National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1635a

Trace Elements in Coal

(Subbituminous)

This Standard Reference Material (SRM) is intended primarily for the evaluation of techniques used in the analysis of coals and materials of a similar matrix. A unit of SRM 1635a consists of 50 g of subbituminous coal that was ground to pass a 250 µm (60 mesh) sieve, homogenized, bottled under an argon atmosphere, and sealed in an aluminized bag.

Certified Mass Fraction Values: The certified mass fraction [1] values for 19 elements, expressed on a dry-mass basis, are provided in Table 1. 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]. A certified value is the present best estimate of the true value.

Reference Mass Fraction Values: The reference mass fraction values for 17 elements, expressed on a dry-mass basis, are provided in Table 2. A reference value is a non-certified value that is the best estimate of the true value; however, the value does not meet NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision and may not include all sources of uncertainty [2].

Information Mass Fraction Value: An information mass fraction value for one element is provided in Table 3. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 1635a** is valid, within the measurement uncertainty specified, until **31 July 2036**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for 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 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.

Technical measurements leading to certification of SRM 1635a were coordinated by J.L. Mann and T.W. Vetter of the NIST Chemical Sciences Division. Analytical measurements leading to certification were made by R.G. Brennan, S.J. Christopher, L. Francini, W.R. Kelly, S.E. Long, J.L. Mann, J.L. Molloy, K.E. Murphy, R. Oflaz, R.L. Paul, S.A. Rabb, J.R. Sieber, R.L. Temple, B.E. Tomlin, L.L. Yu, and R.L. Zeisler of the NIST Chemical Sciences Division.

Statistical analyses were performed by S.D. Leigh and A.L. Pintar 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

Gaithersburg, MD 20899
Certificate Issue Date: 06 December 2017
Certificate Revision History on Page 6

Steven J. Choquette, Director
Office of Reference Materials

INSTRUCTIONS FOR STORAGE AND USE

Storage: The SRM should be stored in its original, tightly sealed bottle away from sunlight, heat, and moisture.

Sampling: The SRM should be thoroughly mixed by carefully inverting and rotating the tightly-sealed bottle before it is sampled. A minimum test portion of 250 mg should be used for analytical determinations to be related to the elemental certified mass fraction values and expanded uncertainties provided. See "Homogeneity Assessment" for information on smaller test portion masses that may be acceptable for certain elements.

Drying: To relate their measurements directly to the certified and reference values, which are expressed on a dry-mass basis, users should determine a drying correction at the time of each analysis. The correction should be determined using oven drying with separate 1 g test portions in a nitrogen atmosphere at 107 °C ± 3 °C to a constant mass [3] or an equivalent technique. Caution: drying of large samples may result in a violent discharge of water vapor and resultant loss of sample.

The dry-mass basis was determined at NIST using a thermogravimetric (TG) method [4], in which drying in a nitrogen atmosphere and an air atmosphere were compared. Using the TG method, the mass loss of SRM 1635a samples ($n = 8$) stabilized after approximately 150 minutes in nitrogen and approximately 120 minutes in air. The average mass loss measured using the TG method, which is reported ***for information purposes only***, was 20.05 % ($1s = 0.54$ %, $n = 16$) in nitrogen and 20.02 % ($1s = 0.54$ %, $n = 16$) in air. The mass loss determined by the user may be significantly different, depending on ambient conditions when the bottle is sampled. A comparison of values calculated on a dry-mass basis using the ASTM method [3] to those listed in this certificate, calculated by the TG method [4], indicated that the calculated mass loss values, within the uncertainty of the measurement, were essentially the same (values agreed within about 0.1 %, relative).

SOURCE, PREPARATION, AND HOMOGENEITY ASSESSMENT[1]

Source and Preparation of Material: Approximately 545 kg of crushed coal was obtained from Peabody Energy's North Antelope Rochelle Mine (Campbell County, WY). This mine produces subbituminous coal from the Wyodak-Anderson seam of the Powder River Basin. The bulk material was shipped to the U.S. Geological Survey (USGS, Denver, CO), where the coal was pulverized to pass a 250 µm (60 mesh) sieve. Then the entire lot was divided using a spinning riffler into multiple sublots contained in plastic buckets with lids. About one quarter of the sublots were divided further into amber glass bottles using the spinning riffler technique. After the material was shipped to NIST, the bottles were opened, flushed with argon in a glove box, and re-capped. The bottles were removed from the glove box and sealed in aluminized bags.

Homogeneity Assessment: Homogeneity was assessed for selected elements based on wavelength-dispersive X-ray fluorescence spectrometry (WDXRF) analysis of duplicate test portions from 20 bottles selected by stratified random sampling from the entire lot of SRM 1635a and, in some cases, by the analytical technique used for an element. For most elements, one-way analyses of variance failed to reject the null hypothesis of no bottle effect, which is consistent with material homogeneity ($P \geq 0.12$).

For three elements, one-way analyses of variance rejected the null hypothesis of no bottle effect, which is indicative of material heterogeneity (for nickel: $P = 0.006$; for potassium: $P = 0.0000001$; for sodium: $P = 0.00001$). In addition, heterogeneity was accounted for in the determination of lead. The expanded uncertainties, U, of the certified values for lead, nickel, and sodium and the reference value for potassium include a component to account for material heterogeneity. Heterogeneity is a major component of the U for lead, nickel, and potassium.

A further analysis of selected elements in 15 bottles of this material by microbeam X-ray fluorescence (XRF) indicates that a minimum test portion mass of 2 mg can be used for the following 11 elements: aluminum, barium, calcium, copper, iron, magnesium, potassium, sulfur, strontium, titanium, and zinc [5].

VALUE ASSIGNMENT

The analytical techniques used for measurement of each element are listed in Table 4. WDXRF was used for homogeneity assessment, but not value assignment.

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[1]Certain commercial equipment, instruments, or materials are identified in this certificate to adequately specify 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.

Metrological Traceability: The measurands for the certified values in Table 1 are the total mass fractions for the elements. The measurands for the reference values in Table 2 are the mass fractions for the elements as determined by the indicated methods. Metrological traceability is to the SI derived unit for mass fraction, expressed as milligrams per kilogram or as a percentage [1].

Certified Mass Fraction Values: Each certified value is given with an expanded uncertainty, $U = ku_c$, calculated according to the method in the ISO/JCGM Guide [6]. The quantity, k, is the coverage factor used to obtain an expanded uncertainty with an approximate 95 % coverage level. The certified values and their corresponding expanded uncertainties are given in Table 1.

The certified mass fraction values for mercury and lead are each based on results from a single NIST primary method for which a complete evaluation of all sources of uncertainty has been performed. For mercury, the value is an un-weighted mean with the coverage factor, $k = 2.16$, determined from the Student's t-distribution with 27 degrees of freedom and 95 % confidence. For lead, heterogeneity was an additional source of uncertainty. The certified value and its associated expanded uncertainty were based on a random effects model for the experimental data that also included the components of uncertainty assessed by Type B methods [6–9]. The certified value and the combined standard uncertainty are the mean and the standard deviation of the appropriate Bayesian posterior distribution [10]. The expanded uncertainty is the half width of a symmetric 95 % credible interval. The effective coverage factor is $k = 2.13$.

The certified value and expanded uncertainty for sulfur are calculated (with the coverage factor, $k = 2$) by combining two sets of results using the approach in reference 11, the first from sample decomposition by microwave-induced combustion with measurements by isotope dilution sector field inductively coupled plasma mass spectrometry (ID-SF-ICP-MS), and the second from a CANSPEX interlaboratory study, which is described in the "Supplemental Information" section. Before combining the ID-SF-ICP-MS and CANSPEX measurements, the CANSPEX measurements were reduced to a Bayesian posterior distribution for the consensus value using a random effects model.

Except as noted for mercury, lead, and sulfur, the certified values are weighted means [12,13] of the results from two critically-evaluated independent analytical methods and the standard uncertainty, u_c, represents, at the level of one standard deviation, a combined uncertainty that results from coupling the measurements and their associated uncertainties. The expanded uncertainties are approximately the half-widths of a symmetric 95 % parametric bootstrap confidence interval [14], and they are consistent with the ISO/JCGM Guide [6]. For these expanded uncertainties, $k = 2$, except for Rb, where $k = 2.26$. The expanded uncertainties for nickel and sodium also include a component to account for material heterogeneity.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for SRM 1635a

Minor Constituents

Element	Mass Fraction (%)			Element	Mass Fraction (%)		
Barium (Ba)	0.03578	±	0.00091	Sulfur (S)	0.2909	±	0.0080
Iron (Fe)	0.2472	±	0.0022	Strontium (Sr)	0.0160	±	0.0011
Sodium (Na)	0.1031	±	0.0078				

Trace Elements

Element	Mass Fraction (mg/kg)			Element	Mass Fraction (mg/kg)		
Antimony (Sb)	0.251	±	0.036	Mercury (Hg)	0.0836	±	0.0064
Arsenic (As)	0.860	±	0.019	Nickel (Ni)	5.37	±	0.30
Chromium (Cr)	3.56	±	0.18	Rubidium (Rb)	1.226	±	0.043
Cobalt (Co)	2.004	±	0.044	Selenium (Se)	0.662	±	0.096
Copper (Cu)	11.42	±	0.74	Thorium (Th)	1.299	±	0.059
Lead (Pb)	2.85	±	0.51	Uranium (U)	0.4792	±	0.0090
Manganese (Mn)	6.69	±	0.14	Vanadium (V)	13.34	±	0.59

Reference Mass Fraction Values: Reference mass fraction values are based on results from a single NIST analytical method for all elements except zinc, for which the value is a weighted mean [12, 13] and the expanded uncertainty is approximately the half-width of a symmetric 95 % parametric bootstrap confidence interval [14]. Each reference value is expressed with an expanded uncertainty, $U = ku_c$, calculated according to the method in the ISO/JCGM Guide [6]. The coverage factor, k, for each element is listed in Table 2. The expanded uncertainties for chlorine and potassium also include a component to account for material heterogeneity.

Table 2. Reference Mass Fraction Values (Dry-Mass Basis) for SRM 1635a

Major Constituents

Element	Mass Fraction (%)			k	Element	Mass Fraction (%)			k
Aluminum (Al)	0.5437	±	0.0063	2	Magnesium (Mg)	0.2303	±	0.0031	2
Calcium (Ca)	1.087	±	0.014	2	Potassium (K)	0.01874	±	0.00079	2
Hydrogen (H)	3.92	±	0.27	2	Titanium (Ti)	0.05240	±	0.00090	2.05

Trace Elements

Element	Mass Fraction (mg/kg)			k	Element	Mass Fraction (mg/kg)			k
Boron (B)	36.0	±	1.5	2	Hafnium (Hf)	3.14	±	0.23	2.11
Cadmium (Cd)	0.282	±	0.020	2.36	Molybdenum (Mo)	6.36	±	0.97	2.36
Cerium (Ce)	5.45	±	0.10	2.09	Samarium (Sm)	0.483	±	0.017	2.36
Cesium (Cs)	0.0998	±	0.0041	2	Scandium (Sc)	1.240	±	0.017	2
Chlorine (Cl)	15.4	±	3.0	2	Zinc (Zn)	7.3	±	1.5	2
Europium (Eu)	0.1115	±	0.0021	2.18					

Information Mass Fraction Value: The information mass fraction value in Table 3, given as additional information on the matrix, is provided without an uncertainty estimate, because insufficient information is available to assess the uncertainty.

Table 3. Information Mass Fraction Value (Dry-Mass Basis) for SRM 1635a

Element	Mass Fraction (mg/kg)
Bromine (Br)	1

Table 4. Analytical Methods Used at NIST

Element	Method	Element	Method
Aluminum	INAA, WDXRF	Magnesium	INAA, WDXRF
Antimony	ICP-MS, INAA	Manganese	ICP-OES, INAA, WDXRF
Arsenic	ICP-MS, INAA	Mercury	ID-CV-ICP-MS
Barium	ICP-OES, INAA, WDXRF	Molybdenum	ICP-MS
Boron	PGAA	Nickel	ICP-MS, ICP-OES, WDXRF
Bromine	INAA	Potassium	ICP-OES, WDXRF
Cadmium	ICP-MS	Rubidium	ICP-MS, INAA
Calcium	INAA, WDXRF	Samarium	INAA
Cerium	INAA	Scandium	INAA
Cesium	INAA	Selenium	ICP-MS, INAA
Chlorine	INAA	Sodium	ICP-OES, INAA, WDXRF
Chromium	ICP-MS, INAA, WDXRF	Strontium	ICP-OES, INAA, WDXRF
Cobalt	ICP-MS, INAA, WDXRF	Sulfur	ID-SF-ICP MS, ILS
Copper	ICP-MS, ICP-OES, WDXRF	Thorium	ICP-MS, INAA
Europium	INAA	Titanium	INAA, WDXRF
Hafnium	INAA	Uranium	ICP-MS, INAA
Hydrogen	PGAA	Vanadium	ICP-OES, INAA
Iron	ICP-OES, INAA, WDXRF	Zinc	ICP-MS, INAA, WDXRF
Lead	ID-ICP-MS		

Methods:

ICP-MS	Inductively coupled plasma mass spectrometry
ICP-OES	Inductively coupled plasma optical emission spectrometry
ID-CV-ICP-MS	Isotope dilution cold vapor inductively coupled plasma mass spectrometry
ID-ICP-MS	Isotope dilution inductively coupled plasma mass spectrometry
ID-SF-ICP MS	Isotope dilution sector field inductively coupled plasma mass spectrometry
ILS	Interlaboratory Study
INAA	Instrumental neutron activation analysis
PGAA	Prompt-gamma activation analysis
WDXRF	Wavelength-dispersive X-ray fluorescence (used for homogeneity assessment only)

SUPPLEMENTAL INFORMATION

Summary statistics reported by Quality Associates International, Ltd. (Sechelt, BC, Canada) for the Coal and Ash Sample Proficiency Exchange (CANSPEX) 2008-3 interlaboratory study using SRM 1635a as an unknown coal sample are provided in the Appendix (Tables A1 and A2) to this certificate to demonstrate user experience with this material using conventional methods and to better characterize the matrix. The CANSPEX interlaboratory study results were not used in calculating any assigned values and should **NOT** be used as substitutes for certified or reference values.

REFERENCES

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Certificate Revision History: 06 December 2017 (Added certified value for sulfur and reference value for chlorine; editorial changes); 19 July 2012 (Corrected units from mg/kg for potassium; editorial changes); 29 June 2012 (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 http://www.nist.gov/srm.

Test portions of SRM 1635a were analyzed as unknown samples in the interlaboratory study CANSPEX 2008–3, conducted by Quality Associates International, Ltd. Values are expressed on a dry-mass basis for all parameters except moisture, which is expressed on an "as received" basis. The tables are included as shown in the summary report by Quality Associates International, Ltd. Table A1 shows the summary results and Table A2 shows the derived standard deviations and a tally of published methods used in the study. The values have not been altered. Table A2 was formatted to fit on the page and minor editorial corrections for text and websites were completed. These results are included to demonstrate user experience with this material using conventional methods and to better characterize the matrix. Results from this study should **NOT** be used as substitutes for certified or reference values.

Table A1. SRM 1635a CANSPEX Interlaboratory Study Results

SRM 1635x CANSPEX™ Interlaboratory Study Results					
Parameter	Most Likely Value	95 % Coverage Interval of Mean value	Pooled Within Lab Standard Deviation (s_w)	Pooled Between Lab Standard Deviation (s_B)	Total Number of Labs
Moisture %	20.54	0.15	0.10	0.59	118
Ash % dry basis	6.29	0.045	0.063	0.184	119
Volatile % dry basis	44.75	0.46	0.30	1.59	92
Btu/lb dry basis	11664	19	25	74	110
Carbon % dry basis	68.97	0.34	0.25	0.89	52
Hydrogen % dry basis	4.642	0.135	0.058	0.320	47
Nitrogen % dry basis	0.946	0.051	0.029	0.127	48
Total Sulfur % dry basis	0.294	0.006	0.008	0.022	112
Pyritic Sulfur % dry basis	0.020	0.005	0.002	0.007	16
Sulfate Sulfur % dry basis	0.009	0.005	0.001	0.004	13
Chlorine µg/g dry basis	51	19	8	41	41
Fluorine µg/g dry basis	63	11	5	22	31
Mercury ng/g dry basis	76	9	4	16	28
Selenium µg/g dry basis	0.94	0.79	0.12	0.78	12

Table A2. CANSPEX Supplied Data

Derived Standard Deviations (in %) of Repeatability (s_r) and Reproducibility (s_R), and Tally of Published Methods Used in CANSPEX™ Interlaboratory Study*

Parameter	Total No. of Labs	AS	s_r	s_R	No.	ASTM	s_r	s_R	No.	BSI	s_r	s_R	No.	DIN	s_r	s_R	No.	GB	s_r	s_R	No.	ISO	s_r	s_R	No.	NF	s_r	s_R	No.	SABS	s_r	s_R	No.	In-house No.
Moisture (%)	118	1038.3	0.04	-	1	D2013	0.10	0.23	1	1016	0.04	-	1	51718	0.07	-	2	212	0.07	-	1	589	0.11	-	2	3-037	-	-	1	925	-	-	1	8
						D3173	0.10	0.23	55													11722	0.04	-	6									
						D3302	0.10	0.23	9													5068	0.07	-	1									
						D5142	0.16	0.33	29																									
Ash (%) dry basis	119	1038.3	0.04	0.05	1	D3174	0.08	0.11	65	1016	0.05	0.11	1	51719	0.07	0.11	2	212	0.07	0.11	1	1171	0.07	0.11	10	3-003	-	-	1		-	-		7
						D5142	0.07	0.10	31																									
Volatile (%) dry basis	92	1038.3	0.07	0.35	1	D3175	0.18	0.35	44	1016	0.11	0.35	1	51720	0.48	0.63	2	212	0.18	0.35	1	562	0.48	0.63	10									9
						D5142	0.33	0.97	24																									
Btu/lb dry basis	110	1038.5	20	46	1	D1989	23	39	5	1016	18	43	1	51900	18	46	4	213	18	46	1	1928	43	106	12									5
						D2015	24	38	3																									
						D3286	18	35	4																									
						D5865	24	38	74																									
Carbon (%) dry basis	52	1038.6.4	0.11	0.21	1	D3178	0.11	-	3					51732	-	-	1	476	0.18	0.35	1	609	0.09	0.18	3									6
						D5373	0.16	0.35	35													12902	-	-	2									
Hydrogen (%) dry basis	47	1038.6.4	0.04	0.07	1	D3178	0.02	-	4					51732	-	-	1	476	0.05	0.09	1	609	0.04	0.09	3									3
						D5373	0.04	0.09	32													12902	-	-	2									
Nitrogen (%) dry basis	48	1038.6.4	0.01	0.03	1	D3179	0.02	0.05	4					51732	-	-	1	476	0.03	0.05	1	333	0.02	0.04	3									4
						D5373	0.02	0.05	32													12902	-	-	2									
Total Sulfur (%) dry basis	112	1038.6.3.3	0.01	0.02	1	D3177	0.02	0.04	4	1016	0.02	0.04	1	51724-3	0.01	0.02	0	214	0.04	0.09	1	351	0.02	0.04	3	3-038	-	-	1		-	-		10
						D4239	0.01	0.02	88																									
						D5016	0.03	0.09	3																									
Pyritic Sulfur (%) dry basis	16	1038.11	0.02	0.05	1	D2492	0.03	0.06	13									215	0.02	0.04	1													1
Sulfate Sulfur (%) dry basis	13	1038.11	0.007	0.011	1	D2492	0.007	0.014	12									215	0.01	0.04	0													0
Chlorine (µg/g) dry basis	41		-	-		D2361	106	213	2	1016	177	177	0	51727	71	106	2	3558	35	71	1					3-009	-	-	1		-	-		10
						D4208	19	75	19																									
						D6721	2	3	6																									
Fluorine (µg/g) dry basis	31					D3761	5	5	14					51723	8	14	2	4663	6	7	1	11724	4	7	2	03-009	-	-	0		-	-		9
						D5987	4	7	3																									
Mercury (ng/g) dry basis	28					D6414	7	8	4					22022	-	-	0																	5
						D6722	3	6	19																									
Selenium (µg/g) dry basis	12					D4606	0.176	0.14	3																									9

* The above precision standard deviations are derived from the division of each method's published precision values by an estimate of the coverage factor used.

** Method is designated "In-house" if lab reports method as In-house; lab reports methods as modified; or does not report a method. CANSPEX does not provide repeatability or reproducibility information for In-house methods.

"-" indicates documentation confirming the repeatability or reproducibility is not available.

The above referenced methods are available through the following websites:

AS http://www.standards.org.au
ASTM http://www.astm.org/
BSI http://www.bsigroup.com/
DIN http://www.din.de/cmd?level=tpl-home&languageid=en
GB http://www.standardsportal.org.cn/zmen/English/Resources/
ISO http://www.iso.org/iso/iso_catalogue.htm
NF http://www2.afnor.org/portail.asp?Lang=English
SABS https://www.sabs.co.za/