A NEW REFERENCE MATERIAL FOR ORGANOHALOGEN MEASUREMENT: SRM 1974C ORGANICS IN MUSSEL TISSUE (*MYTILUS EDULIS*)

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Introduction

Modern environmental analysis methods typically call for the use of natural matrix reference materials with known concentrations of target compounds. Analyses made on these samples help to validate methods and if done routinely, provide a good measure of method performance over time.

The National Institute of Standards and Technology (www.nist.gov) (NIST) prepares and certifies natural matrix Standard Reference Materials (SRMs) for many classes of environmental contaminants, including polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, organochlorine pesticides and brominated flame retardants. One of the most widely used and best-selling environmental SRMs produced by NIST has been the fresh-frozen SRM 1974 Organics in Mussel Tissue (*Mytilus edulis*) series. This material consists of blue mussel (*Mytilus edulis*) that was collected from Dorchester Bay near Boston Harbor, Massachusetts, USA, cryohomogenized and analyzed for numerous man-made contaminants. The material was initially certified in 1989 and has been recollected and recertified as supplies have run out. The history of this series is given in Table 1 (Poster et al. 2004).

Table 1: Summary of SRM 1974 series Organics in Mussel Tissue (*Mytilus edulis*)

Collection and Certificate (Date)	SRM Number	Certified Values	Reference Values	Information Values
1987/(1989)	1974	9 PAHs	19 PAHs; 13 PCB congeners; 9 pesticides; 36 trace elements	none
1992/(1995)	1974a	15 PAHs; 20 PCB congeners; 7 pesticides; total mercury	18 PAHs; 4 PCB congeners, 16 aliphatic hydrocarbons; 32 trace elements	Carbohydrates; fat; fatty acids, calories
1999/(2003)	1974b	22 PAHs; 31 PCB congeners; 7 pesticides; total mercury	16 PAHs; 8 PCB congeners; 6 pesticides; total extractable organics; 11 trace elements	none
2004/(2012)	*1974c	36 PAHs; 37 PCB congeners; 11 pesticides; 5 PBDE congeners	12 PAHs; 14 PCB congeners; 2 pesticides; moisture	none

*certification is still progress; the number of compounds with certified or reference values will change on final certificate and will include trace elements

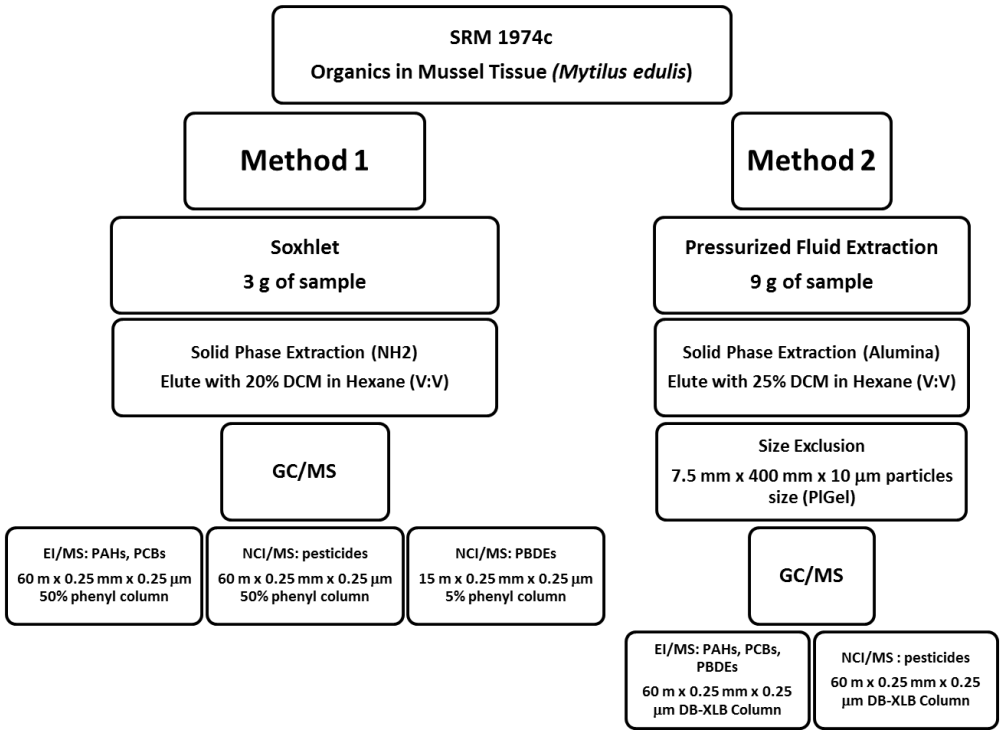
The supply of SRM 1974b that was collected in 1999 is nearly exhausted. Therefore NIST is preparing a new SRM using a new batch of mussel tissue collected from the same location as the previous reference materials in the series. This paper gives a summary of measurements made to date on the material for selected compounds and compares values to the previous SRM 1974 materials.

Materials and methods

Approximately 10,000 individual mussels were collected by hand from Dorchester Bay, Massachusetts USA in 2004. Mussels were shipped to the NIST Environmental Specimen Bank in Charleston, South Carolina USA where they were held in a frozen state until processing. Mussels were shucked, and the meat was transferred to Teflon bags. Teflon bags containing the mussel tissue were frozen in liquid nitrogen vapor freezers until processing. Approximately 70 kg of the material was processed into SRM 1974c. The material from each Teflon bag was first crushed using a weighed "smasher device" (Pugh et al. 2006). The crushed tissue was then transferred to a Palla VM-KT cryomill which is a specialized apparatus that allowed the tissue to be milled in a frozen state. The material was milled several times to provide a homogeneous sample. After milling, the mussel tissue was stored in Teflon bags until bottling. Approximately 4000 jars were produced each containing 10 g of frozen mussel tissue.

Two methods were used to measure the compounds of interest in the samples. A summary of each methods used is given in Figure 1.

Figure 1: Methods used to certify SRM 1974c.



Homogeneity of the material was evaluated using method 1 (Figure 1) by analyzing two subsamples from each of 10 jars of SRM 1974c that were randomly selected from the lot. Previous lots of SRM 1974 (Table 1) were

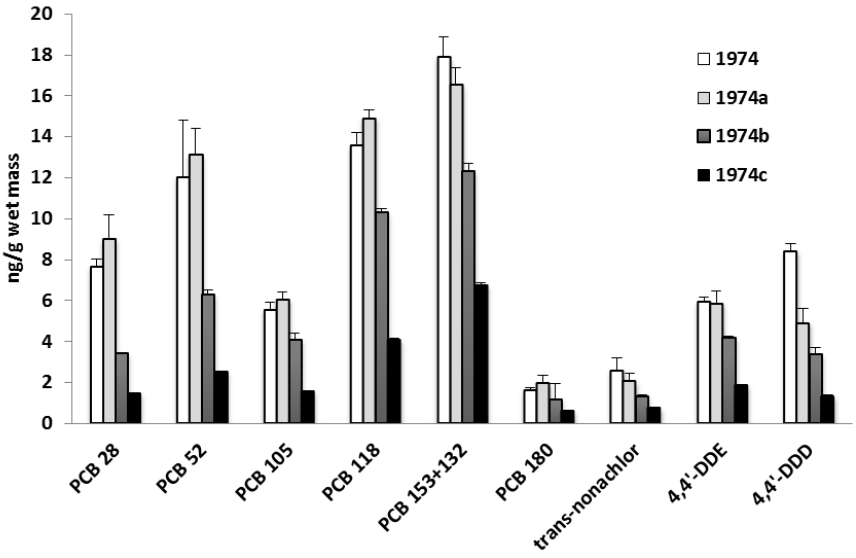
re-analyzed using Method 1 to assess analyte stability and to detect temporal trends at the collection site as all SRM 1974 collections were from the same mussel bed.

Results and Discussion

Certified or reference concentrations of several persistent organic pollutants (POPs) in the SRM 1974 series are shown in Figure 2. POPs have declined substantially in different collections of SRM 1974. The lowest concentrations were observed in SRM 1974c which was collected in 2004 with concentrations of POPs in Figure 2 approximately 28% of the values in the original SRM 1974.

SRM 1974c has more compounds with either certified or reference values compared to its predecessor, SRM 1974b (Table 1). In addition, five PBDE congeners (congeners 17, 25, 47, 49, and 99) were certified in SRM 1974c. PBDE 47 was the dominant PBDE congener with value of 0.94 ng/g wet mass ± 0.02 ng/g wet (95% confidence interval) followed by PBDE 99 (0.375 ng/g wet ± 0.004 ng/g wet). The number of PCB congeners with certified values has increased from 38 in SRM 1974b to 51 in SRM 1974c. Given the large number of organohalogen compounds certified in SRM 1974c and the large number of PAH compounds also with certified values, this material should prove to be useful as an analytical control material for the environmental measurement community.

Figure 2: Concentrations of selected POPs in the SRM 1974 series. Bars are certified or reference values (PCB 28 only in SRM 1974) with error bars being the upper 95% uncertainty limit.



References

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