

Newly Developed Biota and Biota-related Standard Reference Materials for the Determination of Environmental Contaminants

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Biota Standard Reference Materials (SRMs®). Since 1990, the National Institute of Standards and Technology (NIST) has issued a number of cryogenically homogenized tissue SRMs with certified and reference values assigned for organic contaminants (Table 1). The cryogenically homogenized materials are powder-like with the endogenous water retained. We recently reviewed the development and availability of mussel-tissue SRMs and fish-tissue SRMs.^{1,2} The series of natural mussel-tissue SRMs (Organics in Mussel Tissue, *Mytilus edulis*) has been developed from mussels collected in Boston Harbor, MA. SRM 1974b is the third and current material in this series and has certified and reference values for a range of polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners (1 non-*ortho*), total PCBs, chlorinated pesticides, methyl-Hg, Hg, selected trace elements, and total extractable organics. An additional mussel tissue SRM, SRM 2977, is also available (Table 1). This is a freeze-dried tissue homogenate prepared from mussels collected in Guanabara Bay, Brazil.¹

Two cryogenically homogenized fish tissue SRMs have been developed from filleted adult lake trout (*Salvelinus namaycush namaycush*).² SRMs 1946 (Lake Superior Fish Tissue) and 1947 (Lake Michigan Fish Tissue) are characterized for a range of PCB congeners, chlorinated pesticides, methyl-Hg, Hg, selected trace elements, fatty acids, calories, proximates. Both materials have also been examined for selected polybrominated diphenyl ether (PBDE) congeners. SRM 1946 has additionally been examined for total toxaphene and toxaphene congeners.³

Biota-related SRMs. The newest biota-related SRM is a cod liver oil SRM, SRM 1588b. This material is intended for use in developing and validating analytical methods for the determination of PCBs, chlorinated pesticides, 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 alpha-tocopherol, 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 alpha-tocopherol, 34 PCB congeners, and 3 chlorinated pesticides. This series of cod liver oil SRMs consists of a commercial cod liver oil that has been fortified with selected polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDDs/PCDFs) congeners. The concentrations of these components in SRM 1588b are included on the material's Certificate of Analysis as information values. SRM 1588b updates the SRM 1588a certified values and provides certified and reference values for additional analytes, including fatty acids, PBDE congeners, and toxaphene congeners. The material has also been examined for total toxaphene³ and was used in a toxaphene interlaboratory study.⁴ Certified concentration values are also reported for non-*ortho* substituted PCBs.⁵ Concentrations of PCBs in SRM 1588b range from about 3 microgram/kg for PCB 209 to about 275 microgram/kg for PCB 153. Concentrations of pesticides range from about 12 microgram/kg for mirex to about 675 microgram/kg for 4,4'-DDE. SRM 1588b consists of four sealed ampoules per unit, each ampoule containing approximately 1.2 mL of cod liver oil.

The second newest biota-related SRM is a human serum SRM, SRM 1589a. This material was developed in conjunction with the Centers for Disease Control (CDC) with certified concentrations for natural levels of selected PCB congeners and chlorinated pesticides along with reference values for selected PCB congeners, chlorinated pesticides, and polychlorinated dibenzo-*p*-dioxins/dibenzofurans (PCDDs/PCDFs) congeners. Reference concentration values are also provided for total cholesterol, triglycerides, "free" cholesterol, and phospholipids determined by CDC. SRM 1589a is intended to use in evaluating analytical methods for the determination of these compounds in human serum and similar matrices. All of the constituents for which certified and reference values are provided in SRM 1589a are naturally present in the human serum. Plasma was acquired from screened donors who fished on the Great Lakes and ate their catches. Additional samples were selected from individuals who, in their judgement, ate large quantities of fish or the selections were made at random. A unit of the material consists of five

bottles of freeze-dried human serum. SRM 1589a replaces its predecessor, SRM 1589, Polychlorinated Biphenyls (as Aroclor 1260) in Human Serum. SRM 1589 was issued in 1985 and is no longer available.

Table 1. Biota and biota-related SRMs for the determination of trace pollutants^a.

SRM	SRM Name	Year	Compound Classes Characterized
<i>Biota SRMs</i>			
1945	Organics in Whale Blubber	1994	PCBs, pesticides
1946	Lake Superior Fish Tissue	2003	PCBs, pesticides, fatty acids, extractable fat, methyl-Hg, Hg, trace elements, proximates
1947	Lake Michigan Fish Tissue	2005	PCBs, pesticides, trace elements; PBDEs
1974b	Organics in Mussel Tissue (<i>Mytilus edulis</i>)	2003	PAHs, PCBs (1 non-ortho, total PCBs), pesticides, methyl-Hg, Hg, trace elements, total extractable organics
2977	Mussel Tissue (Organic Contaminants and Trace Elements)	2000	PAHs, PCBs, pesticides, methyl-Hg, Hg, trace elements
<i>Biota-related SRMs</i>			
1588b	Organics in Cod Liver Oil	2005	PCBs, pesticides, PBDEs, toxaphene, fatty acids, alpha-tocopherol
1589	PCBs (as Aroclor 1260) in Human Serum	1985 ^b	PCBs (as Aroclor 1260), PCDDs,
1589a	PCBs, Pesticides, Dioxins/Furans in Human Serum	2000	PCBs, pesticides, PCDDs/PCDFs
1957	Natural Levels of Contaminants in Human Serum	in prep.	PCDDs, PCDFs, PCBs, PBDEs, chlorinated pesticides, toxaphene, polychlorinatednaphthalenes, andotherhalogenatedcompounds
1958	Contaminants in Human Serum (fortified)	in prep.	same suite of compounds as in SRM 1957
new	environmental contaminants in mother's milk (two materials)	in prep.	both natural levels and a fortified material for the same suite of compounds as in SRM 1957

^afor a complete listing of available SRMs visit www.nist.gov

^bSRM 1589 is no longer available

Table 2. Sediment and particle-related SRMs for the determination of trace pollutants.

SRM	SRM Name	Year	Compound Classes Characterized
<i>Sediment SRMs</i>			
1939a	PCB Congeners in River Sediment	1998	PCBs, pesticides
1941b	Organics in Marine Sediment	2002	PAHs, PCBs, pesticides, mono-, di-, tributyl tin, total tin, total organic carbon, C, H, N
1944	NY/NJ Waterway Sediment	1999	PAHs, PCBs, Pesticides, PCDDs/ PCDFs, trace elements
<i>Combustion/Particle-relatedSRMs</i>			

1597a	Complex Mixture of PAHs from Coal Tar	2005	PAHs, alkyl-PAHs, nitro-PAHs
1649a	Urban Dust	1998	PAHs, PCBs, pesticides, PCDDs/PCDFs,
1650b	Diesel Particulate Matter	2005	PAHs, alkyl-PAHs, nitro-PAH
1975	Diesel Particulate Extract	2000	PAHs, nitro-PAHs
2585	House Dust	2005	PAHs, PCBs, pesticides, PBDEs
2975	Diesel Particulate Matter, Industrial Forklift	2000	PAHs, alkyl-PAHs, nitro-PAHs
	<i>Air particulate-related SRM Solutions</i>		
2264	Nitro-PAHs I	2005	11 constituents
2265	Nitro-PAHs II	2005	16 constituents
2266	Hopanes and Steranes	2005	10 constituents
2267	Levoglucosan (deuterated) in Ethyl Acetate	2005	
2268	Levoglucosan (carbon-13) in Ethyl Acetate	2005	

Two new SRMs for serum analyses, SRMs 1957 and 1958, are currently in development (Table 1). These SRMs will meet the expanding needs for the measurement of organic contaminants in human serum. One of these serum materials is a “natural” level material while the other has constituents that were added (i.e., the material is fortified). Specifically, SRM 1957 will be characterized for natural levels of selected PCDDs, PCDFs, PCBs, PBDEs, chlorinated pesticides, toxaphene congeners, polychlorinated naphthalenes, and other halogenated compounds such as pentachloronitrobenzene. SRM 1958 will be characterized for the same suite of analytes though these compounds will be added to the material.

A new biota-related matrix in development with CDC scientists is mother’s milk. Mother’s milk contains approximately ten times the lipid compared to human serum so serum is not a good surrogate for milk. NIST is collaborating with CDC to design two new mother’s milk-based SRMs. As with the new serum SRMs, one material will be a “natural” milk (i.e., no analytes added), and the second material will be from the same milk pool as the “natural” milk but will be fortified with the compounds of interest at appropriate levels. A portion of the same spiking solution that CDC is having prepared in methanol for the serum SRM 1958 will be used for spiking the mother’s milk SRM. The mother’s milk SRMs will be characterized for the same suite of analytes as in the new serum SRMs (Table 1).

Non-biota-related SRMs. A range of environmental SRMs that are not biota or biota-related are also available for the determination of trace pollutants (Table 2). Three of the newest materials are combustion/particle-related. One is a house dust SRM, SRM 2585 (House Dust). SRM 2585 is characterized for selected PAHs, PCB congeners, chlorinated pesticides, and PBDE congeners. 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 dust. Several new solution SRMs in support of measuring contaminants in air particulate matter are also in development (Table 2). These include a solution of hopanes and steranes and solutions of selected nitro-PAHs.

DISCLAIMER: Certain commercial equipment or materials are identified to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

References

1. D. L. Poster, J. R. Kucklick, M. J. Lopez de Alda, B. J. Porter, R. S. Pugh, M. M. Schantz, and S. A. Wise, *Analytical and Bioanalytical Chemistry* 378 (2004):1213-1231.
2. D. L. Poster, J. R. Kucklick, M. M. Schantz, B. J. Porter, S. D. Leigh, and S. A. Wise, *Analytical and Bioanalytical Chemistry* 375 (2003):223-241.
3. J. R. Kucklick, K. J. S. Tuerk, S. S. Vander Pol, M. M. Schantz, and S. A. Wise, *Analytical and Bioanalytical Chemistry* 378 (2004):1147-1151.
4. P. Andrews, K. Headrick, J. C. Pilon, B. Lau, and D. Weber, *Chemosphere* 31 (1995):4393-4402.

5. W. W. Jr. Brubaker, M. M. Schantz, and S. A. Wise, *Fresenius' Journal of Analytical Chemistry* 367 (2000):401-406.