



Certificate of Analysis

Standard Reference Material[®] 1588b

Organics in Cod Liver Oil

This Standard Reference Material (SRM) is intended 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 cod liver oil or in similar matrices. SRM 1588b is the same material as SRM 1588, which was originally certified in 1989 for the concentrations of 10 chlorinated pesticides, 5 PCB congeners, and α -tocopherol [1], and SRM 1588a, which was originally certified in 1998 for the concentrations of 14 chlorinated pesticides and 24 PCB congeners and also had reference values for α -tocopherol, 34 PCB congeners, and 3 chlorinated pesticides [2]. SRM 1588b updates the certified values and provides certified and reference values for additional analytes, including fatty acids, PBDE congeners, and toxaphene congeners. SRM 1588b consists of four sealed ampoules per unit, each ampoule containing approximately 1.2 mL of cod liver oil.

Certified Concentration Values: Certified values, expressed as mass fractions [3], for 27 PCB congeners, 15 chlorinated pesticides, and 14 fatty acids found in the cod liver oil are provided in Tables 1 through 3, respectively. The certified values for PCBs and pesticides are based on the agreement of results obtained from two or more independent analytical techniques. The certified values for fatty acids are based on the agreement of results obtained from two different analytical techniques at NIST and results provided in two interlaboratory comparison exercises organized by the Food Products Association's (FPA's) Food Industry Analytical Chemists Subcommittee (FIACS).

Reference Concentration Values: Reference values, also expressed as mass fractions, for α -tocopherol, 47 PCB congeners, 3 chlorinated pesticides, 6 individual fatty acids, 6 PBDE congeners, and 3 toxaphene congeners are provided in Tables 4 through 7. The reference values are noncertified values that do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple methods.

Information Concentration Values: Information concentration values expressed as mass fractions for six polychlorodibenzo-*p*-dioxins and octachlorodibenzofuran added to the cod liver oil are provided in Table 8. These are noncertified values with no uncertainties reported as there is insufficient information to assess the uncertainty.

Expiration of Value Assignment: The certification of this SRM lot is valid until **31 May 2012**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). 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 value assignment before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the recertification was under the direction of M.M. Schantz, K.E. Sharpless, and S.A. Wise of the NIST Analytical Chemistry Division.

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Certificate Issue Date: 09 August 2007
See Certificate Revision History on Page 12

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Preparation and analyses for the original certificate for SRM 1588 were performed by J.M. Brown Thomas, F.R. Guenther, and R.M. Parris of the NIST Analytical Chemistry Division. The analyses for SRM 1588b were performed by W.W. Brubaker, Jr., J.M. Keller, J.R. Kucklick, C.S. Phinney, M.M. Schantz, K.E. Sharpless, H.M. Stapleton, and J.E. Yordy of the NIST Analytical Chemistry Division and by K. Koczanski and B. Grift of the Department of Fisheries and Oceans (DFO) Canada, Freshwater Institute, Winnipeg, Manitoba and D.C.G. Muir of Environment Canada, Burlington, Ontario.

Consultation on the statistical design of the experimental work and evaluation of the data were provided by L.M. Gill, S.D. Leigh, S.B. Schiller, and J.H. Yen of the NIST Statistical Engineering Division.

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

NOTICE AND WARNING TO USERS

CAUTION: This material contains added polychlorodibenzo-*p*-dioxins and octachlorodibenzofuran. Each is present in this material at a concentration of 1 ng/g or less. Although some of these compounds are reported to be toxic and/or carcinogenic, their concentrations are too low to be considered hazardous under OSHA regulations. However, the material should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures less than 30 °C.

INSTRUCTIONS FOR USE

Samples 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. Certified values are not applicable to material in ampoules stored after opening, even if they are resealed.

PREPARATION AND ANALYSIS¹

Preparation: This SRM is a commercial cod liver oil that has been fortified with selected chlorinated dibenzodioxins and dibenzofurans. It was donated to NIST by the University of Ulm (Germany).

Weighed aliquots of gravimetrically prepared 2,2,4-trimethylpentane solutions of six polychlorinated dibenzo-*p*-dioxins and octachlorodibenzofuran were added to a known mass of the cod liver oil. The concentrations of these components, which are included here for information only and are not certified values, were calculated from these gravimetric measurements and are listed in Table 8. The oil was homogenized by stirring for 10 h in a 9-L glass bottle. Each 2-mL amber ampoule was purged with argon just prior to the addition of approximately 1.2 mL of oil to the ampoule, which was then flame sealed.

PCBs and Chlorinated Pesticides: For the certification of SRM 1588a, the cod liver oil was analyzed for selected PCB congeners and chlorinated pesticides using gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity and using GC with mass spectrometric detection (GC/MS). These methods are described in more detail in the certificate for SRM 1588a [2]. This same approach had been used previously for the certification of PCBs and chlorinated pesticides in other environmental matrices [4–6].

¹ Certain commercial equipment, instruments, or materials are identified in this report 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.

For the certification of SRM 1588b, the data from the certification of SRM 1588a along with data from three methods employing GC-ECD analysis (GC-ECD I, II, and III), and from three methods employing GC/MS analysis (GC/MS I, II, and III) were used. For GC-ECD I and II, approximately 1 g samples of SRM 1588b (exact mass known) were extracted using pressurized fluid extraction (PFE) with methylene chloride as the extraction solvent. Following concentration of the extract, the high relative molecular mass compounds were removed using size exclusion chromatography (SEC) on a preparative divinylbenzene-polystyrene column followed by fractionation using 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, J&W Scientific, Folsom, CA) and a 0.25 mm × 60 m fused silica capillary column with a relatively non-polar propriety phase (0.25 µm film thickness) (DB-XLB, J&W Scientific). 4,4'-DDT-*d*₈, 4,4'-DDE-*d*₈, 4,4'-DDD-*d*₈, endosulfan I-*d*₄, PCB 103, and PCB 198 were used as the internal standards. PCB 103 and PCB 198 [7,8] are not significantly present in the native cod liver oil.

For GC-ECD III and GC/MS I, approximately 0.5 g samples of SRM 1588b (exact mass known) were extracted using PFE with methylene chloride. Following concentration, the extracts were eluted through two successive silica solid-phase extraction (SPE) columns using 15 mL of 10 % (volume fraction) methylene chloride in hexane as the eluant. GC-ECD analysis was performed using a 0.25 mm × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB). The GC/MS analysis was performed using a 0.25 mm × 60 m fused silica capillary column with a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17MS, J&W Scientific). 4,4'-DDT-*d*₈, 4,4'-DDD-*d*₈, PCB 103, and PCB 198 were used as the internal standards.

For GC/MS II and GC/MS III, approximately 1 g samples of SRM 1588b (exact mass known) were extracted using PFE with methylene chloride. Following concentration, the high molecular weight compounds were removed using SEC followed by the removal of polar interferences using a 5 % deactivated alumina SPE column. The GC/MS analysis was performed using a 0.25 mm × 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5MS). 4,4'-DDT-*d*₈, 4,4'-DDE-*d*₈, 4,4'-DDD-*d*₈, endosulfan I-*d*₄, ¹³C-labeled PCB 28, 52, 118, 180, 194, and 206; ¹³C-labeled PBDEs 99 and 209, and ¹³C-labeled *trans*-chlordane were used as the internal standards.

Non-ortho-Substituted PCBs: Two sets of results for non-*ortho*-substituted PCBs (NOPCBs) (PCB 77, PCB 126, and PCB169) were obtained by using GC/MS after LC isolation of the NOPCB fraction [9]. Between 0.5 g and 1 g subsamples from six ampoules of SRM 1588b were mixed with sodium sulfate and extracted using PFE with methylene chloride. The extracts were subjected to SEC to remove lipids followed by normal-phase LC on a semi-preparative aminopropylsilane column with hexane as the mobile phase to isolate the PCB fraction. The PCB fraction was then separated into an *ortho*-substituted PCB fraction and a NOPCB fraction using a 2-(pyrenyl)ethyltrimethylsilylated silica (PYE) column (4.6 mm i.d. × 25 cm, 5 µm Comosil-PYE, Nacalai Tesque, Kyoto, Japan) with hexane as the mobile phase. The NOPCB fraction was then analyzed by GC/MS using negative chemical ionization (GC/NCI-MS) on a 0.25 mm i.d. × 30 m fused silica capillary column containing a 5 % (mole fraction) diphenyl dimethylpolysiloxane phase (HP-5, 0.25 µm film thickness, Agilent Technologies, Wilmington, DE). The same samples were also analyzed by GC with high resolution MS with electron impact ionization on a 0.25 mm i.d. × 30 m fused silica capillary column containing a 5 % phenyl methylpolysiloxane phase (DB-5MS, 0.25 µm film thickness). ¹³C-labeled PCBs 77, 126, and 169 were used as the internal standards.

Polybrominated Diphenyl Ethers: Value assignment of concentrations of PBDE congeners was based on three sets of measurements using 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 measurement sets, 1 g subsamples of cod liver oil from each of six ampoules were 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/MS using electron impact ionization (GC/EI-MS) and GC/NCI-MS on a 0.25 mm × 15 m fused silica capillary column with a 5 % phenyl methylpolysiloxane phase (0.25 µm film thickness) (DB-5). For both methods, ¹³C-labeled 4,4'-dibromodiphenyl ether (BDE 15) and ¹³C-labeled 2,2',3,4,5-pentachlorodiphenyl ether (CDE 86) were added to the oil sample prior to SEC for use as internal standards for quantification purposes.

For the third measurement set, 1 g subsamples of cod liver oil from each of six ampoules were extracted using pressurized fluid extraction and 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). Replicates were spiked prior to SEC with ¹³C-labeled 2,2,4,4',5-pentabromodiphenyl ether (BDE 99) for use as an internal standard for quantification purposes.

Toxaphene: Value assignment of concentrations for toxaphene congeners is based on one method [10]. SRM 1588a was also used in an interlaboratory study described by Andrews et al. [11]. The method used for the value assignment was the same as for GC-ECD I and II above except that the fractions from the aminopropylsilane column were recombined prior to analysis by 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-5MS).

Fatty Acids: The concentrations of individual fatty acids were determined at NIST using two GC methods and as part of two FPA FIACS interlaboratory comparison exercises involving the laboratories listed in Appendix A. At NIST, fatty acids were measured in two analyses: one in which duplicate test portions from five ampoules were analyzed and one in which single test portions from three ampoules of material were analyzed. Methyl nonadecanoate (C19:0 fatty acid methyl ester [FAME]) was used as an internal standard. A two-step process employing methanolic sodium hydroxide and boron trifluoride was used to convert the fatty acids to their methyl esters. FAMES were extracted into hexane and analyzed by gas chromatography with flame ionization detection (GC/FID). In the first set of analyses, an SP-2340 100 % poly biscyanopropylsiloxane column, 0.32 mm i.d. × 30 m (Supelco, Bellefonte, PA) was used while in the second set of analyses, an SP-2560 90 % biscyanopropyl/10 % cyanopropylphenyl siloxane column, 0.25 mm i.d. × 100 m × 0.20 µm film (Supelco), was used.

α-Tocopherol: The concentration of α-tocopherol was determined using two methods based on normal-phase (NP) and reversed-phase (RP) liquid chromatography (LC). Fluorescence detection ($\lambda_{ex} = 295$ nm; $\lambda_{em} = 335$ nm) was used for both methods. The NPLC analyses employed a semi-preparative aminopropylsilane column with quantification by the external standard method; aliquots of cod liver oil were diluted in hexane. The RPLC analyses employed a C₁₈ column; the cod liver oil was diluted in ethyl acetate and ethanol and was spiked with tocol as an internal standard.

Table 1. Certified Concentration Values for Selected PCB Congeners in SRM 1588b^(a)

PCB Congener	Mass Fraction ($\mu\text{g}/\text{kg}$)
PCB 18 (2,2',5-Trichlorobiphenyl) ^(b,c,d,e,f)	7.96 \pm 0.45 ^(g)
PCB 28 (2,4,4'-Trichlorobiphenyl) ^(b,c,d)	27.8 \pm 1.4 ^(h)
PCB 31 (2,4',5-Trichlorobiphenyl) ^(b,c,d)	8.52 \pm 0.50 ^(h)
PCB 44 (2,2',3,5'-Tetrachlorobiphenyl) ^(b,c,d,e,f)	32.4 \pm 2.7 ^(g)
PCB 49 (2,2',4,5'-Tetrachlorobiphenyl) ^(b,c,d,e,f)	29.8 \pm 0.6 ^(g)
PCB 52 (2,2',5,5'-Tetrachlorobiphenyl) ^(b,c,d,e,f)	82.4 \pm 1.7 ^(g)
PCB 66 (2,3',4,4'-Tetrachlorobiphenyl) ^(b,c,d,e,f)	51.7 \pm 7.5 ^(g)
PCB 87 (2,2',3,4,5'-Pentachlorobiphenyl) ^(b,c,d,e)	54.6 \pm 2.1 ^(g)
PCB 95 (2,2',3,5',6-Pentachlorobiphenyl) ^(b,c,d,e,f)	37.7 \pm 8.2 ^(g)
PCB 99 (2,2',4,4',5-Pentachlorobiphenyl) ^(c,e,f)	101 \pm 12 ^(h)
PCB 101 (2,2',4,5,5'-Pentachlorobiphenyl) ^(b,c,d,e,f)	127 \pm 9 ^(g)
PCB 105 (2,3,3',4,4'-Pentachlorobiphenyl) ^(b,c,d,e,f)	59.2 \pm 1.2 ^(g)
PCB 110 (2,3,3',4',6-Pentachlorobiphenyl) ^(b,c,d,e,f)	69.3 \pm 4.8 ^(g)
PCB 118 (2,3',4,4',5-Pentachlorobiphenyl) ^(b,c,d,e,f)	172 \pm 7 ^(g)
PCB 128 (2,2',3,3',4,4'-Hexachlorobiphenyl) ^(b,c,d,e,f)	39.9 \pm 6.0 ^(g)
PCB 138 (2,2',3,4,4',5'-Hexachlorobiphenyl) ^(c,d,e)	212 \pm 29 ^(h)
PCB 149 (2,2',3,4',5',6-Hexachlorobiphenyl) ^(b,c,d,e,f)	90 \pm 11 ^(g)
PCB 151 (2,2',3,5,5',6-Hexachlorobiphenyl) ^(b,c,d,e,f)	48.1 \pm 5.4 ^(g)
PCB 153 (2,2',4,4',5,5'-Hexachlorobiphenyl) ^(b,c,d,f)	275 \pm 4 ^(g)
PCB 156 (2,3,3',4,4',5-Hexachlorobiphenyl) ^(c,e,f)	18.0 \pm 2.1 ^(h)
PCB 170 (2,2',3,3',4,4',5-Heptachlorobiphenyl) ^(b,c,d,e,f)	41.9 \pm 3.8 ^(g)
PCB 180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl) ^(b,c,d,e,f)	98.5 \pm 6.3 ^(g)
PCB 183 (2,2',3,4,4',5',6-Heptachlorobiphenyl) ^(b,c,d,e,f)	28.9 \pm 1.8 ^(g)
PCB 187 (2,2',3,4',5,5',6-Heptachlorobiphenyl) ^(b,c,d,e,f)	43.6 \pm 7.2 ^(g)
PCB 194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl) ^(b,c,d,e,f)	13.5 \pm 1.2 ^(g)
PCB 206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) ^(b,c,d,e,f)	3.75 \pm 0.07 ^(g)
PCB 209 (2,2',3,3',4,4',5,5',6,6'-Decachlorobiphenyl) ^(b,c,e,f)	3.21 \pm 0.26 ^(g)

^(a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [7] and later revised by Schulte and Malisch [8] to conform with IUPAC rules.

^(b) GC-ECD on two columns and GC/MS analysis as described in the Certificate of Analysis for SRM 1588a [2].

^(c) GC-ECD I and GC-ECD II on a 5 % phenyl phase and a relatively non-polar proprietary phase.

^(d) GC-ECD III and GC/MS I on a relatively non-polar proprietary phase and a 50 % phenyl phase.

^(e) GC/MS II on a 5 % phenyl phase.

^(f) GC/MS III on a 5 % phenyl phase.

^(g) The certified value is a weighted mean of the results from four or five analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), except for PCB 153 for which a coverage factor of 4 was used, calculated by combining a between-method variance [12] incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [13].

^(h) The certified value is an unweighted mean of the results from three 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 [14] with a pooled, within-method variance following the ISO and NIST Guides [13].

Table 2. Certified Concentration Values for Selected Chlorinated Pesticides in SRM 1588b

Chlorinated Pesticide	Mass Fraction ($\mu\text{g}/\text{kg}$)
4,4'-DDE ^(a,b,c,d)	676 \pm 36 ^(e)
2,4'-DDD ^(a,b,c,d)	34.8 \pm 1.6 ^(e)
4,4'-DDD ^(a,b)	285 \pm 37 ^(f)
2,4'-DDT ^(a,b)	162 \pm 8 ^(f)
4,4'-DDT ^(a,b,c,d)	570 \pm 27 ^(e)
Hexachlorobenzene ^(a,b,c,d)	163 \pm 16 ^(e)
γ -hexachlorohexane (HCH) ^(a,b,c)	23.3 \pm 1.7 ^(f)
α -HCH ^(a,b,c,d)	99 \pm 15 ^(e)
Heptachlor epoxide ^(a,b)	30.0 \pm 1.9 ^(f)
<i>cis</i> -Chlordane ^(a,b)	186 \pm 22 ^(f)
<i>cis</i> -Nonachlor ^(a,b)	92.4 \pm 3.0 ^(f)
<i>trans</i> -Nonachlor ^(a,b)	222 \pm 10 ^(f)
Dieldrin ^(a,b,c,d)	156 \pm 4 ^(e)
Oxychlordane ^(a,b,c,d)	37.5 \pm 4.5 ^(e)
Mirex ^(b,c,d)	11.8 \pm 0.6 ^(f)

^(a) GC-ECD on two columns and GC/MS analysis as described in the Certificate of Analysis for SRM 1588a [2].

^(b) GC-ECD I and GC-ECD II on a 5 % phenyl phase and a relatively non-polar proprietary phase.

^(c) GC/MS II on a 5 % phenyl phase.

^(d) GC/MS III on a 5 % phenyl phase.

^(e) The certified value is a weighted mean of the results from 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 [12] incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [13].

^(f) The certified value is an unweighted mean of the results from two or three 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 [14] with a pooled, within-method variance following the ISO and NIST Guides [13].

Table 3. Certified Concentration Values for Selected Fatty Acids (as the Triglycerides) in SRM 1588b^(a)

Fatty Acid	Mass Fraction (%)
Tetradecanoic Acid (C14:0) (Myristic Acid)	3.35 ± 0.27
Pentadecanoic Acid (C15:0)	0.288 ± 0.021
Hexadecanoic Acid (C16:0) (Palmitic Acid)	8.51 ± 0.97
(Z)-9-Hexadecenoic Acid (C16:1 n-7) (Palmitoleic Acid)	6.80 ± 0.91
Octadecanoic Acid (C18:0) (Stearic Acid)	1.82 ± 0.20
(Z)-9-Octadecenoic Acid (C18:1 n-9) (Oleic Acid)	13.3 ± 1.7
(Z)-11-Octadecenoic Acid (C18:1 n-7) (Vaccenic Acid)	4.34 ± 0.60
(Z,Z)-9,12-Octadecadienoic Acid (C18:2 n-6) (Linoleic Acid)	2.00 ± 0.27
(Z,Z,Z)-6,9,12-Octadecatrienoic Acid (C18:3 n-6) (γ -Linolenic Acid)	0.138 ± 0.019
(Z)-11-Eicosenoic Acid (C20:1 n-9) (Gondoic Acid)	9.5 ± 1.7
Eicosatrienoic Acid (C20:3)	0.137 ± 0.020
Eicosapentaenoic Acid (C20:5)	9.04 ± 0.82
Docosapentaenoic Acid (C22:5)	1.21 ± 0.14
Docosahexaenoic Acid (C22:6)	13.6 ± 1.5
(Z)-Tetracosenoic Acid (C24:1 n-9) (Nervonic Acid)	0.379 ± 0.047

^(a) Each certified concentration value is the weighted mean of results provided by the laboratories listed in Appendix A and NIST. The FPA result is the median of the individual lab means for that analyte, and the uncertainty associated with each such analyte estimate is $(1.2533 * IQR / (2 * 0.67449)) / \sqrt{N}$ where N is the number of lab means and IQR is the interquartile range of the set of lab means for that analyte [15]. Results from the two NIST analyses were the means of the observations for a given analyte; the uncertainty of that estimate is the standard deviation of the mean. Assigned values were calculated using the BOB method [14]. The uncertainty in the certified values, calculated according to the method described in the ISO and NIST Guides [13], is expressed as an expanded uncertainty, U . The expanded uncertainty is calculated as $U = k u_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, k , is determined from the Student's t -distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte.

Table 4. Reference Concentration Values for Additional PCB Congeners and Chlorinated Pesticides in SRM 1588b^(a)

PCB Congeners and Chlorinated Pesticides		Mass Fraction ($\mu\text{g}/\text{kg}$)		
PCB 16	(2,2',3-Trichlorobiphenyl) ^(b)	2.6	±	0.8
32	(2,4',6-Trichlorobiphenyl)			
PCB 17	(2,2',4-Trichlorobiphenyl) ^(b)	6.5	±	1.1
PCB 22	(2,3,4'-Trichlorobiphenyl) ^(b)	3.0	±	0.6
PCB 33	(2',3,4-Trichlorobiphenyl) ^(b)	3.3	±	1.4
PCB 41	(2,2',3,4-Tetrachlorobiphenyl) ^(b)	5.3	±	2.2
71	(2,3',4',6-Tetrachlorobiphenyl)			
PCB 42	(2,2',3,4'-Tetrachlorobiphenyl) ^(b)	14	±	3
PCB 45	(2,2',3,6-Tetrachlorobiphenyl) ^(c,d)	2.17	±	0.29 ^(e)
PCB 46	(2,2',3,6'-Tetrachlorobiphenyl) ^(b)	11	±	2
PCB 55	(2,3,3',4-Tetrachlorobiphenyl) ^(b)	18	±	11
60	(2,3,4,4'-Tetrachlorobiphenyl)			
PCB 63	(2,3,4',5-Tetrachlorobiphenyl) ^(c,d)	2.97	±	0.54 ^(e)
PCB 64	(2,3,4',6-Tetrachlorobiphenyl) ^(b)	14	±	1
PCB 70	(2,3',4',5-Tetrachlorobiphenyl) ^(c,f)	15.9	±	0.7 ^(e)
PCB 74	(2,4,4',5-Tetrachlorobiphenyl) ^(c,d,f,g)	40	±	11 ^(h)
PCB 77	(3,3',4,4'-Tetrachlorobiphenyl) ⁽ⁱ⁾	1.31	±	0.02 ^(e)
PCB 82	(2,2',3,3',4-Pentachlorobiphenyl) ^(c,d,g)	8.2	±	1.6 ^(e)
PCB 83	(2,2',3,3',5-Pentachlorobiphenyl) ^(b)	21	±	5
PCB 85	(2,2',3,4,4'-Pentachlorobiphenyl) ^(b)	27	±	8
PCB 91	(2,2',3,4',6-Pentachlorobiphenyl) ^(b)	21	±	8
PCB 92	(2,2',3,5,5'-Pentachlorobiphenyl) ^(c,d,g)	36.2	±	8.2 ^(e)
PCB 97	(2,2',3',4,5-Pentachlorobiphenyl) ^(b)	42	±	4
PCB 107	(2,3,3',4',5-Pentachlorobiphenyl) ^(c,d,g)	16.2	±	3.9 ^(e)
PCB 114	(2,3,4,4',5-Pentachlorobiphenyl) ^(c,d)	3.79	±	0.55 ^(e)
PCB 126	(3,3',4,4',5-Pentachlorobiphenyl) ⁽ⁱ⁾	1.31	±	0.02 ^(e)
PCB 130	(2,2',3,3',4,5'-Hexachlorobiphenyl) ^(c,d)	7.35	±	0.27 ^(e)
PCB 136	(2,2',3,3',6,6'-Hexachlorobiphenyl) ^(b)	14	±	2
PCB 137	(2,2',3,4,4',5-Hexachlorobiphenyl) ^(c,d)	11.8	±	0.8 ^(e)
PCB 141	(2,2',3,4,5,5'-Hexachlorobiphenyl) ^(b)	24	±	4
PCB 146	(2,2',3,4',5,5'-Hexachlorobiphenyl) ^(c,d,f,g)	47	±	14 ^(h)
PCB 154	(2,2',4,4',5,6'-Hexachlorobiphenyl) ^(c,d)	3.48	±	0.29 ^(e)
PCB 157	(2,3,3',4,4',5'-Hexachlorobiphenyl) ^(c,d,f)	4.3	±	1.2 ^(e)
PCB 158	(2,3,3',4,4',6-Hexachlorobiphenyl) ^(c,f,g)	11.3	±	1.8 ^(e)
PCB 163	(2,3,3',4',5,6-Hexachlorobiphenyl) ^(c,g)	42.2	±	3.2 ^(e)
PCB 167	(2,3',4,4',5,5'-Hexachlorobiphenyl) ^(c,d)	12.2	±	0.8 ^(e)
PCB 169	(3,3',4,4',5,5'-Hexachlorobiphenyl) ⁽ⁱ⁾	0.22	±	0.01 ^(e)
PCB 172	(2,2',3,3',4,5,5'-Heptachlorobiphenyl) ^(c,d)	8.39	±	0.33 ^(e)
PCB 174	(2,2',3,3',4,5,6'-Heptachlorobiphenyl) ^(c,d,g)	15.0	±	2.8 ^(e)
PCB 177	(2,2',3,3',4',5,6-Heptachlorobiphenyl) ^(c,d)	5.18	±	0.36 ^(e)
PCB 178	(2,2',3,3',5,5',6-Heptachlorobiphenyl) ^(b)	28	±	1
129	(2,2',3,3',4,5-Hexachlorobiphenyl)			
PCB 179	(2,2',3,3',5,6,6'-Heptachlorobiphenyl) ^(b)	4.4	±	1.2
PCB 185	(2,2',3,4,5,5',6-Heptachlorobiphenyl) ^(c,d)	2.79	±	0.25 ^(e)
PCB 189	(2,3,3',4,4',5,5'-Heptachlorobiphenyl) ^(c,d)	1.83	±	0.07 ^(e)
PCB 191	(2,3,3',4,4',5,6-Heptachlorobiphenyl) ^(b)	4.5	±	0.7
PCB 193	(2,3,3',4',5,5',6-Heptachlorobiphenyl) ^(b)	14	±	3
PCB 195	(2,2',3,3',4,4',5,6-Octachlorobiphenyl) ^(b)	4.6	±	0.6

PCB 196	(2,2',3,3',4,4',5',6-Octachlorobiphenyl) ^(c,d)	17.1	±	0.7 ^(e)
203	(2,2',3,4,4',5,5',6-Octachlorobiphenyl)			
PCB 199	(2,2',3,3',4,5,6,6'-Octachlorobiphenyl) ^(c,d)	13.3	±	0.2 ^(e)
PCB 207	(2,2',3,3',4,4',5,6,6'-Nonachlorobiphenyl) ^(c,d)	1.53	±	0.06 ^(e)
2,4'-DDE ^(c,d,g,j)		15.6	±	5.5 ^(h)
Octachlorostyrene ^(c,d)		9.14	±	0.74 ^(e)
Pentachlorobenzene ^(c,d)		16.1	±	0.6 ^(e)

- (a) PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [7] and later revised by Schulte and Malisch [8] to conform with IUPAC rules. When two or more congeners are known to coelute under the 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) Concentrations are based on seven analyses done at the Department of Fisheries and Oceans (Winnipeg, Manitoba, Canada) over a period of two years as described in more detail in the Certificate of Analysis for SRM 1588a [2]. Uncertainties are expressed as 95 % confidence intervals of the mean. These concentrations are provided as reference values because the results have not been confirmed by an independent analytical technique as required for certification. The associated uncertainties may reflect only measurement precision and may not include all sources of uncertainty.
- (c) GC/MS II on a 5 % phenyl phase.
- (d) GC/MS III on a 5 % phenyl phase.
- (e) The reference value is an unweighted mean of the results from two or three 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 [14] with a pooled, within-method variance following the ISO and NIST Guides [13].
- (f) GC-ECD III and GC/MS I on a relatively non-polar proprietary phase and a 50 % phenyl phase.
- (g) GC-ECD I and GC-ECD II on a 5 % phenyl phase and a relatively non-polar proprietary phase.
- (h) The reference value is a weighted mean of the results from 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 [12] incorporating inter-method bias with a pooled, within-method variance following the ISO and NIST Guides [13].
- (i) The methods used for PCBs 77, 126, and 169 are described under the non-*ortho*-substituted PCBs section heading.
- (j) GC-ECD on two columns and GC/MS analysis as described in the Certificate of Analysis for SRM 1588a [2].

Table 5. Reference Concentration Values for Selected Fatty Acids (as Triglycerides) in SRM 1588b^(a)

Fatty Acids	Mass Fraction (%)
(Z)-9-Tetradecenoic Acid (C14:1) (Myristoleic Acid)	0.101 ± 0.021
(Z,Z,Z)-9,12,15-Octadecatrienoic Acid (C18:3 n-3) (Linolenic Acid)	1.09 ± 0.26
Eicosanoic Acid (C20:0) (Arachidic Acid)	0.057 ± 0.019
Eicosadienoic Acid (C20:2) (Z,Z,Z,Z)-5,8,11,14-Eicosatetraenoic Acid (C20:4 n-6) (Arachidonic Acid)	0.341 ± 0.075 0.415 ± 0.092

- (a) Each reference concentration value is the weighted mean of results provided by the laboratories listed in Appendix A and NIST. The FPA result is the median of the individual lab means for that analyte, and the uncertainty associated with each such analyte estimate is $(1.2533 * IQR / (2 * 0.67449)) / \sqrt{N}$ where N is the number of lab means and IQR is the interquartile range of the set of lab means for that analyte [15]. Results from the two NIST analyses were the means of the observations for a given analyte; the uncertainty of that estimate is the standard deviation of the mean. Assigned values were calculated using the BOB method [14]. The uncertainty in the certified values, calculated according to the method described in the ISO and NIST Guides [13], is expressed as an expanded uncertainty, U . The expanded uncertainty is calculated as $U = k u_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, k , is determined from the Student's t -distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte.

Table 6. Reference Concentration Value for α -Tocopherol in SRM 1588b^(a)

Compound	Mass Fraction (mg/kg)
α -Tocopherol	134.2 \pm 2.5

^(a) The uncertainty is based on 95 % confidence interval for the mean. It incorporates variability within and between the measurement techniques used. This reference value should be useful for comparison with results obtained using similar procedures (see Preparation and Analysis, α -Tocopherol).

Table 7. Reference Concentrations for Selected PBDE and Toxaphene Congeners in SRM 1588b

PBDE Congener ^(a,b)		Mass Fraction (μ g/kg)
BDE 28	(2,4,4'-Tribromodiphenylether) ^(c,d,e)	1.08 \pm 0.23
BDE 33	(2',3,4-Tribromodiphenylether)	
BDE 47	(2,2',4,4'-Tetrabromodiphenylether) ^(c,d,e)	17.8 \pm 2.0
BDE 49	(2,2',4,5'-Tetrabromodiphenylether) ^(c,d,e)	2.25 \pm 0.24
BDE 99	(2,2',4,4',5-Pentabromodiphenylether) ^(c,d,e)	0.56 \pm 0.20
BDE 100	(2,2',4,4',6-Pentabromodiphenylether) ^(c,d,e)	1.89 \pm 0.45
BDE 154	(2,2',4,4',5,6'-Hexabromodiphenylether) ^(d,e)	0.495 \pm 0.069
Toxaphene Congener ^(f)		
TOX 26	(2-endo, 3-exo, 5-endo, 6-exo, 8,8,10,10-octachlorobornane)	154 \pm 18
TOX 62	(2,25,5,8,9,9,10,10-nonachlorobornane)	106 \pm 13

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

^(b) Reference values are unweighted means of the results from two or more 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 [14] with a pooled, within-method variance following the ISO and NIST Guides [13].

^(c) GC/NCI-MS on a 15 m column containing a 5 % phenyl phase.

^(d) GC/EI-MS (I) on a 15 m column containing a 5 % phenyl phase.

^(e) GC/EI-MS (II) on a 60 m column containing a 5 % phenyl phase.

^(f) Methods described in more detail in reference [10]. 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 8. Information Concentration Values for Dibenzo-*p*-dioxins and Octachlorodibenzofuran in SRM 1588b^(a)

Dibenzo- <i>p</i> -dioxins	Mass Fraction (µg/kg)
1,2,7-Trichlorodibenzo- <i>p</i> -dioxin	0.32
1,2,3,4-Tetrachlorodibenzo- <i>p</i> -dioxin	0.38
2,3,7,8-Tetrachlorodibenzo- <i>p</i> -dioxin	0.21
1,2,3,6,7,8-Hexachlorodibenzo- <i>p</i> -dioxin	0.39
1,2,3,7,8,9-Hexachlorodibenzo- <i>p</i> -dioxin	0.22
Octachlorodibenzo- <i>p</i> -dioxin	1.01
Octachlorodibenzofuran	1.00

^(a) These concentrations are provided as information values because the results, which are based only on gravimetry, have not been confirmed by an independent analytical technique as required for certification. These values are provided for information only.

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Certificate Revision History: 09 August 2007 (Editorial changes); 07 February 2006 (Original certificate date).
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APPENDIX A

The laboratories listed below performed measurements that contributed to the value assignment of fatty acids in SRM 1588b Organics in Cod Liver Oil.

Campbell Soup Company (Camden, NJ, USA)
Cargill, Inc. (Wayzata, MN, USA)
Covance, Inc. (Madison, WI, USA)
Eurofins Scientific (Memphis, TN, USA)
General Mills, Inc. (Golden Valley, MN, USA)
Hormel Foods Corporation (Austin, MN, USA)
Kraft Foods, Inc. (Glenview, IL, USA) (Contracted analyses to Silliker Laboratories)
Krueger Food Laboratories, Inc. (Cambridge, MA, USA)
Kraft Foods, Inc. (East Hanover, NJ, USA)
National Laboratories of Foods and Drugs (Taipei, Taiwan)
Nestlé Food Corporation (Dublin, OH, USA)
Nestlé Purina Company (St. Louis, MO, USA)
Novartis Nutrition Technical Center (St. Louis Park, MN, USA)