National Institute of Standards and Technology
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Standard Reference Material® 1635a

Trace Elements in Coal

(Subbituminous)

CERTIFICATE OF ANALYSIS

Purpose: 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.

Description: 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 Values: A NIST certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias and imprecision have been considered and any contributions they may make to measurement uncertainty have been quantified and are expressed in the reported uncertainty [1]. Certified mass fraction values for elements in SRM 1635a, reported on a dry-mass basis, are provided in Table 1. The measurands in Table 1 are total mass fractions for each analyte reported and metrological traceability is to the International System of Units (SI) derived unit for chemical mass fraction expressed as either milligrams per kilogram or as a percentage [2].

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

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 ± 0.37	Uranium (U)	0.4792 ± 0.0090
Manganese (Mn)	6.69 ± 0.14	Vanadium (V)	13.34 ± 0.59

[(a)] Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty associated with the half width of a symmetric 95 % coverage interval for the mean of all bottles of SRM 1635a because the underlying mass fraction is assumed to be the same for each bottle, except for lead, nickel, and sodium. For lead nickel, and sodium, the quantity $U_{95\%}(x)$ is associated with the mean of a single randomly chosen bottle of SRM 1635a. To propagate the uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/k$ [3–4]. The coverage factor, k, = 2, except that for Hg, k = 2.16; for Pb, k = 1.83; and for Rb, k = 2.26.

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Certificate Revision History on Page 3

Additional Information: Non-certified values and a table of analytical methods are provided in Appendix A. A value for Br and summary statistics reported in an interlaboratory study using SRM 1635a as an unknown coal sample are provided in Appendix B. Source and preparation can be found in Appendix C.

Period of Validity: The certified values delivered by **SRM 1635a** are valid within the measurement uncertainty specified until **31 July 2036**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Safety: SRM 1635a is intended for research use. Please consult the Safety Data Sheet for this product.

Storage: The original unopened bottles of SRM 1635a should be stored tightly sealed and away from sunlight and intense sources of radiation at room temperature (20 °C ± 5 °C). An open bottle can be reused until the material reaches its expiration date, provided that the open bottle is resealed and stored at room temperature (20 °C ± 5 °C).

Use: Before it is sampled, the unit should be thoroughly mixed by carefully inverting and rotating the tightly sealed bottle. 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 Instructions: 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 [5] 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 [6], 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 [5] to those listed in this certificate, calculated by the TG method [6], indicated that the calculated mass loss values, within the uncertainty of the measurement, were essentially the same (values agreed within about 0.1 %, relative).

Certified value assignment for mercury, lead, and sulfur: The certified mass fraction values for mercury and lead are each based on results from a single NIST primary method (see Table A2) for which a complete evaluation of all sources of uncertainty has been performed. The certified value and expanded uncertainty for sulfur are calculated by combining two sets of results using the approach in reference 7, 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 Appendix B Tables B2 and B3.

Certified value assignment for all other elements: Except as noted for mercury, lead, and sulfur, the certified values are weighted means [8,9] 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 [10], and they are consistent with the ISO/JCGM Guide [4]. The expanded uncertainties for nickel and sodium also include a component to account for material heterogeneity.

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[2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/special-publication-811 (accessed Sep 2023).

[3] JCGM 101:2008; *Evaluation of Measurement Data — Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement" — Propagation of Distributions Using a Monte Carlo Method*; JCGM (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Sep 2023).

[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 (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Sep 2023); 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 Sep 2023).

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Certificate Revision History: 06 September 2023 (Revised values for lead in Table 1 and cadmium in Table A1; updated format; editorial changes); 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).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

* * * * * * * End of Certificate of Analysis* * * * * * *

APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the SI or other higher-order reference system [1]. The mass fraction values listed are based on results from a single NIST analytical method for all elements except zinc, for which the value is a weighted mean [8,9].

Table A1. Non-Certified Mass Fraction Values (Dry-Mass Basis) for SRM 1635a[(a)]

Major Constituents

Element	Mass Fraction (%)	k
Aluminum (Al)	0.5437 ± 0.0063	2
Calcium (Ca)	1.087 ± 0.014	2
Hydrogen (H)	3.92 ± 0.27	2

Minor Constituents

Element	Mass Fraction (%)	k
Magnesium (Mg)	0.2303 ± 0.0031	2
Potassium (K)	0.01874 ± 0.00079	2
Titanium (Ti)	0.05240 ± 0.00090	2.05

Trace Elements

Element	Mass Fraction (mg/kg)	k
Boron (B)	36.0 ± 1.5	2
Cadmium (Cd)	0.062 ± 0.020	1.95
Cerium (Ce)	5.45 ± 0.10	2.09
Cesium (Cs)	0.0998 ± 0.0041	2
Chlorine (Cl)	15.4 ± 3.0	2
Europium (Eu)	0.1115 ± 0.0021	2.18
Hafnium (Hf)	3.14 ± 0.23	2.11
Molybdenum (Mo)	6.36 ± 0.97	2.36
Samarium (Sm)	0.483 ± 0.017	2.36
Scandium (Sc)	1.240 ± 0.017	2
Zinc (Zn)	7.3 ± 1.5	2

[(a)] Values are expressed as $x \pm U_{95\%}(x)$, where x is the certified value and $U_{95\%}(x)$ is the expanded uncertainty associated is the expanded uncertainty associated with the half width of a symmetric 95 % coverage interval for the mean of all bottles of SRM 1635a because the underlying mass fraction is assumed to be the same for each bottle, except for cadmium, chlorine, and potassium. For cadmium, chlorine, and potassium, the quantity $U_{95\%}(x)$ is associated with the mean of a single randomly chosen bottle of SRM 1635a. To propagate the uncertainty, treat the certified value as a normally distributed random variable with mean x and standard deviation $U_{95\%}(x)/k$ [3–4]. The coverage factor, k, for each element is listed. The expanded uncertainties for cadmium, chlorine, and potassium also include a component to account for material heterogeneity.

Period of Validity: The non-certified values are valid within the measurement uncertainty specified until **31 July 2036**. The value assignments are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Appendix and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Table A2. Analytical Methods Used

Element	Method	Element	Method
Aluminum (Al)	INAA, WDXRF	Magnesium (Mg)	INAA, WDXRF
Antimony (Sb)	ICP-MS, INAA	Manganese (Mn)	ICP-OES, INAA, WDXRF
Arsenic (As)	ICP-MS, INAA	Mercury (Hg)	ID-CV-ICP-MS
Barium (Ba)	ICP-OES, INAA, WDXRF	Molybdenum (Mo)	ICP-MS
Boron (B)	PGAA	Nickel (Ni)	ICP-MS, ICP-OES, WDXRF
Bromine (Br)	INAA	Potassium (K)	ICP-OES, WDXRF
Cadmium (Cd)	ICP-MS	Rubidium (Rb)	ICP-MS, INAA
Calcium (Ca)	INAA, WDXRF	Samarium (Sm)	INAA
Cerium (Ce)	INAA	Scandium (Sc)	INAA
Cesium (Cs)	INAA	Selenium (Se)	ICP-MS, INAA
Chlorine (Cl)	INAA	Sodium (Na)	ICP-OES, INAA, WDXRF
Chromium (Cr)	ICP-MS, INAA, WDXRF	Strontium (Sr)	ICP-OES, INAA, WDXRF
Cobalt (Co)	ICP-MS, INAA, WDXRF	Sulfur (S)	ID-SF-ICP-MS, ILS
Copper (Cu)	ICP-MS, ICP-OES, WDXRF	Thorium (Th)	ICP-MS, INAA
Europium (Eu)	INAA	Titanium (Ti)	INAA, WDXRF
Hafnium (Hf)	INAA	Uranium (U)	ICP-MS, INAA
Hydrogen (H)	PGAA	Vanadium (V)	ICP-OES, INAA
Iron (Fe)	ICP-OES, INAA, WDXRF	Zinc (Zn)	ICP-MS, INAA, WDXRF
Lead (Pb)	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 (CANSPEX)
INAA	Instrumental neutron activation analysis
PGAA	Prompt gamma-ray activation analysis
WDXRF	Wavelength dispersive X-ray fluorescence (used for homogeneity assessment only)

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APPENDIX B

Mass Fraction Value of Interest: The mass fraction value in Table B1, given as additional information on the matrix, is provided without an uncertainty estimate, because insufficient information is available to assess the uncertainty.

Table B1. Mass Fraction Value of Interest (Dry Mass Basis) for SRM 1635a

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

Interlaboratory Study Summary Statistics: 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 B2 shows the summary results and Table B3 shows the derived standard deviations and a tally of published methods used in the study. The values have not been altered. Table B3 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 B2. SRM 1635a 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 B3. CANSPEX Supplied Data

Parameter	Total Number of Labs	Derived Standard Deviations (in %) of Repeatability (s_r) and Reproducibility (s_R), and Tally of Published Methods Used in CANSPEX™ Interlaboratory Study*																														
		Standards Australia				ASTM International				British Standards Institution				Deutsches Institut für Normung				China National Standards				International Organization for Standardization				Association Francaise de Normalisation				South African Bureau of Standards				In-house**
		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.	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 https://www.standards.org.au/ (accessed Sep 2023)
ASTM https://www.astm.org/ (accessed Sep 2023)
BSI https://www.bsigroup.com/ (accessed Sep 2023)
DIN https://www.din.de/en (accessed Sep 2023)
GB https://www.gbstandards.org/index/ (accessed Sep 2023)
ISO https://www.iso.org/home.html (accessed Sep 2023)
NF https://www.afnor.org/en/ (accessed Sep 2023)
SABS https://www.sabs.co.za/ (accessed Sep 2023)

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APPENDIX C

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 cadmium and lead. The expanded uncertainties, U, of the certified values for lead, nickel, and sodium include a component to account for material heterogeneity.

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 [11].

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