National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 2692

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. It is also certified for ash content and calorific value ($MJ \cdot kg^{-1}$). SRM 2692 consists of a 50-g bottle of a bituminous coal that was ground to pass a 60-mesh (250μm) sieve and homogenized.

The certified value for sulfur is based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis (see drying instructions). The certified calorific value and ash content were determined using procedures recommended in ASTM methods (see references in Table 1). The certified values are given in Table 1, along with the methods used for certification. Noncertified values for major, minor, and trace elements are given in Table 2. These values are provided for information only and are not to be used for analytical purposes.

Table 1
Certified Values for SRM 2692

Sulfur[1,2,3] wt%	Furnace[4] Ash, wt%	HHV2[5,6] $MJ \cdot kg^{-1}$ ($Btu \cdot lb^{-1}$)
1.115 ± 0.019[a]	7.9 ± 0.2	30.61 ± 0.19 (13160 ± 80)

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

1. ASTM D3177 Standard Test Method for Total Sulfur in the Analysis Sample of Coal and Coke.
2. Ion Chromatography with Bomb Combustion.
3. Thermal Ionization Mass Spectrometry, Sealed Glass Tube Digestion.
4. ASTM D3174 Standard Test Method for Ash in the Analysis Sample of Coal and Coke.
5. ASTM D2015 Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Calorimeter.
6. ASTM D3180 Standard Test Method for Calculating Coal and Coke Analyses from As-Determined to Different Bases.

[a]The uncertainty is expressed as two times the standard deviation of the certified value and includes observed variability within and between measurements and any observed material heterogeneity.

Use: The SRM must be thoroughly mixed in the original bottle before sampling. Natural materials such as coal tend to segregate with time.

Notice to Users: The certified calorific value ($MJ \cdot kg^{-1}$) decreases upon the aging and/or normal oxidation of the sample. The National Institute of Standards and Technology (NIST) redetermines the calorific value each year and revises the Certificate of Analysis. The user must be careful to use the most current certificate. The reference date for the calorific data for this SRM is October 1988.

Certification analyses were performed by J.W. Gramlich, W.R. Kelly, W.F. Koch, and Chen Le-Tian (Guest Scientist), of the Inorganic Analytical Research Division and J.C. Colbert of the Chemical Thermodynamics Division.

The statistical analysis of the certification data was performed by R.C. Paule of the National Measurement Laboratory.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the Office of Standard Reference Materials by T.E. Gills.

November 15, 1988
Gaithersburg, MD 20899

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Material Preparation

Approximately one thousand 50-g bottles of a bituminous coal were obtained from a commerical supplier on contract to NIST. These coals were prepared according to NIST procurement specifications and protocols.

Homogeneity testing was performed on 10 randomly selected 50-g bottles of coal using x-ray fluorescence spectrometry. Replicate analysis indicated that there was no evidence of heterogeneity for the elements determined, i.e., Al, Ca, Cu, K, S, Si, and Zn.

The homogeneity studies were performed by P.A. Pella and G.A. Sleater of the Gas and Particulate Science Division.

Analysis

Sulfur: The certified sulfur content is based upon the results of 3 independent methods of analysis: ion chromatography, gravimetry, and thermal ionization isotope dilution mass spectrometry.

Calorific value ($MJ \cdot kg^{-1}$) and Ash Content: The certified values for the calorific value and ash content were determined using measurements made in an adiabatic bomb calorimeter of the type used in commercial laboratories. This calorimeter is capable of reproducing determinations on benzoic acid to a precision of 0.05% (relative). This statement of precision was arrived at by following the standardization procedure outlined in Item 10 of ASTM D2015. Benzoic Acid, SRM 39i, was used as the calibrant.

Major and Minor Elements: Analyses for major and minor elements were performed using instrumental neutron activation analysis (INAA) except for C, H, and N which were determined using an elemental analyzer (EA). These values, provided as Supplemental Information (Table 2), are not certified but are to be used for information only.

Stability

The long-term physical and chemical stability of this SRM, except for the calorific value, has not been rigorously established. It is recommended that the material be stored in the tightly sealed bottle away from sunlight and intense sources of radiation. NIST will continue to monitor this SRM and any substantive change in its certification or analysis will be reported to the user.

It is important that the attached registration form be completed and returned to NIST for obtaining proper notification of any change in the certified values.

Instructions for Drying

The certification of sulfur in this SRM is based upon a properly dried sample. The recommended procedure is vacuum drying at 20 °C to 25 °C and at a pressure not greater than 30 Pa (0.2mm Hg) for 24 hours or oven drying for 2 hours at 105 °C. Typical moisture loss for SRM 2692 is 1.6% relative. However, for the calorific value, the ASTM D3173 Method may be used wherein a moisture determination is made on a duplicate analysis sample of coal and the moisture value is then used for calculating the calorific value to a dry basis.

Supplemental Information

The noncertified values listed in Table 2 are based on measurements made using a single method or technique and are given for information only. While no reason exists to suspect systematic biases in these numbers, no attempt was made to determine if biases attributable to the methods exist.

The instrumental neutron activation (INAA) determinations were performed by D.A. Becker of the Inorganic Analytical Research Division and the C, H, and N results were obtained through a NIST cooperative analysis program with selected industrial laboratories.

Table 2

Noncertified Concentrations of Constituent Elements in SRM 2692

Element	Method	wt.%	Element	Method	µg/g
Al	INAA	1.2	Ce	INAA	22
C	EA	74	Co	INAA	6.3
Ca	INAA	0.09	Cr	INAA	17
Fe	INAA	.49	Cs	INAA	1.5
H	EA	5.1	Eu	INAA	0.5
K	INAA	0.19	Hf	INAA	.7
Mg	INAA	.06	Mn	INAA	14
N	EA	1.7	Rb	INAA	18
Na	INAA	0.03	Sb	INAA	1.9
			Sc	INAA	3.5
			Se	INAA	2.4
			Th	INAA	2.1
			V	INAA	25
			Zn	INAA	35

INAA = Instrumental Neutron Activation Analysis
EA = Elemental Analyzer (combustion)