National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material® 1632b

Trace Elements in Coal

(Bituminous)

This Standard Reference Material (SRM) is intended primarily for use in the calibration of apparatus and the evaluation of techniques employed in the analysis of coal and similar materials. SRM 1632b consists of a 50 g bottle of bituminous coal with a nominal sulfur content of 1.9 %. It was ground to pass a 250 μm (60 mesh) sieve and homogenized before bottling.

Certified Values: The certified values for SRM 1632b are given in Table 1. The certified values are based on measurements using proven analytical techniques and methods. All values are reported as mass fractions [1], on a dry mass basis (see Instructions for Drying) and are based on measurements using a sample mass of at least 250 mg. A list of the analytical techniques and methods used for the different analyses is given in Table 3.

Information Values: Information values for additional trace elements are given in Table 2. These are noncertified values with no uncertainties as there is insufficient information to make an assessment of the uncertainties.

Expiration of Certification: The certification of SRM 1632b is valid, within the measurement uncertainties specified, until **30 June 2000** 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 modified.

Maintenance of SRM Certification: NIST will monitor representative samples of 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. Return of the attached registration card will facilitate notification.

Instructions for Use: The unit should be thoroughly mixed by rotating the bottle before sampling. A minimum sample mass of 250 mg (dry mass) should be used for analytical determinations to be related to the certified values provided. The calorific value and ash content were determined using a minimum sample mass of 1 g. The SRM should be stored in its original tightly sealed bottle away from sunlight and intense sources of radiation.

Statistical analysis of the certification data was performed by R.C. Paule and S.B. Schiller of the NIST Statistical Engineering Division.

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills. Revision of the certification was coordinated through the Standard Reference Material Program by B.S. MacDonald.

Gaithersburg, MD 20899
Certificate Issue Date: 10 December 1998*

Thomas E. Gills, Chief
Standard Reference Materials Program

20 Jun 85 (original certificate date); 26 Mar 90 (calorific value updated; volatile matter changed from certified to information value; ash uncertainty updated); 13 Nov 92 (calorific value updated); 10 Jul 93 (editorial); 12 Apr 95 (certified fluorine added); 17 Oct 95 (informational mercury added); 3 Mar 97 (change in the carbon and hydrogen values from certified to information values); 11 Jun 97 (updated carbon and hydrogen certified values and a change in the expiration date); 27 Jan 98 (correction in the certified value for rubidium)
*This revision reports an updated information value for mercury.

Source and Preparation of Material: The coal for this SRM was obtained from the Humphrey No. 7 mine and coal preparation plant of the Consolidated Coal Company, Christopher Coal Company Division, Osage, WV. This mine produces bituminous coal with a sulfur content of 1.8 % to 1.9 % (dry basis). The coal was obtained from an underground mine that recovers coal from the Pittsburgh seam which is considered the single most valuable and extensive coal seam in the United States. Approximately 900 kg of the coal for SRM 1632b was oven dried prior to processing, in accordance with procedures outlined in ASTM D 2013 [2]. The coal was reduced in size to pass a 250 gm (60 mesh) sieve prior to blending. The coal was then blended in a stainless steel cone blender (approximate capacity 0.85 m^3). After blending, the coal was packaged in polyethylene-lined aluminum cans and was subsequently repackaged (bottled) in 50 g units. Homogeneity testing was done on both the bulk material and the bottled units using X-ray fluorescence spectrometry. The relative intensities of the elements Al, Ca, Fe, K, S, Si, and Ti were measured in randomly selected samples of the bottled units and/or cans and no statistically significant differences were observed within or between bottles or between cans of bulk material.

Instructions for Drying: The recommended procedure for drying is vacuum drying at ambient temperature for 24 h or oven drying for 2 h at 105 °C. Typical moisture loss using the recommended method for drying is approximately 1.6 % relative. However, for the calorific value, a moisture determination should be made on a duplicate analysis sample of the coal and that moisture value then used to convert the calorific value to a dry mass basis.

Table 1. Certified Mass Fractions for Selected Elements

Major Constituents

Elements	Mass Fraction (in %)
Carbon (Total)	76.86 ± 0.26
Hydrogen	4.94 ± 0.13
Nitrogen	1.56 ± 0.07
Sulfur	1.89 ± 0.06

Minor Constituents

Elements	Mass Fraction (in %)
Aluminum	0.855 ± 0.019
Calcium	0.204 ± 0.006
Iron	0.759 ± 0.045
Magnesium	0.0383 ± 0.0008
Potassium	0.0748 ± 0.0028
Sodium	0.0515 ± 0.0011
Titanium	0.0454 ± 0.0017

Trace Elements

Elements	Mass Fraction (in mg/kg)	Elements	Mass Fraction (in mg/kg)
Arsenic	3.72 ± 0.09	Manganese	12.4 ± 1.0
Barium	67.5 ± 2.1	Nickel	6.10 ± 0.27
Cadmium	0.0573 ± 0.0027	Rubidium	5.05 ± 0.11
Cobalt	2.29 ± 0.17	Selenium	1.29 ± 0.11
Copper	6.28 ± 0.30	Thorium	1.342 ± 0.036
Fluorine[a]	41.7 ± 3.2	Uranium	0.436 ± 0.012
Lead	3.67 ± 0.26	Zinc	11.89 ± 0.78

Calorific Value[b] (31.90 ± 0.24) $MJ \cdot kg^{-1}$ [(13715 ± 103) $Btu \cdot lb^{-1}$] [Note: $MJ \cdot kg^{-1} = 429.9226\ Btu_{th} \cdot lb^{-1}$]

Ash, Mass Fraction (in %) 6.8 ± 0.1

[a] The listed uncertainty for fluorine is two standard deviations of the certified value. The listed uncertainties for all other constituents are two standard deviations for the certified values and include an allowance for minor sample heterogeneity. The observed sample variability was generally less than 2 % of the constituent value.

[b] For the certified calorific value, determined as HHV2 (Higher Heating Value-Moisture Free), the uncertainty is a 95 % confidence interval with an additional allowance for sample degradation. The calorific value ($MJ \cdot kg^{-1}$) may decrease upon aging or normal oxidation of the coals. NIST will continue to monitor this value and report any substantive change in the certified calorific value to the purchaser. The reference date for the calorific value on this certificate is October, 1992.

SUPPLEMENTAL INFORMATION

The information values given in Table 2 are provided for information only as additional information on the matrix. They are based on measurements made using a single technique or method. While no reason exists to suspect systematic bias in the information values, no attempt was made to determine if such a bias exists.

Table 2. Information Mass Fractions for Trace Constituents

Element	Mass Fraction (in mg/kg)	Element	Mass Fraction (in mg/kg)
Antimony	0.24	Lithium	10
Bromine	17	Mercury	0.07
Cerium	9	Molybdenum	0.9
Cesium	0.44	Samarium	0.87
Chlorine	1260	Scandium	1.9
Chromium	11	Silicon	14000
Europium	0.17	Strontium	102
Hafnium	0.43	Tungsten	0.48
Lanthanum	5.1	Vanadium	14

Volatile Matter, 35 % (mass fraction)

The following NIST Analytical Chemistry Division technical staff participated in the characterization and certification of this SRM:

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Certification analyses for fluorine were performed by members of the ASTM Committee D05 Fluorine Task Group:

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Table 3. Methods of Analysis

Al	FES, XRF
As	HGAAS
Ash	ASTM D3174, Commercial Coal Analyzer
Ba	INAA
Br	INAA
C (Total)	ASTM D3178, Commercial Coal Analyzer
Ca	IDMS, INAA, FES, XRF
Calorific Value	Commercial Coal Analyzer, Commercial Calorimeter
Cd	AAS, IDMS
Ce	INAA
Cl	INAA
Co	INAA, FES
Cr	INAA, ICP-AES
Cs	INAA
Cu	HGAAS, ICP-AES
Eu	INAA
F	Pyrohydrolysis with IC and ISE (ASTM D3761-91) Bomb Combustion with ISE, Fusion with ISE, PIGE
Fe	AAS, INAA
H	ASTM D3178, Commercial Coal Analyzer
Hf	INAA
Hg	FIA-CVAAS
K	IDMS, INAA, FES
La	INAA
Li	FES
Mg	AAS, IDMS, INAA
Mn	INAA, FES
Mo	INAA
N	ASTM D3179
Na	INAA, FES
Ni	AAS, ICP-AES
Pb	AAS, IDMS
Rb	IDMS, INAA, FES
S	IC, ASTM D3177, Commercial Coal Analyzer, XRF
Sb	INAA
Sc	INAA
Se	INAA, HGAAS
Si	INAA, XRF
Sm	INAA
Sr	INAA
Th	IDMS, INAA
Ti	INAA, FES, XRF, ICP-AES
U	IDMS, INAA
V	INAA
Volatile Matter	ASTM D3175, Commercial Coal Analyzer
W	INAA
Zn	AAS, IDMS, INAA

Methods

AAS	Atomic absorption spectrometry
IDMS	Isotope dilution mass spectrometry
INAA	Instrumental neutron activation analysis
FIA-CVAAS	Flow injection cold vapor atomic absorption spectrometry
FES	Flame emission spectrometry
HGAAS	Hydride generation atomic absorption spectrometry
ETAAS	Electrothermal atomic absorption spectrometry
IC	Ion chromatography
GC	Gas chromatography
XRF	X-ray fluorescence
ICP-AES	Inductively coupled plasma atomic emission spectrometry
PIGE	Proton induced gamma-ray emission
ISE	Ion selective electrode

REFERENCES

[1] Taylor, B.N., "Guide for the Use of International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).

[2] ASTM D 2013-78, "Method for Preparing Coal Samples for Analysis," **Vol. 05.05** ASTM Book of Standards, West Conshohocken, PA.

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) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet http://ts.nist.gov/srm.