



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2974

Organics in Freeze-Dried Mussel Tissue (*Mytilus edulis*)

Standard Reference Material (SRM) 2974 is 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 inorganic constituents. All of the constituents for which certified and reference values are provided in SRM 2974 are naturally present in the freeze-dried mussel tissue homogenate. SRM 2974 was prepared from the same mussel tissue homogenate as SRM 1974a, Organics in Mussel Tissue (*Mytilus edulis*) [1], which is provided as a frozen tissue homogenate. A unit of SRM 2974 consists of one bottle containing approximately 8 g of freeze-dried mussel tissue homogenate.

Certified Concentration Values: Because SRM 2974 was prepared from the same mussel tissue homogenate as SRM 1974a, the certified and reference values for the concentrations for most of the analytes in SRM 2974 are the certified and reference values for SRM 1974a [1], expressed on a dry mass basis, with a small adjustment of the uncertainty interval. Certified values are provided in Tables 1-3 for the concentrations, expressed as mass fractions, for 14 PAHs, 20 PCB congeners, and 7 chlorinated pesticides. The certified values for these constituents are based on the agreement of results obtained from two or more independent analytical techniques used for the certification of SRM 1974a, and these results were confirmed by analyses of SRM 2974 using one or more analytical techniques. Certified 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 concentration values, expressed as mass fractions, are provided in Table 5 for 17 additional PAHs, and in Table 6 for 4 additional PCB congeners and 4 additional chlorinated pesticides. Reference concentration values are provided in Tables 7 and 8 for additional inorganic constituents. The concentrations in Tables 5-8 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. 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: 17 September 2009

The mussels used for SRM 2974 were collected with the assistance of J. Seavey from Battelle Ocean Sciences Laboratory, Duxbury, MA. The collection and preparation of mussels used for SRM 2974 were performed by K.A.

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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 2974 is provided as a freeze-dried tissue homogenate in amber glass bottles. The tissue material should be stored at room temperature or below.

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

Instructions for Use: The freeze-dried mussel tissue homogenate is very hygroscopic and, as received, contains greater than 8 % (mass fraction expressed as percent) residual moisture. A separate subsample of the SRM must be removed from the bottle at the time of analysis and dried to determine the concentration on a dry mass basis.

PREPARATION AND ANALYSIS

Sample Collection and Preparation: The mussels (*Mytilus edulis*) used for the preparation of SRM 2974 were the same batch of mussels used for the preparation of SRM 1974a. The mussels were collected in October 1992 from Dorchester Bay within Boston Harbor, MA as described previously [1-4]. Approximately 81 kg of frozen mussel tissue was pulverized in batches of approximately 700 g each using a cryogenic procedure described previously [5]. The pulverized material was then cryogenically homogenized in an aluminum mixing drum in 30 kg batches. For the preparation of SRM 2974, approximately 20 kg of the frozen tissue homogenate was freeze-dried in batches of approximately 1 kg at 1 Pa, -20 °C shelf temperature and -50 °C condenser temperature. The material was dried until a stable weight was achieved. The batches were pooled and mixed. The material was radiation sterilized (⁶⁰Co) and then aliquoted into jars (~ 8 g each). The freeze-dried mussel tissue was intentionally not sieved to remove fibrous material in order to maintain the dry mass basis equivalence of the freeze-dried material to the frozen homogenate (SRM 1974a).

Analytical Approach: Because SRM 2974 was prepared from the same frozen tissue homogenate as SRM 1974a, the analytical approach for certification of SRM 2974 was designed to use the results from the certification of SRM 1974a, which used several different analytical methods, in conjunction with GC/MS analyses of SRM 2974. The certified values for PAHs in SRM 1974a are based on results obtained from analyses using different extraction/sample preparation procedures and analytical techniques based on GC/MS on two stationary phases of different selectivity and on reversed-phase liquid chromatography with fluorescence detection (See Certificate of Analysis for SRM 1974a [1], and references [3-4] for details). This analytical approach for the measurement of PAHs was described in detail recently for the certification of a marine sediment [6]. The certified values for PCB congeners and chlorinated pesticides in SRM 1974a are based on results obtained from 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 as described previously for other environmental matrix SRMs [7-8]. For selected PCB congeners and chlorinated pesticides,

additional results obtained from two interlaboratory comparison exercises involving about 20 laboratories were also included in the determination of the certified values for SRM 1974a.

The analyses of SRM 2974 described below were performed to confirm the results of the analyses of SRM 1974a to determine whether differences exist between the frozen mussel tissue material (SRM 1974a) and the freeze-dried material (SRM 2974). For the inorganic constituents and methylmercury, results of analyses of both SRM 1974a and SRM 2974 were used to determine the certified and reference values for SRM 2974.

Polycyclic Aromatic Hydrocarbons: For the GC/MS analyses of SRM 2974, subsamples of 5 g to 7 g of the freeze-dried mussel tissue from three 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 methylene chloride. The extract was concentrated, and size exclusion chromatography (SEC) on a divinylbenzene-polystyrene column (10 μ m particle size, 100 X 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 approximately one-half of the sample was injected onto a semipreparative aminopropylsilane column to isolate the PAH fraction by normal-phase LC [9]. 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 expressed as percent) phenyl-substituted methylpolysiloxane phase (0.25 μ m film thickness)(DB-5 MS, J&W Scientific¹, Folsom, CA).

PCBs and Chlorinated Pesticides: The remaining portion of the concentrated eluant from the SEC was injected onto the same aminopropylsilane column to isolate the PCB and lower polarity pesticide fraction from the more polar pesticide fraction by normal-phase LC. Both fractions were analyzed by GC/MS on a 5 % phenyl-substituted methylpolysiloxane phase as described above for the PAH measurements.

Methylmercury and Mercury: The certified values for methylmercury and total 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. [10,11]. 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 [12] 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 [13]. 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 [14-16]. 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

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.

National Measurement Standards, National Research Council (NRC) of Canada, were used [17]. The four different procedures included: microwave digestion followed by flow injection cold-vapor atomic absorption spectrometry (NIST) [18], instrumental neutron activation analysis (INAA) (NIST) [19]; acid digestion at 70 °C followed by double gold trap amalgamation cold vapor atomic fluorescence spectrometry (IAEA) [20]; and acid digestion at 150 °C followed by gold wire amalgamation cold vapor atomic absorption spectrometry (Research Centre of Jülich) [21]. Subsamples from six bottles each of SRM 1974a and SRM 2974 were analyzed by each of the three laboratories.

Additional 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 [17]. 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 [19]. 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.

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Table 1. Certified Concentrations (mass fractions in $\mu\text{g}/\text{kg}$ dry mass basis) for Selected PAHs in SRM 2974^a

Phenanthrene	22.2	⊗	2.5
Anthracene	6.1	⊗	1.7
Fluoranthene	163.7	⊗	10.3
Pyrene	151.6	⊗	8.0
Benz[<i>a</i>]anthracene	32.5	⊗	4.8
Chrysene	44.2	⊗	2.7
Triphenylene	50.7	⊗	6.1
Benzo[<i>b</i>]fluoranthene	46.4	⊗	4.0
Benzo[<i>k</i>]fluoranthene	20.2	⊗	1.0
Benzo[<i>e</i>]pyrene	84.0	⊗	3.2
Benzo[<i>a</i>]pyrene	15.63	⊗	0.80
Perylene	7.68	⊗	0.35
Benzo[<i>ghi</i>]perylene	22.0	⊗	2.3
Indeno[1,2,3- <i>cd</i>]pyrene	14.2	⊗	2.8

^a The certified value for each analyte is equal to the corresponding value (dry mass basis) from SRM 1974a [1], which was derived from the same material as SRM 2974. 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 from the analysis of SRM 1974a and a component that accounts for the small systematic effect between the measured values of SRM 1974a and SRM 2974 as determined by GC/MS analyses.

Table 2. Certified Concentrations (mass fractions in $\mu\text{g}/\text{kg}$ dry mass basis) for Selected PCB Congeners^a in SRM 2974^b

PCB	44	(2,2',3,5'-Tetrachlorobiphenyl)	72.7	⊗	7.7
PCB	49	(2,2',4,5'-Tetrachlorobiphenyl)	88.8	⊗	5.7
PCB	52	(2,2',5,5'-Tetrachlorobiphenyl)	115	⊗	12
PCB	66	(2,3',4,4'-Tetrachlorobiphenyl)	101.4	⊗	5.4
PCB	95	(2,2',3,5',6-Pentachlorobiphenyl)	83	⊗	17
PCB	99	(2,2',4,4',5-Pentachlorobiphenyl)	70.9	⊗	4.5
PCB	101	(2,2',4,5,5'-Pentachlorobiphenyl)	128	⊗	10
PCB	105	(2,3,3',4,4'-Pentachlorobiphenyl)	53.0	⊗	3.8
PCB	110	(2,3,3',4',6-Pentachlorobiphenyl)	127.3	⊗	9.4
PCB	118	(2,3',4,4',5-Pentachlorobiphenyl)	130.8	⊗	5.3
PCB	128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	22.0	⊗	3.5
PCB	138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	134	⊗	10
	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)	87.6	⊗	3.5
PCB	151	(2,2',3,5,5',6-Hexachlorobiphenyl)	25.6	⊗	3.6
PCB	153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	145.2	⊗	8.8
PCB	156	(2,3,3',4,4',5-Hexachlorobiphenyl)	7.4	⊗	1.0
PCB	170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	5.5	⊗	1.1
PCB	180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)	17.1	⊗	3.8
PCB	183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)	16.0	⊗	2.4
PCB	187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	34.0	⊗	2.5
	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 Ballschmitter and Zell [23] and later revised by Schulte and Malisch [24] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmitter-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 certified value for each analyte is equal to the corresponding value from SRM 1974a [1], which was derived from the same material as SRM 2974. 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 from the analysis of SRM 1974a and a component that accounts for the small systematic effect between the measured values of SRM 1974a and SRM 2974 as determined by GC/MS analyses.

Table 3. Certified Concentrations (mass fractions in $\mu\text{g}/\text{kg}$ dry mass basis) for Selected Chlorinated Pesticides in SRM 2974^a

<i>cis</i> -Chlordane (Σ -Chlordane)	17.2	$\Sigma < 2.9$
<i>trans</i> -Chlordane	16.6	$\Sigma < 1.8$
<i>cis</i> -Nonachlor	6.84	$\Sigma < 0.92$
<i>trans</i> -Nonachlor	18.0	$\Sigma < 3.6$
4,4'-DDE	51.2	$\Sigma < 5.7$
4,4'-DDD	43.0	$\Sigma < 6.4$
4,4'-DDT	3.91	$\Sigma < 0.60$

^a The certified value for each analyte is equal to the corresponding value from SRM 1974a [1], which was derived from the same material as SRM 2974. 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 from the analysis of SRM 1974a and a component that accounts for the small systematic effect between the measured values of SRM 1974a and SRM 2974 as determined by GC/MS analyses.

Table 4. Certified Concentrations (mass fractions in $\mu\text{g}/\text{kg}$ dry mass basis) of Methylmercury and Total Mercury in SRM 2974

Methylmercury ^{a,b}	77.3	$\Sigma < 3.1$
Mercury ^c	176	$\Sigma < 13$

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

^b 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 [22] 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.

^c 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 [22] 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 in $\mu\text{g}/\text{kg}$ dry mass basis) for Selected PAHs in SRM 2974

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/MS on a similar column).

Naphthalene	9.63	✗	0.61 ^{a,b}
1-Methylnaphthalene	3.47	✗	0.85 ^{a,b}
2-Methylnaphthalene	6.48	✗	0.85 ^{a,b}
Biphenyl	4.68	✗	0.56 ^{a,b}
Acenaphthylene	4.60	✗	0.88 ^{a,b}
Acenaphthene	2.74	✗	0.52 ^{a,b}
Fluorene	4.69	✗	0.34 ^{a,b}
1-Methylphenanthrene	10.5	✗	4.8 ^c
2-Methylphenanthrene	20.6	✗	8.0 ^c
3-Methylphenanthrene	13.5	✗	9.7 ^c
4-Methylphenanthrene/9-Methylphenanthrene	14.7	✗	9.2 ^c
Benzo[<i>j</i>]fluoranthene	20.5	✗	1.8 ^c
Benzo[<i>a</i>]fluoranthene	4.0	✗	1.9 ^c
Dibenz[<i>a,j</i>]anthracene	1.247	✗	0.084 ^c
Dibenz[<i>a,c</i>]anthracene/Dibenz[<i>a,h</i>]anthracene	3.00	✗	0.22 ^c
Benzo[<i>b</i>]chrysene	1.60	✗	0.16 ^c
Anthanthrene	1.15	✗	0.31 ^c

^a The concentration for this analyte in SRM 2974 is lower than the concentration found in SRM 1974a due to losses during the freeze-drying process.

^b The reference value for this analyte is the mean of three independent GC/MS measurements. The uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [22] 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 three values. The coverage factor, $k = 4.30$, is the Student's t -value for a 95 % confidence interval with two degrees of freedom.

^c The reference value for each analyte is equal to the corresponding value from SRM 1974a [1], which was derived from the same material as SRM 2974. 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 from the analysis of SRM 1974a and a component that accounts for the small systematic effect between the measured values of SRM 1974a and SRM 2974 as determined by GC/MS analyses.

Table 6. Reference Concentrations (mass fractions in $\mu\text{g}/\text{kg}$ dry mass basis) for Selected PCB Congeners and Chlorinated Pesticides in SRM 2974^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 a similar column).

Polychlorinated Biphenyls ^b			$\mu\text{g}/\text{kg}$ dry mass basis ^a
PCB	18	(2,2',5-Trichlorobiphenyl)	26.8 \times 3.3 ^c
PCB	28	(2,4,4'-Trichlorobiphenyl)	79 \times 15
PCB	31	(2,4',5-Trichlorobiphenyl)	76 \times 21
PCB	87	(2,2',3,4,5'-Pentachlorobiphenyl)	54 \times 14
Chlorinated Pesticides			
Dieldrin			6.2 \times 1.3
2,4'-DDE			5.26 \times 0.31
2,4'-DDD			13.7 \times 2.8
2,4'-DDT			8.5 \times 1.9

^a The reference value for each analyte (except PCB 18 as noted below) is equal to the corresponding value from SRM 1974a [1], which was derived from the same material as SRM 2974. 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 from the analysis of SRM 1974a and a component that accounts for the small systematic effect between the measured values of SRM 1974a and SRM 2974 as determined by GC/MS analyses.

^b PCB congeners are numbered according to the scheme proposed by Ballschmitter and Zell [23] and later revised by Schulte and Malisch [24] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmitter-Zell numbers correspond to those of Schulte and Malisch.

^c Concentration for this analyte in SRM 2974 is lower than the concentration found in SRM 1974a due to losses during freeze-drying. The reference value for this analyte is the mean of three independent GC/MS and GC-ECD measurements. The uncertainty in the reference value is equal to $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [22] 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 three values. The coverage factor, $k = 4.30$, is the Student's t -value for a 95 % confidence interval with two degrees of freedom.

Table 7. Reference Concentrations (mass fractions) for Selected Inorganic Constituents in SRM 2974 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 are based on analysis of SRM 1974a; 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 dry mass basis		
Aluminum ^{c,d}	2	4.30	479	⌘	86
Arsenic ^e	8	2.31	7.4	⌘	1.1
Cadmium	7	2.36	1.23	⌘	0.09
Chromium ^e	9	2.26	2.00	⌘	0.11
Copper	7	2.36	10.2	⌘	1.0
Iron ^e	8	2.31	476	⌘	14
Lead	7	2.36	12.1	⌘	1.4
Nickel	5	2.57	0.83	⌘	0.15
Selenium ^e	8	2.31	2.01	⌘	0.35
Silver ^d	7	2.36	0.517	⌘	0.048
Tin	4	2.78	0.282	⌘	0.054
Zinc ^e	8	2.31	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 $U = ku_c$ where u_c is the combined standard uncertainty calculated according to the ISO Guide [22] 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 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.

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

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

Table 8. Reference Concentrations (mass fractions) for Selected Inorganic Constituents in SRM 2974 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. However, based on results for other elements in SRM 2974, SRM 1974a, and SRM 2974 are expected to be identical on a dry mass basis.

Element	percent (dry mass basis)		
Chlorine	7.5	⌘	0.2
Magnesium	0.051	⌘	0.002
Sodium	4.6	⌘	0.1
Potassium	1.15	⌘	0.04
	mg/kg (dry mass basis)		
Antimony	0.033	⌘	0.004
Bromine	318	⌘	9
Cerium	0.58	⌘	0.05
Cesium	0.028	⌘	0.002
Cobalt	0.33	⌘	0.01
Europium	0.0134	⌘	0.0006
Gold	0.024	⌘	0.002
Hafnium	0.038	⌘	0.004
Lanthanum	0.24	⌘	0.01
Manganese	10.6	⌘	0.5
Samarium	0.030	⌘	0.004
Scandium	0.090	⌘	0.003
Strontium	80	⌘	5
Tantalum	0.025	⌘	0.003
Thorium	0.062	⌘	0.008
Vanadium	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 [22] 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.

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>.