



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1649b

Urban Dust

This Standard Reference Material (SRM) is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), nitro-substituted PAHs (nitro-PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and inorganic constituents in atmospheric particulate material and similar matrices. In addition to certified values, reference or information values are also provided for decabromodiphenyl ether and selected toxaphene congeners, polychlorinated dibenzo-*p*-dioxin and dibenzofuran congeners, and inorganic constituents. Information values are provided for hydrocarbons, hopanes, steranes, ketones, and particle-size characteristics. All of the constituents for which certified, reference, and information values are provided in SRM 1649b are naturally present in the particulate material. SRM 1649b was prepared from the same particulate material that was issued in 1982 as SRM 1649 [1] and re-issued in 1999 as SRM 1649a [2]; however, the bulk material was sieved to a smaller particle size fraction. A unit of SRM 1649b consists of a bottle containing 2 g of particulate material.

Certified Mass Fraction Values: Certified values for PAHs, nitro-PAHs, PCB congeners, chlorinated pesticides, and inorganic constituents are provided in Tables 1 to 5, respectively. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [3]. The certified values for the PAHs, nitro-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. The certified values for cadmium, lead, and mercury are based on measurements performed at NIST using a single primary method.

Reference Mass Fraction Values: Reference values are provided for additional PAHs, nitro-PAHs, PCB congeners, and chlorinated pesticides (includes toxaphene congeners) in Tables 6 to 9, respectively. In Table 6 and 7, the reference values for some PAHs and nitro-PAHs, respectively, are listed more than once depending on the extraction conditions that are used (see "Preparation and Analysis"). Table 10 contains a reference value for decabromodiphenyl ether. Reference values are provided in Table 11 for selected dibenzo-*p*-dioxin and dibenzofuran congeners and total, tetra-, penta-, hexa-, and hepta-substituted congeners of polychlorinated dibenzo-*p*-dioxin and dibenzofuran. Table 12 contains additional inorganic constituents. Reference values are noncertified values that are the best estimates of the true values; 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 [3].

Information Values: An information value for aluminum is provided in Table 13, and information values for selected aliphatic hydrocarbons, hopanes, steranes, and ketones are provided in Table 14. An information value is considered to be a value that will be of interest 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 [3]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1649b** is valid, within the measurement uncertainties specified, until **31 July 2030**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Gaithersburg, MD 20899
Certificate Issue Date: 30 August 2016
Certificate Revision History on Last Page

Coordination of the technical measurements leading to the updated certification was under the direction of M.M. Schantz, B.A. Benner, Jr., S.E. Long, L.C. Sander, and S.A. Wise of the NIST Chemical Sciences Division.

Analytical measurements for the certification of SRM 1649b were performed by J.M. Keller, J.R. Kucklick, S.E. Long, K.E. Murphy, B.J. Porter, S.A. Rabb, M.M. Schantz, S.S. Vander Pol, R. Zeisler, and L.J. Wood of the NIST Chemical Sciences Division. Analytical measurements were also performed by G. Poole and C. Chiu of Environment Canada, Environmental Technology Centre, Analysis and Air Quality Division (Ottawa, Ontario, Canada). Analytical measurements for the aliphatic hydrocarbons, hopanes, steranes, and ketones include results from an interlaboratory comparison study conducted as part of the Intercomparison Program for Organic Speciation in PM_{2.5} Air Particulate Matter [4,5].

Collection and preparation of the material for SRM 1649b were supported in part by the U.S. Environmental Protection Agency, Office of Health Research (Research Triangle Park, NC).

Consultation on the experiment design and evaluation of data were provided by N.A. Heckert, S.D. Leigh, and A.L. Pintar of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This readily aerosolized material is a naturally occurring particulate material and may contain constituents of known and unknown toxicities and mutagenicities; therefore, safety precautions should be followed and care should be exercised during its handling and use. A significant portion of the material is in the range of inhalable airborne particulate matter based on its particle size. For health and safety information, see the Safety Data Sheet (SDS).

Storage: Store SRM 1649b in its original amber glass bottle at temperatures less than 30 °C and away from direct sunlight.

Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed thoroughly. The evaluation of the homogeneity of SRM 1649b, at small sample sizes for PAHs, is described in "Homogeneity Assessment for PAHs" and in further detail in reference 6. Based on this work, a minimal sample size for PAH determinations can be assessed at 150 mg. The mass fractions of constituents in SRM 1649b are reported on a dry-mass basis. 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 convert the mass fraction to a dry-mass basis.

PREPARATION AND ANALYSIS⁽¹⁾

Sample Collection and Preparation: This SRM was prepared from atmospheric particulate material collected in the Washington, DC area in 1976 and 1977 using a baghouse specially designed for the purpose. The particulate material was collected over a period greater than 12 months and 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 63 µm (230 mesh) sieve to remove bag fibers and other extraneous materials. The sieved material was then thoroughly mixed in a V-blender and bottled. SRM 1649b is prepared from the same bulk material as SRM 1649 and SRM 1649a, but it has been sieved to a smaller particle size resulting in some differences in the concentrations of the target analytes.

A considerable amount of information on the characterization of SRM 1649 and SRM 1649a has been published during the duration of its availability from 1982 to 2008. Summaries of some of the studies reporting characterization of this air particulate matter material are provided in Wise et al. [7,8], Poster et al. [9], and Currie et al. [10]. Therefore, some values previously reported on the Certificates of Analysis for SRM 1649 and SRM 1649a may not be reported in this Certificate of Analysis.

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

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1649b are reported on a dry-mass basis. The material “as received” contains residual moisture. The consensus mean moisture content in SRM 1649b was determined by measuring the mass loss after forced air drying at 95 °C for one hour and desiccation at 25 °C to 30 °C for 35 d. The moisture content in SRM 1649b at the time of the certification analyses was 2.35 % ± 0.07 % at the 95 % confidence level.

Polycyclic Aromatic Hydrocarbons (PAHs)

The general approach used for the determination of PAHs in SRM 1649b was similar to that reported for the certification of several environmental-matrix SRMs [7]. This approach consisted of Soxhlet extraction or pressurized-fluid extraction (PFE) followed by analysis of the extracts using gas chromatography/mass spectrometry (GC/MS) on columns of differing selectivity. Multiple sets of GC/MS results, designated as GC/MS (Ia), GC/MS (Ib), GC/MS (Ic), GC/MS (IIa), GC/MS (IIb), GC/MS (III), and GC/MS (IV) were used in the certification process. Throughout this Certificate of Analysis, GC/MS in the electron ionization mode (EI) is noted as GC/MS.

GC/MS (I): For GC/MS (I) analyses, duplicate subsamples of between 150 mg and 250 mg from 10 bottles were Soxhlet extracted for 20 h with dichloromethane. The concentrated extract was passed through an aminopropylsilane solid-phase extraction (SPE) cartridge and eluted with 20 % (volume fraction) dichloromethane in hexane. The PAH fraction was then analyzed by GC/MS using three columns with differing selectivity: (1) a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-17MS, Agilent Technologies, Wilmington, DE), designated as GC/MS (Ia); (2) a 0.25 mm i.d. × 60 m fused silica capillary column with a proprietary relatively nonpolar phase (0.25 µm film thickness; DB-XLB, Agilent Technologies), designated as GC/MS (Ib); and (3) a 0.25 mm i.d. × 20 m dimethyl 50 % liquid crystal polysiloxane phase (0.25 µm film thickness; LC-50, J&K Environmental, Milton, Ontario, Canada), designated as GC/MS (Ic).

GC/MS (II): The GC/MS (II) analyses were conducted on test portions of 180 mg to 300 mg from each of three bottles extracted using PFE with dichloromethane at 100 °C and 13.8 MPa (2000 psi), designated as GC/MS (IIa) and test portions of 180 mg to 300 mg from three bottles extracted using PFE with dichloromethane at 200 °C and 13.8 MPa, designated as GC/MS (IIb). The concentrated extracts were passed through a silica SPE cartridge and eluted with 10 % (volume fraction) dichloromethane in hexane. The PAH fraction was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a proprietary relatively nonpolar phase (0.25 µm film thickness; DB-XLB).

GC/MS (III): For GC/MS (III), test portions of approximately 500 mg from each of six bottles were extracted using PFE with dichloromethane at 100 °C and 13.8 MPa. The samples were cleaned-up using automated SPE with 1.8 g alumina columns and eluting with 9 mL of 35 % (volume fraction) dichloromethane in hexane. The samples were analyzed by GC/MS with a 0.25 mm i.d. × 60 m fused silica capillary column containing a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-17, Agilent Technologies).

GC/MS (IV): For GC/MS (IV), test portions of approximately 100 mg from each of three bottles were extracted using PFE with toluene at 100 °C and 13.8 MPa, 150 °C and 13.8 MPa, 200 °C and 13.8 MPa, and 200 °C and 20.7 MPa. The concentrated extract was passed through an aminopropylsilane SPE cartridge and eluted with 20 % (volume fraction) dichloromethane in hexane. The PAH fraction was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a proprietary relatively nonpolar phase (0.25 µm film thickness; DB-XLB, Agilent Technologies).

Internal Standards: For the GC/MS measurements described below in this section, 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 1649b was assessed by analyzing duplicate test portions of 150 mg to 250 mg from 10 randomly selected bottles. Samples were extracted, processed, and analyzed as described above for the GC/MS (I). No statistically significant differences between bottles at the 95 % confidence level were observed for the PAHs at a 150 mg to 250 mg sample size.

Nitro-Substituted Polycyclic Aromatic Hydrocarbons (Nitro-PAHs)

SRM 1649b was analyzed at NIST for the determination of nitro-PAHs. The general procedure for determination of nitro-PAHs at NIST has been reported previously [8] and is described briefly below. Duplicate test portions of 150 mg to 250 mg from 10 bottles were Soxhlet extracted for 20 h with dichloromethane. The concentrated extract was passed through an aminopropylsilane SPE cartridge and eluted with 20 % (volume fraction) 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 % (volume fraction) dichloromethane in hexane to isolate the nitro-PAH fraction as described previously [11]. 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. × 60 m fused silica capillary column containing a 50 % phenyl-substituted methylpolysiloxane stationary phase (0.25 µm film thickness).

In addition, test portions of approximately 100 mg from each of three bottles were extracted using PFE with toluene at 100 °C and 13.8 MPa, 150 °C and 13.8 MPa, 200 °C and 13.8 MPa, and 200 °C and 20.7 MPa. The nitro-PAHs were isolated from the co-extracted matrix using the SPE and LC procedures described above and analyzed by 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).

Internal Standards: For all methods, selected perdeuterated nitro-PAHs were added to the air particulate matter prior to extraction for use as internal standards for quantification purposes.

Value Assignment for PAHs and Nitro-PAHs

The current value assignments of PAHs and nitro-PAHs in SRM 1649b are based on the measurements from when SRM 1649b was issued in 2009 and recent additional measurements using different extraction conditions. Prior studies on solvent extraction of air particulate matter for determination of PAHs demonstrated that Soxhlet extraction and PFE at 100 °C provided similar results [12]. Recent studies on solvent extraction of air particulate matter [13,14] have shown that using PFE at 150 °C or 200 °C recovers higher quantities than using PFE at 100 °C for some PAHs and nitro-PAHs. As a result of these studies, value assignments for PAHs and nitro-PAHs in SRM 1649b are based on measurements using Soxhlet extraction and using PFE at 100 °C, 150 °C, and 200 °C. In cases where it was determined that the quantities of the individual PAHs and nitro-PAHs did not change with the extraction method or temperature, the measurements were combined and the resulting values are denoted as certified values. When different results are obtained using PFE at 100 °C, 150 °C, or 200 °C, the values are reported at all three temperatures, and they are denoted as reference values. These reference values should be considered as “method dependent” values because they are dependent on the extraction method and temperature.

PCB Congeners and Chlorinated Pesticides

SRM 1649b was analyzed for selected PCB congeners and chlorinated pesticides using GC/MS. This same approach has been used previously for the certification of PCBs and chlorinated pesticides in environmental-matrix SRMs [7]. Four sets of GC/MS results, designated as GC/MS (Va), GC/MS (Vb), GC/MS (VIa), and GC/MS (VIb) were used in the certification process. For GC/MS (V) analyses, duplicate test portions of 150 mg to 250 mg from 10 bottles were Soxhlet extracted for 20 h with dichloromethane. The concentrated extract was passed through an aminopropylsilane SPE cartridge and eluted with 20 % (volume fraction) dichloromethane in hexane. The chlorinated compounds were then analyzed by GC/MS and in the NICI mode (GC/NICI-MS). For the GC/MS analysis, designated as GC/MS (Va), a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) was used while for the GC/NICI-MS, designated as GC/MS (Vb), a 0.25 mm i.d. × 60 m fused silica capillary column with a proprietary relatively nonpolar phase (0.25 µm film thickness; DB-XLB) was used.

GC/MS (VI): For GC/MS (VI), test portions from six bottles of approximately 500 mg were extracted using PFE with dichloromethane at 100 °C and 13.8 MPa. The samples were cleaned-up using automated SPE with alumina cartridges and then with acidified silica cartridges. The first fraction from the silica cartridges was concentrated and then further cleaned-up using size-exclusion chromatography. The samples were analyzed by GC/MS with a 0.18 mm i.d. × 30 m fused silica capillary column containing a 5 % phenyl-substituted methylpolysiloxane phase (0.18 µm film thickness), designated as GC/MS (VIa) and by GC/NICI-MS with a 0.18 mm i.d. × 30 m fused silica capillary column with a proprietary relatively nonpolar phase (0.18 µm film thickness; DB-XLB), designated as GC/MS (VIb).

For the GC/MS and GC/NICI-MS analyses, two PCB congeners that are not significantly present in the air particulate extract (PCB 103 and PCB 198 [15,16]) and selected ¹³C-labeled PCB congeners and pesticides were added to the air particulate material prior to extraction for use as internal standards for quantification purposes.

Decabromodiphenyl Ether

Test portions of approximately 500 mg from each of six bottles were extracted using PFE with dichloromethane at 100 °C and 13.8 MPa. The samples were cleaned-up using automated SPE with 1.8 g alumina columns and eluting with 9 mL of 35 % (volume fraction) dichloromethane in hexane. Following concentration, the analytes of interest were further isolated using an acidified silica SPE column. Two fractions were collected: (1) containing the PCBs, pesticides, and polybrominated diphenyl ethers and (2) containing the hexabromocyclododecanes. The first fraction was analyzed using GC/NICI-MS with a 0.18 mm i.d. × 10 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.18 µm film thickness).

Polychlorinated Dibenzo-*p*-Dioxins and Dibenzofurans

Value assignment of the mass fractions for 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 is based on the analysis of four subsamples (between 100 mg and 150 mg) of SRM 1649b by Environment Canada. Samples were Soxhlet extracted overnight with toluene. Extracts were concentrated and exchanged to hexane, then passed through a modified silica column followed by a basic alumina column. The dioxin/furan fraction was analyzed by using GC with high-resolution mass spectrometric detection (GC-HRMS) and 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). The 2,3,7,8-tetrachlorodibenzofuran was quantified using a 50 % cyanopropylphenyl-substituted methylpolysiloxane (DB-225) capillary column.

Inorganic Constituents

The mass fraction value assignments of cadmium and lead were performed using isotope dilution-inductively coupled plasma mass spectrometry (ID-ICP-MS). Two 50 mg samples taken from each of four bottles of SRM 1649b were spiked with ¹¹¹Cd and ²⁰⁶Pb and subjected to microwave digestion using 1 g of concentrated hydrofluoric acid and 9 g of concentrated nitric acid. Samples were heated to near-dryness and redissolved in 2 % (volume fraction) nitric acid prior to ICP-MS analysis.

The mass fraction value assignment of mercury is based on cold-vapor ICP-MS. Single subsamples (160 mg to 240 mg) were taken from each of eight bottles and spiked with ²⁰¹Hg followed by microwave digestion. Mercury measurements were made using cold-vapor mercury generation coupled with ICP-MS isotope ratio measurements.

The mass fractions for the remainder of the elements are based on analyses using inductively coupled plasma optical emission spectrometry (ICP-OES). One 50 mg sample was taken from each of six bottles of SRM 1649b for analysis. The samples were microwave digested using 1 mL of concentrated hydrofluoric acid and 9 mL of concentrated nitric acid. Following dilution with water, the samples were analyzed by ICP-OES using optimized parameters for each element.

Particle-Size Information

Particle size distribution measurements for SRM 1649b 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 % (mass fraction) of SRM 1649b in distilled water with 0.001 % Triton (volume fraction), was prepared by ultrasonication 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 procedures described in reference 17. The results obtained are shown in Figure 1.

Certified Mass Fraction Values for Selected PAHs: The certified mass fraction values in Table 1 are for selected PAHs and are reported on a dry-mass basis.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for Selected PAHs in SRM 1649b

| | Mass Fractions ^(a) (mg/kg) |
|--|--|
| 4H-Cyclopenta[<i>def</i>]phenanthrene ^(b,c,d,e,f) | 0.255 ± 0.023 |
| Pyrene ^(b,c,d,e,f,g) | 4.98 ± 0.14 |
| 1-Methylfluoranthene ^(b,c,e,g) | 0.114 ± 0.015 |
| 3-Methylfluoranthene ^(b,c,e,g) | 0.328 ± 0.031 |
| 1-Methylpyrene ^(b,c,e,g) | 0.363 ± 0.005 |
| Retene ^(b,c,e,g) | 0.238 ± 0.011 |
| Benzo[<i>c</i>]phenanthrene ^(b,c,d,e,f,g) | 0.460 ± 0.013 |
| Chrysene ^(b,c,d,e,f,g) | 3.045 ± 0.028 |
| Triphenylene ^(f,g) | 1.324 ± 0.004 |
| Benzo[<i>b</i>]fluoranthene ^(b,d,f) | 6.18 ± 0.18 |
| Benzo[<i>j</i>]fluoranthene ^(b,d,f) | 1.725 ± 0.043 |
| Benzo[<i>k</i>]fluoranthene ^(b,c,d,e,f,g) | 1.702 ± 0.049 |
| Benzo[<i>e</i>]pyrene ^(b,c,d,e,f,g) | 2.974 ± 0.053 |
| Perylene ^(b,c,d,e,f,g) | 0.614 ± 0.011 |
| Indeno[1,2,3- <i>cd</i>]pyrene ^(b,c,d,e,f,g) | 2.89 ± 0.16 |
| Dibenz[<i>a,j</i>]anthracene ^(b,c,e,f,g) | 0.305 ± 0.027 |
| Benzo[<i>b</i>]chrysene ^(b,c,d,e,f,g) | 0.339 ± 0.043 |
| Picene ^(b,c,d,e,f,g) | 0.399 ± 0.022 |
| Dibenzo[<i>b,k</i>]fluoranthene ^(b,c,e,f,g) | 0.670 ± 0.053 |
| Dibenzo[<i>a,e</i>]pyrene ^(b,c,e,f,g) | 0.567 ± 0.025 |

^(a) The certified values are weighted means of the mass fractions from multiple analytical methods [18]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [20] following the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the PAHs listed in Table 1. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram on a dry-mass basis).

^(b) GC/MS (Ia) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

^(c) GC/MS (Ib) on a proprietary relatively non-polar phase using same extracts as GC/MS (Ia).

^(d) GC/MS (Ic) on a dimethyl 50 % liquid crystal polysiloxane phase using same extracts as GC/MS (Ia).

^(e) GC/MS (IIa and IIb) on a proprietary relatively non-polar phase after PFE at 100 °C and at 200 °C with dichloromethane at 13.8 MPa.

^(f) GC/MS (III) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE at 100 °C with dichloromethane at 13.8 MPa.

^(g) GC/MS (IV) on a proprietary relatively non-polar phase after PFE with toluene at 100 °C and 13.8 MPa, 150 °C and 13.8 MPa, 200 °C and 13.8 MPa, and 200 °C and 20.7 MPa.

Certified Mass Fraction Values for Selected Nitro-PAHs: The certified values in Table 2 are for selected Nitro-PAHs and are reported on a dry-mass basis.

Table 2. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Nitro-PAHs in SRM 1649b^(a)

| | Mass Fractions ^(b) (µg/kg) | | |
|-----------------------------------|--|---|------|
| 3-Nitrophenanthrene | 21.8 | ± | 1.2 |
| 9-Nitrophenanthrene | 1.77 | ± | 0.07 |
| 2-Nitrofluoranthene | 304 | ± | 25 |
| 3-Nitrofluoranthene | 4.67 | ± | 0.35 |
| 1-Nitropyrene | 74.8 | ± | 1.9 |
| 2-Nitropyrene | 10.9 | ± | 0.1 |
| 7-Nitrobenz[<i>a</i>]anthracene | 24.4 | ± | 0.4 |
| 6-Nitrochrysene | 3.60 | ± | 0.21 |

^(a) The methods used were GC/NICI MS on a 50 % phenyl substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane and GC/NICI MS on a 50 % phenyl substituted methylpolysiloxane phase after PFE with toluene at 100 °C and 13.8 MPa, 150 °C and 13.8 MPa, 200 °C and 13.8 MPa, and 200 °C and 20.7 MPa.

^(b) The certified values are weighted means of the mass fractions from multiple analytical methods [18]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [20] following the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the Nitro-PAHs listed in Table 2. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

Certified Mass Fraction Values for Selected PCB Congeners: The certified values in Table 3 are reported on a dry-mass basis. The certified value are weighted means of the mass fractions from multiple analytical methods [18]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [20] following the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the PCB congeners listed in Table 3. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

Table 3. Certified Mass Fraction Values (Dry-Mass Basis) for Selected PCB Congeners in SRM 1649b^(a)

| | Mass Fractions (µg/kg) | | |
|---|---------------------------|---|-----|
| PCB 49 (2,2',4,5'-Tetrachlorobiphenyl) ^(b,c) | 9.1 | ± | 1.7 |
| PCB 52 (2,2',5,5'-Tetrachlorobiphenyl) ^(b,c) | 24.3 | ± | 6.1 |
| PCB 101 (2,2',4,5,5'-Pentachlorobiphenyl) ^(b,c,d) | 56.4 | ± | 5.2 |
| PCB 105 (2,3,3',4,4'-Pentachlorobiphenyl) ^(b,c,d) | 10.0 | ± | 1.0 |
| PCB 110 (2,3,3',4',6-Pentachlorobiphenyl) ^(b,c) | 33.7 | ± | 5.0 |
| PCB 149 (2,2',3,4',5',6-Hexachlorobiphenyl) ^(b,d) | 79.4 | ± | 3.2 |
| PCB 151 (2,2',3,5,5',6-Hexachlorobiphenyl) ^(c,d) | 33.4 | ± | 3.6 |
| PCB 153 (2,2',4,4',5,5'-Hexachlorobiphenyl) ^(b,d) | 76.6 | ± | 0.4 |
| PCB 163 (2,3,3',4',5,6-Hexachlorobiphenyl) ^(c,d) | 22.2 | ± | 0.3 |
| PCB 183 (2,2',3,4,4',5',6-Heptachlorobiphenyl) ^(c,d) | 17.2 | ± | 1.5 |
| PCB 187 (2,2',3,4',5,5',6-Heptachlorobiphenyl) ^(b,c,d) | 39.3 | ± | 2.8 |
| PCB 194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl) ^(c,d) | 28.5 | ± | 2.8 |
| PCB 206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) ^(c,d) | 17.0 | ± | 2.1 |

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] 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) GC/MS (Va) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

^(c) GC/MS (VIa) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE with dichloromethane.

^(d) GC/MS (Vb) on a proprietary relatively non-polar phase using same extracts as GC/MS (Va).

Certified Mass Fraction Values for Selected Chlorinated Pesticides: The certified values in Table 4 are reported on a dry-mass basis. The certified value are weighted means of the mass fractions from multiple analytical methods [18]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [20] following the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the chlorinated pesticides listed in Table 4. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

Table 4. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Chlorinated Pesticides in SRM 1649b

| | Mass Fractions ($\mu\text{g/kg}$) | | |
|---|--|-------|-----|
| <i>trans</i> -Chlordane (γ -Chlordane) ^(a,b) | 51.9 | \pm | 9.0 |
| <i>trans</i> -Nonachlor ^(a,b) | 33.7 | \pm | 6.1 |
| 4,4'-DDE ^(c,d) | 50.7 | \pm | 0.4 |
| 4,4'-DDD ^(c,d) | 37.7 | \pm | 3.1 |

^(a) GC/MS (Vb) on a relatively non-polar phase after Soxhlet extraction with dichloromethane.

^(b) GC/MS (VIa) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE with dichloromethane.

^(c) GC/MS (Va) on a 50 % phenyl-substituted methylpolysiloxane phase using the same extracts as GC/MS (Vb).

^(d) GC/MS (VIb) on a relatively non-polar phase using the same extracts as GC/MS (VIa).

Certified Mass Fraction Values for Selected Inorganic Constituents: The certified values in Table 5 are reported on a dry-mass based on the moisture content at the time of the certification. The certified value is an unweighted mean of the results from a definitive technique. The uncertainty listed with each value is an expanded uncertainty, with coverage factor, k , determined by the Student's t -distribution with the appropriate degrees of freedom and a 95 % confidence level for each analyte by following the ISO/JCGM Guides [21,22]. The coverage factor is included in the table for each analyte. The measurand is the total mass fraction for the inorganic constituents in Table 5. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram on a dry-mass basis).

Table 5. Certified Mass Fraction Values (Dry-Mass Basis) for Selected Inorganic Constituents in SRM 1649b

| | Mass Fraction (mg/kg) | | | k |
|------------------------|-------------------------------------|-------|------|------|
| Cadmium ^(a) | 26.10 | \pm | 0.28 | 3.18 |
| Lead ^(a) | 12864 | \pm | 62 | 3.18 |
| Mercury ^(b) | 1.80 | \pm | 0.04 | 2.36 |

^(a) ID-ICP-MS

^(b) Cold-vapor ID-ICP-MS

Reference Mass Fraction Values for Selected PAHs: The reference values in Table 6 are reported on a dry-mass basis. Reference values for PAHs are provided based on extraction method and conditions as previously described.

Table 6. Reference Mass Fraction Values (Dry-Mass Basis) for Selected PAHs in SRM 1649b based on Extraction Method and Conditions

| Extraction Conditions | Mass Fractions ^(a) (mg/kg) | | |
|--|--|---|----------------------|
| Soxhlet extraction or PFE using temperatures between 100 °C and 200 °C | | | |
| 1-Methylnaphthalene ^(b,c,d) | 0.904 | ± | 0.011 |
| 9-Methylphenanthrene ^(b,c,d) | 0.437 | ± | 0.044 |
| 6-Methylchrysene ^(b,c,d,f,g) | 0.160 | ± | 0.003 |
| Soxhlet extraction or PFE using temperatures between 100 °C and 150 °C | | | |
| Naphthalene ^(b,c,f,h,i) | 0.946 | ± | 0.086 |
| 2-Methylnaphthalene ^(b,c) | 0.955 | ± | 0.017 |
| Acenaphthene ^(b,c,h,i) | 0.197 | ± | 0.040 |
| Acenaphthylene ^(b,c,h,i) | 0.193 | ± | 0.023 |
| Phenanthrene ^(b,c,f,g,h,i,j) | 4.03 | ± | 0.06 |
| 2-Methylphenanthrene ^(b,c) | 0.896 | ± | 0.047 |
| 1,7-Dimethylphenanthrene ^(b,c,f,g,h) | 0.216 | ± | 0.024 |
| Fluoranthene ^(b,c,f,g,h,i,j) | 6.24 | ± | 0.08 |
| Benzo[ghi]fluoranthene ^(b,c,f,g,h,i,j) | 0.901 | ± | 0.015 |
| Benz[a]anthracene ^(b,c,f,g,h,i,j) | 2.11 | ± | 0.05 |
| 3-Methylchrysene ^(b,c,e,g) | 0.232 | ± | 0.002 |
| Benzo[a]fluoranthene ^(b,c,f,g,h,i,j) | 0.386 | ± | 0.027 |
| Benzo[ghi]perylene ^(b,c,f,g,h,i,j) | 3.97 | ± | 0.04 |
| Coronene ^(b,c,f,g,h) | 3.16 | ± | 0.05 |
| Soxhlet extraction or PFE at 100 °C | | | |
| Biphenyl ^(b,h,i) | 0.182 | ± | 0.020 |
| Fluorene ^(b,e,g,h,i) | 0.223 | ± | 0.035 |
| Dibenzothiophene ^(b,e,g,h,i) | 0.193 | ± | 0.002 |
| Anthracene ^(b,f,g,h,i,k) | 0.410 | ± | 0.004 |
| 1-Methylphenanthrene ^(b,f,g,h) | 0.335 | ± | 0.061 |
| 3-Methylphenanthrene ^(b,h) | 0.555 | ± | 0.031 |
| 2-Methylpyrene ^(f,g) | 0.527 | ± | 0.014 |
| 4-Methylpyrene ^(b,f,g) | 0.239 | ± | 0.014 |
| 8-Methylfluoranthene ^(f,g) | 0.233 | ± | 0.006 ^(e) |
| Cyclopenta[cd]pyrene ⁽ⁱ⁾ | 0.343 | ± | 0.012 ^(l) |
| 2-Methylchrysene ^(f,g) | 0.354 | ± | 0.006 |
| Benzo[a]pyrene ^(b,f,g,h,i) | 2.47 | ± | 0.24 |
| Dibenz[a,c]anthracene ^(f,i) | 0.204 | ± | 0.016 |
| Dibenz[a,h]anthracene ^(f,i) | 0.294 | ± | 0.002 |
| Benzo[c]chrysene ^(f,g,j) | 0.084 | ± | 0.001 |
| Anthanthrene ^(f,g,i,j) | 0.513 | ± | 0.013 |
| Pentaphene ^(f,g,j) | 0.204 | ± | 0.001 |
| Dibenzo[b,e]fluoranthene ^(f) | 0.084 | ± | 0.003 ^(l) |
| Naphtho[1,2-b]fluoranthene ^(f) | 0.621 | ± | 0.021 ^(l) |
| Naphtho[2,3-b]fluoranthene ^(f) | 0.149 | ± | 0.005 ^(l) |
| Dibenzo[a,k]fluoranthene ^(f) | 0.057 | ± | 0.002 ^(l) |
| Dibenzo[j,l]fluoranthene ^(f) | 0.361 | ± | 0.010 ^(l) |
| Dibenzo[a,l]pyrene ^(f) | 0.055 | ± | 0.001 ^(l) |
| Naphtho[2,3-e]pyrene ^(f) | 0.177 | ± | 0.008 ^(l) |
| Naphtho[2,1-a]pyrene ^(f) | 0.372 | ± | 0.011 ^(l) |
| Dibenzo[e,l]pyrene ^(f) | 0.250 | ± | 0.014 ^(l) |

(continued on next page)

Table 6. Reference Mass Fraction Values (Dry-Mass Basis) for Selected PAHs in SRM 1649b based on Extraction Method and Conditions (Continued)

| Extraction Conditions | | Mass Fractions (mg/kg) ^(a) | | |
|-----------------------|--|--|---|------------------------|
| PFE at 150 °C | | | | |
| | Biphenyl ^(c) | 0.316 | ± | 0.023 ^(l) |
| | Fluorene ^(c) | 0.312 | ± | 0.045 ^(l) |
| | Dibenzothiophene ^(c) | 0.418 | ± | 0.042 ^(l) |
| | Anthracene ^(c) | 0.601 | ± | 0.049 ^(l) |
| | Benzo[<i>a</i>]pyrene ^(c) | 2.81 | ± | 0.38 ^(l) |
| PFE at 200 °C | | | | |
| | Naphthalene ^(d) | 2.60 | ± | 0.29 |
| | 2-Methylnaphthalene ^(d) | 1.37 | ± | 0.04 |
| | Biphenyl ^(d) | 0.710 | ± | 0.025 |
| | Acenaphthene ^(d) | 0.244 | ± | 0.016 |
| | Acenaphthylene ^(d) | 0.269 | ± | 0.023 |
| | Fluorene ^(d) | 0.505 | ± | 0.065 |
| | Dibenzothiophene ^(d,k) | 0.579 | ± | 0.040 |
| | Phenanthrene ^(d,k) | 4.40 | ± | 0.02 |
| | Anthracene ^(d,k) | 0.978 | ± | 0.021 ^(e) |
| | 1-Methylphenanthrene ^(c,d) | 0.554 | ± | 0.017 ^(m) |
| | 2-Methylphenanthrene ^(d) | 0.977 | ± | 0.064 |
| | 3-Methylphenanthrene ^(c,d) | 0.641 | ± | 0.024 ^(e,m) |
| | 1,7-Dimethylphenanthrene ^(d,k) | 0.348 | ± | 0.042 ^(e) |
| | Fluoranthene ^(d) | 6.60 | ± | 0.04 |
| | 4-Methylpyrene ^(k) | 0.321 | ± | 0.036 ^(l) |
| | Benzo[<i>ghi</i>]fluoranthene ^(d) | 0.961 | ± | 0.032 ^(e) |
| | Benz[<i>a</i>]anthracene ^(d) | 2.35 | ± | 0.12 |
| | 3-Methylchrysene ^(d) | 0.278 | ± | 0.015 |
| | Benzo[<i>a</i>]fluoranthene ^(d) | 0.505 | ± | 0.018 ^(e) |
| | Benzo[<i>a</i>]pyrene ^(d) | 3.04 | ± | 0.10 |
| | Benzo[<i>ghi</i>]perylene ^(d) | 4.31 | ± | 0.08 |
| | Dibenz[<i>a,c</i> + <i>a,h</i>]anthracene ^(d) | 0.585 | ± | 0.011 ^(e) |
| | Coronene ^(d,k) | 3.33 | ± | 0.16 ^(e) |

^(a) The reference mass fraction values, except where otherwise footnoted, are weighted means of the mass fractions from multiple analytical methods [18]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [20] following the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the PAHs in Table 6 based on the method or methods indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram on a dry-mass basis).

^(b) GC/MS (IVa) on a proprietary relatively non-polar phase after PFE with toluene at 100 °C and 13.8 MPa.

^(c) GC/MS (IVb) on a proprietary relatively non-polar phase after PFE with toluene at 150 °C and 13.8 MPa.

^(d) GC/MS (IVc and IVd) on a proprietary relatively non-polar phase after PFE with toluene at 200 °C and 13.8 MPa and 200 °C and 20.7 MPa.

^(e) The reference mass fraction values are unweighted means of the results from of two or more analytical methods [18,19]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [23], which is consistent with the ISO/JCGM Guides [21,22]. The effective coverage factor, $k = 2$. The measurand is the total mass fraction for the PAHs in Table 6 based on the method or methods indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram on a dry mass basis).

^(f) GC/MS (Ia) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

^(g) GC/MS (Ib) on a proprietary relatively non-polar phase using same extracts as GC/MS (Ia).

^(h) GC/MS (IIa) on a proprietary relatively non-polar phase after PFE at 100 °C with dichloromethane at 13.8 MPa.

⁽ⁱ⁾ GC/MS (III) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE at 100 °C with dichloromethane.

^(j) GC/MS (Ic) on a dimethyl 50 % liquid crystal polysiloxane phase using same extracts as GC/MS (Ia).

^(k) GC/MS (IIb) on a proprietary relatively non-polar phase after PFE at 200 °C with dichloromethane at 13.8 MPa

^(l) The reference value is the mean of results obtained using one analytical technique. The expanded uncertainty, U , is calculated as $U = k u_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k , is determined from the Student's t -distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte. For these compounds, $k = 2.57$. The measurand is the total mass fraction for the PAHs in Table 6 based on the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram on a dry mass basis).

^(m) Value includes data from PFE at 150 °C.

Reference Mass Fraction Values for Selected Nitro-PAHs: The reference values in Table 7 are reported on a dry-mass basis. Reference values for selected Nitro-PAHs are provided based on extraction method and conditions.

Table 7. Reference Mass Fraction Values (Dry-Mass Basis) for Selected Nitro-PAHs in SRM 1649b based on Extraction Method and Conditions

| Extraction Conditions | Mass Fractions ^(a) (µg/kg) |
|---|--|
| Soxhlet extraction or PFE using temperatures between 100 °C and 200 °C 3-Nitrobiphenyl ^(b,c,d,e) | 3.72 ± 0.16 |
| Soxhlet extraction or PFE using temperatures between 100 °C and 150 °C 1-Nitronaphthalene ^(b,c,d) | 7.26 ± 0.03 |
| 2-Nitronaphthalene ^(b,c,d) | 11.8 ± 0.4 |
| 5-Nitroacenaphthene ^(b,c,d) | 3.09 ± 0.01 |
| Soxhlet extraction or PFE using 100 °C 4-Nitrophenanthrene ^(b) | 0.512 ± 0.021 ^(f) |
| 9-Nitroanthracene ^(b,c) | 37.0 ± 4.4 |
| 4-Nitropyrene ^(b) | 5.56 ± 0.12 ^(f) |
| 8-Nitrofluoranthene ^(b) | 8.60 ± 0.17 ^(f) |
| PFE using 150 °C 9-Nitroanthracene ^(d) | 65.6 ± 4.2 ^(f) |
| PFE using 200 °C 1-Nitronaphthalene ^(e) | 7.90 ± 0.06 |
| 2-Nitronaphthalene ^(e) | 12.4 ± 0.1 |
| 5-Nitroacenaphthene ^(e) | 3.60 ± 0.29 |
| 9-Nitroanthracene ^(e) | 243 ± 9 |

^(a) The reference mass fraction values, except where otherwise footnoted, are weighted means of the mass fractions from multiple analytical methods [18]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [20] following the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the Nitro-PAHs in Table 7 based on the method or methods indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

^(b) GC/NICI-MS on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

^(c) GC/MS (IVa) on a proprietary relatively non-polar phase after PFE with toluene at 100 °C and 13.8 MPa.

^(d) GC/MS (IVb) on a proprietary relatively non-polar phase after PFE with toluene at 150 °C and 13.8 MPa.

^(e) GC/MS (IVc and IVd) on a proprietary relatively non-polar phase after PFE with toluene at 200 °C and 13.8 MPa and 200 °C and 20.7 MPa.

^(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. For these compounds, $k = 2.57$. The measurand is the total mass fraction for the Nitro-PAHs in Table 7 based on the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

Reference Mass Fraction Values for Selected PCB Congeners: The reference values in Table 8 are reported on a dry-mass basis. PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] to conform with IUPAC rules; differences occur for the following congeners: BZ 107 is IUPAC 109; BZ 199 is IUPAC 200; BZ 200 is IUPAC 201; and BZ 201 is IUPAC 199. 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.

Table 8. Reference Mass Fraction Values (Dry-Mass Basis) for Selected PCB Congeners in SRM 1649b

| PCB Congeners | | | Mass Fractions ^(a) (µg/kg) | | |
|---------------|-----|--|--|---|--------------------|
| PCB | 8 | (2,4'-Dichlorobiphenyl) ^(b) | 10.8 | ± | 1.1 |
| PCB | 18 | (2,2',5-Trichlorobiphenyl) ^(b) | 15.8 | ± | 0.4 |
| PCB | 28 | (2,4,4'-Trichlorobiphenyl) ^(c) | 17.8 | ± | 0.8 |
| PCB | 31 | (2,4',5-Trichlorobiphenyl) ^(c) | 14.6 | ± | 0.7 |
| PCB | 44 | (2,2',3,5'-Tetrachlorobiphenyl) ^(b,c) | 14.5 | ± | 5.7 ^(d) |
| PCB | 45 | (2,2',3,6-Tetrachlorobiphenyl) ^(b) | 1.9 | ± | 0.1 |
| PCB | 56 | (2,3,3',4'-Tetrachlorobiphenyl) ^(b) | 9.0 | ± | 0.2 |
| | 60 | (2,3,4,4'-Tetrachlorobiphenyl) | | | |
| PCB | 66 | (2,3',4,4'-Tetrachlorobiphenyl) ^(b,c) | 21 | ± | 16 ^(e) |
| PCB | 70 | (2,3',4',5-Tetrachlorobiphenyl) ^(b) | 18.4 | ± | 1.8 |
| PCB | 74 | (2,4,4',5-Tetrachlorobiphenyl) ^(b) | 6.7 | ± | 0.5 |
| PCB | 82 | (2,2',3,3',4-Pentachlorobiphenyl) ^(b) | 2.6 | ± | 0.1 |
| PCB | 87 | (2,2',3,4,5'-Pentachlorobiphenyl) ^(b) | 13.9 | ± | 0.9 |
| | 81 | (3,4,4',5-Tetrachlorobiphenyl) | | | |
| PCB | 92 | (2,2',3,5,5'-Pentachlorobiphenyl) ^(b) | 7.6 | ± | 0.6 |
| | 84 | (2,2',3,3',6-Pentachlorobiphenyl) | | | |
| | 89 | (2,2',3,4,6'-Pentachlorobiphenyl) | | | |
| PCB | 95 | (2,2',3,5',6-Pentachlorobiphenyl) ^(c) | 49.3 | ± | 1.5 |
| PCB | 99 | (2,2',4,4',5-Pentachlorobiphenyl) ^(b,c) | 11 | ± | 5 ^(d) |
| PCB | 107 | (2,3,3',4',5-Pentachlorobiphenyl) ^(b) | 4.5 | ± | 0.2 |
| PCB | 118 | (2,3',4,4',5-Pentachlorobiphenyl) ^(b,c,f) | 24 | ± | 5 ^(d) |
| PCB | 128 | (2,2',3,3',4,4'-Hexachlorobiphenyl) ^(c,f) | 9.7 | ± | 3.0 ^(d) |
| PCB | 130 | (2,2',3,3',4,5'-Hexachlorobiphenyl) ^(b) | 2.61 | ± | 0.08 |
| PCB | 137 | (2,2',3,4,4',5-Hexachlorobiphenyl) ^(b) | 11.2 | ± | 0.7 |
| PCB | 138 | (2,2',3,4,4',5'-Hexachlorobiphenyl) ^(b,c,f) | 61 | ± | 15 ^(d) |
| PCB | 146 | (2,2',3,4',5,5'-Hexachlorobiphenyl) ^(b) | 10.1 | ± | 0.1 |
| PCB | 156 | (2,3,3',4,4',5-Hexachlorobiphenyl) ^(c,f) | 7.4 | ± | 3.4 ^(d) |

(continued on next page)

Table 8. Reference Mass Fraction Values (Dry-Mass Basis) for PCB Congeners in SRM 1649b
(Continued)

| PCB Congeners | | | Mass Fractions ^(a) (µg/kg) | | |
|---------------|---|--|--|---|--------------------|
| PCB 157 | (2,3,3',4,4',5'-Hexachlorobiphenyl) ^(b) | | 1.63 | ± | 0.09 |
| PCB 158 | (2,3,3',4,4',6-Hexachlorobiphenyl) ^(b) | | 7.0 | ± | 0.5 |
| PCB 170 | (2,2',3,3',4,4',5-Heptachlorobiphenyl) ^(b,c,f) | | 36.3 | ± | 9.4 ^(d) |
| PCB 174 | (2,2',3,3',4,5,6'-Heptachlorobiphenyl) ^(b) | | 30.7 | ± | 0.5 |
| PCB 175 | (2,2',3,3',4,5',6-Heptachlorobiphenyl) ^(b) | | 1.30 | ± | 0.03 |
| PCB 176 | (2,2',3,3',4,6,6'-Heptachlorobiphenyl) ^(b) | | 4.85 | ± | 0.09 |
| PCB 177 | (2,2',3,3',4',5,6-Heptachlorobiphenyl) ^(b) | | 16.6 | ± | 0.3 |
| PCB 178 | (2,2',3,3',5,5',6-Heptachlorobiphenyl) ^(b) | | 6.8 | ± | 0.2 |
| PCB 180 | (2,2',3,4,4',5,5'-Heptachlorobiphenyl) ^(f) | | 74.2 | ± | 1.1 |
| PCB 185 | (2,2',3,4,5,5',6-Heptachlorobiphenyl) ^(b) | | 4.13 | ± | 0.08 |
| PCB 189 | (2,3,3',4,4',5,5'-Heptachlorobiphenyl) ^(b) | | 1.6 | ± | 0.1 |
| PCB 191 | (2,3,3',4,4',5',6-Heptachlorobiphenyl) ^(b) | | 1.37 | ± | 0.06 |
| PCB 195 | (2,2',3,3',4,4',5,6-Octachlorobiphenyl) ^(c,f) | | 9.0 | ± | 2.4 ^(d) |
| PCB 199 | (2,2',3,3',4,5,6,6'-Octachlorobiphenyl) ^(b) | | 32 | ± | 4 |
| PCB 200 | (2,2',3,3',4,5',6,6'-Octachlorobiphenyl) ^(b) | | 2.6 | ± | 0.2 |
| PCB 201 | (2,2',3,3',4,5,5',6'-Octachlorobiphenyl) ^(b) | | 3.9 | ± | 0.4 |
| PCB 203 | (2,2',3,4,4',5,5',6-Octachlorobiphenyl) ^(b) | | 32.9 | ± | 2.8 |
| PCB 196 | (2,2',3,3',4,4',5,6'-Octachlorobiphenyl) | | | | |
| PCB 205 | (2,3,3',4,4',5,5',6-Octachlorobiphenyl) ^(b) | | 1.50 | ± | 0.07 |
| PCB 207 | (2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) ^(b) | | 2.2 | ± | 0.2 |
| PCB 208 | (2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl) ^(b) | | 4.3 | ± | 0.8 |
| PCB 209 | Decachlorobiphenyl ^(c,f) | | 5.7 | ± | 1.4 ^(d) |

^(a) The reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, U , is calculated as $U = k u_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k , is determined from the Student's t distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte. For these compounds, $k = 2.57$. The measurand is the total mass fraction for the PCB congeners in Table 8 based on the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

^(b) GC/MS (VIa) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE with dichloromethane.

^(c) GC/MS (Va) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

^(d) The reference mass fraction values are weighted means of the mass fractions from each of two or three analytical methods [18,19]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by the half width of a symmetric 95 % parametric bootstrap confidence interval [23], which is consistent with the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the PCB congeners in Table 8 based on the method or methods indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

^(e) The reference mass fraction value is the weighted means of the mass fractions from multiple analytical methods [18]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by combining within method variances with a between method variance [20] following the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the PCB congeners in Table 8 based on the method or methods indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry-mass basis).

^(f) GC/MS (Vb) on a proprietary relatively non-polar phase using the same extracts as GC/MS (Va).

Reference Mass Fraction Values for Additional Chlorinated Pesticides, including Toxaphene Congeners: The reference values in Table 9 are reported on a dry-mass basis.

Table 9. Reference Mass Fraction Values for Additional Chlorinated Pesticides, including Toxaphene Congeners, in SRM 1649b

| | Mass Fractions ^(a) (µg/kg) | | |
|--|--|---|--------------------|
| <i>alpha</i> -HCH ^(b,c) | 13.7 | ± | 3.4 ^(d) |
| <i>gamma</i> -HCH ^(b) | 3.1 | ± | 1.1 |
| pentachlorobenzene ^(b,c) | 62 | ± | 32 ^(d) |
| hexachlorobenzene ^(b,c) | 3.0 | ± | 1.2 ^(d) |
| oxychlordane ^(b) | 3.73 | ± | 0.03 |
| <i>cis</i> -chlordane ^(b,e) | 46 | ± | 14 ^(d) |
| <i>cis</i> -nonachlor ^(b) | 13.0 | ± | 0.5 |
| mirex ^(b) | 1.33 | ± | 0.08 |
| 2,4'-DDE ^(c) 4.71 | ± | | 0.40 |
| 2,4'-DDD ^(c) 13.6 | ± | | 0.7 |
| 2,4'-DDT ^(c) | 39 | ± | 3 |
| 4,4'-DDT ^(c,f) | 235 | ± | 59 ^(d) |
| 2- <i>endo</i> ,3- <i>exo</i> ,5- <i>endo</i> ,6- <i>exo</i> ,8,8,10,10-octachlorobornane (Parlar 26) ^(b) | 1.80 | ± | 0.04 |
| 2- <i>endo</i> ,3- <i>exo</i> ,5- <i>endo</i> ,6- <i>exo</i> ,8,8,9,10,10-nonachlorobornane (Parlar 50) ^(b) | 8.00 | ± | 0.37 |
| 2,2,5,5,8,9,9,10,10-nonachlorobornane (Parlar 62) ^(b) | 7.54 | ± | 0.25 |
| 2,2,5- <i>endo</i> ,6- <i>exo</i> ,8,9,10-heptachlorobornane (Parlar 32) ^(b) | 8.54 | ± | 0.14 |
| 2,2,3- <i>exo</i> ,5,5,9,9,10,10-nonachlorobornane ^(b) | 1.34 | ± | 0.02 |
| 2,2,3- <i>exo</i> ,5,5,8,9,10,10-nonachlorobornane (Parlar 58) ^(b) | 8.82 | ± | 0.34 |
| 2,2,3- <i>exo</i> ,5- <i>endo</i> ,6- <i>exo</i> ,8,9,10-octachlorobornane (Parlar 39) ^(b) | 2.44 | ± | 0.05 |
| 2- <i>exo</i> ,3- <i>endo</i> ,5- <i>exo</i> ,8,9,10,10-heptachlorobornane ^(b) | 2.12 | ± | 0.05 |
| 2,2,5,5,8,9,10-heptachlorobornane ^(b) | 17.8 | ± | 0.2 |
| 2,2,3- <i>exo</i> ,5- <i>endo</i> ,6- <i>exo</i> ,8,9,10,10-nonachlorobornane ^(b) | 3.11 | ± | 0.10 |
| 2- <i>exo</i> ,3,3,5- <i>exo</i> ,6- <i>endo</i> ,8,9,10,10-nonachlorobornane ^(b) | 1.11 | ± | 0.03 |
| 2,2,5- <i>endo</i> ,6- <i>exo</i> ,8,8,9,10,10-nonachlorobornane (Parlar 56) ^(b) | 2.86 | ± | 0.09 |
| 2,2,5- <i>endo</i> ,6- <i>exo</i> ,8,8,9,10-octachlorobornane (Parlar 42a) ^(b) | 8.08 | ± | 0.22 |
| 2- <i>endo</i> ,3- <i>exo</i> ,5- <i>endo</i> ,6- <i>exo</i> ,8,9,10-heptachlorobornane ^(b) | 1.07 | ± | 0.03 |
| 2,2,5- <i>endo</i> ,6- <i>exo</i> ,8,9,10,10-octachlorobornane ^(b) | 5.59 | ± | 0.25 |
| 2- <i>endo</i> ,3- <i>exo</i> ,5- <i>endo</i> ,6- <i>exo</i> ,8,9,10,10-octachlorobornane (Parlar 40) ^(b) | 8.96 | ± | 0.18 |
| 2,2,3- <i>exo</i> ,8,8,9,10-heptachlorocamphene (Parlar 25) ^(b) | 1.21 | ± | 0.03 |
| 2,2,3- <i>exo</i> ,8,8,9,9,10-octachlorocamphene (Parlar 31) ^(b) | 1.48 | ± | 0.03 |
| 2- <i>exo</i> ,5,5,8,9,9,10,10-octachlorobornane (Parlar 44) ^(b) | 3.96 | ± | 0.05 |
| 2- <i>exo</i> ,3- <i>endo</i> ,5- <i>exo</i> ,8,9,9,10,10-octachlorobornane (Parlar 41) ^(b) | 6.21 | ± | 0.29 |
| 2- <i>endo</i> ,3- <i>exo</i> ,6- <i>exo</i> ,8,8,9,10,10-octachlorobornane ^(b) | 8.82 | ± | 0.32 |

^(a) The reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, U , is calculated as $U = k u_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k , is determined from the Student's t distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte. For these compounds, $k = 2.57$. The measurand is the total mass fraction for the analytes in Table 9 based on the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry mass basis).

^(b) GC/MS (VIb) on a proprietary relatively non-polar phase after PFE with dichloromethane.

^(c) GC/MS (VIa) on a 5 % phenyl-substituted methylpolysiloxane phase using the same extracts as GC/MS (VIb).

^(d) The reference mass fraction values are weighted means of the mass fractions from two or three analytical methods [18,19]. The uncertainty listed with each value is an expanded uncertainty about the mean [19,20], with coverage factor, $k = 2$, calculated by the calculated by combining a between-method variance [24] with a pooled, within-method variance which is consistent with the ISO/JCGM Guides [21,22]. The measurand is the total mass fraction for the analytes in Table 9 based on the method or methods indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry mass basis).

^(e) GC/MS (Vb) on a proprietary relatively non-polar phase after Soxhlet extraction with dichloromethane.

^(f) GC/MS (Va) on a 50 % phenyl-substituted methylpolysiloxane phase using the same extracts as GC/MS (Vb).

Reference Mass Fraction Value for Decabromodiphenyl Ether: The reference value in Table 10 is reported on a dry-mass basis. 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. For these compounds, $k = 2.57$. The measurand is decabromodiphenyl ether in Table 10 based on the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry mass basis).

Table 10. Reference Mass Fraction Value (Dry-Mass Basis) for Decabromodiphenyl Ether in SRM 1649b

| | Mass Fraction ($\mu\text{g/kg}$) | | |
|--|---------------------------------------|-------|-----|
| Decabromodiphenyl ether (BDE 209) ^(a) | 83.2 | \pm | 4.9 |

^(a) GC/NICI-MS on a 5 % phenyl-substituted methylpolysiloxane phase.

Reference Mass Fraction Values for Selected Dibenzo-*p*-Dioxin and Dibenzofuran Congeners: The reference values in Table 11 are reported on a dry-mass basis. The reference value is the mean of results obtained by Environment Canada 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. For these compounds, $k = 2.57$. The measurand is the total mass fraction for the dibenzo-*p*-dioxin and dibenzofuran congeners in Table 11 based on the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as micrograms per kilogram on a dry mass basis).

Table 11. Reference Mass Fraction Values for Selected Dibenzo-*p*-Dioxin and Dibenzofuran Congeners in SRM 1649b

| | Mass Fractions ($\mu\text{g/kg}$) | | |
|--|--|-------|-------|
| 2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin | 0.008 | \pm | 0.001 |
| 1,2,3,7,8-Pentachlorodibenzo- <i>p</i> -dioxin | 0.088 | \pm | 0.004 |
| 1,2,3,4,7,8-Hexachlorodibenzo- <i>p</i> -dioxin | 0.231 | \pm | 0.007 |
| 1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin | 0.670 | \pm | 0.031 |
| 1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin | 0.535 | \pm | 0.041 |
| 1,2,3,4,6,7,8-Heptachlorodibenzo- <i>p</i> -dioxin | 17.5 | \pm | 0.7 |
| Octachlorodibenzo- <i>p</i> -dioxin | 201 | \pm | 9 |
| 2,3,7,8-Tetrachlorodibenzofuran ^(a) | 0.042 | \pm | 0.004 |
| 1,2,3,7,8-Pentachlorodibenzofuran | 0.090 | \pm | 0.006 |
| 2,3,4,7,8-Pentachlorodibenzofuran | 0.29 | \pm | 0.01 |
| 1,2,3,4,7,8-Hexachlorodibenzofuran | 1.01 | \pm | 0.03 |
| 1,2,3,6,7,8-Hexachlorodibenzofuran | 0.39 | \pm | 0.02 |
| 2,3,4,6,7,8-Hexachlorodibenzofuran | 0.52 | \pm | 0.07 |
| 1,2,3,7,8,9-Hexachlorodibenzofuran | 0.042 | \pm | 0.003 |
| 1,2,3,4,6,7,8-Heptachlorodibenzofuran | 3.52 | \pm | 0.09 |
| 1,2,3,4,7,8,9-Heptachlorodibenzofuran | 0.447 | \pm | 0.011 |
| Octachlorodibenzofuran | 6.30 | \pm | 0.32 |
| Total Tetrachlorodibenzo- <i>p</i> -dioxins | 0.15 | \pm | 0.02 |
| Total Pentachlorodibenzo- <i>p</i> -dioxins | 0.84 | \pm | 0.07 |
| Total Hexachlorodibenzo- <i>p</i> -dioxins | 6.56 | \pm | 0.25 |
| Total Heptachlorodibenzo- <i>p</i> -dioxins | 31.0 | \pm | 1.5 |

(continued on next page)

Table 11. Reference Mass Fraction Values for Selected Dibenzo-*p*-Dioxin and Dibenzofuran Congeners in SRM 1649b (Continued)

| | Mass Fractions (µg/kg) | | |
|---|---------------------------|---|------|
| Total Tetrachlorodibenzofurans | 0.76 | ± | 0.07 |
| Total Pentachlorodibenzofurans | 1.69 | ± | 0.07 |
| Total Hexachlorodibenzofurans | 4.4 | ± | 0.2 |
| Total Heptachlorodibenzofurans | 8.5 | ± | 0.2 |
| Total Dibenzo- <i>p</i> -dioxins ^(b) | 240 | ± | 11 |
| Total Dibenzofurans ^(b) | 21.6 | ± | 0.8 |

^(a) Confirmation using a 50 % cyanopropylphenylpolysiloxane phase column.

^(b) Total of tetra- through octa- chlorinated congeners.

Reference Mass Fraction Values for Inorganic Constituents: The reference values in Table 12 are reported on a dry-mass basis. 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. For these compounds, $k = 4.30$. The measurand is the total mass fraction for the inorganic constituents in Table 12 based on the method indicated. Metrological traceability is to the SI derived unit for mass fraction (expressed as milligrams per kilogram on a dry mass basis).

Table 12. Reference Mass Fraction Values for Selected Inorganic Constituents in SRM 1649b

| | Mass Fraction ^(a) (mg/kg) | | |
|-----------|---|---|---|
| Nickel | 168 | ± | 6 |
| Copper | 311 | ± | 7 |
| Chromium | 210 | ± | 4 |
| Manganese | 337 | ± | 4 |
| Vanadium | 344 | ± | 9 |

^(a) ICP-OES

Information Values: The information values provided in Table 13, 14, and 15 are reported on a dry-mass basis. Information values for particle size characteristics are provided in Table 15. An information value is considered to be a value that will be of interest 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 [3].

Table 13. Information Mass Fraction Value for Aluminum in SRM 1649b

| | Mass Fraction (%) |
|-------------------------|----------------------|
| Aluminum ^(a) | 1.8 |

^(a) ICP-OES

Table 14. Information Mass Fraction Values for Additional Analytes in SRM 1649b^(a)

| | Mass Fractions (mg/kg) |
|---|---------------------------|
| 9-Fluorenone | 1.4 |
| Antraquinone | 1.8 |
| Benzanthrone | 1.6 |
| Benz[a]anthracene-7,12-dione | 3.6 |
| n-eicosane | 1.9 |
| n-docosane | 5.2 |
| n-tricosane | 16 |
| n-tetracosane | 27 |
| n-pentacosane | 65 |
| n-hexacosane | 66 |
| n-heptacosane | 62 |
| n-octacosane | 42 |
| n-nonacosane | 58 |
| n-triacontane | 25 |
| n-hentriacontane | 41 |
| n-dotriacontane | 14 |
| $\alpha\alpha$ (20R)-Cholestane | 2.1 |
| 17 α (H)-22,29,30-Trisnorhopane | 2.8 |
| 17 α (H),21 β (H)-(22R)-Homohopane | 4.9 |
| 17 α (H),21 β (H)-(22S)-Homohopane | 5.4 |

^(a) Data reported is based on interlaboratory studies [4,5] and corrected for moisture.

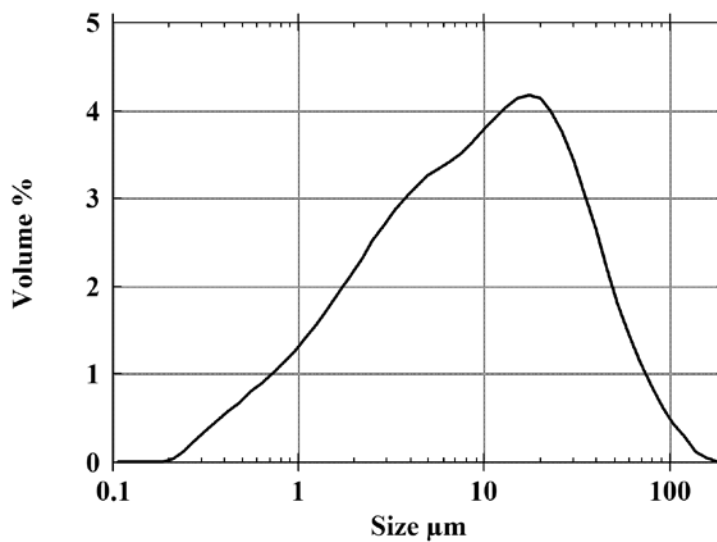


Figure 1. Particle size distribution for SRM 1649b after 1 h sonication in water. Solid line represents the volume in percent.

REFERENCES

- [1] SRM 1649; *Urban Dust/Organics*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (08 April 1982); available at <https://www-s.nist.gov/srmors/certificates/archive/1649.pdf> (accessed Aug 2016).
- [2] SRM 1649a; *Urban Dust/Organics*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (06 December 2007); available at <https://www-s.nist.gov/srmors/certificates/archive/1649a.pdf> (accessed Aug 2016).
- [3] May, W.; Parris, R.; Beck II, 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); available at: <http://www.nist.gov/srm/publications.cfm> (accessed Aug 2016).
- [4] Schantz, M.M.; Wise, S.A.; Lewtas, J.; *Particulate Matter: Description and Results for Trials I and II*; NISTIR 7229 (2005).
- [5] Schantz, M.M.; Poster, D.L.; Kucklick, J.R.; Wise, S.A.; McDow, S.; Lewtas, J.; *Intercomparison Program for Organic Speciation in PM_{2.5} Air Particulate Matter: Description and Results for Trial III*; NISTIR 7303 (2005).
- [6] 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).
- [7] Wise, S.A.; Poster, D.L.; Kucklick, J.R.; Keller, J.M.; Vander Pol, S.S.; Sander, L.C.; Schantz, M.M.; *Standard Reference Materials (SRMs) for Determination of Organic Contaminants in Environmental Samples*; Anal. Biol. Chem., Vol. 386, pp. 1153–1190 (2006).
- [8] 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).
- [9] 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).
- [10] 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).
- [11] 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).
- [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] Schantz, M.M.; McGaw, E.; Wise, S.A.; *Pressurized Liquid Extraction of Diesel and Air Particulate Standard Reference Materials: Effects of Extraction Temperature and Pressure*; Anal. Chem., Vol. 84, pp. 8222–8231 (2012).
- [14] Bergvall, C.; Westerholm, R.; *Determination of 252-302 Da and Tentative Identification of 316-376 Da Polycyclic Aromatic Hydrocarbons in Standard Reference Materials 1649a Urban Dust and 1650b and 2975 Diesel Particulate Matter by Accelerated Solvent Extraction- HPLC-GC-MS*; Anal. Bioanal. Chem., Vol. 391 pp. 2235–2248 (2008).
- [15] 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).
- [16] 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).
- [17] Jones, R.M.; *Particle Size Analysis by Laser Diffraction: ISO 13320, Standard Operating Procedures, and Mie Theory*; Am. Lab., Vol. 35, pages 44–47 (2003).
- [18] Dersimonian, R.; Laird, N.; *Meta-Analysis in Clinical Trials*; Control Clin. Trials, Vol. 7, pp. 177–188 (1986).
- [19] Rukhin, A.L.; *Weighted Means Statistics in Interlaboratory Studies*; Metrologia, Vol. 46, pp. 323–331 (2009).
- [20] Horn, R.A.; Horn, S.A.; Duncan, D.B.; *Estimating Heteroscedastic Variance in Linear Models*; J. Am. Stat. Assoc., Vol. 70, pp. 380–385 (1975).
- [21] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utlis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2016); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at <http://www.nist.gov/pml/pubs/index.cfm> (accessed Aug 2016).

- [22] JCGM 101:2008; *Evaluation of Measurement Data – Supplement 1 to the “Guide to the Expression of Uncertainty in Measurement” - Propagation of Distributions using a Monte Carlo Method*; JCGM (2008); available at http://www.bipm.org/utis/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Aug 2016).
- [23] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall (1993).
- [24] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.-k.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000).

Certificate Revision History: 30 August 2016 (Removed Table 15 information values for particle size distribution characteristics since values were observed to differ from Figure 1 measured distribution; editorial changes); 17 December 2015 (Updated certified and reference values based on new dry mass correction value; PAHs and nitro-PAHs values include the effects of temperature used for extraction; added trace element data; change of expiration date; editorial changes); 23 February 2009 (Original certificate date).

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