

NEW REFERENCE MATERIALS FOR HUMAN BLOOD AND MILK  
PRODUCED BY THE NATIONAL INSTITUTE OF STANDARDS AND  
TECHNOLOGY FOR THE ANALYSIS OF PERSISTENT ORGANIC  
POLLUTANTS

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### Introduction

Assessing the levels of persistent pollutants and their metabolites in people is important both for estimating toxic potential and for determining the effectiveness of controls through monitoring concentrations over time. Consequently, cross sectional investigations such as the National Health and Nutritional Examination Study (NHANES; [www.cdc.gov/nchs/nhanes.htm](http://www.cdc.gov/nchs/nhanes.htm)) have been developed to address such issues through the examination of blood from donors representing a cross section of the US population. Studies such as this require the analysis of a large number of human samples in a setting that provides the highest quality possible. Standard Reference Materials (SRMs) are an important component for assessing the performance of methods applied to the determination of persistent contaminants in samples of human origin. The most recent serum SRM for organic contaminants produced by the National Institute of Standards and Technology (NIST), SRM 1589a, was prepared in 1996. Since this time, polychlorinated biphenyl (PCB) levels in the U.S. population have declined while polybrominated diphenyl ether (PBDE) levels have increased limiting the usefulness of this material<sup>1</sup>. To address this issue, the Centers for Disease Control and Prevention (CDC) and NIST are collaborating on the collection and value assignment of human serum and human milk SRMs. This paper describes the target compounds and approaches to be used for production of these SRMs.

### Materials and Methods

Human serum was collected from eight states in the U.S. and pooled into a 200 L sample. The serum was thoroughly mixed and then split into equal portions. One portion was aliquoted into approximately 9000, 10.7 mL aliquots and freeze-dried. The freeze-dried material was shipped to NIST and stored in a walk-in refrigerator. The remaining 100 L of serum was fortified with the compounds listed in Table 1. This spiking solution for this was prepared by combining 12 stock solutions that were diluted with methanol then added to the 100 L of serum. The fortified serum was thoroughly mixed, divided into 10.7 mL aliquots, then freeze-dried as above. The freeze-dried, fortified serum was also stored in a walk-in refrigerator upon receipt at NIST. The non-fortified material will be SRM 1957, and the fortified material will be SRM 1958.

For the human milk SRM, approximately 100 L of human milk was collected from milk banks across the US, mixed, and split into two 50 L portions. The portions were treated similarly to the serum described above in that one portion was fortified with the compounds listed in Table 1, while one portion was not fortified (this material will not be freeze-dried.). The non-fortified material will be SRM 1953 while the fortified material will be SRM 1954.

The above SRMs will be value assigned using the approach described in detail by Wise et al.<sup>2</sup>. For the certification of concentration values, the results from multiple independent methods will be combined. Values assigned for polychlorinated biphenyls, for example, will be the result of

## Analytical quality control and assurance

several combinations of extraction, cleanup, and determination using gas chromatography with mass spectrometry (GC/MS) with different GC stationary phases (e.g.,<sup>3</sup>).

Table 1: Summary information on compounds added to the fortified serum and milk reference materials

Compound Class	Number of Compounds	Concentration Range in Serum or Milk
Planar Organochlorine Compounds (chlorinated dioxins/furans and coplanar PCBs)	22	0.10 to 2.40 pg/mL
Organochlorine Pesticides	22	500 pg/mL
Chlorobenzenes and octachlorostyrene	8	500 pg/mL
Polychlorinated Biphenyls (PCBs)	38	50 to 500 pg/mL
Brominated Flame Retardants including HBCD, BTBPE	25	500 pg/mL
Polychlorinated Naphthalenes (PCNs)	9	1 pg/mL
Halogenated Phenolic Compounds	12	500 pg/mL
Hydroxylated PCBs	5	500 pg/mL
Brominated Dioxins and Furans	17	0.05 pg/mL
Chloro-bromo Dioxins and Furans	8	0.05 pg/mL
Toxaphene Congeners	6	500 pg/mL

### Results and Discussion

Work is currently underway to begin the value assignment process for the human serum and milk SRMs. Samples of the candidate serum and milk SRMs will be analyzed using multiple analytical methods at NIST (both in Maryland and South Carolina laboratories) and at CDC. In addition to the analytes listed in Table 1, a number of perfluorinated alkyl acids will also be determined in the non-fortified SRMs. Currently no RMs are available for the perfluorinated compounds in human serum or milk, so value assignment of these new SRMs will help to improve perfluorinated compound measurements

### Literature Cited

1. Sjödin, A; Jones, RS; Focant, JF; Lapeza, C; Wang, RY; McGahee, EE; Zhang, YL; Turner, WE; Slazyk, B; Needham, LL; Patterson, DG *Environ Health Perspect* **2004**, *112*, 654-658.
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