



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material<sup>®</sup> 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 percent or milligrams per kilogram) 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 percent or milligrams per kilogram).

**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 uncertainty specified, until **01 May 2028**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and 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.

Carlos A. Gonzalez, Chief  
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Certificate Issue Date: 12 December 2018

Steven J. Choquette, Director  
Office of Reference Materials

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

The agricultural soil and associated blend materials were collected with the assistance of S.J. Nagourney of the New Jersey Department of Environmental Protection (NJDEP), Office of Quality Assurance (Trenton, NJ), who provided expertise in the material design, site selection, and sample shipment, as well as helping to coordinate the multi-laboratory measurements. Additional expertise with the design and development of the material was also provided by L. Phelps of the Office of the Science Advisor, U.S. Environmental Protection Agency (EPA) (Research Triangle Park, NC), S.A. Wilson of the Reference Materials Program, United States Geological Survey (USGS) (Denver CO), Z. Wilk of the NJDEP, J. Bourbon of the Division of Environmental Science and Assessment, EPA Region 2 (Edison, NJ), P. Westrick of Pace Analytical (Greensburg, PA), R. Wyeth of Pace Analytical (Minneapolis, MN), and J. Reiner of the NIST Chemical Sciences Division.

Preparation and bottling of the material was performed by S.A. Wilson of the USGS and M.P. Cronise of the NIST Office of Reference Materials.

Analytical measurements for the certification of SRM 2706 were performed at NIST by B.A. Benner, J.F. Browning, B.L. Kassim, S.E. Long, A.F. Marlow, J.M. Ness, R. Oflaz, R.L. Paul, J.R. Sieber, J. Trevillian, and L.J. Wood. Supporting analytical measurements at the USGS were made by M. Adams, D. Olinger, J. Reitman, and S.A. Wilson. The analytical techniques used for each element are listed in Table 6.

## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

**Handling:** This material is a naturally occurring soil augmented with targeted contaminants and may contain constituents of unknown toxicity. The material was processed to maintain homogeneity and is composed of a particle size distribution that is readily respirable. Therefore, care should be exercised during its handling and use, and precautions to avoid inhalation of dust during material transfer are recommended.

**Storage:** SRM 2706 should be stored in its original, tightly-capped bottle at temperatures less than 30 °C, and away from direct sunlight.

**Sampling:** The SRM should be thoroughly mixed by repeatedly inverting and rotating the bottle horizontally before removing a test portion for analysis. A minimum mass of 250 mg should be used for analytical determinations to be related to the mass fraction values in this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution or extraction. If volatile elements (e.g. arsenic, mercury, selenium) are to be determined, precautions should be taken in the dissolution of SRM 2706 to avoid volatilization losses.

**Determination of Moisture:** The moisture content of SRM 2706 was determined at NIST by measuring the mass loss after oven drying for 2 h at 110 °C. Six bottles were selected according to a stratified randomized scheme for the drying study and measured in duplicate. The moisture content of SRM 2706 at the time of the certification analyses was 0.64 % ± 0.04 % (95 % confidence interval). The analytical results for the constituents were determined on an as-received mass fraction basis and then converted to a dry mass fraction basis by dividing by a conversion factor of 0.9936.

## SOURCE, PREPARATION, AND ANALYSIS<sup>(1)</sup>

**Source and Preparation of Material:** The USGS, under contract to NIST, processed the soil for the production of SRM 2706. Agricultural soil representing a geological profile typical of the Eastern US was collected from the Agricultural Research Station of Rutgers University, NJ, and augmented with waste soil materials collected from four additional sites in Hainsport Township, Burlington, NJ, Richmond County, Staten Island, NY, Passaic County, NJ, and Sayreville, Middlesex County, NJ. The soil materials were delivered to the USGS in 19 L plastic buckets. The contents of each bucket were transferred to a series of plastic lined cardboard trays. The trays were moved to a forced air-drying oven, and the soil was dried for three days at room temperature. After drying, the soil material was disaggregated using a ceramic auger operating at approximately 50 rpm. Soil material with a particle size < 2 mm was separated using a 2 mm stainless steel vibrating screen sieve and further processed in a ceramic lined 115 L ball mill loaded with approximately one inch diameter corundum grinding balls. After an 18 h grinding period, the

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<sup>(1)</sup> Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

contents of the mill were removed, and the ground soil was transferred to a 0.28 m<sup>3</sup> cross flow V-blender for a blending duration of 6 h. The bulk material was then shipped to a gamma-ray irradiation facility for radiation sterilization at an estimated dose of 25 kGy (<sup>60</sup>Co) and returned to USGS for bottling. In the final preparation step, a custom-designed spinning riffler was used to split the blended material into screw-capped wide-mouth glass bottles which had been pre-cleaned at NIST. Every 100th bottle was removed for homogeneity and quantitative testing, and the remainder were returned to NIST.

**Homogeneity Testing:** The homogeneity of the material was assessed at USGS for 51 elements using a combination of wavelength dispersive X-ray fluorescence spectrometry (WDXRF), inductively coupled plasma optical emission spectrometry (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS). The estimated relative standard deviation for material inhomogeneity for all elements with the exception of those elements near the determination limit, is < 2 %. For lead, the estimated relative standard deviation is 2.4 %, which is still considered acceptable from a material homogeneity standpoint. Results from additional analyses during certification of trace elements and PAHs indicated no significant material inhomogeneity.

**Analytical Approach for Inorganic Constituents:** Value assignment for selected trace elements was accomplished by combining results from analyses of stratified random samples of SRM 2706 using one or more critically evaluated analytical methods at NIST and supporting measurements from the USGS. The NIST methods were ICP-OES, WDXRF, cold vapor isotope dilution inductively coupled plasma mass spectrometry (CV-ID-ICP-MS), and instrumental neutron activation analysis (INAA). Analytical methods at the USGS were ICP-OES, ICP-MS and WDXRF.

**NIST Analyses using ICP-OES:** ICP-OES was used to determine Cu, V and Zn. Duplicate 0.5 g test portions were taken from each of six bottles and placed into Teflon microwave vessels. Eight milliliters of concentrated nitric acid and 2 mL of concentrated hydrofluoric acid were added to each vessel prior to microwave digestion. Scandium was added as an internal standard to improve the precision of the instrumental measurements. The resulting solutions were transferred to Teflon beakers, and 2 mL of concentrated perchloric acid were added to each sample. The solutions were then covered and heated on a hot plate with a surface temperature of 200 °C for 4 h. The covers were then removed, and the solutions were heated to dryness. The solutions were diluted to 30 g with 1.5 % mass fraction nitric acid and transferred to polyethylene bottles for analysis by ICP-OES using the method of standard additions.

**NIST Analyses using CV-ID-ICP-MS:** Mercury was determined by CV-ID-ICP-MS. Single 0.3 g test portions were taken from each of six bottles, and placed into microwave quartz vessels, together with an aliquot of <sup>201</sup>Hg and 6 g of high-purity nitric acid. The samples were then digested in a high-pressure microwave under closed system conditions. After cooling to room temperature, the solutions were transferred to cleaned 50 mL polypropylene centrifuge tubes and diluted to a concentration suitable for measurement. Mercury was determined using cold-vapor mercury generation coupled with ICP-MS isotope ratio measurements. The <sup>201</sup>Hg spike was calibrated by reverse isotope dilution using a primary calibrant consisting of SRM 1641d, Mercury in Water.

**NIST Analyses using WDXRF:** WDXRF was used to determine 21 elements consisting of Na, Mg, Al, Si, P, S, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Sr, Zr, Ba and Pb. Measurements were made on two to four replicate test portions taken from each of six bottles of SRM 2706. For quantitative analysis, samples were prepared by borate fusion. A quantity of flux (approximately 4 g of a 50/50 mixture of Li<sub>2</sub>B<sub>4</sub>O<sub>7</sub> and LiBO<sub>2</sub>) was added to a Pt (5 % Au) crucible. A test portion (approximately 3 g) of SRM 2706 was then weighed into the crucible, and a second portion of flux was placed on top of the sample. The fusion procedure consisted of drying and calcination steps by ramping the temperature in steps from 200 °C to 600 °C, followed by a temperature ramp to 980 °C to facilitate the fusion of the SiO<sub>2</sub> in the soil. At this point, the melt was cooled to room temperature and two or three drops of LiI non-wetting agent were added. A second temperature program was used to heat and melt the glass prior to pouring and casting the beaded melt in a platinum dish. A WDXRF spectrometer was used to measure the K-L<sub>2,3</sub> characteristic X-ray lines of Na, Mg, Al, Si, P, S, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Sr, and Zr. For As, the K-M<sub>2,3</sub> line was measured, for Ba, the L<sub>3</sub>-M<sub>4,5</sub> line was measured, and for Pb, the L<sub>2</sub>-M<sub>4</sub> line was measured. In addition to the elements of interest, the Rh K-L Compton scatter line from the X-ray tube was measured for use as an internal reference to compensate for the effects of differences in bead mass thickness. Calibration of the system was performed using synthetic calibrants prepared from high-purity compounds and NIST SRM 3100 series spectrometric solutions. Calibration curves were prepared by linear regression.

**NIST Analyses using INAA:** INAA was used to determine Ag, Co, Cr, Fe, Rb, Sb, Sc and Se. Two pellets were prepared from each of ten bottles of SRM 2706. Aliquots of the powder were poured into a 13 mm diameter stainless steel die and pressed into pellets using a hydraulic press at 10,000 pounds pressure for 2-3 seconds. Each pellet was weighed to ± 0.01 mg using an analytical balance. The element standards were prepared by quantitative deposition on filter papers using previously prepared multi-element solutions and/or single element solutions prepared from SRM materials. Sample irradiations were carried out in the pneumatic tube RT-2 of the NIST reactor at a reactor power of

20 MW. The irradiation time was 2 h with a 180 degree inversion of the irradiation capsule after 1 h to ensure flux homogeneity. Samples were counted using gamma-ray spectrometry. The counting system was equipped with an analog shaping amplifier with a 16 k channel fixed conversion time analog to digital converter, a network data acquisition interface and a germanium detector. Spectral data were converted to mass fraction values for the identified elements using commercially available software routine. All count rates were corrected for radioactive decay, and spectral interferences. The results were transferred to spreadsheets for final statistical evaluation.

**USGS Measurements:** Supporting measurements of trace metals were made at the USGS, Denver, CO, in tandem with homogeneity assessments. A total of 51 elements were measured in 19 test portions of SRM 2706 selected by stratified random sampling. The measurements were made using independent methods based on ICP-OES, ICP-MS and WDXRF. Test portion sizes ranged from 0.2 g for ICP-OES/ICP-MS to 0.7 g for WDXRF.

**Certified Mass Fraction Values for Elements:** Certified mass fraction values for all elements except mercury are the equally weighted means of results from two or more analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is intended to represent, at the level of one standard deviation, the combined effect of between-method and within-method components of uncertainty, following the ISO/JCGM Guide [3,4] and assuming approximately normal distribution. The coverage factor,  $k = 2$  was determined from the Student's  $t$ -distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence for each analyte. The certified value for mercury is based on results from a single NIST method (CV-ID-ICP-MS, see Table 4) for which a complete evaluation of all sources of uncertainty has been performed. The uncertainty provided is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence, consistent with the ISO/JCGM Guide [4]. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is the combined uncertainty that incorporates within-method uncertainty and Type B uncertainty components related to the analysis, assuming approximately normal distribution, and coverage factor,  $k = 2$ , corresponding to approximately 95 % confidence for each analyte.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for Elements in SRM 2706

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum (Al)	2.70 ± 0.19	Antimony (Sb)	149 ± 11
Calcium (Ca)	0.588 ± 0.046	Barium (Ba)	319 ± 43
Iron (Fe)	2.22 ± 0.15	Chromium (Cr)	60.1 ± 5.4
Magnesium (Mg)	0.289 ± 0.014	Cobalt (Co)	5.99 ± 0.23
Potassium (K)	0.946 ± 0.028	Copper (Cu)	88.1 ± 6.8
Silicon (Si)	39.17 ± 0.47	Lead (Pb)	653 ± 36
Sodium (Na)	0.268 ± 0.012	Manganese (Mn)	244 ± 15
Titanium (Ti)	0.290 ± 0.042	Mercury (Hg)	0.1329 ± 0.0033
		Nickel (Ni)	22.8 ± 5.4
		Phosphorus (P)	407 ± 21
		Rubidium (Rb)	37.6 ± 1.9
		Strontium (Sr)	60.3 ± 3.1
		Vanadium (V)	51.9 ± 6.5
		Zinc (Zn)	135.4 ± 6.9
		Zirconium (Zr)	303 ± 25

**NIST Measurements of PAHs:** Measurements of 35 PAHs were made using two independent methods based on pressurized liquid extraction (PLE) and gas chromatography/mass spectrometry (GC/MS). Two separate PLE methods using different solvents - dichloromethane and acetone/hexane (50/50 by volume) were employed to generate extracts of SRM 2706. For both PLE methods, one aliquot (0.4 g to 0.9 g) from each of six bottles of SRM 2706 was weighed by difference into an 11 mL extraction vessel containing pre-extracted diatomaceous earth, fortified with weighed aliquots of SRM 2269, *Perdeuterated PAH-I Solution in Hexane/Toluene*, and SRM 2270, *Perdeuterated PAH-II Solution in Hexane/Toluene*. The loaded vessels were then extracted by PLE using an accelerated solvent extraction system with dichloromethane and acetone/hexane as the extraction solvents, at 100 °C for six cycles, with a 100 % rinse volume, and 90 s purge. Calibrants were prepared by transferring weighed aliquots of SRM 2260a, *Aromatic Hydrocarbons in Toluene*, SRM 2269, *Perdeuterated PAH-I Solution in Hexane/Toluene*, and SRM 2270, *Perdeuterated PAH-II Solution in Hexane/Toluene*, to extraction vessels containing diatomaceous earth sorbent. The extracts were concentrated under nitrogen, transferred to 2 mL glass syringes, and filtered through 13 mm, 0.45 µm Teflon syringe filters into amber glass 2 mL vials. For the measurement of the PAHs in SRM 2706, a gas

chromatograph interfaced with a mass selective detector (MSD) was employed in electron ionization (EI) mode. The two analytical methods employed a 60 m x 0.25 mm ZB-50 column (0.25  $\mu$ m phase, 50 % phenylmethyl-substituted polysiloxane, and a 60 m x 0.25 mm Rxi-PAH column (0.1  $\mu$ m mid-polarity proprietary phase). Both columns were set at a constant flow of 3.0 mL/min He (average linear velocity 44 cm/s) and employed an oven program beginning at 40 °C for a 1 min hold followed by a 50 °C/min ramp to 150 °C, a 2.5 °C/min ramp to 320 °C, and a 15 min hold at this final temperature. The mass spectrometer was operated in selected ion monitoring mode. Relative response factors were determined after multiple GC/MS measurements of four calibration standards. The mass fractions of PAHs in SRM 2706 were calculated using an equation based on the relative peak areas of the analyte, the internal standard, the response factor of the analyte and the mass of the sample taken.

**Certified Mass Fraction Values for PAHs:** Certified mass fraction values are the equally weighted means of results from two analytical methods at NIST based on five to six pressurized fluid extractions using 50 % hexane/50 % acetone, and six pressurized fluid extractions with dichloromethane. The uncertainty listed with each value is an expanded uncertainty about the mean. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is intended to represent, at the level of one standard deviation, the combined effect of between-method and within-method components of uncertainty, following the ISO/JCGM Guide [3,4] and assuming approximately normal distribution. The coverage factor,  $k = 2$  was determined from the Student's  $t$ -distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence for each analyte.

Table 2. Certified Mass Fraction Values (Dry-Mass Basis) for PAHs in SRM 2706

	Mass Fraction (mg/kg)
Naphthalene <sup>(a)</sup>	15.3 ± 3.0
Acenaphthylene	0.29 ± 0.12
Acenaphthene	0.0130 ± 0.0020
Phenanthrene	0.471 ± 0.046
4H-cyclopenta[def]phenanthrene	0.040 ± 0.010
Fluoranthene	0.516 ± 0.066
Pyrene	0.504 ± 0.064
Benzo[ghi]fluoranthene	0.071 ± 0.030
Benzo[c]phenanthrene	0.058 ± 0.010
Benz[a]anthracene <sup>(a)</sup>	0.241 ± 0.068
Benzo[b]fluoranthene	0.314 ± 0.052
Benzo[k]fluoranthene	0.144 ± 0.042
Benzo[j]fluoranthene	0.122 ± 0.018
Benzo[a]fluoranthene	0.129 ± 0.060
Benzo[e]pyrene	0.332 ± 0.040
Benzo[a]pyrene <sup>(a)</sup>	0.255 ± 0.032
Perylene	0.074 ± 0.030
Benzo[ghi]perylene	0.363 ± 0.060
Dibenz[a,j]anthracene	0.055 ± 0.016
Dibenz[a,c]anthracene	0.049 ± 0.012
Dibenz[a,h]anthracene	0.055 ± 0.010
Benzo[b]chrysene	0.064 ± 0.030
Picene	0.076 ± 0.022
Dibenzo[b,k]fluoranthene	0.068 ± 0.012
Dibenzo[a,e]pyrene	0.0720 ± 0.0060
Coronene	0.064 ± 0.010

<sup>(a)</sup> Metrological traceability is to the SI derived unit of mass fraction.

**Reference Mass Fraction Values for Elements:** Reference mass fraction values for all elements are the equally weighted means of results from one or more analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is intended to represent, at the level of one standard deviation, the combined effect of between-method and within-method components of uncertainty, following the ISO/JCGM Guide [3,4], and assuming approximately normal distribution. The coverage factor,  $k = 2$  was determined from the Student's  $t$ -distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence for each analyte. Reference values determined from the mean of results obtained by a single laboratory using one analytical technique are calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the analyte mean, assuming approximately normal distribution, with coverage factor,  $k = 2$ , corresponding to approximately 95 % confidence level for each analyte.

Table 3. Reference Mass Fraction Values (Dry-Mass Basis) for Elements in SRM 2706

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Sulfur (S)	0.217 ± 0.012	Arsenic (As)	30.3 ± 2.7
		Beryllium (Be)	0.84 ± 0.17
		Bismuth (Bi)	0.159 ± 0.012
		Cadmium (Cd)	0.31 ± 0.14
		Cerium (Ce)	34.5 ± 1.7
		Cesium (Cs)	1.35 ± 0.10
		Dysprosium (Dy)	2.41 ± 0.21
		Erbium (Er)	1.45 ± 0.15
		Europium (Eu)	0.602 ± 0.038
		Gadolinium (Gd)	2.63 ± 0.16
		Gallium (Ga)	6.78 ± 0.29
		Holmium (Ho)	0.488 ± 0.052
		Lanthanum (La)	15.50 ± 0.83
		Lithium (Li)	16.4 ± 1.8
		Lutetium (Lu)	0.219 ± 0.024
		Molybdenum (Mo)	1.211 ± 0.076
		Neodymium (Nd)	14.82 ± 0.73
		Niobium (Nb)	7.18 ± 0.49
		Praseodymium (Pr)	3.99 ± 0.18
		Samarium (Sm)	2.93 ± 0.23
		Scandium (Sc)	4.3 ± 1.1
		Terbium (Tb)	0.390 ± 0.028
		Thallium (Tl)	0.24 ± 0.10
		Thorium (Th)	4.37 ± 0.20
		Thulium (Tm)	0.216 ± 0.020
		Tin (Sn)	36 ± 11
		Tungsten (W)	0.90 ± 0.23
		Uranium (U)	1.38 ± 0.21

**Reference Mass Fraction Values for PAHs:** Reference mass fraction values for PAHs are the equally weighted means of results from one or two analytical methods based on five to six pressurized fluid extractions using 50 % hexane/50 % acetone and six pressurized fluid extractions with dichloromethane. The uncertainty listed with each value is an expanded uncertainty about the mean. The expanded uncertainty is calculated as  $U = ku_c$ , where  $u_c$  is intended to represent, at the level of one standard deviation, the combined effect of between-method and within-method components of uncertainty, following the ISO/JCGM Guide [3,4]. The coverage factor,  $k = 2$ , was determined from the Student's  $t$ -distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence for each analyte. Reference values determined from the mean of results obtained using one analytical technique are calculated as  $U = ku_c$ , where  $u_c$  is one standard deviation of the analyte mean, assuming approximately normal distribution, with coverage factor,  $k = 2$ , corresponding to approximately 95 % confidence level for each analyte.

Table 4. Reference Mass Fraction Values (Dry-Mass Basis) for PAHs in SRM 2706

	Mass Fraction (mg/kg)
Biphenyl	0.060 ± 0.036
Fluorene	0.030 ± 0.010
Dibenzothiophene	0.071 ± 0.062
Anthracene	0.060 ± 0.044
Cyclopenta[ <i>cd</i> ]pyrene	0.052 ± 0.036
Triphenylene	0.132 ± 0.044
Chrysene	0.285 ± 0.074
Triphenylene + Chrysene <sup>(a)</sup>	0.345 ± 0.018
Indeno[1,2,3- <i>cd</i> ]pyrene	0.31 ± 0.19

<sup>(a)</sup> Co-elution on ZB-50 chromatography column.

**Information Mass Fraction Values for Additional Measurands:** Information mass fraction values are the mean of measurements from one or more analytical methods. Information mass fraction values cannot be used to establish metrological traceability. No uncertainty is provided because there is insufficient information available for its assessment.

Table 5. Information Mass Fraction Values (Dry-Mass Basis) in SRM 2706

	Value	Units
Selenium	0.3	mg/kg
Silver	<1	mg/kg
Yttrium	11	mg/kg
Loss on Fusion <sup>(a)</sup>	6.5	%

<sup>(a)</sup> Loss on fusion with lithium borate flux at 980 °C.

**Information Values for Particle Size:** Particle size measurements of SRM 2706 were made at NIST using a Malvern Mastersizer 3000 laser-based light scattering system. Approximately 0.5 g of SRM 2706 material was measured using water as the dispersant (refractive index: 1.330). A sample aliquot was introduced into the measurement cell and ten individual measurements were made at an obscuration target of 10 % to 15 % of the laser beam. The calculated 10<sup>th</sup> ( $D_v(10)$ ), 50<sup>th</sup> ( $D_v(50)$ ) and 90<sup>th</sup> ( $D_v(90)$ ) percentile particle sizes are  $D_v(10) = 1.56 \mu\text{m}$ ,  $D_v(50) = 10.4 \mu\text{m}$ , and  $D_v(90) = 43.6 \mu\text{m}$ . The mass median diameter is 10.4  $\mu\text{m}$ . As measured, the size distribution ranges from 0.523  $\mu\text{m}$  to 586  $\mu\text{m}$  in diameter and approximately 49 % of the SRM 2706 material is below 10.1  $\mu\text{m}$  in diameter. The particle size distribution is shown in Figure 1.

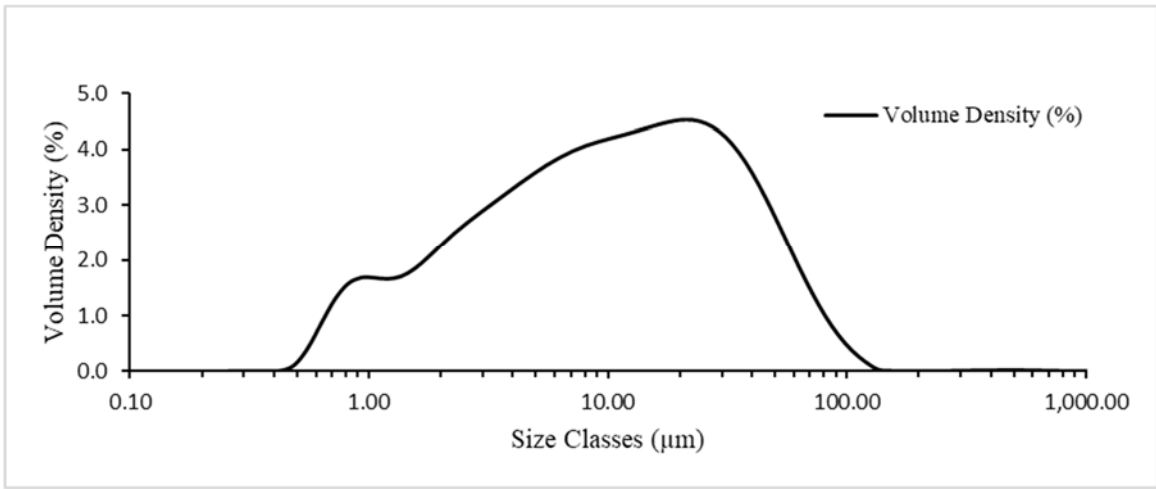


Figure 1. Particle size distribution in SRM 2706



Table 6. Analytical Methods<sup>(a)</sup> Used for the Measurement of Elements in SRM 2706

<b>Element</b>	<b>Analytical Methods</b>	<b>Element</b>	<b>Analytical Methods</b>
Aluminum	ICP-OES; WDXRF	Neodymium	ICP-MS
Antimony	ICP-MS; INAA	Nickel	ICP-OES; WDXRF
Arsenic	ICP-MS	Niobium	ICP-MS
Barium	ICP-OES; WDXRF	Phosphorus	ICP-OES; WDXRF
Beryllium	ICP-OES	Potassium	ICP-OES; WDXRF
Bismuth	ICP-MS	Praseodymium	ICP-MS
Cadmium	ICP-MS	Rubidium	ICP-MS; INAA
Calcium	ICP-OES; WDXRF	Samarium	ICP-MS
Cerium	ICP-MS	Scandium	ICP-MS; INAA
Cesium	ICP-MS	Selenium	INAA
Chromium	INAA; WDXRF	Silicon	WDXRF
Cobalt	ICP-MS; INAA	Silver	ICP-MS; INAA
Copper	ICP-OES; WDXRF	Sodium	ICP-OES; WDXRF
Dysprosium	ICP-MS	Strontium	ICP-OES; WDXRF
Erbium	ICP-MS	Sulfur	ICP-OES
Europium	ICP-MS	Terbium	ICP-MS
Gadolinium	ICP-MS	Thallium	ICP-MS
Gallium	ICP-MS	Thorium	ICP-MS
Holmium	ICP-MS	Thulium	ICP-MS
Iron	ICP-OES; INAA; WDXRF	Tin	ICP-MS
Lanthanum	ICP-MS	Titanium	ICP-OES; WDXRF
Lead	ICP-MS; WDXRF	Tungsten	ICP-MS
Lithium	ICP-OES	Uranium	ICP-MS
Lutetium	ICP-MS	Vanadium	ICP-OES; WDXRF
Magnesium	ICP-OES; WDXRF	Yttrium	ICP-MS
Manganese	ICP-OES; WDXRF	Zinc	ICP-OES; WDXRF
Mercury	CV ID-ICP-MS	Zirconium	ICP-MS; WDXRF
Molybdenum	ICP-MS		

<sup>(a)</sup> Methods

CV ID-ICP-MS	Cold Vapor Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
INAA	Instrumental Neutron Activation Analysis
WDXRF	Wavelength Dispersive X-ray Fluorescence Spectrometry

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*Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 948-3730; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <https://www.nist.gov/srm>.*

## APPENDIX A

### Multi-Laboratory Testing of Standard Reference Material® 2706 Using Approved U.S. Environmental Protection Agency (EPA) Analytical Methods

The certified values for constituents in most National Institute of Standards and Technology (NIST) chemical composition Standard Reference Materials (SRMs) reflect the total absolute mass fraction of each measurand in the given SRM matrix. The certified mass fractions are based on rigorous measurements obtained by well-characterized primary methods, or two or more independent methods or techniques. If dissolution of the matrix is necessary, the measurement approach generally requires complete sample decomposition, which can usually be achieved in a high-pressure digestion or extraction system. Alternatively, and where applicable, the sample may also be analyzed nondestructively.

For environmental monitoring purposes, the mass fractions of labile or extractable fractions of trace elements and organic compounds are often more useful than total mass fractions. Mass fractions of labile or extractable fractions are generally determined using relatively mild leach conditions, which are unlikely to totally decompose the sample. Reported mass fractions of labile or extractable fractions of constituents are generally lower than total absolute mass fractions; recovery can be total only if the constituent species in a given sample is completely labile. The recovery as a percentage of the total mass fraction is a function of several factors which are defined by the sample matrix and the measurement method conditions.

In its monitoring programs, the EPA has promulgated analytical methods to determine toxic elements and organic compounds in soils, sediments and sludges. The EPA publication SW-846, entitled *Test Methods for Evaluating Solid Waste: Physical/Chemical Methods* [5], is the official compendium of analytical and sampling methods compiled by the EPA Office of Solid Waste (OSW). The methods have been evaluated and approved for use in accordance with the 1976 Resource Conservation and Recovery Act (RCRA).

To assess the performance of these SW-846 methods for the determination of elements and organic compounds in SRM 2706, and to provide data relevant to users of these SW-846 methods, a multi-laboratory testing program was conducted jointly by NIST, the NJDEP Office of Quality Assurance, and the EPA. Stratified random samples of SRM 2706 were distributed to a number of expert, participating laboratories, who were asked to measure the target compounds of interest. The names of the participating laboratories, and laboratories providing technical development support for the study, are listed in Table A1. For the measurements of elements, the laboratories reporting data used Method 3050B for sample preparation, which is a strong acid leachable digestion method intended for the dissolution of elements which are environmentally available. Two determinative methods were used for the measurements of the extracted elements, consisting of Method 6010, and Method 6020. One laboratory also used Method 200.2 for sample digestion, and Method 200.8 as the determinative method, which are analytical methods promulgated by the EPA Office of Research and Development for compliance monitoring. For the measurement of mercury, Method 7471 and Method 7473 were used. For the measurement of PAHs and PCBs, participating laboratories used Method 3550, Method 3570, Method 8270, Method 3630 and Method 8082. Measurements of chlorinated dibenzo-*p*-dioxin congeners, and chlorinated dibenzofuran congeners were made using Method 1613 and Method 8290. A glossary of the EPA methods used in the study is provided in Table A2.

Participating laboratories provided replicate analyses from which the mean and median were calculated (to two significant figures). Summary statistics of the data sets forwarded by the laboratories are compiled by analyte class in Tables A3, A4 and A5, and include summary statistics, together with the % recovery relative to the certified or reference value, where applicable. Because analytical data generated from EPA methods are most commonly reported on an as-received basis, it should be noted that the values compiled in Tables A3, A4 and A5 **are not dry-mass corrected**.

Note that none of the values in Tables A3, A4 and A5 are certified, but are given as information on the typical relative performance of the various EPA SW-846 methods for the determination of elements and organic compounds in SRM 2706. The data should not be used for any other purpose. **The certified values provided in the Certificate of Analysis, are the best estimates of the true mass fractions for this material.**

Table A1. Laboratories Supporting the Multi-Laboratory Study of SRM 2706

P. Worby, N. Cole Accutest Laboratories 2235 US Highway 130 Dayton, NJ, USA	J. Todaro, A. Rice Alpha Laboratories 8 Walkup Drive Westborough, MA, USA	R. McLeod, S. Kennedy ALS Life Sciences 1435 Norjohn Court Burlington, ON, Canada
J.R. Bourbon EPA Region 2 2890 Woodbridge Avenue Edison, NJ, USA	F. Wellbourne, J. Hendel EPA Region 4 980 College Station Road Athens, GA, USA	S. Petzinger, C. Bremer, A. Pender Pace Analytical Services 1700 Elm Street Minneapolis, MN, USA
P. Westrick Pace Analytical Services 1638 Roseytown Road Greensburg, PA, USA	M. Acierno Test America Edison 777 New Durham Road Edison, NJ 08817-2859	D. Lowe, M. Bruce Test America Pittsburgh 301 Alpha Drive, RIDC Park Pittsburgh, PA, USA

Table A2. Glossary of EPA Methods used in the Multi-Laboratory Study of SRM 2706

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>
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>

Table A3. Results from Laboratories for Elements by EPA Analytical Methods

Element	<i>n</i>	Range (mg/kg)		Median (mg/kg)	Mean (mg/kg)	Recovery (%) <sup>(a)</sup>
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

<sup>(a)</sup> Recovery (mean value) relative to the certified/reference value, where applicable

Table A4. Results from Laboratories for Polycyclic Aromatic Hydrocarbons and Polychlorinated Biphenyls by SW-846 Methods

	<i>n</i>	Range (µg/kg)		Median (µg/kg)	Mean (µg/kg)	Recovery (%) <sup>(a)</sup>
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	--

<sup>(a)</sup> Recovery (mean value) relative to the certified/reference value, where applicable

Table A5. Results from Laboratories for Chlorinated Dibenzo-*p*-dioxin Congeners and Chlorinated Dibenzofuran Congeners by SW-846 Methods

	<i>n</i>	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