

# Standard Reference Material<sup>®</sup> 1941b

## Organics in Marine Sediment

### CERTIFICATE OF ANALYSIS

**Purpose:** This Standard Reference Material (SRM) is marine sediment collected at the mouth of the Baltimore (MD) Harbor. SRM 1941b is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, and chlorinated pesticides in marine sediment and similar matrices. Non-certified values are also provided for total organic carbon (TOC), total carbon, hydrogen, and nitrogen. All of the constituents for which certified and non-certified values are provided in SRM 1941b were naturally present in the sediment before processing.

**Description:** A unit of SRM 1941b consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment.

**Certified Values:** Certified mass fraction values for PAHs, PCB congeners, and chlorinated pesticides are provided in Table 1 through Table 3. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques along with results from an interlaboratory comparison study [1]. 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].

**Non-Certified Values:** Non-certified values are provided in the Appendix A.

**Additional Information:** Additional information is provided in Appendices B-D.

**Period of Validity:** The certified values delivered by **SRM 1941b** are valid within the measurement uncertainty specified until **14 April 2032**. 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>).

Table 1. Certified Mass Fraction Values for PAHs in SRM 1941b

PAHs	Mass Fractions <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )
Naphthalene <sup>(b,c,d,e,f,g)</sup>	848 $\pm$ 95 <sup>(h)</sup>
Fluorene <sup>(b,c,d,e,f,g)</sup>	85 $\pm$ 15 <sup>(h)</sup>
Phenanthrene <sup>(b,c,d,e,f,g)</sup>	406 $\pm$ 44 <sup>(h)</sup>
Anthracene <sup>(b,c,d,e,f,g)</sup>	184 $\pm$ 18 <sup>(h)</sup>
3-Methylphenanthrene <sup>(b,c,d)</sup>	105 $\pm$ 13 <sup>(h)</sup>
2-Methylphenanthrene <sup>(b,c,d)</sup>	128 $\pm$ 14 <sup>(h)</sup>
1-Methylphenanthrene <sup>(b,c,d,g)</sup>	73.2 $\pm$ 5.9 <sup>(h)</sup>
Fluoranthene <sup>(b,c,d,e,f,g)</sup>	651 $\pm$ 50 <sup>(h)</sup>
Pyrene <sup>(b,c,d,e,f,g)</sup>	581 $\pm$ 39 <sup>(h)</sup>
Benz[ <i>a</i> ]anthracene <sup>(b,c,d,e,f,g)</sup>	335 $\pm$ 25 <sup>(h)</sup>
Chrysene <sup>(d,f)</sup>	291 $\pm$ 31 <sup>(h)</sup>
Triphenylene <sup>(d,f)</sup>	108 $\pm$ 5 <sup>(i)</sup>
Benzo[ <i>b</i> ]fluoranthene <sup>(c,e)</sup>	453 $\pm$ 21 <sup>(h)</sup>
Benzo[ <i>k</i> ]fluoranthene <sup>(b,c,d,e)</sup>	225 $\pm$ 18 <sup>(h)</sup>
Benzo[ <i>e</i> ]pyrene <sup>(b,c,d,g)</sup>	325 $\pm$ 25 <sup>(h)</sup>
Benzo[ <i>a</i> ]pyrene <sup>(b,c,d,f,g)</sup>	358 $\pm$ 17 <sup>(h)</sup>
Perylene <sup>(b,c,d,f,g)</sup>	397 $\pm$ 45 <sup>(h)</sup>
Benzo[ <i>ghi</i> ]perylene <sup>(b,c,d,f,g)</sup>	307 $\pm$ 45 <sup>(h)</sup>
Indeno[1,2,3- <i>cd</i> ]pyrene <sup>(b,c,d,f,g)</sup>	341 $\pm$ 57 <sup>(h)</sup>
Dibenz[ <i>a,j</i> ]anthracene <sup>(b,c,d,f)</sup>	48.9 $\pm$ 4.6 <sup>(h)</sup>
Dibenz[ <i>a,c</i> ]anthracene <sup>(c,f)</sup>	36.7 $\pm$ 5.2 <sup>(h)</sup>
Dibenz[ <i>a,h</i> ]anthracene <sup>(c,f)</sup>	53 $\pm$ 10 <sup>(h)</sup>
Benzo[ <i>b</i> ]chrysene <sup>(b,c,d,f)</sup>	53 $\pm$ 12 <sup>(h)</sup>
Picene <sup>(b,c,d)</sup>	46.6 $\pm$ 4.7 <sup>(h)</sup>

<sup>(a)</sup> Mass fractions reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

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

<sup>(c)</sup> GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

<sup>(d)</sup> GC/MS (III) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(e)</sup> LC-FL (total) of total PAH fraction after PFE with DCM.

<sup>(f)</sup> LC-FL (isomer) of isomeric PAH fractions after PFE with DCM.

<sup>(g)</sup> 1999 Interlaboratory Comparison Study [2] with 21 to 29 laboratories submitting data for each PAH.

<sup>(h)</sup> Certified values are weighted means of the results from two to six analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of the constituent listed and the values are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(i)</sup> The certified value is an unweighted mean of the results from two analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [6] with a pooled, within-method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of the constituent listed and the value is metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

Table 2. Certified Mass Fraction Values for PCB Congeners<sup>(a)</sup> in SRM 1941b

PCB Congeners			Mass Fractions <sup>(b)</sup> (µg/kg)	
PCB	8	(2,4'-Dichlorobiphenyl) <sup>(c,d,e,f,g)</sup>	1.65	± 0.19 <sup>(h)</sup>
PCB	18	(2,2',5-Trichlorobiphenyl) <sup>(c,d,e,f,g)</sup>	2.39	± 0.29 <sup>(h)</sup>
PCB	28	(2,4,4'-Trichlorobiphenyl) <sup>(c,d,e,f,g)</sup>	4.52	± 0.57 <sup>(h)</sup>
PCB	31	(2,4',5-Trichlorobiphenyl) <sup>(c,e,f)</sup>	3.18	± 0.41 <sup>(h)</sup>
PCB	44	(2,2',3,5'-Tetrachlorobiphenyl) <sup>(c,d,e,f,g)</sup>	3.85	± 0.20 <sup>(i)</sup>
PCB	49	(2,2',4,5'-Tetrachlorobiphenyl) <sup>(c,d,e,f)</sup>	4.34	± 0.28 <sup>(i)</sup>
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl) <sup>(c,d,e,f,g)</sup>	5.24	± 0.28 <sup>(i)</sup>
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl) <sup>(c,e,f,g,j)</sup>	4.96	± 0.53 <sup>(i)</sup>
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl) <sup>(c,d,f,j)</sup>	1.14	± 0.16 <sup>(h)</sup>
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl) <sup>(c,e,f,g)</sup>	3.93	± 0.62 <sup>(i)</sup>
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl) <sup>(c,d,e,f,g)</sup>	2.90	± 0.36 <sup>(i)</sup>
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl) <sup>(c,e,f,g,j)</sup>	5.11	± 0.34 <sup>(i)</sup>
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl) <sup>(c,d,e,f,g,j)</sup>	1.43	± 0.10 <sup>(i)</sup>
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl) <sup>(c,e,f,j)</sup>	4.62	± 0.36 <sup>(i)</sup>
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl) <sup>(c,d,e,f,g,j)</sup>	4.23	± 0.19 <sup>(i)</sup>
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl) <sup>(c,d,e,f,g,j)</sup>	0.696	± 0.044 <sup>(i)</sup>
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl) <sup>(c,e,f,j)</sup>	3.60	± 0.28 <sup>(i)</sup>
PCB	149	(2,2',3,4',5',6-Hexachlorobiphenyl) <sup>(c,d,e,j)</sup>	4.35	± 0.26 <sup>(h)</sup>
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl) <sup>(c,d,e,f,g,j)</sup>	5.47	± 0.32 <sup>(i)</sup>
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl) <sup>(c,d,e,f,j)</sup>	0.507	± 0.090 <sup>(h)</sup>
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl) <sup>(c,d,e,f,g,j)</sup>	1.35	± 0.09 <sup>(i)</sup>
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) <sup>(c,d,e,f,g,j)</sup>	3.24	± 0.51 <sup>(i)</sup>
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl) <sup>(c,d,e,j)</sup>	0.979	± 0.087 <sup>(h)</sup>
PCB	187	(2,2',3,4',5,5',6-Heptachlorobiphenyl) <sup>(c,d,e,f,g,j)</sup>	2.17	± 0.22 <sup>(i)</sup>
PCB	194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl) <sup>(c,d,e,j)</sup>	1.04	± 0.06 <sup>(h)</sup>
PCB	195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl) <sup>(c,e,g,j)</sup>	0.645	± 0.060 <sup>(i)</sup>
PCB	201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl) <sup>(c,e,j)</sup>	0.777	± 0.034 <sup>(h)</sup>
PCB	206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) <sup>(c,e,f,g,j)</sup>	2.42	± 0.19 <sup>(i)</sup>
PCB	209	Decachlorobiphenyl <sup>(c,d,e,f,g,j)</sup>	4.86	± 0.45 <sup>(i)</sup>

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [7] and later revised by Schulte and Malisch [8] to conform to IUPAC rules, except PCB 201. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 201 is listed as PCB 200.

<sup>(b)</sup> Mass fractions reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

<sup>(c)</sup> GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(d)</sup> GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

<sup>(e)</sup> GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).

<sup>(f)</sup> GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.

<sup>(g)</sup> 1999 Interlaboratory Comparison Study [2] with 13 to 31 laboratories submitting data for each PCB congener.

<sup>(h)</sup> Certified values are unweighted means of the results from three to five analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [6] with a pooled, within method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of the constituent listed and the values are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(i)</sup> Certified values are weighted means of the results from three to six analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of the constituent listed and the values are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(j)</sup> GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

Table 3. Certified Mass Fraction Values for Chlorinated Pesticides in SRM 1941b

Chlorinated Pesticides	Mass Fractions <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )
Hexachlorobenzene <sup>(b,c,d,e)</sup>	5.83 $\pm$ 0.38 <sup>(f)</sup>
<i>cis</i> -Chlordane <sup>(b,c,d,e,g)</sup>	0.85 $\pm$ 0.11 <sup>(h)</sup>
<i>trans</i> -Chlordane <sup>(b,c,e)</sup>	0.566 $\pm$ 0.093 <sup>(f)</sup>
<i>cis</i> -Nonachlor <sup>(b,e,g)</sup>	0.378 $\pm$ 0.053 <sup>(h)</sup>
<i>trans</i> -Nonachlor <sup>(b,c,d,e,g)</sup>	0.438 $\pm$ 0.073 <sup>(f)</sup>
4,4'-DDE <sup>(b,d,e,g)</sup>	3.22 $\pm$ 0.28 <sup>(h)</sup>
4,4'-DDD <sup>(b,d,e,g)</sup>	4.66 $\pm$ 0.46 <sup>(h)</sup>

<sup>(a)</sup> Mass fractions reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

<sup>(b)</sup> GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(c)</sup> GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

<sup>(d)</sup> GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.

<sup>(e)</sup> 1999 Interlaboratory Comparison Study [2] with 13 to 31 laboratories submitting data for each pesticide.

<sup>(f)</sup> Certified values are unweighted means of the results from three to five analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [6] with a pooled, within-method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of the constituent listed and the values are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(g)</sup> GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

<sup>(h)</sup> Certified values are weighted means of the results from three to five analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of the chlorinated pesticides listed and the values listed are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

**Safety:** This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

**Storage:** SRM 1941b must be stored in its original 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. The mass fractions of constituents in SRM 1941b are reported on a dry-mass basis. The SRM, as received, contains a mass fraction of approximately 2.4 % moisture (see “Conversion to Dry-Mass Basis”). The sediment sample should be dried to a constant mass before weighing for analysis; or a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the mass fraction on a dry-mass basis. If the constituents of interest are volatile, then the moisture must be determined with a separate subsample.

## REFERENCES

- [1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Diewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; U.S. Government Printing Office: Washington, DC (2021); available at <https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf> (accessed Apr 2022).
- [2] Schantz, M.M.; Parris, R.M.; Wise, S.A.; *NIST/NOAA Intercomparison Exercise Program for Organic Contaminants in the Marine Environment: Description and Results of 1999 Organic Intercomparison Exercises*; NOAA Technical Memorandum NOS NCCOS CCMA 146, Silver Spring, MD (2000).
- [3] Ruhkin, A.L.; Vangel, M.G.; *Estimation of a Common Mean and Weighted Means Statistics*; J. Am. Statist. Assoc., Vol. 93, pp. 303–308 (1998).
- [4] 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 (2008); available at <https://www.bipm.org/en/publications/guides> (accessed Apr 2022); 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 <https://www.nist.gov/pml/nist-technical-note-1297> (accessed Apr 2022).
- [5] 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 <https://www.bipm.org/en/publications/guides> (accessed Apr 2022).
- [6] 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).
- [7] 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).
- [8] 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).
- [9] Schubert, P.; Schantz, M.M.; Sander, L.C.; Wise, S.A.; *Determination of Polycyclic Aromatic Hydrocarbons with Molecular Mass 300 and 302 in Environmental-Matrix Standard Reference Materials by Gas Chromatography-Mass Spectrometry*; Anal. Chem., Vol. 75, pp. 234–246 (2003).
- [10] Schantz, M.M.; Kucklick, J.R.; *NIST Interlaboratory Analytical Comparison Study to Support Deepwater Horizon Natural Resource Damage Assessment: Description and Results for Crude Oil QA10OIL01*; NISTIR 7793 (2011).
- [11] Wise, S.A.; Poster, D.L.; Schantz, M.M.; Kucklick, J.R.; Sander, L.C.; Lopez de Alda, M.; Schubert, P.; Parris, R.M.; Porter, B.J.; *Two New Marine Sediment Standard Reference Materials (SRMs) for the Determination of Organic Contaminants*; Anal. Bioanal. Chem., Vol. 378, pp. 1251–1264 (2004).
- [12] Wise, S.A.; Chesler, S.N.; Hertz, H.S.; Hilpert, L.R.; May, W.E.; *Chemically-Bonded Aminosilane Stationary Phase for the High Performance Liquid Chromatographic Separation of Polynuclear Aromatic Hydrocarbons*; Anal. Chem., Vol. 49, pp. 2306–2310 (1977).
- [13] May, W.E.; Wise, S.A.; *Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbons in Air Particulate Extracts*; Anal. Chem., Vol. 56, pp. 225–232 (1984).
- [14] Wise, S.A.; Benner, B.A.; Byrd, G.D.; Chesler, S.N.; Rebbert, R.E.; Schantz, M.M.; *Determination of Polycyclic Aromatic Hydrocarbons in a Coal Tar Standard Reference Material*; Anal. Chem., Vol. 60, pp. 887–894 (1988).
- [15] Wise, S.A.; Deissler, A.; Sander, L.C.; *Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbon Isomers of Molecular Weight 278 and 302 in Environmental Standard Reference Materials*; Polycyclic Aromat. Compd., Vol. 3, pp. 169–184 (1993).
- [16] Brubaker, W.W., Jr.; Schantz, M.M.; Wise, S.A.; *Determination of Non-ortho Polychlorinated Biphenyls in Environmental Standard Reference Materials*; Fresenius’ J. Anal. Chem., Vol. 367, pp. 401–406 (2000).

**Certificate Revision History:** 25 April 2022 (Change of period of validity; updated format; editorial changes); 16 January 2015 (Corrected IUPAC name for PCB-56 and PCB-107; editorial changes); 10 June 2014 (Units corrected from mg/kg to µg/kg in Tables 8 and 9; editorial changes); 10 April 2012 (Reference value added for alkylated PAH groups, hopanes, and steranes; extension of certification period; editorial changes); 01 December 2011 (Extension of certification period; editorial changes); 16 August 2004 (Reference values for the butyl tins removed; editorial changes); 15 July 2002 (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.*

*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or the Internet at <https://www.nist.gov/srm>.*

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

# APPENDIX A

**Non-Certified Values:** Non-certified mass fraction values for additional PAHs (some in combination), additional PCB congeners, and additional chlorinated pesticides are provided in Table A1 through Table A4. Non-certified values for alkylated PAH groups are provided in Table A5 and for selected hopanes and steranes in Table A6. A non-certified value for total organic carbon is provided in Table A7. Non-certified values are values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1].

**Additional Non-Certified Mass Fraction Values:** Non-certified mass fraction values are provided in Table A8 for carbon, hydrogen, and nitrogen.

Table A1. Non-certified Mass Fraction Values for PAHs in SRM 1941b

PAHs	Mass Fractions <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )	
1-Methylnaphthalene <sup>(b,c,d,e)</sup>	127	$\pm 14^{(f)}$
2-Methylnaphthalene <sup>(b,c,d,e)</sup>	276	$\pm 53^{(f)}$
2,6-Dimethylnaphthalene <sup>(b,c,d,e)</sup>	75.9	$\pm 4.5^{(f)}$
2,3,5-Trimethylnaphthalene <sup>(b,c,d,e)</sup>	25.5	$\pm 5.1^{(f)}$
Biphenyl <sup>(b,c,d,e)</sup>	74.0	$\pm 8.0^{(f)}$
Acenaphthylene <sup>(b,c,d,e)</sup>	53.3	$\pm 6.4^{(f)}$
Acenaphthene <sup>(b,c,d,e)</sup>	38.4	$\pm 5.2^{(f)}$
9-Methylphenanthrene <sup>(c)</sup>	63.5	$\pm 2.5^{(g)}$
4-Methylphenanthrene and 9-Methylphenanthrene <sup>(b,d)</sup>	80.1	$\pm 4.8^{(f)}$
2-Methylanthracene <sup>(c,d)</sup>	36	$\pm 15^{(f)}$
8-Methylfluoranthene <sup>(b)</sup>	49.5	$\pm 2.7^{(g)}$
7-Methylfluoranthene <sup>(b)</sup>	45.4	$\pm 1.5^{(g)}$
1-Methylfluoranthene <sup>(b)</sup>	42.4	$\pm 2.1^{(g)}$
3-Methylfluoranthene <sup>(b)</sup>	28.8	$\pm 1.3^{(g)}$
2-Methylpyrene <sup>(b)</sup>	78.7	$\pm 4.0^{(g)}$
4-Methylpyrene <sup>(b)</sup>	66.4	$\pm 2.6^{(g)}$
1-Methylpyrene <sup>(b)</sup>	52.5	$\pm 2.3^{(g)}$
Acephenanthrene <sup>(d)</sup>	30.5	$\pm 1.9^{(g)}$
Benzo[ <i>c</i> ]phenanthrene <sup>(b,c,d)</sup>	58	$\pm 15^{(f)}$
Benzo[ <i>a</i> ]fluoranthene <sup>(b,c,d)</sup>	73	$\pm 18^{(f)}$
Benzo[ <i>j</i> ]fluoranthene <sup>(c)</sup>	217	$\pm 5^{(g)}$
Indeno[1,2,3- <i>cd</i> ]fluoranthene <sup>(d)</sup>	9.63	$\pm 0.34^{(g)}$
Pentaphene <sup>(d)</sup>	25.3	$\pm 1.0^{(g)}$

<sup>(a)</sup> Mass fractions reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

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

<sup>(c)</sup> GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

<sup>(d)</sup> GC/MS (III) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(e)</sup> 1999 Interlaboratory Comparison Study [2] with 14 to 26 laboratories submitting data for each PAH.

<sup>(f)</sup> Non-certified values are weighted means of the results from two to four analytical methods [3]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of PAHs listed as determined by the methods indicated. The values are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(g)</sup> Non-certified values are the means 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 for the associated degrees of freedom (19 for footnote b and 5 for footnotes c and d) and 95 % confidence level for each analyte. The measurand is the total mass fraction of the PAHs listed as determined by the method indicated. The values listed are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

Table A2. Non-certified Mass Fraction Values for PAHs of Molecular Mass 300 and 302 in SRM 1941b

PAHs of Molecular Mass 300 and 302	Mass Fractions <sup>(a,b,c)</sup> ( $\mu\text{g}/\text{kg}$ )
Coronene	72.6 $\pm$ 4.7
Dibenzo[ <i>b,e</i> ]fluoranthene	10.3 $\pm$ 0.3
Naphtho[1,2- <i>b</i> ]fluoranthene	91.0 $\pm$ 3.1
Naphtho[1,2- <i>k</i> ]fluoranthene and Naphtho[2,3- <i>j</i> ]fluoranthene	79.8 $\pm$ 2.5
Naphtho[2,3- <i>b</i> ]fluoranthene	23.5 $\pm$ 0.3
Dibenzo[ <i>b,k</i> ]fluoranthene	95.6 $\pm$ 3.1
Dibenzo[ <i>a,k</i> ]fluoranthene	26.6 $\pm$ 0.4
Dibenzo[ <i>j,l</i> ]fluoranthene	63.8 $\pm$ 1.8
Dibenzo[ <i>a,l</i> ]pyrene	11.1 $\pm$ 1.0
Naphtho[2,3- <i>k</i> ]fluoranthene	10.7 $\pm$ 0.6
Naphtho[1,2- <i>a</i> ]pyrene	16.7 $\pm$ 1.4
Naphtho[2,3- <i>e</i> ]pyrene	33.2 $\pm$ 2.3
Dibenzo[ <i>a,e</i> ]pyrene	76.1 $\pm$ 3.6
Naphtho[2,1- <i>a</i> ]pyrene	59.2 $\pm$ 1.8
Dibenzo[ <i>e,i</i> ]pyrene	35.0 $\pm$ 2.4
Naphtho[2,3- <i>a</i> ]pyrene	16.5 $\pm$ 0.6
Benzo[ <i>b</i> ]perylene	38.2 $\pm$ 1.2
Dibenzo[ <i>a,i</i> ]pyrene	25.5 $\pm$ 1.0
Dibenzo[ <i>a,h</i> ]pyrene	6.94 $\pm$ 0.29

<sup>(a)</sup> Mass fractions reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

<sup>(b)</sup> Non-certified values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty,  $U$ , is calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the analyte mean, and the coverage factor,  $k$ , is determined from the Student's  $t$ -distribution for two degrees of freedom and 95 % confidence level for each analyte. The measurand is the total mass fraction of the constituent listed as determined by the method indicated. The values are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(c)</sup> GC/MS on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM [9].

Table A3. Non-certified Mass Fraction Values for PCB Congeners<sup>(a)</sup> in SRM 1941b

PCB Congeners			Mass Fractions <sup>(b,c)</sup> (µg/kg)		
PCB	45	(2,2',3,6-Tetrachlorobiphenyl) <sup>(d,e)</sup>	0.73	±	0.12
PCB	56	(2,3,3',4'-Tetrachlorobiphenyl) <sup>(d,f,g)</sup>	1.21	±	0.11
PCB	63	(2,3,4',5'-Tetrachlorobiphenyl) <sup>(e,f,g)</sup>	0.213	±	0.040
PCB	70	(2,3',4',5'-Tetrachlorobiphenyl) <sup>(e,f,g)</sup>	4.99	±	0.29
PCB	74	(2,4,4',5'-Tetrachlorobiphenyl) <sup>(e,f,g)</sup>	2.04	±	0.15
PCB	77	(3,3',4,4'-Tetrachlorobiphenyl) <sup>(h)</sup>	0.31	±	0.03
PCB	107	(2,3,3',4',5-Pentachlorobiphenyl) <sup>(d,e,f,g)</sup>	0.628	±	0.028
PCB	132	(2,2',3,3',4,6'-Hexachlorobiphenyl) <sup>(d,f,g)</sup>	1.28	±	0.27
PCB	146	(2,2',3,4',5,5'-Hexachlorobiphenyl) <sup>(e,f,g)</sup>	1.22	±	0.12
PCB	158	(2,3,3',4,4',6-Hexachlorobiphenyl) <sup>(d,e,f,g)</sup>	0.65	±	0.15
PCB	163	(2,3,3',4',5,6-Hexachlorobiphenyl) <sup>(e,f,g)</sup>	1.28	±	0.06
PCB	174	(2,2',3,3',4,5,6'-Heptachlorobiphenyl) <sup>(d,e,f,g)</sup>	1.51	±	0.39
PCB	193	(2,3,3',4',5,5',6-Heptachlorobiphenyl) <sup>(d,e,f,g)</sup>	0.292	±	0.075

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [7] and later revised by Schulte and Malisch [8] to conform with IUPAC rules, except PCB 107. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 107 is listed as PCB 108.

<sup>(b)</sup> Mass fractions reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

<sup>(c)</sup> For these PCB congeners except PCB 77, the non-certified values are unweighted means of the results from two to four analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [6] with a pooled within-method variance following the ISO/JCGM Guide [4,5]. For PCB 77, the non-certified 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 two degrees of freedom and 95 % confidence level for PCB 77. The measurand is the total mass fraction of the PCB Congeners listed as determined by the method or methods indicated. The values listed are metrologically traceable to the SI unit of mass, expressed as microgram per kilogram on a dry-mass basis.

<sup>(d)</sup> GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

<sup>(e)</sup> GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).

<sup>(f)</sup> GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

<sup>(g)</sup> GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

<sup>(h)</sup> GC/MS NICI on a 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (I) fractionated using a PYE column.

Table A4. Non-certified Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1941b

Chlorinated Pesticides	Mass Fractions <sup>(a,b)</sup> (µg/kg)		
2,4'-DDE <sup>(c,d)</sup>	0.38	±	0.12
4,4'-DDT <sup>(e,f)</sup>	1.12	±	0.42

<sup>(a)</sup> Mass Fractions reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

<sup>(b)</sup> The non-certified values are unweighted means of the results from two analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [6] with a pooled, within-method variance following the ISO/JCGM Guide [4,5]. The measurand is the total mass fraction of the chlorinated pesticides listed as determined by the methods indicated. The values listed are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(c)</sup> GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

<sup>(d)</sup> GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).

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

<sup>(f)</sup> 1999 Interlaboratory Comparison Study [2] with 10 laboratories submitting data for 4,4'-DDT.



Table A5. Non-certified Mass Fraction Values for Alkylated PAH Groups in SRM 1941b

Alkylated PAH Group	Mass Fraction <sup>(a,b)</sup> (µg/kg)		
C2-decalins	18	±	5
C4-decalins	41	±	4
C2-naphthalenes	187	±	53
C3-naphthalenes	158	±	42
C1-benzothiophenes	25	±	14
C2-benzothiophenes	20	±	11
C3-benzothiophenes	22	±	13
C4-benzothiophenes	18	±	5
C1-fluorenes	57	±	18
C2-fluorenes	122	±	43
C3-fluorenes	128	±	31
C1-phenanthrenes/anthracenes	313	±	99
C2-phenanthrenes/anthracenes	247	±	62
C3-phenanthrenes/anthracenes	165	±	46
C4-phenanthrenes/anthracenes	87	±	36
C1-dibenzothiophenes	54	±	13
C2-dibenzothiophenes	91	±	18
C3-dibenzothiophenes	84	±	15
C4-dibenzothiophenes	57	±	13
C1-fluoranthenes/pyrenes	252	±	48
C2-fluoranthenes/pyrenes	205	±	38
C3-fluoranthenes/pyrenes	102	±	22
C4-fluoranthenes/pyrenes	121	±	59
C1-benzanthracenes/chrysenes/triphenylenes	208	±	43
C2-benzanthracenes/chrysenes/triphenylenes	120	±	24
C3-benzanthracenes/chrysenes/triphenylenes	73	±	31
C4-benzanthracenes/chrysenes/triphenylenes	41	±	11

<sup>(a)</sup> The non-certified mass fraction value reported on a dry-mass basis is the median of results using one analytical technique. The expanded uncertainty,  $U$ , is calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the median, and the coverage factor,  $k = 2$ . The measurand is the total mass fraction of the alkylated PAH groups listed as determined by the interlaboratory study methods. The values listed are metrologically traceable to the SI unit of mass fraction, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(b)</sup> Data from the interlaboratory study [10].

Table A6. Non-certified Mass Fraction Values for Hopanes and Steranes in SRM 1941b

Hopane or Sterane	Mass Fraction <sup>(a,b)</sup> (µg/kg)		
17 $\alpha$ (H)-22,29,30-Trisnorhopane	54	±	18
17 $\alpha$ (H)-21 $\beta$ (H)-30-Norhopane	137	±	21
17 $\alpha$ (H)-21 $\beta$ (H)-30-Hopane	215	±	44
17 $\alpha$ (H)-21 $\beta$ (H)-22R-Homohopane	44	±	10
17 $\alpha$ (H)-21 $\beta$ (H)-22S-Homohopane	48	±	13
5 $\alpha$ (H)-14 $\alpha$ (H),17 $\alpha$ (H)-Cholestane 20R	41	±	11
5 $\alpha$ (H)-14 $\beta$ (H),17 $\beta$ (H)-Cholestane 20R	27	±	6
5 $\alpha$ (H)-14 $\beta$ (H),17 $\beta$ (H)-24-Methylcholestane 20R	21	±	8
5 $\alpha$ (H)-14 $\alpha$ (H),17 $\alpha$ (H)-24-Ethylcholestane 20R	19	±	5
5 $\alpha$ (H)-14 $\beta$ (H),17 $\beta$ (H)-24-Ethylcholestane 20R	41	±	9

<sup>(a)</sup> The non-certified mass fraction value reported on a dry-mass basis is the median of results using one analytical technique. The expanded uncertainty,  $U$ , is calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the median, and the coverage factor,  $k = 2$ . The measurand is the total mass fraction of the constituent listed as determined by the methods used during the interlaboratory study. The values are metrologically traceable to the SI unit of mass, expressed as micrograms per kilogram on a dry-mass basis.

<sup>(b)</sup> Data from the interlaboratory study [10].

Table A7. Non-certified Mass Fraction Value for Total Organic Carbon in SRM 1941b

Total Organic Carbon (TOC)  $2.99 \% \pm 0.24 \%^{(a,b)}$

<sup>(a)</sup> Mass fraction is reported on a dry-mass basis; material as received contains approximately 2.4 % moisture.

<sup>(b)</sup> The non-certified value for total organic carbon is a weighted mean value from routine measurements made by two laboratories [1]. The uncertainty listed is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance. The reporting follows the ISO/JCGM Guides [11]. The measurand is the total mass fraction of TOC listed as determined by the methods indicated. The values listed are metrologically traceable to the SI unit of mass, expressed as a percent on a dry-mass basis.

Table A8. Additional Non-Certified Mass Fraction Values for SRM 1941b

Elements	Mass Fractions <sup>(a)</sup> (%)
Carbon	3.3
Hydrogen	1.2
Nitrogen	<0.5

<sup>(a)</sup> Mass fraction is reported on a dry-mass basis; material as received contains approximately 2.4 % moisture.

**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 Appendix 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

Coordination of the technical measurements leading to the certification of this material was under the leadership of S.A. Wise of the NIST Chemical Sciences Division and M.M. Schantz, formerly of NIST

Analytical measurements for the certification of SRM 1941b were performed at NIST by J.R. Kucklick, D.L. Poster, and L.L. Yu of the NIST Chemical Sciences Division and B.J. Porter, M.M. Schantz, P. Schubert, and S. Tutschku, formerly of NIST.

Measurements for TOC were provided by a commercial laboratory and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX). The carbon, hydrogen, and nitrogen data were provided by a commercial laboratory. Results for the PAHs, PCBs, and chlorinated pesticides from 38 laboratories (see Appendix C) that participated in an interlaboratory comparison exercise coordinated by NIST were used. Results for the alkylated PAH groups, hopanes, and steranes from 33 laboratories (see Appendix D) that participated in another interlaboratory comparison exercise coordinated by NIST were also used.

Collection and preparation of SRM 1941b were performed by M.P. Cronise and C.N. Fales of the NIST Office of Reference Materials and B.J. Porter and M.M. Schantz, formerly of the NIST Chemical Sciences Division. The sediment material was collected with the assistance of G.G. Lauenstein, J. Collier, and J. Lewis (National Oceanic and Atmospheric Administration, Silver Spring, MD).

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh and J.H. Yen of the NIST Statistical Engineering Division.

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

**Sample Collection and Preparation:** The sediment used to prepare this SRM was collected from the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge (39°12.3'N and 76°31.4'W). This location is very near the site where SRM 1941 and SRM 1941a were collected. The sediment was collected using a Kynar-coated modified Van Veen-type grab sampler. A total of approximately 3300 kg of wet sediment was collected from the site. The sediment was freeze-dried, sieved at 150  $\mu\text{m}$  (100 % passing), homogenized in a cone blender, radiation sterilized ( $^{60}\text{Co}$ ), and then packaged in screw-capped amber glass bottles each containing approximately 50 g.

**Conversion to Dry-Mass Basis:** The results for the constituents in SRM 1941b are reported on a dry-mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1941b was determined by measuring the mass loss after freeze-drying subsamples of 1.1 g to 1.3 g for four days at 1 Pa with a  $-10\text{ }^{\circ}\text{C}$  shelf temperature and a  $-50\text{ }^{\circ}\text{C}$  condenser temperature. The moisture content in SRM 1941b at the time of the certification analyses was  $2.39\% \pm 0.08\%$  (95 % confidence level). Analytical results for the organic constituents were determined on an as-received basis and then converted to a dry-mass basis by dividing by the conversion factor of 0.9761 (gram dry mass per gram as-received mass).

**Polycyclic Aromatic Hydrocarbons:** The general approach used for the value assignment of the PAHs in SRM 1941b was similar to that reported in detail elsewhere [11]. The approach consisted of combining results from analyses using various combinations of different extraction techniques and solvents, clean-up/isolation procedures, and chromatographic separation and detection techniques: Soxhlet extraction and pressurized-fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using solid-phase extraction (SPE) or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (all column compositions are given as mole fractions in %) phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, and a relatively non-polar proprietary phase.

Three sets of GC/MS results, designated as GC/MS (I), GC/MS (II), and GC/MS (III), were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of approximately 1 g from ten bottles of SRM 1941b were extracted using PFE with DCM. Copper powder was added

to the extract to remove elemental sulfur. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 2 % DCM in hexane (all solvent concentrations are given as volume fractions in %). The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 5 g subsamples from six bottles of SRM 1941b. These samples were extracted using PFE with DCM. The high molecular mass compounds were removed from the extracts using size exclusion chromatography (SEC) with a preparative-scale divinylbenzene-polystyrene column (10 µm particle size with 10 nm diameter pores), and the sulfur was removed from the extracts by adding copper powder. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The analysis was by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-17 MS, J&W Scientific). For the GC/MS (III), 9 g subsamples from six bottles of SRM 1941b were Soxhlet-extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur, and the concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness; DB-XLB, J&W Scientific).

Two sets of LC-FL results, designated as LC-FL (total) and LC-FL (isomer), were used in the certification process. For the LC-FL (total), subsamples of approximately 1 g from six bottles of SRM 1941b were extracted using PFE with a mixture of 50 % hexane/50 % acetone. The extracts were concentrated and then processed through an aminopropylsilane SPE cartridge using 2 % DCM in hexane to obtain the total PAH fraction. For the LC-FL (isomer), a 5 g subsample from the six bottles was extracted using PFE with DCM and processed through an aminopropylsilane SPE cartridge using 10 % DCM in hexane; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (µBondapak NH<sub>2</sub>, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [12–15]. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5 µm particle-size polymeric octadecylsilane (C<sub>18</sub>) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength-programmed fluorescence detection [13,14].

For the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1941b was used in an interlaboratory comparison exercise in 1999 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [2]. Results from 38 laboratories that participated in this exercise were used as the sixth data set in the determination of the certified values for PAHs in SRM 1941b. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure the analytes of interest.

**Homogeneity Assessment for PAHs:** The homogeneity of SRM 1941b was assessed by analyzing duplicate samples of approximately 1 g from ten bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at this sample size.

**PAH Isomers of Molecular Mass 300 and 302:** For the determination of the molecular mass 300 and 302 isomers, three subsamples of approximately 5 g each were extracted using PFE with DCM. The extracts were then concentrated with a solvent change to hexane and passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-17MS, J&W Scientific). Perdeuterated dibenzo[*a,i*]pyrene was added to the sediment prior to extraction for use as an internal standard [9].

**PCBs and Chlorinated Pesticides:** The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1941b consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. Techniques and solvents included Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, clean-up/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity for the separation of PCBs and chlorinated pesticides. The analytical methods are described in detail elsewhere [11].

Six sets of results were obtained and designated as GC-ECD (I) A and B, GC/MS (I) A and B, GC/MS (II), and Interlaboratory Comparison Exercise. For the GC-ECD (I) analyses, approximately 10 g subsamples from six bottles of SRM 1941b were extracted using PFE with DCM. Copper powder was added to the extract to remove elemental sulfur, and SEC, as described above, was used to remove the high molecular mass compounds. The concentrated extract was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing:

(1) the PCBs and lower-polarity pesticides and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-5, J&W Scientific), and a 0.25 mm × 60 m fused silica capillary column with a non-polar proprietary phase (0.25 µm film thickness; DB-XLB, J&W Scientific). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the proprietary phase are designated as GC-ECD (IB). For the GC-ECD analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [7,8]) and endosulfan I-*d*<sub>4</sub>, 4,4'-DDE-*d*<sub>8</sub>, 4,4'-DD-*d*<sub>8</sub>, and 4,4'-DDT-*d*<sub>8</sub> were added to the sediment prior to extraction for use as internal standards for quantification purposes.

Two sets of results were obtained by GC/MS. For GC/MS (I), approximately 9 g subsamples from six bottles were Soxhlet- extracted with a mixture of 50 % hexane/50 % acetone for approximately 18 h. Copper powder was added to the extract to remove elemental sulfur, and the concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS with two ionization modes, electron impact (EI) and negative ion chemical ionization (NICI). The GC/MS EI method, GC/MS (IA), used a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness; DB-XLB, J&W Scientific). The GC/MS NICI method, GC/MS (IB), used a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness; DB-5MS, J&W Scientific). The GC/MS (II) results were obtained in the same manner as the GC/MS (IA) analyses except that three subsamples were Soxhlet-extracted with DCM for approximately 18 h. For the GC/MS analyses, selected carbon-13 labeled PCB congeners and chlorinated pesticides were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1941b was used in an interlaboratory comparison exercise in 1999 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [7]. Results from 38 laboratories that participated in this exercise were used as the sixth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1941b. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure the analytes of interest.

The non-certified value for PCB 77 was determined from a separate fraction. The samples were extracted and processed as for GC-ECD (I) above. The first (PCB and lower-polarity pesticide) fraction from the semi-preparative aminopropylsilane column was further fractionated using a Cosmosil PYE (pyrenylethyl group bonded) column (5 µm particle size, 4.6 mm i.d. × 25 cm ; Phenomenex, Torrance, CA) [16]. Three fractions were collected: the first fraction contained the pesticides and multi-*ortho* PCBs, the second fraction contained the polychlorinated naphthalenes, non-*ortho* PCB congeners, and some mono-*ortho* PCB congeners, and the third fraction removed the residual planar compounds from the column. The second fraction was analyzed by GC/MS NICI using the same column as GC/MS (IB) above. Carbon-13 labeled PCB 77 was used as an internal standard for quantification purposes.

**Alkylated PAH Groups, Hopanes, and Steranes:** SRM 1941b was used in an interlaboratory comparison exercise in 2011 [10]. Results from 33 laboratories that participated in this exercise were used in the determination of the non-certified values for alkylated PAH groups, hopanes, and steranes in SRM 1941b. Note that not all laboratories returned data for each analyte. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure the analytes of interest. For the alkylated PAHs, the majority of the laboratories (>90 %) used the parent PAH for determination of the response factor for the corresponding alkylated group.

**Total Organic Carbon (TOC):** Two laboratories provided results for TOC using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6 mol/L hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed subsamples from three bottles of SRM 1941b. One of the laboratories also analyzed three subsamples from three bottles of SRM 1941b for carbon, hydrogen, and nitrogen.

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

# APPENDIX C

The laboratories listed below performed measurements that contributed to the certification of PAHs, PCBs, and chlorinated pesticides in SRM 1941b Organics in Marine Sediment.

- Arthur D. Little, Inc (Cambridge, MA, USA)
- Axys Analytical Services (Sidney, BC, Canada)
- B & B Laboratories (College Station, TX, USA)
- Battelle Ocean Sciences (Duxbury, MA, USA)
- Bedford Institute of Oceanography (Dartmouth, NS, Canada)
- California Department of Fish and Game (Rancho Cordova, CA, USA)
- Central Contra Costa Sanitary District (Martinez, CA, USA)
- Chesapeake Biological Laboratory (Solomons, MD, USA)
- Centro de Investigaciones Energeticas Medioambientales y Tecnologicas (Madrid, Spain)
- City of Los Angeles Environmental Monitoring Division (Playa del Rey, CA, USA)
- City of San Jose Environmental Services Department (San Jose, CA, USA)
- Columbia Analytical Services (Kelso, WA, USA)
- East Bay Municipal Utility District (Oakland, CA, USA)
- Florida Department of Environmental Protection (Tallahassee, FL, USA)
- Manchester Environmental Laboratory (Port Orchard, WA, USA)
- Murray State University (Murray, KY, USA)
- Massachusetts Water Resources Authority Central Lab (Winthrop, MA, USA)
- National Research Council of Canada (Ottawa, Ontario, Canada)
- National Oceanic and Atmospheric Association (NOAA), National Marine Fisheries Service (NMFS), Auke Bay Laboratory (Juneau, AK, USA)
- NOAA, National Ocean Service/Center for Coastal Environmental Health and Biomolecular Research (Charleston, SC, USA)
- NOAA, NMFS, Sandy Hook Marine Laboratory (Highlands, NJ, USA)
- NOAA, NMFS, Northwest Fisheries Science Center (Seattle, WA, USA)
- Orange County Sanitation District (Fountain Valley, CA, USA)
- Philip Analytical Services (Burlington, Ontario, Canada)
- Serv de Hidrografia Naval (Buenos Aires, Argentina)
- Skidaway Institute of Technology (Savannah, GA, USA)
- Southwest Laboratory of Oklahoma (Broken Arrow, OK, USA)
- Sewern Trent Knoxville Laboratory (Knoxville, TN, USA)
- Texas A&M University, Geochemical and Environmental Research Group (College Station, TX, USA)
- Texas Parks and Wildlife Department (San Marcos, TX, USA)
- University of California at Los Angeles, Institute of Geophysics and Planetary Physics (Los Angeles, CA, USA)
- University of Connecticut, Environmental Research Institute (Storrs, CT, USA)
- University of Rhode Island, Graduate School of Oceanography (Narragansett, RI, USA)
- US Department of Agriculture, Environmental Chemistry Laboratory (Beltsville, MD, USA)
- US Environmental Protection Agency, Atlantic Ecology Division (Narragansett, RI, USA)
- US Geological Survey, National Water Quality Laboratory (Denver, CO, USA)
- Woods Hole Group Environmental Lab (Raynham, MA, USA)
- Wright State University (Dayton, OH, USA)

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

# APPENDIX D

The laboratories listed below performed measurements that contributed to the certification of alkylated PAH groups, hopanes, and steranes in SRM 1941b Organics in Marine Sediment.

Alpha Analytical, Inc.; Mansfield, MA  
Analytical Resources, Inc.; Tukwila, WA  
Axys Analytical Services; Sydney, BC, Canada  
Battelle Analytical & Environmental Chemistry Laboratory; Duxbury, MA  
Center for Laboratory Sciences; Pasco, WA  
Columbia Analytical Services; Jacksonville, FL  
Columbia Analytical Services; Rochester, NY  
Columbia Analytical Services, Kelso, WA  
Florida Department of Environmental Protection; Tallahassee, FL  
Florida International University; North Miami, FL  
Michigan Department of Natural Resources and Environment; Lansing, MI  
Mississippi State Chemical Laboratory; Mississippi State, MS  
NIST; Charleston, SC  
NIST; Gaithersburg, MD  
NOAA/NCCOS/NOS; Charleston, SC  
NOAA/NMFS/Alaska Fisheries Science Center; Juneau, AK  
NY State Department of Health; Albany, NY  
Pace Analytical Services, Inc. Minneapolis; Minneapolis, MN  
RJ Lee Group, Inc; Monroeville, PA  
TDI/B&B Laboratories, Inc.; College Station, TX  
TestAmerica Laboratories; Mobile, AL  
TestAmerica Laboratories; West Sacramento, CA  
TestAmerica Laboratories; University Park, IL  
TestAmerica Laboratories; Schriever, LA  
TestAmerica Laboratories; Edison, NJ  
TestAmerica Laboratories; Knoxville, TN  
TestAmerica Laboratories; Pittsburgh, PA  
TestAmerica Laboratories; South Burlington, VT  
TestAmerica Laboratories; Tacoma, WA  
US Army Engineer Research and Development Center; Vicksburg, MS  
USGS Columbia Environmental Research Center; Columbia, MO  
University of Iowa, State Hygienic Laboratory; Iowa City, IO  
Washington State Public Health Laboratories; Shoreline, WA

\*\*\*\*\* End of Appendix D \*\*\*\*\*