



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1588c

Organics in Fish Oil

This Standard Reference Material (SRM) is intended primarily for use in developing and validating analytical methods for the determination of polychlorinated biphenyls (PCBs), chlorinated pesticides, polybrominated diphenyl ethers (PBDEs), and fatty acids in fish oils and similar materials. SRM 1588c can also be used for quality assurance when assigning values to in-house reference materials. A unit of SRM 1588c consists of five ampoules with each ampoule containing approximately 1.2 mL of fish oil.

Certified Mass Fraction Values: Certified mass fraction values for 44 PCB congeners (one pair in combination), 8 chlorinated pesticides, 7 PBDE congeners, and 18 fatty acids are provided in Tables 1 through 4, respectively. 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 [1]. Values are reported on an as-received basis in mass fraction units [2]. The measurands are the total mass fraction for the analytes listed in Tables 1 through 4 and are metrologically traceable to the SI unit for mass, expressed as micrograms per kilogram in Tables 1 through 3 and milligrams per gram in Table 4.

Reference Mass Fraction Values: Reference mass fraction values for 12 PCB congeners, 2 chlorinated pesticides, and 3 fatty acids are provided in Tables 5 through 7, respectively. A NIST reference value is a noncertified value that is the best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification [1] and is 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. Values are reported on an as-received basis in mass fraction units [2]. The measurand is the total mass fraction for the analytes listed in Tables 5 through 7 as determined by the methods indicated and is metrologically traceable to the SI unit for mass, expressed as micrograms per kilogram in Tables 5 and 6 and milligrams per gram in Table 7.

Expiration of Certification: The certification of **SRM 1588c** is valid, within the measurement uncertainty specified, until **03 June 2015**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, 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 on-line) will facilitate notification.

Coordination of the technical measurements leading to the certification of this SRM was performed by M.M. Schantz former of NIST and L.C. Sander of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by J. Hoguet, and C. Luvonga (formerly of NIST Analytical Sciences Division) and J.R. Kucklick, of the NIST Chemical Sciences Division and M.M. Schantz.

Statistical analysis was provided by S.D. Leigh and A. Possolo of the NIST Statistical Engineering Division.

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

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Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

NOTICE AND WARNING TO USERS

SRM 1588c IS INTENDED FOR LABORATORY USE ONLY. IT IS NOT FOR HUMAN CONSUMPTION.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling and Use: Before use, the contents of the ampoule should be mixed thoroughly. Test portions of this SRM for analysis should be drawn from ampoules immediately after opening and used without delay for the certified values to be valid within the stated uncertainties.

Storage: The SRM should be stored in the dark at temperatures less than 30 °C, in an unopened ampoule, until required for use. The certification does not apply to contents of previously opened and stored ampoules as the stability of all analytes has not been investigated.

SOURCE, PREPARATION, AND ANALYSIS⁽¹⁾

Source and Preparation: This SRM is composed of menhaden oil (*Brevoortia tyrannus*) collected in 2003 and 2004 from six rendering plants along the Southeast Atlantic and Gulf Coasts of the United States. The oil was provided by the National Oceanic and Atmospheric Administration, National Centers for Coastal Ocean Research, Hollings Marine Laboratory, Charleston, SC. The oil was filtered and mixed prior to ampouling. Each 2-mL amber glass ampoule was purged with argon prior to addition of approximately 1.2 mL of oil.

Analytical Approach for Determination of PCBs, Chlorinated Pesticides, and PBDEs: Value assignment of PCB congener, chlorinated pesticide, and PBDE congener mass fractions in SRM 1588c was based on the combination of measurements made using multiple analytical methods at NIST, all of which were based on gas chromatography (GC) with mass spectrometric detection (MS) as described below.

For GC/MS I, two 0.5 g test portions from each of 10 ampoules of SRM 1588c were combined with an internal standard solution containing carbon-13 labeled 4,4'-DDE, 4,4'-DDT, γ -HCH, *trans*-nonachlor, PBDE 209, and PCB 28, PCB 52, PCB 101, PCB 138, PCB 153, PCB 180, and PCB 209, and fluorinated PBDE congeners 47 and 208. Samples were mixed with hexane and sulfuric acid, followed by the separation and removal of the hexane phase. The sulfuric acid layer was washed an additional two times with hexane. The hexane phases were combined and concentrated to approximately 0.5 mL. The concentrated extracts were eluted through a silica solid-phase extraction (SPE) column using 20 mL of 10 % (volume fraction) methylene chloride in hexane. The collected fractions were concentrated for GC/MS analysis. The PCB congeners and chlorinated pesticides were quantified using GC/MS in the electron ionization (EI) mode with a 0.25 mm \times 60 m fused silica capillary column containing a 50 % phenyl + 50 % methylpolysiloxane (mole fraction) phase. The PBDE congeners were quantified using GC/MS in the negative chemical ionization (NCI) mode using a 0.25 mm \times 15 m fused silica capillary column containing a 50 % liquid crystal + 50 % methylpolysiloxane (mole fraction) phase. A single internal standard solution was used for the calibrants and samples. Quantitation was based on a calibration curve bracketing the concentrations in the oil samples.

For GC/MS II, single 1 g test portions from each of six ampoules of SRM 1588c were combined with an internal standard solution containing deuterated 4,4'-DDD, carbon-13 labeled *trans*-chlordane, *trans*-nonachlor, oxychlordane, HCB, 4,4'-DDT, 4,4'-DDE, PBDE 209, PCB 28, PCB 52, PCB 77, PCB 188, PCB 126, PCB 153, PCB 169, PCB 180, PCB 194, PCB 206, PBDE 104, and fluorinated PBDE 47, 160, and 208. The oil and internal standard were mixed with sodium sulfate in a pressurized-fluid extraction (PFE) cell. The PFE conditions were: solvent, methylene chloride; cell temperature, 100 °C; equilibration time, 5 min; static time, 5 min; cell pressure, 13.8 MPa; and three cycles. The extracts were concentrated to 1 mL for size exclusion chromatography (SEC). SEC was performed using a preparative-scale column to remove the majority of the lipids and high molecular mass compounds. The SEC fraction was concentrated and eluted through an alumina SPE column using 9 mL of 35 % (volume fraction) methylene chloride in hexane. The SPE fractions were concentrated for GC/MS analysis. GC/MS was run in the EI mode and NCI mode using a 0.18 mm \times 30 m fused silica capillary column containing a 5 % phenyl + 95 % methylpolysiloxane (mole fraction) phase. A single internal standard solution was used for the calibrants and samples. Quantitation was based on a calibration curve bracketing the concentrations in the oil samples.

⁽¹⁾ Certain commercial equipment, instruments, or materials are identified in this certificate in order 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 necessarily the best available for the purpose.

Analytical Approach for Determination of Fatty Acids: Value assignment of the concentrations of fatty acids in 1588c was based on the combination of measurements made using two independent analytical methods at NIST: GC with flame ionization detection (FID) and GC/MS as described below.

For GC-FID, two 0.5 g test portions from each of 10 ampoules of SRM 1588c were combined with an internal standard solution containing octacosanoic acid and myristic- d_{27} acid. MethPrep II [0.1 mol/L methanolic (*m*-trifluoromethylphenyl) trimethylammonium hydroxide, Alltech, Deerfield, IL] and 2,2,4-trimethylpentane were added stepwise for conversion of fatty acids to their methyl esters (FAMES) prior to analysis by GC-FID using a 0.25 mm \times 100 m nonbonded biscyanopropyl polysiloxane fused silica capillary column. Calibrants were prepared gravimetrically, at levels intended to approximate the levels of the fatty acids in the SRM. A single internal standard solution was used for the calibrants and samples. Quantification was based on average response factors for the calibrants.

For GC/MS, two 0.5 g test portions from each of 10 ampoules of SRM 1588c were combined with an internal standard solution containing octacosanoic acid and myristic- d_{27} acid. A two-step process employing methanolic sodium hydroxide and boron trifluoride was used to convert the fatty acids to FAMES. GC/MS was performed using a 0.25 mm \times 60 m fused silica capillary column containing a 50 % cyanopropyl + 50 % methylpolysiloxane (mole fraction) phase. Calibrants were prepared gravimetrically, at levels intended to approximate the levels of the fatty acids in the SRM. A single internal standard solution was used for the calibrants and samples. Calculations are based on average response factors for the calibrants.

Homogeneity Assessment: The homogeneity of PCBs, chlorinated pesticides, and PBDEs was assessed at NIST using the GC/MS I method described above, and the homogeneity of the fatty acids was assessed at NIST using the methods and test portion sizes described above. The analyses of variance did not show statistically significant heterogeneity.

Value Assignment: For calculation of assigned values, the means of the individual sets of data were averaged.

Table 1. Certified Mass Fraction Values for Selected PCB Congeners in SRM 1588c^(a).

		Mass Fraction ^(b) (µg/kg)	
PCB 44	(2,2',3,5'-Tetrachlorobiphenyl)	4.481	± 0.083
PCB 49	(2,2',4,5'-Tetrachlorobiphenyl)	9.369	± 0.078
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl)	12.75	± 0.19
PCB 56	(2,3,3',4'-Tetrachlorobiphenyl)	4.20	± 0.10
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl)	7.16	± 0.15
PCB 70	(2,3',4',5'-Tetrachlorobiphenyl)	7.501	± 0.071
PCB 74	(2,4,4',5'-Tetrachlorobiphenyl)	4.13	± 0.16
PCB 82	(2,2',3,3',4-Pentachlorobiphenyl)	1.541	± 0.035
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl)	6.19	± 0.11
PCB 92	(2,2',3,5,5'-Pentachlorobiphenyl)	6.040	± 0.068
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl)	12.27	± 0.65
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl)	19.77	± 0.38
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl)	22.94	± 0.23
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl)	4.584	± 0.058
PCB 107	(2,3,3',4',5-Pentachlorobiphenyl)	3.012	± 0.050
PCB 110	(2,3,3',4',6-Pentachlorobiphenyl)	20.10	± 0.40
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl)	15.04	± 0.54
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl)	15.2	± 1.2
PCB 130	(2,2',3,3',4,5'-Hexachlorobiphenyl)	2.290	± 0.049
PCB 137	(2,2',3,4,4',5-Hexachlorobiphenyl)	1.003	± 0.034
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl)	25.22	± 0.34
PCB 146	(2,2',3,4',5,5'-Hexachlorobiphenyl)	10.81	± 0.33
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl)	27.56	± 0.27
PCB 151	(2,2',3,5,5',6-Hexachlorobiphenyl)	7.868	± 0.092
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl)	57.38	± 0.33
PCB 156	(2,3,3',4,4',5-Hexachlorobiphenyl)	3.63	± 0.14
PCB 157	(2,3,3',4,4',5'-Hexachlorobiphenyl)	1.237	± 0.027
PCB 158	(2,3,3',4,4',5-Hexachlorobiphenyl)	4.37	± 0.13
PCB 163	(2,3,3',4',5,6-Hexachlorobiphenyl)	11.13	± 0.20
PCB 167	(2,3',4,4',5,5'-Hexachlorobiphenyl)	3.177	± 0.051
PCB 170	(2,2',3,3',4,4',5-Heptachlorobiphenyl)	7.87	± 0.15
PCB 172	(2,2',3,3',4,5,5'-Heptachlorobiphenyl)	3.081	± 0.064
PCB 174	(2,2',3,3',4,5,6'-Heptachlorobiphenyl)	17.3	± 1.3
PCB 177	(2,2',3,3',4',5,6-Heptachlorobiphenyl)	11.07	± 0.27
PCB 178	(2,2',3,3',5,5',6-Heptachlorobiphenyl)	10.26	± 0.27
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) ^(c)	14.21	± 0.25
PCB 193	(2,3,3',4',5,5,6-Heptachlorobiphenyl) ^(c)		
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl)	14.63	± 0.36
PCB 187	(2,2',3,4',5,5',6-Heptachlorobiphenyl)	52.2	± 1.5
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl)	3.63	± 0.16
PCB 195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl)	1.296	± 0.032
PCB 199	(2,2',3,3',4,5,5',6'-Octachlorobiphenyl)	5.634	± 0.087
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)	4.30	± 0.17
PCB 209	(2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl)	3.414	± 0.086

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [3] and later revised by Schulte and Malisch [4] to conform with IUPAC rules.

^(b) The certified mass fraction value is a weighted mean of the mass fractions determined by methods GC/MS I and GC/MS II [5]. The uncertainty listed with each value is an expanded uncertainty about the mean [5,6], with coverage factor $k = 2$, calculated by combining a pooled within-method variance with a between-method variance following the ISO Guide [7,8].

^(c) Under the conditions used PCB 193 coeluted with PCB 180.

Table 2. Certified Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1588c.

	Mass Fraction ^(a) (µg/kg)		
4,4'-DDE	104.0	±	1.6
2,4'-DDD	9.546	±	0.072
4,4'-DDD	65.97	±	0.99
4,4'-DDT	71.5	±	1.6
<i>cis</i> -Chlordane	12.34	±	0.38
<i>cis</i> -Nonachlor	14.64	±	0.49
<i>trans</i> -Nonachlor	17.01	±	0.50
Oxychlordane	1.78	±	0.17

^(a) The certified mass fraction value is a weighted mean of the mass fractions determined by methods GC/MS I and GC/MS II [5]. The uncertainty listed with each value is an expanded uncertainty about the mean [5,6], with coverage factor $k = 2$, calculated by combining a pooled within-method variance with a between-method variance following the ISO Guide [7,8].

Table 3. Certified Mass Fraction Values for Selected PBDE Congeners in SRM 1588c^(a).

	Mass Fraction ^(b) (µg/kg)		
PBDE 28 (2,4,4'-Tribromodiphenylether) ^(c)	1.708	±	0.035
PBDE 33 (2',3,4'-Tribromodiphenylether) ^(c)			
PBDE 47 (2,2',4,4'-Tetrabromodiphenylether)	25.3	±	1.5
PBDE 49 (2,2',4,5'-Tetrabromodiphenylether)	6.29	±	0.16
PBDE 99 (2,2',4,4',5'-Pentabromodiphenylether)	1.70	±	0.14
PBDE 100 (2,2',4,4',6-Pentabromodiphenylether)	5.23	±	0.40
PBDE 154 (2,2',4,4',5,6'-Hexabromodiphenylether)	1.113	±	0.039

^(a) PBDE congeners are numbered according to IUPAC rules.

^(b) The certified mass fraction value is a weighted mean of the mass fractions determined by methods GC/MS I and GC/MS II [5]. The uncertainty listed with each value is an expanded uncertainty about the mean [5,6], with coverage factor $k = 2$, calculated by combining a pooled within-method variance with a between-method variance following the ISO Guide [7,8].

^(c) PBDE 28 coeluted with PBDE 33 under the conditions used.

Table 4. Certified Mass Fraction Values for Fatty Acids as Fatty Acid Methyl Esters (FAMES) in SRM 1588c.

	Mass Fraction ^(a) (mg/g)	
Tetradecanoic Acid (C14:0; Myristic Acid)	98	± 13
Hexadecanoic Acid (C16:0; Palmitic Acid)	166.9	± 1.6
(Z)-9-Hexadecenoic Acid (C16:1 n-7) (Palmitoleic Acid)	117.6	± 5.3
Octadecanoic Acid (C18:0; Stearic Acid)	29.9	± 1.1
(Z)-9-Octadecenoic Acid (C18:1 n-9) (Oleic Acid)	68.2	± 3.6
(Z)-11-Octadecenoic Acid (C18:1 n-7) (Vaccenic Acid)	27.7	± 1.3
(Z,Z)-9,12-Octadecadienoic Acid (C18:2 n-6) (Linoleic Acid)	11.34	± 0.52
(Z,Z,Z)-9,12,15-Octadecatrienoic Acid (C18:3 n-3) (α -Linolenic Acid; ALA)	10.66	± 0.65
(Z,Z,Z)-6,9,12-Octadecatrienoic Acid (C18:3 n-6) (γ -Linolenic Acid; GLA)	2.95	± 0.26
Eicosanoic Acid (C20:0; Arachidic Acid)	2.29	± 0.24
(Z)-11-Eicosenoic Acid (C20:1 n-9) (Gondoic Acid)	8.92	± 0.94
(Z,Z,Z,Z)-5,8,11,14-Eicosatetraenoic Acid (C20:4 n-6) (Arachidonic Acid)	12.78	± 0.70
(Z,Z,Z,Z,Z)-5,8,11,14,17-Eicosapentaenoic Acid (C20:5 n-3; EPA)	120	± 11
Docosanoic Acid (C22:0; Behenic Acid)	1.349	± 0.055
(Z)-13-Docosenoic Acid (C22:1 n-9; Erucic Acid)	1.105	± 0.048
(Z,Z,Z,Z,Z)-7,10,13,16,19-Docosapentaenoic Acid (C22:5; DPA)	25.5	± 2.3
(Z,Z,Z,Z,Z,Z)-4,7,10,13,16,19-Docosahexaenoic Acid (C22:6 n-3; DHA)	87.2	± 5.0
Tetracosanoic Acid (C24:0; Lignoceric Acid)	1.08	± 0.15

^(a) The certified mass fraction value is a weighted mean of the mass fractions determined by GC-FID and GC/MS [5]. The uncertainty listed with each value is an expanded uncertainty about the mean [5,6], with coverage factor $k = 2$ (approximately 95 % confidence), calculated by combining a pooled within-method variance with a between-method variance [9] following the ISO Guide [7,8].

Table 5. Reference Mass Fraction Values for Selected PCB Congeners in SRM 1588c.

		Mass Fraction ^(a) (µg/kg)	
PCB 28	(2,4,4'-Trichlorobiphenyl)	2.807	± 0.064 ^(b)
PCB 31	(2,4',5-Trichlorobiphenyl)	1.479	± 0.056 ^(b)
PCB 154	(2,2',4,4',5,6'-Hexachlorobiphenyl)	3.735	± 0.094 ^(c)
PCB 175	(2,2',3,3',4,5',6-Heptachlorobiphenyl)	1.308	± 0.068 ^(c)
PCB 176	(2,2',3,3',4,6',6-Heptachlorobiphenyl)	0.782	± 0.049 ^(c)
PCB 185	(2,2',3,4,5,5',6-Heptachlorobiphenyl)	1.96	± 0.13 ^(c)
PCB 196	(2,2',3,3',4,4',5,6'-Octachlorobiphenyl) ^(d)	5.01	± 0.30 ^(c)
PCB 203	(2,2',3,4,4',5,5',6-Octachlorobiphenyl) ^(d)		
PCB 201	(2,2',3,3',4,5',6,6'-Octachlorobiphenyl)	2.21	± 0.15 ^(c)
PCB 202	(2,2',3,3',5,5',6,6'-Octachlorobiphenyl)	5.32	± 0.49 ^(c)
PCB 207	(2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl)	0.630	± 0.017 ^(c)
PCB 208	(2,2',3,3',4,5,5',6,6'-Nonachlorobiphenyl)	2.23	± 0.13 ^(c)

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [3] and later revised by Schulte and Malisch [4] to conform with IUPAC rules.

^(b) The reference mass fraction value is the mean of results obtained using one analytical technique, GC/MS I. The expanded uncertainty, U , is calculated as $= ku_c$, 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 19 degrees of freedom and a 95 % confidence level for each analyte.

^(c) The reference mass fraction value is the mean of results obtained using one analytical technique, GC/MS II. The expanded uncertainty, U , is calculated as $= ku_c$, 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 5 degrees of freedom and a 95 % confidence level for each analyte.

^(d) PCB 196 and PCB 203 coeluted under the conditions used.

Table 6. Reference Mass Fraction Values for Selected Chlorinated Pesticides in SRM 1588c.

	Mass Fraction ^(a) (µg/kg)	
2,4'-DDE	6.61	± 0.33
<i>Trans</i> -Chlordane	11.98	± 0.95

^(a) The reference mass fraction value is the mean of results obtained using one analytical technique, GC/MS II. The expanded uncertainty, U , is calculated as $= ku_c$, 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 5 degrees of freedom and a 95 % confidence level for each analyte.

Table 7. Reference Mass Fraction Values for Fatty Acids as Fatty Acid Methyl Esters (FAMES) in SRM 1588c.

	Mass Fraction ^(a) (mg/g)	
Dodecanoic Acid (C12:0; Lauric Acid)	1.33	± 0.10
(Z)-9-Tetradecenoic Acid (C14:1 n-5; Myristoleic Acid)	1.13	± 0.12
(Z)-15-Tetracosenoic Acid (C24:1 n-9; Nervonic Acid)	5.30	± 0.43

^(a) The reference mass fraction value is the mean of results obtained using one analytical technique, GC-FID. The expanded uncertainty, U , is calculated as $= ku_c$, 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 19 degrees of freedom and a 95 % confidence level for each analyte.

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Certificate Revision History: 04 August 2015 (Update of expiration date due to instability; editorial changes); 21 November 2011 (Original certificate date).

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>.