National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2709

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. SRM 2709 is an agricultural soil that was oven-dried, sieved, radiation sterilized, and blended to achieve a high degree of homogeneity. A unit of SRM 2709 consists of 50 g of the dried material.

The certified elements for SRM 2709 are given in Table 1. The values are based on measurements using one definitive method or two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should **NOT** be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

NOTICE AND WARNINGS TO USERS

Expiration of Certification: This certification of SRM 2709 is valid, within the measurement uncertainties specified, until **31 December 2011**, provided the SRM is handled in accordance with 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. Return of the attached registration card will facilitate notification.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and R.L. Watters, Jr. of the NIST Inorganic Analytical Research Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by T.E. Gills and J.S. Kane. Revision of this certificate was coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

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Certificate Issue Date: 18 July 2003
See Certificate Revision History on Page 6

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INSTRUCTIONS FOR USE

Use: A minimum sample weight of 250 mg (dry weight - see *Instructions for Drying*) should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., mercury (Hg), arsenic (As), selenium (Se)) are to be determined, precautions should be taken in the dissolution of SRM 2709 to avoid volatilization losses.

Instructions for Drying: When nonvolatile elements are to be determined, samples should be dried for 2 h at 110 °C. Volatile elements (i.e., Hg, As, Se) 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 to the certified values. This procedure ensures that these elements are not lost during drying. The weight loss on drying has been found to be in the range of 1.8 % to 2.5 %.

PREPARATION AND ANALYSIS

Source and Preparation of Material: The U.S. Geological Survey (USGS), under contract to NIST, collected and processed the material for SRM 2709. The soil was collected from a plowed field, in the central California San Joaquin Valley, at Longitude 120° 15' and Latitude 36° 30'. The collection site is in the Panoche fan between the Panoche and Cantu creek beds. The top 7.5 to 13 cm (3 to 5 in) of soil containing sticks and plant debris was removed, and the soil was collected from the 13 cm level down to a depth of 46 cm (18 in) below the original surface. The material was shoveled into 0.114 m^3 (30 gal) plastic buckets and shipped to the USGS laboratory for processing.

The material was spread on 30.5 cm × 61 cm (1 ft × 2 ft) polyethylene-lined drying trays in an air drying oven and dried for three days at room temperature. The material was then passed over a vibrating 2 mm screen to remove plant material, rocks, and large chunks of aggregated soil. Material remaining on the screen was deaggregated and rescreened. The combined material passing the screen was ground in a ball mill to pass a 74 μm screen and blended for 24 h. Twenty grab samples were taken and measured for the major oxides using X-ray fluorescence spectrometry and for several trace elements using inductively coupled plasma atomic emission analysis to provide preliminary assessment of the homogeneity of the material prior to bottling. The material was bottled into 50 g units and randomly selected bottles were taken for the final homogeneity testing.

Analysis: The homogeneity, using selected elements in the bottled material as indicators, was assessed using X-ray fluorescence spectrometry and neutron activation analysis. In a few cases, statistically significant differences were observed, and the variance due to material inhomogeneity is included in the overall uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity is less than 1 % for those elements for which homogeneity was assessed.

Certified Values and Uncertainties: The certified values are weighted means of results from two or more independent analytical methods, or the mean of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry used by two different laboratories employing different methods of sample preparation prior to measurement. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel [1]. The stated uncertainty includes allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95 % prediction interval and includes an allowance for systematic error among the methods used. In the absence of systematic error, a 95 % prediction interval predicts where the true concentrations of 95 % of the samples of this SRM lie. The certified values were corroborated by analyses from nine Polish laboratories cooperating on the certification under the direction of T. Plebanski and J. Lipinski, Polish Committee for Standardization Measures and Quality Control. The Polish laboratory work was supported by the Maria Sklodowska-Curie Joint Fund.

Table 1. Certified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Aluminum	7.50 ± 0.06	Antimony	7.9 ± 0.6
Calcium	1.89 ± 0.05	Arsenic	17.7 ± 0.8
Iron	3.50 ± 0.11	Barium	968 ± 40
Magnesium	1.51 ± 0.05	Cadmium	0.38 ± 0.01
Phosphorus	0.062 ± 0.005	Chromium	130 ± 4
Potassium	2.03 ± 0.06	Cobalt	13.4 ± 0.7
Silicon	29.66 ± 0.23	Copper	34.6 ± 0.7
Sodium	1.16 ± 0.03	Lead	18.9 ± 0.5
Sulfur	0.089 ± 0.002	Manganese	538 ± 17
Titanium	0.342 ± 0.024	Mercury	1.40 ± 0.08
		Nickel	88 ± 5
		Selenium	1.57 ± 0.08
		Silver	0.41 ± 0.03
		Strontium	231 ± 2
		Thallium	0.74 ± 0.05
		Vanadium	112 ± 5
		Zinc	106 ± 3

Noncertified Values: Noncertified values, shown below, are provided for information only. An element concentration value may not be certified if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available.

Table 2. Noncertified Values

Element	Mass Fraction (%)	Element	Mass Fraction (µg/g)
Carbon	1.2	Cerium	42
		Cesium	5.3
		Dysprosium	3.5
		Europium	0.9
		Gallium	14
		Gold	0.3
		Hafnium	3.7
		Holmium	0.54
		Iodine	5
		Lanthanum	23
		Molybdenum	2.0
		Neodymium	19
		Rubidium	96
		Samarium	3.8
		Scandium	12
		Thorium	11
		Tungsten	2
		Uranium	3
		Ytterbium	1.6
		Yttrium	18
		Zirconium	160

Table 3. Analytical Methods Used for the Analysis of SRM 2709

Element	Certification Methods *	Element	Certification Methods *
Ag	ID ICPMS; RNAA	Mo	**ID ICPMS**
Al	XRF1; XRF2; INAA; DCP; **ICP**	Na	INAA; FAES; **ICP**
As	RNAA; HYD AAS; **INAA**	Nd	**ICP**
Au	**INAA; FAAS**	Ni	ID ICPMS; ETAAS; **INAA**
Ba	XRF2; FAES	P	DCP; COLOR; XRF2
C	**COUL**	Pb	ID TIMS
Ca	XRF1; XRF2; DCP	Rb	**INAA**
Cd	ID ICPMS; RNAA	S	ID TIMS
Ce	**INAA; ICP**	Sb	INAA; ETAAS
Co	INAA; ETAAS; **ICP**	Sc	**INAA; ICP**
Cr	INAA; DCP; **ICP**	Se	RNAA; HYD AAS
Cs	**INAA**	Si	XRF1; XRF2; GRAV
Cu	RNAA; FAES; **ICP**	Sm	**INAA**
Dy	**INAA**	Sr	ID TIMS; **INAA; ICP**
Eu	**INAA**	Th	**ID TIMS; INAA; ICP**
Fe	XRF1; XRF2; INAA; DCP	Ti	INAA; XRF1; XRF2; DCP
Ga	**INAA; ICP**	Tl	ID TIMS; LEAFS
Hf	**INAA**	U	**ID TIMS; INAA**
Hg	CVAAS	V	INAA; ICP
Ho	**INAA**	W	**INAA**
I	**INAA**	Y	**ICP**
K	XRF1; XRF2; FAES; **ICP; INAA**	Yb	**INAA**
La	**INAA; ICP**	Zn	ID TIMS; **ICP: INAA; POLAR**
Mg	INAA; XRF1; ICP	Zr	**INAA**
Mn	INAA; ICP		

*Methods in **bold** were used to corroborate certification methods or to provide information values.

COLOR	Colorimetry; lithium metaborate fusion.
COUL	Combustion coulometry.
CVAAS	Cold vapor atomic absorption spectrometry.
DCP	Direct current plasma atomic emission spectrometry; lithium metaborate fusion.
ETAAS	Electrothermal atomic absorption spectrometry; mixed acid digestion.
FAAS	Flame atomic absorption spectrometry; mixed acid digestion except for Au, leached with HBr-Br_2.
FAES	Flame atomic emission spectrometry; mixed acid digestion.
GRAV	Gravimetry; sodium carbonate fusion.
HYD AAS	Hydride generation atomic absorption spectrometry.
ICP	Inductively coupled plasma atomic emission spectrometry; mixed acid digestion.
ID ICPMS	Isotope dilution inductively coupled plasma mass spectrometry; mixed acid digestion.
ID TIMS	Isotope dilution thermal ionization mass spectrometry; mixed acid digestion.
INAA	Instrumental neutron activation analysis.
LEAFS	Laser enhanced atomic fluorescence spectrometry; mixed acid digestion.
POLAR	Polarography.
RNAA	Radiochemical neutron activation analysis; mixed acid digestion.
XRF1	Wavelength dispersive X-ray fluorescence on fused borate discs.
XRF2	Wavelength dispersive X-ray fluorescence spectrometry on pressed powder.

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Certificate Revision History: 18 July 2003 (The description of the SRM has been updated to include that this SRM was radiation sterilized, which was previously omitted); 18 January 2002 (This revision reflects a change in the certification expiration date); 23 August 1993 (Addendum added); 30 October 1992 (Original certificate date).

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) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

Addendum to Certificates

SRM 2709 San Joaquin Soil
SRM 2710 Montana Soil
SRM 2711 Montana Soil

Leachable Concentrations Using U.S. EPA Method 3050 for Flame Atomic Absorption Spectrometry (FAAS) and Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)

The certified concentrations of constituent elements in essentially all National Institute of Standards and Technology (NIST) chemical composition Standard Reference Materials (SRMs) are given as total concentrations. The certified concentrations are based on measurements obtained by two or more independent methods or techniques. The measurement methods require complete sample decomposition, or the sample may be analyzed nondestructively. 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.

For a number of environmental monitoring purposes, the concentrations of labile or extractable fractions of elements are more useful than total concentrations. Concentrations of labile or extractable fractions are generally determined using relatively mild leach conditions, which are unlikely to totally decompose the sample. It should be noted that results obtained using the mild leach conditions are often erroneously depicted in reports as total concentrations. However, reported concentrations of labile or extractable fractions of elements are generally lower than total concentrations; recovery can be total if an element in a given sample is completely labile. Results are often presented as measured concentration in the leachate in comparison to the total or certified concentration. The recovery of an element as a percent of total concentration is a function of several factors such as the mode of occurrence in the sample, leach medium, leach time and temperature conditions, and pH of the sample-leach medium mixture. References [1] through [27] may be consulted for detailed discussions of these factors and their effect on leach results. Some of these references provide leach data for one or more reference materials.

In its monitoring programs, the U.S. Environmental Protection Agency (EPA) has established a number of leach methods for the determination of labile or extractable elements. They include Methods 3015, 3050, and 3051. A number of cooperating laboratories using the variation to U.S. EPA Method 3050 for Flame Atomic Absorption Spectrometry (FAAS) and Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) measurements, have reported data for SRMs 2709, 2710, and 2711. This variation of the method uses hydrochloric acid in its final step, which is different from Method 3050 for ICP-MS and Hydride Generation-Atomic Absorption Spectrometry (HG-AAS) measurements. The data obtained are presented in Tables 1, 2, and 3 of this addendum. The names of the cooperating laboratories are listed in Table 4. Several laboratories provided replicate (3 to 6) analyses for each of the three soil SRMs. The number of results for a given element varied from only one to as many as nine, as indicated in the data presented in Tables 1 through 3. Because of the wide range of interlaboratory results for most elements, only the data range and median of the individual laboratory means are given. Ranges differ somewhat from those in reference [26], since this addendum is based on a larger data set than had been available previously.

For SRMs 2710 and 2711, 17 laboratories provided data as part of contract work for the U.S. EPA. Each SRM was treated as a blind sample in one quarter of 1992. Since there was no within-laboratory replication of analysis in the design of the exercise, the 17-laboratory means of results were treated as single laboratory results from laboratories using replication, in establishing the median of the full data set. In a few cases, however, the contract laboratories mean was the only result available for a particular element (e.g., Antimony in SRM 2710). In others, the contract laboratories mean is also the median for the full leach data set (e.g., Arsenic in SRM 2710). An asterisk identifies those cases where the contract laboratories' means are given as the median value.

Please note none of the values in Tables 1 through 3 are certified, but are given as information on the performance of the three soils when used to evaluate, or to provide quality control for Method 3050 followed by FAAS and ICP-AES measurements only. The data should not be used for any other purpose. **The certified values, provided as total concentrations, are the best estimate of the true concentrations.**

Gaithersburg, MD 20899 John Rumble, Jr., Chief

Table 1. Leach Data from Cooperating Laboratories for Soil SRM 2709

Element	Range			Median	N	% Leach Recovery†
			Wt %			
Aluminum	2.0	-	3.1	2.6	5	35
Calcium	1.4	-	1.7	1.5	5	79
Iron	2.5	-	3.3	3.0	8	86
Magnesium	1.2	-	1.5	1.4	5	93
Phosphorus	0.05	-	0.07	0.07	3	100
Potassium	0.26	-	0.37	0.32	5	16
Silicon	---		---	< 0.01	1	< 1
Sodium	0.063	-	0.11	0.068	4	6
Titanium	0.03	-	0.04	0.038	3	11
			mg/kg			
Antimony	---		---	< 10	1	...
Arsenic	---		---	< 20	2	...
Barium	392	-	400	398	2	41
Cadmium	---		---	< 1	5	...
Chromium	60	-	115	79	5	61
Cobalt	10	-	15	12	5	90
Copper	26	-	40	32	7	92
Lead	12	-	18	13	5	69
Manganese	360	-	600	470	7	87
Molybdenum	---		---	< 2	2	...
Nickel	65	-	90	78	7	89
Selenium	nr	-	nr	0.014	1	< 1
Strontium	100	-	112	101	3	44
Vanadium	51	-	70	62	3	55
Zinc	87	-	120	100	7	94

$$\text{† \% Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}} \right]$$

--- at or below the detection limit
... no % Leach Recovery calculated
nr no range reported by the laboratory

Table 2. Leach Data from Cooperating Laboratories for Soil SRM 2710

Element	Range			Median	N	% Leach Recovery†
			Wt %			
Aluminum	1.2	-	2.6	1.8	6	28
Calcium	0.38	-	0.48	0.41	7	33
Iron	2.2	-	3.2	2.7	9	80
Magnesium	0.43	-	0.60	0.57	6	67
Phosphorus	0.106	-	0.11	0.11	2	100
Potassium	0.37	-	0.50	0.45	6	21
Silicon	---		---	< 0.01	1	< 1
Sodium	0.049	-	0.062	0.054	5	5
Titanium	0.092	-	0.11	0.10	3	35
			mg/kg			
Antimony	3.4	-	12	7.9*	1*	21
Arsenic	490	-	600	590	3	94
Barium	300	-	400	360	3	51
Cadmium	13	-	26	20	8	92
Chromium	15	-	23	19	6	(49)
Cobalt	6.3	-	12	8.2	7	(82)
Copper	2400	-	3400	2700	8	92
Lead	4300	-	7000	5100	8	92
Manganese	6200	-	9000	7700	8	76
Mercury	27	-	37	32*	1*	98
Molybdenum	13	-	27	20	2	(100)
Nickel	8.8	-	15	10.1	8	71
Silver	24	-	30	28	3	79
Selenium	nr	-	nr	0.002	1	...
Strontium	94	-	110	100	3	(42)
Thallium	0.50	-	0.76	0.63*	1*	(48)
Vanadium	37	-	50	43	4	56
Zinc	5200	-	6900	5900	9	85

$$\text{† \% Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}} \right]$$

( ) indicates that information value was used
--- at or below the detection limit
... no % Leach Recovery could be calculated
nr no range reported by the laboratory
* U.S. EPA contact laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 3. Leach Data from Cooperating Laboratories for Soil SRM 2711

Element	Range			Median	N	% Leach Recovery†
			Wt %			
Aluminum	1.2	-	2.3	1.8	5	28
Calcium	2.0	-	2.5	2.1	5	73
Iron	1.7	-	2.6	2.2	7	76
Magnesium	0.72	-	0.89	0.81	5	77
Phosphorus	0.06	-	0.09	0.088	3	100
Potassium	0.26	-	0.53	0.38	5	16
Silicon	---		---	< 0.01	1	< 1
Sodium	0.020	-	0.029	0.026	4	2.3
Titanium	0.039	-	0.048	0.042	2	14
			mg/kg			
Antimony	...		...	< 10	1	...
Arsenic	88	- -	110	90	3	86
Barium	170	- -	260	200	2	28
Cadmium	32	-	46	40	6	96
Chromium	15	-	25	20	4	(43)
Cobalt	7	-	12	8.2	5	(82)
Copper	91	-	110	100	6	88
Lead	930	- -	1500	1100	7	95
Manganese	400	-	620	490*	7	77
Molybdenum	---		---	< 2	2	...
Nickel	14	-	20	16	7	78
Silver	2.5	-	5.5	4.0	1	86
Selenium	nr	-	nr	0.009	1	< 1
Strontium	48	-	55	50	3	20
Vanadium	34	- -	50	42	3	51
Zinc	290	- -	340	310	7	89

$$\dagger\ \%\ \text{Leach Recovery} = 100 \times \left[\frac{\text{Median Value}}{\text{Certified/Information Value}} \right]$$

( ) indicates that information value was used
--- at or below the detection limit
... no % Leach Recovery could be calculated
nr no range reported by the laboratory
* U.S. EPA contact laboratories mean; treated as one laboratory since no within-laboratory replication; see text

Table 4. Leach Study for Cooperating Laboratories

SRMs 2709, 2710, and 2711

S.A. Wilson: U.S. Geological Survey; Lakewood, CO, USA

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M. Paul: Research Institute of Vegetable Crops; Skierniewice, Poland

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Z. Jonca: Institute of Environmental Protection; Warsaw, Poland

B. Ksiazek: Geological Enterprise; Warsaw, Poland

I. Twardowska: Polish Academy of Sciences, Institute of Environmental Engineering; Zabrze, Poland

SRMs 2710 and 2711

L. Butler and D. Hillman: U.S. Environmental Protection Agency; Las Vegas, NV, and 17 contract laboratories

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