from direct sunlight at room temperature or below.

Handling: This material is naturally occurring urban atmospheric particulate matter 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 1649a are generally reported on a dry mass basis. The SRM, as received, contains approximately 1.2 % moisture. A separate subsample of the SRM should be removed from the bottle at the time of analysis and dried to determine the concentration based on dry mass.

PREPARATION AND ANALYSIS

Sample Collection and Preparation: This SRM was prepared from atmospheric particulate material collected in the Washington DC area in 1976-1977 using a baghouse specially designed for the purpose. The particulate material was collected over a period in excess of 12 months, and therefore represents a time-integrated sample. While the sample is not intended to be representative of the area in which it was collected, it should generally typify atmospheric particulate matter obtained from an urban area. The particulate material was removed from the baghouse filter bags by a specially designed vacuum cleaner and combined into a single lot. This lot was passed through a 125 μm (120 mesh) sieve to remove bag fibers and other extraneous materials. The sieved material was then thoroughly mixed in a V-blender and bottled.

Conversion to Dry Mass Basis: The results for the constituents in SRM 1649a are reported on a dry weight basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1649a was determined by measuring the weight 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 1649a at the time of the certification analyses was 1.23 % ± 0.07 % (95 % confidence level).

Polycyclic Aromatic Hydrocarbons: The general approach used for the determination of PAHs in SRM 1649a was similar to that reported for the recent certification of several environmental matrix SRMs [2-5]. This approach consisted of Soxhlet extraction using dichloromethane or a hexane/acetone mixture followed by analysis of the extract 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 a 5 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase, and (4) GC/MS analysis of the PAH fraction on a smectic liquid crystalline stationary phase. These procedures are described in

detail for SRM 1649a in Wise et al. [6] and are described briefly below. Additional results for selected PAHs were obtained by pressurized fluid extraction (PFE) followed by GC/MS analysis on a 50 % (mole fraction) phenyl-substituted methylpolysiloxane stationary phase.

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. Subsamples of 3 g from six bottles were Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone (volume fractions). The extracts were concentrated and then processed through an aminopropylsilane solid phase extraction (SPE) cartridge to obtain the total PAH fraction. Approximately one third of this fraction was analyzed as the total PAH fraction; the second portion of the extract was then fractionated on a semi-preparative aminopropylsilane column to isolate isomeric PAH fractions as described previously [7-10]. The total PAH fraction and the isomeric PAH fractions were analyzed using both 3 μm and 5 μm particle size polymeric octadecylsilane (C_{18}) columns (4.6 mm i.d. × 15 cm, 3 μm particle size, ChromSpher PAH, Chrompack, Middelburg, The Netherlands and 4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [2,8,9].

Five sets of GC/MS results, designated as GC/MS (I), GC/MS (II), GC/MS (III), GC/MS (IV), 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 10 bottles were Soxhlet extracted for 20 h with dichloromethane. The concentrated extract was passed through an aminopropylsilane SPE cartridge and eluted with 2 % dichloromethane in hexane. The PAH fraction was then isolated from the extract using normal-phase LC [7-10] on a semi-preparative aminopropylsilane column. The PAH fraction was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses consisted of subsamples from six bottles analyzed as a second sample set using the same preparation and analysis procedures as described for GC/MS (I). Two additional sets of GC/MS results for a limited number of PAHs, designated as GC/MS (III) and GC/MS (IV), were obtained using PFE followed by GC/MS. For the GC/MS (III) analyses subsamples of 0.4 g to 1 g from six bottles were extracted with dichloromethane using PFE, as described by Schantz et al. [11], the extracts were processed as described above for GC/MS (I), followed by GC/MS analysis on 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 (IV) analyses two subsamples of 1 g each were extracted with each of three different solvents (dichloromethane, acetonitrile, and 50 % hexane/50 % acetone mixture) using PFE, as described by Schantz et al. [11], the extracts were processed and analyzed by GC/MS on the 5 % phenyl-substituted methylpolysiloxane stationary phase described above for GC/MS (I). The GC/MS (Sm) results were obtained by analyzing selected sample extracts from the GC/MS (I) set on a 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). The liquid crystalline phase provides significantly different selectivity for the separation of PAH isomers when compared with the 5 % phenyl-substituted methylpolysiloxane phase [2]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the particulate matter prior to solvent extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1649a was assessed by analyzing duplicate samples of 1 g from ten randomly selected bottles. Samples were extracted, processed, and analyzed as described above for the GC/MS (I). No statistically significant differences between bottles were observed for the PAHs at the 1 g sample size. Analyses of subsamples of 1 mg to 400 mg show no significant differences in the PAH concentrations.

PCBs and Chlorinated Pesticides: SRM 1649a was analyzed for selected PCB congeners and chlorinated pesticides using gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity and using GC/MS. This same approach has been used previously for the certification of PCBs and chlorinated pesticides in environmental matrix SRMs [3,5,12,13]. For the GC-ECD analyses, subsamples of approximately 1 g from each of six bottles were Soxhlet extracted for 18 h using dichloromethane. 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 x 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 dimethylpolysiloxane phase containing 50 % (mole fraction) C-18 dimethylpolysiloxane (0.1 μm film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands).

A second set of samples was also analyzed by GC-ECD; however, these samples were extracted using PFE with dichloromethane as described by Schantz et al. [11]. Subsamples of 1 g to 2 g were extracted using 15 mL of dichloromethane. The extract was processed and analyzed by GC-ECD on the 5 % phenyl-substituted methylpolysiloxane phase as described above.

For the GC/MS analyses, subsamples of 0.5 g to 1.5 g each from six randomly selected bottles were mixed with 50 g of precleaned sodium sulfate and Soxhlet extracted for 18 h using 50 % hexane/50 % acetone (volume fraction). The extracts were concentrated to 1 mL and then placed on a precleaned silica SPE column and eluted with 15 mL of 10 % dichloromethane in hexane. The concentrated eluent was analyzed by GC/MS on a 5 % phenyl-substituted methylpolysiloxane phase as described above. For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the air particulate extract (PCB 103 and PCB 198 [14,15]), and 4,4'-DDT-d_8 were added to the air particulate material prior to extraction for use as internal standards for quantification purposes. The analyses of SRM 1649a for the determination of PCBs and pesticides is described in detail elsewhere [16].

Inorganic Constituents: The majority of the inorganic constituents were determined using instrumental neutron activation analysis (INAA). For INAA duplicate 100 mg subsamples from six bottles of SRM 1649a were analyzed. Selected trace elements (Pb, Mg, Cu, Mn, Ni, and V) were determined by inductively coupled plasma atomic emission spectrometry (ICP-AES). For the ICP-AES analyses duplicate 250 mg subsamples from four bottles of SRM 1649a were analyzed. For the determination of Cl and S, 250 mg subsamples from six bottles of SRM 1649a were analyzed using high pressure oxygen bomb combustion followed by ion chromatography.

Mutagenic Activity: The reference values for the mutagenic activity of a dichloromethane extract of SRM 1649a were determined as part of an international collaborative study in 1989 sponsored by the International Programme on Chemical Safety (IPCS). The IPCS is jointly sponsored by the World Health Organization (WHO), the United Nations Environmental Programme (UNEP), and the International Labor Organization (ILO). The program was initiated, supported, and technically coordinated by the U.S. Environmental Protection Agency's Office of Health Research. Twenty laboratories from North America, Europe, and Japan participated in the study for which a complete summary is available [17,18]. As part of the protocol, each laboratory used dichloromethane to extract the organic material from SRM 1649a. Half of the laboratories used Soxhlet extraction and the other half used ultrasonication extraction procedures. The extracted material was analyzed using the Salmonella/mammalian microsomal plate-incorporation assay using strains TA98 and TA100 [19]. The mean dichloromethane extractable mass determined in the IPCS collaborative study was 5.0 % ± 0.4 %, and the extract was found to be mutagenic in both strains with and without activation in all 20 laboratories.

Two types of suggested Bioassay Reference Values are provided in Table 8. The first value is the best estimate of the mutagenic activity, from the data available, for a dichloromethane extract of SRM 1649a using the protocol specified for the IPCS collaborative study. For the reference values to apply, the sample should be Soxhlet or ultrasonically extracted with dichloromethane. The dichloromethane extract should be evaporated to near dryness and solvent exchanged to dimethylsulfoxide. The bioassay procedure should follow the *Salmonella typhimurium* plate incorporation protocol as described by Marion and Ames [19] and adhere to the guidelines published by Claxton et al. [20]. Minimal media plates should be made of Difco agar and should contain 30 mL ± 1 mL of base layer agar. The exogenous activation system (S9) should be an Aroclor-1254 induced rat liver homogenate as described by Marion and Ames [19]. Duplicate plates should be used for each of 3-5 dose levels.

The uncertainty in the mutagenic activity, expressed as 95 % confidence limits about the mean potency value, takes into account both between and within laboratory sources of variation. While these confidence limits represent the uncertainty for the best estimate of the mutagenic activity of SRM 1649a, they do not reflect the variation in the values reported by individual participating laboratories. They should also not be taken to represent the range of mutagenic activity values from other laboratories using the protocol of Marion and Ames [19] with some additional constraints [21]. Prediction intervals [22] are provided to characterize differences in the mutagenic activity reported by the 20 laboratories that participated in the IPCS interlaboratory study and to establish a target range for other laboratories that analyze SRM 1649a using the modified Marion and Ames protocol. Additionally, for the

investigator values to be assessed using the tolerance limits given, data should be treated using the same or very similar statistical methods as those used in this study [23,24][a].

An "80 % Prediction Interval" is a range within which 80 % of the mutagenic activity values reported in the interlaboratory study are expected to reside. These limits may be used by all laboratories using the IPCS Salmonella bioassay protocol to determine if their findings are consistent with those reported by the 20 laboratories that participated in the IPCS collaborative study. Although these laboratories may not be representative of all laboratories that conduct the Salmonella bioassay, the tolerance limits given do provide a range of values that all laboratories following the IPCS protocol should strive to obtain. The first set of tolerance limits given are for laboratories that use the same number of replicate extractions and bioassays as were performed in the IPCS collaborative study. The second set of tolerance limits, which are slightly wider, apply to the case where only a single extraction and bioassay is performed.

Particle Size Information: Dry particle-size distribution measurements for SRM 1649a 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 1649a. 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 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 Extractable Mass: Three laboratories provided results for the Total Organic Carbon (TOC) measurements using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6N HCl 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. For the determination of extractable mass, six samples of approximately 15 g of SRM 1649a were Soxhlet extracted with 250 mL of 50 % hexane/50 % acetone (volume/volume) for 20 h. The extraction thimbles were allowed to air dry. After reaching constant mass, the difference in mass before and after extraction was determined.

Carbon Composition Information: Carbon composition values were determined at NIST, Sunset Laboratory Inc. (SLI), University of California (UCI) (Irvine, CA), U.S. Environmental Protection Agency (EPA) National Exposure Laboratory (Research Triangle Park, NC), University of Arizona (UA) (Tucson, AZ), Lawrence Livermore Laboratory (LLNL) (Berkeley, CA), and the Woods Hole Oceanographic Institution (WHOI) (Woods Hole, MA) using the procedures described below.

Combustion-Manometry (NIST) [26]: Samples were combusted to CO_2 in a quartz furnace filled with 101 KPa O_2. Down stream of the combustion furnace is a series of three furnaces: (1) Pt gauze at 900 °C, (2) CuO at 800 °C, and (3) Ag wool at 400 °C to assure complete combustion and to purify the CO_2 of sulfur and halogen containing impurities. The sample gas stream is then reduced to less than 13 kPa to prevent the condensation of liquid O_2 by controlling the gas flow through the system using a throttle valve and a vacuum pump. Before the vacuum pump, the sample CO_2 is cryogenically trapped at liquid N_2 temperature (-196 °C) in a series of spiral glass traps. The resulting CO_2 is cryogenically separated from other gaseous combustion products by distillation from -78 °C and quantified using manometry in a calibrated volume. Low-level ^{14}C decay counting was performed on the CO_2 using a miniature gas proportional counter at NIST.

H_2PO_4-Combustion-Manometry (UCI) [27]: A subsample of SRM 1649a, Ag foil (prefired at 550 °C), and CuO wire (prefired at 850 °C) were added to a quartz tube. Approximately 5 mL of 3 % H_2PO_4 was added to the tube to remove any inorganic carbon. The quartz tube was then attached to a vacuum line, evacuated to a pressure of less than 5 Pa, sealed, and combusted to CO_2 at 850 °C for 4 h. The CO_2 was reduced to graphite over Co catalyst at 850 °C in the presence of H_2. Accelerator mass spectrometry ^{14}C measurements were performed at LLNL [28].

a A computer program developed by the U.S. Environmental Protection Agency to run under MS-DOS entitled *GeneTox Manager* contains the statistical analysis software developed by several research groups for the Salmonella assay including the program described by Krewski et al. [23,24]. The *GeneTox Manager* has been described [25] and is available from the U.S. EPA through a written request to: Dr. Larry D. Claxton, Environmental Carcinogenesis Division, MD-68, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

Thermal-Optical-FID Method (SLI) [29]: In a completely oxygen-free helium atmosphere, the sample (~1 mg) was heated in four increasing steps (from 25 °C to 700 °C) to remove all organic carbon on the filter. Organic compounds that are pyrolytically converted to elemental carbon were continuously monitored by measuring the transmission of He-Ne laser light through the filter. As organic compounds are volatilized, they are immediately oxidized to CO_2 using a plug of MnO_2 at 700 °C, reduced to methane over Ni on firebrick in the presence of H_2, and measured using a flame ionization detector (FID). After cooling the sample to 525 °C, a 2 % O_2/He mixture was introduced and the temperature increased to 850 °C. Based on the FID response and laser-transmission data, the amounts of organic, elemental, and pyrolytic carbon are then calculated for the sample. The chemical fractions, quantified by this method are defined as: (l) organic carbon, that which is volatilized-oxidized and pyrolyzed in He during four increasing temperature steps from 25 °C to 700 °C, (2) elemental carbon, that which is oxidized in O_2/He during the heating from 525 °C to 850 °C minus the pyrolyzed (organic) carbon oxidized during this step, (3) pyrolyzed carbon, the organic carbon pyrolyzed during the four steps of heating in He and subsequently oxidized during the 525 °C to 850 °C heating in O_2 /He, (4) total carbon, the sum of the organic carbon (including pyrolyzed carbon) and elemental carbon.

Combustion-GC-TCD (NIST) [32]: Samples (0.3 mg to 9 mg) were weighed into Al boats, combusted to CO_2 at 900 °C in an atmosphere of O_2, purified by gas chromatography (GC), quantified with GC using a thermal conductivity detector (TCD), and the CO_2 trapped at -196 °C from a single measurement. Sample CO_2 was then reduced to graphite [30] for ^{14}C AMS at UA [31].

Combustion-NDIR (NIST) [33]: The weighed sample was placed in a ceramic crucible which was then purged with O_2 while inductively heating the crucible. The CO and CO_2 produced were measured using a non-dispersive infrared (NDIR) detector.

H_2O-Combustion-Manometry (NIST) [32]: Subsamples of SRM 1649a were placed on prefired quartz filters. Three 200 mL portions of prefiltered distilled water were passed through the sample and filter. The filters were dried at 60 °C for 2 h before closed tube combustion with CuO. The resulting CO_2 was distilled and quantified by manometry and then radiocarbon analysis was performed at UA [31].

Acic/Base-Combustion-Manometry (NIST) [32]: The acid/base method of removing carbonate and organic carbon involves a series of subsequent solutions in the following order, twice with 1M NaOH, once with 70 % HNO_3, three times with 1M NaOH, once with 1 % HCl, and twice with distilled water as described by Kuhlbusch [34]. Lowering the pH in the last steps ensures that carbonates are not reformed. The residue was then sealed in a quartz tube containing 380 mbar of ultra-high purity O_2 and heated for 2 h at 340 °C. Finally, the thermally evolved gases were removed, CuO and Ag were added to the same tube, the tube was evacuated, and the sample was combusted at 950 °C [35]. The resulting CO_2 was cryogenically separated from other gaseous combustion products at -78 °C and quantified using manometry as described above.

HNO_3-Combustion-NDIR (NIST) [33]: The sample was weighed into a beaker to which 70 % HNO_3 per g was added. After boiling for 20 min, 125 mL of 6N HNO_3 per g was added to the mixture and it was allowed to stand overnight [36]. The solution was then transferred to test tubes for centrifuging at 3200 rpm for 30 min. The supernate was decanted, and the original beakers rinsed with distilled water. The water was then transferred to the tubes and centrifuged. This process was repeated once or twice more, then the final supernates were decanted and the residue dried overnight at 100 °C. The dried residues were weighted and their carbon contents determined using a combustion/NDIR analyzer.

H_3PO_4-Oxidation-Manometry (NIST) [32]: A weighed sample (280 mg) and 2 mL of 100 % phosphoric acid were placed in opposite branches of an inverted Y-shaped tube. The stem of the Y-tube was adapted to a vacuum manifold. The system was evacuated, and the acid was frozen by applying dry ice/isopropanol slurry. Vacuum was applied to the frozen acid for a short time, then the acid was warmed to room temperature. The system was closed to vacuum, then the acid was poured onto the sample and the mixture heated with a beaker of boiling water for 2 h - 3 h. A sample bulb immersed in liquid N_2 was used to collect the evolved CO_2, which was then measured using manometry as described above.

Soxhlet Extraction (EPA) [32, 33]: Samples were Soxhlet extracted for 24 h with dichloromethane. The solvent was removed by rotary evaporation and the extract was reconstituted to 10 mL with dichloromethane. Gravimetric determinations were then made by transferring a 200 µL aliquot of the extract solution to a balance, evaporating the

solvent, and weighing the residue to constant weight. A 1 mL aliquot of the extract solution was evaporated to dryness in a quartz tube. CuO and Ag wire were then added to the tube. The tube was attached to a vacuum line, evacuated to 5 Pa, sealed, and the contents combusted to CO_2 at 900 °C. The sample CO_2 was then reduced to graphite [37] for ^{14}C AMS measurements at UA [31].

Soxhlet Extraction/LC (NIST) [32]: Samples were Soxhlet extracted for 24 h with dichloromethane. The extract was concentrated to a small volume under a stream of N_2. The concentrated extract was placed on a silica SPE cartridge and eluted with 10 % dichloromethane in pentane. The PAH fraction was isolated using normal-phase liquid chromatography on an aminopropylsilane column as described above. The PAH fraction was concentrated and a 1 mL aliquot transferred to a quartz tube and evaporated to dryness. CuO was added to the tube which was then attached to a vacuum line, evacuated, sealed, and the contents combusted to CO_2. Low-level ^{14}C decay counting was performed on the sample CO_2 using a miniature gas proportional counter at NIST.

Soxhlet Extraction/GC (NIST/WHOI) [38]: Separation and collection of the individual PAHs were accomplished using an automated preparative capillary GC system described by Eglinton et al. [38].

Table 1. Certified Concentrations for Selected PAHs in SRM 1649a[a,b]

	mass fractions, in mg/kg
Phenanthrene[c,d,e,f]	4.14 ± 0.37
Anthracene[c,d,e,f]	0.432 ± 0.082
Fluoranthene[c,d,e,f]	6.45 ± 0.18
Pyrene[c,d,e,f]	5.29 ± 0.25
Benz[*a*]anthracene[c,d,h]	2.21 ± 0.073
Chrysene[c,h]	3.049 ± 0.060[i]
Triphenylene[c,h]	1.357 ± 0.054
Benzo[*b*]fluoranthene[c,f,g]	6.45 ± 0.64
Benzo[*k*]fluoranthene[c,d,e,f,g]	1.913 ± 0.031
Benzo[*a*]fluoranthene[c,d,e]	0.409 ± 0.035
Benzo[*e*]pyrene[c,d,e]	3.09 ± 0.19
Benzo[*a*]pyrene[c,d,e,f]	2.509 ± 0.087
Perylene[c,d,e,f]	0.646 ± 0.075
Anthanthrene[c,d,e,h]	0.450 ± 0.067
Benzo[*ghi*]perylene[c,d,g,h,j]	4.01 ± 0.91
Indeno[1,2,3-*cd*]pyrene[c,d,g,h,j]	3.18 ± 0.72
Dibenz[*a,j*]anthracene[c,d,g,h]	0.310 ± 0.034
Dibenz[*a,c*]anthracene[c,g,h]	0.200 ± 0.025
Dibenz[*a,h*]anthracene[c,g,h]	0.288 ± 0.023
Pentaphene[c,d,g,h]	0.151 ± 0.035
Benzo[*b*]chrysene[c,d,e,g,h]	0.315 ± 0.013
Picene[c,d,e,g,h]	0.426 ± 0.022

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.

[b] Each certified value is the equally-weighted mean of the means from two or more independent analytical methods. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [39], 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 for the certified value within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[c] GC/MS (I) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

[d] GC/MS (II) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

[e] GC/MS (Sm) analysis using a smectic liquid crystalline phase after Soxhlet extraction with dichloromethane.

[f] LC-FL analysis of total PAH fraction after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[g] GC/MS (III) analysis on 50 % phenyl-substituted methylpolysiloxane after PFE with dichloromethane.

[h] LC-FL analysis of isomeric PAH fractions after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[i] 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 %.

[j] GC/MS (IV) analysis on 5 % phenyl-substituted methylpolysiloxane phase after PFE with three different solvents (dichloromethane, acetonitrile, and 50 % hexane/50 % acetone mixture).

Table 2. Certified Concentrations for Selected PCB Congeners[a] in SRM 1649a[b,c]

			mass fractions, in µg/kg		
PCB	8	(2,4'-Dichlorobiphenyl)	12.28	±	0.29
PCB	18	(2,2',5-Trichlorobiphenyl)	20.44	±	0.84
PCB	28	(2,4,4'-Trichlorobiphenyl)	18.5	±	1.2
PCB	31	(2,4',5-Trichlorobiphenyl)	17.3	±	1.4
PCB	44	(2,2'3,5'-Tetrachlorobiphenyl)	15.4	±	1.6
PCB	49	(2,2'4,5'-Tetrachlorobiphenyl)	12.2	±	1.5
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)	24.65	±	0.97
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)[d]	65	±	12
	95	(2,2',3,5',6-Pentachlorobiphenyl)[d]			
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)	10.65	±	0.62
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)[e]	51.6	±	4.2
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl)	9.58	±	0.69
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)	52.9	±	1.0
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)	8.63	±	0.80
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl)	26.6	±	1.6
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)	25.7	±	1.5
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	6.35	±	0.69
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	69.7	±	7.5
	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)	75.7	±	1.3
PCB	151	(2,2',3,5,5',6-Hexachlorobiphenyl)	34.3	±	3.9
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	82.5	±	8.0
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)	16.25	±	0.77
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	30.8	±	2.2
	190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)			
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	78.7	±	8.2
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)	20.34	±	0.95
PCB	187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	40.1	±	2.5
	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)	28.9	±	3.6
PCB	195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl)	9.63	±	0.37
PCB	206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	20.6	±	4.6
PCB	209	Decachlorobiphenyl	8.04	±	0.77

[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 PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.

[c] Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [40]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [39], is an expanded uncertainty at the 95 % level of confidence, which includes random 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] Concentration for PCB 95 and PCB 66 together was determined using GC-ECD on the 5 % phenyl-substituted methylpolysiloxane phase.

[e] Concentration for PCB 95 was determined using GC-ECD on the 50 % C-18 dimethylpolysiloxane phase and GC/MS on the 5 % phenyl-substituted methylpolysiloxane phase.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1649a[a,b]

	mass fractions, in µg/kg		
Hexachlorobenzene	16.3	±	1.8
trans-Chlordane (γ-Chlordane)	40.3	±	2.8
cis-Chlordane (α-Chlordane)	34.88	±	0.42
trans-Nonachlor	27.6	±	1.6
2,4'-DDE	5.79	±	0.85
4,4'-DDE	40.4	±	1.7
4,4'-DDD	34.01	±	0.48
4,4'-DDT	212	±	15

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.
[b] Each certified value is a mean of the means from two or more analytical methods, weighted as described in Paule and Mandel [40]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [39], is an expanded uncertainty at the 95 % level of confidence, which includes random 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 %.

Table 4. Reference Concentrations for Selected PAHs in SRM 1649a as Determined by GC/MS[a,b]

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

	mass fractions, in mg/kg		
Fluorene[c]	0.23	±	0.05
Dibenzothiophene[d]	0.18	±	0.01
1-Methylphenanthrene[c,d,e]	0.37	±	0.04
2-Methylphenanthrene[c,d,e]	0.73	±	0.12
3-Methylphenanthrene[c,d,e]	0.50	±	0.05
4-Methylphenanthrene and 9-Methylphenanthrene[c,d,e]	0.34	±	0.01
4H-Cyclopenta[*def*]phenanthrene[c,d,e]	0.32	±	0.06
Benzo[*c*]phenanthrene[d,e]	0.46	±	0.03
Benzo[*ghi*]fluoranthene[d]	0.88	±	0.02
Benzo[*j*]fluoranthene[d,f]	1.5	±	0.4
Indeno[1,2,3-*cd*]fluoranthene[c]	0.23	±	0.01
Benzo[*c*]chrysene[d]	0.080	±	0.004

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % 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) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.
[d] GC/MS (Sm) analysis using a smectic liquid crystalline phase after Soxhlet extraction with dichloromethane.
[e] GC/MS (II) analysis on 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.
[f] GC/MS (III) analysis on 50 % phenyl-substituted methylpolysiloxane after PFE with dichloromethane.

Table 5. Reference Concentrations for Selected PAHs in SRM 1649a as Determined by LC[a,b,c]

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 procedure used, the reference values should be useful for comparison with results obtained using similar procedures.

	mass fractions, in µg/kg
Dibenzo[*a*,*e*]pyrene	630 ± 80
Dibenzo[*a*,*h*]pyrene	53 ± 2
Dibenzo[*a*,*i*]pyrene	130 ± 10
Dibenzo[*b*,*k*]fluoranthene	800 ± 100
Naphtho[2,3-*a*]pyrene	57 ± 5
Naphtho[2,3-*e*]pyrene	240 ± 30
Naphtho[2,3-*b*]fluoranthene	230 ± 20
Naphtho1,2-*k*]fluoranthene	550 ± 60
Naphtho[2,3-*k*]fluoranthene	57 ± 3

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.
[b] Expanded uncertainties are sample standard deviations of the mean concentrations.
[c] Concentrations reported by Wise et al. [10] were determined using LC-FL analysis of isomeric PAH fractions; duplicate analyses of two sample extracts.

Table 6. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1649a[a]

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.

	mass fraction, in µg/kg
Heptachlor[b,c]	18.9 ± 0.5[d]

[a] Concentrations reported on dry mass basis; material as received contains approximately 1.2 % moisture.
[b] GC-ECD analysis on the 50 % C-18 dimethylpolysiloxane phase.
[c] GC-ECD analysis on the 5 % phenyl-substituted methylpolysiloxane phase.
[d] The reference value is the equally-weighted mean of the means from two or more analytical methods. 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.

Table 7. Reference Concentrations for Selected Inorganic Constituents in SRM 1649a

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique or only a limited number of analyses were performed; therefore, unrecognized bias may exist for some analytes in this matrix.

Element	mass fractions, in %[a,b]
Bromine[c]	0.119 ± 0.001
Chlorine[d]	0.28 ± 0.01
Iron[c,e]	2.98 ± 0.07
Lead[e]	1.24 ± 0.04
Magnesium[e]	0.92 ± 0.03
Sulfur[d]	3.27 ± 0.09
Zinc[c,e]	0.168 ± 0.004
	mass fractions, in mg/kg[a]
Antimony[c]	29.9 ± 0.7
Arsenic[c]	67 ± 2
Barium[c]	569 ± 21
Cadmium[c,e]	(22)[f]
Cerium[c]	52 ± 4
Cesium[c]	2.84 ± 0.07
Chromium[c]	211 ± 6
Cobalt[c]	16.4 ± 0.4
Copper[e]	223 ± 7
Europium[c]	0.87 ± 0.07
Hafnium[c]	4.4 ± 0.1
Lanthanum[c]	33 ± 3
Manganese[e]	237 ± 8
Molybdenum[c]	13.5 ± 0.9
Nickel[e]	166 ± 7
Rubidium[c]	48 ± 3
Samarium[c]	4.7 ± 0.4
Scandium[c]	8.7 ± 0.2
Selenium[c]	25.6 ± 0.7
Silver[c]	3.5 ± 0.2
Thorium[c]	6.6 ± 0.2
Tin[c]	56 ± 13
Tungsten[c]	3.8 ± 0.3
Uranium[c]	2.65 ± 0.08
Vanadium[e]	345 ± 13

[a] Concentration is reported on an as received basis; material as received contains approximately 1.2 % moisture.

[b] Each reference value is the mean of means of measurements made by INAA on duplicatesubsamples from six bottles or measurements made by ICP on duplicate subsamples from four bottles, or the combination of results from both analytical techniques. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [39], is an expanded uncertainty at the 95 % level of confidence, which includes random uncertainty, as well as a Type-B uncertainty of 1 % to 3 %. The expanded uncertainty defines a range of values with which the true value is believed to lie, at a confidence of approximately 95 %.

[c] Determined using instrumental neutron activation analysis (INAA).

[d] Determined using high pressure oxygen bomb combustion and ion chromatography.

[e] Determined using inductively coupled plasma atomic emission spectrometry (ICP-AES).

[f] Cadmium value is the mean of the results from INAA (18.3 ± 1.1) mg/kg and ICP-AES (26.5 ± 1.0) mg/kg and is provided as an information value only because of the disagreement of the results from the two analytical techniques. An information value is considered to be a value that will be of interest and use to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value.

Table 8. Reference Values for Ames Bioassay Mutagenic Activity of SRM 1649a[a]

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.

			80 % Prediction Intervals	
Strain/Activation	Mutagenic Activity[b]	95 % Confidence Limits[c]	Multiple Extraction/ Bioassay[d]	Single Extraction/ Bioassay[e]
TA100, +S9	102 rev/mg	66 - 158	30 - 351	29 - 365
TA100, -S9	103 rev/mg	73 - 146	39 - 275	36 - 295
TA98, +S9	214 rev/mg	153 - 299	83 - 555	80 - 570
TA98, -S9	237 rev/mg	186 - 301	119 - 471	115 - 488

[a] Results summarized from Claxton et al. [21]. Reference values refer to the mutagenic activity of the dichloromethane extract of SRM 1649a per unit mass of the particulate material extracted. Doses for the IPCS collaborative study were based on the following mg equivalents of SRM 1649a particles: TA100, +/-S9 (0.25, 0.5, 1.0, 1.5, 2.0); TA98, +S9 (1.0, 2.0, 4.0, 6.0, 8.0); and TA98, -S9 (1.25, 2.5, 5.0, 7.5, 10.0). Total extractable mass for SRM 1649a with dichloromethane from the IPCS collaborative study was 5.0 %± 0.4 % (mass fraction) as compared with 4.60 % ± 0.36 % with hexane/acetone in Table 10.

[b] Geometric mean of all replicate mutagenicity potency values reported by participating laboratories after deleting outlying observations. Results reported as revertants per mg of SRM 1649a.

[c] Calculated on a logarithmic scale taking into account both inter- and intra-laboratory variation, excluding outliers, and then re-expressed in the original scale by taking antilogs.

[d] Prediction intervals for mutagenic activity determined in a single laboratory using the same number of replicate extracts/bioassays as in the IPCS collaborative study.

[e] Prediction intervals for mutagenic activity determined in a single laboratory using only one replicate extraction/bioassay.

Table 9. Reference Values for Particle Size Characteristics for SRM 1649a

NOTE: These results are provided as reference values because the results are method specific as defined by the procedure described in the Preparation and Analysis section. Although bias has not been evaluated for the procedure 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]	34.6 ± 0.4
Mean diameter (area distribution, μm)[c]	12.9 ± 0.3
Mean diameter (number distribution, μm)[d]	1.50 ± 0.09
Surface Area (m^2/cm^3)[e]	0.47 ± 0.01

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

Percentile	Particle Diameter (μm)[a]
95	100 ± 3
90	73 ± 1
80	49.9 ± 0.2
70	38.3 ± 0.1
60	30.5 ± 0.2
50[f]	24.4 ± 0.2
40	19.3 ± 0.2
30	15.2 ± 0.2
20	11.3 ± 0.2
10	6.7 ± 0.2

[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 Guide [39], 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 approximately 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.

[f] Median diameter (50 % of the volume is less than 24.4 μm).

Table 10. Reference Values for Total Organic Carbon and Percent Extractable Mass in SRM 1649a

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.

	mass fractions, in %
Total Organic Carbon (TOC)	16 ± 5[a,b]
Extractable Mass[c]	4.6 ± 0.4[a,d]

[a] Concentration is reported on an as received basis; material as received contains approximately 1.2 % 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 Guide [39], 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 approximately 95 %.

[c] Extractable mass value was determined from Soxhlet extraction using 50 % hexane/50 % acetone (volume/volume).

[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 Guide [39], 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 approximately 95 %.

Table 11. Reference Values for Carbon Composition of SRM 1649a

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.

Chemical Fraction	Analytical Method	Concentration (mass fractions, g/g)[a]	Modern Carbon[b] (mass fractions, in %, pMC)
Total Carbon	Combustion-Manometry		61 ± 4[c,d] (1) [41]
	H_2PO_4-Combustion-Manometry		50.2 ± 0.3[c,f] (1) [27]
	Thermal-Optical-FID	0.175 ± 0.031 (3) [29]	
	Combustion-GC-TCD	0.179 ± 0.004 (39) [32]	51.6 ± 0.3[c,g,h] (1) [32]
	Combustion-NDIR	0.177 ± 0.008 (15) [33]	
Insoluble Carbon	H_2O-Combustion-Manometry	0.152 ± 0.002 (2) [32]	55.7 ± 1.4[c,j] (2) [32]
Organic Carbon[i]	Thermal-Optical-FID	0.13 ± 0.03 (3) [29]	
Elemental Carbon[i]	Thermal-Optical-FID	0.046 ± 0.005 (3) [29]	
	Acid/Base-Combustion-Manometry	0.047 ± 0.001 (6) [32]	
	HNO_3-Combustion-NDIR	0.052 ± 0.006 (5) [33]	
Pyrolyzed Carbon[i]	Thermal-Optical-FID	0.037 ± 0.003 (3) [29]	
Carbonate Carbon[l]	H_3PO_4 Oxidation-Manometry	0.001 17 ± 0.000 03 (2) [32]	
Extractable Mass[k]	Soxhlet Extraction	0.044 ± 0.003 (3) [33]	33 ± 1[c] (3) [32]
PAH Fraction[m]	Soxhlet Extraction/LC		17 ± 4[c] (1) [41]
Individual PAHs	Soxhlet Extraction/GC		
Fluoranthene		(see Table 1)	20[n] [42]
Benz[*a*]anthracene		(see Table 1)	12[n] [42]
Benzofluoranthenes (*b*, *j*, and *k*)		(see Table 1)	15[n] [42]
Benzo[*ghi*]perylene		(see Table 1)	14[n] [42]

[a] The reference value is the mean value of *n* measurements (*n* is the number in parentheses). Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [39], 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 approximately 95 %.

[b] Percent Modern Carbon: The percentage of modern carbon (pMC) is based on $^{14}C/^{12}C$ ratio measurements of the chemical fraction (X) relative to $^{14}C/^{12}C$ ratio of SRM 4990b, Oxalic Acid for Radiocarbon Dating, using the following equation:
$pMC = [^{14}C/^{12}C_{(X)} / (0.95\ ^{14}C/^{12}C_{SRM\ 4990b})] \times 100$.

[c] Low-level counting; uncertainty is one standard deviation based on Poisson counting statistics.

[d] $\delta^{13}C = -25.3$ ‰ ± 0.1 ‰ [40]; Carbon-13 composition is expressed in delta (δ) notation as the relative deviation of a sample $^{13}C/^{12}C$ ratio (R_x) from a specified standard $^{13}C/^{12}C$ ratio (R_{std}) in per mill (‰), using the following equation: $\delta^{13}C = [(R_x - R_{std})/ R_{std}] \times 1000$ where R_{std} is VPDB (Vienna Pee Dee Belemnite) [43].

[e] The uncertainties for the AMS measurements are one standard deviation based on Poisson counting statistics. For *n* greater than 1, the value is the average with an uncertainty reported as the standard deviation of the mean which includes sampling effects.

[f] Percent modern carbon value age corrected to December 1997.

[g] Percent modern carbon value age corrected to March 1998.

[h] $\delta^{13}C = -25.18$ ‰ ± 0.03 ‰ [32]. See above footnote for definition of carbon-13 composition.

[i] Definitions of organic, elemental, and pyrolyzed carbon are method dependent (see Preparation and Analysis section).

[j] Percent modern carbon value age corrected to February 1998. The individual pMC's are 57.1 ± 0.4 and 54.3 ± 0.4.

[k] Extractable Organic Mass: Mass extracted from the bulk particulate material usingSoxhlet extraction for 24 h with dichloromethane. (Compare to value of 0.0460 g/g for extractable mass in Table 10 determined using Soxhlet extraction with hexane/acetone.)

[l] Carbonate Carbon: Validation based on total carbon recovery of SRM 1a, Argillaceous Limestone.

[m] PAH Fraction: Fraction isolated by normal-phase LC representing the fraction in which the majority of the PAHs elute (see Preparation and Analysis section).

[n] These values are reported as information values only. An information value is considered to be a value that will be of interest and use to the SRM user, but for which insufficient information is available to assess adequately the uncertainty associated with the value. The uncertainties of these measurements are typically 0.5 % to 0.6 % modern carbon.

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