National Institute of Standards & 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 test 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 furnace coke that was ground to pass a 250 μm (60 mesh) sieve, homogenized, and bottled under an argon atmosphere.

Table 1. Certified Value (Mass Fraction)

Sulfur	0.825 % ± 0.016 %

Certified Value: A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1]. The certified value is based on measurements by isotope dilution thermal ionization mass spectrometry (ID-TIMS). The certified value for sulfur is reported in Table l as a mass fraction [2] on a dry-mass basis (see "Instructions for Drying"). 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/JCGM Guide [3] and k is a coverage factor. The value of u_c represents, at the level of one standard deviation, the combined effect of uncertainty components associated with material inhomogeneity and ID-TIMS measurement uncertainty. The expanded uncertainty U is based on a 95 % prediction interval. The coverage factor, $k = 2.20$, is the value from the Student's t-distribution corresponding to 11 degrees of freedom and 95 % confidence.

Reference Values: Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty. Reference values for ash [4,5], volatile matter [5,6], carbon [8] hydrogen [8], and nitrogen [8] are reported in Table 2 as mass fractions [2] on a dry basis (see "Instructions for Drying"). 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 % [7].

Expiration of Certification: The certification of **SRM 2776** is valid, within the measurement uncertainty specified, until **30 June 2027**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of SRM Certification: NIST will monitor 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. Registration (see attached sheet or register online) will facilitate notification.

Overall direction and coordination of the technical measurements leading to certification were performed by R.D. Vocke, Jr. and J.D. Fassett of the NIST Chemical Sciences Division and W.R. Kelly and P.A. Pella, formerly of NIST.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
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Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Metrological Traceability: The measurand for the sulfur certified value in Table 1 is the total mass fraction on a dry-mass basis. The measurands for the reference values in Table 2 are the mass fractions for the elements on a dry-mass basis as determined by the methods described. Metrological traceability is to the International System of Units (SI) derived unit for mass fraction, expressed as a percentage [2].

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

The blast furnace (metallurgical) coke for this SRM was donated by U.S. Steel Clairton Works[(1)] (Carbon, PA).

INSTRUCTIONS FOR HANDLING, STORAGE, AND 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 "Instructions for Drying". To relate analytical determinations to ash and volatile matter reference values, a nominal sample weight of 1 g should be used. To relate analytical determinations to carbon, hydrogen, and nitrogen reference values, a minimum sample mass of 90 mg should be used. When not in use, the 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 on 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 for 1 h, to a constant weight. The weight of the moisture samples began to stabilize after approximately 40 min. 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 measurements obtained by ASTM Subcommittee D05.15 interlaboratory testing ranged from 0.26 % to 0.39 % when determined in a nitrogen atmosphere, and from 0.28 % to 0.46 % when determined in air.

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. The material was then 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 (XRF) 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 R.D. Vocke of the NIST Chemical Sciences Division and W.R. Kelly, formerly of NIST. The X-ray fluorescence homogeneity analysis was performed by A.F. Marlow of the NIST Chemical Sciences Division and P.A. Pella, formerly of NIST.

Reference Values and Uncertainties: The reference values provided for ash and volatile matter are based on results from interlaboratory testing 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/JCGM Guide [3]. The uncertainties for ash and volatile content were calculated as 95 % confidence intervals and represent the combined effects of between-laboratories and within-laboratories components of uncertainty. The percent volatile matter obtained by interlaboratory testing was confirmed by measurements made with platinum crucibles under tightly controlled conditions according to ASTM D 3175-89a [6]. Cooperating analysts and laboratories are listed following the table.

The reference values for C, H, and N are based on measurements performed according to a NIST experimental plan by the LECO[(1)] Corporation (St. Joseph, MI) using ASTM D5373 *Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke* [8]. The uncertainty in

[(1)]Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

the reference values for carbon, hydrogen, and nitrogen are expressed as an expanded uncertainty, $U = ku_c$, calculated according to the methods in the ISO/JCGM Guide [3] for a prediction interval. The quantity u_c represents, at the level of one standard deviation, the combined uncertainty due to the potential effects of random measurement errors in the assessment of carbon, hydrogen, and nitrogen and between bottle variance. The quantity $k = 2$ is the coverage factor used to specify a confidence level of 95 %.

Table 2. Reference Values (Dry-Mass Basis)

Ash [4,5]	8.06 % ± 0.08 %
Volatile Matter [5,6]	0.98 % ± 0.15 %
Carbon [8]	89.15 % ± 1.65 %
Hydrogen [8]	0.26 % ± 0.04 %
Nitrogen [8]	1.21 % ± 0.06 %

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. Patalsky; 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 Plan (Birmingham, AL)

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Apr 2024).

[2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at https://www.nist.gov/pml/special-publication-811 (accessed Apr 2024).

[3] JCGM 100:2008; *Evaluation of Measurement Data – Guide to the Expression of in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Apr 2024) ; see also Taylor, B.N.; 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); available at https://www.nist.gov/pml/nist-technical-note-1297 (accessed Apr 2024).

[4] ASTM D 3174-93, *Test Method for Ash in the Analysis Sample of Coal and Coke from Coal*; Annual Book of ASTM Standards, Vol. 05.05, pp. 291–294 (1996).

[5] ASTM D 5142-90, *Standard Test Methods for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures*; Annual Book of ASTM Standards, Vol. 05.05, pp. 438–442 (1996).

[6] ASTM D 3175-89a; *Test Method for Volatile Matter in the Analysis Sample of Coal and Coke*; Annual Book of ASTM Standards, Vol. 05.05, pp. 295–297 (1996).

[7] ASTM D 3172-89, *Standard Practice for Proximate Analysis of Coal and Coke*; Annual Book of ASTM Standards, Vol. 05.05, p. 291 (1993).

[8] ASTM D 5373-93, *Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke*; Annual Book of ASTM Standards, Vol. 05.05, pp. 453–456 (1996).

Certificate Revision History: 17 April 2024 (Change of expiration date; editorial changes); **23 December 2020** (Change of expiration date, editorial changes); **30 August 2010** (Extension of the certification period, editorial changes); **01 March 2006** (This revision reflects the addition of reference values for C, H, and N); **19 March 1998** (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; e-mail srminfo@nist.gov; or via the Internet at https://www.nist.gov/srm.