CURRENT ACTIVITIES IN ENVIRONMENTAL STANDARD REFERENCE MATERIALS FOR TRACE HALOGENATED ORGANIC POLLUTANTS

Michele M. Schantz, Dianne L. Poster, and Stephen A. Wise

Analytical Chemistry Division, National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899-8392 USA

Introduction

Since 1980, the National Institute of Standards and Technology (NIST) has issued a number of Standard Reference Materials (SRMs) for use in determination of organic contaminants in environmental samples. These SRMs include simple calibration solutions that contain a number of analytes and are useful for calibrating the measurement system and natural matrix materials that are useful for validating analytical procedures and providing quality control of organic contaminant analyses.

Calibration solutions are available containing 28 polychlorinated biphenyl (PCB) congeners and 15 chlorinated pesticides at two levels approximately an order of magnitude different in concentration. Additional calibration solutions that will soon be available include one containing 11 additional PCB congeners and one containing 9 additional chlorinated pesticides. In addition, NIST is preparing a number of calibration solutions in support of the externalization of U.S. EPA's Water Proficiency Testing Studies. These solutions will include Aroclors in methanol and transformer oil, chlorinated pesticides in methanol, haloacetic acids in MTBE, and carbamates in acetonitrile.

Current activities in the area of natural matrix materials include updating the Certificates of Analysis (COAs) for a number of existing SRMs and issuing several new materials. These activities are summarized in Table 1.

Table 1. Recently released natural matrix SRMs for trace organic halogenated pollutants

SRM number		Compound classes with **certified values** and reference values
1588a	Organics in Cod Liver Oil	**PCBs, Pesticides**, PCDDs/ PCDFs
1589a	Polychlorinated Biphenyls / Pesticides in Human Serum	**PCBs, Pesticides**, PCDDs/PCDFs
1649a	Urban Dust	**PAHs, PCBs, Pesticides**, PCDDs/PCDFs, trace elements
1939a	Polychlorinated Biphenyl (Congeners) in River Sediment	**PCBs, Pesticides**
1941a	Organics in Marine Sediment	**PAHs, PCBs, Pesticides**, Trace Elements
1944	NY/NJ Waterway Sediment	**PAHs, PCBs, Pesticides**, PCDDs/ PCDFs, **trace elements**
1945	Organics in Whale Blubber	**PCBs, Pesticides**
1974a	Organics in Mussel Tissue (*Mytilus edulis*)	**PAHs, PCBs, Pesticides, Methyl-Hg, Hg** trace elements
2974	Organics in Freeze-dried Mussel Tissue	**PAHs, PCBs, Pesticides, Methyl-Hg, Hg**

trace elements

In addition to those SRMs listed in Table 1, three materials are in preparation including the replacement sediment for SRM 1941a, which will be SRM 1941b, and two fish tissue materials, SRM 1946 and SRM 1947. SRM 1941b was collected from the same site as SRM 1941a and will have a similar suite of compounds certified. SRM 1946 and SRM 1947 are being prepared from lake trout fillets collected in Lake Superior and Lake Michigan, respectively, and will be issued as fresh frozen homogenates.

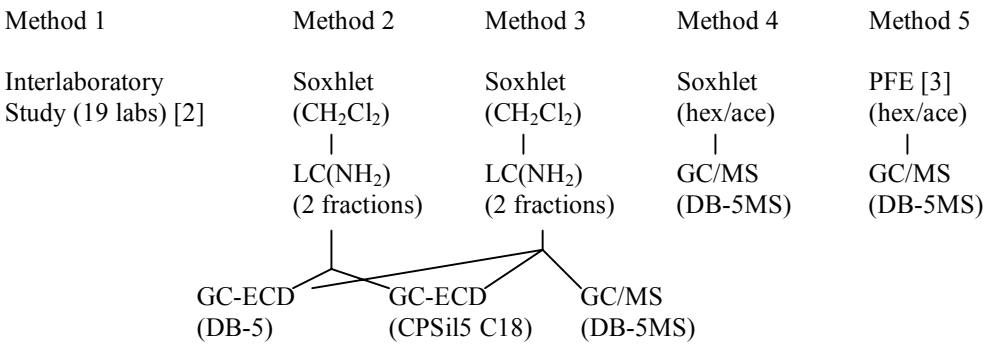
Materials and Methods

The SRMs mentioned in this abstract are available from the Standard Reference Materials Program (SRMP), NIST (Gaithersburg, MD). The methods used for certification are described in detail in the corresponding Certificates of Analysis available from SRMP [1].

Results and Discussion

During the certification of SRMs at NIST, the results from at least two "chemically independent" analytical techniques are typically used to determine the certified concentrations of the analytes. This includes independent extraction, isolation, and chromatographic techniques. An example is given in Figure 1 for the certification of the PCBs and chlorinated pesticides in SRM 1944, a sediment.

Figure 1. Methods used for the analysis of PCB congeners and pesticides in SRM 1944



For this SRM, data from an interlaboratory exercise (Method 1) coordinated by NIST and the National Oceanic and Atmospheric Administration (NOAA) were used along with data from four methods done at NIST. At NIST, both Soxhet extraction and pressurized fluid extraction (PFE), using either dichloromethane or hexane/acetone (1:1, v:v), were used followed by the isolation of two fractions, one containing the PCBs and lower polarity pesticides and the other the more polar pesticides for the two methods using gas chromatography with electron capture detection as the final analysis step.

There are currently three sediment SRMs available, two harbor sediments (SRM 1941a and SRM 1944) which differ in concentration by about an order of magnitude and a river sediment (SRM 1939a) which is representative of a PCB spill. There is an urban dust, SRM 1649a, which was just recently reissued with certified concentrations for selected PCB congeners and chlorinated

pesticides. Three marine tissue materials are available, two provided as fresh frozen materials (SRM 1945, whale blubber, and SRM 1974a, mussel tissue) and one as freeze-dried (SRM 2974, which is the freeze-dried version of SRM 1974a). SRM 1588a, a cod liver oil, was also just recently reissued with an expanded list of PCB congeners and chlorinated pesticides having certified concentrations. Certification measurements are currently underway, in conjunction with the Centers for Disease Control (CDC), on a new issuance of freeze-dried human serum which will have certified concentrations for natural levels of selected PCB congeners and chlorinated pesticides along with reference values (determined using one method at CDC) of selected PCDD and PCDF congeners.

Table 2 shows the relative concentrations of selected PCB congeners and chlorinated pesticides in five of the above mentioned materials. Only a limited number of congeners and pesticides are shown in this table, but typically 20 to 25 PCB congeners and 5 to 10 pesticides have values listed in the Certificates of Analysis.

Acknowledgements
The authors acknowledge the help of Donald Patterson and Wayman Turner at CDC on the certification of SRM 1589a.

References

1. Certificates of Analysis available from http://ts.nist.gov/srm
2. Parris, R.M., Schantz, M.M., and Wise, S.A., NIST/NOAA NS&T/EPA EMAP Intercomparison Exercise Program for Organic Contaminants in the Marine Environment: Description and Results of 1995 Organic Intercomparison Exercises, NOAA Tech Memo NOS ORCA 104, **1996**.
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Table 2. Concentrations (ng/g dry weight) of Selected PCB Congeners and Chlorinated Pesticides in Sediment, Urban Dust, and Mussel Tissue SRMs

Compound	SRM 1941a	SRM 1944	SRM 1939a	SRM 1649a	SRM 1974a
PCB 28	*9.8 ± 3.7*	80.8 ± 2.7	*2461 ± 78*	18.5 ± 1.2	*79 ± 15*
PCB 52	6.89 ± 0.56	79.4 ± 2.0	4320 ± 130	24.65 ± 0.97	115 ± 11
PCB 99	4.17 ± 0.51	37.5 ± 2.4	380 ± 96	9.58 ± 0.69	70.9 ± 4.0
PCB 101/90	11.0 ± 1.6	73.4 ± 2.5		52.9 ± 1.0	128.3 ± 9.7
PCB 105	3.65 ± 0.27	24.5 ± 1.1	201 ± 28	8.63 ± 0.80	53.0 ± 3.4
PCB 118	10.0 ± 1.1	58.0 ± 4.3	423 ± 88	25.7 ± 1.5	130.8 ± 3.6
PCB 138/163/164	13.38 ± 0.97	62.1 ± 3.0	258.1± 6.9	69.7 ± 7.5	133.5 ± 9.5
PCB 153	17.6 ± 1.9	74.0 ± 2.9	297 ± 19	82.5 ± 8.0	145.2 ± 7.6
PCB 170/190	3.00 ± 0.46	22.6 ± 1.4	107 ± 17	30.8 ± 2.2	5.5 ± 1.1
PCB 180	5.83 ± 0.58	44.3 ± 1.2	140.3± 6.1	78.7 ± 8.2	17.1 ± 3.8
cis-chlordane	2.33 ± 0.56	16.51± 0.83	4.8 ± 1.3	34.88± 0.42	17.2 ± 2.8
trans-nonachlor	1.26 ± 0.13	8.20 ± 0.51		27.6 ± 1.6	18.0 ± 3.6
4,4'-DDE	6.59 ± 0.56	*86 ± 12*		40.4 ± 1.7	51.2 ± 5.5
4,4'-DDD	5.06 ± 0.58	*108 ± 16*	5.50± 0.97	34.01± 0.48	43.0 ± 6.3
4,4'-DDT	*1.25 ± 0.10*	119 ± 11	2.72± 0.42	212 ± 15	3.91± 0.59

[a] Concentrations are the certified concentrations or *reference concentrations* as determined by statistically combining the data from the different methods used for certification. For each SRM the method used for combining the data and the definition of the associated uncertainties are given in the Certificates of Analysis[1].