



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), nitro-substituted polycyclic aromatic hydrocarbons (nitro-PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and total carbon in atmospheric particulate material and similar matrices. Reference and information values are also provided for selected polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners, inorganic constituents, total extractable material, particle-size characteristics, and chemical and isotopic (¹⁴C) carbon composition. All of the constituents for which certified, reference, and information 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, reference, and information values for additional constituents. A unit of SRM 1649a consists of an amber glass bottle containing 2.5 g of particulate material.

Certified Concentration Values: Certified values for the concentrations, expressed as mass fractions, for 25 PAHs, 6 nitro-PAHs, 35 PCB congeners (some in combination), and 8 chlorinated pesticides are provided in Tables 1, 2, 3, and 4, respectively and for total carbon in Table 11. 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 [2]. 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, and the certified values for the nitro-PAHs are based on the agreement of results obtained from multiple analytical techniques performed at NIST and at three collaborating laboratories.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 30 additional PAHs and 10 additional nitro-PAHs in Tables 5 and 6, respectively, and for one additional chlorinated pesticide and levoglucosan in Table 7. Reference values are provided in Table 8 for the seventeen 2,3,7,8-substituted polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners and the total tetra-, penta-, hexa-, and hepta-substituted congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Reference values for 32 selected inorganic constituents are provided in Table 9, and a reference value for total extractable mass is provided in Table 10. Reference values for chemical carbon and isotopic carbon (¹⁴C) composition are summarized in Tables 11 and 12, respectively. 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 [2].

Information Concentration Values: Information concentration values, expressed as mass fractions, for selected components of the chemical and isotopic carbon composition are provided in Tables 11 and 12, respectively. Information values for particle-size characteristics are provided in Table 13. An information value is considered to be a value that will be of 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 [2].

Expiration of Certification: The certification of SRM 1649a is valid, within the measurement uncertainties specified, until **30 April 2012**, provided the SRM is handled and stored in accordance with the instructions (see "Notice and Warning to User") 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. Registration (see attached sheet) will facilitate notification.

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters Jr., Chief
Standard Reference Materials Program

Gaithersburg, MD 20899
Certificate Issue Date: 06 December 2007
See Certificate Revision History on Last Page

Coordination of the technical measurements leading to the updated certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division. Coordination of the measurements and evaluation of the data for chemical and isotopic carbon composition were performed by L.A. Currie and G.A. Klouda of the NIST Surface and Microanalysis Science Division.

Analytical measurements for the certification of SRM 1649a were performed by H.A. Bamford, B.A. Benner, Jr., A. Deissler, R.R. Greenberg, M.J. Hays, B.J. Porter, D.L. Poster, L.C. Sander, P. Schubert, M.M. Schantz, R. L. Watters, Jr., and R.L. Zeisler of the NIST Analytical Chemistry Division. Analytical measurements for the nitro-PAHs included results from three laboratories (B. Zielinska and L. Rinehart of Desert Research Institute, Reno, Nevada; C. Chiu and G. Poole of Environment Canada, Environmental Technology Centre, Analysis and Air Quality Division, Ottawa, Ontario, Canada; and J. Arey, Air Pollution Research Center, University of California, Riverside, California). Analytical measurements for the polychlorinated dibenzo-*p*-dioxins and dibenzofurans were the results of an interlaboratory comparison study among 13 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). Contributors to the analytical measurements for isotopic and particulate carbon composition are listed in Appendix B. The results were derived primarily from an interlaboratory comparison study coordinated by L.A. Currie, with assistance from G.A. Klouda and J.D. Kessler of the NIST Surface and Microanalysis Science Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh, M.S. Levenson, 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, Office of Health Research.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

NOTICE AND WARNING TO USERS

Storage: SRM 1649a is provided in an amber glass bottle 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 moisture content to correct the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS¹

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-mass basis; however, the material “as received” contains residual moisture. The amount of moisture in SRM 1649a 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

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

-10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1649a at the time of the certification analyses was $1.23 \% \pm 0.07 \%$ at the 95 % confidence level.

Polycyclic Aromatic Hydrocarbons (Tables 1 and 5): 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 [3–6]. 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 reference 7 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 [8–11]. 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. \times 15 cm, 3 μm particle size, ChromSpher PAH, Chrompack, Middelburg, The Netherlands and 4.6 mm i.d. \times 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [3,9,10].

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 [8–11] on a semi-preparative aminopropylsilane column. The PAH fraction was then analyzed by GC/MS using a 0.25 mm i.d. \times 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 in reference 12, 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. \times 60 m, 0.25 μm film thickness) (DB-17MS, J&W Scientific). 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 [12], 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. \times 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 [3]. 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. A sample size of approximately 120 mg will contribute less than 2 % error due to sample homogeneity for the PAHs for which certified values are provided. A more extensive evaluation of the homogeneity of SRM 1649a for PAHs at small sample sizes is described in reference 13.

PAH Isomers of Molecular Mass 300 and 302 (Tables 1 and 5): For the determination of the molecular mass 300 and 302 isomers, up to six sets of data were used. Four sets of data used the method described in reference 14 with GC/MS analysis using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (GC/MS V). Perdeuterated dibenzo[*a,i*]pyrene was added to the air particulate matter prior to extraction for use as an internal standard. One of the sets of samples was also analyzed on a dimethyl 50 % polysiloxane liquid crystalline stationary phase (LC-50, J&K Environmental, Milton, Ontario, Canada) for the fifth data set (GC/MS VI). The sixth set of data came from LC-FL analyses as described in reference 11.

Nitro-Substituted PAHs (Tables 2 and 6): SRM 1649a was analyzed at NIST and in an interlaboratory study with three participants for the determination of nitro-substituted PAHs. The general procedure for determination of nitro-PAHs at NIST has been reported previously [15] and is described briefly below. Two sets of subsamples of approximately 100 mg from each of six bottles of SRM 1649a were mixed with Hydromatrix and extracted with dichloromethane using PFE at 100 °C and 2000 psi. The extracts were concentrated to about 0.5 mL, solvent exchanged to hexane, and placed on an aminopropylsilane solid phase extraction (SPE) cartridge and eluted with 40 mL of 20 % dichloromethane in hexane. The concentrated eluant was then subjected to normal-phase liquid chromatography (LC) using a semi-preparative amino/cyano phase column with a mobile phase of 20 % dichloromethane in hexane to isolate the nitro-PAH fraction as described previously [15]. The nitro-PAH fraction was analyzed by GC with negative ion chemical ionization mass spectrometry (GC/NICI-MS) using a 0.25 mm i.d. × 30 m fused silica capillary column containing a 50 % phenyl-substituted methylpolysiloxane stationary phase (0.25 µm film thickness). Selected perdeuterated nitro-PAHs were added to the air particulate matter prior to extraction for use as internal standards for quantification purposes.

PCBs and Chlorinated Pesticides (Tables 3, 4, and 7): 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 [4,6,16,17]. 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 × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) 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 in reference 12. Subsamples of 1 g to 2 g were extracted using approximately 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 [18,19]), 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 in reference 20.

Polychlorinated Dibenzo-*p*-dioxins and Dibenzofurans (Table 8): Value assignment of the concentrations of the seventeen 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 1649a by 13 laboratories that participated in an interlaboratory comparison study (see Appendix A). Each laboratory analyzed three subsamples (typically 0.5 g) of SRM 1649a using their routine analytical procedures and high resolution gas chromatography with high resolution mass spectrometry detection (GC-HRMS). The analytical procedures used by all of the laboratories included spiking with ¹³C-labeled surrogates (internal standards); Soxhlet extraction with toluene; sample extract cleanup with acid/base silica, alumina, and

carbon columns; and finally, analysis of the cleaned up extract with GC-HRMS. Most of the laboratories used a 5 % phenyl-substituted methylpolysiloxane phase capillary column (DB-5), and about half of the laboratories confirmed 2,3,7,8-tetrachlorodibenzofuran using a 50 % cyanopropylphenyl-substituted methylpolysiloxane (DB-225, J&W Scientific) capillary column. The results from the interlaboratory study are described in more detail in reference 21.

Inorganic Constituents (Table 9): 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 (copper, lead, magnesium, manganese, nickel, and vanadium) 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 chlorine and sulfur, 250 mg subsamples from six bottles of SRM 1649a were analyzed using high pressure oxygen bomb combustion followed by ion chromatography.

Extractable Mass (Table 10): 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.

Isotopic and Particulate Carbon Composition (Tables 11 and 12): Laboratories and analysts contributing to the isotopic and particulate carbon composition results are listed in Appendix B. Data for this section were compiled by L.A. Currie and G.A. Klouda of the NIST Surface and Microanalysis Science Division.

Nomenclature [22]: Nomenclature for “total” and “elemental” fractions of particulate carbon tends to be ambiguous and varied across disciplines. As used here, “total carbon (TC)” refers to the total, non-carbonate fraction of particulate carbon; as such, it includes what are commonly called “organic” and “elemental” carbon fractions. The meaning of TC is identical to what is frequently called “total organic carbon (TOC)” in some areas of geoscience, a term used to highlight the removal of inorganic (carbonate) carbon.

Problems with metrology and nomenclature are even more serious for the Refractory Pyrogenic Carbon (RPC) phase labeled EC here. As a result, only information values can be given for this constituent at this time. Among the terms having wide usage are: char, black carbon, elemental carbon, soot carbon, refractory carbon, and graphitic carbon [23]. In some cases, these terms have specialized meanings that are method-specific or material/structure-specific; in other cases, they are used interchangeably. For a discussion and recommendations concerning the RPC nomenclature problem, see reference 22.

Thermal-Optical-FID Method (SLI, NIST): In a completely oxygen-free helium atmosphere, the sample (~1 mg) was heated in four increasing steps (from approximately 500 °C to 900 °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 helium-neon laser light through the filter. As organic compounds are volatilized, they are immediately oxidized to carbon dioxide using a plug of manganese dioxide at 700 °C, reduced to methane over nickel on firebrick in the presence of hydrogen, and measured using a flame ionization detector (FID). After cooling the sample to approximately 525 °C, a 2 % or 5 % oxygen/helium mixture was introduced and the temperature increased to approximately 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 [24].

Combustion-GC-TCD (NIST): Samples (0.3 mg to 9 mg) were weighed into aluminum boats, combusted to carbon dioxide at 900 °C in an atmosphere of oxygen, purified by gas chromatography (GC), and quantified with GC using a thermal conductivity detector (TCD).

Combustion-NDIR (NIST): The weighed sample was placed in a ceramic crucible which was then purged with oxygen while inductively heating the crucible. The carbon monoxide and carbon dioxide produced were measured using a non-dispersive infrared (NDIR) detector [25].

Combustion-CHN (SU, URI): Samples were placed in silver boats and fully oxidized and quantified by flash combustion/gas chromatographic analysis using a commercial CHN-analyzer. This process served also as the final carbon quantification step for the “Gustafsson procedure” to determine elemental (“soot”) carbon (SC). For SC assay, the final CHN combustion step was preceded by thermal oxidation for 24 h at 375 °C in the presence of excess oxygen and treatment with hydrochloric acid for carbonate removal if necessary [26].

H₂O Extraction, Combustion-Manometry (NIST): Subsamples of SRM 1649a were placed on pre-fired quartz filters. Three 200-milliliter 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 copper oxide. The resulting carbon dioxide was distilled and quantified by manometry and then ¹⁴C analysis was performed at UA [27].

H₃PO₄ Acidification-Manometry (NIST): 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 to 3 h. A sample bulb immersed in liquid nitrogen was used to collect the evolved carbon dioxide, which was then measured using manometry as described above.

Soxhlet Extraction (EPA) [25]: 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 removing a 200 µL aliquot of the extract solution, 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. Copper oxide and silver 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 carbon dioxide at 900 °C. The sample carbon dioxide was then reduced to a graphite-iron bead [28] for ¹⁴C AMS measurements at UA [27].

Combustion-Manometry (NIST) [29]: Samples were combusted to carbon dioxide in a quartz furnace filled with 101 kPa oxygen. Downstream of the combustion furnace is a series of three furnaces: (1) platinum gauze at 900 °C, (2) copper oxide at 800 °C, and (3) silver wool at 400 °C to assure complete combustion and to purify the carbon dioxide of sulfur and halogen containing impurities. The sample gas stream is then reduced to less than 13 kPa to prevent the condensation of liquid oxygen by controlling the gas flow through the system using a throttle valve and a vacuum pump. Before the vacuum pump, the sample carbon dioxide is cryogenically trapped at liquid nitrogen temperature (−196 °C) in a series of spiral glass traps. The resulting carbon dioxide is cryogenically separated from other gaseous combustion products by distillation from −78 °C and quantified using manometry in a calibrated volume. Low level ¹⁴C decay counting was performed on the carbon dioxide using a miniature gas proportional counter at NIST [30,31].

H₃PO₄-Combustion-Manometry (UCI): A subsample of SRM 1649a, silver foil (pre-fired at 550 °C), and copper oxide wire (pre-fired at 850 °C) were added to a quartz tube. Approximately 5 mL of 3 % phosphoric acid (H₃PO₄) as 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 carbon dioxide at 850 °C for 4 h. The carbon dioxide was reduced to graphite over cobalt catalyst at 850 °C in the presence of hydrogen. Accelerator mass spectrometry ¹⁴C measurements were performed at Lawrence Livermore National Laboratory (LLNL).

Combustion-GC (NIST) [32]: Samples were placed in tin boats and fully oxidized and quantified by flash combustion/gas chromatographic analysis using a commercial CHN-analyzer, by essentially the same procedure used for TC quantification by Combustion-GC-TCD. The purified carbon dioxide was then trapped at −196 °C and transferred to UA for preparation of a graphite target [35] and ¹⁴C AMS [27].

Soxhlet Extraction/LC Isolation of Aromatic Fraction (NIST): Samples were Soxhlet extracted for 24 h with dichloromethane. The extract was concentrated to a small volume under a stream of nitrogen. The concentrated extract was placed on a silica SPE cartridge and eluted with 10 % dichloromethane in pentane. The aromatic fraction was isolated using normal-phase LC on an aminopropylsilane column as described above. This fraction was concentrated and a 1 mL aliquot transferred to a quartz tube and evaporated to dryness. Copper oxide was added to the tube, which was then attached to a vacuum line, evacuated, sealed, and the contents combusted to carbon dioxide. Low-level ¹⁴C decay counting was performed on the sample carbon dioxide using a miniature gas proportional counter at NIST [30,31].

Soxhlet Extraction/LC/PCGC (NIST, WHOI): Extraction followed by LC was used for isolating polar, aromatic, and aliphatic fractions for ¹⁴C analysis. The aromatic fraction was specially purified using silica gel flash chromatography and LC PAH ring size fractionation [8], prior to separation and collection of individual PAH using an automated preparative capillary GC (PCGC) system described in reference 34. The individual PAH fractions were then subjected to closed-tube combustion and ¹⁴C determined by AMS [22, 35–37].

¹⁴C Methods for Elemental Carbon [22]

Thermal oxidation/residue (WHOI): ¹⁴C is measured in the residual carbon after thermal oxidation at 375 °C for 24 h (to remove labile organic carbon) and acidification (to remove inorganic carbonates). The residual carbon is placed in a quartz tube containing copper oxide and elemental silver and combusted at 850 °C for 5 h. The ¹⁴C content of the resulting carbon dioxide was measured by accelerator mass spectrometry [37].

Chemical oxidation, dichromate/residue (UCI): Wet oxidation with 0.1 M Cr₂O₇²⁻ in 2 M H₂SO₄ at 50 °C was performed for periods up to 406 h. The ¹⁴C AMS result is given for the residual carbon for the longest (406 h) reaction period. Because of the gentler oxidation treatment (chemical vs. thermal), the ¹⁴C must reflect somewhat less refractory components of the “black carbon spectrum” [38].

Thermal kinetic oxidation/intercept (NIST): EC is defined as the refractory (intercept) component that survives isothermal oxidation at 560 °C in a stream of helium (5 % oxygen). The intercept is estimated by fitting a five parameter model (2 exponentials + intercept) to the residual carbon rate function. Intercept ¹⁴C is estimated as the end point of a series of three intermediate samples taken for ¹⁴C AMS [39].

Other Thermal EC Methods (LBNL, LSCE, DRI, TUW): Four additional thermal and thermal-optical methods were employed by laboratories contributing to the EC/TC results in Table 13. Two-stage to multi-stage thermal steps were employed, with the earlier stages including lower temperature oxidation (air, oxygen) or volatilization (helium), and the later stages including high temperature oxidation (air, oxygen, helium-oxygen) and, in some cases, laser transmission or reflectance to correct for charring [40–43]. Full details are given in the review of the international comparison [22].

Particle-Size Information (Table 13): Particle size distribution measurements for SRM 1649a were carried out using a laser diffraction instrument (Mastersizer 2000, Malvern Instruments, Southborough, MA) and the liquid suspension method with the instrument manufacturer’s small volume sample dispersion unit (Hydro 2000 SM). A suspension of 0.1 % by mass of SRM 1649a in distilled water with 0.001 % Triton (volume fraction), was prepared by ultra-sonication for 1 h. A measurement sequence of background and sample measurement was used. After the recording of the background, a portion of the suspension was added to the measurement cell to achieve an obscuration of 5 %. Three passes of the sample solution were recorded and averaged. A refractive index of 1.5 and absorption index of 0.1 were selected for the measurements. Results were calculated using the General Purpose Model provided by the instrument manufacturer; the results obtained are shown in Figure 1.

Supplemental Information for SRM 1649a: Because SRM 1649 and SRM 1649a have been available since 1982, a considerable amount of information on the characterization of these materials has been published. A summary of some of the studies reporting characterization of this air particulate matter SRM is provided in Wise et al. [44]. Descriptions of selected studies are provided below as supplemental information for SRM 1649a.

Mutagenicity Assay: 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) and supported and technically coordinated by the U.S. Environmental Protection Agency's (EPA) Office of Health Research. Twenty laboratories from North America, Europe, and Japan participated in the study for which a complete summary has been published [45,46]. Mutagenicity data were provided by J. Lewtas and L.D. Claxton of the National Health and Environmental Effects Research Laboratory, U.S. Environmental Protection Agency (Research Triangle Park, NC).

Additional Characterization: Several papers have reported the concentrations of selected oxygenated polycyclic aromatic hydrocarbons (OPAHs) in SRM 1649a. Fernandez and Bayona [47] reported measurements of six OPAHs using GC/MS after isolating the OPAH fraction. Later, Durant et al. [48] quantified eight OPAHs (ketones and quinines) as part of an investigation of the mutagenicity of various fractions isolated from SRM 1649a. Recently, Albinet et al. [49] reported eight OPAHs in SRM 1649a as part of the validation for a GC/MS method to simultaneously measure nitro-PAHs and OPAHs.

Table 1. Certified Concentrations for Selected PAHs in SRM 1649a^(a,b)

Nitro-substituted PAH	Mass Fraction (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[<i>a</i>]anthracene ^(c,d,h)	2.208 ± 0.073
Chrysene ^(e,h)	3.049 ± 0.060 ⁽ⁱ⁾
Triphenylene ^(e,h)	1.357 ± 0.054
Benzo[<i>b</i>]fluoranthene ^(e,f,g)	6.45 ± 0.64
Benzo[<i>k</i>]fluoranthene ^(c,d,e,f,g)	1.913 ± 0.031
Benzo[<i>a</i>]fluoranthene ^(c,d,e)	0.409 ± 0.035
Benzo[<i>e</i>]pyrene ^(c,d,e)	3.09 ± 0.19
Benzo[<i>a</i>]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[<i>ghi</i>]perylene ^(c,d,g,h,j)	4.01 ± 0.91
Indeno[1,2,3- <i>cd</i>]pyrene ^(c,d,g,h,j)	3.18 ± 0.72
Dibenz[<i>a,j</i>]anthracene ^(c,d,g,h)	0.310 ± 0.034
Dibenz[<i>a,c</i>]anthracene ^(e,g,h)	0.200 ± 0.025
Dibenz[<i>a,h</i>]anthracene ^(e,g,h)	0.288 ± 0.023
Pentaphene ^(c,d,g,h)	0.151 ± 0.035
Benzo[<i>b</i>]chrysene ^(c,d,e,g,h)	0.315 ± 0.013
Picene ^(c,d,e,g,h)	0.426 ± 0.022
Dibenzo[<i>b,k</i>]fluoranthene ^(k,l)	0.724 ± 0.076 ^(m)
Dibenzo[<i>a,e</i>]pyrene ^(k,l,n)	0.565 ± 0.060 ^(m)
Dibenzo[<i>a,h</i>]pyrene ^(k,l,n)	0.047 ± 0.010 ^(m)

^(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 [50], 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).

^(k) Four sets of data using GC/MS(V) analysis for molecular mass 302 isomers on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with dichloromethane as described in reference 14.

^(l) LC/FL analysis of the molecular mass 302 isomers as described in reference 11.

^(m) The certified value is a weighted mean of the results from four to six analytical methods [51]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO *Guide to the Expression of Uncertainty in Measurement* [50].

⁽ⁿ⁾ GC/MS (VI) analyses for the molecular mass 302 isomers on a dimethyl 50 % polysiloxane liquid crystalline stationary phase after PFE with dichloromethane.

Table 2. Certified Concentrations for Selected Nitro-substituted PAHs in SRM 1649a^(a,b)

Nitro-substituted PAH	Mass Fraction ($\mu\text{g}/\text{kg}$)
9-Nitroanthracene ^(c,d)	33.4 \pm 6.1
1-Nitropyrene ^(c,d)	70.9 \pm 4.3
2-Nitrofluoranthene ^(c,d)	313 \pm 38
3-Nitrofluoranthene ^(c)	4.5 \pm 1.8
7-Nitrobenz[<i>a</i>]anthracene ^(c,d)	27.8 \pm 6.7
6-Nitrochrysene ^(c,d)	4.01 \pm 0.52

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.2 % moisture.

^(b) The certified value is a weighted mean of the results from three to four analytical methods [51]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [50].

^(c) Three sets of data using GC/NICI-MS for nitro-PAHs on a 50% phenyl-substituted methylpolysiloxane stationary phase after PFE with dichloromethane as described in reference 15.

^(d) Data from two interlaboratory studies in which three laboratories participated for nitro-PAHs.

Table 3. Certified Concentrations for Selected PCB Congeners^(a) in SRM 1649a^(b,c)

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

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [18] and later revised by Schulte and Malisch [19] to conform with IUPAC rules; for the specific congeners identified 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 [52]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [50], 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 4. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1649a^(a,b)

Chlorinated Pesticide	Mass Fraction ($\mu\text{g}/\text{kg}$)		
Hexachlorobenzene	16.3	\pm	1.8
<i>trans</i> -Chlordane (γ -Chlordane)	40.3	\pm	2.8
<i>cis</i> -Chlordane (α -Chlordane)	34.88	\pm	0.42
<i>trans</i> -Nonachlor	27.6	\pm	1.6
2,4'-DDE	5.79	\pm	0.85
4,4'-DDE	40.4	\pm	1.7
4,4'-DDD	34.01	\pm	0.48
4,4'-DDT	212	\pm	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 [52]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [50], 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 5. Reference Concentrations for Selected PAHs in SRM 1649a

PAH	Mass Fraction ^(a) (mg/kg)	
Fluorene ^(c)	0.23	± 0.05 ^(b)
Dibenzothiophene ^(d)	0.18	± 0.01 ^(b)
1-Methylphenanthrene ^(c,d,e)	0.37	± 0.04 ^(b)
2-Methylphenanthrene ^(c,d,e)	0.73	± 0.12 ^(b)
3-Methylphenanthrene ^(c,d,e)	0.50	± 0.05 ^(b)
4-Methylphenanthrene and 9-Methylphenanthrene ^(c,d,e)	0.34	± 0.01 ^(b)
Retene ^(f)	0.304	± 0.019 ^(g)
4H-Cyclopenta[<i>def</i>]phenanthrene ^(c,d,e)	0.32	± 0.06 ^(b)
Benzo[<i>c</i>]phenanthrene ^(d,e)	0.46	± 0.03 ^(b)
Benzo[<i>ghi</i>]fluoranthene ^(d)	0.88	± 0.02 ^(b)
Benzo[<i>j</i>]fluoranthene ^(d,f)	1.5	± 0.4 ^(b)
Indeno[1,2,3- <i>cd</i>]fluoranthene ^(e)	0.23	± 0.01 ^(b)
Benzo[<i>c</i>]chrysene ^(d)	0.080	± 0.004 ^(b)
Corenene ^(h)	3.59	± 0.58 ⁽ⁱ⁾
Dibenzo[<i>b,e</i>]fluoranthene ^(h)	0.0755	± 0.0031 ⁽ⁱ⁾
Naphtho[1,2- <i>b</i>]fluoranthene ^(h,j)	0.615	± 0.185 ⁽ⁱ⁾
Naphtho[1,2- <i>k</i>]fluoranthene and naphtho[2,3- <i>j</i>]fluoranthene ^(h,k)	0.511	± 0.068 ⁽ⁱ⁾
Naphtho[2,3- <i>b</i>]fluoranthene ^(h,k)	0.181	± 0.020 ⁽ⁱ⁾
Dibenzo[<i>a,k</i>]fluoranthene ^(h,j)	0.078	± 0.014 ⁽ⁱ⁾
Dibenzo[<i>j,l</i>]fluoranthene ^(h,j)	0.369	± 0.092 ⁽ⁱ⁾
Dibenzo[<i>a,l</i>]pyrene ^(h)	0.0612	± 0.0074 ⁽ⁱ⁾
Naphtho[2,3- <i>k</i>]fluoranthene ^(h,j,k)	0.0563	± 0.0009 ⁽ⁱ⁾
Naphtho[2,3- <i>e</i>]pyrene ^(h,j)	0.215	± 0.024 ⁽ⁱ⁾
Naphtho[2,1- <i>a</i>]pyrene ^(h,i)	0.453	± 0.043 ⁽ⁱ⁾
Dibenzo[<i>e,l</i>]pyrene ^(h,i)	0.361	± 0.062 ⁽ⁱ⁾
Naphtho[2,3- <i>a</i>]pyrene ^(h,j,k)	0.0503	± 0.0054 ⁽ⁱ⁾
Benzo[<i>b</i>]perylene ^(h,i)	0.279	± 0.042 ⁽ⁱ⁾
Dibenzo[<i>a,i</i>]pyrene ^(h,j,k)	0.153	± 0.015 ⁽ⁱ⁾

(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.

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

(h) Four sets of data using GC/MS(V) analysis for molecular mass 302 isomers on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with dichloromethane as described in reference 14.

(i) The reference value is a weighted mean of the results from four to six analytical methods [51]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO *Guide to the Expression of Uncertainty in Measurement* [50].

(j) GC/MS (VI) analyses for the molecular mass 302 isomers on a dimethyl 50 % polysiloxane liquid crystalline stationary phase after PFE with dichloromethane.

(k) LC/FL analysis of the molecular mass 302 isomers as described in reference 11.

Table 6. Reference Concentrations for Selected Nitro-substituted PAHs in SRM 1649a

Nitro-substituted PAH	Mass Fraction ^(a,b,c)	
	(µg/kg)	
1-Nitronaphthalene	6.8	± 0.3
2-Nitronaphthalene	10.0	± 0.5
3-Nitrobiphenyl	3.6	± 0.5
5-Nitroacenaphthene	3.1	± 0.3
4-Nitrophenanthrene	0.47	± 0.03
3-Nitrophenanthrene	22.0	± 0.6
9-Nitrophenanthrene	1.7	± 0.1
2-Nitropyrene	24.4	± 4.0
4-Nitropyrene	5.5	± 0.6
8-Nitrofluoranthene	8.8	± 1.4

^(a) Concentrations reported on dry-mass basis; material as received contains approximately 1.2 % moisture.

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

^(c) GC/NICI-MS for nitro-PAHs on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with dichloromethane as described in reference 15.

Table 7. Reference Concentrations for an Additional Chlorinated Pesticide and Levoglucosan in SRM 1649a^(a)

	Mass Fraction (µg/kg)
Heptachlor ^(b,c)	18.9 ± 0.5 ^(d)
	Mass Fraction (mg/kg)
Levoglucosan ^(e)	81.1 ± 6.1 ^(f)

^(a) Concentration 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 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.

^(e) GC/MS analysis following derivatization as described in reference 53.

^(f) The reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, U , is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k , is determined from the Student's t -distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte.

Table 8. Reference Concentrations for Selected Dibenzo-*p*-dioxin and Dibenzofuran Congeners in SRM 1649a

	Mass Fraction ($\mu\text{g}/\text{kg}$) ^(a,b)		
2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin	0.011	±	0.004 ^(c)
1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin	0.091	±	0.012
1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	0.26	±	0.02
1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	0.68	±	0.05
1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin	0.64	±	0.11
1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin	18.8	±	1.5
Octachlorodibenzo- <i>p</i> -dioxin	201	±	20
2,3,7,8-Tetrachlorodibenzofuran ^(d)	0.068	±	0.15 ^(e)
1,2,3,7,8-Pentachlorodibenzofuran	0.090	±	0.010
2,3,4,7,8-Pentachlorodibenzofuran	0.28	±	0.03
1,2,3,4,7,8-Hexachlorodibenzofuran	0.87	±	0.26
1,2,3,6,7,8-Hexachlorodibenzofuran	0.43	±	0.06
2,3,4,6,7,8-Hexachlorodibenzofuran	0.69	±	0.03 ^(f)
1,2,3,7,8,9-Hexachlorodibenzofuran	0.066	±	0.029 ^(f)
1,2,3,4,6,7,8-Heptachlorodibenzofuran	3.8	±	0.2 ^(f)
1,2,3,4,7,8,9-Heptachlorodibenzofuran	0.46	±	0.07 ^(f)
Octachlorodibenzofuran	6.8	±	0.8 ^(f)
Total Toxic Equivalents (TEQ) ^(g)	0.86	±	0.06
Total Tetrachlorodibenzo- <i>p</i> -dioxins	0.16	±	0.08 ^(f)
Total Pentachlorodibenzo- <i>p</i> -dioxins	0.88	±	0.16
Total Hexachlorodibenzo- <i>p</i> -dioxins	6.0	±	0.5
Total Heptachlorodibenzo- <i>p</i> -dioxins	36.4	±	3.4
Total Tetrachlorodibenzofurans	0.52	±	0.13
Total Pentachlorodibenzofurans	1.6	±	0.2
Total Hexachlorodibenzofurans	5.0	±	0.6
Total Heptachlorodibenzofurans	9.8	±	0.8
Total Dibenzo- <i>p</i> -dioxins ^(h)	244	±	22
Total Dibenzofurans ^(h)	23.9	±	1.9

^(a) Each reference value is the mean of the results from up to 13 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 Guide [50] and k is the coverage factor. The value 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 (12 unless noted otherwise). The coverage factor, k , is the value from a Student's t -distribution corresponding to a 95 % level of confidence.

^(b) Concentrations reported on dry-mass basis; material as received contains approximately 1.2 % moisture.

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

^(d) Confirmation results using a 50 % cyanopropylphenylpolysiloxane or 90 % *bis*-cyanopropyl 10 % cyanopropylphenylpolysiloxane phase columns.

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

^(f) Degrees of freedom = 11 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 in reference 54. 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 octa-chlorinated congeners.

Table 9. Reference Concentrations for Selected Elements in SRM 1649a

Element	Mass Fraction (%) ^(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 Fraction (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 instrumental neutron activation analysis (INAA) on duplicate subsamples from six bottles or measurements made by inductively coupled plasma atomic emission spectrometry (ICP-AES) 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 [50], 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 INAA.

^(d) Determined using high pressure oxygen bomb combustion and ion chromatography.

^(e) Determined using 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.

Table 10. Reference Value for Percent Extractable Mass in SRM 1649a

	Mass Fraction (%)
Extractable Mass	4.6 ± 0.4 ^(a,b,c)

- ^(a) Concentration is reported on an as-received basis; material as received contains approximately 1.2 % moisture.
- ^(b) Extractable mass value was determined from Soxhlet extraction using 50 % hexane/50 % acetone (volume/volume).
- ^(c) 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 [50], 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. Certified (in bold), Reference, and Information Values for Particulate Carbon Composition of SRM 1649a

Chemical Component	Mass Fraction (g/g) ^(a)	
Total Carbon (TC) ^(b)	0.1768	± 0.0019
Insoluble Carbon ^(c,d)	0.152	(<i>n</i> = 2)
Carbonate Carbon ^(d,e)	0.00118	(<i>n</i> = 2)
Extractable Mass ^(f,g)	0.044	± 0.006 (<i>n</i> = 3)
Elemental carbon, fraction of total (EC/TC) ^(h)	Median	Inter-quartile range
Cluster-1	0.075	0.071, 0.078 (<i>n</i> = 4)
Cluster-2	0.28	0.27, 0.29 (<i>n</i> = 4)
Cluster-3	0.46	0.44, 0.50 (<i>n</i> = 5)
	Complete range:	0.069, 0.520 (<i>n</i> = 19)

- ^(a) Concentration is reported on an as received basis; material as received contains approximately 1.2 % moisture. For the identification and codes of laboratories contributing to the data, and literature citations for the reference methods, please consult the section, "Preparation and Analysis" and "Appendix B".
- ^(b) The certified value for TC is the 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 [50], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty within each analytical method. The expanded uncertainty defines a range of values for the certified value, which is expected to cover the true value with a level of confidence of approximately 95 %. Methods contributing to the certified value are Thermal-Optical-FID (SLI, *n* = 3; NIST, *n* = 2), Combustion-GC-TCD (NIST, *n* = 39), Combustion-NDIR (NIST, *n* = 15), Combustion-Coulometry (LSCE, *n* = 4), and Combustion-CHN (SU, *n* = 4; URI, *n* = 10). (The symbol *n* represents the respective number of measurements for each mean.)
- ^(c) H₂O Extraction, Combustion-Manometry (NIST).
- ^(d) Information value. For a single method, with just one degree of freedom (*n* = 2) and no additional uncertainty information, NIST has chosen to tabulate only the median (mean), as an information value. This is justified because of the extreme variation of the expanded uncertainty (95 % confidence interval) in this case, i.e., greater than a factor of 30 (90 % interval) for normal data, and the minimal impact of the Central Limit Theorem. The alternative, which NIST does not recommend, despite its statistical validity for normal, random data, is to use a coverage factor of 12.7 to construct the (95 %) expanded uncertainty.
- ^(e) H₃PO₄ Acidification Manometry (NIST).
- ^(f) Reference value, which equals the mean value of *n* measurements (*n* is number in parentheses). Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [50], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty.
- ^(g) Soxhlet Extraction (EPA). Although the mass fraction of extractable carbon in dichloromethane given here is consistent with that given in Table 10, the compounds extracted in the two cases are not necessarily the same. The numerical similarity may be fortuitous because different solvents were used for the extractions.
- ^(h) Information values for the fraction of "elemental" carbon (EC/TC) are derived from an interlaboratory and intermethod comparison [22,39]. Three broad classes of methods were represented: optical (absorbance, reflectance), dry oxidation ("thermal"), and wet oxidation ("chemical"). Also represented were hybrid methods (thermal-chemical, thermal-optical) and kinetic modeling approaches. One conclusion of the exercise is that one should not expect consistent results across methods, because they are (appropriately) measuring different regions of the "black carbon spectrum," which ranges from char, at the one extreme, to graphitic soot at the other. In this table, NIST presents only the principal features of the results, namely the range observed, and three clusters of EC/TC fractions, which together included about 70 % of the data. Exploratory descriptive statistics (medians, interquartile ranges) are used to characterize the three clusters [55]. A complete exposition of the structure and results of the exercise is given in reference 22.

Table 12. Reference and Information Values for ¹⁴C Isotopic Carbon Composition of SRM 1649a

Isotopic Component	¹⁴ C, fraction of modern (f_M) ^(a)
Total Carbon (TC) ^(b)	
Combustion-Manometry (NIST)	0.61 ± 0.08 (Poisson)
H ₃ PO ₄ -Combustion-Manometry (UCI)	0.505 ± 0.006 (Poisson) ^(c)
Combustion-GC (NIST)	0.517 ± 0.007 (Poisson) ^(d)
Elemental Carbon (EC) ^(e)	
Thermal oxidation/residue (WHOI)	0.065 ± 0.014 ($n = 3$)
Chemical oxidation/residue (UCI)	0.153 ± 0.004 (Poisson)
Thermal kinetic oxidation/intercept (NIST)	0.038 ± 0.024 (Poisson)
Insoluble Carbon ^(f,g)	
Polar Carbon ^(b,h)	0.56 ($n = 2$)
Extractable Carbon ^(b,i)	0.43 ± 0.02 (Poisson)
Aliphatic Carbon ^(b,h)	0.33 ± 0.04 ($n = 3$)
Aromatic Carbon ^(b,j)	0.024 ± 0.012 (Poisson)
Individual PAH ^(b,h)	
Phenanthrene	0.17 ± 0.08 (Poisson)
Methylphenanthrenes	0.43 ± 0.02 (Poisson)
Fluoranthene	0.0406 ± 0.0096 (Poisson)
Pyrene	0.0434 ± 0.0112 (Poisson)
Benz[<i>a</i>]anthracene	0.0637 ± 0.0051 (Poisson)
Chrysene/Triphenylene	0.0372 ± 0.0043 (Poisson)
Benzofluoranthenes (<i>b,j,k</i>)	0.0413 ± 0.0073 (Poisson)
Benzo[<i>ghi</i>]perylene	0.0553 ± 0.0059 (Poisson)
	0.0842 ± 0.0053 (Poisson)
	0.0864 ± 0.0090 (Poisson)

^(a) The fraction of modern carbon (f_M) is derived from the ¹⁴C/¹²C ratio observed, relative to 0.95 times that of the contemporary (oxalic acid) standard for radiocarbon dating, SRM 4990B. For detailed information on the definition of f_M , taking into account adjustment for the ¹³C/¹²C ratio (for precise work), see references 59 and 60. Note that all f_M values are corrected for ¹⁴C decay to the mid-date of sampling (1977.0) using the physical mean life: 8268 years. Corrections are quite small, with correction factors ranging from 1.00073 to 1.00273. Note that f_M reflects the ¹⁴C content compared to the artifactually (SRM) defined reference state "Modern." At the time of collection of SRM 1649a (1976-1977), the ¹⁴C content of the living biosphere was approximately 1.35 × Modern as a result of atmospheric nuclear testing [22].

^(b) Reference value, which equals the mean value of n measurements (n is number in parentheses). Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [50], is an expanded uncertainty at the 95 % level of confidence, which includes random sources of uncertainty. For those ¹⁴C reference values based strictly on "counting statistics," ($n =$) is replaced by (Poisson), and the expanded uncertainty is taken as 1.96 times the Poisson standard uncertainty. This approach was not taken if replication showed a significant non-Poisson component of variance. (Note that AMS ¹⁴C uncertainties lacking external replication [independent samples] are treated as Poisson uncertainties for the purpose of this certificate [58].)

^(c) The corresponding $\delta^{13}\text{C}$ value is -25.5 ± 0.2 .

^(d) The corresponding $\delta^{13}\text{C}$ value is -25.18 ± 0.12 .

^(e) Information values. Although the ¹⁴C measurements, *per se*, might qualify as reference values having the stated expanded uncertainties, values for ¹⁴C speciation in EC must be considered as information values because of their dependence on the fraction of residual carbon (RC) and lack of consensus on the quantitative link between RC and EC. For the Thermal oxidation/residue method, a weighted mean is given, based on relative weights from the individual values and their expanded Poisson uncertainties: 0.0606 ± 0.0027 , 0.0793 ± 0.0074 , and 0.0663 ± 0.0024 .

^(f) Information value. For a single method, with just one degree of freedom ($n = 2$) and no additional uncertainty information, NIST has chosen to tabulate only the median (mean), as an information value. This is justified because of the extreme variation of the expanded uncertainty (95 % confidence interval) in this case, i.e., greater than a factor of 30 (90 % interval) for normal data, and the minimal impact of the Central Limit Theorem. The alternative, which NIST does not recommend, despite its statistical validity for normal, random data, is to use a coverage factor of 12.7 to construct the (95 %) expanded uncertainty.

^(g) Information value, as is the Insoluble Carbon mass fraction. The individual ¹⁴C (f_M) values and Poisson expanded uncertainties are 0.571 ± 0.008 and 0.543 ± 0.008 .

^(h) Soxhlet extraction/LC/PCGC (WHOI, NIST).

⁽ⁱ⁾ Soxhlet Extraction (EPA).

^(j) Soxhlet Extraction/LC (NIST). Fraction isolated by normal-phase LC representing the fraction in which the majority of the PAHs elute. (See section, "Preparation and Analysis".)

Table 13. Information Values for Particle Size Characteristics for SRM 1649a (see Figure 1). Note that this is an instrument specific measurement of SRM 1649a dispersed in water with a 1h sonication.

Mean Particle Diameter, $d(0.5)^{(a)}$	12.9 μm
Particle Diameter, $d(0.1)^{(b)}$	2.2 μm
Particle Diameter, $d(0.9)^{(c)}$	48.6 μm
Volume Weighted Mean ^(d)	20.8 μm

- ^(a) $d(0.5)$ is a shape parameter for the particle-size distribution indicating the particle size below which 50 % of the volume is present.
- ^(b) $d(0.1)$ is a shape parameter for the particle-size distribution indicating the particle size below which 10 % of the volume is present.
- ^(c) $d(0.9)$ is a shape parameter for the particle-size distribution indicating the particle size below which 90 % of the volume is present.
- ^(d) The volume weighted mean is the particle size in a uniform distribution.

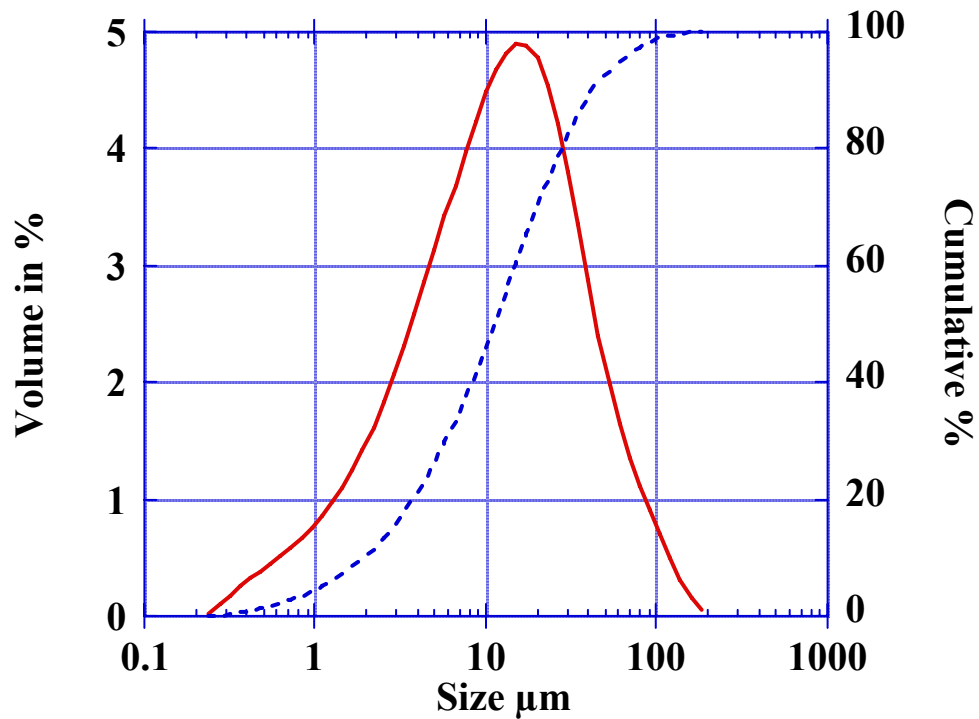


Figure 1. Particle size distribution for SRM 1649a after 1 h. Red solid line represents the volume in % while the blue dashed line represents the cumulative %. See “Particle-Size Information” for additional information.

REFERENCES

- [1] Certificate of Analysis; *Standard Reference Material (SRM) 1649 Urban Dust/Organics*; National Institute of Standards and Technology, Gaithersburg, MD (1982).
- [2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000).
- [3] Wise, S.A.; Schantz, M.M.; Benner, B.A. Jr.; Hays, M.J.; Schiller, S.B.; *Certification of Polycyclic Aromatic Hydrocarbons in a Marine Sediment Standard Reference Material*; *Anal. Chem.*, Vol. 67, pp. 1171–1178 (1995).
- [4] Schantz, M.M.; Benner, B.A. Jr.; Hays, M.J.; Kelly, W.R.; Vocke, R.D. Jr.; Demiralp, R.; Greenberg, R.R.; Schiller, S.B.; Lauenstein, G.G.; Wise, S.A.; *Certification of Standard Reference Material (SRM) 1941a, Organics in Marine Sediment*; *Fresenius J. Anal. Chem.*, Vol. 352, pp. 166–173 (1995).
- [5] Wise, S.A.; Schantz, M.M.; Hays, M.J.; Koster, B.J.; Sharpless, K.S.; Sander, L.C.; Schiller, S.B.; *Certification of Polycyclic Aromatic Hydrocarbons in Mussel Tissue Standard Reference Materials*; *Polycyclic Aromat. Compd.*, Vol. 12, pp. 21–26 (1997).
- [6] Schantz, M.M.; Demiralp, R.; Greenberg, R.R.; Hays, M.J.; Parris, R.M.; Porter, B.J.; Poster, D.L.; Sander, L.C.; Schiller, S.B.; Sharpless, K.S.; Wise, S.A.; *Certification of a Frozen Mussel Tissue Standard Reference Material (SRM 1974a) for Trace Organic Constituents*; *Fresenius J. Anal. Chem.*, Vol. 358, pp. 431–440 (1997).
- [7] Wise, S.A.; Schantz, M.M.; Hays, M.J.; Koster, B.J.; Sharpless, K.S.; Sander, L.C.; Benner, B.A. Jr.; Schiller, S.B.; *Certification of Polycyclic Aromatic Hydrocarbons in Mussel Tissue and Air Particulate Standard Reference Materials*; *Polycyclic Aromat. Compd.*, Vol. 9, pp. 209–216 (1996).
- [8] Wise, S.A.; Chesler, S.N.; Hertz, H.S.; Hilpert, L.R.; May, W.E.; *Chemically-Bonded Aminosilane Stationary Phase for the High Performance Liquid Chromatographic Separation of Polynuclear Aromatic Hydrocarbons*; *Anal. Chem.*, Vol. 49, pp. 2306–2310 (1977).
- [9] May W.E.; Wise, S.A.; *Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbons in Air Particulate Extracts*; *Anal. Chem.*, Vol. 56, pp. 225–232 (1984).
- [10] Wise, S.A.; Benner, B.A.; Byrd, G.D.; Chesler, S.N.; Rebbert, R.E.; Schantz, M.M.; *Determination of Polycyclic Aromatic Hydrocarbons in a Coal Tar Standard Reference Material*; *Anal. Chem.*, Vol. 60, pp. 887–894 (1988).
- [11] Wise, S.A.; Deissler, A.; Sander, L.C.; *Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbon Isomers of Molecular Weight 278 and 302 in Environmental Standard Reference Materials*; *Polycyclic Aromat. Compd.*, Vol. 3, pp. 169–184 (1993).
- [12] Schantz, M.M.; Nichols, J.J.; Wise, S.A.; *Evaluation of Pressurized Fluid Extraction for the Extraction of Environmental Matrix Reference Materials*; *Anal. Chem.*, Vol. 69, pp. 4210–4219 (1997).
- [13] Lippa, K.A.; Schantz, M.M.; *Microhomogeneity Evaluation of Polycyclic Aromatic Hydrocarbons in Particulate Standard Reference Materials*; *Anal. Bioanal. Chem.*, Vol. 387, pp. 2389–2399 (2007).
- [14] Schubert, P.; Schantz, M.M.; Sander, L.C.; Wise, S.A.; *Determination of Polycyclic Aromatic Hydrocarbons with Molecular Weight 300 and 302 in Environmental-Matrix Standard Reference Materials by Gas Chromatography/Mass Spectrometry*; *Anal. Chem.*, Vol. 75, pp. 234–246 (2003).
- [15] Bamford, H.A.; Bezabeh, D.Z.; Schantz, M.M.; Wise, S.A.; Baker, J.E.; *Determination and Comparison of Nitrated-Polycyclic Aromatic Hydrocarbons Measured in Air and Diesel Particulate Reference Materials*; *Chemosphere*, Vol. 50, pp. 575–587 (2003).
- [16] Schantz, M.M.; Parris, R.M.; Kurz, J.; Ballschmiter, K.; Wise, S.A.; *Comparison of Methods for the Gas-Chromatographic Determination of PCB Congeners and Chlorinated Pesticides in Marine Reference Materials*; *Fresenius J. Anal. Chem.*, Vol. 346, pp. 766–778 (1993).
- [17] Schantz, M.M.; Koster, B.J.; Oakley, L.M.; Schiller, S.B.; Wise, S.A.; *Certification of Polychlorinated Biphenyl Congeners and Chlorinated Pesticides in a Whale Blubber Standard Reference Material*; *Anal. Chem.*, Vol. 67, pp. 901–910 (1995).
- [18] Ballschmiter, K.; Zell, M.; *Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography - Composition of Technical Aroclor- and Clophen-PCB Mixtures*; *Fresenius Z. Anal. Chem.*, Vol. 302, pp. 20–31 (1980).
- [19] Schulte E.; Malisch, R.; *Calculation of the Real PCB Content in Environmental Samples. I. Investigation of the Composition of Two Technical PCB Mixtures*; *Fresenius Z. Anal. Chem.*, Vol. 314, pp. 545–551 (1983).
- [20] Poster, D.L.; Schantz, M.M.; Wise, S.A.; Vangel, M.G.; *Analysis of Urban Particulate Standard Reference Materials for the Determination of Chlorinated Organic Contaminants and Additional Chemical and Physical Properties*; *Fresenius J. Anal. Chem.*, Vol. 363, pp. 380–390 (1999).

- [21] Chiu, C.H.; Turle, R.; Poole, G.; Thibert, B.; Brubaker, W.W. Jr; Schantz, M.M.; Wise, S.A.; *Determination of Polychlorinated Dibenzo-p-dioxin and Dibenzofuran Congeners in Air Particulate and Marine Sediment Standard Reference Materials (SRMs)*; Fresenius J. Anal. Chem., Vol. 369, pp. 356–363 (2001).
- [22] Currie, L.A.; Benner, B.A. Jr.; Cachier, H.; Cary, R.; Chow, J.C.; Druffel, E.R.M.; Eglinton, T.I.; Gustafsson, O.; Hartmann, P.C.; Hedges, J.I.; Kessler, J.D.; Kirchstetter, T.W.; Klinedinst, D.B.; Klouda, G.A.; Kohl, S.D.; Marolf, J.V.; Masiello, C.A.; Novakov, T.; Pearson, A.; Prentice, K.M.; Puxbaum, H.; Quinn, J.G.; Reddy, C.M.; Schmid, H.; Slater, J.F.; Wise, S.A.; *A Critical Evaluation of Interlaboratory Data on Elemental and Isotopic Carbon in the Carbonaceous Particle Reference Material, NIST SRM 1649a*; J. Res. Natl. Inst. Stand. Technol., Vol 107, pp 279–298 (2002).
- [23] Masiello, C; Schmidt, M.W.I. (co-chairs); International Steering Committee for the Development of Black Carbon Reference Materials, personal communication (1999); <http://www.du.edu/~dwsmith/bcsteer.html>.
- [24] Birch, M.E.; Cary, R.A.; *Elemental Carbon-Based Method for Monitoring Occupational Exposures to Particulate Diesel Exhaust*; Aerosol Sci. Technol., Vol. 25, pp. 221–241 (1996).
- [25] Klouda, G.A.; Klinedinst, D.D.; Steel, E.B.; Benner, B.A. Jr.; Parish, H.J.; *Exploring a Method to Produce an Urban Dust Particle Filter Standard*; J. Aerosol Sci., Vol. 27, Suppl. 1, pp. S351–S352 (1996).
- [26] Gustafsson, O.; Haghseta, F.; Chan, C.; MacFarlane, J.; Gschwend, P.; *Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability*; Environ. Sci. Technol., Vol. 31, pp. 203–209 (1997).
- [27] Donahue, D.J.; Beck, J.W.; Burr, G.S.; Courtney, C.; Damon, P.E.; Hatheway, A.L.; Hewitt, L.; Jull, A.J.T.; Lange, T.; Lifton, N.; Maddock, R.; McHargue, L.R.; O'Malley, J.M.; Toolin, L.J.; *Status of the NSF-Arizona AMS Laboratory*; Nucl. Instrum. Methods Phys. Res., Vol. B123, p. 5156 (1997).
- [28] Verkouteren, R.M.; Klouda, G.A.; Donahue, D.J.; Jull, A.J.T.; Linick, T.W.; *Preparation of Microgram Samples on Iron Wool for Radiocarbon Analysis via Accelerator Mass Spectrometry: A Closed System Approach*; Nucl. Instrum. Methods Phys. Res., Vol. B29, pp. 41–44 (1987).
- [29] Currie, L.A.; Kunen, S.M.; Voorhees, K.J.; Murphy, R.B.; Koch, W.F.; *Analysis of Carbonaceous Particulates and Characterization of Their Sources by Low-level Radiocarbon Counting and Pyrolysis/Gas Chromatography/Mass Spectrometry*; In: *Proceedings of the Carbonaceous Particles in the Atmosphere*, Lawrence Berkeley Laboratory, University of California (June 1979).
- [30] Currie, L.A.; Gerlach, R.W.; Klouda, G.A.; Ruegg, F.C.; Tompkins G.B.; *Miniature Signals and Miniature Counters: Accuracy Assurance via Microprocessors and Multiparameter Control Techniques*; Radiocarbon, Vol. 25, pp. 553–564 (1983).
- [31] Currie, L.A.; Klouda, G.A.; Voorhees, K.J.; *Atmospheric Carbon: The Importance of Accelerator Mass Spectrometry*; Nucl. Instrum. Methods Phys. Res., Vol. 233 (B5), pp. 371–379 (1984).
- [32] Currie, L.A.; Klinedinst, D.B.; Burch, R.; Feltham, N.; Dorsch, R.; *Authentication and Dating of Biomass Components of Industrial Materials; Links to Sustainable Technology*; Nucl. Instrum. Meth. Phys. Res., Vol. B172, pp. 281–287 (2000).
- [33] Vogel, J.S.; Southon, J.R.; Nelson, D.E.; Brown, T.A.; *Performance of Catalytically Condensed Carbon for Use in Accelerator Mass Spectrometry*; Nucl. Instrum. Methods Phys. Res., Vol. 233 (B5), pp. 289–293, (1984).
- [34] Eglinton, T.I.; Aluwihare, L.I.; Bauer, J.E.; Druffel, E.R.M.; McNichol, A.P.; *Gas Chromatographic Isolation of Individual Compounds from Complex Matrices for Radiocarbon Dating*; Anal. Chem., Vol. 68, pp. 904–912 (1996).
- [35] Currie, L.A.; Klouda, G.A.; Benner, B.A.; Garrity, K.; Eglinton, T.I.; *Isotopic and Molecular Fractionation in Combustion; Three Routes to Molecular Marker Validation, Including Direct Molecular 'Dating' (GC/AMS)*; Atm. Environ., Vol. 33, pp. 2789–2806 (1999).
- [36] Currie, L.A.; Eglinton, T.I.; Benner, B.A. Jr.; Pearson, A.; *Radiocarbon Dating of Individual Chemical Compounds in Atmospheric Aerosol: First Results Comparing Direct Isotopic and Multivariate Statistical Apportionment of Specific Polycyclic Aromatic Hydrocarbons*; Nucl. Instrum. Methods Phys. Res., Vol. B123, pp. 475–486 (1997).
- [37] Reddy, C.M.; Pearson, A.; Eglinton, T.I.; Xu, L.; McNichol, A.P.; Benner, B.A. Jr.; Wise, S.A.; Klouda, G.A.; Currie, L.A.; Eglinton, T.I.; *Radiocarbon as a Tool to Apportion Sources of Polycyclic Aromatic Hydrocarbons and Black Carbon in Environmental Samples*; Environ. Sci. Technol., Vol. 36, pp. 1774–1782 (2002).
- [38] Masiello, C.A.; Druffel, E.R.M.; Currie, L.A.; *Radiocarbon Measurements of Black Carbon in Aerosols and Ocean Sediments*; Geochim. Cosmochim. Acta, Vol. 66, pp. 1025–1036 (2002).
- [39] Currie, L.A.; Kessler, J.D.; *On the Isolation of Elemental Carbon (EC) for Micro-Molar ¹⁴C Accelerator Mass Spectrometry: Development of a Hybrid Reference Material for ¹⁴C-EC Accuracy Assurance, and a Critical Evaluation of the Thermal Optical Kinetic (TOK) EC Isolation Procedure*; Atmos. Chem. Phys., Vol. 5, pp. 2833–2845 (2005).
- [40] Gundel, L.A.; Dod, R.L.; Rosen, H.; Novakov, T.; *The Relationship Between Optical Attenuation and Black Carbon Concentration for Ambient and Source Particles*; Sci. Total Environ., Vol. 36, pp. 197–202 (1984).

- [41] Cachier, H.; Bremond, M.P.; Buat-Ménard, P.; *Determination of Atmospheric Soot Carbon with a Simple Thermal Method*; *Tellus*, Vol. 41B, pp. 379–390 (1989).
- [42] Chow, J.C.; Watson, J.G.; Prichett, L.C.; Pierson, W.R.; Frazier, C.A.; Purcell, R.G.; *The DRI Thermal/Optical Reflectance Carbon Analysis System. Description, Evaluation and Application in the U.S. Air Quality Studies*; *Atmospheric Environ.*, Vol. 27A, pp. 1185–1201 (1993).
- [43] Puxbaum, H.; *Thermo-Gasanalysator zur Charakterisierung von Kohlenstoff- und Schwefelverbindungen in luftgetragenen Stäuben*; *Fresenius Zeitschrift für Analytische Chemie*, Vol. 298, pp. 250–259 (1979).
- [44] Wise, S.A.; Sander, L.C.; Schantz, M.M.; Hays, M.J.; Benner, B.A.; *Recertification of Standard Reference Material (SRM) 1649, Urban Dust, for the Determination of Polycyclic Aromatic Hydrocarbons (PAHs)*; *Polycyclic Aromat. Compd.*, Vol. 13, pp. 419–456 (2000).
- [45] Lewtas, J.; Claxton, L.D.; Rosenkranz, H.S.; Schuetzle, D.; Shelby, M.; Matsushita, H.; Wurgler, F.E.; Zimmermann, F.K.; Lofroth, G.; May, W.E.; Krewski, D.; Matsushima, T.; Ohnishi, Y.; Gopalan, H.N.G.; Sarin, R.; Becking, G.C.; *Design and Implementation of a Collaborative Study of the Mutagenicity of Complex Mixtures in Salmonella Typhimurium*; *Mutat. Res.*, Vol. 276, pp. 3–9 (1992).
- [46] Claxton, L.D.; Douglas, G.; Krewski, D.; Lewtas, J.; Matsushita, H.; Rosenkranz, H.; *Overview, Conclusions, and Recommendations of the IPCS Collaborative Study on Complex Mixtures*; *Mutat. Res.*, Vol. 276, pp. 61–80 (1992).
- [47] Fernandez, P.; Bayona, J.M.; *Use of Offline Gel Permeation Chromatography-Normal-Phase Liquid Chromatography for the Determination of Polycyclic Aromatic Compounds in Environmental Samples and Standard Reference Materials (Air Particulate Matter and Marine Sediment)*; *J. Chromatogr.*, Vol. 625, pp. 141–149 (1992).
- [48] Durant, J.L.; Lafleur, A.L.; Plummer, E.F.; Taghizadeh, K.; Busby, W.F.; Thilly, W.G.; *Human B-Lymphoblastoid Cell Mutagens in Urban Airborne Particles*; *Environ. Sci. Technol.*, Vol. 32, pp. 1894–1906 (1998).
- [49] Albinet, A.; Leoz-Garziandia, E.; Budzinski, H.; Villenave, E.; *Simultaneous Analysis of Oxygenated and Nitrated Polycyclic Aromatic Hydrocarbons on Standard Reference Material 1649a (Urban Dust) and on Natural Ambient Air Samples by Gas Chromatography-Mass Spectrometry with Negative Ion Chemical Ionization*; *J. Chromatogr. A*, Vol. 1121, pp. 106–113 (2006).
- [50] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.
- [51] Ruhkin, A.L.; Vangel, M.G.; *Estimation of a Common Mean and Weighted Means Statistics*; *J. Am. Statist. Assoc.*, Vol. 93, pp. 303–308 (1998).
- [52] Paule, R.C.; Mandel, J.; *Consensus Values and Weighting Factors*; *J. Research NBS*, Vol. 87, pp. 377–385 (1982).
- [53] Larsen, R.K. III; Schantz, M.M.; Wise, S.A.; *Determination of Levoglucosan in Particulate Matter Reference Materials*; *Aerosol Sci. Technol.*, Vol. 40, pp. 781–787 (2006).
- [54] Van den Berg, M.; Birnbaum, L.; Bosveld, A.T.C.; Brunström, B.; Cook, P.; Feeley, M.; Giesy, J.P.; Hanberg, A.; Hasegawa, R.; Kennedy, S.W.; Kubiak, T.; Larsen, J.C.; van Leeuwen, F.X.R.; Liem, A.K.D.; Nolt, C.; Peterson, R.E.; Poelinger, L.; Safe, S.; Schrenk, D.; Tillitt, D.; Tysklind, M.; Younes, M.; Waern, F.; Zacharewski, T.; *Toxic Equivalency Factors (TEFs) for PCBs, PCDDs, PCDFs for Humans and Wildlife*; *Environ. Health Perspect.*, Vol. 106, pp. 775–792 (1998).
- [55] Tukey, J. W.; *Exploratory Data Analysis*, Addison-Wesley, Reading, MA (1977).
- [56] Stuiver, M.; Polach, H.; *Reporting of ¹⁴C Data*; *Radiocarbon*, Vol. 19, pp. 355–363 (1977).
- [57] Hut, G.; *Consultants Group Meeting on Stable Isotope Reference Samples for Geochemical and Hydrological Investigations*; International Atomic Energy Agency, Vienna, Austria, p. 42 (1987).
- [58] Currie, L.A.; *Optimal Estimation of Uncertainty in Reporting of Accelerator Mass Spectrometry Results*; *Nucl. Instr. Meth. Phys. Res.*, Vol. 92, pp. 188–93 (1994).

Certificate Revision History: 06 December 2007 (Added certified and reference values for molecular mass 302 PAHs and for nitro-substituted PAHs; extended expiration date); 31 January 2001 (Added certified value for total carbon, reference and information values for chemical and isotopic (¹⁴C) carbon composition, and reference values for 17 polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners; removed reference value for total organic carbon.); 19 November 1998 (original report date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

APPENDIX A

The laboratories and analysts listed below participated in the interlaboratory comparison exercise for the determination of polychlorinated dibenzo-*p*-dioxins and dibenzofurans in SRM 1649a (Table 8).

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

APPENDIX B

The laboratories and analysts listed below contributed to the results presented for isotopic carbon and particulate composition for SRM 1649a (Tables 11 and 12).

Atmospheric Aerosol Research, Lawrence Berkeley National Laboratory (LBNL) (Berkeley, CA, USA):
T. Novakov and T. Kirchstetter
Centre des Faibles Radioactivites (LSCE), CNRS-CEA (Gif sur Yvette, France): H. Cachier
Department of Earth System Science, University of California (UCI) (Irvine, CA, USA): C.A. Masiello and
E.R.M. Druffel
Department of Marine Chemistry and Geochemistry, Woods Hole Oceanographic Institution (WHOI) (Woods Hole,
MA, USA): C.M. Reddy, T.I. Eglinton, and A. Pearson
Energy and Environmental Engineering Center, Desert Research Institute (DRI) (Reno, NV, USA): S.D. Kohl and
J.C. Chow
Environmental Research Institute, Stockholm University (SU) (Stockholm, Sweden): O. Gustafsson
Graduate School of Oceanography, University of Rhode Island (URI) (Narragansett, RI, USA): P.C. Hartmann and
J.G. Quinn
Institute for Analytical Chemistry, Vienna University of Technology (TUW) (Vienna, Austria): H. Puxbaum and
H. Schmid
National Institute of Standards and Technology (NIST) (Gaithersburg, MD, USA): B.A. Benner, Jr., L.A. Currie,
J.D. Kessler, D.B. Klinedinst, G.A. Klouda, J.V. Marolf, A.E. Sheffield, J.F. Slater, and S.A. Wise
School of Oceanography, University of Washington (UW) (Seattle, WA): K.M. Prentice and J.I. Hedges
Sunset Laboratories, Inc. (SLI) (Forest Grove, OR, USA): R. Cary
U.S. Environmental Protection Agency (EPA), National Exposure Laboratory (Research Triangle Park, NC, USA):
R.B. Zweidinger

Accelerator measurements were performed at the National Science Foundation Accelerator Mass Spectrometry (AMS) Facility, University of Arizona (UA) (Tucson, AZ); Lawrence Livermore National Laboratory (LLNL) (Livermore, CA) and the National Ocean Sciences AMS Facility (NOSAMS), Woods Hole Oceanographic Institution (WHOI) (Woods Hole, MA).