



# Certificate of Analysis

## Standard Reference Material<sup>®</sup> 2683c

### Bituminous Coal (Nominal Mass Fraction 2 % Sulfur)

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of techniques employed in the determination of sulfur, mercury, and chlorine in coal and materials of a similar matrix. A unit of SRM 2683c consists of 50 g of bituminous coal ground to pass a 250  $\mu\text{m}$  (60 mesh) sieve, homogenized, packaged in an amber glass bottle under an argon atmosphere, and sealed in an aluminized bag.

**Certified Mass Fraction Values:** The certified values for mercury and sulfur, expressed as mass fractions [1] on a dry-mass basis, are provided in Table 1. The mercury certified value is based on or directly traceable to a single NIST primary method. 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 Mass Fraction Value:** The reference value for chlorine, expressed as a mass fraction [1] on a dry-mass basis, is 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].

**Expiration of Certification:** The certification of SRM 2683c is valid, within the measurement uncertainty specified, until **01 July 2030**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The 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 or register online) will facilitate notification.

Technical measurements leading to certification of this SRM were coordinated by T.W. Vetter of the NIST Chemical Sciences Division. Analytical measurements were performed by S.J. Christopher and J.L. Mann of the NIST Chemical Sciences Division, S.E. Long, formerly of NIST, and E.A. Mackey of the NIST Office of Safety, Health and Environment.

Statistical analyses of sulfur were performed by A.L. Pintar of the NIST Statistical Engineering Division. Statistical analyses of mercury and chlorine were performed by S.D. Leigh, formerly of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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Chemical Sciences Division

Gaithersburg, MD 20899  
Certificate Issue Date: 07 May 2021  
*Certificate Revision History on Page 4*

Steven J. Choquette, Director  
Office of Reference Materials

## INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

**Sampling:** 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 certified value for mercury, 100 mg minimum should be used for analytical determinations to be related to the certified value for sulfur, and 750 mg minimum should be used to be related to the reference value for chlorine. The SRM should be stored in its original bottle, tightly sealed and away from sunlight and intense sources of radiation.

**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 the analysis. The correction is determined by oven drying separate 1 g test portions in a nitrogen atmosphere at  $107\text{ °C} \pm 3\text{ °C}$  to a constant mass [3], or use of an equivalent technique. The dry-mass basis was determined at NIST using a thermogravimetric method [4]. During drying at NIST, the mass loss of SRM 2683c samples ( $n = 8$ ) stabilized after approximately 75 minutes in nitrogen and approximately 60 minutes in air.

The mass loss determined in both a nitrogen and air atmosphere, which is reported *for information purposes only*, was nominally 3.3 %. The mass loss determined by the user may be different, depending on ambient conditions when the bottle is sampled.

## SOURCE, PREPARATION, AND HOMOGENEITY ASSESSMENT<sup>(1)</sup>

**Preparation:** SRM 2683c is a 50/50 blend of Illinois State Geological Survey IBC-112 coal (Herrin, II No. 6 coal) and Electric Fuels Corporation SPC-B coal (a blend of coal from various seams found in Harlan County, KY). Each coal was crushed to a nominal 4.76 mm (8 mesh) particle size and subsequently pulverized until the entire lot passed a 250  $\mu\text{m}$  sieve (60 mesh). Next, the two coals were combined and mixed into a single lot. The entire lot was then divided by the spinning riffle technique into two portions. One portion was divided further by the spinning riffle technique into units of SRM 2683b (the previous issue of this material) and bottled under an argon atmosphere. The other portion was stored in bulk under an argon atmosphere and, at a later date, was divided further by the spinning riffle technique into units of SRM 2683c and bottled under an argon atmosphere.

**Homogeneity Testing:** Twenty-four bottles of SRM 2683c were selected for homogeneity assessment. Duplicate test portions from each bottle were analyzed by X-ray fluorescence spectrometry for sulfur and chlorine. Measured gross count rates were used to assess homogeneity. For both elements, one-way analyses of variance fail to reject the null hypothesis of no bottle effect, which is indicative of material homogeneity (sulfur  $p$ -value = 0.465, chlorine  $p$ -value = 0.171).

## VALUE ASSIGNMENT

Measurements were performed to determine uniformity between SRM 2683c and SRM 2683b, which are from the same original bulk material (see "Preparation"). No significant differences were found as discussed below. Each mass fraction value is expressed with an expanded uncertainty,  $U = ku_c$ , calculated according to the method in the ISO/JCGM Guide [5]. The quantity  $u_c$  represents, at the level of one standard deviation, the combined effects of uncertainty due to components associated with material and measurement variability. The quantity,  $k$ , is the coverage factor used to obtain an expanded uncertainty with an approximately 95 % level of confidence. The value is  $k = 2$ , unless stated otherwise.

**Certified Mass Fraction Values:** Metrological traceability is to the International System of Units (SI) derived units for mass fraction (expressed as micrograms per kilogram or as percent).

The mercury mass fraction value of SRM 2683c was measured by isotope dilution cold-vapor inductively coupled plasma mass spectrometry (ID-CV-ICP-MS) [6] and no statistically significant difference from the certified value determined for SRM 2683b was found. The certified mercury value and uncertainty for SRM 2683b (also determined by ID-CV-ICP-MS) have been assigned to SRM 2683c. The coverage factor,  $k = 2.424$ , is determined from the Student's  $t$ -distribution for 6.24 degrees of freedom and 95 % confidence.

The sulfur mass fraction value of SRM 2683c was measured by isotope dilution microwave induced combustion inductively coupled plasma mass spectrometry (ID-MIC-ICP-MS) [7], and in a Coal and Ash Sample Proficiency Exchange (CANSPEX) interlaboratory study (ILS). A CANSPEX ILS result was calculated based on a Bayesian data

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<sup>(1)</sup>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.

analysis of the CANSPEX ILS measurements. The statistical model for this reduction includes between-laboratory and within-laboratory differences. The ID-MIC-ICP-MS measurement and uncertainty is combined with the CANSPEX ILS result as an equally-weighted mixture of two probability distributions.

Table 1. Certified Mass Fraction Values (Dry-Mass Basis) for Mercury and Sulfur in SRM 2683c

Element	Mass Fraction
Mercury (Hg)	$90.0 \pm 3.6$ ( $\mu\text{g}/\text{kg}$ )
Sulfur (S)	$1.952 \pm 0.026$ (%)

**Reference Mass Fraction Value:** The reference mass fraction value for chlorine is based on measurements by prompt gamma-ray activation analysis (PGAA) of SRM 2683c. The measurand is the chlorine mass fraction on a dry-mass basis as listed in Table 2. The reference value should not be used to establish metrological traceability to the International System of Units.

Table 2. Reference Mass Fraction Value (Dry-Mass Basis) for Chlorine in SRM 2683c

Element	Mass Fraction
Chlorine (Cl)	$1127 \pm 20$ ( $\text{mg}/\text{kg}$ )

#### SUPPLEMENTAL INFORMATION

Summary statistics reported by Quality Associates International, Ltd. (Sechelt, BC Canada) for the CANSPEX 2010-4 ILS using SRM 2683c 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. See text in the Appendix about limitations on using this data.

## REFERENCES

- [1] 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 May 2021).
- [2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at <https://www.nist.gov/system/files/documents/srm/SP260-136.PDF> (accessed May 2021).
- [3] ASTM D7582-10, *Standard Test Methods for Proximate Analysis of Coal and Coke by Macro Thermogravimetric Analysis*; Annu. Book ASTM Stand., Vol. 05.06 (2010).
- [4] Mann, J.L.; Kelly, W.R.; MacDonald, B.S.; *Observations of Anomalous Mass-Loss Behavior in SRM Coals and Cokes on Drying*; Anal. Chem., Vol. 74, pp. 3585–3591 (2002).
- [5] 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 (JCGM) (2008); available at <https://www.bipm.org/en/publications/guides> (accessed May 2021); 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 May 2021).
- [6] Long, S.E.; Kelly, W.R.; *Determination of Mercury in Coal by Isotope Dilution Cold-Vapor Generation Inductively Coupled Plasma Mass Spectrometry*; Anal. Chem., Vol. 74, pp. 1477–1483 (2002).
- [7] Christopher, S.J.; Vetter, T.W.; *Application of Microwave-Induced Combustion and Isotope Dilution Strategies for Quantification of Sulfur in Coals via Sector-Field Inductively Coupled Plasma Mass Spectrometry*; Anal. Chem., Vol 88, pp. 4635–4643 (2016).

<p><b>Certificate Revision History:</b> <b>07 May 2021</b> (Change of expiration date; sulfur certified value and uncertainty updated in Table 1; minimum sample requirements for sulfur changed; mass loss from drying changed to information value; editorial changes); <b>10 September 2019</b> (Change of expiration date; editorial changes); <b>19 December 2016</b> (Change of expiration date; editorial changes); <b>22 May 2012</b> (Revision of uncertainty for sulfur; editorial changes); <b>20 April 2011</b> (Original certificate date).</p>
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*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; e-mail [srminfo@nist.gov](mailto:srminfo@nist.gov); or via the Internet at <https://www.nist.gov/srm>.*

APPENDIX A

Test portions of SRM 2683c were analyzed as unknown samples in the CANSPEX interlaboratory study (ILS) 2010-4, conducted by Quality Associates International, Ltd. Summary results shown in Tables A1 and A2 were provided by Quality Associates International, Ltd. These data provide complementary information about the material, which may be useful to the SRM user. However, the CANSPEX ILS results should **NOT** be used as substitutes for NIST values or for calibration, validation, or establishing metrological traceability.

Table A1. SRM 2683c CANSPEX ILS Results

Parameter	Most Likely Value <sup>(a)</sup>	Unit	95 % Coverage Interval of Most Likely Value	Pooled Within-Lab Standard Deviation (sw)	Pooled Between-Lab Standard Deviation (sb)	Total Number of Labs
Moisture	3.185	%	0.057	0.057	0.270	124
Ash	9.870	%	0.017	0.050	0.062	122
Volatile	35.84	%	0.18	0.18	0.77	100
Btu	13 003	per lb	11	20	44	111
Carbon	73.38	%	0.22	0.26	0.72	62
Hydrogen	4.886	%	0.040	0.042	0.120	58
Nitrogen	1.635	%	0.018	0.022	0.054	58
Total Sulfur	1.954	%	0.016	0.018	0.073	119
Pyritic Sulfur	0.541	%	0.033	0.011	0.062	21
Sulfate Sulfur	0.199	%	0.014	0.006	0.024	21
Chlorine	1053	µg/g	37	36	102	48
Fluorine	82	µg/g	8.2	4.2	18.3	30
Mercury	89	ng/g	3	5	7	37
Selenium	1.92	µg/g	0.34	0.12	0.49	14
FSI <sup>(b)</sup>	2.2		0.2	0.4	0.3	55

<sup>(a)</sup>Values are expressed on a dry-mass basis for all parameters except moisture. The moisture value is expressed on an “as received” basis.

<sup>(b)</sup>Free Swelling Index

Parameter	Total Number of Labs	Table A2. Derived Standard Deviations (in %) of Repeatability ( $s_r$ ) and Reproducibility ( $s_R$ ), and Tally of Published Methods Used in CANSPEX ILS <sup>(a)</sup>																																	
		Standards Australia (AS)				ASTM International				British Standards Institution (BSI)				Deutsches Institut für Normung (DIN)				China National Standards (GB)				International Organization for Standardization				Association Francaise de Normalisation (NF)				South African Bureau of Standards (SABS)				In-House <sup>(b)</sup>	
		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 (%)	124	1038.3	0.04	- <sup>(c)</sup>	1	D2013	0.04	0.10	1	1016	0.04	-	1	51718	0.07	-	2	212	0.07	-	1	589	0.11	-	1	3-037	-	-	1	925	-	-	1	9	
						D3173	0.04	0.10	55												11722	0.04	-	7											
						D3302	0.04	0.10	13													5068	0.07	-	1										
						D5142	0.08	0.12	27																										
Ash (%)	122					D7582	0.09	0.2801	3																										
		1038.3	0.04	0.05	1	D3174	0.08	0.11	62	1016	0.05	0.11	1	51719	0.07	0.11	2	212	0.07	0.11	1	1171	0.07	0.11	11	3-003	-	-	1		-	-		8	
						D5142	0.09	0.13	32																										
Volatile (%)	100					D7582	0.07	0.11	3																										
		1038.3	0.07	0.35	1	D3175	0.18	0.35	47	1016	0.11	0.35	1	51720	0.38	0.51	2	212	0.18	0.35	1	562	0.38	0.51	12									7	
						D5142	0.28	0.82	27																										
Btu (per lb)	111					D7582	0.13	0.47	2																										
		1038.5	20	46	1	D1989	23	39	4	1016	18	43	1	51900	18	46	5	213	18	46	1	1928	43	106	13									4	
						D2015	24	38	3																										
						D3286	18	35	2																										
Carbon (%)	62					D5865	24	38	77																										
		1038.6.4	0.11	0.21	1	D3178	0.11	-	0					51732	-	-	1	476	0.18	0.35	1	609	0.09	0.18	2									5	
Hydrogen (%)	58					D5373	0.16	0.35	49												12902	-	-	3											
		1038.6.4	0.04	0.07	1	D3178	0.02	-	1					51732	-	-	1	476	0.05	0.09	1	609	0.04	0.09	2									3	
Nitrogen (%)	58					D5373	0.04	0.09	46												12902	-	-	3											
		1038.6.4	0.01	0.03	1	D3179	0.09	0.15	1					51732	-	-	1	476	0.03	0.05	1	333	0.02	0.04	2									4	
Total Sulfur (%)	119					D5373	0.02	0.05	45												12902	-	-	3											
		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	1	214	0.04	0.09	1	351	0.02	0.04	1	3-038	-	-	1		-	-		15	
						D4239	0.03	0.07	92																										
Pyritic Sulfur (%)	21	1038.11	0.02	0.05	1	D2492	0.05	0.10	19											215	0.02	0.04	1											0	
Sulfate Sulfur (%)	21	1038.11	0.007	0.011	1	D2492	0.007	0.014	19											215	0.01	0.04	1											0	
Chlorine (µg/g)	48		-	-		D2361	106	213	2	1016	177	177	0	51727	71	106	2	3558	35	71	1					3-009	-	-	0		-	-		16	
						D4208	66	157	20																										
						D6721	23	28	7																										
Fluorine (µg/g)	30					D3761	5	5	14					51723	8	14	1	4663	6	7	1	11724	4	7	3	03-009	-	-	1					7	
						D5987	4	7	3																										
Mercury (ng/g)	37					D6414	8	9	5					22022	-	-	0																		9
						D6722	3	7	23																										
Selenium (µg/g)	14					D4606	0.246	0.24	4																									10	
FSI	55	1038.17	0.18	0.35	1	D720	0.35	0.71	45	1016	-	-	1	51741	-	-	1	5448	0.35	0.53	1	501	0.35	0.18	4									2	

- (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. Values are expressed on a dry-mass basis for all parameters except moisture. The moisture value is expressed on an "as received" basis.
- (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 May 2021)  
 ASTM <https://www.astm.org/> (accessed May 2021)  
 BSI <https://www.bsigroup.com/> (accessed May 2021)  
 DIN <https://www.din.de/en> (accessed May 2021)

GB <https://www.gbstandards.org/> (accessed May 2021)  
 ISO <https://www.iso.org/standards-catalogue/browse-by-ics.html> (accessed May 2021)  
 NF <https://www.iso.org/member/1738.html> (accessed May 2021)  
 SABS <https://www.sabs.co.za/> (accessed May 2021)