



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2710a

Montana I Soil

Highly Elevated 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 2710a consists of 50 g of the dried, powdered soil, blended with lead oxide.

Certified Values: The certified concentrations for 22 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 13 constituents, expressed as mass fractions on a dry-mass basis, are provided in Table 2. Ten reference values are based on results obtained from a single NIST analytical method, and three are based on results from two NIST analytical methods. 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 13 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 2710a is valid, within the measurement uncertainties specified, until **1 January 2019**, 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.

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) will be determined, precautions should be taken in the dissolution of SRM 2710a 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 must 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 mass loss on drying for this material as bottled was approximately 2 %, 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 material for SRM 2710a. The original collection site used for SRM 2710 was no longer available due to remediation efforts by the Montana Department of Environmental Quality. An alternative nearby site, located within the flood plain of the Silver Bow Creek, was selected. The site is approximately five miles west of Butte, Montana. Soil for SRM 2710a was placed in 22 plastic-lined five-gallon buckets using a common garden spade. The buckets were sealed and transferred to the USGS using a commercial freight carrier. At the USGS, the SRM 2710a 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 together with an amount of lead oxide sufficient to achieve a mass fraction of 0.55 % lead in the final product. The entire ball-milled 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).

Analysis: The homogeneity was assessed for selected elements in the bottled material using X-ray fluorescence spectrometry and instrumental neutron activation analysis (INAA). The estimated relative standard deviation for material inhomogeneity is <1 % and no component for inhomogeneity was included in the expanded uncertainties of the certified or reference values.

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.

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,b) (Dry-Mass Basis) for Selected Elements in SRM 2710a

Element	Mass Fraction (%)			Element	Mass Fraction (mg/kg)		
Aluminum	5.95	±	0.05	Antimony	52.5	±	1.6
Arsenic	0.154	±	0.010	Barium	792	±	36
Calcium	0.964	±	0.045	Cadmium	12.3	±	0.3
Copper	0.342	±	0.005	Cobalt	5.99	±	0.14
Iron	4.32	±	0.08	Lanthanum	30.6	±	1.2
Lead	0.552	±	0.003	Mercury	9.88	±	0.21
Magnesium	0.734	±	0.038	Strontium	255	±	7
Manganese	0.214	±	0.006	Uranium	9.11	±	0.30
Phosphorus	0.105	±	0.004				
Potassium	2.17	±	0.13				
Silicon	31.1	±	0.4				
Sodium	0.894	±	0.019				
Titanium	0.311	±	0.007				
Zinc	0.418	±	0.015				

^(a) Certified values for all elements except lead and mercury 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 Guide [3,4]. 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.

^(b) The certified values for lead and mercury are each results from a single NIST method (see Table 4) for which a complete evaluation of all sources of uncertainty has been performed. The uncertainties for the certified values for these elements represent expanded uncertainties with a coverage factor of 2, with uncertainty components combined following the ISO Guide [4].

Table 2. Reference Values^(a,b,c) (Dry-Mass Basis) for Selected Elements in SRM 2710a

Element	Mass Fraction (mg/kg)	
Cesium	8.25 ±	0.11
Chromium	23 ±	6
Europium	0.82 ±	0.01
Gadolinium	3.0 ±	0.1
Lutetium	0.31 ±	0.01
Neodymium	22 ±	2
Nickel	8 ±	1
Rubidium	117 ±	3
Samarium	4.0 ±	0.2
Scandium	9.9 ±	0.1
Thallium	1.52 ±	0.02
Thorium	18.1 ±	0.3
Vanadium	82 ±	9

^(a) Reference values for all elements except chromium, nickel, samarium, and vanadium are based on results from one analytical method at NIST (see Table 4) and the uncertainties 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) Reference values for nickel and samarium are the equally weighted means of results from two analytical methods for nickel and two INAA experiments for samarium. 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 Guide [3,4]. 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.

^(c) Reference values for chromium and vanadium are based on a weighted mean calculated based on the Dersimonian-Laird method [5], which incorporates an estimate of the between-method variance into the weights. The expanded uncertainty listed with these values is calculated as $U = ku_c$, where $k = 2$, and 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.

Table 3. Information Values^(a) (Dry-Mass Basis) for Selected Elements in SRM 2710a

Element	Mass Fraction (mg/kg)
Boron	20
Cerium	60
Dysprosium	3
Gold	0.2
Hafnium	7
Indium	7
Selenium	1
Silver	40
Tantalum	0.9
Terbium	0.5
Tungsten	190
Ytterbium	2
Zirconium	200

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

Table 4. NIST Methods Used for the Analysis of SRM 2710a

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

WD-XRF-2	Wavelength dispersive X-ray fluorescence spectrometry at USGS
ICP-OES-2	Inductively coupled plasma optical emission spectrometry at USGS
ICP-MS-2	Inductively coupled plasma mass spectrometry at USGS

^(a)Two different INAA experiments, performed using different sub-samples and different analytical conditions, were used to provide certified and reference values for lanthanum and samarium, respectively.

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

Table 5. Participating NIST Analysts:

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

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

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- [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://www.physics.nist.gov/Pubs/contents.html>.
- [5] DerSimonian, R.; Laird, N.; *Controlled Clinical Trials* 7, 177-188 (1986).

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[®] 2710a

Montana I Soil

Highly Elevated 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 2710a 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. Six laboratories participated, five 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. All 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 (QATS), 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	6	8200	- 12000	10000	17
Antimony	6	5.0	- 12	9.6	18
Arsenic	6	1300	- 1600	1400	92
Barium	6	490	- 540	510	65
Beryllium	6	0.24	- 0.51	0.48	--
Cadmium	5	9.6	- 12	11	86
Calcium	6	1700	- 2000	1800	19
Chromium	6	9.2	- 11	10	41
Cobalt	6	2.8	- 5.2	3.8	64
Copper	6	3100	- 3500	3300	95
Iron	6	30000	- 36000	34000	79
Lead	6	4700	- 5800	5100	93
Magnesium	6	3200	- 3600	3500	48
Manganese	6	1500	- 1800	1700	77
Mercury	6	9.3	- 11.7	10	104
Nickel	5	4.8	- 6.1	5.5	69
Potassium	6	3800	- 4700	4100	19
Selenium	2	1.5	- 2.6	2.0	200
Silver	6	31	- 39	36	91
Sodium	6	550	- 650	590	7
Thallium	3	1.3	- 3.6	3.2	213
Vanadium	6	35	- 43	38	48
Zinc	6	3300	- 4400	3800	90

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
SVL Analytical, Inc.