

# Standard Reference Material<sup>®</sup> 1649b

## Urban Dust

### CERTIFICATE OF ANALYSIS

**Purpose:** The certified values delivered by this Standard Reference Material (SRM) are intended for use in evaluating analytical methods for the determination of 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.

**Description:** A unit of SRM 1649b consists of a bottle containing 2 g of particulate material. All the constituents for which values are provided in SRM 1649b are naturally present in the particulate material.

**Certified Values:** Certified mass fraction 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 [1]. 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. These values are traceable to International System of Units (SI).

**Non-Certified Values:** Non-certified values are provided in Appendix A. These values 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 [1]. Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the International System of Units (SI) or other higher order reference system.

**Additional Information:** Values of potential interest to users and additional information are provided in Appendices B and C.

**Period of Validity:** The certified values delivered by **SRM 1649b** are valid within the measurement uncertainty specified until **31 July 2030**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

**Maintenance of Certified Values:** NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (<https://www.nist.gov/srm>) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

**Safety:** 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, consult the Safety Data Sheet (SDS) for hazard information.

**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 2. 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.

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

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

<sup>(a)</sup> The certified values are weighted means of the mass fractions from multiple analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by combining within method variances with a between method variance [5] following the ISO/JCGM Guides [6,7].

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

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

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

<sup>(e)</sup> 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.

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

<sup>(g)</sup> 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.

Table 2. Certified Mass Fraction Values (Dry-Mass Basis) for Nitro-PAHs in SRM 1649b<sup>(a)</sup>

	Mass Fractions <sup>(b)</sup> ( $\mu\text{g}/\text{kg}$ )	
3-Nitrophenanthrene	21.8	$\pm$ 1.2
9-Nitrophenanthrene	1.77	$\pm$ 0.07
2-Nitrofluoranthene	304	$\pm$ 25
3-Nitrofluoranthene	4.67	$\pm$ 0.35
1-Nitropyrene	74.8	$\pm$ 1.9
2-Nitropyrene	10.9	$\pm$ 0.1
7-Nitrobenz[ <i>a</i> ]anthracene	24.4	$\pm$ 0.4
6-Nitrochrysene	3.60	$\pm$ 0.21

<sup>(a)</sup> 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.

<sup>(b)</sup> The certified values are weighted means of the mass fractions from multiple analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by combining within method variances with a between method variance [5] following the ISO/JCGM Guides [6,7].

Table 3. Certified Mass Fraction Values (Dry-Mass Basis) for PCB Congeners in SRM 1649b<sup>(a)</sup>

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

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [8] and later revised by Schulte and Malisch [9] 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. The certified value are weighted means of the mass fractions from multiple analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by combining within method variances with a between method variance [5] following the ISO/JCGM Guides [6,7].

<sup>(b)</sup> GC/MS (Va) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with dichloromethane.

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

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

Table 4. Certified Mass Fraction Values (Dry-Mass Basis) for Chlorinated Pesticides in SRM 1649b<sup>(a)</sup>

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

<sup>(a)</sup> The certified value are weighted means of the mass fractions from multiple analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated, combining within method variances with a between method variance [5] following the ISO/JCGM Guides [6,7]

<sup>(b)</sup> GC/MS (Vb) on a relatively non-polar phase after Soxhlet extraction with dichloromethane.

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

<sup>(d)</sup> GC/MS (Va) on a 50 % phenyl-substituted methylpolysiloxane phase using the same extracts as GC/MS (Vb).

<sup>(e)</sup> GC/MS (VIb) on a relatively non-polar phase using the same extracts as GC/MS (VIa).

Table 5. Certified Mass Fraction Values (Dry-Mass Basis) for Inorganic Constituents in SRM 1649b<sup>(a)</sup>

	Mass Fraction ( $\text{mg}/\text{kg}$ )	Coverage Factor, $k$
Cadmium (Cd) <sup>(b)</sup>	26.10 $\pm$ 0.28	3.18
Lead (Pb) <sup>(b)</sup>	12864 $\pm$ 62	3.18
Mercury (Hg) <sup>(c)</sup>	1.80 $\pm$ 0.04	2.36

<sup>(a)</sup> The certified values in Table 5 are reported on a dry-mass basis 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,  $k$ , following the ISO/JCGM Guides [6,7]. The coverage level associated with each expanded uncertainty is 95 %, and the coverage factor is included in the table for each analyte.

<sup>(b)</sup> ID-ICP-MS, calculated dry-mass basis using a moisture content of  $2.67 \% \pm 0.17 \%$ .

<sup>(c)</sup> Cold-vapor ID-ICP-MS, calculated dry-mass basis using a moisture content of  $0.97 \% \pm 0.06 \%$  for Cd and Pb.

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**Certificate Revision History:** 16 May 2023 (Updated non-certified values and uncertainties for Ni, Cu, Cr, Mn, and V in Table A7; added particle size values of potential interest to users in Appendix B; updated format; editorial changes); 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).

*Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.*

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\*\*\*\*\* End of Certificate of Analysis \*\*\*\*\*

# APPENDIX A

## Non-Certified Values

Table A1. Non-Certified Mass Fraction Values (Dry-Mass Basis) for PAHs in SRM 1649b based on Extraction Method and Conditions

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

(continued on next page)

Table A1. Non-Certified Mass Fraction Values (Dry-Mass Basis) for PAHs in SRM 1649b based on Extraction Method and Conditions (Continued)

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

<sup>(a)</sup> The non-certified mass fraction values, except where otherwise footnoted, are weighted means of the mass fractions from multiple analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by combining within method variances with a between method variance [5] following the ISO/JCGM Guides [6,7].

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

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

<sup>(d)</sup> 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.

<sup>(e)</sup> The non-certified mass fraction values are unweighted means of the results from two or more analytical methods [3,4]. The expanded uncertainty is the half width of a symmetric 95 % parametric bootstrap confidence interval [10], which is consistent with the ISO/JCGM Guides [6,7]. The effective coverage factor,  $k = 2$ . The measurand is the total mass fraction for the PAHs in Table A1 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) as realized by the method used.

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

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

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

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

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

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

<sup>(l)</sup> The non-certified value is the mean of results obtained 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,  $k = 2.57$ .

<sup>(m)</sup> Value includes data from PFE at 150 °C.

Table A2. Non-Certified Mass Fraction Values (Dry-Mass Basis) for Nitro-PAHs in SRM 1649b based on Extraction Method and Conditions

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

<sup>(a)</sup> The non-certified mass fraction values, except where otherwise footnoted, are weighted means of the mass fractions from multiple analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by combining within method variances with a between method variance [5] following the ISO/JCGM Guides [6,7].

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

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

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

<sup>(e)</sup> 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.

<sup>(f)</sup> The non-certified 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,  $k = 2.57$ .



**Non-Certified Mass Fraction Values for PCB Congeners:** The non-certified values in Table A3 are reported on a dry-mass basis. PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [8] and later revised by Schulte and Malisch [9] 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 A3. Non-Certified Mass Fraction Values (Dry-Mass Basis) for PCB Congeners in SRM 1649b

PCB Congeners			Mass Fractions <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )	
PCB	8	(2,4'-Dichlorobiphenyl) <sup>(b)</sup>	10.8	$\pm$ 1.1
PCB	18	(2,2',5-Trichlorobiphenyl) <sup>(b)</sup>	15.8	$\pm$ 0.4
PCB	28	(2,4,4'-Trichlorobiphenyl) <sup>(c)</sup>	17.8	$\pm$ 0.8
PCB	31	(2,4',5-Trichlorobiphenyl) <sup>(c)</sup>	14.6	$\pm$ 0.7
PCB	44	(2,2',3,5'-Tetrachlorobiphenyl) <sup>(b,c)</sup>	14.5	$\pm$ 5.7 <sup>(d)</sup>
PCB	45	(2,2',3,6-Tetrachlorobiphenyl) <sup>(b)</sup>	1.9	$\pm$ 0.1
PCB	56	(2,3,3',4'-Tetrachlorobiphenyl) <sup>(b)</sup>	9.0	$\pm$ 0.2
	60	(2,3,4,4'-Tetrachlorobiphenyl)		
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl) <sup>(b,c)</sup>	21	$\pm$ 16 <sup>(e)</sup>
PCB	70	(2,3',4',5-Tetrachlorobiphenyl) <sup>(b)</sup>	18.4	$\pm$ 1.8
PCB	74	(2,4,4',5-Tetrachlorobiphenyl) <sup>(b)</sup>	6.7	$\pm$ 0.5
PCB	82	(2,2',3,3',4-Pentachlorobiphenyl) <sup>(b)</sup>	2.6	$\pm$ 0.1
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl) <sup>(b)</sup>	13.9	$\pm$ 0.9
	81	(3,4,4',5-Tetrachlorobiphenyl)		
PCB	92	(2,2',3,5,5'-Pentachlorobiphenyl) <sup>(b)</sup>	7.6	$\pm$ 0.6
	84	(2,2',3,3',6-Pentachlorobiphenyl)		
	89	(2,2',3,4,6'-Pentachlorobiphenyl)		
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl) <sup>(c)</sup>	49.3	$\pm$ 1.5
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl) <sup>(b,c)</sup>	11	$\pm$ 5 <sup>(d)</sup>
PCB	107	(2,3,3',4',5-Pentachlorobiphenyl) <sup>(b)</sup>	4.5	$\pm$ 0.2
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl) <sup>(b,c,f)</sup>	24	$\pm$ 5 <sup>(d)</sup>
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl) <sup>(c,f)</sup>	9.7	$\pm$ 3.0 <sup>(d)</sup>
PCB	130	(2,2',3,3',4,5'-Hexachlorobiphenyl) <sup>(b)</sup>	2.61	$\pm$ 0.08
PCB	137	(2,2',3,4,4',5-Hexachlorobiphenyl) <sup>(b)</sup>	11.2	$\pm$ 0.7
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl) <sup>(b,c,f)</sup>	61	$\pm$ 15 <sup>(d)</sup>
PCB	146	(2,2',3,4',5,5'-Hexachlorobiphenyl) <sup>(b)</sup>	10.1	$\pm$ 0.1
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl) <sup>(c,f)</sup>	7.4	$\pm$ 3.4 <sup>(d)</sup>
PCB	157	(2,3,3',4,4',5'-Hexachlorobiphenyl) <sup>(b)</sup>	1.63	$\pm$ 0.09
PCB	158	(2,3,3',4,4',6-Hexachlorobiphenyl) <sup>(b)</sup>	7.0	$\pm$ 0.5
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl) <sup>(b,c,f)</sup>	36.3	$\pm$ 9.4 <sup>(d)</sup>
PCB	174	(2,2',3,3',4,5,6'-Heptachlorobiphenyl) <sup>(b)</sup>	30.7	$\pm$ 0.5
PCB	175	(2,2',3,3',4,5',6-Heptachlorobiphenyl) <sup>(b)</sup>	1.30	$\pm$ 0.03
PCB	176	(2,2',3,3',4,6,6'-Heptachlorobiphenyl) <sup>(b)</sup>	4.85	$\pm$ 0.09
PCB	177	(2,2',3,3',4',5,6-Heptachlorobiphenyl) <sup>(b)</sup>	16.6	$\pm$ 0.3
PCB	178	(2,2',3,3',5,5',6-Heptachlorobiphenyl) <sup>(b)</sup>	6.8	$\pm$ 0.2
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) <sup>(f)</sup>	74.2	$\pm$ 1.1
PCB	185	(2,2',3,4,5,5',6-Heptachlorobiphenyl) <sup>(b)</sup>	4.13	$\pm$ 0.08
PCB	189	(2,3,3',4,4',5,5'-Heptachlorobiphenyl) <sup>(b)</sup>	1.6	$\pm$ 0.1
PCB	191	(2,3,3',4,4',5',6-Heptachlorobiphenyl) <sup>(b)</sup>	1.37	$\pm$ 0.06
PCB	195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl) <sup>(c,f)</sup>	9.0	$\pm$ 2.4 <sup>(d)</sup>
PCB	199	(2,2',3,3',4,5,6,6'-Octachlorobiphenyl) <sup>(b)</sup>	32	$\pm$ 4

(continued on next page)

Table A3. Non-Certified Mass Fraction Values (Dry-Mass Basis) for PCB Congeners in SRM 1649b  
(Continued)

PCB Congeners			Mass Fractions <sup>(a)</sup> (µg/kg)
PCB	200	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl) <sup>(b)</sup>	2.6 ± 0.2
PCB	201	(2,2',3,3',4,5,5',6'-Octachlorobiphenyl) <sup>(b)</sup>	3.9 ± 0.4
PCB	203	(2,2',3,4,4',5,5',6-Octachlorobiphenyl) <sup>(b)</sup>	32.9 ± 2.8
	196	(2,2',3,3',4,4',5,6'-Octachlorobiphenyl)	
PCB	205	(2,3,3',4,4',5,5',6-Octachlorobiphenyl) <sup>(b)</sup>	1.50 ± 0.07
PCB	207	(2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) <sup>(b)</sup>	2.2 ± 0.2
PCB	208	(2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl) <sup>(b)</sup>	4.3 ± 0.8
PCB	209	Decachlorobiphenyl <sup>(c,f)</sup>	5.7 ± 1.4 <sup>(d)</sup>

<sup>(a)</sup> The non-certified 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,  $k = 2.57$ .

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

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

<sup>(d)</sup> The non-certified mass fraction values are weighted means of the mass fractions from each of two or three analytical methods [3,4]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by the half width of a symmetric 95 % parametric bootstrap confidence interval [2,3] which is consistent with the ISO/JCGM Guides [6,7]. The measurand is the total mass fraction for the PCB congeners in Table A3 based on the method or methods indicated.

<sup>(e)</sup> The non-certified mass fraction value is the weighted means of the mass fractions from multiple analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by combining within method variances with a between method variance [5] following the ISO/JCGM Guides [6,7].

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

Table A4. Non-Certified Mass Fraction Values for Additional Chlorinated Pesticides, including Toxaphene Congeners, in SRM 1649b

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

<sup>(a)</sup> The non-certified 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,  $k = 2.57$ .

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

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

<sup>(d)</sup> The non-certified mass fraction values are weighted means of the mass fractions from two or three analytical methods [3,4]. The uncertainty listed with each value is an expanded uncertainty about the mean [4,5], with coverage factor,  $k = 2$ , calculated by the calculated by combining a between-method variance [11] with a pooled, within-method variance which is consistent with the ISO/JCGM Guides [6,7].

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

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

**Non-Certified Mass Fraction Value for Decabromodiphenyl Ether:** The non-certified value in Table A5 is reported on a dry-mass basis. The non-certified 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,  $k = 2.57$ .

Table A5. Non-Certified Mass Fraction Value (Dry-Mass Basis) for Decabromodiphenyl Ether in SRM 1649b

	Mass Fractions <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )	
Decabromodiphenyl ether (BDE 209) <sup>(a)</sup>	83.2	$\pm$ 4.9

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

**Non-Certified Mass Fraction Values for Dibenzo-*p*-Dioxin and Dibenzofuran Congeners:** The non-certified values in Table A6 are reported on a dry-mass basis. The non-certified 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,  $k = 2.57$ .

Table A6. Non-Certified Mass Fraction Values for Dibenzo-*p*-Dioxin and Dibenzofuran Congeners in SRM 1649b

	Mass Fractions <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )	
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 <sup>(a)</sup>	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
Total Tetrachlorodibenzofurans	0.76	$\pm$ 0.07
Total Pentachlorodibenzofurans	1.69	$\pm$ 0.07
Total Hexachlorodibenzofurans	4.4	$\pm$ 0.2
Total Heptachlorodibenzofurans	8.5	$\pm$ 0.2
Total Dibenzo- <i>p</i> -dioxins <sup>(b)</sup>	240	$\pm$ 11
Total Dibenzofurans <sup>(b)</sup>	21.6	$\pm$ 0.8

<sup>(a)</sup> Confirmation using a 50 % cyanopropylphenylpolysiloxane phase column.

<sup>(b)</sup> Total of tetra- through octa- chlorinated congeners.

**Non-Certified Mass Fraction Values for Inorganic Constituents:** The non-certified values in Table A7 are reported on a dry-mass basis. The non-certified 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,  $k = 4.3$ .

Table A7. Non-Certified Mass Fraction Values for Inorganic Constituents in SRM 1649b

	Mass Fraction <sup>(a)</sup> (mg/kg)
Nickel (Ni)	166 ± 6
Copper (Cu)	308 ± 7
Chromium (Cr)	208 ± 4
Manganese (Mn)	333 ± 4
Vanadium (V)	341 ± 9

<sup>(a)</sup> ICP-OES, calculated dry-mass basis using a moisture content of  $0.97\% \pm 0.06\%$ .

**Maintenance of Non-Certified Values:** NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Certificate of Analysis and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (<https://www.nist.gov/srm>).

\* \* \* \* \* End of Appendix A \* \* \* \* \*

# APPENDIX B

**Values of Potential Interest to Users:** Values of potential interest to users are provided in Table B1, B2, B3 and Figure B1.

Table B1. Mass Fraction Value of Potential Interest to Users for Aluminum in SRM 1649b

	Mass Fraction <sup>(a)</sup> (%)
Aluminum (Al)	1.8

<sup>(a)</sup> ICP-OES

Table B2. Mass Fraction Values of Potential Interest to Users for Additional Analytes in SRM 1649b<sup>(a)</sup>

	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 $\alpha$ (H)-22,29,30-Trisnorhopane	2.8
17 $\alpha$ (H),21 $\beta$ (H)-(22R)-Homohopane	4.9
17 $\alpha$ (H),21 $\beta$ (H)-(22S)-Homohopane	5.4

<sup>(a)</sup> Data reported is based on interlaboratory studies [12,13] and corrected for moisture.

Table B3. Particle Size Values of Potential Interest to Users for SRM 1649b<sup>(a)</sup>

Mean Particle Diameter, d(0.5) <sup>(b)</sup>	24.3	$\mu\text{m}$
Particle Diameter, d(0.1) <sup>(c)</sup>	7.07	$\mu\text{m}$
Particle Diameter, d(0.9) <sup>(d)</sup>	66	$\mu\text{m}$

<sup>(a)</sup> These values are of potential interest to users and are based on and instrument-specific measurement of SRM 1649b dispersed in ethanol and Triton X-100. The values have not been confirmed by an independent analytical technique as required for certification. See Figure B1 for particle-size distribution.

<sup>(b)</sup> d(0.5) is the particle-size distribution parameter indicating the particle size below which 50 % of the volume is present.

<sup>(c)</sup> d(0.1) is the particle-size distribution parameter indicating the particle size below which 10 % of the volume is present.

<sup>(d)</sup> d(0.9) is the particle-size distribution parameter indicating the particle size below which 90 % of the volume is present

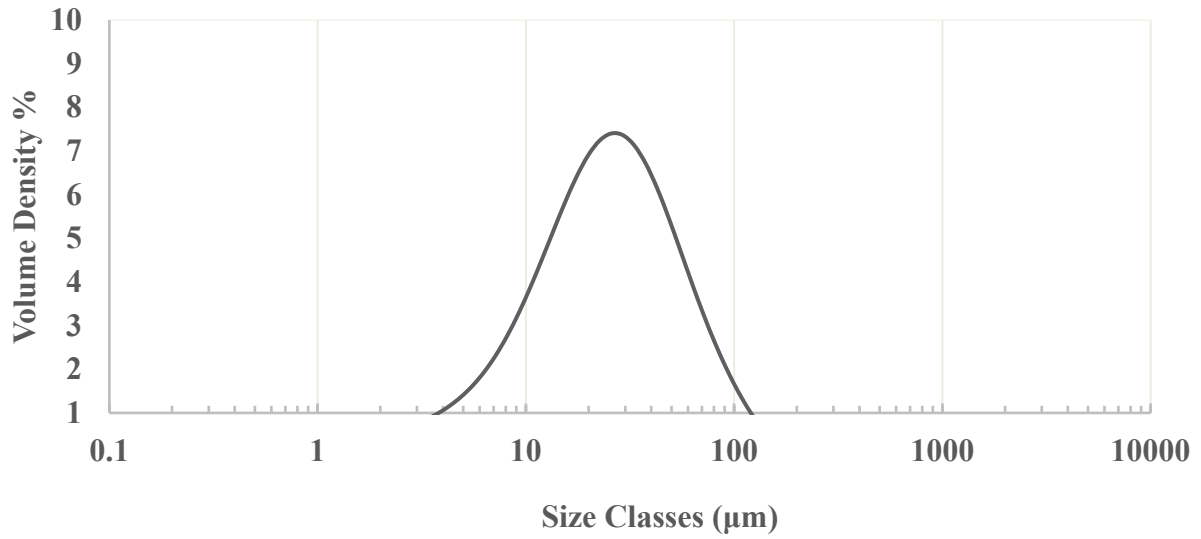


Figure B1. Average Volume Particle Size Distribution of Potential Interest to Users SRM 1649b Urban Dust.  
 Solid line represents volume density % with respect to particle size.

\*\*\*\*\* End of Appendix B \*\*\*\*\*

# APPENDIX C

**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  $\mu\text{m}$  (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. [14,15], Poster et al. [16], and Currie et al. [17]. 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.

**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 %  $\pm$  0.07 % at the 95 % confidence level. If the analyst measuring a specific analyte performed an assessment of the moisture content, that value is stated in the footnote of the analyte table.

**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 [14]. 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.  $\times$  60 m fused silica capillary column with a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25  $\mu\text{m}$  film thickness; DB-17MS, Agilent Technologies, Wilmington, DE), designated as GC/MS (Ia); (2) a 0.25 mm i.d.  $\times$  60 m fused silica capillary column with a proprietary relatively nonpolar phase (0.25  $\mu\text{m}$  film thickness; DB-XLB, Agilent Technologies), designated as GC/MS (Ib); and (3) a 0.25 mm i.d.  $\times$  20 m dimethyl 50 % liquid crystal polysiloxane phase (0.25  $\mu\text{m}$  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.  $\times$  60 m fused silica capillary column with a proprietary relatively nonpolar phase (0.25  $\mu\text{m}$  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.  $\times$  60 m fused silica capillary column containing a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25  $\mu\text{m}$  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, 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 [15] 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 [18]. 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, 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 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 [19]. Recent studies on solvent extraction of air particulate matter [20,21] 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 non-certified values. These non-certified 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 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 [8,9]) and <sup>13</sup>C-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 Dibenzop-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 <sup>111</sup>Cd and <sup>206</sup>Pb 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 <sup>201</sup>Hg 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 determined using a laser diffraction instrument fitted with a wet measurement cell [Mastersizer 3000 (MS 3000), Malvern Instruments, Southborough, MA]. The MS 3000 is fitted with a red-light laser (λ = 632.8 nm) and a blue light laser (λ = 470 nm). As particles pass through the measurement cell, detection of the angular distribution of scattered light produced by the incident beam and analyzed at the detectors gives the average size distribution represented as an equivalent spherical diameter in an average volume distribution. The measurement was conducted in ethanol with Triton X-100 surfactant at 0.01 % concentration. Background measurements were automatically subtracted before sample addition. The sample bottles were mixed by a combination of inversion and rolling 10-15 times. Approximately 0.5 gram of material from two jars were combined. A small amount of the ethanol/Triton X-100 solution was used to produce a slurry and the material was added to the measurement cell until an obscuration of 14 % was achieved. The sample was allowed to disperse for 10 seconds before ten (10) individual measurements were made. Results were calculated using procedures described in reference 22 and the particle size distribution is shown in Figure B1.

*Additional Information:* Analytical measurements for the certification of SRM 1649b were performed by NIST, and by 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<sub>2.5</sub> Air Particulate Matter [12,13].

\*\*\*\*\* End of Appendix C \*\*\*\*\*