



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1941a

Organics in Marine Sediment

Standard Reference Material (SRM) 1941a is a marine sediment intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and sulfur. A unit of SRM 1941a contains approximately 50 g of dry sediment in one screw-capped amber glass bottle.

Certified values are provided in Tables 1-3 for the concentrations of 23 PAHs, 21 PCB congeners, 6 chlorinated pesticides, and sulfur naturally present in the sediment. The certified values for the PAHs (Table 1) are based on the results obtained from the analyses of this material using different extraction/sample preparation procedures and analytical techniques based on gas chromatography with mass spectrometric detection (GC-MS) on two stationary phases of different selectivity and reversed-phase liquid chromatography with fluorescence detection (LC-FL). The certified values for the PCB congeners and chlorinated pesticides (Table 2) are based on the results obtained from the analyses of this material using different extraction/sample preparation procedures and analytical techniques based on gas chromatography with electron capture detection (GC-ECD) on two stationary phases of different selectivity and GC-MS. The certified value for sulfur content of SRM 1941a (Table 3) is based on measurements by isotope dilution thermal ionization mass spectrometry (ID-TIMS).

Noncertified concentrations are provided in Appendix A for 14 additional PAHs and in Appendix B for 7 additional PCB congeners and 3 additional chlorinated pesticides. Noncertified concentrations are provided in Appendix C for 17 aliphatic hydrocarbons. Noncertified concentrations for 27 inorganic constituents are provided in Appendix D and a noncertified value for percent total organic carbon is provided in Appendix E. Summaries of the analytical results obtained by using the different analytical techniques during the certification are provided in Appendices F, G, and H for the PAHs, PCB congeners, and chlorinated pesticides, respectively.

The collection, preparation, and certification of SRM 1941a were supported in part by the Coastal Monitoring and Bioeffects Assessment Division, National Ocean Service, National Oceanic and Atmospheric Administration (NOAA).

Collection and preparation of the SRM were performed by M.M. Schantz and S.A. Wise of the NIST Organic Analytical Research Division and by M.P. Cronise and C.N. Fales of the NIST Standard Reference Materials Program. The sediment was collected with the assistance of G.G. Lauenstein (NOAA) and the U.S. Coast Guard.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.B. Schiller of the NIST Statistical Engineering Division. The coordination of the technical measurements leading to certification was under the direction of M.M. Schantz and S.A. Wise.

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

Gaithersburg, MD 20899
March 30, 1994

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

Analytical measurements were performed in the NIST Organic Analytical Research Division by B.A. Benner, Jr., M.J. Hays, B.J. Koster, R.E. Rebbert, and M.M. Schantz, and in the NIST Inorganic Analytical Research Division by R. Demiralp, R.R. Greenberg, W.R. Kelly, and R.D. Vocke, Jr. Measurements for percent total organic carbon were provided by two commercial laboratories and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University.

NOTICE AND WARNING TO USERS

Handling: This material consists of fine particles (150-250 μm); therefore, the transfer of the material should be performed with care. Use proper methods for disposal of wastes.

Expiration of Certification: This certification is valid within the specified uncertainty limits for three years from the date of purchase. In the event that the certification should become invalid before then, purchasers will be notified by NIST. Please return the attached registration form to facilitate notification.

Storage: The sediment is provided in an amber glass bottle and should be stored in the dark at temperatures less than 30 °C.

Use: Prior to analysis, the bottle should be shaken. Samples should be withdrawn from the bottle immediately after opening and used without delay for the certified values listed in Tables 1-3 to be valid within the stated uncertainties. The bottle should be tightly closed immediately after removal of the subsamples; the remaining material may be used for subsequent analyses. While analyses are typically performed on the sediment as received, the concentrations of constituents in SRM 1941a are reported on a dry weight basis. The SRM sediment, as received, contains approximately 2.2% moisture. Therefore, a separate aliquot of the SRM should be removed from the bottle at the time of analysis and dried to determine the moisture content and analytical results should be corrected to the dry weight basis (see Moisture Determination section).

PREPARATION AND ANALYSIS

SRM Preparation: The marine sediment used to prepare this SRM was collected in the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge (39°12.68'N and 76°31.33'W). The sediment was collected using a modified Van Veen type grab sampler designed to sample the sediment to a depth of 10 cm. The sediment was freeze dried, sieved (150-250 μm particles used for the SRM), homogenized in a cone blender, radiation (^{60}Co) sterilized, and then packaged in screw-capped amber glass bottles (~50 g/bottle).

Moisture Determination: The results for both the organic and inorganic constituents in SRM 1941a are reported on a dry weight basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1941a was determined by measuring the weight loss after oven drying at 90 °C for 18 h for subsamples of 1-2 g. The moisture content in SRM 1941a at the time of the certification analyses was approximately 2.21%.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1941a was assessed by analyzing duplicate samples of 10 g each from 10 randomly selected bottles. Samples were extracted, processed, and analyzed as described below for the GC-MS analyses (5% phenyl-substituted methylpolysiloxane phase). No statistically significant differences between bottles were observed for the PAHs at the 10 g sample size.

Polycyclic Aromatic Hydrocarbons: SRM 1941a was analyzed for selected PAHs using gas chromatography with mass spectrometric detection (GC-MS) and reversed-phase liquid chromatography with fluorescence detection (LC-FL). GC-MS analyses were performed on two columns with different selectivities for the separation of PAHs: a 5% phenyl-substituted methylpolysiloxane stationary phase and a smectic liquid crystalline stationary phase. Two different sample preparation/cleanup procedures were used prior to the analysis by GC-MS and LC-FL. A similar approach was used previously for the certification of PAHs in SRM 1941. [1]

For the GC-MS analyses, two sets of six samples (10-25 g each) from twelve randomly selected bottles were Soxhlet extracted for 18-20 h using methylene chloride. A silica solid phase extraction column was used to remove the polar interferences from each sediment extract. Finely divided copper was then added to the extracts to remove elemental sulfur. The PAH fraction was isolated from each sediment extract by normal-phase liquid chromatography using a semi-preparative aminopropylsilane column. [2] The GC-MS analyses were performed on one set of six sample extracts using a 0.25 mm i.d. x 60 m fused silica capillary column with a 5% phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness), (DB-5 MS, J&W Scientific, Folsom, CA). The second set of six sample extracts was prepared as described above and then analyzed by GC-MS on a 0.2 mm i.d. x 25 m fused silica capillary column with a smectic liquid crystalline phase (0.15 μ m film thickness) (SB-Smectic, Dionex, Inc., Salt Lake City, UT).

For the LC-FL analyses, subsamples of 10 g of sediment from six randomly selected bottles were Soxhlet extracted for 20 h using *n*-hexane:acetone (1:1 v/v). Each concentrated extract was placed on an aminopropylsilane solid phase extraction column and eluted with 2% methylene chloride in *n*-hexane to remove the polar constituents. The extract was then analyzed by reversed-phase LC using a polymeric octadecylsilane (C₁₈) column (4.6 mm i.d. x 25 cm, 5 μ m particle size, Hypersil PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [3-5]; these results are designated as LC-FL (Total) in Appendix F. To quantify several PAHs that have low fluorescence sensitivity and/or selectivity, six additional subsamples were extracted and prepared as described above, and then the extract was fractionated on a semi-preparative aminopropylsilane column to isolate isomeric PAH fractions as described previously. [3-5] These isomeric PAH fractions were analyzed by reversed-phase LC-FL on a similar octadecylsilane column; these results are designated as LC-FL (Fractions) in Appendix F.

For both the GC-MS and LC-FL analyses, selected perdeuterated PAHs were added to the sediment prior to extraction for use as internal standards for quantification purposes. Calibration response factors for the analytes relative to the internal standards were determined by analyzing aliquots of SRMs 1491 and 2260 (Aromatic Hydrocarbons in Hexane/Toluene), in the case of GC-MS analyses and SRM 1647b (Polycyclic Aromatic Hydrocarbons in Acetonitrile), in the case of LC-FL analyses; gravimetrically prepared solutions of additional analytes not contained in SRMs 1491, 1647b, or 2260; and the internal standards.

PCBs and Chlorinated Pesticides: SRM 1941a was analyzed for selected PCB congeners and chlorinated pesticides using GC-ECD and GC-MS using the general approach described previously. [6] For the GC-ECD analyses, a portion of the extract prepared for the PAH analyses by GC-MS (see above) was used. For the GC-MS analyses, additional samples of ~100 g each from six pairs of randomly selected bottles were Soxhlet extracted for 18 h using 1:1 *n*-hexane/acetone (v/v). The extracts were fractionated on the 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 selectivity for PCB separations: 0.25 mm x 60 m fused silica capillary column with a 5% phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness) and 0.32 mm x 100 m fused silica capillary column with a dimethylpolysiloxane phase containing 50% methyl C-18 (0.1 μ m film thickness), (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands). The GC-MS analyses were performed on a 5% phenyl-substituted methylpoly-siloxane phase as described above for the GC-ECD analyses.

Two PCB congeners, which are not significantly present in the sediment extract (PCB 103 and PCB 198 [7,8]), and perdeuterated 4,4'-DDT were added to the sediment prior to extraction for use as internal standards for quantification purposes. Calibration response factors for the analytes relative to the internal standards were determined by analyzing aliquots of SRMs 2261 (Concentrated Chlorinated Pesticides in Hexane), SRM 2262 (Concentrated PCB Congeners in Iso-octane), gravimetrically prepared solutions of additional analytes not contained in SRMs 2261 and 2262, and the internal standards.

Aliphatic Hydrocarbons: Ten sediment extracts prepared for the homogeneity study of PAHs by GC-MS and six extracts prepared for the measurement of PAHs by GC-MS were analyzed on the same 5% phenyl-substituted methylpolysiloxane column for the determination of aliphatic hydrocarbons. Perdeuterated dodecane, eicosane, and triacontane were added prior to extraction as internal standards for quantification purposes. Calibration response factors for the analytes relative to the internal standards were determined by analyzing SRM 1494 (Aliphatic Hydrocarbons in 2,2,4-Trimethylpentane).

Sulfur Determination: The general procedure for the determination of sulfur by isotope dilution thermal ionization mass spectrometry (ID-TIMS) has been described in detail previously. [9,10] A subsample of ~100 mg of SRM 1941a was removed from each of six different bottles and added to Pyrex Carius tubes along with enriched ^{34}S tracer and NIST high-purity nitric acid. The Carius tubes were then sealed and heated at 240 °C for 16 h. This procedure oxidizes all sulfur to sulfate and completely equilibrates the spike, which is in the sulfate form, with the sulfur in the sample. The sulfate formed in the samples was reduced to H_2S , which was then precipitated as As_2S_3 . The As_2S_3 was dissolved in aqueous ammonia and a small amount of this solution, equivalent to about 1.5 μg S, was mixed with silica gel on a rhenium filament and the sulfur was determined as the AsS^+ molecular ion.

Additional Inorganic Constituents: SRM 1941a was analyzed for 27 inorganic constituents using instrumental neutron activation analysis (INAA). The INAA was performed using previously developed procedures for sequential instrumental multi-element determinations in biological materials. [11,12] Two subsamples (~300 mg) from each of six bottles of the sediment were pelletized and analyzed using the sequential INAA procedure.

Total Organic Carbon Determination: Three laboratories provided results for the Total Organic Carbon (TOC) measurements using similar procedures. Briefly, subsamples of ~200 mg were reacted with 6 N hydrochloric acid to remove carbonate, and rinsed with deionized water prior to combustion of the residue 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.

REFERENCES

- [1] Schantz, M.M., Benner, B.A., Jr., Chesler, S.N., Koster, B.J., Stone, S.F., Zeisler, R., and Wise, S.A., Preparation and Analysis of a Marine Sediment Reference Material for the Determination of Organic Contaminants, **Fresenius J. Anal. Chem.** **338**, 501-514 (1990).
- [2] Wise, S.A., Chesler, S.N., Hertz, H.S., Hilpert, L.R., and May, W.E., Chemically-Bonded Aminosilane Stationary Phase for the High Performance Liquid Chromatographic Separation of Polynuclear Aromatic Hydrocarbons, **Anal. Chem.** **49**, 2306-2310 (1977).
- [3] May, W.E. and Wise, S.A., Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbons in Air Particulate Extracts, **Anal. Chem.** **56**, 225-232 (1984).
- [4] Kline, W.F., Wise, S.A., and May, W.E., The Application of Perdeuterated Polycyclic Aromatic Hydrocarbons (PAH) as Internal Standards for the Liquid Chromatographic Determination of PAH in Petroleum Crude Oil and Other Complex Mixtures, **J. Liquid Chromatogr.** **8**, 223-237 (1985).
- [5] Wise, S.A., Benner, B.A., Byrd, G.D., Chesler, S.N., Rebbert, R.E., and Schantz, M.M., Determination of Polycyclic Aromatic Hydrocarbons in a Coal Tar Standard Reference Material, **Anal. Chem.** **60**, 887-894 (1988).
- [6] Schantz, M.M., Parris, R.M., Kurz, J., Ballschmiter, K., and Wise, S.A., Comparison of Methods for Gas-Chromatographic Determination of PCB Congeners and Chlorinated Pesticides in Marine Reference Materials, **Fresenius J. Anal. Chem.** **346**, 766-778 (1993).
- [7] Ballschmiter, K. and Zell, M., Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography, **Fresenius Z. Anal. Chem.** **302**, 20-31 (1980).
- [8] Schulte, E. and 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.** **314**, 545-551 (1983).
- [9] Paulsen, P.J. and Kelly, W.R., Determination of Sulfur as Arsenic Monosulfide Ion by Isotope Dilution Thermal Ionization Mass Spectrometry, **Anal. Chem.** **56**, 708-713 (1984).
- [10] Kelly, W.R. and Paulsen, P.J., Precise and Accurate Determination of High Concentrations of Sulfur by Isotope Dilution Thermal Ionization Mass Spectrometry, **Talanta** **31**, 1063-1068 (1984).
- [11] Zeisler, R., Stone, S.F., and Sanders, R.W., Sequential Determination of Biological and Pollutant Elements in Marine Bivalves, **Anal. Chem.** **60**, 2760-2765 (1988).
- [12] Stone, S.F., Koster, B.J., and Zeisler, R., Instrumental Neutron Activation Analysis of SRM 1941 "Organics in Marine Sediment": Elemental Content and Homogeneity, **Biol. Trace Elem. Res.** **26/27**, 579-587 (1990).
- [13] Schiller, S.B. and Eberhardt, K.R., Combining Data from Independent Chemical Analysis Methods, **Spectrochimica Acta**, **46B** (12), 1607-1613 (1991).

Table 1. Certified Concentrations of PAHs in SRM 1941a

Compound	Concentration	
	$\mu\text{g/kg}$ (dry weight) ^{a,b}	
Naphthalene	1010	± 140
Fluorene	97.3	± 8.6
Phenanthrene	489	± 23
Anthracene	184	± 14
Fluoranthene	981	± 78
Pyrene	811	± 24
Benz[<i>a</i>]anthracene	427	± 25
Chrysene	380	± 24
Triphenylene	197	± 11
Benzo[<i>b</i>]fluoranthene	740	± 110
Benzo[<i>k</i>]fluoranthene	361	± 18
Benzo[<i>a</i>]fluoranthene	118	± 11
Benzo[<i>e</i>]pyrene	553	± 59
Benzo[<i>a</i>]pyrene	628	± 52
Perylene	452	± 58
Benzo[<i>ghi</i>]perylene	525	± 67
Indeno[1,2,3- <i>cd</i>]pyrene	501	± 72
Dibenz[<i>a,i</i>]anthracene	74.3	± 6.8
Dibenz[<i>a,c</i>]anthracene	43.1	± 3.7
Dibenz[<i>a,h</i>]anthracene	73.9	± 9.7
Pentaphene	42	± 12
Benzo[<i>b</i>]chrysene	99	± 20
Picene	80.0	± 9.0

^a Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture.

^b The certified values are weighted means of results from two or more analytical techniques as described by Schiller and Eberhardt.[13] The uncertainty is based on a 95% confidence interval for the true concentration, and includes an allowance for differences among the analytical methods used.

Table 2. Certified Concentrations of PCB Congeners and Chlorinated Pesticides in SRM 1941a

Polychlorinated Biphenyls ^a		Concentration μg/kg (dry weight) ^{b,c}	
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl)	4.80	± 0.62
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl)	9.5	± 2.1
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)	6.89	± 0.56
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)	6.8	± 1.4
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)	6.70	± 0.37
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)	7.5	± 1.1
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)	4.17	± 0.51
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)	11.0	± 1.6
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)	3.65	± 0.27
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)	9.47	± 0.85
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)	10.0	± 1.1
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	1.87	± 0.32
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	13.38	± 0.97
163	(2,3,3',4',5,6-Hexachlorobiphenyl)		
164	(2,3,3',4',5',6-Hexachlorobiphenyl)		
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)	9.2	± 1.1
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	17.6	± 1.9
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)	0.93	± 0.14
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	3.00	± 0.46
190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)		
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	5.83	± 0.58
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	1.78	± 0.23
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	3.67	± 0.87
PCB 209	(Decachlorobiphenyl)	8.34	± 0.49
Chlorinated Pesticides			
Hexachlorobenzene		70	± 25
<i>cis</i> -Chlordane (α-Chlordane)		2.33	± 0.56
<i>trans</i> -Nonachlor		1.26	± 0.13
2,4'-DDE		0.73	± 0.11
4,4'-DDE		6.59	± 0.56
4,4'-DDD		5.06	± 0.58

^a 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; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

^b Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture.

^c The certified values are weighted means of results from two analytical techniques as described by Schiller and Eberhardt.[13] The uncertainty is based on a 95% confidence interval for the true concentration, and includes an allowance for differences between the analytical methods used.

Table 3. Certified Concentration of Sulfur in SRM 1941a

Sulfur	$0.9589 \pm 0.0058\%$ (by weight) ^{a,b}
--------	--

^a Concentration is reported on a dry weight basis; material as received contains approximately 2.2% moisture.

^b The uncertainty was calculated according to NIST Technical Note 1297 "Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results" and has an approximate level of confidence of 95%.

SUPPLEMENTAL INFORMATION

Noncertified Quantitative Values: Appendices A through H contain supplementary analytical results obtained during the course of the certification of SRM 1941a. Noncertified concentration values are listed in Appendices A and B for additional PAHs, PCB congeners, and chlorinated pesticides. Noncertified concentrations are listed in Appendix C for selected aliphatic hydrocarbons, in Appendix D for selected inorganic constituents, and in Appendix E for total organic carbon (TOC). The noncertified values reported in these appendices are the results obtained by the measurement technique(s) indicated and may include unrecognized bias; therefore, they are provided for information only. NIST does not recommend that this information be used for calibration, bias evaluation, or similar purposes for which certified values are used. Appendices F, G, and H contain summaries of the analytical results obtained using the various analytical techniques for the determination of PAHs, PCB congeners, and chlorinated pesticides, respectively.

APPENDIX A

Noncertified Concentrations of PAHs in SRM 1941a

NOTE: The noncertified values have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the noncertified concentrations should be useful for comparison with results obtained using similar procedures (i.e., Soxhlet extraction and GC-MS or LC-FL analyses on similar columns).

Polycyclic Aromatic Hydrocarbons	Concentration $\mu\text{g/kg}$ (dry weight) ^{a,b}	
Riphenyl ^{c,d}	175	± 18
Acenaphthene ^{c,d}	41	± 10
Acenaphthylene ^{c,d}	37	± 14
Dibenzothiophene ^{c,d}	70.0	± 9.4
3-Methylphenanthrene ^{c,d}	97	± 32
2-Methylphenanthrene ^{c,d}	158	± 32
1-Methylphenanthrene ^{c,d}	101	± 27
4H-Cyclopenta[def]phenanthrene ^{c,d}	92	± 15
Acephenanthrylene ^{c,d}	48.1	± 1.2
Benzo[ghi]fluoranthene ^d	97.9	± 3.1
Benzo[c]phenanthrene ^{c,d}	80	± 39
Benzo[j]fluoranthene ^d	341	± 22
Indeno[1,2,3-cd]fluoranthene ^c	20.0	± 2.3
Anthanthrene ^{c,e}	129	± 10

^a Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture.

^b Concentrations are the mean determined by the technique indicated or the weighted mean from the results of two techniques. Uncertainties for the measurements are 95% confidence interval for the mean plus an allowance for differences between the analytical methods when two methods were used; six samples were extracted and analyzed in duplicate using the technique(s) indicated.

^c Concentration determined by GC-MS on DB-5 column.

^d Concentration determined by GC-MS on SB-Smectic column.

^e Concentration determined by LC-FL.

APPENDIX B

Noncertified Concentrations of PCB Congeners and Chlorinated Pesticides in SRM 1941a

NOTE: The noncertified values have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the noncertified concentrations should be useful for comparison with results obtained using similar procedures (i.e., Soxhlet solvent extraction and GC-ECD or GC-MS on a similar column).

Polychlorinated Biphenyls ^a		Concentration $\mu\text{g/kg}$ (dry weight) ^{b,c}
PCB 8	(2,4'-Dichlorobiphenyl) ^d	1.39 $\pm$ 0.19
PCB 18	(2,2',5-Trichlorobiphenyl) ^d	1.15 $\pm$ 0.16
PCB 28	(2,4,4'-Trichlorobiphenyl) ^d	9.8 $\pm$ 3.7
PCB 31	(2,4',5-Trichlorobiphenyl) ^d	6.2 $\pm$ 2.4
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl) ^c	2.62 $\pm$ 0.22
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl) ^c	1.63 $\pm$ 0.15
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl) ^{d,e}	7.0 $\pm$ 2.6
182	(2,2',3,4,4',5,6'-Heptachlorobiphenyl)	

Chlorinated Pesticides

Oxychlordan ^d	2.59 $\pm$ 0.19
Dieldrin ^d	1.26 $\pm$ 0.37
4,4'-DDT ^d	1.25 $\pm$ 0.10

^a 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; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

^b Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture.

^c Concentrations are the mean values determined by the technique(s) indicated; subsamples from six bottles were extracted and analyzed in duplicate; uncertainties for the measurements are a 95% confidence interval for the mean plus an allowance for differences between the analytical methods when two methods were used.

^d Concentration determined by GC-ECD on C-18 column.

^e Concentration determined by GC-MS on DB-5 column.

APPENDIX C

Noncertified Concentrations of Aliphatic Hydrocarbons in SRM 1941a

NOTE: The noncertified values have not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedures used, the noncertified concentrations should be useful for comparison with results obtained using similar procedures (i.e., Soxhlet solvent extraction and GC-MS on a similar column).

Compound	Concentration $\mu\text{g/kg}$ (dry weight) ^{a,b}
<i>n</i> -Decane (<i>n</i> -C ₁₀)	230 ± 15
<i>n</i> -Dodecane (<i>n</i> -C ₁₂)	164 ± 10
<i>n</i> -Tridecane (<i>n</i> -C ₁₃)	207 ± 25
<i>n</i> -Tetradecane (<i>n</i> -C ₁₄)	264 ± 35
<i>n</i> -Pentadecane (<i>n</i> -C ₁₅)	470 ± 120
<i>n</i> -Hexadecane (<i>n</i> -C ₁₆)	147 ± 19
<i>n</i> -Heptadecane (<i>n</i> -C ₁₇)	269 ± 38
Pristane (2,6,10,14-Tetramethylpentadecane)	61 ± 25
<i>n</i> -Octadecane (<i>n</i> -C ₁₈)	151 ± 15
Phytane (2,6,10,14-Tetramethylhexadecane)	74 ± 22
<i>n</i> -Nonadecane (<i>n</i> -C ₁₉)	129 ± 14
<i>n</i> -Eicosane (<i>n</i> -C ₂₀)	167 ± 17
<i>n</i> -Docosane (<i>n</i> -C ₂₂)	128 ± 11
<i>n</i> -Tetracosane (<i>n</i> -C ₂₄)	168 ± 16
<i>n</i> -Hexacosane (<i>n</i> -C ₂₆)	186 ± 48
<i>n</i> -Octacosane (<i>n</i> -C ₂₈)	138 ± 24
<i>n</i> -Triacontane (<i>n</i> -C ₃₀)	108 ± 11

^a Concentration is reported on a dry weight basis; material as received contains approximately 2.2% moisture.

^b Concentrations are the mean values determined by GC-MS; uncertainties for the measurements are a 95% confidence interval for the mean. Subsamples from 16 bottles were extracted and analyzed in duplicate.

APPENDIX D

Noncertified Concentrations of Inorganic Constituents in SRM 1941a

NOTE: These noncertified values were obtained using INAA procedures that have been used previously to provide certified values for SRMs with similar matrices. However, this SRM was not analyzed using a second independent analytical procedure as required to provide a certified value; therefore, unrecognized bias may exist for the determination of some analytes in this matrix.

Element	Concentration mg/kg (dry weight) ^{a,b}			Element	Concentration mg/kg (dry weight) ^{a,b}		
Na (%)	1.16	±	0.03	Rb	86	±	6
Al (%)	7.4	±	0.3	Sb	11.4	±	0.4
Cl (%)	1.36	±	0.08	Cs	5.7	±	0.2
K (%)	1.63	±	0.08	La	147	±	6
Sc	29.4	±	0.8	Ce	117	±	3
Ti (%)	1.8	±	0.2	Sm	12	±	3
V	830	±	30	Eu	1.95	±	0.07
Cr	600	±	20	Tb	1.2	±	0.2
Mn	870	±	20	Hf	33	±	1
Fe (%)	8.1	±	0.2	Ta	26.9	±	0.7
Co	24.8	±	0.8	Hg	0.5	±	0.2
Zn	610	±	20	Au	0.017	±	0.006
As	55	±	9	Th	26.3	±	0.9
Se	3.9	±	0.6				

^a Results are reported in mg/kg, except where noted in percent. Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture. Two subsamples (~300 mg each) were analyzed from each of six bottles.

^b Overall estimated analytical uncertainty at the 95% confidence level.

APPENDIX E

Noncertified Value for Total Organic Carbon

NOTE: The noncertified value has not been confirmed by an independent analytical technique as required for certification. Although bias has not been evaluated for the procedure used, the noncertified value should be useful for comparison with results obtained using a similar procedure.

Total Organic Carbon 4.8 ± 1.2 % (by weight)^{a,b}

^a Concentration is reported on a dry weight basis; material as received contains approximately 2.2% moisture.

^b The TOC value is an equally weighted mean value from routine measurements made by three laboratories; the uncertainty was calculated according to NIST Technical Note 1297 "Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results" and has an approximate level of confidence of 95%.

APPENDIX F

Summary of Analytical Results for the Determination of PAHs in SRM 1941a

Compound	Concentration, $\mu\text{g/kg}$ (dry weight) ^{a,b}							
	GC-MS (DB-5)		GC-MS (SB-Smectic)		LC-FL (Total)		LC-FL (Fractions)	
Naphthalene	1095	(62)	1031	(34)	893	(66)		
Fluorene	94.3	(7.3)	101.5	(9.5)				
Phenanthrene	492	(32)	476	(31)	491	(11)		
Anthracene	193	(12)	184	(11)	182.9	(6.1)		
Fluoranthene	1012	(57)	1009	(58)	929	(44)		
Pyrene	814	(33)	812	(51)	808	(38)		
Benz[<i>a</i>]anthracene	423	(19)	444	(26)			423	(12)
Chrysene	[592] ^c	(35)	396	(35)			379.3	(7.7)
Triphenylene			191	(10)	202.3	(3.0)		
Benzo[<i>b</i>]fluoranthene	[979] ^d	(82)	739	(50)	800	(150)		
Benzo[<i>j</i>]fluoranthene			341	(22)				
Benzo[<i>k</i>]fluoranthene	416	(45)	362	(26)	361	(21)		
Benzo[<i>a</i>]fluoranthene	114	(13)	119.0	(7.8)				
Benzo[<i>e</i>]pyrene	522	(46)	587	(45)				
Benzo[<i>a</i>]pyrene	611	(44)	656	(93)	639	(34)		
Perylene	495	(41)	434	(25)	434	(22)		
Indeno[1,2,3- <i>cd</i>]pyrene	446	(23)	537	(38)			520	(25)
Benzo[<i>ghi</i>]perylene	480	(34)	576	(43)			522	(11)
Dibenz[<i>a,j</i>]anthracene	76.2	(4.8)	70.0	(7.7)			76.1	(4.7)
Dibenz[<i>a,c</i>]anthracene	[123] ^e	(10)	41.3	(4.3)			44.8	(3.0)
Dibenz[<i>a,h</i>]anthracene			76	(10)			73.6	(4.9)
Pentaphene	47.8	(2.7)	35.4	(2.6)			46.7	(8.4)
Benzo[<i>b</i>]chrysene	94.3	(5.8)	106	(13)			112	(17)
Picene	80.4	(8.2)	75.9	(9.4)			80.8	(6.6)

^a Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture.

^b Uncertainties (in parentheses) are reported as the standard deviation of a single measurement; subsamples from six bottles were extracted and analyzed in duplicate for all four techniques.

^c Concentration is the sum of the chrysene and triphenylene.

^d Concentration is the sum of benzo[*b*]fluoranthene and benzo[*j*]fluoranthene.

^e Concentration is the sum of dibenz[*a,c*]anthracene and dibenz[*a,h*]anthracene.

APPENDIX G

Summary of Analytical Results for the Determination of PCB Congeners in SRM 1941a

Polychlorinated Biphenyls ^a		Concentration µg/kg (dry weight) ^{b,c}			
		GC-ECD		GC-MS	
PCB 8	(2,4'-Dichlorobiphenyl)	1.39	(0.15)		
PCB 18	(2,2',5-Trichlorobiphenyl)	1.15	(0.13)		
PCB 28	(2,4,4'-Trichlorobiphenyl)	9.8	(1.3)	[13.9] ^d	(1.0)
PCB 31	(2,4',5-Trichlorobiphenyl)	6.20	(0.85)		
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl)	4.99	(0.46)	4.60	(0.60)
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl)	10.89	(0.48)	8.04	(0.62)
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)	6.89	(0.36)	6.89	(0.50)
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)	7.45	(0.59)	6.08	(0.48)
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)	6.71	(0.43)	6.70	(0.80)
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)	8.17	(0.38)	6.78	(0.53)
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)	4.03	(0.33)	4.30	(0.32)
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)	11.87	(0.86)	10.1	(0.63)
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)	3.69	(0.18)	3.61	(0.41)
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)	9.88	(0.46)	9.07	(0.60)
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)	10.69	(0.51)	9.38	(0.79)
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	2.09	(0.13)	1.64	(0.24)
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	13.70	(0.47)	13.07	(0.61)
163	(2,3,3',4',5,6-Hexachlorobiphenyl)				
164	(2,3,3',4',5',6-Hexachlorobiphenyl)				
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)	9.71	(0.55)	8.66	(0.60)
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl)			2.62	(0.17)
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	17.9	(1.2)	17.31	(0.83)
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)	0.835	(0.068)	1.028	(0.069)
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	3.20	(0.20)	2.79	(0.29)
190	(2,3,3',4,4',5,6-Heptachlorobiphenyl)				
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	5.60	(0.25)	6.06	(0.49)
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)			1.63	(0.16)
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	9.30	(0.42)	4.64	(0.50)
182	(2,2',3,4,4',5,6'-Heptachlorobiphenyl)				
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	1.89	(0.19)	1.66	(0.23)
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	4.28	(0.23)	3.07	(0.26)
PCB 209	(Decachlorobiphenyl)	8.57	(0.31)	8.11	(0.49)

^a 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; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

^b Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture.

^c Uncertainties (in parentheses) are reported as the standard deviation of a single measurement; subsamples from six bottles were extracted and analyzed in duplicate for both techniques.

^d PCB 28 and PCB 31 coeluted in the GC-MS analyses.

APPENDIX H

Summary of Analytical Results for the Determination of Chlorinated Pesticides in SRM 1941a

Chlorinated Pesticides	Concentration $\mu\text{g/kg}$ (dry weight) ^{a,b}			
	GC-ECD		GC-MS	
Hexachlorobenzene	82.0	(6.3)	58.6	(8.6)
<i>cis</i> -Chlordane (α -Chlordane)	2.69	(0.27)	1.97	(0.21)
<i>trans</i> -Nonachlor	1.30	(0.11)	1.21	(0.12)
Oxychlordane	2.59	(0.22)		
Dieldrin	1.26	(0.55)		
2,4'-DDE	0.798	(0.040)	0.653	(0.058)
4,4'-DDE	6.85	(0.32)	6.33	(0.35)
4,4'-DDD	4.96	(0.23)	5.16	(0.47)
4,4'-DDT	1.25	(0.12)		

^a Concentrations reported on dry weight basis; material as received contains approximately 2.2% moisture.

^b Uncertainties (in parentheses) are reported as the standard deviation of a single measurement; subsamples from six bottles were extracted and analyzed in duplicate for both techniques.