NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

Date of Issue:
01 May 2024

Standard Reference Material® 2775
Sulfur in Foundry Coke
CERTIFICATE OF ANALYSIS

Purpose: 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 foundry (metallurgical) coke.

Description: A unit of SRM 2775 consists of 50 g of foundry 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	0.5816 % ± 0.0051 %

Certified Value: A certified value is a value for which NIST has the highest confidence in that all known or suspected sources of bias and imprecision have been considered and any contributions they may make to measurement uncertainty have been quantified and are expressed in the reported uncertainty [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 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, $k = 2.78$, is the value from the student's t-distribution corresponding to four degrees of freedom and 95 % confidence.

Metrological Traceability: The measurand for the certified value in Table 1 is the total mass fraction for the element on a dry-mass basis. Metrological traceability for the certified value is to the SI derived unit for mass fraction, expressed as a percentage [2]. The measurands for the non-certified values in Table A1 are the mass fractions for the elements on a dry-mass basis as determined by the methods described.

Period of Validity: The certified value delivered by **SRM 2775** is valid within the measurement uncertainty specified until **30 June 2027**. The certified value is nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Non-Certified Values: Non-certified values are provided in Appendix A.

Additional Information: Additional information is provided in Appendix B.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Steven J. Choquette, Director
Office of Reference Materials

Certificate Revision History on Page 2

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 non-certified values, a nominal sample mass of 1 g should be used. To relate analytical determinations to carbon, hydrogen, and nitrogen non-certified values, a minimum sample mass of 90 mg should be used.

Storage: When not in use, the SRM must be stored in an air conditioned or similar 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 mass. The mass of the moisture samples began to stabilize after approximately 40 min. The average moisture measured at NIST for SRM 2775 was 0.66 %, with individual determinations ranging from 0.60 % to 0.74 %. SRM 2775 moisture measurements obtained by ASTM Subcommittee D05.15 interlaboratory testing ranged from 0.53 % to 0.67 %, when determined in a nitrogen atmosphere, and from 0.54 % to 0.84 % when determined in an air atmosphere.

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 May 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 May 2024).

[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed May 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 May 2024).

[4] ASTM D 3174-93; *Test Method for Ash in the Analysis Sample of Coal and Coke from Coal*; ASTM Book of 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*; ASTM Book of 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*; ASTM Book of Standards, Vol. 05.05, pp. 295–297 (1996).

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

[8] ASTM D 3172-8; *Standard Practice for Proximate Analysis of Coal and Coke*; ASTM Book of Standards, Vol. 05.05, p. 291 (1993).

Certificate Revision History: 01 May 2024 (Change of period of validity; format changes; editorial changes); **27 April 2021** (Change of expiration date; editorial changes); **30 August 2010** (Extension of the certification period; editorial changes); **10 November 2005** (This revision adds reference values for C, H, and N); **01 May 1997** (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

* * * * * * * End of Certificate of Analysis * * * * * * *

APPENDIX A

Non-Certified Values: Non-certified values are suitable for use in method development, method harmonization, and process control but do not provide metrological traceability to the SI or other higher-order reference system. They are best estimates based on currently available information; however, they do not meet NIST's criteria for certification [1]. Non-certified values for ash [4,5], volatile matter [5,6], carbon [7] hydrogen [7], and nitrogen [7] are reported in Table A1 as mass fractions [2] on a dry basis (see "Instructions for Drying"). The dry fixed carbon, expressed as a percentage, for SRM 2775 can be calculated by subtracting the sum of the dry ash and dry volatile matter from 100 % [8].

The non-certified values provided for ash and volatile matter are based on results from interlaboratory testing done in cooperation with ASTM Subcommittee D05.15 with 12 participating laboratories. The uncertainties in Table A1 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 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 in Appendix B.

The non-certified values for C, H, and N are based on measurements performed according to a NIST experimental plan by the LECO Corporation (St. Joseph, MI), using ASTM D 5373-93, Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke [7]. The uncertainty in the non-certified 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 A1. Non-Certified Values (Dry Basis)

Ash[a,b]	5.77 % ± 0.05 %
Volatile Matter[b,c]	1.31 % ± 0.20 %
Carbon[d]	91.34 % ± 0.49 %
Hydrogen[d]	0.41 % ± 0.06 %
Nitrogen[d]	1.16 % ± 0.05 %

[a] Measurements are based on test methods found in reference 4.
[b] Measurements are based on test methods found in reference 5.
[c] Measurements are based on test methods found in reference 6.
[d] Measurements are based on test methods found in reference 7.

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Appendix and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

* * * * * * * * * * * End of Appendix A * * * * * * * * * * *

APPENDIX B

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

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.

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

The foundry (metallurgical) coke for this SRM was donated by ABC COKE, Birmingham, AL.

Preparation: Foundry coke was collected and crushed to a nominal 2.36 mm (8 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. With the exception of barium, no significant bottle-to-bottle differences were found for any of the elements tested. For barium, a small component of bottle-to-bottle variability was detected. The standard deviation corresponding to this component of variation is estimated to be less than 0.84 % relative, based on a 95 % confidence interval for the standard deviation.

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)

* * * * * * * * * * End of Appendix B * * * * * * * * * *