National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1945

Organics in Whale Blubber

This Standard Reference Material (SRM) is a frozen whale blubber homogenate intended for use in evaluating analytical methods for the determination of selected polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, polybrominated diphenyl ether (PBDE) congeners, and toxaphene congeners [1]. SRM 1945 has been reanalyzed, and the current Certificate of Analysis has values assigned for additional PCB congeners and pesticides, as well as values assigned for selected PBDE and toxaphene congeners. A unit of SRM 1945 consists of two screw-capped glass bottles, each containing approximately 15 g of frozen whale blubber homogenate.

Certified Mass Fraction Values: The measured is the total mass fraction for each PCB congeners, chlorinated pesticides, and PBDE congeners provided in Tables 1, 2, and 3, respectively. Metrological traceability is the SI derived unit for mass fraction (expressed as micrograms per kilogram on a wet basis). 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 taken into account [2]. The certified values for the PCB congeners, chlorinated pesticides, and PBDE congeners are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques along with results from an interlaboratory comparison study.

Reference Mass Fraction Values: The measured is the total mass fraction based on the indicated method or methods for additional PCB congeners, a chlorinated pesticide, additional PBDE congeners, and 2 methoxylated-PBDEs (MeO-PBDEs) in Table 4 and for 3 toxaphene congeners and total toxaphene in Table 5. Metrological traceability is the SI derived unit for mass fraction (expressed as micrograms per kilogram on a wet basis). Reference values are noncertified values that represent a best estimate of the true value; however, the values do not meet the NIST criteria for certification [2] 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.

Expiration of Certification: The certification of **SRM 1945** is valid, within the measurement uncertainty specified, until **31 July 2026**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Storage and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise 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. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements leading to certification of SRM 1945 were performed by M.M. Schantz and J.R. Kucklick of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by J.M. Keller, J.R. Kucklick, D.L. Poster, H.M. Stapleton, M.M. Schantz, and J.E. Yordy of the NIST Chemical Sciences Division. Results were also used from 26 laboratories (see Appendix A) that participated in an interlaboratory comparison exercise coordinated by NIST.

Evaluation of the data was provided by S.D. Leigh of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Collection and preparation of the SRM were performed by B.J. Porter and S.A. Wise of the NIST Chemical Sciences Division and by M.P. Cronise, C.N. Fales, J.T. Fort, G.V. Proulx, and T.P. Shuggars of the NIST Office of Reference Materials.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

NOTICE AND WARNING TO USERS

WARNING: FOR RESEARCH USE; NOT FOR HUMAN CONSUMPTION.

INSTRUCTIONS FOR STORAGE AND USE

Storage: SRM 1945 is provided as a frozen tissue homogenate in glass bottles. The tissue homogenate should not be allowed to thaw prior to subsampling for analysis. This material has been stored at NIST at –80 °C (or lower) since it was prepared. SRM 1945 should be stored at or less than –80 °C , if possible, since the validity of the certified values at higher temperatures is unknown.

Use: This material is a frozen tissue homogenate; after extended storage at temperatures of –25 °C or higher, or if allowed to warm, it 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 on the containers by rapid handling and wiping of the containers prior to weighing.) Transfer subsamples to another container for weighing, using 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.

Subsamples of this SRM for analysis should be withdrawn from bottles immediately after opening and used without delay for the certified values listed in Tables 1, 2, and 3 to be valid within the stated uncertainties. The bottles should be tightly closed immediately after removal of the subsamples and the remaining material may be frozen for use in later analyses. The concentrations of constituents in SRM 1945 are reported on a wet-mass basis.

PREPARATION AND ANALYSIS[1]

SRM Preparation: The whale blubber used to prepare this SRM was collected from an adult female pilot whale which was stranded on Cape Cod, MA in September 1991. The blubber tissue was placed in Teflon bags, frozen on dry ice, shipped to NIST, and placed in a liquid nitrogen freezer (–150 °C). At NIST, any remaining skin was removed using a titanium knife, and the blubber was again placed in Teflon bags and immediately returned to a liquid nitrogen freezer. Approximately 15 kg of whale blubber were prepared for use as the SRM. The frozen whale blubber was pulverized in batches of approximately 150 g each, using a cryogenic grinding procedure described previously [3]. The total 15 kg of pulverized material was then combined and thoroughly mixed with a Teflon paddle. Subsamples (10 g to15 g) of the whale blubber homogenate were aliquoted into pre-cooled glass bottles and stored at –80 °C.

ANALYTICAL METHODS USED FOR THE ANALYTES OF INTEREST

Up to nine sets of data were used for determination of the analytes of interest in SRM 1945. One method used gas chromatography with electron capture detection (GC-ECD) on dual columns described below; six methods used GC with mass spectrometry (GC/MS) on three columns of differing polarity described below; and one data set was the combination of consensus means from four interlaboratory comparison studies coordinated by NIST as described below. For all of the NIST methods described below (GC-ECD and GC/MS), multi-point calibration response curves for the PCB congeners, chlorinated pesticides, and PBDE congeners relative to the internal standards were determined by processing gravimetrically-diluted solutions of SRM 2261 *Chlorinated Pesticides in Hexane (Nominal Mass Concentration 2 µg/mL)*, SRM 2262 *Chlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane (Nominal Concentration 2 µg/mL)*, SRM 2274 *PCB Congener Solution-II in Isooctane*, and SRM 2275 *Chlorinated Pesticide Solution-II in Isooctane* plus gravimetrically prepared solutions of the additional analytes of interest with the internal standards added.

---

[1] Certain commercial equipment, instruments or materials are identified in this certificate to adequately describe 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 products identified are necessarily the best available for this purpose.

GC-ECD: For the GC-ECD analyses, approximately 2 g subsamples from 24 bottles of SRM 1945 were extracted using pressurized fluid extraction (PFE) with dichloromethane (DCM). Size exclusion chromatography (SEC) with a preparative-scale divinylbenzene-polystyrene column (10 μm particle size with 100 Å diameter pores) was used to remove the high molecular mass compounds. The concentrated extract was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm × 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5, Agilent Technologies, Wilmington, DE) and a 0.25 mm × 60 m fused silica capillary column with a non-polar proprietary phase (0.25 μm film thickness) (DB-XLB, Agilent Technologies). For the GC-ECD analyses, two PCB congeners that are not significantly present in the blubber extract (PCB 103 and PCB 198 [4,5]), and endosulfan I-d_4, 4,4'-DDE-d_8, 4,4'-DDD-d_8, and 4,4'-DDT-d_8 were added to the blubber prior to extraction for use as internal standards for quantification purposes.

GC/MS (I): For the GC/MS (I) analyses, approximately 1.5 g subsamples from six bottles of SRM 1945 were extracted using PFE with DCM. SEC with a preparative-scale divinylbenzene-polystyrene column (10 μm particle size with 100 Å diameter pores) was used to remove the high molecular mass compounds. The analytes of interest were then further isolated using a silica solid phase extraction (SPE) column. GC/MS analyses in the electron impact (EI) mode were performed on a 0.25 mm × 30 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (HP-5MS, Agilent Technologies). PCB 103, PCB 198, and carbon-13 labeled 4,4'-DDE, 4,4'-DDT, lindane, and *trans*-nonachlor were added to the blubber prior to extraction for use as internal standards for quantification purposes.

GC/MS (II): For the GC/MS (II) analyses, approximately 4 g subsamples from three bottles of SRM 1945 were Soxhlet extracted using with 1:1 hexane:acetone (volume fraction) for approximately 20 h. Concentrated sulfuric acid was used to remove the majority of the lipid and biogenic material. The analytes of interest were then further isolated using a silica SPE column. GC/MS analyses were performed on a 0.25 mm × 60 m fused silica capillary column with a non-polar proprietary phase (0.25 μm film thickness) (DB-XLB, Agilent Technologies) using two ionization modes, EI and negative chemical ionization (NCI). PCB 103, PCB 198, and carbon-13 labeled 4,4'-DDT, lindane, PCB 28, PCB 101, PCB 118, PCB 138, PCB 153, and PCB 169 were added to the blubber prior to extraction for use as internal standards for quantification purposes.

GC/MS (IIIa and IIIb): For the GC/MS (III) analyses, two sets of samples were run using the same method. One set included nine subsamples and the other set included six subsamples of SRM 1945 (approximately 1 g each) that were extracted using PFE with DCM. SEC with a preparative-scale divinylbenzene-polystyrene column (10 μm particle size with 100 Å diameter pores) was used to remove the high molecular mass compounds. The concentrated extract was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides and, (2) the more polar pesticides. GC/MS (EI) analyses were performed on a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5MS, Agilent Technologies). PCB 103, PCB 198, 4,4'-DDD-d_8, 4,4'-DDT-d_8, and carbon-13 labeled *cis*-chlordane were added to the blubber prior to extraction for use as internal standards for quantification purposes.

GC/MS (IV): For the GC/MS (IV) analyses, approximately 1 g subsamples from six bottles of SRM 1945 were extracted using PFE with DCM. SEC with a preparative-scale divinylbenzene-polystyrene column (10 μm particle size with 100 Å diameter pores) was used to remove the high molecular mass compounds. The analytes of interest were then further isolated using a silica SPE column. GC/MS (EI) analyses were performed on a 0.25 mm × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-17MS, Agilent Technologies). Carbon-13 labeled 4,4'-DDE, 4,4'-DDT, lindane, *trans*-nonachlor, PCB 28, PCB 101, PCB 105, PCB 153, and PCB 170 and 4,4'-DDD-d_8 were added to the blubber prior to extraction for use as internal standards for quantification purposes.

GC/MS (V): For the GC/MS (V) analyses, approximately 1 g subsamples from three bottles of SRM 1945 were extracted using PFE with DCM. SEC with a the following series of columns (PhenoGel, Phenomenex, Torrance, CA) was used to remove the high molecular mass compounds: guard column (50 mm × 7.80 mm i.d., 10 μm particle size), first column (600 mm × 21.2 mm i.d., 10 μm particle size and 100 Å diameter pores), and second column (300 mm × 21.2 mm i.d., 10 μm particle size and 100 Å diameter pores). The analytes of interest were then further isolated using an alumina SPE column. GC/MS analyses in both the EI and NCI mode were performed on a 0.18 mm × 30 m fused silica capillary column with a non-polar proprietary phase (0.18 μm film thickness) (DB-XLB, Agilent Technologies). Carbon-13 labeled PCB 28, PCB 52, PCB 77, PCB 118, PCB 126, PCB 153, PCB 169, PCB 180, PCB 194, PCB 206, *trans*-chlordane, BDE 99, and BDE 209 plus endosulfan I-d_4, 4,4'-DDE-d_8, 4,4'-DDD-d_8, and 4,4'-DDT-d_8 were added to the blubber prior to extraction for use as internal standards for quantification purposes.

GC/MS (VI): For the GC/MS (VI) analyses, approximately 1 g subsamples from three bottles of SRM 1945 were Soxhlet extracted using with DCM for approximately 20 h. SEC with a preparative-scale divinylbenzene-polystyrene column (10 µm particle size with 100 Å diameter pores) was used to remove the high molecular mass compounds. The analytes of interest were then further isolated using a silica SPE column. GC/MS (EI) analyses were performed on a 0.25 mm°× 60 m fused silica capillary column with a non-polar proprietary phase (0.25 µm film thickness) (DB-XLB, Agilent Technologies).   Carbon-13 labeled 4,4'-DDE, 4,4'-DDT, lindane, *trans*-nonachlor, PCB 28, PCB 101, PCB 105, PCB 153, and PCB 170 and 4,4'-DDD-d_8 were added to the blubber prior to extraction for use as internal standards for quantification purposes.

Non-ortho PCBs: The reference values for PCB 77, PCB 126, and PCB 169 were determined from a separate fraction. The samples were extracted and processed as for GC/MS (III) above. The first (PCB and lower polarity pesticide) fraction from the semi-preparative aminopropylsilane column was further fractionated using a Cosmosil PYE column (5 µm particle size, 4.6 mm i.d. × 25 cm, Phenomenex, Torrance, CA). Three fractions were collected: the first fraction contained the pesticides and multi-*ortho* PCBs, the second fraction contained the polychlorinated naphthalenes, non-*ortho* PCB congeners, and some mono-*ortho* PCB congeners, and the third fraction removed the residual planar compounds from the column.   The second fraction was analyzed by GC/MS NICI using a 0.25 mm × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5MS, Agilent Technologies).   Carbon-13 labeled PCB 77, PCB 126, and PCB 169 were used as internal standards for quantification purposes.

PBDEs and MeO-PBDEs GC/MS(VII): For one set of the PBDE analyses and for the MeO-PBDE analyses, approximately one gram subsamples from five bottles of SRM 1945 were extracted using PFE with DCM. SEC with a preparative-scale divinylbenzene polystyrene column (10 µm particle size with 100 Å diameter pores) was used to remove the high molecular mass compounds. GC/MS analyses in both the EI and NICI modes were performed on a 0.25 mm × 15 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5MS, Agilent Technologies).   Carbon-13 labeled BDE 15, BDE 209, and 2,2,3,4,5-pentachlorodiphenyl ether (CDE-86) were added to the blubber prior to extraction for use as internal standards for quantification purposes.

Interlaboratory Comparison Studies (IL): In addition to the analyses performed at NIST, SRM 1945 was used in an interlaboratory comparison exercise in 1999, 2000, 2001, and 2003 as part of the NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants in the Marine Mammal Tissues [6–8]. Results from 26 laboratories that participated in these exercises were used as the ninth data set in the determination of the certified values for PCB congeners, chlorinated pesticides, and PBDEs in SRM 1945. The laboratories participating in these exercises employed the analytical procedures routinely used in their laboratories to measure PCB congeners, chlorinated pesticides, and PBDEs.

Non-volatile Extractable Mass Determination: The percent of non-volatile extractable material (or total extractable organics) was determined gravimetrically for the nine data sets described above for the determination of PCBs and pesticides. The percent non-volatile extractable material is 71.9 % ± 1.3 % (95 % confidence interval for the mean).

Table 1. Certified Mass Fraction Values for Selected PCB Congeners[a] in SRM 1945

PCB Congener		Mass Fraction (µg/kg wet mass)[b]		
PCB 18	(2,2',5-Trichlorobiphenyl)[c,d,e,f,g,h,i,j,k]	4.35	±	0.56
PCB 28	(2,4,4'-Trichlorobiphenyl)[c,d,e,f,h,i,j,k]	13.1	±	1.1
PCB 31	(2,4',5-Trichlorobiphenyl)[c,d,e,h,i,j,k]	3.56	±	0.36
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	12.1	±	0.5
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	18.3	±	0.1
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	40.7	±	1.3
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	22.4	±	0.5
PCB 70	(2,3',4',5-Tetrachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	11.0	±	1.1
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)[c,f,g,i,j,k]	20.6	±	2.6
PCB 92	(2,2',3,5,5'-Pentachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	23.4	±	3.9
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)[c,f,g,i,j]	33.9	±	0.5
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	58.5	±	5.2
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	78	±	12
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	28.6	±	1.2
PCB 107	(2,3,3',4',5-Pentachlorobiphenyl)[c,f,g,i,j,k]	5.04	±	0.94
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)[c,f,g,i,j,k]	33.8	±	2.3
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	76.5	±	2.9
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	23.0	±	1.1
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	146	±	13
PCB 146	(2,2',3,4',5,5'-Hexachlorobiphenyl)[c,f,g,i,j,k]	45.1	±	4.0
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	89.0	±	6.9
PCB 151	(2,2',3,4,4',5-Hexachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	28.6	±	1.3
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)[c,h]	228	±	10[l]
PCB 154	(2,2',4,4',5,6'-Hexachlorobiphenyl)[c,f,g,i,j,k]	3.97	±	0.22
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	11.4	±	0.9
PCB 158	(2,3,3',4,4',6-Hexachlorobiphenyl)[c,g,i,j,k]	9.1	±	1.0
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	42.6	±	2.2
PCB 174	(2,2',3,3',4,5,6'-Heptachlorobiphenyl)[c,f,g,i,j,k]	25.2	±	0.1
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	138	±	10
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	38.0	±	1.8
PCB 187	(2,2',3,4,5,5',6-Heptachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	121	±	11
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	53.5	±	5.2
PCB 195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	14.3	±	2.2
PCB 201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	16.8	±	1.3
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	44.9	±	4.2
PCB 209	(Decachlorobiphenyl)[c,d,e,f,g,h,i,j,k]	17.2	±	1.9

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [4] and later revised by Schulte and Malisch [5] to conform with IUPAC rules. For the specific congeners mentioned in this table, the Ballschmiter-Zell numbers correspond to those of Schulte and Malisch. When two or more congeners are known to coelute under the GC analysis conditions used, the PCB congener listed first is the major component and the additional congeners may be present as minor components. The quantitative results are based on the response of the congener listed first.

[b] Certified values are weighted means of the results for up to nine data sets [9]. 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-source variance following the ISO/JCGM and NIST Guides [10].

[c] GC-ECD analysis on dual columns, DB-5 and DB-XLB following PFE using DCM.

[d] GC/MS(I) analysis on HP-5MS column following PFE using DCM.

[e] GC/MS(II) analysis on DB-XLB column following Soxhlet extraction using 1:1 hexane:acetone (volume fraction).

[f] GC/MS(IIIa) analysis on DB-5MS column following PFE using DCM.

[g] GC/MS(IIIb) analysis on DB-5MS column following PFE using DCM.

[h] GC/MS(IV) analysis on DB-17MS column following PFE using DCM.

[i] GC/MS(V) analysis on DB-XLB column following PFE using DCM.

[j] GC/MS(VI) analysis on DB-XLB column following Soxhlet extraction using DCM.

[k] Combination of consensus means from four interlaboratory comparison exercises.

[l] Certified value is the unweighted mean of the results for three data sets. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/JCGM and NIST Guides [10].

Table 2. Certified Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1945

	Mass Fraction (µg/kg wet mass)[a]		
2,4'-DDT[b,c,d,e,f,g,h,i,j]	91	±	14
4,4'-DDT[b,c,d,e,f,g,h,i,j]	233	±	8
2,4'-DDE[b,c,d,e,f,g,h,i,j]	14.2	±	1.4
4,4'-DDE[b,c,d,e,f,g,h,j]	497	±	19
2,4'-DDD[b,c,d,e,f,g,h,i,j]	19.5	±	1.2
4,4'-DDD[b,c,d,e,f,g,h,j]	120	±	5
Hexachlorobenzene[b,c,d,e,f,g,h,i,j]	30.6	±	1.5
α-hexachlorocyclohexane (HCH)[b,c,d,e,f,g,h,j]	16.9	±	1.4
γ-HCH[b,c,d,e,f,g,h,j]	3.18	±	0.01
heptachlor epoxide[b,c,d,e,f,g,i,j]	10.7	±	0.09
cis-chlordane[b,c,d,e,f,g,h,j]	48.1	±	1.6
trans-chlordane[b,c,d,e,f,g,h,j]	11.8	±	0.5
oxychlordane[b,c,d,e,f,g,h,i,j]	21.2	±	1.1
cis-nonachlor[b,c,d,e,f,g,h,i,j]	45.8	±	3.3
trans-nonachlor[b,c,d,e,f,g,h,i,j]	198	±	16
dieldrin[b,c,e,f,g,h,i,j]	50.1	±	4.1
mirex[b,c,d,e,f,g,h,i,j]	31.0	±	3.4

[a] Certified values are weighted means of the results for up to nine data sets [9]. 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-source variance following the ISO/JCGM and NIST Guides [10].

[b] GC-ECD analysis on dual columns, DB-5 and DB-XLB following PFE using DCM.

[c] GC/MS(I) analysis on HP-5MS column following PFE using DCM.

[d] GC/MS(II) analysis on DB-XLB column following Soxhlet extraction using 1:1 hexane:acetone (volume fraction).

[e] GC/MS(IIIa) analysis on DB-5MS column following PFE using DCM.

[f] GC/MS(IIIb) analysis on DB-5MS column following PFE using DCM.

[g] GC/MS(IV) analysis on DB-17MS column following PFE using DCM.

[h] GC/MS(V) analysis on DB-XLB column following PFE using DCM.

[i] GC/MS(VI) analysis on DB-XLB column following Soxhlet extraction using DCM.

[j] Combination of consensus means from four interlaboratory comparison exercises.

Table 3. Certified Mass Fraction Values for Selected PBDE Congeners[a] in SRM 1945

		Mass Fraction (µg/kg wet mass)		
PBDE 47	(2,2',4,4'-Tetrabromodiphenyl ether)[b,c,d,e]	39.6	±	0.2[f]
PBDE 99	(2,2',4,4',5-Pentabromodiphenyl ether)[b,d,e]	18.9	±	2.3[g]
PBDE 100	(2,2',4,4',6-Pentabromodiphenyl ether)[b,c,d,e]	10.3	±	1.1[f]
PBDE 153	(2,2',4,4',5,5'-Hexabromodiphenyl ether)[b,c,d,e]	8.34	±	0.55[f]
PBDE 154	(2,2',4,4',5,6'-Hexabromodiphenyl ether)[b,c,d,e]	13.3	±	1.7[f]

[a] PBDE congeners are numbered according to IUPAC rules.

[b] GC/MS(IIIa) analysis on DB-5MS column following PFE using DCM.

[c] GC/MS(V) analysis on DB-XLB column following PFE using DCM.

[d] GC/MS(VII) analysis on DB-5MS column following PFE using DCM.

[e] Combination of consensus means from four interlaboratory comparison exercises.

[f] Certified values are weighted means of the results for four data sets [9]. 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-source variance following the ISO/JCGM and NIST Guides [10].

[g] Certified values are unweighted means of the results for three data sets. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/JCGM and NIST Guides [10].

Table 4. Reference Mass Fraction Values for Selected PCB Congeners[a], Chlorinated Pesticide, and PBDE Congeners[a] in SRM 1945

		Mass Fraction (µg/kg wet mass)		
PCB 56	(2,3,3',4'-Tetrachlorobiphenyl)[b,c,d]	7.5	±	1.8[e]
PCB 74	(2,3,3',4'-Tetrachlorobiphenyl)[b,c,d,f,g,h]	21.6	±	7.8[i]
PCB 77	(3,3',4,4'-Tetrachlorobiphenyl)[j]	0.416	±	0.061[e]
PCB 82	(2,2',3,3',4-Pentachlorobiphenyl)[b,c,d,g,h]	4.5	±	1.1[i]
PCB 119	(2, 3',4,4',6-Pentachlorobiphenyl)[c,d,g]	3.7	±	1.0[e]
PCB 126	(3,3',4,4',5-Pentachlorobiphenyl)[j]	0.182	±	0.048[e]
PCB 130	(2,2',3,3',4,5'-Hexachlorobiphenyl)[c,g]	10.2	±	0.6[e]
PCB 132	(2,2',3,3',4,6'-Hexachlorobiphenyl)[d,f,k]	21.1	±	4.8[e]
PCB 137	(2,2',3,4,4',5-Hexachlorobiphenyl)[c,d,g]	7.7	±	1.4[e]
PCB 157	(2,3,3',4,4',5'-Hexachlorobiphenyl)[b,c,f,g,h]	3.27	±	0.67[i]
PCB 163	(2,3,3',4',5,6-Hexachlorobiphenyl)[c,f,g,h]	46.7	±	8.1[i]
PCB 169	(3,3',4,4',5,5'-Hexachlorobiphenyl)[j]	0.158	±	0.041[e]
PCB 172	(2,2',3,3',4,5,5'-Heptachlorobiphenyl)[b,c,d,g]	12.4	±	3.4[i]
PCB 175	(2,2',3,3',4,5,6'-Heptachlorobiphenyl)[c,d,g]	4.25	±	0.98[e]
PCB 176	(2,2',3,3',4,6,6'-Heptachlorobiphenyl)[c,d,g]	6.2	±	1.0[e]
PCB 177	(2,2',3,3',4',5,6-Heptachlorobiphenyl)[c,d,g]	28.6	±	1.2[e]
PCB 178	(2,2',3,3',5,5',6-Heptachlorobiphenyl)[c,d,g]	20.7	±	1.7[e]
PCB 185	(2,2',3,4,5,5',6-Heptachlorobiphenyl)[c,d]	3.40	±	0.32[e]
PCB 191	(2,3,3',4,4',5',6-Heptachlorobiphenyl)[c,d]	1.57	±	0.45[e]
PCB 193	(2,3,3',4',5,5',6-Heptachlorobiphenyl)[g,h]	8.7	±	1.5[e]
PCB 196	(2,2',3,3',4,4',5,6'-Octachlorobiphenyl)[c,d,g]	74.5	±	8.4[e]
PCB 197	(2,2',3,3',4,4',6,6'-Octachlorobiphenyl)[c,d,g]	5.74	±	0.79[e]
PCB 199	(2,2',3,3',4,5,5',6'-Octachlorobiphenyl)[c,d,g]	84.2	±	8.0[e]
PCB 200	(2,2',3,3',4,5,6,6'-Octachlorobiphenyl)[c,d,g]	4.30	±	0.32[e]
PCB 202	(2,2',3,3',5,5',6,6'-Octachlorobiphenyl)[c,d,g]	29.0	±	7.0[e]
PCB 205	(2,3,3',4,4',5,5',6-Octachlorobiphenyl)[c,g]	2.30	±	0.14[e]
PCB 207	(2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl)[c,d,g]	14.5	±	2.4[e]
PCB 208	(2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl)[c,d,g]	23.9	±	1.7[e]
Pentachlorobenzene[c,g]		2.09	±	0.53[e]
PBDE 28	(2,4,4'-Tribromodiphenyl ether)[g,l]	2.66	±	0.37[e]
PBDE 155	(2,2',4,4',6,6'-Hexabromodiphenyl ether)[g,l]	4.75	±	0.93[e]
2'-MeOPBDE 68 (2'-methoxy-2,3',4,5'-Tetrabromodiphenyl ether)[l]		53.5	±	3.3[m]
6'-MeO-PBDE 47 (6'-methyoxy-2,2',4,4'Tetrabromodiphenyl ether)[l]		66.5	±	4.7[m]

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [4] and later revised by Schulte and Malisch [5] to conform with IUPAC rules. 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. PBDE congeners are numbered according to IUPAC rules.

[b] GC/MS(IIIa) analysis on DB-5MS column following PFE using DCM.

[c] GC/MS(IIIb) analysis on DB-5MS column following PFE using DCM.

[d] Combination of consensus means from four interlaboratory comparison exercises.

[e] Reference values are unweighted means of the results for two or three data sets. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [11] with a pooled, within-method variance following the ISO/JCGM and NIST Guides [10].

[f] GC-ECD analysis on dual columns, DB-5 and DB-XLB following PFE using DCM.

[g] GC/MS(V) analysis on DB-XLB column following PFE using DCM.

[h] GC/MS(VI) analysis on DB-XLB column following Soxhlet extraction using DCM.

[i] Reference values are weighted means of the results for up to six data sets [9]. 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-source variance following the ISO/JCGM and NIST Guides [10].

[j] Non-ortho PCB method plus two sets of data from interlaboratory study.

[k] GC/MS(IV) analysis on DB-17MS column following PFE using DCM.

[l] GC/MS(VII) analysis for PBDEs and MeO-PBDEs.

[m] The reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte.

Table 5. Reference Concentrations for Selected Toxaphene Congeners and Total Toxaphene[(a)] in SRM 1945

	Mass Fraction (µg/kg wet mass)		
TOX 26[(b)] (2-*endo*, 3-*exo*, 5-*endo*, 6-*exo*, 8,8,10,10-octachlorobornane)	41.8	±	2.8
TOX 50[(b)] (2-*endo*, 3-*exo*, 5-*endo*, 6-*exo*, 8,8,9,10,10-nonachlorobornane)	69.7	±	5.5
TOX 62[(b)] (2,25,5,8,9,9,10,10-nonachlorobornane)	37.2	±	3.8
Total Toxaphene	1220	±	100

[(a)] Methods described in more detail in reference 12. The reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to the associated degrees of freedom and 95 % confidence level for each analyte.

[(b)] The two-digit nomenclature is the "Parlar System" described in reference 13.

REFERENCES

[1]   Schantz, M.; Koster, B.J.; Oakley, L.M.; Schiller, S.B.; Wise, S.A.; *Certification of Polychlorinated Biphenyl Congeners and Chlorinated Pesticides in a Whale Blubber Standard Reference Material*; Anal. Chem., Vol. 67, pp. 901–910 (1995).

[2]   May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at: http://www.nist.gov/srm/publications.cfm (accessed Apr 2016).

[3]   Zeisler, R.; Langland, J.K.; Harrison, S.H.; *Cryogenic Homogenization of Biological Tissues*; Anal. Chem.; Vol. 55, pp. 2431–2434 (1983).

[4]   Ballschmiter, K.; Zell, M.; *Analysis of Polychlorinated Biphenyls (PCB) by Glass Capillary Gas Chromatography - Composition of Technical Aroclor- and Clophen-PCB Mixtures*; Fresenius Z. Anal. Chem., Vol. 302, pp. 20–31 (1980).

[5]   Schulte, E.; Malisch, R.; *Calculation of the Real PCB Content in Environmental Samples. I. Investigation of the Composition of Two Technical PCB Mixtures*; Fresenius Z. Anal. Chem., Vol. 314, pp. 545–551 (1983).

[6]   Kucklick, J.R.; Christopher, S.J.; Becker, P.R.; Pugh, R.S.; Porter, B.J.; Schantz, M.M.; Mackey, E.A.; Wise, S.A.; Rowles, T.K.; *Description and Results of the 2000 NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants and Trace Elements in Marine Mammal Tissues*; NISTIR 6849; National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2002).

[7]   Kucklick, J.R.; Pugh, R.S.; Becker, P.R.; Schantz, M.M.; Wise, S.A.; Rowles, T.K.; *Description and Results of the 2001 NIST/NOAA Interlaboratory Comparison Exercise Program for Organic Contaminants in Marine Mammal Tissues*, NISTIR 7318; National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2006).

[8]   Kucklick, J.R.; Pugh, R.S.; Becker, P.R.; Schantz, M.M.; Wise, S.A.; Rowles, T.K.; *Description and Results of the 2003 Interlaboratory Comparison Exercise Program for Organic Contaminants in Marine Mammal Tissues*, NISTIR 7269; National Institute of Standards and Technology, U.S. Department of Commerce: Gaithersburg, MD (2005).

[9]   Ruhkin, A.L.; Vangel, M.G.; *Estimation of a Common Mean and Weighted Means Statistics*; J. Am. Stat. Assoc., Vol. 93, pp. 303–308 (1998).

[10] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2016); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office: Washington, DC (1994); available at http://www.nist.gov/pml/pubs/index.cfm (accessed Apr 2016).

[11] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571–579 (2000).

[12] Kucklick, J.R.; Tuerk, K.J.S.; Vander Pol, S.S.; Schantz, M.M.; Wise, S.A.; *Polybrominated Diphenyl Ether Congeners and Toxaphene in Selected Marine Standard Reference Materials*; Anal. Bioanal. Chem., Vol. 378, pp. 1147–1151 (2004).

[13] Hainzl, D.; Burhenne, J.R.; Barlas, H.; Parlar, H.; *Spectroscopic Characterization of Environmentally Relevant C_{10}-Chloroterpenes from a Photochemically Modified Toxaphene Standard*; Anal. Bioanal. Chem., Vol. 351, pp. 271–285 (1995).

The laboratories listed below performed measurements that contributed to the certification of SRM 1945 *Organics in Whale Blubber*.

Baltic Sea Research Institute (Rostock, Germany)
Center for Environment, Fisheries, and Aquaculture Science (Burnam-on-Crounch, Essex, United Kingdom)
Department of Biology, University of Ottawa (Ottawa, Canada)
Ehime University, Center for Marine Environmental Studies (Ehime, Japan)
Environment Canada, Fisheries and Oceans Canada, Freshwater Institute (Winnipeg, Canada)
Environment Canada, National Water Research Insititute (Burlington, Ontario, Canada)
Fisheries and Oceans Canada, Institute of Ocean Sciences (Sidney, British Columbia, Canada)
Kumamoto University, Department of Environmental Sciences (Kumamoto, Japan)
Lancaster University, Department of Environmental Sciences (Bailrigg, Lancaster, United Kingdom)
Michigan State University, National Food Safety and Toxicology Center (East Lansing, Michigan, USA)
Mississippi State Chemical Lab. (Mississippi, State, Mississippi, USA)
National Environmental Research Institute, Department of Environmental Chemistry (Roskilde, Denmark)
National Oceanic and Atmospheric Administration (NOAA), CCEHBR (Charleston, South Carolina, USA)
NOAA, Northeast Fisheries Science Center (Sandy Hook, New Jersey, USA)
NOAA, Northwest Fisheries Science Center (Seattle, Washington, USA)
Norwegian School of Veterinary Science (Oslo, Norway)
Oklahoma Animal Disease and Diagnostic Laboratory, Oklahoma State University (Stillwater, Oklahoma, USA)
Organic Chemistry Institute, Deparment of Instrumental Analysis and Environmental Chemistry (Madrid, Spain)
Texas A&M University, Geochemical and Environmental Research Group (College Station, Texas, USA)
Toxicology Center, University of Antwerp (Antwerp, Begium)
University of Barcelona (Barcelona, Spain)
University of Connecticut, Environmental Research Institute (Storrs, Connecticut, USA)
University of Georgia System, Skidaway Institute of Oceanography (Savannah, Georgia, USA)
University of Pennsylvania, School of Veterinary Medicine (Kennett Square, Pennsylvania, USA)
University of Utah, Energy and Geosciences Institute (Salt Lake City, Utah, USA)
Wadsworth Center, New York State Department of Health (Albany, New York, USA)

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.