National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2978

Mussel Tissue (Organic Contaminants – Raritan Bay, New Jersey)

Standard Reference Material (SRM) 2978 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 bivalve mollusk tissue and similar matrices. All of the constituents for which certified and reference values are provided are naturally present in the freeze-dried mussel tissue. A unit of SRM 2978 consists of one bottle containing approximately 10 g of freeze-dried mussel tissue.

The development of this material was in response to the recommendations of the Group of Experts on Standards and Reference Materials (GESREM) established by the Intergovernmental Oceanographic Commission (IOC), United Nations Environment Program (UNEP), and the International Atomic Energy Agency (IAEA) [1]. Two additional mussel tissue materials have been developed as part of the GESREM recommendations: SRM 2976 Mussel Tissue (Trace Elements and Methylmercury) [2], which is intended for use in the determination of trace elements and methylmercury; and SRM 2977 Mussel Tissue (Organic Contaminants and Trace Elements) [3], which is intended for use in the determination of organic contaminants, trace elements, and methylmercury. These freeze-dried mussel tissue materials complement SRM 1974a Organics in Mussel Tissue (*Mytilus edulis*) [4], which is provided as a frozen tissue homogenate. SRM 2978 has concentrations of organic contaminants that are similar to SRM 1974a; the concentrations of these contaminants in SRM 2978 are a factor of 2 to 4 times higher than in SRM 2977.

Certified Concentration Values: Certified concentration values, expressed as mass fractions, are provided in Tables 1, 2, and 3 for seven PAHs, 22 PCB congeners, some in combination, and 12 chlorinated pesticides, respectively. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on results obtained at NIST from two or more analytical techniques and, for selected compounds, results from an interlaboratory comparison study.

Reference Concentration Values: Reference concentration values, expressed as mass fractions, are provided in Table 4 for 20 additional PAHs, some in combination, and in Table 5 for two additional PCB congeners. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods. Explanations in support of each reference value are given as notes in Tables 4 and 5.

Expiration of Certification: The value assignment of this SRM lot is valid until **31 December 2009**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified. NIST reserves the right to withdraw, amend, or extend this certification at anytime.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.C. Colbert.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 01 May 2000

Nancy M. Trahey, Chief
Standards Reference Materials Program

The coordination of the technical measurements leading to certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by M.G. Vangel of the NIST Statistical Engineering Division.

The mussels used for the preparation of SRM 2978 were collected under the supervision of G. Lauenstein, Coastal Monitoring and Bioeffects Assessment Division, National Ocean Service, National Oceanic and Atmospheric Administration. Preparation of the freeze-dried material was performed by R. Dawson of the University of Maryland and M.P. Cronise and C.N. Fales of the NIST Standard Reference Materials Program.

Analytical measurements at NIST were performed by M.J. Lopez de Alda, B.J. Porter, L.C. Sander, and M.M. Schantz of the NIST Analytical Chemistry Division.

Results for selected PAHs, PCB congeners, and chlorinated pesticides were also used from 15 laboratories that participated in an intercomparison exercise coordinated by R.M. Parris of the NIST Analytical Chemistry Division.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

NOTICE AND WARNING TO USERS

Storage: SRM 2978 is provided as a freeze-dried tissue homogenate in glass bottles. The tissue material should be stored at or below room temperature.

Handling: Normal biohazard safety precautions for the handling of biological tissues should be exercised.

Instructions for Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 2978 are reported on a dry-mass basis. The freeze-dried mussel tissue homogenate is hygroscopic and, as received, contains greater than 4 % (mass fraction expressed as percent) residual moisture. The mussel tissue sample should be dried to a constant mass before weighing for analysis, or if the constituents of interest are volatile, a separate subsample of the mussel tissue should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis.

PREPARATION AND ANALYSIS[1]

Sample Collection and Preparation: The mussels used for the preparation of SRM 2978 were collected from Raritan Bay, NJ. The mussels were shucked and freeze-dried at the University of Maryland, and the freeze-dried tissue was shipped to NIST. The dry material was broken into smaller chunks and then jet milled to produce a fine powder. The powder was sieved with the -40 mesh fraction (smaller than 425 μm) and saved for the SRM. The mussel tissue was then blended for homogeneity in a polyethylene bag, radiation sterilized, and aliquoted into jars.

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 2978 was similar to that reported for the recent certification of several environmental matrix SRMs [5-9] and consisted of combining results from analyses using various combinations of different extraction solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using solid phase extraction (SPE), size exclusion chromatography (SEC), or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) for analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC) and (2) gas chromatography/mass spectrometry (GC/MS) for analysis of the PAH fraction on two stationary phases of different selectivity, (i.e., a 5 % mole fraction) phenyl methylpolysiloxane phase and a smectic liquid crystalline phase.

Three sets of GC/MS results, designated as GC/MS (IA), GC/MS (IB), and GC/MS (II), were obtained using two columns with different selectivities for the separation of PAHs. For GC/MS (IA) analyses, subsamples of approximately 7 g from each of six bottles of SRM 2978 were Soxhlet extracted for 18 h using DCM. Size exclusion chromatography (SEC) on a preparative-scale divinylbenzene-polystyrene column (10 μm particle size, 10 nm (100 Å) pore size, 2.5 cm i.d. x 60 cm, PL-Gel, Polymer Labs, Inc., Amherst, MA) was used to remove the majority of the lipid and biogenic

[1]Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

material. The extract was further fractionated using normal-phase LC [10-13] on a semi-preparative aminopropylsilane column to isolate the PAH fraction. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. x 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 μm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (IB) analyses were performed using the same processed extracts as for GC/MS (IA) with analysis by GC/MS using a 0.20 mm i.d. x 25 m fused silica capillary column with a smectic liquid crystalline phase (0.15 μm film thickness, Dionex, Lee Scientific Division, Salt Lake City, UT). For the GC/MS (II) analyses, one sample of 7 g from each of three bottles was Soxhlet extracted for 18 h using DCM. The SEC and normal-phase LC steps, as well as the GC/MS analyses, were the same as detailed for GC/MS (IA); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

For the LC-FL analyses, subsamples of approximately 3 g from each of six bottles of SRM 2978 were Soxhlet extracted for 20 h using DCM (three samples) and hexane/acetone 1:1 volume fraction (three samples). The extracts were concentrated and then processed through six aminopropylsilane SPE cartridges and a 0.2 μm inorganic membrane filter connected in series. The PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (μBondapak NH_2, 9 mm i.d. x 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [10-13]. The isomeric PAH fractions were analyzed using a 5 μm particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. x 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [11,12]. For all of the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the mussel tissue prior to solvent extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 2978 was used in an interlaboratory comparison exercise in 1994 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [14]. Results from 15 laboratories that participated in this exercise were used as the fifth data set in the determination of the reference values for PAHs in SRM 2978. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure PAHs.

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 2978 was similar to that reported for the recent certification of several environmental matrix SRMs [6,8,15,16] and consisted of combining results from analyses using various combinations of different extraction solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction using DCM or a hexane/acetone mixture, cleanup/isolation using SPE, SEC, or LC, followed by analysis using gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity and GC/MS.

Four sets of GC-ECD results were obtained designated as GC-ECD (IA), GC-ECD (IB), GC-ECD (IIA), and GC-ECD (IIB). For the GC-ECD (IA) and GC-ECD (IB) analyses, subsamples of approximately 7 g from each of six bottles of SRM 2978 were Soxhlet extracted for 18 h using DCM. SEC was used to remove the majority of the lipid and biogenic material. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides, and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: a 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 μm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.25 mm x 50 m fused silica capillary column with a 10 % (mole fraction) octadecyl (C-18) methylpolysiloxane phase (0.1 μm film thickness, CPSil 5/C18 CB, Chrompack International, Middelburg, The Netherlands). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the C-18 phase are designated as GC-ECD (IB). For GC-ECD (IIA) and GC-ECD (IIB), one sample (7 g) from each of three bottles was Soxhlet extracted for 18 h using DCM. The SEC and normal-phase LC steps, as well as the GC-ECD analyses, were the same as detailed for GC-ECD (IA) and GC-ECD (IB); however, the subsamples were extracted, processed and analyzed as part of three different sample sets at different times using different calibrations for each set.

For the GC/MS analyses [GC/MS (III)], subsamples of approximately 5 g from each of four bottles were Soxhlet extracted for 20 h with a mixture of hexane/acetone 1:1 volume fraction. The concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The SPE step was repeated two additional times. The extract was then analyzed by GC/MS using a 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 μm film thickness, DB-5MS, J&W Scientific, Folsom, CA). For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the mussel extract (PCB 103 and PCB 198), and 4,4'-DDT-d_8 were added to the mussel tissue prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 2978 was used in an interlaboratory comparison exercise in 1994 as

part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [14]. Results from laboratories that participated in this exercise (13 laboratories for the PCB congeners and 15 laboratories for the chlorinated pesticides) were used as the sixth data set in the determination of the reference values for PCB congeners and chlorinated pesticides in SRM 2978. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure these analytes.

Table 1. Certified Concentrations for Selected PAHs in SRM 2978

	Mass Fraction μg/kg (dry-mass basis)[a]
Fluoranthene[b,c,d,e]	166 ± 12
Pyrene[b,c,d,e]	256 ± 21
Benzo[*k*]fluoranthene[b,c,e,f]	24.1 ± 3.4
Benzo[*e*]pyrene[b,c,d,f]	89.3 ± 6.3
Perylene[b,c,d,e,f]	4.09 ± 0.32
Benzo[*ghi*]perylene[b,c,d,e]	19.7 ± 4.4
Indeno[1,2,3-*cd*]pyrene[b,c,d,e]	12.2 ± 2.9

[a] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [17]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [18], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method, as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[b] GC/MS (IA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction (6 samples) with DCM.

[c] GC/MS (II) on 5 % phenyl methylpolyxiloxane phase after Soxhlet extraction (3 samples) with DCM.

[d] Results from 15 laboratories participating in an interlaboratory comparison exercise.

[e] LC-FL of isomeric PAH fractions after Soxhlet extraction with DCM (3 samples) and with hexane/acetone 1:1 volume fraction (3 samples).

[f] GC/MS (IB) on smectic liquid crystalline phase; same extracts analyzed as GC/MS (IA).

Table 2. Certified Concentrations for Selected PCB Congeners[a] in SRM 2978

			Mass Fraction μg/kg (dry-mass basis)[b]
PCB	28	(2,4,4'-Trichlorobiphenyl)[c,d,f,g,h]	7.91 ± 0.90
PCB	31	(2,4',5-Trichlorobiphenyl)[c,d,f,g]	21.40 ± 0.43[i]
PCB	44	(2,2',3,5'-Tetrachlorobiphenyl)[c,d,e,f,g,h]	11.80 ± 0.64
PCB	49	(2,2',4,5'-Tetrachlorobiphenyl)[c,d,e,f,g]	16.84 ± 0.86
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)[c,d,e,f,g,h]	17.7 ± 2.8
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)[d,e,g]	18.4 ± 1.5
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)[c,d,e,f,g]	10.20 ± 0.29
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)[d,e,g]	20.8 ± 2.1
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl)[c,d,e,f,g]	18.84 ± 0.44
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)[c,d,e,f,g,h]	35.9 ± 1.6
	90	(2,2',3,4',5-Pentachlorobiphenyl)	
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)[c,d,e,f,g,h]	10.85 ± 0.45
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl)[c,d,e,f,g]	35.34 ± 0.71[i]
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)[c,d,e,f,g,h]	35.1 ± 1.0
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)[c,d,e,f,g,h]	5.25 ± 0.17
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)[c,d,e,f,g,h]	35.7 ± 1.5
	163	(2,3,3',4',5,6-Hexachlorobiphenyl)	
	164	(2,3,3',4',5',6-Hexachlorobiphenyl)	
PCB	149	(2,2',3,4',5',6-Hexachlorobiphenyl)[c,d,e,f,g]	34.73 ± 0.69[i]
PCB	151	(2,2',3,5,5',6-Hexachlorobiphenyl)[c,d,e,f,g]	10.92 ± 0.25
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)[c,d,e,f,g,h]	56.9 ± 3.5
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)[c,d,e,f,g]	1.97 ± 0.11
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)[c,d,e,f,g,h]	7.81 ± 0.63
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)[c,d,e,f,g]	5.25 ± 0.15
PCB	187	(2,2',3,4',5,5'6-Heptachlorobiphenyl)[c,d,e,f,g,h]	16.7 ± 1.3
	159	(2,3,3',4,5,5'-Hexachlorobiphenyl)	
	182	(2,2',3',4,4',5,6'-Heptachlorobiphenyl)	

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [19] and later revised by Schulte and Malisch [20] 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 under the GC analysis conditions used, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [17]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [18], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[c] GC-ECD (IA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with DCM (6 samples).

[d] GC-ECD (IB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IA).

[e] GC/MS (III) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with hexane/acetone 1:1 volume fraction (4 samples).

[f] GC-ECD (IIA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction (3 samples) with DCM.

[g] GC-ECD (IIB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

[h] Results from 13 laboratories participating in an interlaboratory comparison exercise.

[i] The uncertainty interval for this congener was widened in accordance with expert consideration of the analytical procedures, along with the analysis of the data as a whole, which suggests that the half-widths of the expanded uncertainties for these analytes should not be less than 2%.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 2978

	Mass Fraction[a] µg/kg (dry-mass basis)
Oxychlordane[b,c,d,e]	2.13 ± 0.27
cis-Chlordane (α-Chlordane)[b,c,d,e,f,g]	15.56 ± 0.83
trans-Chlordane (γ-Chlordane)[b,c,d,e,f,g]	11.38 ± 0.56
cis-Nonachlor[b,c,d,e,f,g]	8.23 ± 0.56
trans-Nonachlor[b,c,d,e,f,g]	11.5 ± 1.0
Dieldrin[b,c,e,f]	6.30 ± 0.67
2,4'-DDE[b,c,d,e,f,g]	4.41 ± 0.56
4,4'-DDE[b,c,d,e,f,g]	37.5 ± 1.5
2,4'-DDD[b,c,d,e,f,g]	10.5 ± 1.0
4,4'-DDD[b,c,d,e,f,g]	38.8 ± 2.3
2,4'-DDT[b,c,d,e,f,g]	9.2 ± 1.6
4,4'-DDT[b,c,d,e,f,g]	3.84 ± 0.28

[a] The results are expressed as the certified value ± the expanded uncertainty. Each certified value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [17]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [18], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method, as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[b] GC-ECD (IA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with DCM (6 samples).

[c] GC-ECD (IB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IA).

[d] GC-ECD (IIA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with DCM (3 samples).

[e] GC-ECD (IIB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

[f] Results from 15 laboratories participating in an interlaboratory comparison exercise.

[g] GC/MS (III) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with hexane/acetone 1:1 volume fraction (4 samples).

Table 4. Reference Concentrations for Selected PAHs in SRM 2978

These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the disagreement among the methods was greater than expected for certified values. These reference values should be useful for comparison with results obtained using similar procedures.

	Mass Fraction μg/kg (dry-mass basis)[a]
Naphthalene[b,c,d]	31 ± 6
2-Methylnaphthalene[b,c,d]	23 ± 4
1-Methylnaphthalene[b,c,d]	21 ± 5
Biphenyl[b,c,d]	8 ± 1
Acenaphthylene[b,c,d]	4 ± 1
Acenaphthene[b,c,d]	6 ± 2
Fluorene[b,c,d]	7 ± 1
Phenanthrene[b,c,d]	74 ± 7
Anthracene[b,c,e]	5.4 ± 2.2
1-Methylphenanthrene[b,c]	6.8 ± 0.1
Benzo[*c*]phenanthrene[f]	31 ± 2
Benz[*a*]anthracene[b,c,d,e,f]	25 ± 7
Chrysene[e,f]	59 ± 10
Triphenylene[e,f]	63 ± 9
Benzo[*b*]fluoranthene[e,f]	58 ± 15
Benzo[*j*]fluoranthene[f]	23 ± 2
Benzo[*a*]pyrene[b,c,d,e,f]	7 ± 3
Dibenz[*a,c*+*a,h*]anthracene[b,d]	3.5 ± 0.5
Benzo[*b*]chrysene[f]	2.1 ± 0.4
Picene[f]	4.5 ± 0.5

[a] The results are expressed as the reference value ± the expanded uncertainty. Each reference value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [17]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [18], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method, as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[b] GC/MS (IA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction (6 samples) with DCM.

[c] GC/MS (II) on 5 % phenyl methylpolyxiloxane phase after Soxhlet extraction (3 samples) with DCM.

[d] Results from 15 laboratories participating in an interlaboratory comparison exercise.

[e] LC-FL of isomeric PAH fractions after Soxhlet extraction with DCM (3 samples) and with hexane/acetone 1:1 volume fraction (3 samples).

[f] GC/MS (IB) on smectic liquid crystalline phase; same extracts analyzed as GC/MS (IA).

Table 5. Reference Concentrations for Selected PCB Congeners[a] in SRM 2978

These concentrations are provided as reference values because either the results have not been confirmed by an independent analytical technique as required for certification or the disagreement among the methods was greater than expected for certified values. These reference values should be useful for comparison with results obtained using similar procedures.

			Mass Fraction μg/kg (dry-mass basis)[b]
PCB	18	(2,2',5-Trichlorobiphenyl)[c,d,e,f,g,h]	7 ± 2
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)[c,d,e,f,g,h]	2.4 ± 0.6

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [19] and later revised by Schulte and Malisch [20] 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 under the GC analysis conditions used, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] The results are expressed as the reference value ± the expanded uncertainty. Each reference value is a mean of the means from two or more analytical methods, weighted as described by Paule and Mandel [17]. Each uncertainty, computed according to the CIPM approach as described in the ISO Guide [18], is an expanded uncertainty at the 95 % confidence level which includes random uncertainty within each analytical method as well as uncertainty due to the drying study. The expanded uncertainty defines a range of values within which the true value is believed to lie, at a level of confidence of approximately 95 %.

[c] GC-ECD (IA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with DCM (6 samples).

[d] GC-ECD (IB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IA).

[e] GC/MS (III) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction with hexane/acetone 1:1 volume fraction (4 samples).

[f] GC-ECD (IIA) on 5 % phenyl methylpolysiloxane phase after Soxhlet extraction (3 samples) with DCM.

[g] GC-ECD (IIB) on 50 % C-18 dimethylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

[h] Results from 13 laboratories participating in an interlaboratory comparison exercise.

REFERENCES

[1] Calder, J.A. and Jamieson, W.D., "The Purpose and Program of the IOC/UNEP/IAEA Group of Experts on Standards and Reference Materials", Fresenius J. Anal. Chem. **338**, pp. 378-379, (1990).

[2] Certificate of Analysis, Standard Reference Material 2976 Mussel Tissue (Trace Elements and Methylmercury), National Institute of Standards and Technology, Gaithersburg, MD, (8 Sept. 1998).

[3] Certificate of Analysis, Standard Reference Material 2977 Mussel Tissue (Organic Contaminants and Trace Elements), National Institute of Standards and Technology, Gaithersburg, MD, (01 May. 2000).

[4] Certificate of Analysis, Standard Reference Material 1974a Organics in Mussel Tissue (*Mytilus edulis*), National Institute of Standards and Technology, Gaithersburg, MD, 20 Nov 1995 (Revised 5 May 1999).

[5] Wise, S.A., Schantz, M.M., Benner, B.A. Jr., Hays, M.J., and Schiller, S.B., "Certification of Polycyclic Aromatic Hydrocarbons in a Marine Sediment Standard Reference Material", Anal. Chem. **67**, pp. 1171-1178, (1995).

[6] Schantz, M.M., Benner, B.A. Jr., Hays, M.J., Kelly, W.R., Vocke, R.D. Jr., Demiralp, R., Greenberg, R.R., Schiller, S.B., Lauenstein, G.G., and Wise, S.A., "Certification of Standard Reference Material (SRM) 1941a, Organics in Marine Sediment", Fresenius J. Anal. Chem. **352**, pp. 166-173, (1995).

[7] Wise, S.A., Schantz, M.M., Hays, M.J., Koster, B.J., Sharpless, K.S., Sander, L.C., and Schiller, S.B., "Certification of Polycyclic Aromatic Hydrocarbons in Mussel Tissue Standard Reference Materials", Polycyclic Aromat. Compd. **12,** pp. 21-26, (1997).

[8] Schantz, M.M., Demiralp, R., Greenberg, R.R., Hays, M.J., Parris, R.M., Porter, B.J., Poster, D.L., Sander, L.C., Schiller, S.B., Sharpless, K.S., and Wise, S.A., "Certification of a Frozen Mussel Tissue Standard Reference Material (SRM 1974a) for Trace Organic Constituents", Fresenius J. Anal. Chem. **358**, pp. 431-440, (1997).

[9] Wise, S.A., Schantz, M.M., Hays, M.J., Koster, B.J., Sharpless, K.S, Sander, L.C., Benner, B.A. Jr., and Schiller, S.B., "Certification of Polycyclic Aromatic Hydrocarbons in Mussel Tissue and Air Particulate Standard Reference Materials", Polycyclic Aromat. Compd. **9,** pp. 209-216, (1996).

[10] 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**, pp. 2306-2310, (1977).
[11] May W.E. and Wise, S.A., "Liquid Chromatographic Determination of Polycyclic Aromatic Hydrocarbons in Air Particulate Extracts", Anal. Chem. **56**, pp. 225-232, (1984).
[12] Wise, S.A. Wise, 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**, pp 887-894, (1988).
[13] Wise, S.A., Deissler, A., and 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. **3**, pp. 169-184, (1993).
[14] Parris, R.M., Schantz, M., and Wise, S.A., "NIST/NOAA NS&T/EPA EMAP Intercomparison Exercise Program for Organic Contaminants in the Marine Environment: Description and Results of 1994 Organic Intercomparison Exercises", Analytical Chemistry Division, National Institute of Standards and Technology, (February 1998).
[15] Schantz, M.M., Parris, R.M., Kurz, J., Ballschmiter, K., and Wise, S.A., "Comparison of Methods for the Gas-Chromatographic Determination of PCB Congeners and Chlorinated Pesticides in Marine Reference Materials", Fresenius J. Anal. Chem. **346**, pp. 766-778, (1993).
[16] Schantz, M.M., Koster, B.J., Oakley, L.M., Schiller, S.B., and Wise, S.A., "Certification of Polychlorinated Biphenyl Congeners and Chlorinated Pesticides in a Whale Blubber Standard Reference Material", Anal. Chem. **67**, pp. 901-910, (1995).
[17] Paule, R.C. and Mandel, J., "Consensus Values and Weighting Factors", J. Res. NBS. **87**, pp. 377-385, (1982).
[18] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Switzerland, 1993; see also Taylor, B.N., and Kuyatt, C.E., "Guidelines for Evaluating and Expressing Uncertainty of NIST Measurement Results", NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/.
[19] Ballschmiter, K. and Zell, M., "Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography - Composition of Technical Aroclor- and Clophen-PCB Mixtures", Fresenius Z. Anal. Chem. **302**, pp. 20-31, (1980).
[20] 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**, pp. 545-551, (1983).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (30l) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the internet http://www.nist.gov/srm.