



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 2977

### Mussel Tissue (Organic Contaminants and Trace Elements)

This Standard Reference Material (SRM) 2977 is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, polybrominated diphenyl ether (BDE) congeners, methylmercury, and inorganic constituents in marine bivalve mollusk tissue and similar matrices. All of the constituents for which certified, reference, and information values are provided are naturally present in the freeze-dried mussel tissue. A unit of SRM 2977 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]. The collection, preparation, and value assignment of SRM 2977 was a collaboration between the National Institute of Standards and Technology (NIST) and National Research Council of Canada (NRCC).

**Certified Values:** Certified values for concentrations, expressed as mass fractions, for 13 PAHs, 19 PCB congeners, 6 chlorinated pesticides, 5 BDE congeners (some in combination), 6 trace elements, and methylmercury are provided in Tables 1 to 6. 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 the agreement of results obtained at NIST from two independent analytical techniques and from an interlaboratory comparison study. The certified values for the BDE congeners are based on the agreement of results obtained at NIST, from a collaborating laboratory, and from an interlaboratory comparison study. The certified values for the trace elements and methylmercury are based on NIST measurements by one technique and additional results from several collaborating laboratories.

**Reference Values:** Reference concentration values, expressed as mass fractions, are provided in Table 7 for 10 additional PAHs, 3 additional PCB congeners, 2 additional chlorinated pesticides, and five additional BDE congeners. Reference concentration values are provided in Table 8 for nine additional inorganic constituents. Reference values are noncertified values that represent best estimates 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.

**Information Values:** Information values for concentrations, expressed as mass fractions, are provided in Table 9 for 23 additional trace elements. An information value is considered to be a value that will be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed.

**Expiration of Certification:** The certification of SRM 2977 is valid, within the measurement uncertainty specified, until **31 December 2017**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

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 S. D. Leigh, M.G Vangel and M.S. Levenson of the NIST Statistical Engineering Division.

Stephen A. Wise, Chief  
Analytical Chemistry Division

Robert L. Watters, Jr., Chief  
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Gaithersburg, MD 20899  
Certificate Issue Date: 12 September 2008  
*See Certificate Revision History on Last Page*

The mussels were collected under the supervision of A. Wagener from the Pontificia Universidade Catolica, Do Rio De Janeiro, Brazil. The mussel tissue was freeze-dried at the Natural Products Support Group at the Frederick Cancer Research and Development Center (Frederick, MD) under the direction of T. McCloud. Preparation of the freeze-dried material was performed by M.P. Cronise and C.N. Fales of the NIST Measurement Services Division.

Analytical measurements at NIST were performed by W.C. Davis, J.M. Keller, J.R. Kucklick, M.J. Lopez de Alda, B.J. Porter, M.M. Schantz, S. Tutschku, and L. Yu of the NIST Analytical Chemistry Division.

Analytical measurements for selected PCB congeners were also performed at the Institute for National Measurement Standards, NRCC (Ottawa, Canada) by G. Gardner and C. Frasier. Results for selected PAHs, PCB congeners, chlorinated pesticides, and BDE congeners were also used from 12 laboratories that participated in an intercomparison exercise coordinated by M. M. Schantz of the NIST Analytical Chemistry Division. Analytical measurements for selected trace elements and methylmercury were also performed at the Institute of Applied Physical Chemistry, Research Centre Jülich (Jülich, Germany) by H. Emons and at the Department of Environmental Sciences, Jožef Stefan Institute (Ljubljana, Slovenia) by M. Horvat. Analytical measurements for selected BDE congeners were also performed at Indiana University (Bloomington, IN) by Y.L. Zhu and R.A. Hites. Results for selected trace elements were also used from six laboratories that participated in an intercomparison exercise coordinated by S. Willie of the Institute for National Measurement Standards, NRCC.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services 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. Registration (see attached sheet) will facilitate notification.

## INSTRUCTIONS FOR USE

Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 2977 are reported on a dry-mass basis. The freeze-dried mussel tissue homogenate is hygroscopic, and as received, contains greater than 3 % (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.

## NOTICE AND WARNING TO USERS

**Storage:** SRM 2977 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.

## PREPARATION AND ANALYSIS<sup>1</sup>

**Sample Collection and Preparation:** The mussels (*Perna perna*, edible brown mussel) used for the preparation of SRM 2977 were collected in Guanabara Bay, Brazil. The mussels were shucked, and the tissue was shipped to NIST on dry ice in two batches, each containing approximately 35 kg. For processing, the tissue was allowed to partially thaw and was transferred into a Robot Coupe Vertical Cutter Mixer until it was half full. The mussel tissue was blended for 5 min into a puree form and then poured into metal trays and frozen. The material was then freeze-dried with a starting temperature of -10 °C and slowly warmed to a temperature of 10 °C. The dry material was broken into smaller chunks and then jet milled to produce a fine powder. The powder was blended for homogeneity by processing through the jet mill twice. The material was radiation sterilized (<sup>60</sup>Co) and then aliquoted into jars (~ 10 g each).

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<sup>1</sup>Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

**PAHs, PCBs , and Chlorinated Pesticides:** The general approach used for the value assignment of the PAHs, PCBs, and chlorinated pesticides in SRM 2977 was similar to that reported for the recent certification of several environmental matrix SRMs [2] and consisted of combining results from analyses using various combinations of different extraction techniques, cleanup/isolation procedures, and chromatographic separation and detection techniques.

Two sets of gas chromatography/mass spectrometry (GC/MS) results, designated as GC/MS (I) and GC/MS (II), were obtained at NIST. For GC/MS (I) analyses, single subsamples of 3 g from three bottles of SRM 2977 were extracted using PFE with DCM as described by Schantz et al. [3]. 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. × 60 cm, PL-Gel, Polymer Labs, Inc., Amherst, MA) was used to remove the majority of the lipid and biogenic material. The extract was further fractionated using a silica solid phase extraction (SPE) column to isolate the fraction of interest. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 30 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.25 µm film thickness) (HP-5 MS, Agilent Technologies, Wilmington, DE) and a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % (mole fraction) phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-17 MS, Agilent Technologies). For the GC/MS (II) analyses, one sample (2 g) from each of three bottles was extracted using PFE with DCM. The fraction of interest was isolated using an alumina column (5% deactivated) followed by an amiopropylsilane SPE column. The isolated fraction was then analyzed by GC/MS using a 0.18 mm i.d. × 30 m fused silica capillary column with a proprietary non-polar phase (0.18 µm film thickness) (DB-XLB, Agilent Technologies). For both methods described above, selected perdeuterated PAHs, carbon-13 labeled PCBs, and perdeuterated pesticides 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 2977 was used in 2005 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [4]. Results from 12 laboratories that participated in this exercise were used as the third data set in the determination of the assigned values for PAHs, PCBs, and chlorinated pesticides in SRM 2977. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure the analytes of interest.

**Homogeneity Assessment for PAHs, PCBs, and Chlorinated pesticides:** The homogeneity of SRM 2977 was assessed by analyzing duplicate 3 g samples from eight bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at the 3 g sample size.

**BDEs:** Value assignment of concentrations for BDE congeners was based on three sets of data (one set from NIST, one set from a collaborating laboratory, and one set from an interlaboratory comparison study) using a variety of different extraction, cleanup, and quantification methods. All measurements were performed by using GC/MS operated in either electron impact (GC/EI-MS) or negative chemical ionization (GC/NCI-MS) mode.

For the NIST data set (GC/MS III), 3 g to 4 g subsamples of tissue from each of three bottles were extracted using PFE with DCM. The extracts were processed as above using SEC followed by a second cleanup step using a 5 % deactivated alumina SPE column. The extracts were analyzed by using GC/EI-MS on a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5MS, Agilent Technologies). <sup>13</sup>C-Labeled 2,2,4,4',5'-pentabromodiphenyl ether (BDE 99) was added to the tissue samples prior to extraction for use as an internal standard for quantification of the BDEs.

For the measurements from the collaborating laboratory (Indiana University, Bloomington, IN) (GC/MS IV), five subsamples of SRM 2977 were Soxhlet extracted using hexane:acetone (1:1, volume fraction) after spiking with two internal standards, <sup>13</sup>C-labeled 2,3,3',4,4',5-hexachlorodiphenyl ether (CDE 156) and <sup>13</sup>C-labeled 2,2',3,3',4,4',5,5'-octachlorodiphenyl ether (CDE 194). Lipids were removed by adding concentrated H<sub>2</sub>SO<sub>4</sub> and shaking; the organic phase was collected and the extracts were further cleaned using a 3 % deactivated silica column and an alumina column in series. The extracts were analyzed by using GC/NCI-MS on a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5, Agilent Technologies). Details of the analyses by the collaborating laboratory are presented by Zhu and Hites [5].

SRM 2977 was used in 2005 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [4]. Results from 12 laboratories that participated in this exercise were used as the third data set in the determination of the assigned values for BDEs in SRM 2977. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure the analytes of interest.

**Analytical Approach for Inorganic Constituents:** Value assignment of the concentrations of selected trace elements was accomplished by combining results of the analyses of SRM 2977 at NIST, NRCC, Research Centre Jülich, Jožef Stefan Institute, and six selected laboratories that participated in an interlaboratory comparison exercise coordinated by the NRCC [6].

For the certified concentration values listed in Table 5, results were combined from analyses at NIST using inductively coupled plasma mass spectrometry (ICP-MS), analyses at NRCC using isotope dilution (ID) ICP-MS and graphite furnace atomic absorption spectrometry (GFAAS), analyses at Research Centre Jülich using one to four techniques, analyses at Jožef Stefan Institute using one or two techniques, and the mean of the results from six laboratories that participated in the NRCC interlaboratory comparison exercise. For the reference values provided in Table 8, results were combined from NIST, NRCC, Jožef Stefan Institute, Research Centre Jülich, and the NRCC interlaboratory comparison exercise. The information values in Table 9 are based on results of analyses at NRCC, Jožef Stefan Institute, and/or Research Centre Jülich. The analytical techniques used for the analysis of SRM 2977 for inorganic constituents are summarized in Table 10.

**NIST Trace Element Analyses:** The elements cadmium, cobalt, nickel, lead, copper, manganese, and strontium were determined using ICP-MS, quantified by the method of standard addition. Five mL of concentrated HNO<sub>3</sub> was added to 0.5 g subsamples from each of five bottles of SRM 2977. These samples were digested in closed vessels using programmed heating in a microwave oven. The resulting tissue digests were quantitatively diluted into two concentration ranges; rhodium was added to each as an internal standard. The elements copper, manganese, and strontium were determined in the more dilute solution; cadmium, copper, nickel, and lead were determined in the more concentrated solution. Two spike solutions containing each of these groups of elements were prepared and added to a split portion of each digest solution for the purpose of quantification by the method of standard addition. Prior to the quantitative determination of the analyte elements, an ICP-MS semi-quantitative analysis was performed to assess possible isobaric interferences. A correction was made for a molybdenum oxide interference on cadmium, the only interference observed.

**NRCC Trace Element Analyses:** Subsamples (0.25 g) from each of six bottles were placed in polytetrafluoroethylene (PTFE) digestion vessels with nitric acid; the vessels were sealed and heated in a microwave oven. (For the samples intended for ICP-MS analyses, a suitable amount of each enriched isotope solution was added to each sample prior to digestion). The digestion vessels were opened, H<sub>2</sub>O<sub>2</sub> was added to the samples for GFAAS and H<sub>2</sub>O<sub>2</sub> and HF were added to the samples for ICP-Atomic Emission Spectrometry (AES), and the contents were evaporated to dryness. The residues were dissolved in nitric acid and double distilled water. The samples were analyzed by ID-ICP-MS for the determination of silver, cadmium, copper, nickel, lead, tin, and zinc. GFAAS was used for determination of silver, arsenic, cadmium, chromium, copper, nickel, and selenium, and ICP-AES was used for the determination of aluminum, iron, and zinc.

**Research Centre Jülich Trace Element Analyses:** The elements manganese, strontium, iron, zinc, calcium, magnesium, phosphorus, potassium, sodium, sulfur, and barium were determined by ICP-AES after pressure digestion (0.2 g of sample + 2 mL of HNO<sub>3</sub>) in PTFE vessels. Aqueous acid-matched standard solutions containing scandium as an internal standard were used for calibration. The elements phosphorus and sulfur were determined without an internal standard. The elements cadmium, lead, and copper were determined in aliquots of corresponding digestion solutions by GFAAS using the method of standard addition [7,8]. ID-TIMS was used for the determination of cadmium, lead, copper, zinc, and thallium in solutions from pressure digestion (0.2 g of sample + 2 mL of HNO<sub>3</sub> + 0.2 mL HF) [9].

For mercury determination by cold vapor atomic absorption spectrometry (CVAAS), a subsample of 0.3 g to 0.5 g of material was digested with 10 mL concentrated nitric acid in heated quartz vessels closed with a cap [10]. The measuring system was calibrated using mercury (II) standard solutions in nitric acid. After high-pressure digestion (HPA) in quartz vessels (0.2 g of sample + 2 mL of HNO<sub>3</sub>), cadmium, lead, copper, nickel, and thallium were determined by ICP-MS using aqueous standard solutions for calibration. In aliquots of HPA digestion solutions, electrochemical techniques were used for the determination of lead (differential pulse anodic stripping voltammetry (DPASV)), nickel (adsorptive stripping voltammetry (ADSV)), and selenium (cathodic stripping voltammetry (CSV)) at the hanging mercury drop electrode by standard addition method [11]. Selenium was quantified in HPA digestion and arsenic after open wet digestion (0.2 g of sample + 3 mL of HNO<sub>3</sub>) by HG-AAS using aqueous standard solutions for calibration.

**Jožef Stefan Institute Trace Element Analyses:** Subsamples from each of six bottles of SRM 2977 were analyzed by electrothermal atomic absorption spectrometry (ETAAS), flame atomic absorption spectrometry (FAAS), instrumental neutron activation analysis (INAA), and radiochemical neutron activation analysis (RNAA). For the determination of trace elements by FAAS (iron, manganese, zinc, and copper) and ETAAS (cadmium, lead, and vanadium), subsamples of 300 mg were placed in PTFE Parr bombs with nitric acid and heated at 105 °C for 12 h. After digestion, the samples were equilibrated to room temperature and diluted with double distilled water.

For INAA, subsamples of 150 mg to 200 mg were sealed in plastic containers and irradiated for 20 h at a fluence rate of  $1.0 \times 10^{12} \text{ cm}^{-2} \cdot \text{s}^{-1}$ . For the short-lived radionuclides, samples were irradiated for 1 min. The irradiated samples were transferred to clean polyethylene containers and counted after 2, 8, and 30 days. For the short-lived radionuclides, samples

were counted at 2 min after irradiation for 5 min and at 3 h for 30 min. Samples were counted with a germanium detector. For the determination of mercury and selenium by RNAA, subsamples were sealed in quartz ampoules and irradiated for 16 h to 20 h at the fluence rate above. The samples were pyrolyzed resulting in volatilization of the mercury and selenium; selenium was trapped on soda lime and mercury was trapped on selenium-impregnated paper. The gamma activity of the isolated radionuclides was counted with a NaI(Tl) detector.

**Methylmercury:** The certified value for methylmercury is based on results of analyses of SRM 2977 at NIST and two other laboratories: Institute of Applied Physical Chemistry, Research Centre Jülich, (Jülich, Germany) and the Jožef Stefan Institute (Ljubljana, Slovenia). For the determination of methylmercury, SRM 2977 was analyzed at NIST using solid phase microextraction (SPME) with speciated isotope dilution GC/inductively coupled plasma mass spectrometry (GC/ICPMS). For the speciated isotope dilution GC/ICPMS analyses, approximately 1.0 g to 2.0 g subsamples were spiked with an appropriately diluted sample of IRMM-670 <sup>202</sup>Hg enriched methylmercury isotopic CRM and subjected to an alkaline microwave digestion (using 25 % volume fraction tetraammoniumhydroxide in water). Sodium tetraethylborate was used for ethylation. The derivatized methylmercury was back-extracted into iso-octane and injected into a GC/ICPMS. The GC analysis used a 30 m × 0.32 mm column with a 100 % dimethylpolysiloxane phase (0.17 μm film thickness) (HP-1, Agilent Technologies) [12]. At the Research Centre Jülich, the analytical procedure for methylmercury consisted of water steam distillation under acid conditions, anion exchange chromatographic separation of inorganic mercury and methylmercury, followed by CVAAS detection before and after ultraviolet radiation [13-15]. Triplicate subsamples (~ 300 mg) from each of three bottles were analyzed. Three methods were used for the determination of methylmercury at the Jožef Stefan Institute: (1) HCl extraction for 12 h anion exchange chromatographic separation of inorganic mercury and organomercury followed by cold vapor atomic absorption spectrometric detection before and after ultraviolet radiation (IEC-CVAAS) [13,14,16]; (2) H<sub>2</sub>SO<sub>4</sub> extraction followed by ethylation at room temperature pre-collection, GC-pyrolysis with cold vapor atomic fluorescence spectrometric detection (GC-CVAFS) [17-20]; and (3) solid-liquid extraction into toluene followed by GC-ECD [16,19,21]. Six subsamples (200 mg to 500 mg) from one bottle of SRM 2977 were analyzed for each of the three analytical techniques and a subsample (500 mg) from each of six bottles of SRM 2977 was analyzed by one technique (GC-ECD).

Table 1. Certified Concentrations for Selected PAHs in SRM 2977

	Mass Fraction μg/kg (dry mass basis)	
Fluorene <sup>(b,c,d)</sup>	10.30	± 0.13 <sup>(a)</sup>
Phenanthrene <sup>(b,c,d)</sup>	36.2	± 2.5 <sup>(a)</sup>
1-Methylphenanthrene <sup>(b,c,d)</sup>	39.0	± 1.9 <sup>(e)</sup>
Fluoranthene <sup>(b,c,d)</sup>	38.90	± 0.63 <sup>(e)</sup>
Pyrene <sup>(b,c,d)</sup>	77.4	± 2.1 <sup>(a)</sup>
Benz[ <i>a</i> ]anthracene <sup>(b,c,d)</sup>	20.19	± 0.87 <sup>(a)</sup>
Benzo[ <i>b</i> ]fluoranthene <sup>(b,c,d)</sup>	11.10	± 0.50 <sup>(e)</sup>
Benzo[ <i>j</i> ]fluoranthene <sup>(b,c,d)</sup>	4.48	± 0.15 <sup>(a)</sup>
Benzo[ <i>e</i> ]pyrene <sup>(b,c,d)</sup>	13.29	± 0.43 <sup>(a)</sup>
Benzo[ <i>a</i> ]pyrene <sup>(b,c,d)</sup>	5.30	± 0.61 <sup>(a)</sup>
Perylene <sup>(b,c,d)</sup>	3.69	± 0.38 <sup>(e)</sup>
Benzo[ <i>ghi</i> ]perylene <sup>(b,c,d)</sup>	9.45	± 0.37 <sup>(e)</sup>
Indeno[1,2,3- <i>cd</i> ]pyrene <sup>(b,c,d)</sup>	4.76	± 0.15 <sup>(a)</sup>

<sup>(a)</sup> The certified value is a weighted mean of the results from three analytical methods [22]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [23].

<sup>(b)</sup> GC/MS I

<sup>(c)</sup> GC/MS II

<sup>(d)</sup> Results from up to 12 laboratories participating in an interlaboratory comparison exercise.

<sup>(e)</sup> The certified value is an unweighted mean of the results from three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance [24] with a pooled, within-method variance following the ISO and NIST Guides [23].

Table 2. Certified Concentrations for Selected PCB Congeners<sup>(a)</sup> in SRM 2977

		Mass Fraction µg/kg (dry mass basis)
PCB 8	(2,4'-Dichlorobiphenyl) <sup>(c,d,e)</sup>	1.99 ± 0.14 <sup>(b)</sup>
PCB 28	(2,4,4'-Trichlorobiphenyl) <sup>(c,d,e)</sup>	5.17 ± 0.36 <sup>(f)</sup>
PCB 31	(2,4',5-Trichlorobiphenyl) <sup>(c,d,e)</sup>	3.86 ± 0.29 <sup>(f)</sup>
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl) <sup>(c,d,e)</sup>	3.22 ± 0.21 <sup>(f)</sup>
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl) <sup>(c,d,e)</sup>	2.44 ± 0.27 <sup>(b)</sup>
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl) <sup>(c,d,e)</sup>	8.02 ± 0.56 <sup>(b)</sup>
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl) <sup>(c,d,e)</sup>	3.55 ± 0.18 <sup>(f)</sup>
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl) <sup>(c,d,e)</sup>	5.17 ± 0.53 <sup>(b)</sup>
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl) <sup>(c,d,e)</sup>	10.6 ± 0.9 <sup>(b)</sup>
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl) <sup>(c,d,e)</sup>	10.0 ± 0.41 <sup>(b)</sup>
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl) <sup>(c,d,e)</sup>	2.38 ± 0.28 <sup>(b)</sup>
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl) <sup>(c,d,e)</sup>	7.94 ± 0.63 <sup>(f)</sup>
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl) <sup>(c,d,e)</sup>	8.95 ± 0.67 <sup>(b)</sup>
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl) <sup>(c,d,e)</sup>	14.1 ± 1.3 <sup>(b)</sup>
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl) <sup>(c,d,e)</sup>	0.959 ± 0.036 <sup>(f)</sup>
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl) <sup>(c,d,e)</sup>	2.74 ± 0.25 <sup>(b)</sup>
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) <sup>(c,d,e)</sup>	6.32 ± 0.72 <sup>(b)</sup>
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl) <sup>(c,d,e)</sup>	4.47 ± 0.32 <sup>(b)</sup>
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl) <sup>(c,d,e,f)</sup>	0.881 ± 0.032 <sup>(f)</sup>

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [25] and later revised by Schulte and Malisch [26] 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.

<sup>(b)</sup> The certified value is a weighted mean of the results from three analytical methods [22]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [23].

<sup>(c)</sup> GC/MS I

<sup>(d)</sup> GC/MS II

<sup>(e)</sup> Results from up to 12 laboratories participating in an interlaboratory comparison exercise.

<sup>(f)</sup> The certified value is an unweighted mean of the results from three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance [24] with a pooled, within-method variance following the ISO and NIST Guides [23].

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 2977

	Mass Fraction µg/kg (dry mass basis)
<i>trans</i> -Nonachlor <sup>(b,c,d)</sup>	1.25 ± 0.17 <sup>(a)</sup>
Dieldrin <sup>(b,c,d)</sup>	5.55 ± 0.61 <sup>(a)</sup>
4,4'-DDE <sup>(b,c,d)</sup>	11.8 ± 1.2 <sup>(e)</sup>
2,4'-DDD <sup>(b,c,d)</sup>	3.15 ± 0.25 <sup>(e)</sup>
4,4'-DDD <sup>(b,c,d)</sup>	3.92 ± 0.56 <sup>(a)</sup>
4,4'-DDT <sup>(b,c,d)</sup>	1.32 ± 0.16 <sup>(a)</sup>

<sup>(a)</sup> The certified value is a weighted mean of the results from three analytical methods [22]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [23].

<sup>(b)</sup> GC/MS I

<sup>(c)</sup> GC/MS II

<sup>(d)</sup> Results from up to 12 laboratories participating in an interlaboratory comparison exercise.

<sup>(e)</sup> The certified value is an unweighted mean of the results from three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance [24] with a pooled, within-method variance following the ISO and NIST Guides [23].

Table 4. Certified Concentrations for Selected BDE Congeners<sup>(a)</sup> in SRM 2977

	Mass Fraction µg/kg (dry mass basis)
BDE 28 (2,4,4'-Tribromodiphenyl ether) <sup>(c,d,e)</sup>	2.54 ± 0.40 <sup>(b)</sup>
BDE 33 (2',3,4-Tribromodiphenyl ether)	
BDE 47 (2,2',4,4'-Tetrabromodiphenyl ether) <sup>(c,d,e)</sup>	36.5 ± 4.0 <sup>(b)</sup>
BDE 49 (2,2',4,5'-Tetrabromodiphenyl ether) <sup>(c,d,e)</sup>	1.20 ± 0.19 <sup>(b)</sup>
BDE 66 (2,3',4,4'-Tetrabromodiphenyl ether) <sup>(c,d,e)</sup>	0.453 ± 0.046 <sup>(b)</sup>

<sup>(a)</sup> BDE congeners are numbered according to IUPAC rules. When two or more congeners are known to coelute under the GC analysis conditions used, the BDE 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.

<sup>(b)</sup> The certified value is a weighted mean of the results from three analytical methods [22]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [23].

<sup>(c)</sup> GC/MS III

<sup>(d)</sup> GC/MS IV [5]

<sup>(e)</sup> Results from up to 12 laboratories participating in an interlaboratory comparison exercise.



Table 5. Certified Concentrations for Selected Inorganic Constituents in SRM 2977

Element	Degrees of Freedom	Mass Fraction mg/kg (dry mass basis) <sup>(a)</sup>
Cadmium <sup>(b,c,d,e,f,g,h)</sup>	4	0.179 ± 0.003
Copper <sup>(b,c,d,e,f,g,h,j)</sup>	5	9.42 ± 0.52
Lead <sup>(b,c,d,e,f,g,h,k,l)</sup>	4	2.27 ± 0.13
Manganese <sup>(b,i,m)</sup>	2	23.93 ± 0.29
Nickel <sup>(b,c,d,e,g,n)</sup>	4	6.06 ± 0.24
Strontium <sup>(b,i,m)</sup>	2	69.3 ± 4.2

<sup>(a)</sup> The results are expressed as the certified value ± the expanded uncertainty. The certified value is the mean of three to six results from the following: (1) the mean of ICP-MS analyses performed at NIST; (2) the mean of ID-ICP-MS analyses performed at NRCC; (3) the mean of GFAAS analyses performed at NRCC; (4) the mean of results from six selected laboratories participating in the NRCC intercomparison exercise; (5) the mean of results from analyses by HGAAS, GFAAS, ICP-MS, ID-TIMS, ADSV, and/or DPASV performed at Research Centre Jülich; and (6) the mean of results from analyses by INAA, GFAAS, and FAAS performed at Jožef Stefan Institute. The expanded uncertainty in the certified value is equal to  $U = ku_c$ , where  $u_c$  is the combined standard uncertainty calculated according to the ISO and NIST Guides [23] 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 available values.

<sup>(b)</sup> Measured at NIST using ICP-MS.

<sup>(c)</sup> Measured at NRCC using ID-ICP-MS.

<sup>(d)</sup> Measured at NRCC using GFAAS.

<sup>(e)</sup> Measured by six laboratories as part of the NRCC interlaboratory comparison exercise.

<sup>(f)</sup> Measured at Research Centre Jülich using GFAAS.

<sup>(g)</sup> Measured at Research Centre Jülich using ICP-MS.

<sup>(h)</sup> Measured at Research Centre Jülich using ID-TIMS.

<sup>(i)</sup> Measured at Jožef Stefan Institute using INAA.

<sup>(j)</sup> Measured at Jožef Stefan Institute using FAAS.

<sup>(k)</sup> Measured at Jožef Stefan Institute using GFAAS.

<sup>(l)</sup> Measured at Research Centre Jülich using DPASV.

<sup>(m)</sup> Measured at Research Centre Jülich using ICP-AES.

<sup>(n)</sup> Measured at Research Centre Jülich using ADSV.

Table 6. Certified Concentration of Methylmercury in SRM 2977

	Mass Fraction µg/kg (dry mass basis)
Methylmercury <sup>(a,b)</sup>	36.6 ± 1.0

<sup>(a)</sup> Results for methylmercury are reported as µg/kg mercury.

<sup>(b)</sup> The certified value is a weighted mean of the results from three analytical methods [22]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [23].

Table 7. Reference Concentrations for Selected PAHs, PCB Congeners, Chlorinated Pesticides, and BDE Congeners in SRM 2977

	Mass Fraction μg/kg (dry mass basis)
Naphthalene <sup>(b,c,d)</sup>	21.1 ± 1.4 <sup>(a)</sup>
1-Methylnaphthalene <sup>(b,c,d)</sup>	15.6 ± 1.5 <sup>(e)</sup>
2-Methylnaphthalene <sup>(b,c,d)</sup>	17.3 ± 1.7 <sup>(e)</sup>
Biphenyl <sup>(b,c,d)</sup>	6.0 ± 1.3 <sup>(a)</sup>
Acenaphthene <sup>(b,c,d)</sup>	4.9 ± 1.2 <sup>(a)</sup>
Anthracene <sup>(b,c,d)</sup>	6.2 ± 1.4 <sup>(a)</sup>
Chrysene <sup>(b,c,d)</sup>	42.2 ± 5.5 <sup>(a)</sup>
Triphenylene <sup>(b,c,d)</sup>	36.1 ± 2.4 <sup>(a)</sup>
Benzo[ <i>k</i> ]fluoranthene <sup>(b,c,d)</sup>	4.02 ± 0.75 <sup>(a)</sup>
Dibenz[ <i>a,h</i> ]anthracene <sup>(b,c,d)</sup>	1.47 ± 0.33 <sup>(a)</sup>
PCB 18 <sup>f</sup> (2,2',5-Trichlorobiphenyl) <sup>(b,c,d)</sup>	2.24 ± 0.74 <sup>(a)</sup>
PCB 99 <sup>f</sup> (2,2',4,4',5-Pentachlorobiphenyl) <sup>(b,c,d)</sup>	3.0 ± 1.2 <sup>(a)</sup>
PCB 105 <sup>f</sup> (2,3,3',4,4'-Pentachlorobiphenyl) <sup>(b,c,d)</sup>	2.93 ± 0.46 <sup>(a)</sup>
<i>cis</i> -Chlordane (α-Chlordane) <sup>(b,c,d)</sup>	1.14 ± 0.39 <sup>(a)</sup>
<i>trans</i> -Chlordane (γ-Chlordane) <sup>(b,c,d)</sup>	2.01 ± 0.39 <sup>(a)</sup>
BDE 17 <sup>f</sup> (2,2',4-Tribromodiphenyl ether) <sup>(d,g,h)</sup>	1.04 ± 0.19 <sup>(a)</sup>
BDE 99 <sup>f</sup> (2,2',4,4',5-Pentabromodiphenyl ether) <sup>(d,g,h)</sup>	4.68 ± 0.92 <sup>(e)</sup>
BDE 100 <sup>f</sup> (2,2',4,4',6-Pentabromodiphenyl ether) <sup>(d,g,h)</sup>	1.82 ± 0.64 <sup>(a)</sup>
BDE 153 <sup>f</sup> (2,2',4,4',5,5'-Hexabromodiphenyl ether) <sup>(d,g,h)</sup>	0.16 ± 0.04 <sup>(e)</sup>
BDE 154 <sup>f</sup> (2,2',4,4',5,6'-Hexabromodiphenyl ether) <sup>(d,g,h)</sup>	0.20 ± 0.09 <sup>(e)</sup>

<sup>(a)</sup> The reference value is a weighted mean of the results from three analytical methods [22]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) calculated by combining a between-method variance incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [23].

<sup>(b)</sup> GC/MS I

<sup>(c)</sup> GC/MS II

<sup>(d)</sup> Results from up to 12 laboratories participating in an interlaboratory comparison exercise.

<sup>(e)</sup> The reference value is an unweighted mean of the results from three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance [24] with a pooled, within-method variance following the ISO and NIST Guides [23].

<sup>(f)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmitter and Zell [25] and later revised by Schulte and Malisch [26] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, the Ballschmitter-Zell numbers correspond to those of Schulte and Malisch. BDE congeners are numbered according to IUPAC rules.

<sup>(g)</sup> GC/MS III

<sup>(h)</sup> GC/MS IV [5]

Table 8. Reference Concentrations for Selected Inorganic Constituents in SRM 2977 as Determined by Multiple Laboratories

Element	Degrees of Freedom	Mass Fraction mg/kg (dry mass basis) <sup>(a)</sup>
Arsenic <sup>(b,c,d,e)</sup>	3	8.83 ± 0.91
Chromium <sup>(b,c,d)</sup>	2	3.91 ± 0.47
Cobalt <sup>(c,f,g)</sup>	2	0.48 ± 0.13
Iron <sup>(b,c,d,h)</sup>	4	274 ± 18
Mercury <sup>(d,i,j,k,l)</sup>	3	0.101 ± 0.004
Selenium <sup>(b,c,d,e,k,m)</sup>	3	1.78 ± 0.16
Silver <sup>(b,c,d,n)</sup>	3	4.58 ± 0.33
Tin <sup>(d,n)</sup>	5	1.47 ± 0.27
Zinc <sup>(b,c,d,h,n,p)</sup>	4	135 ± 5

<sup>(a)</sup> The results are expressed as the reference value ± the expanded uncertainty. The reference value is the mean of three to five values from the following values: (1) the mean of ID-ICP-MS analyses performed at NIST; (2) the mean of ID-ICP-MS analyses performed at NRCC; (3) the mean of GFAAS or ICP-AES analyses performed at NRCC; (4) the mean of results from five or six selected laboratories participating in the NRCC intercomparison exercise; (5) the mean of results from analyses by CSV, HGAAS, ICP-AES, and/or ID-TIMS performed at Research Centre Jülich; and (6) the mean of results from analyses by CVAAS, INAA, and/or RNAA performed at Ljubljana. The expanded uncertainty in the certified value is equal to  $U = ku_c$  where  $u_c$  is the combined standard uncertainty calculated according to the ISO and NIST Guides [23] 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 available values. The coverage factor,  $k$ , is the Student's  $t$ -value for a 95 % confidence interval with four degrees of freedom.

<sup>(b)</sup> Measured at NRCC using GFAAS or ICP-AES.

<sup>(c)</sup> Measured at Jožef Stefan Institute using INAA.

<sup>(d)</sup> Measured by five or six laboratories as part of the NRCC interlaboratory comparison exercise.

<sup>(e)</sup> Measured at Research Centre Jülich using HGAAS.

<sup>(f)</sup> Measured at NIST using ICP-MS.

<sup>(g)</sup> Measured at Research Centre Jülich using ICP-MS.

<sup>(h)</sup> Measured at Research Centre Jülich using ICP-AES.

<sup>(i)</sup> Measured at NRCC using CVAAS.

<sup>(j)</sup> Measured at Research Centre Jülich using CVAAS.

<sup>(k)</sup> Measured at Jožef Stefan Institute using RNAA.

<sup>(l)</sup> Measured at Jožef Stefan Institute using CVAAS.

<sup>(m)</sup> Measured at Research Centre Jülich using CSV.

<sup>(n)</sup> Measured at NRCC using ID-ICP-MS.

<sup>(o)</sup> The reference value for tin is the mean of the results from NRCC using ID-ICP-MS and the individual results from five laboratories participating in the NRCC interlaboratory exercise ( $n = 6$ ).

<sup>(p)</sup> Measured at Research Centre Jülich using ID-TIMS.

Table 9. Information Values for the Concentrations for Selected Inorganic Constituents in SRM 2977

Element	Mass Fraction % (dry mass basis)
Calcium <sup>(a,b)</sup>	0.83
Chlorine <sup>(b)</sup>	4.3
Magnesium <sup>(a,b)</sup>	3.9
Phosphorus <sup>(a)</sup>	1.1
Potassium <sup>(a,b)</sup>	1.2
Sodium <sup>(a,b)</sup>	2.4
Sulfur <sup>(a,b)</sup>	2.9
	mg/kg (dry mass basis)
Aluminum <sup>(b,c)</sup>	400
Antimony <sup>(b)</sup>	0.048
Barium <sup>(a)</sup>	4.7
Bromine <sup>(b)</sup>	215
Cerium <sup>(b)</sup>	0.93
Cesium <sup>(b)</sup>	0.039
Gold <sup>(b)</sup>	0.013
Iodine <sup>(b)</sup>	26
Lanthanum <sup>(b)</sup>	0.44
Rubidium <sup>(b)</sup>	6.7
Samarium <sup>(b)</sup>	0.064
Scandium <sup>(b)</sup>	0.055
Thorium <sup>(b)</sup>	0.19
Uranium <sup>(b)</sup>	0.083
Vanadium <sup>(b)</sup>	1.1
	µg/kg (dry-mass basis)
Thallium <sup>(d,e)</sup>	10.2

<sup>(a)</sup> Measured at Research Centre Jülich using ICP-AES.

<sup>(b)</sup> Measured at Jožef Stefan Institute using INAA.

<sup>(c)</sup> Measured at NRCC using ICP-AES.

<sup>(d)</sup> Measured at Research Centre Jülich using ICP-MS.

<sup>(e)</sup> Measured at Research Centre Jülich using ID-TIMS.

Table 10. Analytical Methods Used for the Analysis of SRM 2977 for Inorganic Constituents

Elements	Analytical Methods
Aluminum	ICP-AES, INAA
Antimony	INAA
Arsenic	CSV, GFAAS, HGAAS, ICP-AES, ICP-MS, INAA
Barium	ICP-AES
Bromine	INAA
Cadmium	FAAS, GFAAS, ICP-MS, ICP-AES, ID-ICP-MS, ID-TIMS
Calcium	INAA, ICP-AES
Cerium	INAA
Cesium	INAA
Chlorine	INAA
Chromium	GFAAS, ICP-MS, INAA
Cobalt	ICP-MS, INAA, RNAA
Copper	FAAS, GFAAS, ICP-AES, ICP-MS, ID-ICP-MS, ID-TIMS
Iodine	INAA
Iron	FAAS, ICP-AES, ICP-MS, INAA
Lanthanum	INAA
Lead	DPASV, GFAAS, ICP-MS, ID-TIMS, XRF
Magnesium	ICP-AES, INAA
Manganese	FAAS, ICP-AES, ICP-MS, INAA
Mercury	CVAAS, ICP-MS INAA, RNAA
Nickel	ADSV GFAAS, ICP-AES, ICP-MS
Potassium	ICP-AES, INAA
Rubidium	INAA
Samarium	INAA
Scandium	INAA
Selenium	CSV, GFAAS, HGAAS, ICP-MS, INAA, RNAA
Silver	GFAAS, ID-ICP-MS, ICP-MS, INAA
Sodium	ICP-AES, INAA
Strontium	ICP-MS, HG-AAS, INAA
Sulfur	ICP-AES, INAA
Thallium	ICP-MS, ID-TIMS, INAA
Tin	GFAAS, ICP-AES, ICP-MS, ID-ICP-MS
Thorium	INAA
Uranium	INAA
Vanadium	INAA
Zinc	FAAS, ICP-AES, ICP-MS, ID-ICP-MS, ID-TIMS, XRF, INAA
Methods	
ADSV	Adsorptive stripping voltammetry
CSV	Cathodic stripping voltammetry
CVAAS	Cold vapor atomic absorption spectrometry
DPASV	Differential pulse anodic stripping voltammetry
ETAAS	Electrothermal atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
GFAAS	Graphite furnace atomic absorption spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ICP-AES	Inductively coupled plasma atomic emission spectrometry
ICP-MS	Inductively coupled plasma mass spectrometry
ID-ICP-MS	Isotope dilution inductively coupled plasma mass spectrometry
ID-TIMS	Isotope dilution thermal ionization mass spectrometry
INAA	Instrumental neutron activation analysis
RNAA	Radiochemical neutron activation analysis
XRF	X-ray fluorescence spectrometry

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**Certificate Revision History:** 12 September 2008 (Updated existing certified and reference values for PAHs, PCBs, pesticides and methylmercury; added certified and reference values for PBDEs and for additional PAHs and PCBs; extension of certification period); 01 May 2000 (Original certificate date).

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