

Development of SRM 2706 (New Jersey Soil, Organics and Trace Elements), a New Reference Material for the Monitoring and Assessment of Soils.

Stephen E. Long

National Institute of Standards and Technology, 331 Fort Johnson Road, Charleston, SC

Stuart J. Nagourney (Ret)

New Jersey Department of Environmental Protection, 401 East State Street, Trenton, NJ

Stephen A. Wilson

U.S. Geological Survey, PO Box 25046, Denver, CO

Lara P. Phelps

U.S. Environmental Protection Agency, 109 T.W. Alexander Drive, Research Triangle Park, NC

National Institute of Standards and Technology - History

Origins

1824 Office of Standard Weights and Measures

1901 National Bureau of Standards (NBS)

Federal Government's first physical science research laboratory
Number of Staff - 12

1988 National Institute of Standards and Technology (NIST) 1988



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NIST TODAY: MISSION

“To promote U.S. innovation and industrial competitiveness by advancing measurement science, standards, and technology in ways that enhance economic security and improve our quality of life”

Early Driver for New Standards



1904

- Out-of-town fire companies arriving at a Baltimore fire cannot couple their hoses to the hydrants. 1526 buildings destroyed

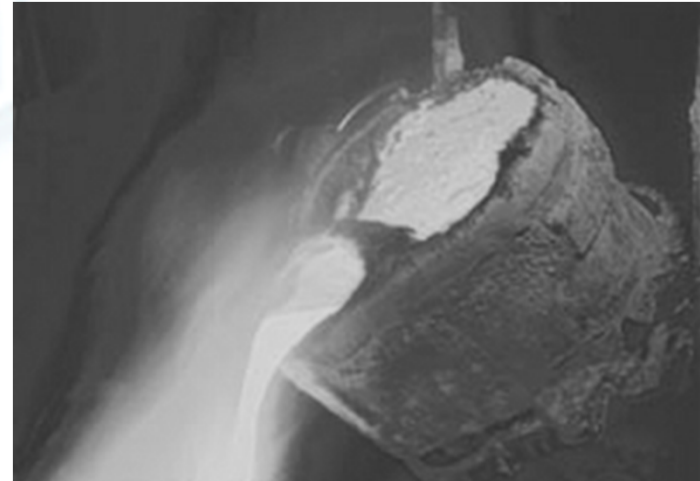
1905

- National Fire Protection Association adopted NBS-developed national hose coupling standard

Birth of the “Standard Samples” Program

1905

Standard samples program begins with “standardized irons” in collaboration with **the American Foundrymans Association**



1906

At the request of the **Association of American Steel Manufacturers**, the Bureau began work on certification of 17 types of steel

- By 1951, there were 502 Standard Samples, 98 of these were steels
- Today there are more than 1400 different SRMs



Standard Reference Materials (SRMs)

- **Standard Reference Materials (SRMs)** are **Certified Reference Materials (CRMs)** issued by the National Institute of Standards and Technology (NIST)
- Homogeneous, stable materials well-characterized for one or more chemical and/or physical properties
- Assist laboratories worldwide in validating analytical measurements of chemical and physical composition

How Reference Materials Can Augment Current Method QC

- Reference materials (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 US EPA SW846 methods and their use has been required as part of CLP projects
- **NIST SRMs**
 - **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.

Soil and Sediment SRMs

Soils

- SRM 2709a San Joaquin Soil
- SRM 2710a Montana I Soil
- SRM 2711a Montana II Soil
- SRM 2700 Hexavalent Chromium in Contaminated Soil (Low)
- SRM 2701 Hexavalent Chromium in Contaminated Soil (High)



Sediments

- SRM 1646a Estuarine Sediment
- SRM 1941b Organics in Marine Sediment
- SRM 1944 New York/new Jersey Waterway Sediment
- SRM 2702 Inorganics in Marine Sediment
- RM 8704 Buffalo River Sediment

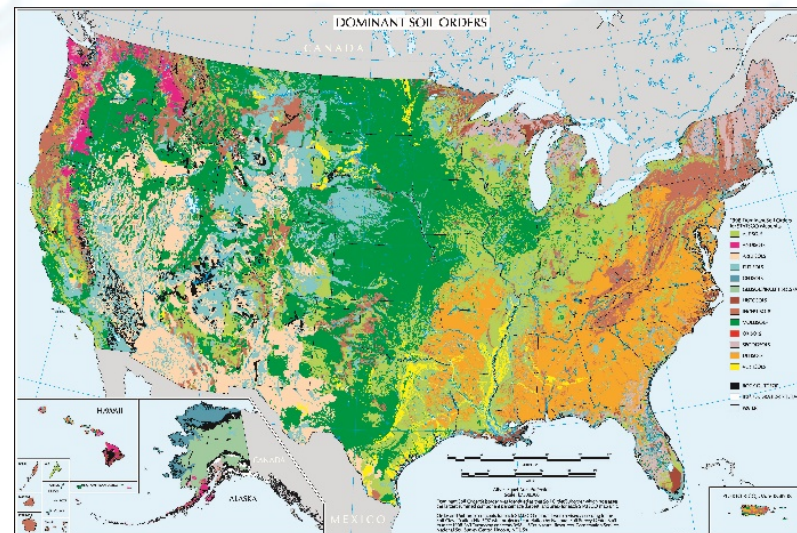


Why We Need Another Soil SRM

Current NIST soil SRM inventory is mostly from Western US sources (Montana, California)

Existing soil SRMs do not contain all analytes of interest, eg organics

Concentrations are often much higher than found in “typical” soil samples



Objective: To create a new soil SRM from Eastern US Sources containing a wide range of inorganic and organic contaminants at typical environmental concentrations

NIST SRM 2706, New Jersey Soil Organics and Trace Elements

Primary Project Stakeholders

NJDEP

Trenton, NJ

NIST

Gaithersburg, MD
Charleston, SC

USGS

Denver, CO

US EPA

Research Triangle Park, NC

US EPA Region 2

Edison, NJ

Pace Analytical

Greensburg, PA

SRM 2706 Project Process I

- Assemble group of interested parties representing government, academia and the certified laboratory community
- Identify key contaminants of concern
- Locate “suitable” sampling locations
 - Publicly-funded sites
 - Easy access for sampling
- Analyze sample retains to assess concentrations
 - USEPA Region 2
 - Pace Analytical
- Select source for uncontaminated background soil (Rutgers University, NJ)

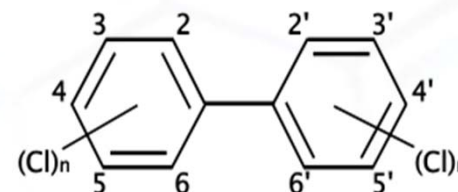
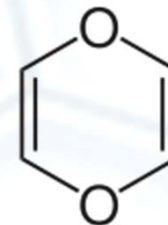
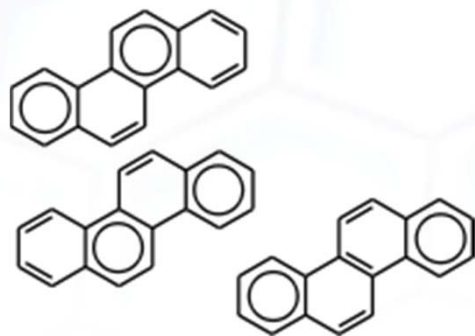


SRM 2706 Project Process II

- Using the results from the preliminary analyses, develop a “recipe” for mixing/blending samples from the various sites to create a SRM with the desired analyte concentrations - **10-15X “typical analyte concentrations”**
- Ship samples to USGS for processing
- USGS and NIST studies assess “bottle-to-bottle” chemical homogeneity and stability
- Value assignment at NIST – primary methods (eg IDMS) / combined methods
- Intercomparison study to develop ancillary values using SW846 methods for elements and organics

SRM 2706 Soil Sampling Sites

As, Pb,
trace elements



Site	Location	Primary Contaminants	Concentration Range (mg/kg)
1. Paul Tank	Burlington, NJ	PCBs	0.01- 84
2. Mariners Marsh	Staten Island, NY	PAHs	0.04 -10,000
3. Melon Leasing	Kearney, NJ	Dioxins/Furans	0.00001 – 0.2
4. Raritan Bay Slag	Sayreville, NJ	As, Pb, other metals	3 – 20,000



Processing

- Collected material retains were transferred to five-gallon plastic buckets and shipped to the USGS laboratory with blank soil for processing.
- At USGS, the materials were dried at room temperature, disaggregated, and sieved to remove coarse material (≥ 2 mm). The dried materials were then ball-milled in 50 kg portions, and blended with dried background soil using a cross-flow V-blender for mixing.
- Blended soil SRM was radiation sterilized prior to bottling as 50 g units using a custom-designed spinning riffler.
- 2400 units produced

SRM 2706 Certified Values Inorganic

SRM 2706 = 23
SRM 2709a = 19



Constituent	Value	<i>U</i>	Units	<i>k</i>	Assigned	Methods
Aluminum	2.70	0.19	%	2	Certified	ICP-OES; WDXRF
Calcium	0.588	0.046	%	2	Certified	ICP-OES; WDXRF
Iron	2.22	0.15	%	2	Certified	ICP-OES; INAA; WDXRF
Magnesium	0.289	0.014	%	2	Certified	ICP-OES; WDXRF
Potassium	0.946	0.028	%	2	Certified	ICP-OES; WDXRF
Silicon	39.17	0.47	%	2	Certified	WDXRF
Sodium	0.268	0.012	%	2	Certified	ICP-OES; WDXRF
Titanium	0.290	0.042	%	2	Certified	ICP-OES; WDXRF
Antimony	149	11	mg/kg	2	Certified	ICP-MS; INAA
Barium	319	43	mg/kg	2	Certified	ICP-OES; WDXRF
Chromium	60.1	5.4	mg/kg	2	Certified	INAA; WDXRF
Cobalt	5.99	0.23	mg/kg	2	Certified	ICP-MS; INAA
Copper	88.1	6.8	mg/kg	2	Certified	ICP-OES; WDXRF
Lead	653	36	mg/kg	2	Certified	ICP-MS; WDXRF
Manganese	244	15	mg/kg	2	Certified	ICP-OES; WDXRF
Mercury	0.1329	0.0033	mg/kg	2	Certified	CV ID-ICP-MS
Nickel	22.8	5.4	mg/kg	2	Certified	ICP-OES; WDXRF
Phosphorus	407	21	mg/kg	2	Certified	ICP-OES; WDXRF
Rubidium	37.6	1.9	mg/kg	2	Certified	ICP-MS; INAA
Strontium	60.3	3.1	mg/kg	2	Certified	ICP-OES; WDXRF
Vanadium	51.9	6.5	mg/kg	2	Certified	ICP-OES; WDXRF
Zinc	135.4	6.9	mg/kg	2	Certified	ICP-OES; WDXRF
Zirconium	303	25	mg/kg	2	Certified	ICP-MS; WDXRF

Key to Methods

CV ID-ICP-MS Cold vapor isotope dilution inductively coupled plasma mass spectrometry at NIST
 ICP-MS Inductively coupled plasma mass spectrometry at the USGS
 ICP-OES Inductively coupled plasma optical emission spectrometry at NIST and USGS
 INAA Instrumental neutron activation analysis at NIST
 WDXRF Wavelength dispersive X-ray fluorescence spectrometry at NIST and USGS

SRM 2706 Reference Values Inorganic

SRM 2706 = 29

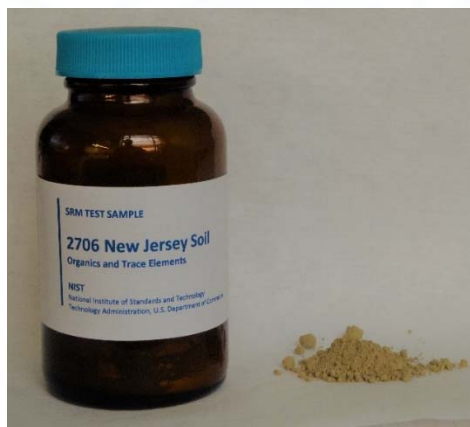
SRM 2709a = 15



Constituent	Value	<i>U</i>	Units	<i>k</i>	Assigned	Methods
Sulfur	0.217	0.012	%	2	Reference	ICP-OES
Arsenic	30.3	2.7	mg/kg	2	Reference	ICP-MS
Beryllium	0.84	0.17	mg/kg	2	Reference	ICP-OES
Bismuth	0.159	0.012	mg/kg	2	Reference	ICP-MS
Cadmium	0.31	0.14	mg/kg	2	Reference	ICP-MS
Cerium	34.5	1.7	mg/kg	2	Reference	ICP-MS
Cesium	1.35	0.10	mg/kg	2	Reference	ICP-MS
Dysprosium	2.41	0.21	mg/kg	2	Reference	ICP-MS
Erbium	1.45	0.15	mg/kg	2	Reference	ICP-MS
Europium	0.602	0.038	mg/kg	2	Reference	ICP-MS
Gadolinium	2.63	0.16	mg/kg	2	Reference	ICP-MS
Gallium	6.78	0.29	mg/kg	2	Reference	ICP-MS
Holmium	0.488	0.052	mg/kg	2	Reference	ICP-MS
Lanthanum	15.50	0.83	mg/kg	2	Reference	ICP-MS
Lithium	16.4	1.8	mg/kg	2	Reference	ICP-OES
Lutetium	0.219	0.024	mg/kg	2	Reference	ICP-MS
Molybdenum	1.211	0.076	mg/kg	2	Reference	ICP-MS
Neodymium	14.82	0.73	mg/kg	2	Reference	ICP-MS
Niobium	7.18	0.49	mg/kg	2	Reference	ICP-MS
Praseodymium	3.99	0.18	mg/kg	2	Reference	ICP-MS
Samarium	2.93	0.23	mg/kg	2	Reference	ICP-MS
Scandium	4.3	1.1	mg/kg	2	Reference	ICP-MS; INAA
Terbium	0.390	0.028	mg/kg	2	Reference	ICP-MS
Thallium	0.24	0.10	mg/kg	2	Reference	ICP-MS
Thorium	4.37	0.20	mg/kg	2	Reference	ICP-MS
Thulium	0.216	0.020	mg/kg	2	Reference	ICP-MS
Tin	36	11	mg/kg	2	Reference	ICP-MS
Tungsten	0.90	0.23	mg/kg	2	Reference	ICP-MS
Uranium	1.38	0.21	mg/kg	2	Reference	ICP-MS

SRM 2706, Certified and Reference Values, PAHs

35 PAH Congeners



Constituent	Value	U	k	Assigned	Methods
Naphthalene	15.3	3.0	2	Certified	1;2
Acenaphthylene	0.29	0.12	2	Certified	1;2
Acenaphthene	0.0130	0.0020	2	Certified	1;2
Phenanthrene	0.471	0.046	2	Certified	1;2
4H-cyclopenta[def]phenanthrene	0.040	0.010	2	Certified	1;2
Fluoranthene	0.516	0.066	2	Certified	1;2
Pyrene	0.504	0.064	2	Certified	1;2
Benzo[ghi]fluoranthene	0.071	0.030	2	Certified	1;2
Benzo[c]phenanthrene	0.058	0.010	2	Certified	1;2
Benzo[a]anthracene	0.241	0.068	2	Certified	1;2
Benzo[b]fluoranthene	0.314	0.052	2	Certified	1;2
Benzo[k]fluoranthene	0.144	0.042	2	Certified	1;2
Benzo[j]fluoranthene	0.122	0.018	2	Certified	1;2
Benzo[a]fluoranthene	0.129	0.060	2	Certified	1;2
Benzo[e]pyrene	0.332	0.040	2	Certified	1;2
Benzo[a]pyrene	0.255	0.032	2	Certified	1;2
Perylene	0.074	0.030	2	Certified	1;2
Benzo[ghi]perylene	0.363	0.060	2	Certified	1;2
Dibenz[a,j]anthracene	0.055	0.016	2	Certified	1;2
Dibenz[a,c]anthracene	0.049	0.012	2	Certified	1;2
Dibenz[a,h]anthracene	0.055	0.010	2	Certified	1;2
Benzo[b]chrysene	0.064	0.030	2	Certified	1;2
Picene	0.076	0.022	2	Certified	1;2
Dibenzo[b,k]fluoranthene	0.068	0.012	2	Certified	1;2
Dibenzo[a,e]pyrene	0.0720	0.0060	2	Certified	1;2
Coronene	0.064	0.010	2	Certified	1;2
Biphenyl	0.060	0.036	2	Reference	1;2
Fluorene	0.030	0.010	2	Reference	1
Dibenzothiophene	0.071	0.062	2	Reference	1;2
Anthracene	0.060	0.044	2	Reference	1;2
Cyclopenta[cd]pyrene	0.052	0.036	2	Reference	1;2
Triphenylene	0.132	0.044	2	Reference	1
Chrysene	0.285	0.074	2	Reference	1
Triphenylene + Chrysene	0.345	0.018	2	Reference	2
Indeno[1,2,3-cd]pyrene	0.31	0.19	2	Reference	1;2

Key to Methods

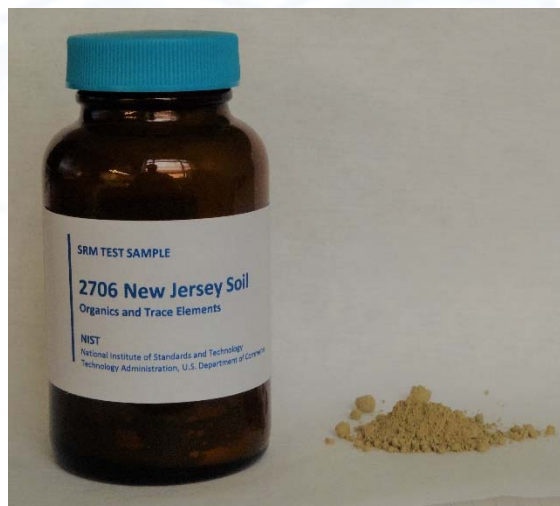
1. Hexane/acetone pressurized fluid extraction GC/MS at NIST with Rxi-PAH GC column
2. Dichloromethane pressurized fluid extraction GC/MS at NIST with ZB-50 GC column

SRM 2706, EPA Methods used in the Appendix Study

EPA Method 3050B	<i>Acid Digestion of Sediments, Sludges and Soils</i>
EPA Method 200.2	<i>Sample Preparation Procedure for Spectrochemical Determination of Total Recoverable Elements</i>
EPA Method 200.8	<i>Determination of Trace Elements in Water and Wastes by Inductively Coupled Plasma Mass Spectrometry</i>
EPA Method 6010	<i>Inductively Coupled Plasma-Atomic Emission Spectrometry</i>
EPA Method 6020	<i>Inductively Coupled Plasma Mass Spectrometry</i>
EPA Method 7471	<i>Mercury in Solid or Semisolid Waste (Manual Cold- Vapor Technique)</i>
EPA Method 7473	<i>Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrometry</i>
<hr/>	
EPA Method 3550	<i>Ultrasonic Extraction</i>
EPA Method 3570	<i>Microscale Solvent Extraction (MSE)</i>
EPA Method 3630	<i>Silica Gel Cleanup</i>
EPA Method 8082	<i>Polychlorinated Biphenyls (PCBs) by Gas Chromatography</i>
EPA Method 8270	<i>Semivolatile Organic Compounds by GC/MS</i>
EPA Method 1613	<i>Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS</i>
EPA Method 8290	<i>Polychlorinated Dibenzo-dioxins (PCDDs) and Polychlorinated Dibenzo-furans (PCDFs) by High-Resolution Gas Chromatography/High Resolution Mass Spectrometry (HRGC/HRMS)</i>

SRM 2706 Appendix Values

Major and Trace Elements (39 elements)



Element	n	Range (mg/kg)		Median (mg/kg)	Mean (mg/kg)	Recovery (%) ^(a)
Aluminum	6	6100	- 10000	7800	8100	30
Antimony	5	40	- 150	54	70	47
Arsenic	6	20	- 31	28	27	89
Barium	6	84	- 110	93	96	30
Beryllium	6	0.4	- 0.55	0.47	0.47	57
Boron	1	---	---	7.8	7.8	--
Cadmium	5	0.26	- 0.35	0.3	0.31	99
Calcium	6	3700	- 4500	4000	4100	70
Cerium	1	---	---	22	22	64
Chromium	6	14	- 21	19	18	31
Cobalt	5	4.1	- 5.3	5.2	4.8	81
Copper	6	72	- 96	86	84	96
Iron	6	11000	- 19000	17000	16000	74
Lanthanum	1	---	---	9	9	58
Lead	6	530	- 670	610	610	94
Lithium	3	7	- 8.1	7.9	7.7	47
Magnesium	6	1300	- 2000	1600	1600	56
Manganese	6	140	- 180	170	160	67
Mercury	6	0.12	- 0.14	0.12	0.13	96
Molybdenum	4	0.83	- 1.7	1.2	1.2	101
Neodymium	1	---	---	9.5	9.5	65
Nickel	6	12	- 18	17	16	69
Phosphorus	2	280	- 360	320	320	79
Potassium	6	980	- 2000	1400	1400	15
Praseodymium	1	---	---	2.5	2.5	63
Selenium	4	0.13	- 1.8	0.46	0.71	--
Silver	3	0.11	- 0.14	0.14	0.13	--
Sodium	4	180	- 250	200	210	8
Strontium	5	23	- 27	25	25	41
Sulfur	1	---	---	1500	1500	70
Thallium	2	0.11	- 0.12	0.11	0.11	47
Thorium	1	---	---	2.3	2.3	53
Tin	5	24	- 37	31	30	84
Titanium	5	120	- 300	190	200	7
Uranium	1	---	---	0.66	0.66	48
Vanadium	6	24	- 34	30	30	58
Yttrium	2	4.7	- 6.3	5.5	5.5	--
Zinc	6	110	- 130	120	120	88
Zirconium	1	---	---	3.9	3.9	1

^(a) Recovery (mean value) relative to the certified/reference value, where applicable

SRM 2706 Appendix Values

Chlorinated Dibenzo-*p*-dioxin Congeners

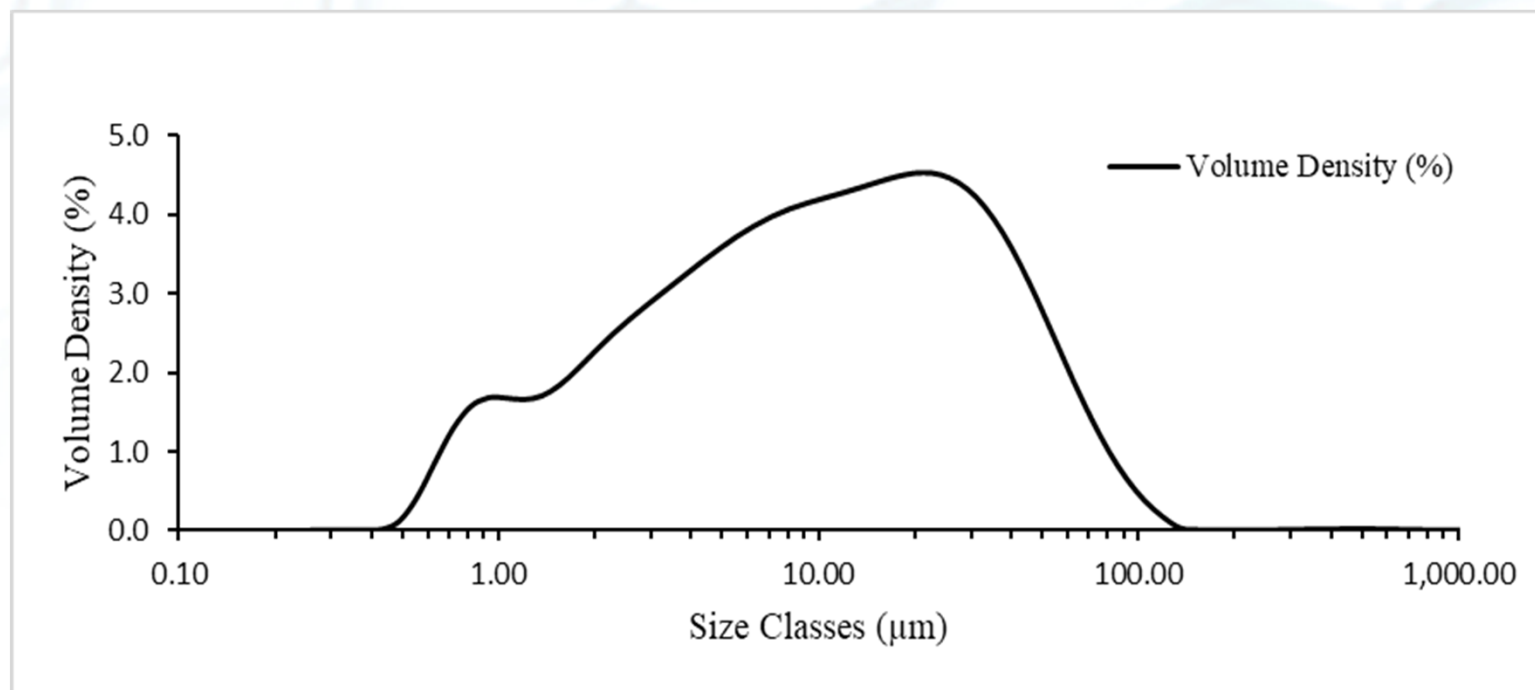
PAHs/PCBs	n	Range (µg/kg)			Median (µg/kg)	Mean (µg/kg)	Recovery (%) ^(a)
Acenaphthene	1	---		---	9.1	9.1	70
Acenaphthylene	2	53	-	57	55	55	19
Anthracene	1	---		---	41	41	69
Benzo(<i>a</i>)anthracene	1	---		---	110	110	46
Benzo(<i>a</i>)pyrene	1	---		---	88	88	35
Benzo(<i>b</i>)fluoranthene	4	210	-	280	240	240	77
Benzo(<i>g,h,i</i>)perylene	2	130	-	140	130	130	36
Benzo(<i>k</i>)fluoranthrene	3	64	-	160	64	97	68
Chrysene	2	160	-	190	180	180	64
Dibenzo(<i>a,h</i>)anthracene	1	---		---	34	34	62
Fluoranthrene	4	230	-	370	290	290	57
Fluorene	1	---		---	21	21	70
Indeno(1,2,3- <i>cd</i>)pyrene	1	---		---	93	93	30
Naphthalene	3	3600	-	7400	5200	5400	36
Phenanthrene	2	200	-	200	200	200	43
Pyrene	2	220	-	220	220	220	44
Aroclor 1260	3	750	-	820	800	790	--

^(a) Recovery (mean value) relative to the certified/reference value, where applicable

Chlorinated Dibenzofuran Congeners

	n	Range (ng/kg)			Median (ng/kg)	Mean (ng/kg)
2,3,7,8-TCDD	1	---		---	34	34
1,2,3,7,8-PeCDD	1	---		---	81	81
1,2,3,4,7,8-HxCDD	1	---		---	79	79
1,2,3,6,7,8-HxCDD	1	---		---	170	170
1,2,3,7,8,9-HxCDD	1	---		---	67	67
1,2,3,4,6,7,8-HpCDD	3	920	-	1000	970	960
OCDD	3	8100	-	8800	8700	8500
2,3,7,8-TCDF	2	280	-	670	480	480
1,2,3,7,8-PeCDF	3	5400	-	7600	5500	6200
2,3,4,7,8-PeCDF	2	8800	-	13000	11000	11000
1,2,3,4,7,8-HxCDF	3	150000	-	210000	150000	170000
1,2,3,6,7,8-HxCDF	2	32000	-	43000	38000	38000
2,3,4,6,7,8-HxCDF	3	7700	-	11000	11000	9700
1,2,3,7,8,9-HxCDF	2	2300	-	2500	2400	2400
1,2,3,4,6,7,8-HpCDF	3	470000	-	660000	480000	540000
1,2,3,4,7,8,9-HpCDF	3	17000	-	22000	18000	19000
OCDF	3	280000	-	430000	370000	360000
Total TCDF	1	---		---	44000	44000
Total TCDD	1	---		---	740	740
Total PeCDF	1	---		---	100000	100000
Total PeCDD	1	---		---	1100	1100
Total HxCDF	2	300000	-	410000	360000	360000
Total HxCDD	1	---		---	1300	1300
Total HpCDF	2	540000	-	740000	640000	640000
Total HpCDD	2	2300	-	2400	2400	2400

SRM 2706, Particle Size Distribution



Malvern laser-based light scattering system

The mass median diameter is 10.4 μm. Size Range 0.5 μm to 590 μm in diameter

Approximately 49 % is below 10.1 μm in diameter



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2706

New Jersey Soil

Organics and Trace Elements

This Standard Reference Material (SRM) is intended primarily for use in the analysis of selected inorganic constituents, polycyclic aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB), chlorinated dibenzo-*p*-dioxin congeners, and chlorinated dibenzofuran congeners in soils, sediments, or other materials of a similar matrix. SRM 2706 is a natural agricultural soil blended with waste soil materials containing target constituents. A unit of SRM 2706 consists of 50 g of dried, powdered soil.

Certified Values: Certified mass fraction values [1] of trace elements and PAHs, reported on a dry-mass basis, are provided in Tables 1 and 2. Certified values are based on results obtained from critically evaluated independent analytical techniques. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [2]. The measurands are the total mass fractions of the elements or PAHs reported in Tables 1 and 2. Metrological traceability is to the SI derived unit of mass fraction (expressed as either % or mg/kg) for trace elements and for selected PAHs.

Reference Values: Reference mass fraction values for additional trace elements and PAHs, reported on a dry-mass basis, are provided in Tables 3 and 4. The reference values are based on results obtained from NIST analytical methods, or participating external laboratories. Reference values are non-certified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty [2]. The measurands are the total mass fractions of the trace elements or PAHs reported in Tables 3 and 4. Metrological traceability is to the SI derived unit of mass fraction (expressed as either % or mg/kg).

Information Values: Information values for selected trace elements and loss on fusion, are provided in Table 5. An information value is considered to be a value that will be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of SRM 2706 is valid, within the measurement uncertainties specified, until **1 May 2028**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). This certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to the certification was performed by S.E. Long of the NIST Chemical Sciences Division.

Consultation on the statistical design and evaluation of the data was provided by Z.Q.J. Lu of the NIST Statistical Engineering Division.

SRM 2706
Expected
availability
October 2018



Conclusions and Future Work

The successful development of SRM 2706 (New Jersey Soil) was a highly complex project involving close cooperation between five different major stakeholders, and numerous additional contributors including nine external laboratories

Future Work

- Addition of perfluorinated compounds eg. PFOA, PFAS
 - Method development underway at NIST

Thank you to:

- NELAC Institute - for assistance with travel to this meeting
- All those who helped with the samples and distribution

Acknowledgements

S. Nagourney, Z. Wilk	NJDEP
S. Wilson, M. Adams, D. Olinger, and J. Reitman	USGS
L. Phelps	US EPA ORD
J. Bourbon	EPA Region 2
B. Benner, J. Browning, M. Cronise, B. Kassim, A. Marlow, J. Ness, R. Oflaz, R. Paul, J. Sieber, J. Trevillian, and L. Wood	Chemical Sciences Division NIST
M. Cronise	Office of Reference Materials, NIST
J. Lu	Statistical Engineering Division, NIST
P. Westrick and R. Wyeth	Pace Analytical

Participating Laboratories

P. Worby, N. Cole

Accutest Laboratories
2235 US Highway 130
Dayton, NJ, USA

J.R. Bourbon

EPA Region 2
2890 Woodbridge Avenue
Edison, NJ, USA

P. Westrick

Pace Analytical Services
1638 Roseytown Road
Greensburg, PA, USA

J. Todaro, A. Rice

Alpha Laboratories
8 Walkup Drive
Westborough, MA, USA

F. Wellbourne, J. Hendel

EPA Region 4
980 College Station Road
Athens, GA, USA

M. Acierno

Test America Edison
777 New Durham Road
Edison, NJ 08817-2859

R. McLeod, S. Kennedy

ALS Life Sciences
1435 Norjohn Court
Burlington, ON, Canada

S. Petzinger, C. Bremer, A. Pender

Pace Analytical Services
1700 Elm Street
Minneapolis, MN, USA

D. Lowe, M. Bruce

Test America Pittsburgh
301 Alpha Drive, RIDC Park
Pittsburgh, PA, USA