



Certificate of Analysis

Standard Reference Material® 2709a

San Joaquin Soil

Baseline Trace Element Concentrations

This Standard Reference Material (SRM) is intended primarily for use in the analysis of soils, sediments, or other materials of a similar matrix. A unit of SRM 2709a consists of 50 g of dried, powdered, agricultural soil.

Certified Values: The certified concentrations of elements, expressed as mass fractions [1] on a dry-mass basis, are provided in Table 1. 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 concentrations of the elements reported in Table 1. Metrological traceability is to the SI unit of mass expressed as the derived unit of mass fraction.

Reference Values: The reference values for elements, expressed as mass fractions on a dry-mass basis, are provided in Table 2. The reference values are based on results obtained from a single NIST analytical method. 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 concentrations of the elements reported in Table 2. Metrological traceability is to the SI unit of mass expressed as the derived unit of mass fraction.

Information Values: Information values for elements based on results obtained from one NIST method, are provided in Table 3. Particle size measurements are provided in Figure 1. 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 2709a** is valid, within the measurement uncertainties specified, until **01 November 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.

E.A. Mackey and R.R. Greenberg, formerly of the NIST Analytical Chemistry Division, and S.E. Long of the NIST Chemical Sciences Division were responsible for coordination of the technical measurements.

Statistical analyses were performed by J.H. Yen of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

INSTRUCTIONS FOR USE

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 (dry mass - see *Drying*) 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. If volatile elements (i.e., arsenic, mercury, selenium) are to be determined, precautions should be taken in the dissolution of SRM 2709a to avoid volatilization losses.

Drying: To relate measurements to the certified, reference, and information values that are expressed on a dry-mass basis, users should determine a drying correction at the time of each analysis. The recommended drying procedure is oven drying for 2 h at 110 °C. Note that analytical determination of volatile elements (i.e., arsenic, mercury, selenium) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture is to be made to the data for volatile elements before comparing them to the certified values. This procedure ensures that these elements are not lost during drying. The mass loss on drying for this material as bottled was approximately 3 %, but this value may change once the bottle is opened and the soil is exposed to air.

SOURCE, PREPARATION, AND ANALYSIS

Source and Preparation of Material⁽¹⁾: The U.S. Geological Survey (USGS), under contract to NIST, collected and processed the soil for SRM 2709a with assistance from the U.S. Bureau of Reclamation's Sacramento CA office. The agricultural soil used to produce SRM 2709a was collected from a fallow field, in the central California San Joaquin Valley. Three separate collection sites were used to obtain the necessary amount of material. Each collection site covered an area of approximately 4 m². Prior to sample collection the area was scraped clean of surface vegetation. Collected material was transferred to 20 plastic-lined five-gallon plastic buckets and shipped to the USGS laboratory for processing. At USGS, the SRM 2709a soil was dried at room temperature, disaggregated, and sieved to remove coarse material (≥ 2 mm). The resulting soil was ball-milled in 50 kg portions, and then the entire batch of soil was transferred to a cross-flow V-blender for mixing. The blended soil was radiation sterilized prior to bottling. In the final preparation step the blended material was split into containers using a custom-designed spinning riffler, which was used to divide the material into smaller batches, and then used to apportion approximately 50 g into each pre-cleaned bottle.

Every 100th bottle was set aside for chemical analyses designed to assess material homogeneity using X-ray fluorescence spectrometry (XRF), inductively coupled plasma optical emission spectrometry (ICP-OES), and inductively coupled plasma mass spectrometry (ICP-MS) at the USGS. Homogeneity assessments were performed at NIST as well, and results indicated that additional processing was needed to achieve optimum homogeneity. The material from all bottles was combined, and then ground in batches between stainless steel plates for a time sufficient to produce a powder of which ≥ 95 %, by mass, passed through a 200 mesh (74 μ m) sieve. The resulting powder was blended, and 50 g portions were dispensed into bottles using the spinning riffler. Results from additional analyses indicated material homogeneity was acceptable (see below).

Homogeneity Testing: The homogeneity was assessed for elements in the bottled material using X-ray fluorescence spectrometry and instrumental neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the expanded uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity for most elements is ≤ 1 %, for calcium it is approximately 2 %, and for chromium it is approximately 3 %. Significant material heterogeneity was observed for mercury, for which a reference value with a prediction interval is provided; see Table 2.

Analysis: Analyses of this material were performed at NIST and at the USGS (Denver, CO). Results from NIST were used to provide the certified, reference, and information values shown in Tables 1, 2, and 3 respectively. Results from the USGS were used to confirm those values. The analytical techniques used for each element are listed in Table 4; the analysts are listed in Tables 5 and 6.

Particle Size Measurements: Particle size measurements for SRM 2709a were made using a Malvern Mastersizer 3000 laser-based light scattering system and the particle size distribution is shown in Figure 1.

⁽¹⁾ 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.

Table 1. Certified Values^(a) (Dry-Mass Basis) for Elements in SRM 2709a

Element	Mass Fraction (%)	Coverage Factor, <i>k</i>	Element	Mass Fraction (mg/kg)	Coverage Factor, <i>k</i>
Aluminum (Al)	7.37 ± 0.16	2.04	Antimony (Sb)	1.55 ± 0.06	2.36
Calcium (Ca)	1.91 ± 0.09	2.05	Barium (Ba)	979 ± 28	2.57
Iron (Fe)	3.36 ± 0.07	2.23	Cadmium (Cd)	0.371 ± 0.002	2
Magnesium (Mg)	1.46 ± 0.02	2.02	Chromium (Cr)	130 ± 9	2.05
Phosphorus (P)	0.0688 ± 0.0013	2.12	Cobalt (Co)	12.8 ± 0.2	2.45
Potassium (K)	2.11 ± 0.06	2.45	Lead (Pb)	17.3 ± 0.1	2
Silicon (Si)	30.3 ± 0.4	2.57	Manganese (Mn)	529 ± 18	2.16
Sodium (Na)	1.22 ± 0.03	2.02	Strontium (Sr)	239 ± 6	2.26
Titanium (Ti)	0.336 ± 0.007	2.26	Vanadium (V)	110 ± 11	2.10
			Zirconium (Zr)	195 ± 46	2.10

^(a) Certified values for all elements except cadmium and lead are the equally weighted means of results from two or three 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]. A component for material heterogeneity is incorporated into the uncertainties of aluminum, calcium, chromium, manganese, and sodium. The coverage factor (k) is determined from the Student's t -distribution corresponding to the appropriate associated degrees of freedom and approximately 95 % confidence for each analyte. The certified values for cadmium and lead are each results from a single NIST method (isotope dilution (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, and k is the coverage factor corresponding to approximately 95 % confidence for each analyte.

Table 2. Reference Values^(a) (Dry-Mass Basis) for Elements in SRM 2709a

Element	Mass Fraction (mg/kg)
Arsenic (As)	10.5 ± 0.3
Cerium (Ce)	42 ± 1
Cesium (Cs)	5.0 ± 0.1
Copper (Cu)	33.9 ± 0.5
Europium (Eu)	0.83 ± 0.02
Gadolinium (Gd)	3.0 ± 0.1
Lanthanum (La)	21.7 ± 0.4
Mercury (Hg) ^(b)	0.9 ± 0.2
Nickel (Ni)	85 ± 2
Rubidium (Rb)	99 ± 3
Scandium (Sc)	11.1 ± 0.1
Thallium (Tl)	0.58 ± 0.01
Thorium (Th)	10.9 ± 0.2
Uranium (U)	3.15 ± 0.05
Zinc (Zn)	103 ± 4

^(a) Reference values for all elements are based on results from one analytical method at NIST. Uncertainty values represent the expanded uncertainties 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, and k is the coverage factor ($k = 2$) corresponding to approximately 95 % confidence for each analyte.

^(b) The reference value for mercury represents the average value from analysis of portions from six bottles using ID-cold vapor (CV)-ICP-MS. Results indicate significant material heterogeneity; values ranged from 0.8 mg/kg to 1.1 mg/kg. The uncertainty for this reference value is in the form of a prediction interval.

Table 3. Information Values^(a) (Dry Mass Basis) for Elements in SRM 2709a

Element	Mass Fraction (mg/kg)
Boron (B)	74
Dysprosium (Dy)	3
Hafnium (Hf)	4
Lutetium (Lu)	0.3
Neodymium (Nd)	17
Samarium (Sm)	4
Selenium (Se)	1.5
Tantalum (Ta)	0.7
Terbium (Tb)	0.5
Ytterbium (Yb)	2

^(a) Information values are based on results from one analytical method at NIST.

SUPPLEMENTAL INFORMATION FOR SRM 2709a

Particle Size Measurements: Particle size measurements for SRM 2709a were made using a Malvern Mastersizer 3000 laser-based light scattering system. Approximately 0.5 g of SRM 2709a material was measured using water as the dispersant (refractive index: 1.330). Sample was introduced into the measurement cell before ten individual measurements were made at an obscuration of 14 % – 18 % of the laser beam. The information values calculated 10th ($D_v(10)$), 50th ($D_v(50)$) and 90th ($D_v(90)$) percentile particle sizes are $D_v(10) = 2.62 \mu\text{m}$, $D_v(50) = 12.6 \mu\text{m}$, and $D_v(90) = 71.7 \mu\text{m}$. The volume fraction of material smaller than $10.1 \mu\text{m}$ in diameter is approximately 44 %. The particle size distribution is shown in Figure 1.

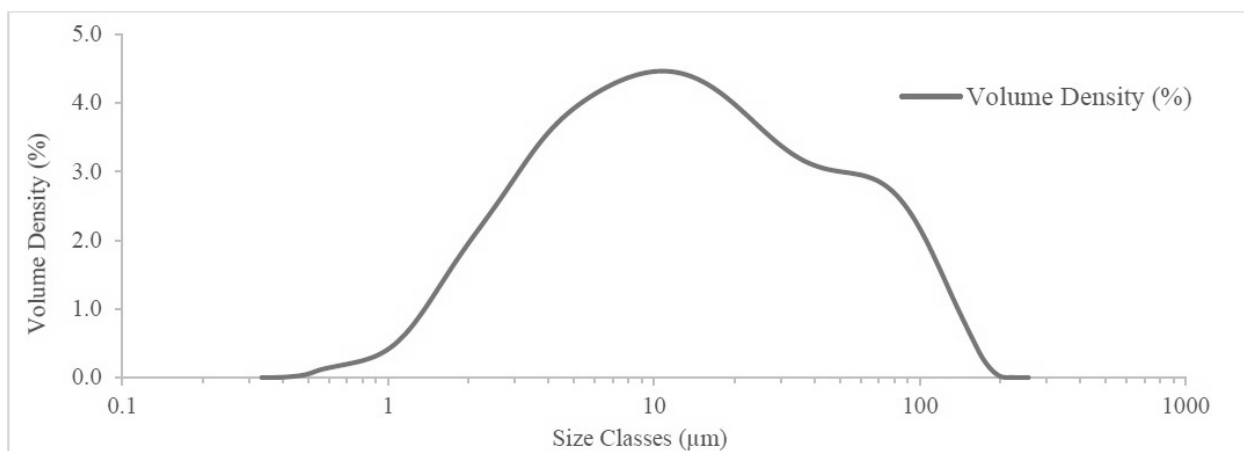


Figure 1. Particle size distribution in SRM 2709a

Table 4. Methods^(a,b) Used for the Analysis of SRM 2709a

Element	Methods	Element	Methods
Al	INAA; XRF	Na	NAA; XRF
As	INAA	Nd	INAA
B	PGAA	Ni	ICP-MS
Ba	ICP-OES; INAA; XRF	P	ICP-OES; XRF
Ca	INAA; XRF	Pb	ID-ICP-MS
Cd	ID-ICP-MS; PGAA	Rb	INAA
Ce	INAA	Sb	INAA; ICP-MS
Co	INAA; ICP-OES	Sc	INAA
Cr	INAA; XRF	Se	CCT-ICP-MS
Cs	INAA	Si	PGAA; XRF
Cu	ICP-MS	Sm	INAA
Dy	INAA	Sr	INAA; ICP-OES; XRF
Eu	INAA	Ta	INAA
Fe	INAA; PGAA; XRF	Tb	INAA
Gd	PGAA	Th	INAA
Hf	INAA	Ti	INAA; PGAA; XRF
Hg	CV-ID-ICP-MS	Tl	ICP-MS
K	INAA; PGAA; XRF	U	ICP-MS
La	INAA	V	INAA; XRF
Lu	INAA	Yb	INAA
Mg	INAA; XRF	Zn	INAA
Mn	INAA; PGAA; XRF	Zr	INAA; XRF

^(a) NIST Methods of Analysis:

CCT-ICP-MS	Collision Cell Inductively Coupled Plasma Mass Spectrometry
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
ID-ICP-MS	Isotope Dilution Inductively Coupled Plasma Mass Spectrometry
INAA	Instrumental Neutron Activation Analysis
PGAA	Prompt Gamma-Ray Activation Analysis
XRF	X-ray Fluorescence Spectrometry

^(b) USGS Methods of Analysis (used to confirm results from certification methods).

WD-XRF	Wavelength Dispersive X-ray Fluorescence Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectrometry
ICP-MS	Inductively Coupled Plasma Mass Spectrometry

Table 5. Participating NIST Analysts:

S.J. Christopher	A.F. Marlow	S.A. Rabb	B.E. Tomlin
D.L. Ellisor	K.E. Murphy	J.R. Sieber	L.J. Wood
R.M. Lindstrom	J.M. Ness	R.O. Spatz	L.L. Yu
S.E. Long	R.L. Paul	R.S. Popelka-Filcoff	R. Zeisler
E.A. Mackey			

Table 6. Participating USGS Laboratory and Analysts

Laboratory	Analysts
U.S. Geological Survey Branch of Geochemistry (Denver, CO)	M.G. Adams; Z.A. Brown; P.L. Lamothe; J.E. Taggart; S.A. Wilson

REFERENCES

- [1] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*, NIST Special Publication 811 (2008); available at <https://www.nist.gov/pml/special-publication-811> (accessed Nov 2018).
- [2] May, W.E.; Gills, T.E.; Parris, R.; Beck, II, C.M.; Fassett, J.D.; Gettings, R.J.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.; MacDonald, B.S.; Wise, S.A.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (1999); available at <https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF> (accessed Nov 2018).
- [3] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105, pp. 571-579 (2000).
- [4] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utis/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 2018); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at <https://www.nist.gov/pml/nist-technical-note-1297> (accessed Nov 2018).

Certificate Revision History: 02 November 2018 (Updated uncertainty calculation information; added particle size distribution; change of expiration date; editorial changes); 07 April 2009 (Original certificate date).

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; or via the Internet at <https://www.nist.gov/srm>.

Appendix A

Leachable Concentrations Determined Using USEPA Methods 200.7 and 3050B

The mass fraction values contained in the NIST Certificate of Analysis for SRM 2709a represent the total element content of the material. The measurement results used to provide the certified, reference, or information values are obtained from methods that require complete sample decomposition, or from nondestructive analytical methods such as instrumental neutron activation analysis or prompt gamma-ray activation analysis. Where complete sample decomposition is required, it can be accomplished by digestion with mixed acids or by fusion. For mixed-acid decomposition, hydrofluoric acid must be included in the acid mixture used to totally decompose siliceous materials such as soils and sediments.

In its monitoring programs, the U.S. Environmental Protection Agency (USEPA) has established a number of leach methods for the preparation of soil samples for the determination of extractable elements. Eight laboratories participated, seven of which used USEPA Method 200.7; the remaining laboratory used USEPA SW-846 Method 3050B for preparation of soil samples. All elements were determined in leachates by inductively coupled plasma optical emission spectrometry. Six of the eight laboratories provided individual results from duplicate portions, and these results were averaged together to provide one result for each element from each participating laboratory. Results rejected as outliers by the USEPA Contract Laboratory Program (CLP) officials were not included. Results are summarized in Table A1. The ranges of mass fraction values, median values (to two significant figures), and the number of results included for each are given for 23 elements. The percent recovery values based on the ratios of the median values to the total element content (from the certified, reference, or information values in the Certificate of Analysis) are listed in the last column of Table A1. **Note that the certified values provided as total mass fractions in the Certificate of Analysis are the best estimate of the true mass fraction values for this material.**

This USEPA CLP Study was coordinated by Clifton Jones, Quality Assurance and Technical Support Program, Shaw Environmental & Infrastructure Group (Las Vegas, NV) under the direction of J. Nebelsick, USEPA, Analytical Services Branch. The participating laboratories are listed below the table.

Table A1. Results from Laboratories Participating in the EPA Contract Laboratory Program Study.

Element	n	Range (mg/kg)	Median (mg/kg)	Recovery (%)
Aluminum	7	13000 – 17000	16000	22
Antimony	2	1.2 – 1.5	1.4	88
Arsenic	8	6.4 – 10	7.8	74
Barium	8	350 – 400	380	39
Beryllium	7	0.50 – 0.72	0.61	—
Cadmium	5	0.33 – 0.66	0.40	110
Calcium	8	12000 – 14000	12000	65
Chromium	8	46 – 67	53	41
Cobalt	8	8.2 – 13	10	81
Copper	7	24 – 28	27	81
Iron	8	22000 – 26000	24000	70
Lead	7	8.1 – 11	9.2	53
Magnesium	7	9700 – 11000	10000	71
Manganese	8	380 – 450	420.0	79
Mercury	8	0.79 – 0.92	0.87	97
Nickel	8	59 – 71	66	77
Potassium	8	2600 – 4000	2900	14
Selenium	5	0.69 – 1.9	0.95	63
Silver	4	0.14 – 4.1	0.64	—
Sodium	7	460 – 610	500	4
Thallium	2	0.74 – 1.6	1.2	200
Vanadium	8	43 – 71	48	44
Zinc	8	69 – 87	79	77

List of CLP and non-CLP Participating Laboratories: A4 Scientific, Inc.; Bonner Analytical Testing Co.; Chem Tech Consulting Group; Datachem Laboratories, Inc.; Liberty Analytical Corporation; MSE Laboratory Services; Shealy Environmental; SVL Analytical Inc.