



National Institute of Standards & Technology

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. One unit of SRM 2709a consists of 50 g of dried, powdered, agricultural soil.

Certified Values: The certified concentrations for 19 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].

Reference Values: The reference values for 15 constituents, 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].

Information Values: The values for 10 elements are provided in Table 3 for information purposes only. These are non-certified values with no uncertainty assessed. The information values included in this certificate are based on results obtained from one NIST method.

Expiration of Certification: The certification of SRM 2709a is valid, within the measurement uncertainties specified, until **1 November 2018**, 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) will facilitate notification.

E.A. Mackey and R.R. Greenberg of the NIST Analytical Chemistry Division were responsible for coordination of the technical measurements leading to certification.

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

The support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 7 April 2009

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 *Instructions for 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 Material¹: 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 $\geq 95\%$, by mass, passed through a 200 mesh (74 μm) 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).

Analysis: The homogeneity was assessed for selected 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 $\leq 1\%$, 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.

Analyses of this material were performed at NIST (Gaithersburg, MD) 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.

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 Selected Elements in SRM 2709a

Element	Mass Fraction (%)	Element	Mass Fraction (mg/kg)
Aluminum	7.37 ± 0.16	Antimony	1.55 ± 0.06
Calcium	1.91 ± 0.09	Barium	979 ± 28
Iron	3.36 ± 0.07	Cadmium	0.371 ± 0.002
Magnesium	1.46 ± 0.02	Chromium	130 ± 9
Phosphorus	0.0688 ± 0.0013	Cobalt	12.8 ± 0.2
Potassium	2.11 ± 0.06	Lead	17.3 ± 0.1
Silicon	30.3 ± 0.4	Manganese	529 ± 18
Sodium	1.22 ± 0.03	Strontium	239 ± 6
Titanium	0.336 ± 0.007	Vanadium	110 ± 11
		Zirconium	195 ± 46

^(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, with coverage factor determined by the Student's t distribution, calculated by combining a between-method variance with a pooled within-method variance [3] following the ISO Guide [4]. A component for material heterogeneity is incorporated into the uncertainties of aluminum, calcium, chromium, manganese, and sodium. The certified values for cadmium and lead are each results from a single NIST method (isotope dilution (ID)-ICP-MS) for which a complete evaluation of all sources of uncertainty has been performed. The uncertainty for the certified value for cadmium and lead represents an expanded uncertainty with a coverage factor of 2, with uncertainty components combined following the ISO Guide [4].

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

Element	Mass Fraction (mg/kg)
Arsenic	10.5 ± 0.3
Cerium	42 ± 1
Cesium	5.0 ± 0.1
Copper	33.9 ± 0.5
Europium	0.83 ± 0.02
Gadolinium	3.0 ± 0.1
Lanthanum	21.7 ± 0.4
Mercury ^(b)	0.9 ± 0.2
Nickel	85 ± 2
Rubidium	99 ± 3
Scandium	11.1 ± 0.1
Thallium	0.58 ± 0.01
Thorium	10.9 ± 0.2
Uranium	3.15 ± 0.05
Zinc	103 ± 4

^(a) Reference values for all elements are based on results from one analytical method at NIST. Uncertainty values represent the expanded uncertainties which include the combined Type A and Type B with a coverage factor of 2, following the ISO Guide [4].

^(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 Selected Elements in SRM 2709a

Element	Mass Fraction (mg/kg)
Boron	74
Dysprosium	3
Hafnium	4
Lutetium	0.3
Neodymium	17
Samarium	4
Selenium	1.5
Tantalum	0.7
Terbium	0.5
Ytterbium	2

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

Table 4. Methods Used for the Analysis of SRM 2709a

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

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

USGS Methods of Analysis^(a)

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

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

Table 5. Participating NIST Analysts:

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

Table 6. Participating USGS Laboratory and Analysts

Laboratory	Analysts
U.S. Geological Survey	M.G. Adams
Branch of Geochemistry	Z.A. Brown
Denver, CO, USA	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 <http://physics.nist.gov/Pubs/>
- [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 http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.
- [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.; J. Res. NIST 105, pp. 571-579 (2000).
- [4] ISO; *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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 <http://physics.nist.gov/Pubs/>.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <http://www.nist.gov/srm>.

Addendum to Certificate

Standard Reference Material® 2709a

San Joaquin Soil

Baseline Trace Element Concentrations

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 John Nebelsick, USEPA, Analytical Services Branch. The participating laboratories are listed in Table A2.

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

Table A2. 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.