National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2585

Organic Contaminants in House Dust

This Standard Reference Material (SRM) is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, chlorinated pesticides, polybrominated diphenyl ether (PBDE) congeners, synthetic musks, perfluorinated alkyl acids (PFAAs), hexabromocyclododecane (HBCDD) isomers, and phosphorous flame retardants in house dust and similar matrices. All of the constituents for which certified, reference, and information values are provided in SRM 2585 were naturally present in the dust material before processing. A unit of SRM 2585 consists of one bottle containing approximately 10 g of house dust.

Certified Mass Fraction Values: The certified values for selected PAHs, PCB congeners, chlorinated pesticides, and PBDE congeners in SRM 2585 are provided in Tables 1 through 4. A NIST certified value is a value for which NIST has the highest confidence in its accuracy and that all known or suspected sources of bias have been investigated or taken into account [1]. The measurands are the total mass fraction for each analyte on a dry-mass basis and are metrologically traceable to the International System of Units (SI). The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques while the certified values for the PBDE congeners are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques and results from a collaborating laboratory [1,2].

Expiration of Certification: This certification of **SRM 2585** is valid, within the measurement uncertainty specified, until **01 June 2025**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handing, 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.

Overall coordination of the technical measurements leading to the certification of SRM 2585 was provided by J.L Reiner, M.M. Schantz, and S.A. Wise of the NIST Chemical Sciences Division.

Analytical measurements for the certification of SRM 2585 were performed at NIST by J.R. Kucklick, J.M. Lynch, J.A. Murray, M.S. Nocun, A.M. Peck, B.J. Porter, D.L. Poster, J.L. Reiner, M.M. Schantz, S.S. Schuur, and H.M. Stapleton of the NIST Chemical Sciences Division. Additional measurements for selected PBDE congeners were performed at the Meteorological Service of Canada, Environment Canada (Toronto, Canada) by T. Harner and M. Shoeib.

Statistical consultation was provided by N.A. Heckert and S.D. Leigh 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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Table 1. Certified Mass Fraction for Selected PAHs in SRM 2585

	Mass Fraction (dry-mass basis)[a] (µg/kg)		
Naphthalene[d,e,f]	266	±	8[b]
Dibenzothiophene[d,e,f,g]	109	±	8[c]
Phenanthrene[d,e,f,g,h,i]	1920	±	20[c]
Anthracene[d,e,f,g,h,i]	96.0	±	5.2[c]
4H-cyclopenta[def]phenanthrene[d,e,f,g]	117	±	10[c]
3-Methylphenanthrene[e,f,g,i]	293	±	36[c]
2-Methylphenathrene[e,f,g]	352	±	40[b]
9-Methylphenanthrene[e,f,g,i]	205	±	16[c]
1-Methylphenanthrene[e,f,g]	197	±	29[b]
Fluoranthene[d,e,f,g,h,i]	4380	±	100[c]
Pyrene[d,e,f,g,i]	3290	±	30[c]
Benzo[ghi]fluoranthene[d,e,f,g,h,i]	317	±	11[c]
Benzo[c]phenanthrene[d,e,f,g,i]	288	±	10[c]
Benz[a]anthracene[d,e,f,g,h,i]	1160	±	54[c]
Chrysene[e,h,i]	2260	±	60[b]
Triphenylene[e,h,i]	589	±	17[b]
Benzo[b]fluoranthene[d,f,g,h,i,j]	2700	±	90[c]
Benzo[j]fluoranthene[d,f,g,j]	1320	±	110[c]
Benzo[k]fluoranthene[d,f,g,h,j]	1330	±	70[c]
Benzo[a]fluoranthene[d,f,g,j]	74.5	±	8.1[c]
Benzo[e]pyrene[d,f,g,h,i,j]	2160	±	80[c]
Benzo[a]pyrene[d,f,g,h,i,j]	1140	±	10[c]
Perylene[d,f,g,h,i,j]	387	±	23[c]
Benzo[ghi]perylene[d,f,g,h,i,j]	2280	±	40[c]
Indeno[1,2,3-cd]pyrene[d,f,g,h,i,j]	2080	±	100[c]
Dibenz[a,j]anthracene[d,f,g,h,i,j]	267	±	9[c]
Dibenz[a,c]anthracene[d,f,g,h,i,j]	183	±	25[c]
Dibenz[a,h]anthracene[d,f,g,h,i,j]	301	±	50[c]
Benzo[b]chrysene[d,f,g,h,i]	182	±	6[c]
Picene[d,f,g,h,j]	413	±	15[c]
Coronene[d,f,g,h,i,j]	603	±	38[c]
Dibenzo[b,k]fluoranthene[d,f,g,h,i,j]	596	±	22[c]
Dibenzo[a,e]pyrene[d,f,g,h,i,j]	477	±	67[c]

[a] Mass fraction reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

[b] Certified values are unweighted means of the results from three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO/JCGM Guides [2].

[c] Certified values are weighted means of the results from four to six analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), except for phenanthrene and benzo[a]pyrene for which a coverage factor of 4 was used, calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/JCGM Guide [2].

[d] GC/MS (Ia) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[e] GC/MS (IIIa) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[f] GC/MS (IV) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[g] GC/MS (IIa) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[h] GC/MS (Ib) on a liquid crystalline phase after PFE with DCM.

[i] GC/MS (IIb) on a liquid crystalline phase after PFE with DCM.

[j] GC/MS (IIIb) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

Table 2. Certified Mass Fraction for Selected PCB Congeners in SRM 2585

PCB Congener[a]	Mass Fraction (dry-mass basis)[b] (µg/kg)		
PCB 18 (2,2',5-Trichlorobiphenyl)[e,f,g,h]	12.8	±	1.0[c]
PCB 28 (2,4,4'-Trichlorobiphenyl)[f,g]	13.4	±	0.5[d]
PCB 31 (2,4',5-Trichlorobiphenyl)[f,g]	14.0	±	0.5[d]
PCB 44 (2,2'3,5'-Tetrachlorobiphenyl)[e,g,h]	18.1	±	1.9[c]
PCB 52 (2,2',5,5'-Tetrachlorobiphenyl)[e,f,g,h]	21.8	±	1.9[c]
PCB 56 (2,3,3',4-Tetrachlorobiphenyl)[e,f,g]	4.42	±	0.28[d]
PCB 70 (2,3',4',5-Tetrachlorobiphenyl)[e,f,g,h]	13.1	±	1.2[c]
PCB 74 (2,4,4',5-Tetrachlorobiphenyl)[e,f,g,h]	5.22	±	0.51[c]
PCB 87 (2,2',3,4,5'-Pentachlorobiphenyl)[e,f,g,h]	16.6	±	0.8[c]
PCB 92 (2,2',3,5,5'-Pentachlorobiphenyl)[f,g,h]	5.48	±	0.72[d]
PCB 95 (2,2',3,5',6-Pentachlorobiphenyl)[f,g,h]	22.7	±	2.6[d]
PCB 99 (2,2',4,4',5-Pentachlorobiphenyl)[,f,g,h]	11.6	±	0.4[c]
PCB 101 (2,2',4,5,5'-Pentachlorobiphenyl)[f,g,h]	29.8	±	2.3[d]
PCB 105 (2,3,3',4,4'-Pentachlorobiphenyl)[f,g,h]	13.2	±	1.4[d]
PCB 107 (2,3,3',4,5'-Pentachlorobiphenyl)[e,f,g]	4.14	±	0.47[d]
PCB 110 (2,3,3',4',6-Pentachlorobiphenyl)[e,f,g,h]	28.1	±	3.7[c]
PCB 118 (2,3',4,4',5-Pentachlorobiphenyl)[e,f,g,h]	26.3	±	1.7[c]
PCB 138 (2,2',3,4,4',5'-Hexachlorobiphenyl)[e,f,g,h]	27.6	±	2.1[c]
PCB 146 (2,2',3,4',5,5'-Hexachlorobiphenyl)[e,f,g,h]	4.89	±	0.38[c]
PCB 149 (2,2',3,4',5',6-Hexachlorobiphenyl)[e,f,g,h]	24.4	±	1.9[c]
PCB 151 (2,2',3,5,5',6-Hexachlorobiphenyl)[f,g,h]	6.92	±	0.64[d]
PCB 153 (2,2',4,4',5,5'-Hexachlorobiphenyl)[e,f,g,h]	40.2	±	1.8[c]
132 (2,2',3,3',4,6-Hexachlorobiphenyl)[i]			
PCB 158 (2,3,3',4,4',6-Hexachlorobiphenyl)[e,f,g,h,i]	4.50	±	0.43[c]
PCB 163 (2,3,3',4',5,6-Hexachlorobiphenyl)[e,f,g,h]	7.2	±	1.2[c]
PCB 170 (2,2',3,3',4,4',5-Heptachlorobiphenyl)[e,f,g,h]	8.8	±	1.0[c]
PCB 174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl)[e,f,g,h]	8.83	±	0.47[c]
PCB 180 (2,2',3,4,4',5,5'-Heptachlorobiphenyl)[e,f,h]	18.4	±	3.2[d]
PCB 183 (2,2',3,4,4',5',6-Heptachlorobiphenyl)[e,f,g,h]	5.27	±	0.39[c]
PCB 187 (2,2',3,4',5,5',6-Heptachlorobiphenyl)[e,f,g,h]	11.3	±	1.4[c]
PCB 206 (2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl)[e,h]	3.81	±	0.13[d]

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, PCB 107 is different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 107 is listed as PCB 108.

[b] Mass fraction reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

[c] Certified values are weighted means of the results from four analytical methods [14]. 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 Guide [2].

[d] Certified values are unweighted means 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, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO/JCGM Guide [2].

[e] GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

[f] GC/MS (I) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (I).

[g] GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[h] GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

[i] PCB 153 is the primary component with PCB 132 contributing at most 10 % to the mass fraction based on the data from method GC/MS (II).

Table 3. Certified Mass Fraction for Selected Chlorinated Pesticides in SRM 2585

	Mass Fraction (dry-mass basis)[a] (µg/kg)		
4,4'-DDE[d,e,f,g]	261	±	2[b]
4,4'-DDD[d,f]	27.3	±	0.8[c]
2,4'-DDT[d,f]	44.5	±	3.9[c]
4,4'-DDT[d,f,g]	111	±	23[c]

[a] Mass fraction reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

[b] Certified values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) except for 4,4'-DDE with a coverage factor of 10, calculated by combining a between-method variance incorporating inter-method bias with a pooled within-source variance following the ISO/JCGM Guide [2].

[c] Certified values are unweighted means 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, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO/JCGM Guide [2].

[d] GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

[e] GC/MS (I) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (I).

[f] GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[g] GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

Table 4. Certified Mass Fraction for Selected PBDE Congeners in SRM 2585

PBDE Congener[a]		Mass Fraction (dry-mass basis) (µg/kg)		
PBDE 17	(2,2',4-Tribromodiphenyl Ether)[c,d,e,f]	11.5	±	1.2[b]
PBDE 28	(2,4,4'-Tribromodiphenyl Ether)[c,d,e,f]	46.9	±	4.4[b]
33	(2',3,4-Tribromodiphenyl Ether)[g]			
PBDE 47	(2,2',4,4'-Tetrabromodiphenyl Ether)[c,d,e,f]	497	±	46[b]
PBDE 49	(2,2',4,5'-Tetrabromodiphenyl Ether)[c,d,e]	53.5	±	4.2[h]
PBDE 85	(2,2',3,4,4'-Pentabromodiphenyl Ether)[c,d,e,f]	43.8	±	1.6[b]
PBDE 99	(2,2',4,4',5-Pentabromodiphenyl Ether)[c,d,e,f]	892	±	53[b]
PBDE 100	(2,2',4,4',6-Pentabromodiphenyl Ether)[c,d,e,f]	145	±	11[b]
PBDE 138	(2,2',3,4,4',5'-Hexabromodiphenyl Ether)[c,d,e,f]	15.2	±	2.0[b]
PBDE 153	(2,2',4,4',5,5'-Hexabromodiphenyl Ether)[c,d,e,f]	119	±	1[b]
PBDE 154	(2,2',4,4',5,6-Hexabromodiphenyl Ether)[d,e]	83.5	±	2.0[h]
PBDE 155	(2,2',4,4',6,6'-Hexabromodiphenyl Ether)[d,e]	3.94	±	0.34[h]
PBDE 183	(2,2',3,4,4',5',6-Heptabromodiphenyl Ether)[c,d,e,f]	43.0	±	3.5[b]
PBDE 203	(2,2',3,4,4',5,6',6-Octabromodiphenyl Ether)[c,e]	36.7	±	6.4[h]
PBDE 206	(2,2',3,3',4,4',5,5',6-Nonabromodiphenyl Ether)[c,e]	271	±	42[h]
PBDE 209	(Decabromodiphenyl Ether)[c,e,f]	2510	±	190[h]

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

[b] Mass fraction reported on dry-mass basis; material as received contains approximately 2.1 % moisture. Certified values are weighted means of the results from four analytical methods [14]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence) except for PBDE 153 with a coverage factor of 10, calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/JCGM Guide [2].

[c] GC/NCI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

[d] GC/EI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM (same extracts as GC/NCI-MS (I)).

[e] GC/EI-MS (II) on a 60 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

[f] GC/NCI-MS (II) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[g] Using the 15 m column, PBDE 28 and PBDE 33 coelute; however, using the 60 m column, the two isomers are separated. Based on the data from the 60 m column, the mass fraction of PBDE 33 is less than the detection limit of 2.2 µg/kg.

[h] Certified values are unweighted means of the results from two or three analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [23] with a pooled, within-method variance following the ISO/JCGM Guide [2].

NOTICE AND WARNING TO USERS

SRM 2585 IS INTENDED FOR RESEARCH USE.

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: This material is naturally occurring house dust collected from a number of locations and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

Storage: SRM 2585 must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Use: Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The mass fractions of constituents in SRM 2585 are reported on a dry-mass basis. The SRM, as received, contains approximately 2.1 % moisture. The dust sample should be dried to a constant mass before weighing for analysis, or if the constituents of interest are volatile, a separate subsample of the dust should be removed from the bottle at the time of analysis and dried to determine the mass fraction on a dry-mass basis.

Conversion to Dry-Mass Basis: The moisture content of the dust was determined by measuring the mass loss after freeze drying. Six bottles of SRM 2585 were selected according to a stratified randomization scheme for the drying study. Approximately 1 g of each glass bottle was transferred to a glass weighing vial and dried for 7 days at 1 Pa with 5 °C shelf temperature and a –46.9 °C condenser temperature. The moisture content in SRM 2585 at the time of the certification analyses was 2.11 % ± 0.06 % (95 % confidence level). Analytical results for the organic constituents were determined on an as received mass fraction basis and then converted to a dry-mass fraction basis by dividing by the conversion factor of 0.9789 (g dry mass/g as received mass).

Non-Certified Values: Noncertified values are provided for additional constituents in SRM 2585.

Reference Mass Fraction Values: Based on the method used for each analyte, reference values are provided for additional measurands in Tables 5 through 11. These values are metrologically traceable to calibration procedures and standards used by experienced analysts. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Mass Fraction Values: A NIST information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed. Information mass fraction values, expressed as mass fractions, for 9 additional PBDE congeners are provided in Table 12. Information values cannot be used to establish metrological traceability.

Table 5. Reference Mass Fraction for Selected PAHs in SRM 2585

	Mass Fraction (dry-mass basis)[a] (µg/kg)		
1-Methylnaphthalene[d,e,f]	150	±	37[b]
2-Methylnaphthalene[d,e,f]	227	±	20[b]
Biphenyl[e,f,g]	88	±	21[b]
Retene[d]	588	±	34[c]
1,7-Dimethylphenanthrene[f,g]	219	±	19[b]
1-Methylfluoranthene[d,f,h]	94	±	11[b]
3-Methylfluoranthene[d,f,h]	235	±	67[b]
8-Methylfluoranthene[d]	132	±	7[c]
4-Methylpyrene[d,f,h]	235	±	27[b]
2-Methylpyrene[d]	345	±	10[c]
1-Methylpyrene[d,f,h]	209	±	69[b]
3-Methylchrysene[d,f,h]	146	±	18[b]
2-Methylchrysene[d]	181	±	4[c]
6-Methylchrysene[d,f]	88	±	14[b]
4-Methyl- and 1-Methylchrysene[d]	94.8	±	4.8[c]
9-Methyl- and 3-Methylbenz[a]anthracene[d]	92.3	±	2.5[c]
6-Methyl- and 1-Methylbenz[a]anthracene[d]	155	±	5[c]
Anthanthrene[f,g,i]	91	±	27[b]
Dibenzo[b,e]fluoranthene[f,g,j]	59.6	±	7.5[b]
Naphtho[1,2-b]fluoranthene[g,j]	312	±	10[b]
Naphtho[1,2-k] and naphtho[2,3-j]fluoranthene[f,g,j]	382	±	18[b]
Naphtho[2,3-b]fluoranthene[f,g,j]	93	±	30[b]
Naphtho[2,3-k]fluoranthene[i]	24.7	±	1.2[c]
Dibenzo[a,k]fluoranthene[f,j]	14.3	±	3.4[b]
Dibenzo[j,l]fluoranthene[f,g,i,j]	260	±	26[k]
Dibenzo[a,l]pyrene[f,g,j]	42.3	±	3.1[b]
Naphtho[2,3-k]fluoranthene and Naphtho[1,2-a]pyrene[f,g,j]	44.3	±	1.9[b]
Naphtho[2,3-e]pyrene[f,g,j]	145	±	29[b]
Naphtho[2,1-a]pyrene[f,g,i,j]	379	±	47[k]
Dibenzo[e,l]pyrene[f,g,i,j]	208	±	14[k]
Benzo[b]perylene[g,i]	103	±	24[b]
Dibenzo[a,i]pyrene[g,i]	105	±	11[b]
Dibenzo[a,h]pyrene[f]	20.9	±	0.7[c]

[a] Mass fraction reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

[b] Reference values are unweighted means 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, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO/JCGM Guide [2].

[c] 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.

[d] GC/MS (IIa) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[e] GC/MS (IIIa) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[f] GC/MS (IV) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[g] GC/MS (Ia) on a 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

[h] GC/MS (IIb) on a liquid crystalline phase after PFE with DCM.

[i] GC/MS (Ib) on a liquid crystalline phase after PFE with DCM.

[j] GC/MS (IIIb) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[k] Reference values are weighted means of the results from four analytical methods [14]. 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 Guide [2].

Table 6. Reference Mass Fraction for Selected PCB Congeners in SRM 2585

PCB Congener[a]	Mass Fraction (dry-mass basis)[b] (µg/kg)		
PCB 49 (2,2'4,5'-Tetrachlorobiphenyl)[e,f,g,h]	16.4	±	3.3[c]
PCB 63 (2,3,4',5-Tetrachlorobiphenyl)[e]	6.69	±	0.26[d]
PCB 66 (2,3',4,4'-Tetrachlorobiphenyl)[f,g,h]	8.5	±	1.9[e]
PCB 121 (2,3',4,5',6-Pentachlorobiphenyl)[h]	18.7	±	0.4[d]
PCB 128 (2,2',3,3',4,4'-Hexachlorobiphenyl)[g,h]	8.1	±	1.6[e]
PCB 177 (2,2',3,3',4,5,6-Heptachlorobiphenyl)[h]	5.50	±	0.44[d]
PCB 178 (2,2',3,3',5,5',6-Heptachlorobiphenyl)[h]	2.17	±	0.16[d]
PCB 185 (2,2',3,4,5,5',6-Heptachlorobiphenyl)[h]	5.32	±	0.39[d]
PCB 193 (2,3,3',4',5,5',6-Heptachlorobiphenyl)[e]	1.23	±	0.070[d]
PCB 194 (2,2',3,3',4,4',5,5'-Octachlorobiphenyl)[e,h]	4.47	±	0.76[e]
PCB 199 (2,2',3,3',4,5,6,6'-Octachlorobiphenyl)[h]	5.81	±	0.38[d]
PCB 209 Decachlorobiphenyl[e]	2.14	±	0.11[d]

[a] PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [15] and later revised by Schulte and Malisch [16] to conform with IUPAC rules; for the specific congeners mentioned in this SRM.

[b] Mass fractions reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

[c] Reference values are weighted means of the results from four analytical methods [14]. 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 Guide [2].

[d] 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.

[e] Reference values are unweighted means of the results from two to three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO/JCGM Guide [2].

[f] GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

[g] GC/MS (I) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (I).

[h] GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[i] GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

Table 7. Reference Mass Fraction for Selected Chlorinated Pesticides in SRM 2585

	Mass Fraction (dry-mass basis)[a] (µg/kg)		
cis-Chlordane[d,e,f]	174	±	45[b]
trans-Chlordane[d,e,f]	277	±	96[b]
cis-Nonachlor[f]	28.0	±	0.6[c]
trans-Nonachlor[d,e,f]	130	±	38[b]
Heptachlor[d,f]	166	±	34[b]
Heptachlor Epoxide[f]	11.3	±	0.6[c]
Dieldrin[d,f]	88	±	21[b]
gamma-hexachlorocyclohexane (HCH)[d]	4.06	±	0.55[c]
Mirex[d]	6.89	±	0.25[c]
Pentachlorobenzene[f]	20.9	±	1.6[c]

[a] Mass fractions reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

[b] Reference values are unweighted means of the results from two to three analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO/JCGM Guide [2].

[c] 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.

[d] GC-ECD (I) on a relatively non-polar proprietary phase after PFE extraction with DCM.

[e] GC/MS (II) on a 50 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

[f] GC/MS (III) on a 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

Table 8. Reference Mass Fraction for Selected PBDE Congeners in SRM 2585

PBDE Congener[a]	Mass Fraction (dry-mass basis)[b] (µg/kg)		
PBDE 66 (2,3',4,4'-Tetrabromodiphenyl Ether)[d,e,f,g]	29.5	±	6.2[c]
PBDE 75 (2,4,4',6-Tetrabromodiphenyl Ether)[e,f]	4.5	±	1.2[h]
PBDE 190 (2,3,3',4,4',5,6-Heptabromodiphenyl Ether)[d,g]	5.1	±	2.9[h]

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

[b] Mass fractions reported on dry-mass basis; material as received contains approximately 2.1 % moisture.

[c] Reference values are weighted means of the results from four analytical methods [14]. 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-source variance incorporating inter-method bias with a pooled within-source variance following the ISO/JCGM Guide [2].

[d] GC/NCI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

[e] GC/EI-MS (I) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM (same extracts as GC/NCI-MS (I)).

[f] GC/EI-MS (II) on a 60 m 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

[g] GC/NCI-MS (II) on a 15 m 5 % phenyl-substituted methylpolysiloxane phase after Soxhlet extraction with DCM.

[h] Reference values are unweighted means of the results from two analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [13] with a pooled, within-method variance following the ISO/JCGM Guide [2].

Table 9. Reference Mass Fraction for Selected Polycyclic Musks in SRM 2585

	Mass Fraction (dry-mass basis)[a] (µg/kg)		
1,3,4,6,7,8-Hexahydro-4,6,6,7,8,8-hexa-methyl-cyclopenta-(γ)-2-benzopyran (HHCB)	1470	±	60
7-Acetyl-1,1,3,4,4,6-hexamethyltetralin (AHTN)	1700	±	50[b]
4-Acetyl-1,1-dimethyl-6-tert-butylindan (ADBI)	163	±	10
6-Acetyl-1,1,2,3,3,5-hexamethylindan (AHMI)	201	±	11[b]
5-Acetyl-1,1,2,6-tetramethyl-3-isopropylindan (ATII)	144	±	4[b]

[a] Mass fractions reported on dry-mass basis; material as received contains approximately 2.1 % moisture. The reference mass fraction value is a weighted mean of the mass fractions determined for each analyte [14]. The uncertainty listed with each value is an expanded uncertainty about the mean [14,17], with coverage factor, $k = 2$, calculated by combining a pooled within-method variance with a between-method variance [18] following the ISO/JCGM Guide [2,19]. The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

[b] The expanded uncertainty is the half-width of a symmetric 95 % parametric bootstrap confidence interval [20], which is consistent with the ISO Guide [2,19]. The effective coverage factor, k, is 2.

Table 10. Reference Mass Fraction for Selected Perfluorinated Alkyl Acids (PFAAs) in SRM 2585

	Mass Fraction (dry-mass basis)[a] (µg/kg)		
Perfluorobutanoic Acid (PFBA)	230	±	16
Perfluorohexanoic Acid (PFHxA)	260	±	25
Perfluoroheptanoic Acid (PFHpA)	249	±	32
Perfluorononanoic Acid (PFNA)	99.4	±	4.9)
Perfluorododecanoic Acid (PFDoA)	34.6	±	4.5
Perfluorotridecanoic Acid (PFTriA)	29.4	±	4.6
Perfluorohexanesulfonic Acid (PFHxS)	1440	±	250
Perfluorooctanesulfonic Acid (PFOS)	2310	±	420

[a] Mass fractions reported on dry-mass basis; material as received contains approximately 2.1 % moisture. The reference mass fraction value is a weighted mean of the mass fractions determined for each analyte [14]. The uncertainty listed with each value is an expanded uncertainty about the mean [14,17], with coverage factor, $k = 2$, calculated by combining a pooled within-method variance with a between-method variance [18] following the ISO/JCGM Guide [2,19]. The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

Table 11. Reference Mass Fraction for Selected Flame Retardants in SRM 2585

	Mass Fraction (dry-mass basis)[a] (µg/kg)		
α-hexabromocyclododecane (α-HBCD)	21.2	±	3.6
Tributyl phosphate	276	±	14
Tris (2-chloroethyl) phosphate	925	±	149
Tris (chloropropyl) phosphate[b]	1220	±	350
Triphenyl phosphate	1190	±	130

[a] Mass fractions reported on dry-mass basis; material as received contains approximately 2.1 % moisture. For reference values to be valid, the material must be dried according to the instructions provided above. The reference mass fraction value is a weighted mean of the mass fractions determined for each analyte [14]. The uncertainty listed with each value is an expanded uncertainty about the mean [14,17], with coverage factor, $k = 2$, calculated by combining a pooled within-method variance with a between-method variance [18] following the ISO/JCGM Guide [2,19].

[b] Tris (chloropropyl) phosphate was quantified as a mixture of the following isomers: tris(1-chloro-2-propyl) phosphate (major isomer), bis(1-chloro-2-propyl)(2-chloropropyl) phosphate and (1-chloro-2-propyl)bis(2-chloropropyl) phosphate.

Table 12. Information Mass Fraction for Selected PBDE Congeners in SRM 2585[a]

PBDE Congener[b]		Mass Fraction (dry-mass basis)[c] (µg/kg)
PBDE 25	(2,3',4-Tribromodiphenyl Ether)	<0.2
PBDE 30	(2,4,6-Tribromodiphenyl Ether)	<0.2
PBDE 71	(2,3',4',6-Tetrabromodiphenyl Ether)	<0.2
PBDE 116	(2,3,4,5,6-Pentabromodiphenyl Ether)	<0.2
PBDE 119	(2,3',4,4',6-Pentabromodiphenyl Ether)	<0.2
PBDE 156	(2,3,3',4,4',5-Hexabromodiphenyl Ether)	<0.2
PBDE 181	(2,2',3,4,4',5,6-Heptabromodiphenyl Ether)	<0.3
PBDE 191	(2,3,3',4,4'5',6-Heptabromodiphenyl Ether)	<0.3
PBDE 205	(2,3,3',4,4',5,5',6-Octabromodiphenyl Ether)	<0.5

[a] Mass fractions reported on dry-mass basis; material as received contains approximately 2.1 % moisture.
[b] PBDE congeners are numbered according to the IUPAC rules.
[c] The information values are from the method detection limits using a signal to noise value = 100 for method GC/NCI-MS (I).

PREPARATION AND ANALYSIS[1]

Sample Collection: The collection of SRM 2585 was under the direction of the Research Triangle Institute and the U.S. Environmental Protection Agency (Research Triangle Park, NC). The dust used for the preparation SRM 2585 was taken from vacuum cleaner bags collected from homes, cleaning services, motels, and hotels in the states of North Carolina, Maryland, Ohio, New Jersey, Montana, and Wisconsin during 1993 and 1994. The dust used for the preparation of SRM 2585 was collected at the same time and from the same locations as the dust used for the preparation of SRM 2583 *Trace Elements in Indoor Dust, Nominal 90 mg/kg Lead* [3] and SRM 2584 *Trace Elements in Indoor Dust, Nominal 1 % Lead* [4].

Sample Preparation: The vacuum cleaner bags were first sterilized by gamma irradiation, receiving a minimum total dose of 2.5 mrads. For the low lead material, SRM 2583, the contents of each vacuum cleaner bag was passed through a coarse screen (2 mm hole size) to remove cotton and debris. Then using a vibrating stainless steel sieve apparatus, the resultant material was screened in two successive steps, first through a 250 µm sieve and then a 100 µm sieve. For the high lead material, SRM 2584, the raw material from each bag was mixed and tumbled in a modified food processor using chopping blades and a compressed air jet. While still tumbling, the dust was separated from unwanted debris by vacuuming through a series of screens into a clean HEPA vacuum cleaner. The dust collected in this manner was then screened through a 90 µm stainless steel sieve using vibration and a vacuum. Processed sublots of approximately 5 kg each were set aside and analyzed for lead by X-ray fluorescence to develop a blending protocol for the target lead concentrations in SRM 2584. SRM 2585 was prepared from approximately 70 % of the material found to have 90 mg/kg nominal lead levels used for SRM 2583 and 30 % of the material that had the high lead content used for SRM 2584. The selected sublots were blended in a cone blender and then bottled (approximately 10 g of dust per bottle) as SRM 2585.

Polycyclic Aromatic Hydrocarbons

The general approach used for the value assignment of the PAHs in SRM 2585 was similar to that reported for the recent certification of several environmental matrix SRMs [5−8] and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. The methods utilized Soxhlet extraction and pressurized fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts by solid phase extraction (SPE), and analysis by gas chromatography/mass spectrometry (GC/MS) of the PAH fraction on three stationary phases of different selectivity, i.e., a 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase, a relatively non-polar proprietary phase, and a liquid crystalline phase.

Seven sets of GC/MS results, designated as GC/MS (Ia), (Ib), (IIa), (IIb), (IIIa), (IIIb), and (IV) were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I and II) analyses, one subsample of approximately 1 g from six bottles of SRM 2585 was extracted using PFE with DCM [9]. The concentrated extract was passed through a silica SPE cartridge and eluted with 2 % DCM in hexane (volume fraction). The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17ms, J&W Scientific, Folsom, CA). This method is designated as GC/MS (Ia) and GC/MS (IIa). For GC/MS (Ib) and (IIb), the same extracts were analyzed by GC/MS using a 0.25 mm i.d. × 15 m fused silica capillary column with dimethyl 50 % (mole fraction) liquid crystalline polysiloxane phase (0.25 µm film thickness) (LC-50, J&K Environmental, Nova Scotia, Canada). The GC/MS (III) analyses were performed using duplicate subsamples of approximately 1 g from six bottles of SRM 2585. These samples were Soxhlet extracted for 18 h with 250 mL of 50 % hexane and 50 % acetone (volume fraction). The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The fractions were concentrated, and the SPE step was repeated. GC/MS analysis was performed using 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB, J&W Scientific) for method GC/MS (IIIa) and a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17ms) for method GC/MS (IIIb). For the GC/MS (IV) analyses, approximately 1 g subsamples from six bottles of SRM 2585 were extracted using PFE with DCM. The concentrated extract was passed through an alumina cartridge and eluted with 9 mL of 35 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17ms). For the GC/MS measurements described above, selected perdeuterated PAHs were added to the dust prior to solvent extraction for use as internal standards for quantification purposes.

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

Homogeneity Assessment for PAHs

The homogeneity of SRM 2585 was assessed by analyzing duplicate samples of approximately 1 g from six bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (IIIa and IIIb). No statistically significant differences among bottles were observed for the PAHs at this sample size.

PCBs and Chlorinated Pesticides

The general approach used for the determination of PCBs and chlorinated pesticides in SRM 2585 was similar to that reported for the recent certification of several environmental matrix SRMs [5,7,10−12], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, cleanup/isolation using normal-phase liquid chromatography (LC) or SPE followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on three columns with different selectivity for the separation of PCBs and chlorinated pesticides.

Four sets of results were obtained, designated as GC-ECD (I), GC/MS (I), GC/MS (II), and GC/MS (III). For GC-ECD (I) and GC/MS (I), subsamples of approximately 1 g from six bottles of SRM 2585 were extracted using PFE with DCM. The concentrated extract was fractionated by LC on a semi-preparative aminopropylsilane column to isolate two fractions containing (1) the PCB congeners and lower polarity pesticides using hexane as the mobile phase and (2) the more polar pesticides using 25 % DCM in hexane as the mobile phase. Each concentrated fraction was passed through a silica SPE cartridge with 25 % DCM in hexane, concentrated, and then analyzed by GC-ECD and by GC/MS both using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 μm film thickness) (DB-XLB). PCB 30, PCB 103, and perdeuterated 4,4'-DDT were added prior to extraction for use as internal standards for quantification purposes. For GC/MS (II), duplicate subsamples of approximately 1 g from six bottles of SRM 2585 were Soxhlet extracted for 18 h with 250 mL of 50 % hexane and 50 % acetone (volume fraction). The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The fractions were concentrated, and the SPE step was repeated. GC/MS analysis was performed using 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-17ms). For the GC/MS (II) analyses, selected ^{13}C-labeled PCB congeners and chlorinated pesticides were added to the dust prior to extraction for use as internal standards for quantification purposes. For the GC/MS (III) analyses, approximately 1 g subsamples from six bottles of SRM 2585 were extracted using PFE with DCM. The concentrated extract was passed through an alumina cartridge and eluted with 9 mL of 35 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5ms, J&W Scientific). Electron impact mass spectrometry was used for all of the analytes except for the chlordanes and nonachlors, which were quantified using negative chemical ionization for GC/MS (III). For the GC/MS (III) analyses, selected ^{13}C-labeled PCB congeners, ^{13}C-labeled *trans*-chlordane, and selected deuterated pesticides were added to the dust prior to extraction for use as internal standards for quantification purposes.

Homogeneity Assessment for PCBs and Chlorinated Pesticides: The homogeneity of SRM 2585 was assessed by analyzing duplicate samples of approximately 1 g from six bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (II). No statistically significant differences among bottles were observed for the chlorinated analytes at this sample size.

Polybrominated Diphenyl Ethers

Value assignment of mass fractions for PBDE congeners was based on four sets of measurements (three sets from NIST and one set from a collaborating laboratory) using a variety of different extraction, cleanup, and quantification methods. All measurements were performed by using GC/MS operated in either electron impact (GC/EI-MS) or negative chemical ionization (GC/NCI-MS) mode.

For two of the NIST measurement sets, GC/NCI-MS (I) and GC/EI-MS (I), between 0.5 g and 1 g subsamples of dust from each of four bottles were extracted using PFE with DCM. The concentrated extract was placed on a silica SPE cartridge and eluted with hexane. The extracts were analyzed by using both GC/EI-MS and GC/NCI-MS on a 0.25 mm × 15 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5ms). For both methods ^{13}C-labeled 2,2',3,4,5-pentachlorodiphenyl ether (CDE 86L) and ^{13}C-labeled 2,2',3,3',4,4',5,5',6,6'-decabromodiphenyl ether (BDE 209L) were added to the dust sample prior to extraction for use as internal standards for quantification purposes.

For the third NIST measurement set, GC/EI-MS (II), between 0.5 g and 1 g subsamples of dust from each of six bottles were extracted using PFE with DCM. The extracts were first subjected to a cleanup step using a 5 % deactivated alumina SPE column and then injected onto a size exclusion chromatographic column. The extracts were analyzed by using GC/EI-MS on a 0.25 mm x 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5ms). ^{13}C-labeled 2,2,4,4',5-pentabromodiphenyl ether (PBDE 99L) and PBDE 209L were added to the dust samples prior to extraction for use as an internal standard for quantification of the PBDEs.

For the measurements from the collaborating laboratory (Environment Canada), GC/NCI-MS (II), four subsamples of between 0.5 g and 1 g from a single bottle were Soxhlet extracted using DCM after spiking with mirex as an internal standard. The extracts were cleaned using 2 g of 6 % deactivated alumina with petroleum ether. The extracts were analyzed by using GC/NCI-MS on a 0.25 mm × 15 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5ms).

Polycyclic Musks

Value assignment of mass fractions for polycyclic musks was based on two sets of measurements using a variety of different extraction, cleanup, and quantification methods. All measurements were performed by using GC/EI-MS.

For two of the NIST measurement sets between 0.5 g and 1 g subsamples of dust from each of six bottles were extracted using PFE with DCM. The extracts were first subjected to a cleanup step using a 5 % deactivated alumina SPE column and then injected onto a size exclusion chromatographic column. The extracts were analyzed by using GC/EI-MS on either a 0.25 mm x 60 m fused silica capillary column with a 5 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-5ms) or a 0.18 mm i.d. × 30 m fused silica capillary column with a relatively non-polar proprietary phase (0.18 μm film thickness) (DB-XLB). The internal standard d_{10}-fluoranthene was added to the dust samples prior to extraction and used for the quantification of polycyclic musks.

Perfluorinated alkyl acids

Value assignment of mass fractions for PFAAs was based on four sets of measurements using a variety of different extraction, cleanup, and quantification methods. All measurements were performed by using LC/MS/MS operated in the negative ionization mode.

For one NIST measurement set between 0.5 g and 1 g of dust were extracted using sonication with 0.1 M potassium hydroxide in methanol for 30 min. The extracts were subjected to a weak anion exchange SPE column for cleanup. The extracts were analyzed by using LC/MS/MS on a 2.1 mm x 100 mm x 5 μm C8 column. ^{13}C-labeled PFAAs were added to the dust samples prior to extraction and used for the quantification of PFAAs.

For one set of NIST measurements between 0.5 g and 1 g of dust were extracted using sonication with acetonitrile for 10 min. The extracts were subjected to a graphitized carbon black SPE column for cleanup. The extracts were analyzed by using LC/MS/MS on a 3.0 mm x 50 mm x 2.6 μm pentafluorophenyl phase column. ^{13}C-labeled PFAAs were added to the dust samples prior to extraction and used for the quantification of PFAAs.

For two NIST measurement sets between 0.5 g and 1 g of dust were extracted using PFE with 0.1 M potassium hydroxide in methanol. The extracts were subjected to a graphitized carbon black SPE column for cleanup. The extracts were analyzed by using LC/MS/MS on either a 2.1 mm x 100 mm x 5 μm C8 column or a 3.0 mm x 50 mm x 2.6 μm pentafluorophenyl phase column. ^{13}C-labeled PFAAs were added to the dust samples prior to extraction and used for the quantification of PFAAs.

Hexabromocyclododecanes

Value assignment of mass fractions for hexabromocyclododecanes was based on four sets of measurements using a variety of different extraction, cleanup, and quantification methods. All measurements were performed by using LC/MS/MS operated in the negative ionization mode.

For one of the NIST measurement sets approximately 0.2 g subsamples of dust were extracted using PFE with DCM. The extracts were first subjected to a cleanup on a size exclusion chromatographic column and then a cleanup using a 5 % deactivated alumina SPE column. The extracts were analyzed by using LC/MS/MS on a 3.0 mm x 150 mm x 3.5 μm C18 column. ^{13}C-labeled hexabromocyclododecanes were added to the dust samples prior to extraction for use as an internal standard for quantification of the hexabromocyclododecanes.

For three of the NIST measurement sets between 0.8 g and 1 g subsamples of dust were extracted using ultrasonic extraction with DCM for one hour. The extracts were subjected to cleanup using a silica SPE cartridge and eluted with DCM and methanol. The extracts were analyzed using LC/MS/MS on a 3.0 mm x 150 mm x 3.5 μm C18 column. ^{13}C-labeled hexabromocyclododecanes were added to the dust samples prior to extraction for use as an internal standard for quantification of the hexabromocyclododecanes

Organophosphorus flame retardants

Value assignment of mass fractions for organophosphorus flame retardants was based on four sets of measurements using a variety of different extraction, cleanup, and quantification methods. Measurements were performed by using GC/EI-MS or LC/MS/MS operated in the negative ionization mode.

For one NIST measurement set between 0.5 g and 1 g of dust were extracted using ultrasonic extraction with DCM. The extracts were subjected to cleanup using a silica SPE cartridge and eluted with ethyl acetate. The extracts were analyzed using GC/EI-MS on a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 μm film thickness) (DB-XLB). The internal standards d_{27}-Tributyl phosphate and d_{15}-Triphenyl phosphate were added to the dust samples prior to extraction for use as an internal standard for quantification of the organophosphorus flame retardants.

For three NIST measurement sets between 0.5 g and 1 g of dust were extracted using Soxhlet extraction for 18 h. Both sets of Soxhlet extraction were performed with 250 mL of 50 % hexane and 50 % acetone (volume fraction). The extracts were subjected to cleanup using a florisil SPE cartridge and eluted with ethyl acetate. Two sets of the processed extract were then analyzed by GC/MS using a 0.25 mm i.d. × 30 m fused silica capillary column with 50 % (mole fraction) phenyl-substituted methylpolysiloxane phase (0.25 μm film thickness) (DB-17ms). The third set of process extract was analyzed using LC/MS/MS on a 2.1 mm x 100 mm x 3 μm C18 column. The internal standards d_{27}-Tributyl phosphate and d_{15}-Triphenyl phosphate were added to the dust samples prior to extraction for use as an internal standard for quantification of the organophosphorus flame retardants.

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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.