

STANDARD REFERENCE MATERIALS CHARACTERIZED FOR HALOGENATED ORGANIC CONTAMINANTS

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Introduction

The National Institute of Standards and Technology (NIST) issued its first Certified Reference Material (CRM) for halogenated organic contaminants in 1982. This material, Standard Reference Material (SRM) 1581 Polychlorinated Biphenyls in Oil has certified values for Aroclor 1242 and Aroclor 1260 in motor oil and in transformer oil¹. Since 1982, the analytical methods used at NIST and collaborating institutes have improved such that recent materials, such as SRM 1958 Organic Contaminants in Fortified Human Serum have values assigned (both certified and reference values, see below) for more than 40 polychlorinated biphenyl (PCB) congeners, 20 chlorinated pesticides, 10 polybrominated diphenyl ethers (PBDE) congeners, and 15 polychlorinated dibenzo-*p*-dioxin (PCDD) and dibenzofuran (PCDF) congeners.

Materials and methods

Descriptions of the materials and methods are summarized in each Certificate of Analysis¹.

Results and discussion

NIST has available solution SRMs that are useful for validating and calibrating the chromatographic separation and detection steps, as well as natural-matrix SRMs that are useful for evaluating and validating the complete analytical procedure². Table 1 lists the SRMs characterized for halogenated organic contaminants with an indication of what groups of compounds are listed on the Certificates of Analysis as certified, reference, or information values.

The principal mode used for the certification of SRMs for halogenated organic contaminants is the analysis of the material using two or more chemically independent analytical techniques^{2,3}. If the results from the two or more independent analytical techniques are in agreement, it is assumed that the possibility of biases in the value is minimized so the result is considered a certified value on the Certificate of Analysis. When the results are obtained by one laboratory method or the results are obtained from two or more analytical methods that either do not agree or are not considered independent methods, the value is listed as a reference value on the Certificate of Analysis. As the name implies, information values appear on the Certificates of Analysis to provide additional information to the user, often without an associated uncertainty.

As shown in Table 1, there are a wide variety of solution and natural matrix SRMs available with values for PCB congeners, Aroclors, pesticides, PBDE congeners (including methoxy PBDE congeners in whale blubber SRM 1945), as well as a more limited number with values for PCDDs and PCDFs. The PCDD and PCDF values that appear on the Certificates of Analysis for the natural matrix materials are all reference values typically obtained through an interlaboratory study or by one laboratory outside of NIST. For additional classes of compounds, perfluorinated compounds (PFCs) have been characterized in the human serum SRMs 1957 and 1958 through an interlaboratory study so appear as reference values, and hexabromocyclododecane (HBCD) congeners have been characterized at NIST in the fine particulate matter SRMs 2786 and 2787 and in the freeze-dried mussel tissue SRM 2974a and appear as information values for SRMs 2786 and 2787 and reference values for SRM 2974a. One issue for the PFCs and HBCDs, that may delay the values from appearing on any Certificate of Analysis as certified values, is the purity of the commercial compounds used to prepare the calibration solutions. Most of the PFCs are sold commercially as the linear plus branched forms, and there are not many commercial suppliers for neat PFCs or HBCDs.

Since the materials summarized in Table 1, as well as other SRMs, are homogeneous, they are suitable for use in developing methods for and characterizing other analytes of interest. The stability of the materials is monitored over time, and the Certificates of Analysis are updated periodically as new data are generated.

Acknowledgements

Many individuals at NIST and other organizations have contributed to the certification of these SRMs. Their names and affiliations are noted on the Certificates of Analysis.

References:

1. For the Certificates of Analysis, see <http://www.nist.gov/srm/>
2. Wise, S.A.; Poster, D.L.; Kucklick, J.R.; Keller, J.M.; Vander Pol, S.S.; Sander, L.C.; Schantz, M.M.; *Standard Reference Materials (SRMs) for Determination of Organic Contaminants in Environmental Samples*; Anal. Bioanal. Chem.; Vol. 386, pp. 1153-1190 (2006).
3. May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136, U.S. Government Printing Office: Gaithersburg, MD (2000); <http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm>.

Table 1. SRMs Characterized for Halogenated Organic Contaminants

SRM	Title	Certified, Reference, Information Values
Solutions		
1492	Chlorinated Pesticides in Hexane	Pesticides (nominal 0.3 µg/g)
1493	Polychlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane	PCBs (nominal 0.3 µg/g)
1614	Dioxin (2,3,7,8-TCDD) in Isooctane	Native and ¹³C₁₂ 2,3,7,8-TCDD
2257	PBDE Congeners in 2,2,4-Trimethylpentane	PBDEs (1 µg/g to 6 µg/g)
2258	BDE 209 in 2,2,4- Trimethylpentane	BDE 209 (nominal 7 µg/g)
2259	PCB Congeners in 2,2,4-Trimethylpentane	PCBs (0.3 µg/g to 53 µg/g)
2261	Chlorinated Pesticides in Hexane (Nominal Concentration 2 µg/mL)	Pesticides
2262	Chlorinated Biphenyl Congeners in 2,2,4-Trimethylpentane (Nominal Concentration 2 µg/mL)	PCBs
2273	Chlorinated Pesticides (DDTs) and Metabolites in Isooctane	DDTs (nominal 3 µg/g)
2274	PCB Congener Solution-II in Isooctane	PCBs (nominal 3 µg/g)
2275	Chlorinated Pesticide Solution-II in Isooctane	Pesticides (nominal 3 µg/g)
2276	Three Planar Polychlorinated Biphenyl (PCB) Congeners in Isooctane	PCBs (nominal 3 µg/g)
3067	Toxaphene in Methanol	Total Toxaphene; toxaphene congeners
3068	Total Chlordane in Methanol	Total Chlordane
3075	Aroclor 1016 in Transformer Oil	Aroclor 1016
3076	Aroclor 1232 in Transformer Oil	Aroclor 1232
3077	Aroclor 1242 in Transformer Oil	Aroclor 1242
3078	Aroclor 1248 in Transformer Oil	Aroclor 1248
3079	Aroclor 1254 in Transformer Oil	Aroclor 1254
3080	Aroclor 1260 in Transformer Oil	Aroclor 1260
3081	Aroclor 1016 in Methanol	Aroclor 1016
3082	Aroclor 1232 in Methanol	Aroclor 1232
3083	Aroclor 1242 in Methanol	Aroclor 1242
3084	Aroclor 1248 in Methanol	Aroclor 1248
3085	Aroclor 1254 in Methanol	Aroclor 1254
3086	Aroclor 1260 in Methanol	Aroclor 1260
3090	Aroclors in Transformer Oil	Aroclors 1016, 1232, 1242, 1248, 1254, 1260
3091	Aroclors in Methanol	Aroclors 1016, 1232, 1242, 1248, 1254, 1260
Natural Matrix Materials		
1581	Polychlorinated Biphenyls in Oil	Aroclor 1242 and 1260
1588c	Organics in Cod Liver Oil	PCBs, pesticides, PBDEs, fatty acids; PCBs, pesticides, fatty acids
1649b	Urban Dust	PCBs, pesticides, PAHs; PCBs, pesticides, BDE 209, PCDDs, PCDFs, PAHs, nitro-PAHs; aliphatics, hopanes, steranes, ketones, particle size
1939a	Polychlorinated Biphenyls (Congeners) in River Sediment A	PCBs; PCBs, pesticides, PAHs
1941b	Organics in Marine Sediment	PCBs, pesticides, PAHs; PCBs, pesticides, PAHs, TOC; carbon, hydrogen, nitrogen
1944	New York/New Jersey Waterway Sediment	PCBs, pesticides, PAHs, inorganics; Pesticides, PCDDs, PCDFs, PAHs, inorganics, particle size, TOC, % extractable; inorganics

Table 1 (cont). SRMs Characterized for Halogenated Organic Contaminants

SRM	Title	Certified, Reference, Information Values
1945	Organics in Whale Blubber	PCBs, pesticides, PBDEs; <i>PCBs, pesticides, PBDEs, MeO-PBDEs</i>
1946	Lake Superior Fish Tissue	PCBs, pesticides, extractable fat, fatty acids, MeHg, Hg, As, Fe; <i>PCBs, pesticides, fatty acids, proximates, inorganics;</i> carbohydrates, inorganics, fatty acids
1947	Lake Michigan Fish Tissue	PCBs, pesticides, PBDEs, inorganics, MeHg; <i>PCBs, pesticides, PBDEs, proximates, fatty acids</i>
1953	Organic Contaminants in Non-Fortified Human Milk	PCBs, pesticides, PBDEs; <i>PCBs, Pesticides, PCDDs, PCDFs, inorganics;</i> Aroclor 1260
1954	Organic Contaminants in Fortified Human Milk	PCBs, pesticides, PBDEs; <i>PCBs, Pesticides, PCDDs, PCDFs, inorganics;</i> Aroclor 1260
1957	Organic Contaminants in Non-Fortified Human Serum	PCBs, pesticides, PBDEs; <i>PCBs, pesticides, PBDEs, PFCs, PCDDs, PCDFs;</i> Aroclor 1260
1958	Organic Contaminants in Fortified Human Serum	PCBs, pesticides, PBDEs; <i>PCBs, pesticides, PBDEs, PFCs, PCDDs, PCDFs;</i> Aroclor 1260
1974b	Organics in Mussel Tissue (<i>Mytilus edulis</i>)	PCBs, pesticides, PAHs; <i>PCBs, Pesticides, PAHs, % extractable, MeHg, inorganic,</i>
2585	Organic Contaminants in House Dust	PCBs, pesticides, PBDEs, PAHs,; <i>PCBs, Pesticides, PBDEs, PAHs;</i> PBDEs
2786	Fine Particulate Matter (<4 μm)	PBDEs, PAHs, nitro-PAHs; <i>PBDEs, PCDDs, PCDFs, PAHs, nitro-PAHs, inorganics, sugars;</i> HBCDs, inorganics, particle-size
2787	Fine Particulate Matter (<10 μm)	PBDEs, PAHs, nitro-PAHs; <i>PBDEs, PCDDs, PCDFs, PAHs, nitro-PAHs, inorganics, sugars;</i> HBCDs, inorganics, particle-size
2974b	Organics in Freeze-dried Mussel Tissue (<i>Mytilus edulis</i>)	PCBs, pesticides, PAHs, MeHg, total & inorganic Hg; <i>PCBs, PBDEs, HBCDs, PAHs</i>