



National Institute of Standards and Technology

Certificate of Analysis

Standard Reference Material[®] 2776

Sulfur in Furnace Coke

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of methods and for the calibration of instruments used to determine sulfur in furnace (metallurgical) coke. Each unit of SRM 2776 consists of 50 g of blast furnace coke that was ground to pass a 250 μm (60 mesh) sieve, homogenized, and bottled under an argon atmosphere.

The certified value for sulfur, expressed as a mass fraction [1] on a dry basis, is given in Table 1 (see Instructions for Drying). The certified value is based on measurements by isotope dilution thermal ionization mass spectrometry (ID-TIMS). The uncertainty in the certified value is calculated as $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO Guide [2] and k is a coverage factor. The value of u_c represents, at the level of one standard deviation, the combined effects of uncertainty components associated with material inhomogeneity and ID-TIMS measurement uncertainty. The coverage factor, $k = 2.20$ is the value from the t -distribution corresponding to 11 degrees of freedom and 95 % confidence.

Table 1. Certified Value (Mass Fraction)

Sulfur 0.825 % \pm 0.016 %

Reference values for ash [3,4] and volatile matter [4,5], expressed as mass fractions [1] on a dry basis, are provided in Table 2 (see Instructions for Drying). Reference values are based on results from a round robin exercise done in cooperation with ASTM Subcommittee D05.15 with 12 participating laboratories. The uncertainties in Table 2 are given as expanded uncertainties, as described in the ISO Guide [2]. They were calculated as 95 % confidence intervals, and represent the combined effects of between-lab and within-lab components of uncertainty. The percent volatile matter obtained by round robin testing was confirmed by measurements made with platinum crucibles under tightly controlled conditions according to ASTM D 3175-89a [5]. The percent dry fixed carbon for SRM 2776 can be calculated by subtracting the sum of the dry ash and dry volatile matter from 100 % [6].

Table 2. Reference Values (Mass Fraction)

Ash	8.06 % \pm 0.08 %
Volatile Matter	0.98 % \pm 0.15 %

Expiration of Certification: This certification is valid until **31 December 2010**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Instructions for Use). However, the certification will be nullified if the SRM is contaminated or modified. Return of the attached registration card will facilitate notification.

The support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899
Certificate Issue Date: 19 March 1998

Thomas E. Gills, Chief
Standard Reference Materials Program

The overall direction and coordination of the technical measurements leading to this certification were performed by W.R. Kelly, P.A. Pella, R.D. Vocke, Jr., and J.D. Fassett of the NIST Analytical Chemistry Division.

Statistical consultation was provided by K.R. Eberhardt and R.C. Hagwood of the NIST Statistical Engineering Division.

The blast furnace (metallurgical) coke for this SRM was donated by U.S. Steel Clairton Works, Carbon, PA.

NOTICE AND WARNINGS TO USERS

Stability: This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

Instructions for Use: To relate analytical determinations to the certified value on this Certificate of Analysis, a minimum sample mass of 150 mg should be used and the sample should be dried according to the procedures given in the section "Instructions for Drying". To relate analytical determinations to reference values, a nominal sample weight of 1 g should be used. When not in use, this SRM must be stored in a cool and dry environment away from sunlight and fumes.

Instructions for Drying: In order for users to directly relate their measurements to the certified value, loss-after-drying corrections should be measured and applied at the time of the analysis. The moisture correction for sulfur analysis was determined by drying separate 1 g samples in a nitrogen atmosphere at 107 °C to a constant weight. The average moisture measured at NIST for SRM 2776 was 0.33 % with individual determinations ranging from 0.29 % to 0.37 %.

SRM 2776 moisture results obtained from the ASTM Subcommittee D05.15 round robin ranged from 0.26 % to 0.39 %, when determined in a nitrogen atmosphere, and from 0.28 % to 0.46 %, when determined in an air atmosphere.

PREPARATION, HOMOGENEITY TESTING, AND ANALYSIS

Preparation: Blast furnace coke was collected and crushed to a nominal 4.76 mm (4 mesh) particle size. The crushed coke was subsequently pulverized with ceramic plates until the entire lot passed a 250 µm (60 mesh) sieve. Next, the material was divided by the spinning riffle technique into two portions. One portion was stored in bulk under an argon atmosphere. The other portion was further divided by the spinning riffle technique and bottled under an argon atmosphere.

Homogeneity Testing: Twelve bottles from the lot were selected for homogeneity testing. Samples from each bottle were analyzed by X-ray fluorescence for sulfur, strontium, iron, titanium, barium, silicon, calcium, potassium, and aluminum. No significant bottle-to-bottle differences were found for any of the elements tested. The effective sample size for sulfur seen by XRF analysis is approximately 75 mg.

Analysis: Certification analyses by ID-TIMS were performed by W.R. Kelly and R.D. Vocke, Jr. of the NIST Analytical Chemistry Division. The X-ray fluorescence homogeneity analysis was performed by A.F. Marlow and P.A. Pella of the NIST Analytical Chemistry Division. Reference values are based on ASTM Subcommittee D05.15 round robin testing. Cooperating analysts and laboratories are listed on page 3.

Cooperating Analysts and Laboratories:

J.R. Spaeth; Acme Steel Company, Chicago, IL
J. Yoak; Commercial Testing & Engineering, Sophia, WV
T. Todoschuk; Dofasco, Hamilton, Ontario, Canada
M. Snow; Empire Coke Company, Tuscaloosa, AL
J. Serdy; National Steel Inc., Trenton, MI
T. Pike; ABC Coke Company, Birmingham, AL
R. Patasky; Coal Petrographic Associates, Pittsburgh, PA
E.B. McClain; Koppers Industries, Inc., Dolomite, AL
D. Lowenhaupt; Consol Inc., Library, PA
L. Janke; Canmet, Ottawa, Ontario, Canada
T. Hopkins; New Boston Coke Corp., New Boston, OH
D. Chmielewski; Armco Research and Technology, Middletown, OH
G. Canoles; Sloss Industries Coke Plant, Birmingham, AL

REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and 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).
- [3] ASTM D 3174-93 "Test Method for Ash in the Analysis Sample of Coal and Coke from Coal," **Vol. 05.05**, ASTM Book of Standards, West Conshohocken, PA.
- [4] ASTM D 5142-90 "Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures," **Vol. 05.05**, ASTM Book of Standards, West Conshohocken, PA.
- [5] ASTM D 3175-89a "Test Method for Volatile Matter in the Analysis Sample to Coal and Coke," **Vol. 05.05**, ASTM Book of Standards, West Conshohocken, PA.
- [6] ASTM D 3172-89 "Standard Practice for Proximate Analysis of Coal and Coke," **Vol. 05.05**, ASTM Book of Standards, West Conshohocken, PA.

It is the responsibility of users of this SRM to assure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: Phone (301) 975-6776 (select "Certificates"), Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet <http://ts.nist.gov/srm>.