National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2684a

Sulfur in Coal

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of methods and the calibration of instruments used in the determination of sulfur in coal. In addition to sulfur, it is also certified for ash content and calorific value ($MJ \cdot kg^{-1}$). SRM 2684a consists of a 50-g bottle of bituminous coal that was ground to pass a 250-μm (60 mesh) sieve and homogenized.

Certified Values: The certified values for SRM 2684a are given in Table 1. Except for sulfur, the certified values are based on measurements using two or more independent, reliable techniques or methods. The sulfur content is based solely on measurements from isotope dilution thermal ionization mass spectrometry, one of NIST's most accurate analytical techniques.

The methods used for the certification analyses are given in Table 2. Noncertified values for major and minor elements are given in Table 3. These noncertified values are provided for information only. They are based on measurements made by using a single technique or method.

Notice to Users: The certified calorific value ($MJ \cdot kg^{-1}$) decreases with aging and normal oxidation of the coals. NIST redetermines the calorific value annually and revises the Certificate of Analysis accordingly. The reference date for the calorific data in this certificate is October, 1992.

Use: The bottle of coal should be thoroughly mixed by rotating the bottle before sampling. The certified sulfur value is based on a sample size of at least 100 mg of the dried material (see drying instructions) and is reported on a "dry-weight" basis. The calorific value and ash content were determined using minimum sample weights of 1 g.

Expiration of Certification: The certification of 2684a, except for the calorific value, will be valid up to 5 years from the purchase date. Should any of the certified values or physical parameters become invalid prior to that date, purchasers will be notified.

The overall direction and coordination of technical measurements leading to the certification of this SRM were performed in the NIST Inorganic Analytical Research Division under the chairmanship of W.F. Koch.

The 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 preparation, certification, and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by T. E. Gills.

Gaithersburg, MD 20899
November 13, 1992
(Revision of certificate dated 6-3-91)

William P. Reed, Chief
Standard Reference Materials Program

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Special Publication 260-84, Sampling, Material Handling, Processing, and Packaging of NBS Sulfur in Coal Standard Reference Materials. The coal was oven dried prior to processing in accordance with procedures outlined in ASTM D2013. At least 500 kg of the coal was reduced in size to -250 μm (-60 mesh) and screened prior to blending. The coal was blended in a stainless steel cone blender (approximate capacity 0.85 cubic meter). SRM 2684a was further homogenized by a spinning riffling technique and then bottled into 50-g units. Homogeneity testing was done on bottled units using x-ray fluorescence spectrometry. Replicate analyses indicated the material variability for sulfur to be within ± 0.2% (relative).

Stability: This SRM is considered to be stable for its intended use if properly stored in its original tightly sealed bottle away from sunlight and intense sources of radiation. NIST will continue to monitor representative samples from the SRM "lot". Any substantive change in its certification or analysis will be reported to the purchaser.

Instructions for Drying: The certification of sulfur in this SRM is reported on a dry-weight basis; thus the concentration determined on undried samples should be adjusted for the moisture content of the sample. The recommended procedures for drying are vacuum drying at ambient temperature for 24 hours or oven drying for 2 hours at 105 °C. Typical moisture loss using the recommended methods for drying is approximately 3.8%. However, for the calorific value, a moisture determination is made on a duplicate analysis sample of coal and that moisture value is then used to convert the calorific value to a dry-weight basis.

SUPPLEMENTAL INFORMATION

The concentration values listed in Table 3 for the major and minor elements were determined using thermal neutron activation analysis and neutron capture prompt-gamma activation analysis. These values are not certified, but are provided as additional information on the matrix. While no reason exists to suspect systematic bias in these numbers, no attempt was made to determine if bias, attributable to the methods, exists.

Table 3. Noncertified Values for SRM 2684a

(Mean Concentrations (mg/kg) Unless Noted)

Element	Mean Concentration	Element	Mean Concentration
Al, wt.%	1.1	La	6.7
As	3.9	Mg, wt.%	0.08
B	114	Mn	36
Ba	41	N, wt.%	1.6
Br	11	Na, wt.%	0.03
C, wt.%	68	Rb	15
Ca, wt.%	0.44	Sb	0.35
Ce	12	Sc	2.7
Co	3.9	Se	1.9
Cr	17	Sm	1.1
Cs	1.2	Th	2.0
Eu	0.23	Ti, wt.%	0.06
Fe, wt.%	1.5	U	0.90
H, wt.%	4.8	V	22
Hf	0.57	W	0.56
K, wt.%	0.20	Zn	110

Table 1. Certified Values for SRM 2684a

SRM No.	Coal Type	Sulfur Wt.%	Furnace Ash Wt.%	Gross Calorific Value* $MJ \cdot kg^{-1}$ $(Btu \cdot lb)^{-1}$
2684a	Bituminous	3.06±0.03	11.0±0.1	28.50±0.24 (12253±103)

[Note: $MJ \cdot kg^{-1} = 429.9226\ Btu_{th} \cdot lb^{-1}$ was used for the calorific conversion].

*Higher Heating Value-Moisture Free

The uncertainty of a certified value, except for the calorific value, is expressed as two times the standard deviation of the certified value and includes observed variability within and between measurement methods and any observed material heterogeneity. For the certified calorific value, the uncertainty is a 95% confidence interval with an additional allowance for sample degradation.

Table 2. Analytical Methods and Techniques Used for the Determination of Sulfur, Furnace Ash, and Gross Calorific Value in SRM 2684a.

Certified Constituents/ Physical Property	Methods or Techniques Used
Sulfur†	A, B, C, D, E
Furnace Ash	E, F
Calorific Content - Higher Heating Value - Moisture Free (HHV2)	E, G, H, I, J

† Note: The certified sulfur value is based solely on measurements from Method A. Methods B,C,D,E were used to confirm the sulfur value.

A. Isotope Dilution Thermal Ionization Mass Spectrometry
B. X-ray Fluorescence Spectrometry
C. ASTM D3177 Standard Test Method for Total Sulfur in the Analysis Sample of Coal and Coke
D. ASTM D4239 Standard Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods (Method C)
E. Commercial Coal/ Sulfur Analyzer
F. ASTM D3174 Standard Test Method for Ash in the Analysis Sample of Coal and Coke
G. ASTM D2015 Standard Test Method for Gross Calorific Value of Coal and Coke by the Adiabatic Bomb Calorimeter
H. ASTM D3286 Standard Test Method for Gross Calorific Value of Coal and Coke by the Isoperibol Bomb Calorimeter
I. ASTM D3180 Standard Test Method for Calculating Coal and Coke Analyses from As-Determined to Different Bases
J. ASTM 3173 Standard Test Methods for Moisture in the Analysis Sample of Coal and Coke

Preparation and Testing: Approximately 900 kg of coal was obtained from the Delta mine of the Amax Coal Company, Marion, Illinois. The mine is a surface mine that produces bituminous coal with a sulfur content, after working, of 2.8-2.9 percent (dry basis). The coal mined at the Delta mine was obtained from the Illinois No. 6 Herrin seam. Additional information on sampling and preparation can be obtained from the NIST

ANALYSTS

Analyses for the certification of this SRM were performed in the following laboratories:

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