

CREATION OF NEW SOIL SRM FOR METALS AND ORGANICS

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NIST

**National Institute of
Standards and Technology**

Technology Administration
U.S. Department of Commerce

HOW DO WE KNOW THAT THE “MEASUREMENT SYSTEM” WORKS?



✓ **Laboratory Certification**


- On-site audits every 2-4 years

✓ **Performance Testing Samples**

- Usually not natural samples containing actual concentrations of contaminants as they exist in the environment

✓ **Method and Sample Specific QC**

METHOD QC: INSTRUMENTATION-ORIENTED



✓ Instrument Stability

- Pre-analysis Performance Checks

✓ Calibration Accuracy

- Analysis of Second Source Materials
- LCS

✓ Calibration Stability


- CCV

✓ Low Level Detection

- Blanks, MDLs, MRLs, LLOQ Check Samples

Control limits in EPA methods are generally established by “consensus” and not by empirical data

METHOD QC: SAMPLE-ORIENTED



✓ Contamination

- Field, Trip and Lab. (Prep.) Blanks

✓ Sample Homogeneity

- Matrix Duplicates

✓ Method Accuracy

- Matrix Spikes*

***This is the only indicator that directly addresses the question “are we accurately measuring how much contaminant is in the sample”**

BUT THERE ARE ISSUES WITH MATRIX SPIKES



- ✓ Only 1/20 of a sample batch is spiked; how representative is that sample spike of the behavior of the entire batch?
- ✓ Spiking regime usually involves the addition of a “simple” chemical, usually as a liquid, often to a non-aqueous sample. This raises many questions, including:
 - Equilibration of the spike
 - **How well does recovery of the spike represent the behavior of indigenous analyte?**

HOW RMs CAN AUGMENT CURRENT METHOD QC

- ✓ RMs (especially SRMs produced by NIST) are routinely utilized in method QC in non-EPA method compendiums (AOAC, ASTM, NIOSH)
- ✓ NIST SRMs are routinely used to validate USEPA SW846 methods and their use has been required as part of CLP projects
- ✓ **NIST Standard Reference Material® (SRM)**
 - **NIST Certified Value** - A value reported on an SRM certificate or certificate of analysis for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been fully investigated or accounted for by NIST.
 - **NIST Reference Value** - A best estimate of the true value provided on a NIST certificate, certificate of analysis, or report of investigation where all known or suspected sources of bias have not been fully investigated by NIST.
- ✓ **Both the Certified and Reference Values can be effective QC tools**

HOW TO USE RMs TO EVALUATE USEPA METHODS

- ✓ Reference values, found in Appendices to NIST SRM COAs, often provide data obtained by USEPA Methods
- ✓ These data can be compared with the Certified Values on the COA to predict how effective digestion and/or determinate methods are in measuring concentrations of contaminants in environmental samples.
- ✓ Reference values for NIST SRM 2710A (Montana Soil):

Element	% Leach
Ag	91
As	92
Cd	86
Cr	41
Co	64
Cu	95
Pb	93
Mn	77
Ni	69
Sb	18
Se	200
Tl	213
V	48
Zn	90

“GENERIC” BARRIERS TO RM USE



- ✓ Despite the use of SRMs to validate methods, only Method 7199 [Cr(VI) by IC] requires QC involving RMs
- ✓ Too expensive
- ✓ Analyte concentrations do not match anticipated sample values (SRMs values too high)
- ✓ Current SRM inventory does not cover the range of all SW846 methods
- ✓ NELAC/TNI standard does not require RM use

WHY WE NEED ANOTHER SOIL SRM

- ✓ Current NIST soil SRM inventory is exclusively from Western US sources (Montana, CA, ...)
- ✓ Existing SRMs do not contain all analytes of interest
- ✓ COA parameter concentrations are often much higher than that found in “typical” soil samples
- ✓ **SRMs are not now used for method QC for these and other reasons**



**OBJECTIVE: TO CREATE A NEW SRM FROM EASTERN US SOURCES
CONTAINING A WIDE RANGE OF INORGANIC AND ORGANIC
CONTAMINANTS AT TYPICAL ENVIRONMENTAL CONCENTRATIONS**

NIST SRM 2706

PRIMARY PROJECT STAKEHOLDERS



✓ NIST

Gaithersburg, MD

✓ USGS

Denver, CO

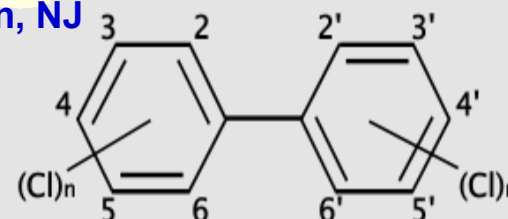
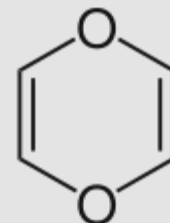
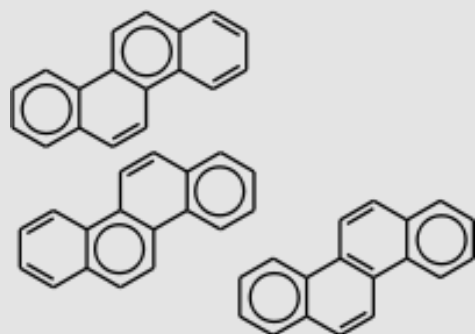
✓ USEPA

Research Triangle Park, NC

✓ NJDEP

Trenton, NJ

SAMPLING SITES AND PRIMARY ANALYTE CONCENTRATIONS



Site	Location	Primary Contaminant(s)	Concentration Range (mg/kg)
Paul Tank	Burlington, NJ	PCBs	0.01 – 84
Mariners Marsh	Staten Island, NY	PAHs	0.04 – 10,000
Melon Leasing	Kearney, NJ	Dioxins/Furans	0.0001- 0.2
Raritan Bay Slag	Sayreville, NJ	As, Pb, other metals	3-20,000

PROJECT PROCESS I

- ✓ Assemble group of interested parties representing government, academia and the certified laboratory community
- ✓ Identify key contaminants of concern
- ✓ “Suitable” sampling locations have been located
 - Publicly-funded sites
 - Easy access for sampling
- ✓ Analyze retains to confirm concentrations
 - USEPA R2
 - Pace Analytical
- ✓ Select source for uncontaminated bkg. soil



PROJECT PROCESS II



- ✓ Using the results from the preliminary analyses, develop a “recipe” for mixing/blending samples from various sites to create a SRM with the desired analyte concentrations - **10-15X “typical analyte concentrations”**
- ✓ Samples shipped to USGS; ~4000 80g. samples have been prepared
- ✓ USGS and NIST studies have/will confirm “bottle-to-bottle” geochemical and chemical homogeneity
- ✓ NIST analyses will provide the certified values for metals
 - INAA – 3 Elements
 - ICP-MS – 9 Elements
 - ID ICP-MS – 1 Element (Hg)
 - ICP-OES – 10 elements
 - XRF – 16 Elements
 - Other labs. will provide data to be used for reference and information values

PROJECT PROCESS III

- ✓ NIST will provide certified and appendix values for organics

Measurand	Method 1	Method 2	Type
PCBs, OCs, BDEs	GC/MS HML	GC/MS HML	Certified
PAHs	GC/MS Gaithersburg	GC/MS Gaithersburg	Certified
Dioxins/Furans	Intercomparison Study		Appendix
PFCs	LC-MS/MS HML		Reference

- ✓ An intercomparison study will develop reference values using USEPA SW846 methods for metals and organics – **participating labs. have been asked to quantify analytes below their traditional reporting levels**

PARTICIPATING LABORATORIES



- ✓ **Accutest – Phil Worby**
- ✓ **Alpha Laboratories – Jim Todaro**
- ✓ **ALS – Ron McLeod**
- ✓ **EMSL – Bill Chamberlin**
- ✓ **USEPA Region 4 – Floyd Wellbourne**
- ✓ **Pace Analytical – Bob Wyeth**
- ✓ **TestAmerica – Mark Bruce**

TARGET CONCENTRATIONS - METALS

Element	ICP-MS 1	ICP-MS 2	Units
Ag	< 1	<1	mg/kg
As	30.25	30	mg/kg
Be	0.842		mg/kg
Cd	0.307		mg/kg
Cr	40.79		mg/kg
Cu	87.78	86.82	mg/kg
Hg	0.133 IDMS		mg/kg
Ni	20.64		mg/kg
Pb	625.1	654.1	mg/kg
Se			mg/kg
Sb	145.8	158.1	mg/kg
Tl	0.244		mg/kg
Zn	133.0	129.0	mg/kg

Only EPA Regulated Pollutants shown above
Data will also be available for other major, transition & rare earth elements

TARGET CONCENTRATIONS - ORGANICS

Blended Soil PCBs			
Source	Primary Group	Predicted 2706 µg/kg	Reporting Limit µg/kg
Site 1	Aroclor 1260	4200	16
Blended Soil PAHs			
Source	Primary Group	Predicted 2706 µg/kg	Reporting Limit µg/kg
Site 2	Benzofluoranthene	301	20.1
Site 2	Fluoranthene	117	20.1
Site 2	Napthalene	52500	100
Blended Soil Dioxins			
Source	Primary Group	Predicted 2706 ng/kg	Reporting Limit ng/kg
Site 3	TCDF	23333	1100
Site 3	TCDD	360	1200
Site 3	PeCDF	86667	3000
Site 3	PECDD	547	3000
Site 3	HxCDF	226667	11000
Site 3	HxCDD	1533	3300
Site 3	HpCDF	933333	13000
Site 3	HpCDD	933	2500

ADDITIONAL CONSIDERATIONS



- ✓ VOCs not being considered due to obvious stability concerns
- ✓ Other organics, such as PFCs, will be analyzed by NIST
- ✓ If the cost of the product is an issue, secondary RMs with a link to the NIST SRM could be developed

THANK YOUs



- ✓ Lara Phelps – USEPA
- ✓ NELAC for funding my travel
- ✓ All who helped with sample acquisition & distribution
- ✓ Labs. who have and are helping with analyses
- ✓ Dr. Bernie Wilk