NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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Standard Reference Material® 2693a

Bituminous Coal

(Nominal Mass Fraction 0.5 % Sulfur)

CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended primarily for use in the evaluation of techniques used in the analysis of coals and materials of a similar matrix.

Description: A unit of SRM 2693a consists of 50 g of bituminous coal ground to pass a 212 µm (70 mesh) sieve, homogenized, and packaged in an amber glass bottle under argon and then sealed in an aluminized bag.

Certified Values: A 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]. The certified mass fraction values are reported on a dry-mass basis. The certified values in Tables 1 and 2 are based on analyses by two or more independent methods as listed in Table B1. The measurands in Tables 1 and 2 are total mass fractions for each constituent listed and metrological traceability is to the International System of Units (SI) derived unit for mass fraction expressed as a percentage [2].

Table 1. Certified Mass Fraction Values for Sulfur in SRM 2693a

Element	Mass Fraction[(a)] (%)	Coverage Factor, k
Sulfur (S)	0.3334 ± 0.0078	2.19

[(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 2693a because the underlying mass fraction is assumed to be the same for each bottle. The value of $U_{95\%}$calculated as ku_c, where the quantity u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guides [3–4].

Table 2. Certified Mass Fraction Values for Hydrogen and Nitrogen in SRM 2693a

Element	Mass Fraction (%)	Uncertainty Interval[(a)] (%)	Standard Uncertainty, u_c (%)
Hydrogen (H)	4.39	4.33 to 4.54	0.056
Nitrogen (N)	1.179	1.155 to 1.193	0.010

[(a)] The use of the asymmetric intervals about the assigned values depends on the context. Instructions for two common uses, (1) direct comparison of the interval to a result of a user determination and (2) propagation of uncertainty when using SRM 2693a as a calibration standard using the value of u_c, are provided in more detail in NIST SP 260-230 [5].

Non-Certified Values: Non-certified values are provided in Appendix A.

Additional Information: Additional information is provided in Appendix B and Appendix C.

Period of Validity: The certified values delivered by **SRM 2693a** are valid within the measurement uncertainty specified until **01 September 2036**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

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 2693a is intended for research use. Please consult the Safety Data Sheet for this product.

Storage: The original unopened bottles of SRM 2693a should be stored tightly sealed and away from sunlight and intense sources of radiation at room temperature (20 °C ± 5 °C). Although a tightly-sealed bottle should prevent absorption of moisture, storage at relative humidities above 60 % are not recommended. An opened bottle can be reused until the material reaches its expiration date, provided that the opened 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 size of 25 mg for carbon, hydrogen, and nitrogen, 50 mg for sulfur, 100 mg for mercury, and 250 mg for aluminum, bromine, calcium, chlorine, dysprosium, manganese, sodium, and vanadium should be used for analytical determinations.

Drying Instructions: To relate measurements directly to the certified and non-certified values, which are expressed on a dry-mass basis, users must determine a drying correction at the time of the analysis. The correction is determined by oven-drying a separate 1 g sample in an air, argon, or nitrogen atmosphere at 107 °C ± 3 °C to a constant mass [6]. Attainment of constant mass is defined according to the ASTM thermogravimetric (TG) method as either a mass loss of ≤ 0.05 %, relative, over a nine-minute period or the mass loss after one hour of heating [6]. At NIST, the mass losses determined in both manners, and in both nitrogen and air, were similar.

The mass loss determined in both a nitrogen and air atmosphere, which is reported ***for information purposes only***, was nominally 1 %. The mass loss determined by the user may be different, depending on ambient conditions when the bottle is sampled.

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Apr 2024).

[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 Apr 2024).

[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 Apr 2024).

[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 Apr 2024); 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 Apr 2024).

[5] Vetter, T.W.; Bryan Sallee, C.E.; Lang, B.; Marlow, A.F.; Ness, J.M.; Paul, R.L.; Pintar, A.L.; Scruggs, B.E.; Sharp, N., Sieber, J.R.; Vega, M.; *Certification of Standard Reference Material® 2693a Bituminous Coal (Nominal Mass Fraction 0.5 % Sulfur)*; NIST Special Publication (NIST SP) 260-230; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-230.pdf (accessed Apr 2024).

[6] ASTM D7582-15; *Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis*; Annu. Book ASTM Stand., Vol 05.06, pp. 965–973 (2021).

If you use this SRM in published work, please reference:

Vetter TW, Bryan Sallee CE, Lang BE, Marlow AF, Ness JM, Paul RL, Pintar AL, Scruggs BE, Sharp N, Sieber JR, Vega M (2023) Certification of Standard Reference Material® 2693a Bituminous Coal (Nominal Mass Fraction 0.5 % Sulfur). (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-230. https://doi.org/10.6028/NIST.SP.260-230

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. They are best estimates based on currently available information; however, they do not meet NIST's criteria for certification [1]. Non-certified mass fraction values are provided in Tables A1 and A2.

Table A1. Non-Certified Mass Fraction Values with Symmetric Uncertainty Intervals in SRM 2693a

Element	Mass Fraction[(a)]	Units	Coverage Factor, k
Aluminum (Al)	15 080 ± 110	mg/kg	2
Bromine (Br)	0.418 ± 0.016	mg/kg	2
Calcium (Ca)	981 ± 45	mg/kg	2
Chlorine (Cl)	59.7 ± 1.8	mg/kg	2.08
Dysprosium (Dy)	1.550 ± 0.072	mg/kg	2
Mercury (Hg)	56.3 ± 6.2	µg/kg	2.22
Manganese (Mn)	24.3 ± 2.4	mg/kg	2.18
Sodium (Na)	34.4 ± 2.1	mg/kg	2.11
Vanadium (V)	52.22 ± 0.41	mg/kg	1.85

[(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 2693a because the underlying mass fraction is assumed to be the same for each bottle. The value of $U_{95\%}$calculated as ku_c, where the quantity u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guides [3–4].

Table A2. Non-Certified Mass Fraction Values with Non-symmetric Uncertainty Intervals in SRM 2693a

Element	Mass Fraction (%)	Uncertainty Interval[(a)] (%)	Standard Uncertainty, u_c (%)
Carbon (C)	79.86	79.76 to 80.22	0.13

[(a)] The use of the asymmetric intervals about the assigned values depends on the context. Instructions for two common uses, (1) direct comparison of the interval to a result of a user determination and (2) propagation of uncertainty when using SRM 2693a as a calibration standard using the value of uc, are provided in more detail in NIST SP 260-230 [5].

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).

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APPENDIX B

Values of Potential Interest to Users: Particle size measurements were made using a laser-based light scattering system. Approximately 0.5 g of material (refractive index: 1.746, absorption index: 1.0) was measured using water as the dispersant, (refractive index: 1.33) and 0.01 % volume fraction Triton X-100 as a pre-wetting surfactant. Calculated 10th percentile ($d_{0.1}$), 50th percentile ($d_{0.5}$), and 90th percentile ($d_{0.9}$) particle sizes (volume fraction, in percent, of particles smaller than the value) are $d_{0.1}$ = 8.72 µm, $d_{0.5}$ = 51.4 µm, and $d_{0.9}$ = 147 µm. The fraction of material smaller than 9 µm in diameter is 10 %. The particle size distribution is shown in Figure B1.

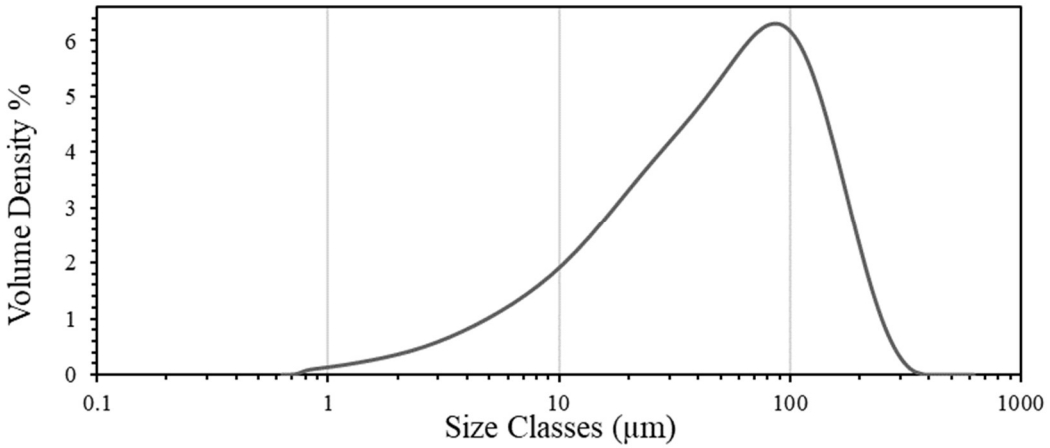

Figure B1. Average Volume Particle Size Distribution for SRM 2693a as Measured by a Laser-based Light Scattering System.

Table B1. Methods of Analysis

Method	Element
Atomic absorption spectrometry (AAS) using direct combustion	Hg
CANSPEX interlaboratory study	C, H, Hg, N, S
Combustion with thermal conductivity detection using an elemental analyzer	C, H, N
Instrumental neutron activation analysis (INAA)	Al, Br, Ca, Cl, Dy, Mn, Na, V
Prompt gamma-ray activation analysis (PGAA)	H, N, S

Source of Material: Approximately 500 kg of washed metallurgical coal was obtained from the Tech Coal Line Creek Mine in Sparwood, British Columbia, Canada. This multi-seam coal was crushed and air-dried prior to being pulverized and screened at 212 µm (70 mesh). The resulting fraction of coal, less than 212 µm, was divided into two portions using the spinning riffler technique. One portion was stored in bulk. The other portion was divided using the spinning-riffler technique into 50 g units and bottled.

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APPENDIX C

Interlaboratory Study Summary Statistics: Test portions of SRM 2693a were analyzed as unknown samples in the interlaboratory study (CANSPEX) 2018-1 conducted by Quality Assurance International, Ltd. The tables are included as shown in the summary report by Quality Associates International, Ltd. Table C1 shows the summary results and Table C2 shows the derived standard deviations and a tally of published methods used in the study. The values have not been altered. Table C2 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 non-certified values.

Table C1. SRM 2693a CANSPEX Interlaboratory Study Results

Parameter	Most Likely Value [(a)]	Unit	95 % Coverage Interval of Most Likely Value	Pooled Within Lab Standard Deviation (s_w)	Pooled Between Lab Standard Deviation (s_B)	Total Number of Labs
Moisture	1.20	%	0.029	0.051	0.134	116
Ash	9.93	%	0.012	0.043	0.055	113
Volatile	21.99	%	0.12	0.12	0.50	89
Btu	13 862 [(b,c)]	per lb	7	19	31	106
Carbon	79.81	%	0.17	0.21	0.56	66
Hydrogen	4.362	%	0.028	0.038	0.090	60
Nitrogen	1.171	%	0.017	0.018	0.054	61
Total Sulfur	0.333	%	0.004	0.007	0.016	113
Pyritic Sulfur	0.27	%	0.007	0.004	0.011	17
Sulfate Sulfur	0.014	%	0.009	0.006	0.010	10
Chlorine	56	µg/g	6	5	17	48
Fluorine	88	µg/g	6	5	15	38
Mercury	57	ng/g	2	3	7	46
Selenium	0.84	µg/g	0.17	0.05	0.25	16

[(a)] Values are expressed on a dry-mass basis for all parameters except moisture. The moisture value is expressed on an "as received" basis.

[(b)] The gross calorific value may decrease upon aging, and this decrease will accelerate after the unit has been removed from the aluminized bag packaging (see "Use").

[(c)] Btu_{th}/lb • 2324.444 = J/kg [2].

Table C2. Derived Standard Deviations (in %) of Repeatability (s_r) and Reproducibility (s_R), and Tally of Published Methods Used in CANSPEX Interlaboratory Study[a]

Parameter	Total Number of Labs	Standards Australia (AS)[c]				ASTM International				British Standards Institution (BSI)[c]				Deutsches Institut für Normung (DIN)[c]				China National Standards (GB)[c]				International Organization for Standardization (ISO)[c]				Association Francaise de Normalisation (NF)[c]				South African Bureau of Standards (SABS)[c]				In-house[b]
		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 %	116	**1038.3**	0.04	-	1	**D2013**	0.04	0.09	0	**1016**	0.04	-		**51718**	0.07	-	3	**212**	0.07	-	1	**589**	0.11	-	1	**3-037**	-	-		**925**	-	-	0	6
						D3173	0.04	0.09	54													**11722**	0.04	-	18									
						D3302	0.04	0.09	0													**5068**	0.07	-	1									
						D5142	0.08	0.10	10																									
						D7582	0.09	0.28	21																									
Ash % dry basis	113	**1038.3**	0.04	0.05	1	**D3174**	0.08	0.11	55	**1016**	0.05	0.11		**51719**	0.07	0.11	3	**212**	0.07	0.11	1	**1171**	0.07	0.11	19	**3-003**	-	-			-	-		3
						D5142	0.10	0.13	10																									
						D7582	0.07	0.11	21																									
Volatile % dry basis	89	**1038.3**	0.07	0.35	1	**D3175**	0.18	0.35	35	**1016**	0.11	0.35		**51720**	0.23	0.31	2	**212**	0.18	0.35	1	**562**	0.23	0.31	24									1
						D5142	0.21	0.59	7																									
						D7582	0.13	0.47	18																									
Btu/lb dry basis	106	**1038.5**	20	46	1	**D1989**	23	39	2	**1016**	18	43		**51900**	18	46	4	**213**	18	46	1	**1928**	43	106	22									3
						D2015	24	38																										
						D3286	18	35	2																									
						D5865	24	38	71																									
Carbon % dry basis	66	**1038.6.4**	0.11	0.21	1	**D3178**	0.11	-						**51732**	-	-	2	**476**	0.18	0.35	1	**609**	0.09	0.18										3
						D5373	0.16	0.35	46													**29541**	0.18	0.46	13									
Hydrogen % dry basis	60	**1038.6.4**	0.04	0.07	1	**D3178**	0.02	-						**51732**	-	-	2	**476**	0.05	0.09	1	**609**	0.04	0.09										1
						D5373	0.04	0.09	42													**29541**	0.04	0.14	13									
Nitrogen % dry basis	61	**1038.6.4**	0.01	0.03	1	**D3179**	0.04	0.08						**51732**	-	-	2	**476**	0.03	0.05	1	**333**	0.02	0.04	1									2
						D5373	0.02	0.05	42													**29541**	0.01	0.05	12									
Total Sulfur % dry basis	113	**1038.6.3.3**	0.00	0.01	1	**D3177**	0.02	0.04		**1016**	0.02	0.04		**51724-3**	0.01	0.02	2	**214**	0.04	0.09	1	**351**	0.02	0.04	4	**3-038**	-	-	0		-	-		9
						D4239	0.01	0.02	82													**19579**	0.01	0.02	12									
						D5016	0.03	0.09	2																									
Pyritic Sulfur % dry basis	17	**1038.11**	0.02	0.05	1	**D2492**	0.03	0.06	14									**215**	0.02	0.04	1													1
Sulfate Sulfur % dry basis	10	**1038.11**	0.007	0.011	1	**D2492**	0.007	0.014	8									**215**	0.01	0.04	1													
Chlorine µg/g dry basis	48	**1038.8.2**	35	71	1	**D2361**	106	213		**1016**	177	177		**51727**	71	106	1	**3558**	35	71	1	**587**	-	-	1	**3-009**	-	-	1		-	-		16
						D4208	20	76	19																									
						D6721	2	4	9																									
Fluorine µg/g dry basis	38					**D3761**	5	5	17					**51723**	8	14	1	**4663**	6	7	1	**11724**	4	7	2	**03-009**	-	-						14
						D5987	4	7	3																									
Mercury ng/g dry basis	46					**D6414**	6	6	2					**22022**	-	-																		9
						D6722	3	5	35																									
Selenium µg/g dry basis	16					**D4606**	0.170	0.13	3	**5.000**																								13

[a] 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.
[b] 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.
[c] "-" 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 Apr 2024)	GB	https://www.gbstandards.org/index (accessed Apr 2024)
ASTM	https://www.astm.org/ (accessed Apr 2024)	ISO	https://www.iso.org/standards.html (accessed Apr 2024)
BSI	https://www.bsigroup.com/ (accessed Apr 2024)	NF	https://www.afnor.org/en/ (accessed Apr 2024)
DIN	https://www.din.de/en/about-standards (accessed Apr 2024)	SABS	https://www.sabs.co.za/ (accessed Apr 2024)

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