



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 1947

#### Lake Michigan Fish Tissue

Standard Reference Material (SRM) 1947 is a frozen fish tissue homogenate, which was prepared from fish collected from Lake Michigan, and is intended primarily for use in evaluating analytical methods for the determination of selected trace elements, methylmercury, total mercury, polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, and polybrominated diphenyl ether (PBDE) congeners, proximates, caloric content and fatty acids in fish tissue and similar matrices. All of the constituents for which certified, reference, and information values are provided are naturally present in the fish tissue homogenate. A unit of SRM 1947 consists of five bottles, each containing approximately 8 g (wet basis) of frozen tissue homogenate.

**Certified Concentration Values:** Certified concentration values are provided in Table 1 for selected trace elements including total mercury, and methylmercury. Certified concentration values are provided in Tables 2 through 4 for 32 PCB congeners, 15 chlorinated pesticides, and 7 PBDE congeners, respectively. The certified values for trace elements, PCBs, and chlorinated pesticides are based on results obtained from two or more independent analytical techniques. 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 [1].

**Reference Concentration Values:** Reference concentration values are provided in Table 5 for an additional 13 PCB congeners, 2 chlorinated pesticides, and 2 PBDE congeners. Reference concentration values are provided for proximates, caloric content, and selected fatty acids in Tables 6 and 7. Reference values are noncertified values which represent the best estimate of the true values based on available data; however, the values do not meet the NIST criteria for certification [1] and are provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

**Expiration of Certification:** The certification of SRM 1947 is valid until **31 May 2015**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this report. The certification is nullified if the SRM is damaged, contaminated, or modified.

**Maintenance of SRM Value Assignment:** 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.

Coordination of the preparation and technical measurements leading to the certification of this SRM was performed by S.J. Christopher, G.C. Turk, M.M. Schantz, and S.A. Wise of the NIST Analytical Chemistry Division.

Statistical analysis was provided by S.D. Leigh and J. H. Yen of the NIST Statistical Engineering Division.

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Certificate Issue Date: 30 August 2007

Analytical measurements at NIST were performed by S.J. Christopher, R.D. Day, J.M. Keller, J.R. Kucklick, S.E. Long, E.A. Mackey, W.C. Davis, B.J. Porter, D.L. Poster, M.M. Schantz, and H.M. Stapleton of the NIST Analytical Chemistry Division. Additional PBDE measurements were provided by R.A. Hites and Y.L. Zhu of Indiana University (Bloomington, IN). Measurements from the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment were coordinated by M.M. Schantz of the NIST Analytical Chemistry Division (see Appendix A for participating laboratories). Measurements by the Food Products Association (FPA) Food Industry Analytical Chemists Subcommittee were coordinated by K.E. Sharpless of the NIST Analytical Chemistry Division and H.B. Chin and D.W. Howell of the FPA (Washington, DC) (see Appendix B for participating laboratories). Measurements from the 2001 NIST/NOAA Interlaboratory Comparison Exercise for Trace Elements in Marine Mammals were coordinated by S.J. Christopher of the NIST Analytical Chemistry Division (see Appendix C for participating laboratories). Analytical measurements for methylmercury were also performed at the University of Pau (Pau, France) by E. Krupp, D. Point, and O. Donard.

The fish used for SRM 1947 were collected with the assistance of the Michigan Department of Natural Resources (DNR) (J. Jonas) and the Michigan DNR survey vessel *Steelhead* (Captain J. Meggison, J. Ranville, and J. Harris). The coordination for the collection was performed by J.R. Kucklick and B.J. Porter of the NIST Analytical Chemistry Division. Field collection was performed by B. Flood and J. Stevens of the Michigan DNR, B.J. Porter of the NIST Analytical Chemistry Division, and M.P. Cronise and C.N. Fales of the NIST Measurement Services Division. Cryogenic homogenization of the fish tissue was performed by B.J. Porter, R.S. Pugh, and D.J. Struntz of the NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

## **NOTICE AND WARNING TO USERS**

**WARNING:** FOR LABORATORY USE ONLY. NOT FOR HUMAN CONSUMPTION.

## **STORAGE**

SRM 1947 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  $-80\text{ }^{\circ}\text{C}$  (or lower) since it was prepared and should be stored by the user at this temperature for the certified values to be valid within the stated uncertainties.

## **INSTRUCTIONS FOR USE**

This material is a frozen tissue homogenate. After extended storage at temperatures of  $-25\text{ }^{\circ}\text{C}$  or higher, 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 mass 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-coated spatulas, for transferring the powder. Normal biohazard safety precautions for the handling of biological tissues should be exercised. 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 through 4 to be valid within the stated uncertainties. The concentrations of constituents in SRM 1947 are reported on a wet-mass basis. The SRM tissue homogenate, as received, contains approximately 73 % moisture.

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

**Sample Collection:** SRM 1947 was prepared from fillets from adult lake trout (*Salvelinus namaycush*) collected in October 1997 at Fisherman's Island and Big Reef near Charlevoix, MI in northern Lake Michigan. The fillets were removed from the fish using stainless steel knives and placed in Teflon bags. The tissue was placed on wet ice and transported to NIST where it was stored in liquid nitrogen vapor freezers ( $-120\text{ }^{\circ}\text{C}$ ) until processed and bottled. A total of 79 kg of fillets was obtained. The frozen fillets were pulverized in batches of approximately 350 g using the cryogenic procedure described previously [3]. The pulverized fish tissue was then homogenized in an aluminum mixing drum in two batches of approximately 40 kg each [4]. The mixing drum was designed to fit inside a liquid nitrogen vapor freezer and to rotate in the freezer thereby mixing the frozen tissue powder. After mixing for 2 h, subsamples of approximately 8 g of fish tissue homogenate were aliquoted into pre-cooled glass bottles.

**Moisture Content:** The moisture content of the fish tissue homogenate was determined by measuring the mass loss from freeze drying. Twelve bottles (six from each batch) of SRM 1947 were selected according to a stratified randomization scheme for the drying study. The entire contents of each glass bottle were transferred to a Teflon bottle and dried for seven days at 1 Pa with a  $-20\text{ }^{\circ}\text{C}$  shelf temperature and a  $-50\text{ }^{\circ}\text{C}$  condenser temperature. Based on these studies, the mean moisture content of SRM 1947 is  $73.00\% \pm 0.15\%$  (mass fraction expressed as percent  $\pm$  expanded uncertainty with  $k = 2$ , approximately 95 % confidence). The concentration values are reported on a wet-mass (as-received) basis. If necessary, the results can be converted to a dry-mass basis by dividing by the conversion factor of 0.2700 (g dry mass per g wet mass).

**Trace Elements:** Trace element data are derived from three sources: (1) NIST measurements, (2) data from the 2001 NIST/NOAA Interlaboratory Comparison Exercise for Trace Elements in Marine Mammals, and (3) data obtained in 2002 from an interlaboratory comparison exercise conducted by the Food Products Association. The NIST measurements were collected using collision cell inductively coupled plasma mass spectrometry and the method of standard additions. A maximum likelihood solution algorithm was used to determine the consensus mean estimates and analytical uncertainties associated with the interlaboratory data, after rejection of outlier laboratories. This algorithm weights data based on inverse variance. Certified values for As, Cu, Fe, Mn, Rb, Se, and Zn were then assigned by combining the NIST values and interlaboratory consensus mean estimates, using an equal weighting scheme. Analytical uncertainties were calculated using the bound-on-bias method [5]. The certified value for Hg is derived from isotope dilution cold vapor inductively coupled plasma mass spectrometry measurements performed at NIST [6].

**Methylmercury:** The general approach for the assignment of a value for methylmercury was similar to that used in recent marine tissue SRMs [7,8]. The certified value for methylmercury is based on results of analyses of SRM 1947 at NIST and the University of Pau using four analytical techniques. The three NIST methods used for methylmercury measurements were based on (1) solid phase microextraction (SPME) with gas chromatography/mass spectrometry (GC/MS), (2) SPME with standard additions quantification and GC/inductively coupled plasma mass spectrometry (GC/ICPMS) [9], and (3) microwave extraction with speciated isotope dilution and GC/ICPMS [10]. The fourth method also utilized microwave extraction with speciated isotope dilution GC/ICPMS. For the SPME GC/MS analyses, approximately 1 g subsamples of SRM 1947 were spiked with an appropriately diluted solution of IRMM-670  $^{202}\text{Hg}$  enriched methylmercury isotopic Certified Reference Material (CRM) (Institute of Reference Materials and Measurements [IRMM], Geel, Belgium) followed by an acidic microwave digestion using 1 mol/L hydrochloric acid. Sodium tetraphenylborate was used for phenylation. After headspace SPME sampling, the SPME fiber was desorbed using a GC injection temperature of  $250\text{ }^{\circ}\text{C}$ . The GC analysis used a  $30\text{ m} \times 0.25\text{ mm}$  column with a 5 % (mole fraction) phenyl methylpolysiloxane phase ( $0.25\text{ }\mu\text{m}$  film thickness) (DB-5MS, J&W Scientific, Folsom, CA). For the SPME GC/ICPMS analyses, approximately 0.5 g subsamples of SRM 1947 were subjected to an acidic microwave digestion using 1 mol/L hydrochloric acid. Sodium tetraphenylborate was used for phenylation. After headspace SPME sampling, the SPME fiber was desorbed using a GC injection temperature of  $210\text{ }^{\circ}\text{C}$ . The GC analysis used a  $30\text{ m} \times 0.28\text{ mm}$  column with a 100 % dimethylpolysiloxane phase ( $0.50\text{ }\mu\text{m}$  film thickness) (MXT-1, Restek, Bellefonte, PA). For the speciated isotope dilution GC/ICPMS analyses, approximately 1.0 g to 2.0 g subsamples were spiked with an appropriately

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<sup>1</sup> Certain commercial equipment, instruments, or materials are identified to specify adequately 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 the best available for the purpose.

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 isoctane 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, J&W Scientific, Folsom, CA). SRM 1946 Lake Superior Fish Tissue was used as a control in each of the methods described.

**PCBs and Chlorinated Pesticides:** The general approach used for the value assignment of concentrations for PCBs and chlorinated pesticides in SRM 1947 was similar to that reported for the recent certification of SRM 1946 [11] and consisted of combining results from analyses at NIST using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. For SRM 1947 the approach consisted of Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture; cleanup/isolation using solid-phase extraction (SPE), size-exclusion chromatography (SEC), or normal-phase liquid chromatography (LC); and finally analysis by using gas chromatography with electron capture detection (GC-ECD) or gas chromatography with mass spectrometric detection (GC/MS) on three columns with different selectivity for the separation of PCBs and chlorinated pesticides.

Three sets of results were obtained by GC-ECD and are designated as GC-ECD (I), GC-ECD (IIA), and GC-ECD (IIB). For the GC-ECD (I) analyses, between 2 g and 3 g subsamples from each of six bottles of SRM 1947 were extracted using Soxhlet with DCM. SEC was used to remove the majority of the lipid material. The concentrated eluant was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and the less polar pesticides and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 μm film thickness) (DB-5, J&W Scientific, Folsom, CA). For GC-ECD (IIA) and GC-ECD (IIB), 3.5 g subsamples from each of six bottles were extracted using PFE with DCM [12]. The SEC and normal-phase LC cleanup steps were the same as those used for GC-ECD (I). GC-ECD (IIA) analyses were performed on a 0.25 mm × 60 m fused silica capillary column with nonpolar proprietary phase (0.25 μm film thickness) (DB-XLB, J&W Scientific), and GC-ECD (IIB) analyses were performed on a 5 % phenyl methylpolysiloxane phase as described above. For both GC-ECD analyses, two PCB congeners that are not significantly present in the fish extract [PCB 30 and PCB 198 for GC-ECD(I) or PCB 103 and PCB 198 for GC-ECD (II)], and 4,4'-DDT-*d*<sub>8</sub>, 4,4'-DDE-*d*<sub>8</sub>, and 4,4'-DDD-*d*<sub>8</sub> were added to the fish tissue prior to extraction for use as internal standards for quantification purposes.

Two sets of results were obtained by GC/MS. The samples used for GC/MS (I) analyses were the same extracts as analyzed for GC-ECD (I). The GC/MS (I) analyses were performed using a DB-XLB column as described above and MS detection. For GC/MS (II) analyses, two subsamples of between 1 g and 2 g were used from three bottles of SRM 1947. The six samples were extracted using PFE with hexane:acetone (1:1, volume fraction). The concentrated extract was subjected to cleanup on a silica solid phase extraction (SPE) cartridge with 10 % DCM in hexane. Following concentration, a second silica SPE cartridge was used for additional sample cleanup. The GC/MS (II) analyses were performed using a 50 % (mole fraction) phenyl methylpolysiloxane phase (0.25 μm film thickness) (DB-17MS, J&W Scientific, Folsom, CA). For the GC/MS (II) analyses, PCB 103, PCB 198, and <sup>13</sup>C-labeled 4,4'-DDT, lindane, PCB 28, PCB 101, PCB 118, PCB 138, PCB 153, and PCB 169 were added to the fish tissue prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1947 was used in an interlaboratory comparison exercise in 2002 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [13]. Results from 28 laboratories that participated in this exercise (see Appendix A) were used as the sixth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1947. The laboratories participating in this exercise used the analytical procedures routinely used in their laboratories to measure these analytes.

**Polybrominated Diphenyl Ethers:** Value assignment of concentrations for PBDE congeners was based on four sets of data (three sets from NIST and one set from a collaborating laboratory) 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 two of the NIST data sets, 1 g to 2 g subsamples of tissue from each of five bottles were extracted using PFE with DCM. The concentrated extract was subjected to SEC to remove the majority of the lipids, followed by an additional cleanup step employing silica SPE cartridges. The extracts were analyzed by using both GC/EI-MS and GC/NCI-MS on a 0.25 mm × 15 m fused silica capillary column with a 5 % (mole fraction) phenyl methylpolysiloxane phase (0.25 μm film thickness) (DB-5). For both methods <sup>13</sup>C-labeled 4,4'-dibromodiphenyl ether (BDE 15) and <sup>13</sup>C-labeled 2,2',3,4,5-pentachlorodiphenyl ether (CDE 86) were added to the tissue sample prior to extraction for use as internal standards for quantification purposes.

For the third NIST data set, 3 g to 4 g subsamples of tissue from each of six 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). <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 PBDEs.

For the measurements from the collaborating laboratory (Indiana University, Bloomington, IN), four subsamples of 8 g 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). Details of the analyses by the collaborating laboratory are presented by Zhu and Hites [14].

**FPA Interlaboratory Comparison Exercise:** Results for proximates, extractable fat, fatty acids, and selected trace elements were obtained from an interlaboratory comparison exercise organized in 2002 by the Food Products Association (FPA) Food Industry Analytical Chemists Subcommittee (FIACS; 10 participating laboratories, listed in Appendix B). The laboratories listed in Appendix B were asked to use AOAC methods or their equivalent, to make single measurements from each of two bottles, and to report the analytical method that was used.

**Extractable Fat Determination:** The reference value for extractable fat was determined from the combination of results from analyses performed at NIST and the results from the interlaboratory comparison exercise. At NIST, six samples were extracted with hexane:acetone (1;1, volume fraction) using PFE. The extracts were evaporatively concentrated to approximately 20 mL (known mass) and an aliquot of 90 μL was placed on an aluminum pan. The extract on the pan was air dried, and the mass of the dried extract determined. For the interlaboratory study, laboratories used their typical extraction methods and then determined the extractable fat by drying the extract and determining the mass of the remaining residue.

**Proximates:** Results for proximates (solids, ash, protein, and fat) were obtained from the FPA interlaboratory comparison exercise described above. The value for solids was a combination of the mean of the FPA measurements and the NIST moisture determination measurements described above.

Table 1. Certified Concentrations for Selected Elements and Methylmercury in SRM 1947

Element	Mass Fraction (wet-mass basis) <sup>(a)</sup> (mg/kg)
As	0.732 ± 0.039
Cu	0.411 ± 0.029
Fe	3.79 ± 0.42
Hg	0.254 ± 0.005 <sup>(b)</sup>
Mn	0.076 ± 0.004
Rb	4.51 ± 0.09
Se	0.475 ± 0.084
Zn	2.66 ± 0.08
Methylmercury	0.233 ± 0.010 <sup>(c)</sup>

<sup>(a)</sup> Unless otherwise noted, the certified mass fraction is an unweighted mean of NIST and round-robin consensus mean results. 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 [5] incorporating inter-method bias with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [2].

<sup>(b)</sup> The certified mass fraction for mercury is based solely on isotope dilution cold vapor inductively coupled plasma mass spectrometry measurements performed at NIST.

<sup>(c)</sup> Certified mass fraction for methylmercury is reported as mg of mercury/kg. The certified value is an unweighted mean of the results from four analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance [5] with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [2].

Table 2. Certified Concentrations for Selected PCB Congeners in SRM 1947

PCB Congener <sup>(a)</sup>	Mass Fraction (wet-mass basis) <sup>(b)</sup> (µg/kg)
PCB 28 (2,4,4'-Trichlorobiphenyl) <sup>(d,e,f,g,h)</sup>	14.1 ± 1.0
PCB 31 (2,4',5-Trichlorobiphenyl) <sup>(d,e,f,g,h)</sup>	10.4 ± 1.4
PCB 44 (2,2',3,5'-Tetrachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	20.4 ± 1.7
PCB 49 (2,2',4,5'-Tetrachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	27.3 ± 3.8
PCB 52 (2,2',5,5'-Tetrachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	36.4 ± 4.3
PCB 63 (2,3,4',5-Tetrachlorobiphenyl) <sup>(d,e,f,g)</sup>	4.75 ± 0.60
PCB 66 (2,3',4,4'-Tetrachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	69.4 ± 5.3
PCB 74 (2,4,4',5-Tetrachlorobiphenyl) <sup>(c,d,g,h)</sup>	33.7 ± 3.1
PCB 87 (2,2',3,4,5'-Pentachlorobiphenyl) <sup>(c,e,g,h)</sup>	27.9 ± 1.5
PCB 99 (2,2',4,4',5-Pentachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	78.0 ± 6.0
PCB 101 (2,2',4,5,5'-Pentachlorobiphenyl) <sup>(c,e,f,g)</sup>	90.8 ± 0.3
PCB 105 (2,3,3',4,4'-Pentachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	50.3 ± 3.7
PCB 107 (2,3,3',4',5-Pentachlorobiphenyl) <sup>(d,e,f,g)</sup>	17.1 ± 1.2
PCB 110 (2,3,3',4',6-Pentachlorobiphenyl) <sup>(d,f,g)</sup>	94.6 ± 4.3
PCB 118 (2,3',4,4',5-Pentachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	112 ± 6
PCB 128 (2,2',3,3',4,4'-Hexachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	31.6 ± 2.1
PCB 132 (2,2',3,3',4,6'-Hexachlorobiphenyl) <sup>(c,d,e,g)</sup>	20.8 ± 2.1
PCB 138 (2,2',3,4,4',5'-Hexachlorobiphenyl) <sup>(d,f,g,h)</sup>	162.0 ± 6.9
PCB 146 (2,2',3,4',5,5'-Hexachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	40.5 ± 2.0
PCB 149 (2,2',3,4',5',6-Hexachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	67.1 ± 3.7
PCB 153 (2,2',4,4',5,5'-Hexachlorobiphenyl) <sup>(c,d,e,g,h)</sup>	201 ± 3
PCB 156 (2,3,3',4,4',5-Hexachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	13.3 ± 0.9
PCB 158 (2,3,3',4,4',6-Hexachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	11.3 ± 0.9
PCB 170 (2,2',3,3',4,4',5-Heptachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	29.2 ± 2.4
PCB 174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	18.6 ± 1.7
PCB 180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	80.8 ± 5.0
PCB 183 (2,2',3,4,4',5',6-Heptachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	23.3 ± 1.9
PCB 187 (2,2',3,4',5,5',6-Heptachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	54.8 ± 2.6
PCB 193 (2,3',3,4',5,5',6-Heptachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	6.04 ± 0.23
PCB 194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	13.2 ± 0.9
PCB 195 (2,2',3,3',4,4',5,6-Octachlorobiphenyl) <sup>(d,e,f,g,h)</sup>	4.95 ± 0.77
PCB 206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) <sup>(c,d,e,f,g,h)</sup>	6.24 ± 0.88

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] to conform with IUPAC rules; for the specific congeners listed in this table, only PCB 107 and PCB 201 are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 107 is listed as PCB 108 and the IUPAC PCB 201 is listed as PCB 200.

<sup>(b)</sup> The certified value is a weighted mean of the results from four to six 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 [17] incorporating inter-method bias with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [2].

<sup>(c)</sup> GC-ECD (I) on 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC/MS (I).

<sup>(d)</sup> GC-ECD (IIA) on a proprietary nonpolar phase after PFE with DCM.

<sup>(e)</sup> GC-ECD (IIB) on 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

<sup>(f)</sup> GC/MS (I) on a proprietary nonpolar phase after Soxhlet extraction with DCM.

<sup>(g)</sup> GC/MS (II) on a 50 % phenyl methylpolysiloxane phase after PFE with hexane/acetone mixture.

<sup>(h)</sup> Results from up to 28 laboratories participating in an interlaboratory comparison exercise.

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1947

Pesticide	Mass Fraction (wet-mass basis) <sup>(a)</sup> ( $\mu\text{g}/\text{kg}$ )	
Hexachlorobenzene <sup>(b,c,d,e,f,g)</sup>	7.48	$\pm$ 0.66
$\alpha$ -HCH <sup>(b,c,d,g)</sup>	1.06	$\pm$ 0.12
Heptachlor epoxide <sup>(b,c,d,e,f,g)</sup>	13.4	$\pm$ 0.8
Oxychlorodane <sup>(b,c,e,f,g)</sup>	23.6	$\pm$ 1.5
<i>trans</i> -Chlordane <sup>(b,c,d,e,f,g)</sup>	12.8	$\pm$ 1.2
<i>cis</i> -Nonachlor <sup>(b,c,d,e,f,g)</sup>	54.1	$\pm$ 7.3
<i>trans</i> -Nonachlor <sup>(b,c,d,e,f,g)</sup>	127	$\pm$ 6
Dieldrin <sup>(b,c,d,e,f,g)</sup>	80.8	$\pm$ 3.8
Mirex <sup>(b,c,e,f,g)</sup>	5.09	$\pm$ 0.73
2,4'-DDE <sup>(b,c,e,f,g)</sup>	3.39	$\pm$ 0.28
4,4'-DDE <sup>(c,d,e,f,g)</sup>	720	$\pm$ 43
2,4'-DDD <sup>(c,d,e,f)</sup>	3.31	$\pm$ 0.16
4,4'-DDD <sup>(b,c,d,e,f,g)</sup>	45.9	$\pm$ 3.6
2,4'-DDT <sup>(c,e,f,g)</sup>	15.7	$\pm$ 0.89
4,4'-DDT <sup>(b,c,d,e,f,g)</sup>	59.5	$\pm$ 6.7

<sup>(a)</sup> The certified value is a weighted mean of the results from four to six 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 [17] incorporating inter-method bias with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [2].

<sup>(b)</sup> GC-ECD (I) on 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC/MS (I).

<sup>(c)</sup> GC-ECD (IIA) on a proprietary nonpolar phase after PFE with DCM.

<sup>(d)</sup> GC-ECD (IIB) on 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

<sup>(e)</sup> GC/MS (I) on a proprietary nonpolar phase after Soxhlet extraction with DCM.

<sup>(f)</sup> GC/MS (II) on a 50 % phenyl methylpolysiloxane phase after PFE with hexane/acetone mixture.

<sup>(g)</sup> Results from up to 28 laboratories participating in an interlaboratory comparison exercise.

Table 4. Certified Concentrations for Selected Polybrominated Diphenyl Ether (PBDE) Congeners in SRM 1947

PBDE Congener <sup>(a)</sup>			Mass Fraction (wet-mass basis) <sup>(b)</sup> ( $\mu\text{g}/\text{kg}$ )	
BDE	47	(2,2',4,4'-Tetrabromodiphenylether) <sup>(c,d,e,f)</sup>	73.3	$\pm$ 2.9
BDE	49	(2,2',4,5'-Tetrabromodiphenylether) <sup>(c,d,e,f)</sup>	4.01	$\pm$ 0.10
BDE	66	(2,3',4,4'-Tetrabromodiphenylether) <sup>(c,d,e,f)</sup>	1.85	$\pm$ 0.13
BDE	99	(2,2',4,4',5-Pentabromodiphenylether) <sup>(c,d,e,f)</sup>	19.2	$\pm$ 0.8
BDE	100	(2,2',4,4',6-Pentabromodiphenylether) <sup>(c,d,e,f)</sup>	17.1	$\pm$ 0.6
BDE	153	(2,2',4,4',5,5'-Hexabromodiphenylether) <sup>(c,d,e,f)</sup>	3.83	$\pm$ 0.04
BDE	154	(2,2',4,4',5,6'-Hexabromodiphenylether) <sup>(d,e,f)</sup>	6.88	$\pm$ 0.52

<sup>(a)</sup> PBDE congeners are numbered according to IUPAC rules.

<sup>(b)</sup> The certified value is a weighted mean of the results from three or four 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 [17] incorporating inter-method bias with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [2].

<sup>(c)</sup> GC/NCI-MS on a 15 m 5 % phenyl methylpolysiloxane phase.

<sup>(d)</sup> GC/EI-MS (I) on a 15 m 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC/NCI-MS.

<sup>(e)</sup> GC/EI-MS (II) on a 60 m 5 % phenyl methylpolysiloxane phase.

<sup>(f)</sup> GC/NCI-MS results reported by Zhu and Hites [14].

Table 5. Reference Concentrations for Selected PCB Congeners, PBDE Congeners, and Pesticides in SRM 1947

PCB Congeners <sup>(a)</sup>		Mass Fraction (wet-mass basis) ( $\mu\text{g}/\text{kg}$ )
PCB 18	(2,2',5-Trichlorobiphenyl) <sup>(d,e,f,g,h,i)</sup>	2.72 $\pm$ 0.95 <sup>(b)</sup>
PCB 45	(2,2',3,6-Tetrachlorobiphenyl) <sup>(e,f,g)</sup>	1.76 $\pm$ 0.76 <sup>(c)</sup>
PCB 56	(2,3,3',4'-Tetrachlorobiphenyl) <sup>(g,h)</sup>	12.8 $\pm$ 0.4 <sup>(c)</sup>
PCB 70	(2,3',4',5-Tetrachlorobiphenyl) <sup>(d,e,g,h)</sup>	50 $\pm$ 12 <sup>(b)</sup>
PCB 82	(2,2',3,3',4-Pentachlorobiphenyl) <sup>(c,d,f,g)</sup>	3.87 $\pm$ 0.67 <sup>(b)</sup>
PCB 92	(2,2',3,5,5'-Pentachlorobiphenyl) <sup>(d,f,g,h)</sup>	32.6 $\pm$ 5.2 <sup>(b)</sup>
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl) <sup>(f,g,h,i)</sup>	33.6 $\pm$ 5.1 <sup>(b)</sup>
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl) <sup>(d,e,f,g)</sup>	23.3 $\pm$ 5.3 <sup>(b)</sup>
PCB 154	(2,2',4,4',5,6'-Hexachlorobiphenyl) <sup>(f,g)</sup>	3.51 $\pm$ 0.46 <sup>(c)</sup>
PCB 157	(2,3,3',4,4',5'-Hexachlorobiphenyl) <sup>(e,g,h)</sup>	4.08 $\pm$ 0.77 <sup>(c)</sup>
PCB 163	(2,3,3',4',5,6-Hexachlorobiphenyl) <sup>(e,g,h)</sup>	40.0 $\pm$ 5.2 <sup>(c)</sup>
PCB 201	(2,2',3,3',4,5,5',6'-Octachlorobiphenyl) <sup>(g,h)</sup>	3.59 $\pm$ 0.43 <sup>(c)</sup>
PCB 209	(Decachlorobiphenyl) <sup>(d,e,f,g,h,i)</sup>	2.45 $\pm$ 0.68 <sup>(b)</sup>
PBDE Congeners <sup>(i)</sup>		
BDE 28	(2,4,4'-Tribromodiphenylether) <sup>(k,l,m,n)</sup>	2.26 $\pm$ 0.46 <sup>(b)</sup>
33	(2',3,4-Tribromodiphenylether)	
BDE 155	(2,2',4,4',6,6'-Hexabromodiphenylether) <sup>(k,m)</sup>	0.45 $\pm$ 0.10 <sup>(c)</sup>
Pesticides		
$\gamma$ -HCH <sup>(d,e,f,i)</sup>		0.355 $\pm$ 0.095 <sup>(b)</sup>
<i>cis</i> -Chlordane ( $\alpha$ -Chlordane) <sup>(d,g,h,i)</sup>		49.0 $\pm$ 5.5 <sup>(b)</sup>

<sup>(a)</sup> PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] to conform with IUPAC rules; for the specific congeners listed in this table, only PCB 107 and PCB 201 are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 107 is listed as PCB 108 and the IUPAC PCB 201 is listed as PCB 200.

<sup>(b)</sup> The reference value is a weighted mean of the results from four to six 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 [17] incorporating inter-method bias with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [2].

<sup>(c)</sup> The reference value is an unweighted mean of the results from two to 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 [5] with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurement* [2].

<sup>(d)</sup> GC-ECD (I) on 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC/MS (I).

<sup>(e)</sup> GC-ECD (IIA) on a proprietary nonpolar phase after PFE with DCM.

<sup>(f)</sup> GC-ECD (IIB) on 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC-ECD (IIA).

<sup>(g)</sup> GC/MS (I) on a proprietary nonpolar phase after Soxhlet extraction with DCM.

<sup>(h)</sup> GC/MS (II) on a 50 % phenyl methylpolysiloxane phase after PFE with hexane/acetone mixture.

<sup>(i)</sup> Results from up to 28 laboratories participating in an interlaboratory comparison exercise.

<sup>(j)</sup> BDE congeners are numbered according to IUPAC rules.

<sup>(k)</sup> GC/NCI-MS on a 15 m 5 % phenyl methylpolysiloxane phase.

<sup>(l)</sup> GC/EI-MS (I) on a 15 m 5 % phenyl methylpolysiloxane phase; same extracts analyzed as GC/NCI-MS.

<sup>(m)</sup> GC/EI-MS (II) on a 60 m 5 % phenyl methylpolysiloxane phase.

<sup>(n)</sup> GC/NCI-MS results reported by Zhu and Hites [14].

Table 6. Reference Concentration Values for Proximates and Caloric Content in SRM 1947

	Mass Fraction (wet-mass basis) <sup>(a)</sup> (%)		
Solids <sup>(b)</sup>	27.1	±	0.2 <sup>(b)</sup>
Ash	1.07	±	0.07
Protein	17.0	±	0.5
Calories <sup>(c)</sup>	(152	±	6) kcal/100 g
Fat (Extractable)	10.4	±	0.5
Carbohydrate <sup>(d)</sup>	0.9 <sup>(d)</sup>		

- <sup>(a)</sup> The reference value is a weighted mean of the results provided by the laboratories in Appendix B. The uncertainty listed with each value is an expanded uncertainty about the mean with coverage factor determined from the Student's *t*-distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence, and calculated to include the combined effect of between-laboratory and within-laboratory components of uncertainty, following the *ISO Guide to the Expression of Uncertainty in Measurements* [2].
- <sup>(b)</sup> The reference value for solids is the unweighted mean of the mean of the average of results provide by laboratories listed in Appendix B and the mean of the NIST measurements. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor determined from the Student's *t* distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence, and calculated by combining a between-method variance [5] with a pooled, within-method variance following the *ISO Guide to the Expression of Uncertainty in Measurements* [2].
- <sup>(c)</sup> The value for caloric content is the mean of individual caloric calculations from the laboratories listed in Appendix B. If the proximate values above are used for calculation, with caloric equivalents of 9, 4, and 4 for fat, protein, and carbohydrate, respectively, the mean caloric content is 165 kcal/100 g.
- <sup>(d)</sup> The concentration for carbohydrates is provided as an information value only; information values are typically provided with no uncertainty because of the lack of sufficient information to assess adequately the uncertainty associated with the value. It may be assumed that the uncertainty is relatively large.

Table 7. Reference Concentrations for Fat and Selected Fatty Acids in SRM 1947

Fat	Mass Fraction (wet-mass basis) <sup>(a)</sup> (%)
Fat (Sum of Fatty Acids) <sup>(b)</sup>	8.50 ± 0.54
Saturated Fat	1.75 ± 0.22
Monosaturated Fat	3.55 ± 0.22
Polyunsaturated Fat	2.84 ± 0.44

  

Fatty Acid	Mass Fraction (wet-mass basis) <sup>(a)</sup> (as the triglyceride) (%)
Tetradecanoic Acid (C14:0) (Myristic Acid)	0.312 ± 0.027
Pentadecanoic Acid (C15:0)	0.025 ± 0.004
Hexadecanoic Acid (C16:0) (Palmitic Acid)	1.14 ± 0.08
(Z)-9-Hexadecenoic Acid (C16:1) (Palmitoleic Acid)	0.946 ± 0.050
Heptadecanoic Acid (C17:0) (Margaric Acid)	0.024 ± 0.007
Octadecanoic Acid (C18:0) (Stearic Acid)	0.230 ± 0.019
(Z)-9-Octadecenoic Acid (C18:1) (Oleic Acid)	2.34 ± 0.15
(Z,Z)-9,12-Octadecadienoic Acid (C18:2) (Linoleic Acid)	0.375 ± 0.014
(Z,Z,Z)-9,12,15-Octadecatrenoic Acid (C18:3) (Linolenic Acid)	0.287 ± 0.026
(Z,Z,Z,Z)-6,9,12,15-Octadecatetraenoic Acid (C18:4) (Stearidonic Acid)	0.120 ± 0.025
(Z)-9-Eicosenoic Acid (C20:1) (Gadoleic Acid)	0.138 ± 0.028
(Z,Z)-11,14-Eicosadienoic Acid (C20:2)	0.100 ± 0.015
(Z,Z,Z,Z)-5,8,11,14-Eicosatetraenoic Acid (C20:4) (Arachidonic Acid)	0.247 ± 0.035
(Z,Z,Z,Z,Z)-5,8,11,14,17-Eicosapentaenoic Acid (C20:5) (EPA)	0.395 ± 0.034
(Z,Z,Z,Z,Z)-7,10,13,16,19-Docosapentaenoic Acid (C22:5) (DPA)	0.320 ± 0.017
(Z,Z,Z,Z,Z,Z)-4,7,10,13,16,19-Docosahexaenoic Acid (C22:6) (DHA)	0.874 ± 0.059

<sup>(a)</sup> The reference value is a weighted mean of the results provided by five to ten laboratories in Appendix B. The uncertainty listed with each value is an expanded uncertainty about the mean with coverage factor determined from the Student's *t*-distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence, and calculated to include the combined effect of between-laboratory and within-laboratory components of uncertainty, following the *ISO Guide to the Expression of Uncertainty in Measurements* [2].

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## APPENDIX A

The laboratories listed below performed measurements that contributed to the value assignment for PCBs and pesticides in SRM 1947.

Academy of Natural Sciences; Philadelphia, PA, USA  
ALS Environmental; Vancouver, BC, Canada  
Arthur D. Little, Inc.; Cambridge, MA, USA  
Axys Analytical Services; Sidney, BC, Canada  
B & B Laboratories; College Station, TX, USA  
Battelle Columbus; Columbus, OH, USA  
Battelle Ocean Sciences; Duxbury, MA, USA  
Bedford Institute of Oceanography; Dartmouth, NS, Canada  
Chesapeake Biological Laboratory; Solomons, MD, USA  
City of Los Angeles, Environmental Monitoring Division; Playa del Rey, CA, USA  
East Bay Municipal Utility District; Oakland, CA, USA  
EnChem, Inc.; Madison, WI, USA  
Environment Canada, Environmental Sciences Centre; Moncton, New Brunswick, Canada  
King County Environmental Laboratory; Seattle, WA, USA  
Manchester Environmental Laboratory; Port Orchard, WA, USA  
Mississippi State Chemical Laboratory; Mississippi State, MS, USA  
Murray State University; Murray, KY, USA  
National Oceanic and Atmospheric Administration, National Marine Fisheries Service (NOAA/NMFS), Center for Coastal Environmental Health and Biomolecular Research (CCEHBR); Charleston, SC, USA  
NOAA/NMFS, Sandy Hook Marine Laboratory; Highlands, NJ, USA  
NOAA/NMFS, Northwest Fisheries Science Center; Seattle, WA, USA  
Orange County Sanitation District; Fountain Valley, CA, USA  
Philip Analytical Services; Burlington, Ontario, Canada  
STL Knoxville; Knoxville, TN, USA  
STL Sacramento; Sacramento, CA, USA  
Texas A & M University, Geochemical and Environmental Research Group (GERG); College Station, TX, USA  
U.S. Geological Survey, National Water Quality Laboratory; Denver, CO, USA  
Woods Hole Group Environmental Laboratory; Raynham, MA, USA

## APPENDIX B

The laboratories listed below performed measurements that contributed to the value assignment for proximates, caloric content, extractable fat, fatty acids, and trace elements in SRM 1947.

Campbell Soup; Camden, NJ, USA  
Covance Laboratories; Madison, WI, USA  
Eurofins; Memphis, TN, USA  
General Mills, Inc.; Golden Valley, MN, USA  
Hormel Foods Corporation; Austin, MN, USA  
Kraft Foods Inc.; Hanover, NJ, USA  
Kraft Foods Inc.; Glenview, IL, USA  
Krueger Food Laboratories, Inc.; Cambridge, MA, USA  
Nestlé USA; Dublin, OH, USA  
Novartis Nutrition Technical Center; St. Louis Park, MN, USA

## APPENDIX C

The laboratories listed below performed measurements that contributed to the value assignment for trace elements in SRM 1947.

NOAA/NMFS, Northwest Fisheries Science Center; Seattle, WA, USA  
USDA Beltsville Human Nutrition Center; Beltsville, MD, USA  
Australian Nuclear Science and Technology Organization; Sydney, Australia  
Centre for Environment, Fisheries and Aquaculture Science; Burnham-on-Crouch, Essex, UK  
Curtin University Center for Excellence in Mass Spectrometry; Perth, Australia  
University of Connecticut Environmental Research Institute; Storrs, CT, USA  
Geological Survey of Canada; Ottawa, Canada  
Hill Laboratories; Hamilton, New Zealand  
University of Alaska Fairbanks, Institute of Arctic Biology; Fairbanks, Alaska, USA  
Karl-Franzens Universität Graz Institute of Chemistry; Graz, Austria  
Midwest Research Institute (Florida Division); Palm Bay, FL, USA  
Politechnika Poznanska; Poznań, Poland  
PSC Analytical; Mississauga, Canada  
R. J. Reynolds Tobacco Company; Winston-Salem, NC, USA  
Research Triangle Institute; Chapel Hill, NC, USA  
Savannah River Ecology Laboratory; Aiken, SC, USA  
Texas A&M University Trace Element Research Laboratory; College Station, TX, USA  
Texas A&M University Department of Veterinary Anatomy and Public Health; College Station, TX, USA  
Ultra-Traces Analyses Aquitaine, Laboratoire de Chimie Analytique Bio-Inorganique et Environnement;  
University of Pau, Pau, France  
University of Massachusetts Department of Chemistry; Amherst, MA, USA  
University of Massachusetts University Research Institute for Analytical Chemistry; Amherst, MA, USA  
Universidad de Coruña; Coruña, Spain  
University of Iowa Hygienic Laboratory; Des Moines, IA, USA  
University of Maine; Orono, ME, USA  
University of Nevada Las Vegas; Las Vegas, NV, USA