RECENT AND UPCOMING ENVIRONMENTAL MATRIX STANDARD REFERENCE MATERIALS FOR ORGANOHALOGEN COMPOUNDS

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Introduction

The National Institute of Standards and Technology (NIST) has previously produced numerous environmental matrix Standard Reference Materials (SRM) including materials from sediment, mussel tissue, fish tissue, house dust, sewage sludge, air particulate material, whale blubber, and human blood [1]. New SRMs are needed to replenish depleted SRMs and to respond to the need for new types of materials with contemporary concentrations of organohalogen chemicals. NIST has recently released or is in the process of assigning concentration values to several new materials including four air particulate materials and two materials derived from marine biota. The objective of this presentation is to summarize the information available or soon to be available on these new SRMs.

Materials and Methods

Mussel tissue and fish oil were obtained in order to produce the new SRMs. Mussels (*Mytilus edulis*) were collected in 2004 from Boston Harbor, Massachusetts, from the same location where the SRM 1974 series were collected [2]. Mussels were shucked, the resulting tissue was pureed, spread across aluminum trays, and then freeze-dried. After freeze-drying, the mussel tissue was jet-milled, radiation sterilized, and divided into individual jars each containing about 5 g of the dried product to create SRM 2974a. Raw menhaden (*Brevoortia sp*.) oil was collected in 2003 and 2004 from six rendering plants along the US Southeast Atlantic coast and the Gulf of Mexico coast. Oil (6.5 L) was combined, filtered, and then stirred in a large glass bottle overnight prior to dividing into 2 mL ampoules with approximately 1.2 mL of oil per ampoule and labeled as candidate SRM 1588c.

Four air particulate materials were obtained from multiple sources. SRMs 2786 and 2787 Fine Particulate Matter (<4 μm and <10 μm, respectively) were collected in 2005 in Prague, Czech Republic, from an air intake filtration system. The material was size fractionated by using an ultra high volume sampler by adjusting the face velocity across a collection filter. The materials were divided amongst 4 mL amber bottles each containing between 100 mg and 140 mg of SRM. SRM 1649b was prepared from atmospheric particulate material collected in the Washington, DC area in 1976 and 1977 using a baghouse specially designed for the purpose. The particulate material was collected over a period in excess of 12 months, and therefore represents a time-integrated sample. While the sample is not intended to be representative of the area in which it was collected, it should generally typify atmospheric particulate matter obtained from an urban area. The particulate material was removed from the baghouse filter bags by a specially designed vacuum cleaner and combined into a single lot. This lot was passed through a 63 μm (230 mesh) sieve to remove bag fibers and other extraneous materials. The sieved material was then thoroughly mixed in a V-blender and bottled. SRM 1648 was collected in the mid-1970s (prior to the SRM 1649b collection), using a similar collection device but from a different location—St. Louis, Missouri.

Certified values for organohalogen compounds and polycyclic aromatic hydrocarbons were obtained by combining results from at least two independent methods. Methods were varied by using different combinations of extraction, cleanup, or instrumental analysis. For example, PCBs in SRM 2974a were certified using two methods. The first method used Soxhlet extraction followed by GC/MS analysis using a DB-17 column while the second method used pressurized fluid extraction followed by GC/MS analysis on a DB-5 column. Data from the methods were statistically combined to provide a certified value. If the data set did not meet the criteria for assignment of a certified value, reference values were calculated where values may not include all sources of uncertainty. An information value is a value of use to the user but the sources of uncertainty have not been evaluated.

Results and Discussion

Table 1 summarizes the information available for the different materials. The concentrations of organohalogen compounds in the biological materials are generally lower than the ones they replace. For instance, the total PCB concentration in SRM 1588c is 23% of the total PCB concentration in SRM 1588b, which was collected approximately 30 years ago. Likewise, concentration of PCBs were higher in SRM 1974b (collected 1999) versus SRM 2974a (collected 2004) from the same location. For example PCB 153 concentrations were 121 ± 8 ng/g dry mass versus 78.8 ± 2.5 ng/g in SRM 1974b and SRM 2974a. Likewise PBDE 47 concentrations were higher in SRM 1974b (33 ± 2.1 ng/g dry mass) relative to SRM 2974a (14.3 ± 2.8 ng/g dry mass).

Table 1: Information on air particulate and biological Standard Reference Materials.

SRM number	Name	Certified Values	Reference Values	Information Values
2974a	Organics in Freeze-dried Mussel Tissue (*Mytilus edulis*)	22 PAHs, 29 PCBs, 8 organochlorine pesticides, 6 PBDEs	18 PAHs, 14 PCBs, 8 PBDEs, 2 HBCD isomers	
1588c	Organics in Fish Oil	In progress: Data will be available for PCBs, PBDEs, HBCD, and organochlorine pesticides	In progress	In progress
2786	Fine Particulate Matter (<4 μm)	24 PAHs, 3 PBDEs	25 PAHs, 4 nitro PAHs, 4 PBDEs, 3 HBCD isomers, 17 PCDD/Fs	
2787	Fine Particulate Matter (<10 μm)	24 PAHs, 3 PBDEs	25 PAHs, 4 nitro PAHs, 4 PBDEs, 3 HBCD isomers, 17 PCDD/Fs	
1648a	Urban Particulate Matter	In Progress: A similar suite of compounds as shown for SRM 1649b will be determined	In progress	In progress
1649b	Urban Dust	23 PAHs, 13 PCBs, 4 organochlorine pesticides,	38 PAHs, 17 nitro PAHs, 49 PCBs, 13 organochlorine pesticides, 21 toxaphene congeners, PBDE 209	Aliphatic hydrocarbons, hopanes, steranes, ketones

Several brominated flame retardants of interest were quantified in the air particulate materials. For example, α-HBCD and PBDE 209 were determined in SRM 2786 to be 146 ng/g ±7 ng/g and 243 ng/g ± 10 ng/g, respectively. Values of organohalogen compounds in SRM 1649b were similar to those found previously for SRM 1649a, which was a different sieve fraction from the same material. SRM 1648a tends to be about a factor of two lower for the organohalogen compounds relative to the SRM 1649 series.

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Literature Cited

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