National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2682a

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 content it is also certified for ash content and calorific value ($MJ \cdot kg^{-1}$). SRM 2682a consists of a 50 g bottle of subbituminous coal that was ground to pass a 60 mesh sieve and homogenized.

Certified Values: The certified values for SRM 2682a 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 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 coal. NIST redetermines the calorific value routinely and revises the Certificate of Analysis accordingly. The user must be careful to use the most recent certificate value. The reference date for the calorific data in this certificate is September, 1993.

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 a 100 mg sample 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 SRM 2682a, except for the calorific value, will be valid up to 5 years from the date of shipment from NIST. 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 Inorganic Analytical Research Division under the chairmanship of W.F. Koch.

Gaithersburg, MD 20899
May 3, 1994
(Revision of certificate dated 6-3-91)

Thomas E. Gills, Chief
Standard Reference Materials Program

(over)

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

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by T.E. Gills. Updating of the BTU certification was coordinated by J.S. Kane.

Table 1. Certified Values for SRM 2682a

SRM No.	Coal Type	Sulfur[1] Wt. %	Furnace[1] Ash Wt. %	HHV2*[2] $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)
2682a	Subbituminous	0.486 ± 0.006	6.3 ± 0.1	25.48 ± 0.45 (10954 ± 193)

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

[1]The uncertainty is expressed as two times the standard deviation of the certified value; it includes observed variability within measurement methods and observed material heterogeneity.

[2]The certified calorific value and its uncertainty form a 95% confidence interval for 24 months of anticipated sample degradation. The certified value is the midpoint of this interval, which began September, 1993.

*HHV2: Higher Heating Value - Moisture Free

Table 2. Analytical Methods and Techniques Used for the Determination of Sulfur, Furnace Ash, and Calorific Content in SRM 2682a

Certified Constituent/ Physical Property	Technique(s) Used
Sulfur	A,B,C
Furnace Ash	D
Calorific Content, Higher Heating Value-Moisture Free (HHV2)	E,F

A. Isotope Dilution Thermal Ionization Mass Spectrometry
B. X-ray Fluorescence Spectrometry
C. ASTM D 4239 Method C - High Temperature Combustion with Infrared Absorption
D. ASTM D 3174 Standard Test Method for Ash in the Analysis Sample of Coal and Coke
E. ASTM D 2015 Standard Test Method for Gross Calorific Value of Coal and Coke by the Adiabatic Bomb Calorimeter
F. ASTM D 3286 Standard Test Method for Gross Calorific Value of Coal and Coke by the Isoperibol Bomb Calorimeter

Preparation and Testing: Approximately 900 kg of coal was obtained from the Amax Coal Company's Belle Ayr Mine near Gillette, in Campbell County, WY. This mine, opened for production in 1973, is an open pit mine that produces subbituminous coal from the Wyodak-Anderson coal seam, which is a part of the Powder River Coal Basin. Additional information on sampling and preparation can be obtained from the NBS Special Publication 260-84, Sampling, Materials 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 D 2013. At least 500 kg of the coal was reduced in size to -60 mesh and screened prior to blending. The -60 mesh coal was blended in a stainless steel cone blender (approximate capacity 0.85 cubic meter). The 2682a lot was further homogeneized by a spinning riffling technique and then bottled into 50 g units. Homogeneity testing was done on both the bulk materials and 50 g bottled units using X-ray fluorescence analysis. Replicate analyses indicated the material variability for sulfur to be within $\pm$ 0.2% (relative).

Stability: This SRM is considered to be stable for 5 years 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 procedure for drying is oven drying for 2 h at 105 °C. Typical moisture loss using the recommended method for drying is 16.7 $\pm$ 0.4 wt. %. However, for the calorific value, a moisture determination was made on a duplicate analysis sample of coal and that moisture value was then used for converting the calorific value to a dry weight basis.

ANALYSTS

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

National Institute of Standards & Technology, R. Greenberg, K.E. Hehn, W.R. Kelly, W.F. Koch, R.M. Lindstrom, P.A. Pella, Inorganic Analytical Research Division; D.R. Kirklin and E.L. Diaz-Chemical Thermodynamics Division.

Consolidation Coal Company, Library, PA 15129, T. Altman, K. Buchman, S. Clore, J.F. Cryster, L. Hrovatic, L.W. Rosendale, and R. Strong.

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 <u>not</u> certified but 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 2682a

Mean Concentrations (μg/g Unless Noted)

Element	Mean Concentration	Element	Mean Concentration
Al, wt.%	0.46	La	5.2
As	1.0	Mg, wt.%	0.2
B	39	Mn	26
Ba	382	N, wt.%	0.8
Br	3.7	Na, wt.%	0.10
C, wt.%	75	Rb	<2
Ca, wt.%	1.1	Sb	0.19
Ce	10	Sc	1.5
Co	1.7	Se	0.91
Cr	15	Sm	0.78
Cs	<0.1	Th	1.5
Eu	0.17	Ti, wt.%	0.05
Fe, wt.%	0.24	U	0.52
H, wt.%	4.7	V	15
Hf	0.60	W	1.8
Km, wt.%	0.01	Zn	8.6