



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1974a

Organics in Mussel Tissue (*Mytilus edulis*)

Standard Reference Material (SRM) 1974a is a frozen mussel tissue homogenate intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, methylmercury, and total mercury in marine bivalve mollusk tissue and similar matrices. Information is also provided for selected aliphatic hydrocarbons and inorganic constituents. All of the constituents for which certified and reference values are provided in SRM 1974a are naturally present in the frozen mussel tissue homogenate. A second mussel tissue material, SRM 2974, Organics in Freeze-dried Mussel Tissue (*Mytilus edulis*), was prepared from the same mussel tissue homogenate as SRM 1974a, and is provided as a freeze-dried tissue [1]. A unit of SRM 1974a consists of three bottles, each containing approximately 15 g to 20 g (wet basis) of frozen tissue homogenate.

Certified Concentration Values: Certified values are provided in Tables 1-3 for the concentrations, expressed as mass fractions, of 15 PAHs, 20 PCB congeners, and 7 chlorinated pesticides. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained from two or more independent analytical techniques. Certified values for concentrations, expressed as mass fractions, for methylmercury and mercury (mercury refers to "total" mercury, not only elemental mercury) are provided in Table 4. The certified values for mercury and methylmercury are based on agreement of results from two or more independent analytical techniques used in the analysis of both SRM 1974a and SRM 2974.

Reference Concentration Values: Reference values, also expressed as mass fractions, are provided in Table 5 for 18 additional PAHs, and in Table 6 for 4 additional PCB congeners and 4 additional chlorinated pesticides. Reference values are provided in Table 7 for 16 aliphatic hydrocarbons. Reference values for selected inorganic constituents are provided in Tables 8 and 9. The concentrations in Tables 5-9 are provided only as reference values because either the results have not been confirmed by an independent analytical technique as required for certification, or no (or only limited) analyses were performed by NIST.

Expiration of Certification: The certification of this SRM lot is valid until 30 April 2002, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Instructions for Use). However, the certification is invalid if the SRM is damaged, contaminated, or modified.

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

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

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

Gaithersburg, MD 20899
Certificate Issue Date: 18 July 1997*
20 Nov 95 (original certificate date)

Thomas E. Gills, Chief
Standard Reference Materials Program

*This technical revision reports a change from noncertified to certified values for mercury and methylmercury; reference values for five additional trace elements, and new reference values for selected elements.

The mussels used for SRM 1974a were collected with the assistance of J. Seavey from Battelle Ocean Sciences Laboratory, Duxbury, MA. The collection and preparation of mussels used for SRM 1974a were performed by K.A. Fitzpatrick, B.J. Porter, M.M. Schantz, and S.A. Wise of the NIST Analytical Chemistry Division and by M.P. Cronise and C.N. Fales of the NIST Standard Reference Materials Program.

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

Analytical measurements were performed by D.A. Becker, R. Demiralp, M.K. Donais, R.R. Greenberg, M.J. Hays, E.A. Mackey, R.M. Parris, D.L. Poster, L.C. Sander, R. Saraswati, M.M. Schantz, and K.S. Sharpless of the NIST Analytical Chemistry Division.

Analytical measurements for mercury and methylmercury were also performed at the Institute of Applied Physical Chemistry, Research Centre of Jülich (Jülich, Germany) by K. May and H. Emons and at the Marine Environment Laboratory, International Atomic Energy Agency (Monaco) by V. Mandic, S. Azemard, and M. Horvat. Results for selected trace elements were also used from seven laboratories that participated in an intercomparison exercise coordinated by S. Berman and S. Willie of the Institute for National Measurement Standards, National Research Council (NRC) of Canada. The seven laboratories from the NRC interlaboratory comparison exercise providing data for selected trace elements were: NOAA Northwest Fisheries Science Center (Seattle, WA); Skidaway Institute of Oceanography (Savannah, GA); Department of Chemistry, Texas A&M University (College Station, TX); Battelle Pacific Northwest (Sequim, WA); Pennsylvania Department of Environmental Services (Harrisburg, PA); Queensland Department of Primary Industries (Queensland, Australia); and Institute for National Measurement Standards, National Research Council of Canada (Ottawa, Ontario).

NOTICE AND WARNING TO USERS

Storage: SRM 1974a is packaged as a frozen tissue homogenate in glass bottles. The tissue homogenate should not be allowed to thaw prior to subsampling for analysis. This material has been stored at NIST at -80 °C (or lower) since it was prepared and should be stored by the user at this temperature, or the certified values will be invalidated.

Handling: This material is a frozen tissue homogenate. After extended storage at temperatures of -25 °C or higher, or if allowed to warm, the tissue homogenate will lose its powder-like form. For the handling of this material during sample preparation, the following procedures and precautions are recommended. If weighing relatively large quantities, remove a portion from the bottle and reweigh the bottle to determine the weight of the subsample. (Avoid heavy frost buildup by handling the bottles rapidly and wiping them prior to weighing.) For weighing, transfer subsamples to a pre-cooled, thick-walled glass container rather than a thin-walled plastic container to minimize heat transfer to the sample. If possible, use a cold work space, e.g., an insulated container with dry ice or liquid nitrogen coolant on the bottom and pre-cooled implements, such as Teflon[®]1-coated spatulas, for transferring the powder. Normal biohazard safety precautions for the handling of biological tissues should be exercised.

Instructions for Use: Subsamples of this SRM for analysis 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 concentrations of constituents in SRM 1974a are reported on both a wet mass and a dry mass basis for user convenience. The SRM tissue homogenate, as received, contains approximately 89 % moisture. A separate subsample of the SRM should be removed from the bottle at the time of analysis and dried to determine the concentration based on dry mass.

¹Certain commercial materials and equipment are identified in order to adequately specify the experimental procedure. Such identification does not imply a recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment are the best available for the purpose.

PREPARATION AND ANALYSIS

Sample Collection: The mussels (*Mytilus edulis*) used for the preparation of SRM 1974a were collected on October 1992 from Dorchester Bay within Boston Harbor, MA (Position 42°18.25'N, 71°02.31'W) following the same procedures as described previously for the collection of mussels for SRM 1974 [2]. Approximately 6 000 individual mussels were collected by hand at low tide. The samples were transported to the Battelle Ocean Sciences Laboratory (Duxbury, MA) where the mussels were rinsed with water to remove rocks and other debris. The samples were placed in insulated, Teflon-lined wooden containers, frozen and transported to NIST on dry ice. The samples were transferred to Teflon bags and stored in a liquid nitrogen vapor freezer (-120 °C) until they were shucked.

Sample Preparation: The mussel tissue was removed from the shell using the following procedure. The mussels were allowed to warm up to about 0 °C; the tissue was removed from the shell using a titanium knife and placed in Teflon bags (approximately 1 kg per bag) and immediately returned to a liquid nitrogen freezer. Approximately 81 kg total of mussel tissue were prepared for use as SRM 1974a and SRM 2974. The frozen mussel tissue was pulverized in batches of approximately 700 g each using a cryogenic procedure described previously [3]. The pulverized material was then homogenized in an aluminum mixing drum in 30 kg batches. The mixing drum was designed to fit inside the liquid nitrogen vapor freezer and to rotate in the freezer thereby mixing the frozen tissue powder. After mixing for 2 h, subsamples (15 g to 20 g) of the mussel tissue homogenate were aliquoted into pre-cooled glass bottles.

Conversion to Dry Mass Basis: The moisture content of the mussel homogenate was determined by measuring the mass loss from freeze drying. Twenty bottles of SRM 1974a were selected for the drying study according to a stratified randomization scheme. The entire contents of each glass bottle were transferred to a Teflon bottle and dried for five days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. Based on these studies, a 95 % confidence interval for the mean moisture content of SRM 1974a is 88.61 % \pm 0.08 % (mass fraction expressed as percent). Analytical results for constituents determined on a wet mass basis were converted to a dry mass basis by dividing by the conversion factor of 0.1139 (kilogram dry mass per kilogram wet mass).

Polycyclic Aromatic Hydrocarbons: The general approach used for the determination of PAHs in SRM 1974a was similar to that reported previously for the certification of a marine sediment material [4]. This approach consisted of: (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 a 5 % phenyl-substituted methylpolysiloxane stationary phase, and (4) GC/MS analysis of the PAH fraction on a smectic liquid crystalline stationary phase that provides excellent shape selectivity for the separation of PAH isomers. The procedures, which are described in detail for SRM 1974a in references [5-6], are described briefly below.

Three sets of GC/MS results, designated as GC/MS (I), GC/MS (II), and GC/MS (III), were obtained using two columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of 7 g to 9 g of the frozen mussel homogenate from 12 randomly selected bottles were mixed with approximately 100 g of sodium sulfate, internal standards were added to the sodium sulfate-tissue mixture, and then the mixture was Soxhlet extracted for 18 h using 250 mL of dichloromethane. The extract was concentrated, and size-exclusion chromatography on a preparative-scale divinylbenzene-polystyrene column (10 μ m particle size, 10 nm pore size, 2.5 cm i.d. x 60 cm) was used to remove the majority of the lipid and biogenic materials. The eluant was concentrated and injected onto a semi-preparative aminopropylsilane column to isolate the PAH fraction by normal phase LC [7]. The PAH fraction was then analyzed by GC/MS using a 0.25 mm x 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). For the GC/MS (II) analyses, subsamples of 14 g to 16 g from three bottles of SRM 1974a were extracted and one half of the extract was analyzed using the same procedures as described above for GC/MS (I). However, these extractions and analyses were performed as part of three different sample sets at different times using different calibrations for each set. The GC/MS (III) results were obtained by analyzing seven of the sample extracts from the GC/MS (I) set using a 0.2 mm i.d. x 25 m (0.15 μ m film thickness) smectic liquid

crystalline stationary phase (SB-Smectic, Dionex, Lee Scientific Division, Salt Lake City, UT). The liquid crystalline phase provided significantly different selectivity for the separation of PAH isomers when compared with the 5 % phenyl methylpolysiloxane phase.

Two sets of LC-FL results, designated as LC-FL (Total) and LC-FL (Fraction), were used in the certification process. For the LC-FL (Total) analyses, 15 g to 20 g of the frozen mussel tissue homogenate from each of eight randomly selected bottles were mixed with approximately 100 g of sodium sulfate, internal standards were added to the sodium sulfate-tissue mixture, and then the mixture was Soxhlet extracted for 20 h using 200 mL of 50 % hexane/50 % acetone (volume fractions). The extract was concentrated and then passed through an aminopropylsilane solid phase extraction (SPE) column to remove the lipid and more polar interferences. The eluant from the SPE column was concentrated and the SPE procedure was repeated five times on different SPE columns. The cleaned-up extract was then analyzed by reversed-phase LC using a polymeric octadecylsilane (C₁₈) column (4.6 mm i.d. x 15 cm, 3 μm particle size, ChromSpher PAH, Chrompack, Middelburg, The Netherlands) with wavelength programmed fluorescence detection [4,5,8]. To quantify several additional PAHs that are difficult to measure accurately in the total PAH fraction, six additional samples of SRM 1974a were extracted and prepared as described above. The extract was then fractionated on a semi-preparative aminopropylsilane column to isolate isomeric PAH fractions as described previously [4,5,7]. These isomeric PAH fractions were analyzed by reversed-phase LC-FL on a similar C₁₈ column. For both the GC/MS and LC-FL measurements, selected perdeuterated PAHs were added to the mussel tissue sodium sulfate mixture immediately prior to Soxhlet extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1974a was assessed by analyzing duplicate samples of 7 g to 9 g each from 12 randomly selected bottles. Samples were extracted, processed, and analyzed as described above for the GC/MS (I). No statistically significant differences between bottles were observed for the PAHs at the 7 g to 9 g sample size.

PCBs and Chlorinated Pesticides: SRM 1974a was analyzed for selected PCB congeners and chlorinated pesticides using gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity and using GC/MS. This same approach has been used previously for the certification of PCBs and chlorinated pesticides in environmental matrices [9-11]. Two sets of GC-ECD analyses, designated as GC-ECD (I) and GC-ECD (II), were performed using similar procedures. For the GC-ECD (I) analyses, subsamples of 14 g to 16 g from eight bottles were mixed with 100 g of precleaned sodium sulfate and Soxhlet extracted using 250 mL of dichloromethane for 18 h. Size exclusion chromatography on a preparative-scale divinylbenzene-polystyrene column 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: 0.25 mm x 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5, J&W Scientific, Folsom, CA) and a 0.32 mm x 100 m fused silica capillary column with a dimethylpolysiloxane phase containing 50 % (mole fraction) methyl C-18 (0.1 μm film thickness) (CPSil 5 C18 CB, Chrompack International, Middelburg, The Netherlands). For the GC-ECD (II) analyses, subsamples of 14 g to 16 g from three bottles of SRM 1974a were extracted and one half of the extract was analyzed using the same procedures as described for GC-ECD (I). However, these extractions and analyses were performed as part of three different sample sets at different times using different calibrations for each set.

For the GC/MS analyses, subsamples of 14 g to 16 g each from eight randomly selected bottles were Soxhlet extracted for 18 h using 50 % hexane/50 % acetone. The extracts were concentrated and placed on a precleaned silica SPE column and eluted with 15 mL of 10 % dichloromethane in hexane. The SPE cleanup procedure was performed sequentially on three separate SPE columns. The final fraction was analyzed by GC/MS on a 5 % phenyl-substituted methylpolysiloxane phase as described above for PAH measurements. For both the GC-ECD and GC/MS analyses, two PCB congeners that are not significantly present in the mussel tissue extract (PCB 103 and PCB 198 [12,13]), octachloronaphthalene, 4,4'-DDT-*d*₈, and endosulfan-*d*₄ were added to the mussel tissue/sodium sulfate mixture prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1974a was used in two interlaboratory comparison exercises as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment. The results from these two exercises, which were conducted in 1993 and 1994 and included results from 22 and 19 laboratories respectively, were also used in the determination of the certified values for selected PCB congeners and chlorinated pesticides. The laboratories participating in these exercises used the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

Aliphatic Hydrocarbons: The fractions isolated for the measurement of PAHs by GC/MS (I) were analyzed on the same 5 % phenyl-substituted methylpolysiloxane column by GC/MS for the determination of selected aliphatic hydrocarbons. Perdeuterated dodecane, eicosane, and triacontane were added prior to extraction as internal standards for quantification purposes.

Methylmercury and Mercury: The certified values for methylmercury and mercury are based on results of analyses of SRM 1974a and SRM 2974 at NIST and two other laboratories: Institute of Applied Physical Chemistry, Research Centre of Jülich, (Jülich, Germany) and the Marine Environment Laboratory, International Atomic Energy Agency (Monaco). The results from the analyses of both SRMs (i.e., the frozen tissue and the freeze-dried tissue) were combined to provide one certified value for both materials.

For the determination of methylmercury, SRM 1974a and SRM 2974 were analyzed at NIST using liquid-solid extraction under acidic conditions and size exclusion chromatography followed by GC with atomic emission detection (GC-AED) as described in detail by Donais et al. [14,15]. The GC-AED analyses were performed using a 0.53 mm x 15 m fused silica capillary column with a 14 % (mole fraction expressed as percent) cyanopropyl-substituted methylpolysiloxane phase (3.0 μ m film thickness)(OV-1701, Quadrex, New Haven, CT). SRM 2974 was also analyzed at NIST using a second analytical procedure [16] which consisted of the distillation of the organomercury compounds from a mixture of the powdered sample, sulfuric acid, and cupric sulfate followed by binding of the organomercury compounds to cysteine fixed onto filter paper. The filter paper was then subjected to neutron activation analysis for mercury determination. The method used at IAEA consisted of saponification at 70 °C followed by ethylation-room temperature precollection, GC pyrolysis, cold vapor atomic fluorescence spectrometric detection [17]. At the Research Centre of Jülich the analytical procedure for methylmercury consisted of a water steam distillation under acid conditions, anion exchange chromatographic separation of inorganic and methylmercury followed by cold vapor atomic absorption spectrometric detection before and after ultraviolet radiation [18-20]. Subsamples from six bottles each of SRM 1974a and SRM 2974 were analyzed by each of the three laboratories.

For the determination of the certified value for mercury, results from four different analytical procedures and from seven laboratories that participated in an interlaboratory comparison exercise coordinated by the Institute for National Measurement Standards, National Research Council (NRC) of Canada, were used [21]. The four different procedures included: microwave digestion followed by flow injection cold vapor atomic absorption spectrometry (NIST) [22], instrumental neutron activation analysis (INAA) (NIST) [23]; acid digestion at 70 °C followed by double gold trap amalgamation cold vapor atomic fluorescence spectrometry (IAEA) [24]; and acid digestion at 150 °C followed by gold wire amalgamation cold vapor atomic absorption spectrometry (Research Centre of Jülich) [25]. Subsamples from six bottles each of SRM 1974a and SRM 2974 were analyzed by each of the three laboratories.

Inorganic Constituents: For inorganic constituents (except mercury), the reference values are based on the results of analyses at NIST using instrumental neutron activation analysis (INAA) and the results from analyses of SRM 2974 by seven laboratories that participated in an interlaboratory comparison exercise coordinated by the NRC of Canada [21]. NRC provided results from two different analytical techniques as part of the interlaboratory study.

At NIST, selected inorganic constituents were determined in SRM 1974a and SRM 2974 using INAA procedures as described previously for the analysis of marine bivalves [23]. Subsamples from eight randomly selected bottles of SRM 1974a were freeze-dried and duplicate portions of approximately 200 mg each from each bottle were pelletized and analyzed. A total of 6-8 subsamples from 3-5 bottles of SRM 2974 were analyzed using INAA.

REFERENCES

- [1] Certificate of Analysis, Standard Reference Material (SRM) 2974, Organics in Freeze-dried Mussel Tissue, National Institute of Standards and Technology, Gaithersburg MD, 1997.
- [2] Wise, S.A., Christensen, R.G., Benner, B.A. Jr., Koster, B.J., Schantz, M.M., and Zeisler, R., "Preparation and Analysis of a Frozen Mussel Tissue Reference Material for the Determination of Trace Organic Constituents," *Environ. Sci. Technol.* **25**, pp. 1695-1704, (1991).
- [3] Zeisler, R. Langland, J.K., and Harrison, S.H., "Cryogenic Homogenization of Biological Tissues," *Anal. Chem.* **55**, pp. 2431-2434, (1983).
- [4] 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).
- [5] 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," *J. Polycyclic Aromatic Compounds* **12**, pp. 21-36, (1997).
- [6] 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).
- [7] 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).
- [8] 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).
- [9] 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).
- [10] 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).
- [11] 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).
- [12] 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).
- [13] 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).
- [14] Donais (Behlke), M.K., Uden, P.C., Schantz, M.M., and Wise, S.A., "Development, Validation, and Application of a Method for Quantification of Organomercury Species in Biological Marine Materials using Gas Chromatography-Atomic Emission Detection," *Anal. Chem.* **68**, pp. 3859-3866, (1996).
- [15] Donais, M.K., Saraswati, R., Mackey, E.A., Demiralp, R., Porter, B.J., Vangel, M., Levenson, M., Mandic, V., Azemard, S., Horvat, M., May, K., Emons, H., and Wise, S.A., "Certification of Three Mussel Tissue Standard Reference Materials for Methylmercury and Total Mercury Content," *Fresenius' J. Anal. Chem.* **358**, pp. 424-430, (1997).
- [16] Mackey, E.A. and Becker, D.A., "Determination of Methylmercury in Two Mussel Tissue Standard Reference Materials by Pre-irradiation Separation and Neutron Activation Analysis," (in preparation).
- [17] Horvat, M., Liang, L., and Bloom, N.S., "Comparison of Distillation with Other Current Isolation Methods for the Determination of Methylmercury Compounds in Low Level Environmental Samples," *Anal. Chim. Acta* **282**, pp. 153-168, (1993).
- [18] May, K., Stoeppler, M., and Reisinger, K., "Studies of the Ratio of Total Mercury/Methylmercury in the Aquatic Food Chain," *Toxicol. Environ. Chem.* **13**, pp. 153-159, (1987).

- [19] Ahmed, R., May, K., and Stoeppler, M., "Ultratrace Analysis of Mercury and Methylmercury (MM) in Rain Water using Cold Vapour Atomic Absorption Spectrometry," *Fresenius J. Anal. Chem.* **326**, pp. 510-516, (1987).
- [20] Padberg, S., Burow, M., and Stoeppler, M., "Methylmercury Determination in Environmental and Biological Reference and Other Materials by Quality Control with Certified Reference Materials," *Fresenius J. Anal. Chem.* **346**, pp. 686-688, (1993).
- [21] Willie, S. and Berman, S., "NOAA National Status and Trends Program Ninth Round Intercomparison Exercise Results for Trace Metals in Marine Sediments and Biological Tissues," NOAA Technical Memorandum NOS ORCA 93, Silver Spring MD, (1995).
- [22] Saraswati, R., Vetter, T.W., Jr., and Watters, R.L., "Determination of Arsenic, Selenium and Mercury in an Estuarine Sediment Standard Reference Material using Flow Injection and Atomic Absorption Spectrometry," *Mikrochim. Acta* **118**, pp. 163-175, (1995).
- [23] Zeisler, R., Stone, S.F., and Sanders, R.W., "Sequential Determination of Biological and Pollutant Elements in Marine Bivalves," *Anal. Chem.* **60**, pp. 2760-2765, (1988).
- [24] Liang, L. and Bloom, N.S., "Determination of Total Mercury by Single-Stage Gold Amalgamation with Cold Vapour Atomic Absorption Spectrometric Detection," *J. Anal. Atom. Spect.* **8**, pp. 591-594, (1993).
- [25] Padberg, S. Burow, M., May, K., and Stoeppler, M., "Methylquecksilberbestimmung in Böden, In: CAS-6 Colloquium Atomspektromitrische Spurenanalytik," B. Welz (ed), Bodenseewerk Perkin Elmer, Überlingen, Germany, pp. 751-759, (1995).
- [26] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, 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).

Table 1. Certified Concentrations (mass fractions) for Selected PAHs in SRM 1974a^a

	$\mu\text{g}/\text{kg}$ wet mass basis ^b		$\mu\text{g}/\text{kg}$ dry mass basis ^b	
Naphthalene ^{d,e,g}	2.68	\pm 0.50 ^c	23.5	\pm 4.4 ^c
Anthracene ^{d,e,g}	0.69	\pm 0.20 ^c	6.1	\pm 1.7 ^c
Phenanthrene ^{d,e,g}	2.53	\pm 0.28	22.2	\pm 2.4
Fluoranthene ^{d,e,g}	18.6	\pm 1.0 ^c	163.7	\pm 9.1 ^c
Pyrene ^{d,e,g}	17.26	\pm 0.74 ^c	151.6	\pm 6.6 ^c
Benz[<i>a</i>]anthracene ^{d,e,f,h}	3.71	\pm 0.54 ^c	32.5	\pm 4.7 ^c
Chrysene ^{f,h}	5.04	\pm 0.26	44.2	\pm 2.3
Triphenylene ^{f,h}	5.77	\pm 0.67	50.7	\pm 5.9
Benzo[<i>b</i>]fluoranthene ^{f,g}	5.28	\pm 0.42 ^c	46.4	\pm 3.7 ^c
Benzo[<i>k</i>]fluoranthene ^{d,e,f,g}	2.30	\pm 0.10	20.18	\pm 0.84
Benzo[<i>a</i>]pyrene ^{d,e,f}	1.780	\pm 0.073	15.63	\pm 0.65
Benzo[<i>e</i>]pyrene ^{d,e,f}	9.56	\pm 0.21	84.0	\pm 1.9
Perylene ^{d,e,f,g}	0.874	\pm 0.030	7.68	\pm 0.27
Benzo[<i>ghi</i>]perylene ^{d,e,g,h}	2.50	\pm 0.25 ^c	22.0	\pm 2.2 ^c
Indeno[1,2,3- <i>cd</i>]pyrene ^{d,e,g,h}	1.62	\pm 0.32 ^c	14.2	\pm 2.8 ^c

^a The certified value for each analyte is the equally-weighted mean of the means from two or more independent analytical methods. The uncertainty in the certified value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study (for the dry mass basis values).

^b The results are reported on both wet and dry mass basis; the sample as received contains 88.61 % \pm 0.08 % water.

^c An additional allowance for differences between methods was included in the total uncertainty for this analyte.

^d GC/MS (I) = GC/MS analyses on 5 % phenyl methylpolysiloxane phase; duplicate samples from twelve bottles analyzed.

^e GC/MS (II) = GC/MS analysis on 5 % phenyl methylpolysiloxane phase; samples from three bottles analyzed on three different occasions.

^f GC/MS (III) = GC/MS analysis using a liquid crystalline phase; samples from eight bottles analyzed.

^g LC-FL (Total) = LC-FL analysis of total PAH fraction; samples from eight bottles analyzed.

^h LC-FL (Fraction) = LC-FL analysis of isomeric PAH fractions; samples from six bottles analyzed.

Table 2. Certified Concentrations (mass fractions) for Selected PCB Congeners^a in SRM 1974a^b

		$\mu\text{g}/\text{kg}$ wet mass basis ^c	$\mu\text{g}/\text{kg}$ dry mass basis ^c
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl)	8.28 ± 0.84	72.7 ± 7.4
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl) ^d	10.12 ± 0.59	88.8 ± 5.0
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)	13.1 ± 1.3	115 ± 11
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl) ^e	11.54 ± 0.50	101.4 ± 4.4
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl) ^{d,e}	9.5 ± 1.9	83 ± 17
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl) ^d	8.08 ± 0.46	70.9 ± 4.0
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)	14.6 ± 1.1	128.3 ± 9.7
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)	6.04 ± 0.39	53.0 ± 3.4
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl) ^{d,f}	14.5 ± 1.0	127.3 ± 8.6
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)	14.90 ± 0.40	130.8 ± 3.6
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	2.50 ± 0.39	22.0 ± 3.4
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	15.2 ± 1.1	133.5 ± 9.5
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) ^d	9.98 ± 0.27	87.6 ± 2.3
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl) ^d	2.91 ± 0.40	25.6 ± 3.5
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	16.54 ± 0.86	145.2 ± 7.6
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl) ^d	0.85 ± 0.11	7.43 ± 0.99
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	0.63 ± 0.12	5.5 ± 1.1
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	1.95 ± 0.43	17.1 ± 3.8
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl) ^d	1.82 ± 0.27	16.0 ± 2.4
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	3.87 ± 0.27	34.0 ± 2.3
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 [12] and later revised by Schulte and Malisch [13] 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 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 certified value for each analyte is the equally-weighted mean of the means from three to seven different analytical methods. The uncertainty in the certified value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study (for the dry mass basis values). The seven analytical methods used were as follows: GC-ECD (I) and (II) on the 5 % phenyl methylpolysiloxane phase, GC-ECD (I) and (II) on the 50 % C-18 dimethylpolysiloxane phase, GC/MS on the 5 % phenyl methylpolysiloxane phase, and Interlaboratory Exercises (I) and (II). The certified value was determined using the results of all seven analytical data sets except as noted.

^c Results reported on both wet and dry mass basis; the sample as received contains 88.61 % ± 0.08 % water.

^d Results from Interlaboratory Exercises (I) and (II) were not used in the determination of the certified value for this PCB congener.

^e PCB 66 and PCB 95 coeluted in the GC-ECD (I) and (II) analyses on the 5 % phenyl methylpolysiloxane phase; results were not used to determine certified value.

^f PCB 110 and PCB 77 coeluted in the GC-ECD (I) and (II) analyses on the 5 % phenyl methylpolysiloxane phase; results were not used to determine certified value.

Table 3. Certified Concentrations (mass fractions) for Selected Chlorinated Pesticides in SRM 1974a^a

	$\mu\text{g}/\text{kg}$ wet mass basis ^b	$\mu\text{g}/\text{kg}$ dry mass basis ^b
<i>cis</i> -Chlordane (α -Chlordane)	1.96 \pm 0.32	17.2 \pm 2.8
<i>trans</i> -Chlordane ^c	1.89 \pm 0.19	16.6 \pm 1.7
<i>cis</i> -Nonachlor ^c	0.78 \pm 0.10	6.84 \pm 0.90
<i>trans</i> -Nonachlor	2.05 \pm 0.41	18.0 \pm 3.6
4,4'-DDE	5.84 \pm 0.63	51.2 \pm 5.5
4,4'-DDD	4.90 \pm 0.72	43.0 \pm 6.3
4,4'-DDT	0.445 \pm 0.067	3.91 \pm 0.59

^a The certified value for each analyte is the equally-weighted mean of the means from three to seven different analytical methods. The uncertainty in the certified value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study (for the dry mass basis values). The seven analytical methods used were as follows: GC-ECD (I) and (II) on the 5 % phenyl methylpolysiloxane phase, GC-ECD (I) and (II) on the 50 % C-18 dimethylpolysiloxane phase, GC/MS on the 5 % phenyl methylpolysiloxane phase, and Interlaboratory Exercises (I) and (II). The certified value was determined using the results of all seven analytical data sets except as noted.

^b Results reported on both wet and dry mass basis; the sample as received contains 88.61 % \pm 0.08 % water.

^c Results from Interlaboratory Exercises (I) and (II) were not used in the determination of the certified value for this compound.

Table 4. Certified Concentrations (mass fractions) of Methylmercury and Total Mercury in SRM 1974a

	$\mu\text{g}/\text{kg}$ wet mass basis ^a	$\mu\text{g}/\text{kg}$ dry mass basis ^a
Methylmercury ^{b,c}	8.80 \pm 0.35	77.3 \pm 3.1
Mercury ^d	20.1 \pm 1.5	176 \pm 13

^a The results are reported on both wet and dry mass basis. The sample as received contains 88.61 % \pm 0.08 % water. The uncertainty in the water determination is negligible relative to the other sources of uncertainty.

^b Results for methylmercury are reported as μg mercury/kg.

^c The certified value is the mean of the three different laboratory analyses of SRM 1974a and the four different laboratory analyses of SRM 2974. The seven values were judged to be independent measurements. The uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [26] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c is given by the standard error of the mean of the seven analyses. The coverage factor, $k = 2.45$, is the Student's t -value for a 95 % confidence interval with six degrees of freedom.

^d The certified value is the mean of three analyses of SRM 1974a, four analyses of SRM 2974, and the mean of an interlaboratory exercise on SRM 2974. The eight values were judged to be independent measurements. The uncertainty in the certified value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [26] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the certified value. Here u_c is given by the standard error of the mean of the seven analyses and the round robin exercise. The coverage factor, $k = 2.36$, is the Student's t -value for a 95 % confidence interval with seven degrees of freedom.

Table 5. Reference Concentrations (mass fractions) for Selected PAHs in SRM 1974a^a

NOTE: 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 between the methods was greater than expected for certified values. These reference values should be useful for comparison with results obtained using similar procedures (i.e., solvent extraction and GC/MS or LC-FL on similar columns).

	$\mu\text{g}/\text{kg}$ wet mass basis	$\mu\text{g}/\text{kg}$ dry mass basis
1-Methylnaphthalene ^{d,e}	0.61 ± 0.20	5.3 ± 1.8
2-Methylnaphthalene ^{d,e}	1.16 ± 0.17 ^c	10.2 ± 1.5 ^c
Biphenyl ^{d,e}	0.582 ± 0.038	5.11 ± 0.33
Acenaphthylene ^{d,e}	0.598 ± 0.043 ^c	5.25 ± 0.38 ^c
Acenaphthene ^d	0.359 ± 0.038	3.15 ± 0.26
Fluorene ^{d,e}	0.65 ± 0.10 ^c	5.72 ± 0.91 ^c
1-Methylphenanthrene ^{d,e}	1.20 ± 0.55	10.5 ± 4.8
2-Methylphenanthrene ^d	2.34 ± 0.92	20.6 ± 8.0
3-Methylphenanthrene ^d	1.5 ± 1.1	13.5 ± 9.7
4-Methylphenanthrene/9-Methylphenanthrene ^d	1.7 ± 1.0	14.7 ± 9.2
Benzo[ghi]fluoranthene ^d	3.22 ± 0.62	28.3 ± 5.5
Benzo[c]phenanthrene ^{d,f}	2.22 ± 0.76 ^c	19.5 ± 6.7 ^c
Benzo[a]fluoranthene ^{d,e}	0.45 ± 0.22 ^c	4.0 ± 1.9 ^c
Benzo[j]fluoranthene ^f	2.33 ± 0.20	20.5 ± 1.7
Anthracene ^g	0.131 ± 0.036	1.15 ± 0.31
Benzo[b]chrysene ^d	0.182 ± 0.016	1.60 ± 0.15
Dibenz[a, c]anthracene/Dibenz[a, h]anthracene ^{d,e}	0.342 ± 0.022	3.00 ± 0.20
Dibenz[a, j]anthracene ^d	0.142 ± 0.010	1.247 ± 0.075

^a The reference value for each analyte is the equally-weighted mean of the means from two analytical methods or the mean from one analytical technique. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study (for the dry mass basis values).

^b Results reported on both wet and dry mass basis; the sample as received contains 88.61 % ± 0.08 % water.

^c An additional allowance for differences between methods was included in the total uncertainty for this analyte.

^d GC/MS (I) = GC/MS analyses on 5 % phenyl methylpolysiloxane phase; duplicate samples from 12 bottles analyzed.

^e GC/MS (II) = GC/MS analysis on 5 % phenyl methylpolysiloxane phase; samples from three bottles analyzed on three different occasions.

^f GC/MS (III) = GC/MS analysis using a liquid crystalline phase; samples from eight bottles analyzed.

^g LC-FL (Fraction) = LC-FL analysis of isomeric PAH fractions; samples from six bottles analyzed.

Table 6. Reference Concentrations (mass fractions) for Selected PCB Congeners and Chlorinated Pesticides in SRM 1974a^a

NOTE: 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 (i.e., solvent extraction and GC-ECD or GC/MS on similar columns).

Polychlorinated Biphenyls ^b	$\mu\text{g}/\text{kg}$ wet mass basis ^c	$\mu\text{g}/\text{kg}$ dry mass basis ^c
PCB 18 (2,2',5-Trichlorobiphenyl)	3.7 \pm 1.2	33 \pm 11
PCB 28 (2,4,4'-Trichlorobiphenyl) ^d	9.0 \pm 1.7	79 \pm 15
PCB 31 (2,4',5-Trichlorobiphenyl) ^{d,e}	8.6 \pm 2.4	76 \pm 21
PCB 87 (2,2',3,4,5'-Pentachlorobiphenyl) ^e	6.1 \pm 1.6	54 \pm 14
Chlorinated Pesticides	$\mu\text{g}/\text{kg}$ wet mass basis ^c	$\mu\text{g}/\text{kg}$ wet mass basis ^c
Dieldrin	0.70 \pm 0.15	6.2 \pm 1.3
2,4'-DDE	0.599 \pm 0.031	5.26 \pm 0.27
2,4'-DDD	1.56 \pm 0.32	13.7 \pm 2.8
2,4'-DDT	0.96 \pm 0.21	8.5 \pm 1.9

^a The reference value for each analyte is the equally-weighted mean of the means from four to seven different analytical methods. The uncertainty in the reference value defines a range of values that is intended to function as an interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within each analytical method, among methods, and from the drying study (for the dry mass basis values). The seven analytical methods used were as follows: GC-ECD (I) and (II) on the 5 % phenyl methylpolysiloxane phase, GC-ECD (I) and (II) on the 50 % C-18 dimethylpolysiloxane phase, GC/MS on the 5 % phenyl methylpolysiloxane phase, and Interlaboratory Exercises (I) and (II). The reference value was determined using the results of all seven analytical data sets except as noted.

^b PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [12] and later revised by Schulte and Malisch [13] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch.

^c Results reported on both wet and dry mass basis; the sample as received contains 88.61 % \pm 0.08 % water.

^d PCB 28 and PCB 31 coeluted in the GC/MS analyses; results were not used to determine the certified value.

^e Results from Interlaboratory Exercises (I) and (II) were not used in the determination of the certified value for this PCB congener.

Table 7. Reference Concentrations (mass fractions) for Selected Aliphatic Hydrocarbons in SRM 1974a^a

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

	$\mu\text{g}/\text{kg}$ wet mass basis ^b	$\mu\text{g}/\text{kg}$ dry mass basis ^b
<i>n</i> -Tetradecane (<i>n</i> -C ₁₄)	9.54 ± 0.35	83.8 ± 3.0
<i>n</i> -Pentadecane (<i>n</i> -C ₁₅)	12.4 ± 1.4	108 ± 12
<i>n</i> -Hexadecane (<i>n</i> -C ₁₆)	18.4 ± 3.2	161 ± 28
<i>n</i> -Heptadecane (<i>n</i> -C ₁₇)	31.8 ± 6.5	280 ± 57
Pristane (2,6,10,14-Tetramethylpentadecane)	8.44 ± 0.67	74.1 ± 5.8
<i>n</i> -Octadecane (<i>n</i> -C ₁₈)	17.4 ± 2.0	153 ± 17
Phytane (2,6,10,14-Tetramethylhexadecane)	6.48 ± 0.78	56.9 ± 6.8
<i>n</i> -Nonadecane (<i>n</i> -C ₁₉)	4.60 ± 0.11	40.4 ± 1.1
<i>n</i> -Eicosane (<i>n</i> -C ₂₀)	7.41 ± 0.69	65.1 ± 6.0
<i>n</i> -Docosane (<i>n</i> -C ₂₂)	5.32 ± 0.22	46.7 ± 1.9
<i>n</i> -Tetracosane (<i>n</i> -C ₂₄)	6.51 ± 0.66	57.2 ± 5.7
<i>n</i> -Hexacosane (<i>n</i> -C ₂₆)	5.61 ± 0.72	49.3 ± 6.3
<i>n</i> -Octacosane (<i>n</i> -C ₂₈)	7.34 ± 0.39	64.5 ± 3.3
<i>n</i> -Triacontane (<i>n</i> -C ₃₀)	5.44 ± 0.84	47.8 ± 7.3
<i>n</i> -Dotriacontane (<i>n</i> -C ₃₂)	6.36 ± 0.48	55.9 ± 4.2
<i>n</i> -Tetratriacontane (<i>n</i> -C ₃₄)	3.25 ± 0.22	28.5 ± 1.9

^a The reference value for each analyte is the mean value determined by GC/MS. The uncertainty in the reference value defines a range of values that is intended to function as a interval that contains the true value at a level of confidence of 95 %. This uncertainty includes sources of uncertainty within the method and from the drying study (for the dry mass basis values).

^b Results reported on both wet and dry mass basis; the sample as received contains 88.61 % ± 0.08 % water.

Table 8. Reference Concentrations (mass fractions) for Selected Inorganic Constituents in SRM 1974a as Determined by Multiple Laboratories^a

NOTE: 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 insufficient analyses have been performed at NIST to confirm the results of the outside laboratories. Some of the results from the outside laboratories are based on results for SRM 2974; however, SRM 1974a and SRM 2974 are expected to be identical on a dry mass basis for the trace element content.

Element	Degrees of Freedom ^b	Coverage Factor, <i>k</i>	mg/kg (wet mass basis) ^c			mg/kg (dry mass basis) ^c		
			Value	±	Uncertainty	Value	±	Uncertainty
Aluminum ^{d,e}	2	4.30	54.6	±	9.8	479	±	86
Arsenic ^f	8	2.31	0.84	±	0.12	7.4	±	1.1
Cadmium	7	2.36	0.140	±	0.010	1.23	±	0.09
Chromium ^f	9	2.26	0.228	±	0.013	2.00	±	0.11
Copper	7	2.36	1.16	±	0.11	10.2	±	1.0
Iron ^f	8	2.31	54.2	±	1.6	476	±	14
Lead	7	2.36	1.38	±	0.16	12.1	±	1.4
Nickel	5	2.57	0.0945	±	0.017	0.83	±	0.15
Selenium ^f	8	2.31	0.229	±	0.040	2.01	±	0.35
Silver ^e	7	2.36	0.0589	±	0.0055	0.517	±	0.048
Tin	4	2.78	0.0321	±	0.0062	0.282	±	0.054
Zinc ^f	8	2.31	11.6	±	0.5	102	±	4

^a The reference value is the mean of the available results from seven laboratories participating in the NRC intercomparison exercise using SRM 2974 and two NIST INAA studies, one on each of SRMs 1974a and 2974. The analyses were judged to be independent measurements. The uncertainty in the reference value is equal to $U = k u_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [26] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the reference value. Here u_c is given by the standard error of the mean of the available values. The coverage factor, k , is the Student's t -value for a 95 % confidence interval with the degrees of freedom equal to the number of different method values minus one.

^b This value is equal to the number of independent results minus one. Six laboratories provided a single independent result and two laboratories provided two independent results.

^c The results are reported on both wet and dry mass basis. The sample as received contains 88.61 % ± 0.08 % water. The uncertainty in the water determination is negligible relative to the other sources of uncertainty.

^d Results from only three laboratories were used for this element because other laboratories used sample digestion techniques that were incomplete for the accurate determination of aluminum.

^e Value includes one set of INAA results from NIST.

^f Value includes two sets of INAA results from NIST.

Table 9. Reference Concentrations (mass fractions) for Selected Inorganic Constituents in SRM 1974a as Determined by INAA^a

NOTE: These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification; therefore, unrecognized bias may exist for some analytes in this matrix. These results are based on the analysis of SRM 1974a after samples were freeze-dried.

	percent (wet mass basis) ^b			percent (dry mass basis) ^b		
Chlorine	0.85	±	0.02	7.5	±	0.2
Magnesium	0.057	±	0.002	0.051	±	0.002
Sodium	0.52	±	0.01	4.6	±	0.1
Potassium	0.131	±	0.004	1.15	±	0.04
	mg/kg (wet mass basis)			mg/kg (dry mass basis)		
Antimony	0.0038	±	0.0005	0.033	±	0.004
Bromine	36	±	1	318	±	9
Cerium	0.066	±	0.006	0.58	±	0.05
Cesium	0.0032	±	0.0002	0.028	±	0.002
Cobalt	0.038	±	0.001	0.33	±	0.01
Europium	0.00153	±	0.00007	0.0134	±	0.0006
Gold	0.0027	±	0.0002	0.024	±	0.002
Hafnium	0.0043	±	0.0005	0.038	±	0.004
Lanthanum	0.028	±	0.002	0.24	±	0.01
Manganese	1.21	±	0.06	10.6	±	0.5
Samarium	0.0034	±	0.0004	0.030	±	0.004
Scandium	0.0103	±	0.0004	0.090	±	0.003
Strontium	9.1	±	0.6	80	±	5
Tantalum	0.0029	±	0.0004	0.025	±	0.003
Thorium	0.0071	±	0.0009	0.062	±	0.008
Vanadium	0.17	±	0.02	1.5	±	0.2

^a Each reference value is the mean of 16 repetitions of INAA analyses of SRM 1974a. The uncertainty in a reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [26] and k is the coverage factor. The value of u_c is intended to represent at the level of one standard deviation the combined effect of all the uncertainties in the reference value. The coverage factor of 2.0 was used to produce the 95 % confidence interval.

^b The results are reported on both wet and dry mass basis. The sample as received contains 88.61 % ± 0.08 % water. The uncertainty in the water determination is negligible relative to the other sources of uncertainty.

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax: (301) 926-4751, e-mail: srminfo@nist.gov, or, WWW: <http://ts.nist.gov/srm>.