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

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Standard Reference Material® 2719
Calcined Petroleum Coke
CERTIFICATE OF ANALYSIS

Purpose: This Standard Reference Material (SRM) is intended primarily for use in the calibration of apparatus and the evaluation of techniques employed in the analysis of calcined petroleum coke and other materials with a similar matrix.

Description: A unit of SRM 2719 consists of 50 g of calcined petroleum coke ground to pass a 250 µm (60 mesh) sieve, homogenized, and bottled under an argon atmosphere.

Certified Values: 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 values, expressed as mass fractions [2] on a dry basis (see "Instructions for Drying"), are provided in Table 1. The certified values for aluminum, calcium, iron, nickel, and vanadium are based on two independent NIST methods. The certified value for sulfur is based on a single NIST primary method.

Table 1. Certified Values (Dry Basis)

Elements	Mass Fraction (mg/kg)
Aluminum	58.9 ± 5.7
Calcium	57.7 ± 4.4
Iron	201.6 ± 5.4
Nickel	204 ± 12
Sulfur	8877 ± 10
Vanadium	58.6 ± 3.4

Metrological Traceability: The measurands for the certified values in Table 1 are the total mass fractions for the elements on a dry-mass basis. Metrological traceability for the certified values is to the SI derived unit for mass fraction, expressed as milligrams per kilogram [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.

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

Additional Information: Values of potential interest to users and additional information are provided in Appendix B.

Period of Validity: The certified values delivered by **SRM 2719** are valid within the measurement uncertainty specified until **30 June 2027**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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
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Use: The unit should be thoroughly mixed by rotating the bottle before sampling. A minimum sample mass of 250 mg should be used for analytical determinations to be related to the certified values and to the non-certified values for cobalt and sodium. To relate analytical determinations to carbon, hydrogen, and nitrogen non-certified values, a minimum sample mass of 90 mg should be used.

Storage: The SRM should be stored in its original, tightly sealed bottle away from sunlight and intense sources of radiation.

Instructions for Drying: In order for users to directly relate their measurements to the certified and non-certified values, drying corrections should be measured and applied at the time of analysis. The correction is determined by drying separate 1 g samples in a nitrogen atmosphere at 107 °C ± 3 °C to a constant mass. Air is also an acceptable carrier gas for drying this material. The average mass loss measured at NIST for SRM 2719 was 0.07 % (standard deviation = 0.04 %, $n = 6$).

Certified Values and Uncertainties: Certification analyses for aluminum, calcium, iron, nickel, vanadium, and sulfur were performed by NIST. The certified values for aluminum, calcium, iron, nickel, and vanadium are the equally weighted mean of two independent analytical methods. Aluminum, calcium, iron, nickel, and vanadium values are based on inductively coupled plasma-optical emission spectrometry (ICP-OES) and instrumental neutron activation analysis (INAA). The certified value for sulfur is based on a single NIST primary method, isotope dilution-thermal ionization mass spectrometry (ID-TIMS) [3].

The uncertainty in the mass fractions certified by two NIST methods is calculated as $U = ku_c + B$, as described by Schiller and Eberhardt [4]. The quantity u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [5], which accounts for the combined effect of the variance for the two methods at one standard deviation. The coverage factor k is determined from the Student's t-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte. B is a bias adjustment for the difference between methods, which is the maximum difference between the certified value and the method means [4].

The uncertainty in the value certified by a NIST primary method is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO/JCGM Guide [5]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with the measurement and material inhomogeneity, and k is a coverage factor corresponding to 95 % confidence.

REFERENCES

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[3] Kelly, W.R.; Paulsen, P.J.; Murphy, K.E.; Vocke, R.D.; Chen, L.-T.; *Determination of Sulfur in Fossil Fuels by Isotope Dilution Thermal Ionization Mass Spectrometry*; Anal. Chem., Vol. 66, p. 2505−2513 (1994).

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[6] 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).

[7] 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).

[8] 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).

[9] ASTM D 4421-89; *Test Method for Volatile Matter in Petroleum Coke*; ASTM Book of Standards, Vol. 05.02.
[10] ASTM D 2015-93; *Test Method for Gross Calorific Value of Coal and Coke by Adiabatic Bomb*; ASTM Book of Standards, Vol. 05.05.
[11] ASTM D 3286-91a; *Test Method for Gross Calorific Value of Coal and Coke by the Isoperibol Bomb Calorimeter*; ASTM Book of Standards, Vol. 05.05, pp. 317–325 (1996).

Certificate Revision History: 01 May 2024 (Change of period of validity; updated format; editorial changes); **28 April 2021** (Change of expiration date; editorial changes); **30 August 2010** (Extension of the certification period; editorial changes); **01 March 2006** (This revision reflects a change from information to reference value for C, H, and N, and an extension of the certification period.); **20 May 2002** (Note regarding volatile matter and disclaimer added); **15 July 1999** (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.

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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]. The non-certified values, expressed as a mass fraction [2] on a dry basis (see "Instructions for Drying") are provided in Table A1. The non-certified values for cobalt and sodium are based on a single NIST method. The values for carbon, hydrogen, and nitrogen are based on a single method performed by the LECO Corporation (St. Joseph, MI) and corroborated by results from the interlaboratory analysis study administrated on behalf of NIST by Laboratory Quality Services International.

Table A1. Non-Certified Values (Dry Basis)

Elements	Mass Fraction		
Carbon[(a)] (C)	97.06 %	±	0.92 %
Cobalt (Co)	18.6 mg/kg	±	0.5 mg/kg
Hydrogen[(a)] (H)	0.16 %	±	0.09 %
Nitrogen[(a)] (N)	1.17 %	±	0.05 %
Sodium (Na)	15.1 mg/kg	±	0.9 mg/kg

[(a)] Measurements are based on test methods found in reference 5.

Non-Certified Value and Uncertainty: The non-certified values for cobalt and sodium were determined by INAA. The uncertainty in the non-certified values is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO/JCGM Guide [5]. The expanded uncertainty is calculated as $U = ku_c$, where u_c represents, at the level of one standard deviation, the combined effect of uncertainty components associated with the measurement uncertainty and material inhomogeneity, and k is a coverage factor. The coverage factor k is determined from the Student's t-distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each analyte.

The values for carbon, hydrogen, and nitrogen are based on measurements performed according to a NIST experimental plan by the LECO Corporation using ASTM D 5373-93, *Standard Test Methods for Instrumental Determination of Carbon, Hydrogen, and Nitrogen in Laboratory Samples of Coal and Coke* [6]. These results were corroborated by an SRM 2719 interlaboratory analysis study. 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 [5] 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 %.

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).

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APPENDIX B

Values of Potential Interest: Values of potential interest are reported in Table B1. The silicon, ash content, and gross caloric values are based on an interlaboratory analysis study for this SRM, administrated on behalf of NIST by Laboratory Quality Services International. These values cannot be used to establish traceability.

Table B1. Values of Potential Interest (Dry Basis)

Silicon	138 mg/kg
Ash[(a)]	0.12 %
Volatile Matter[(b,c)]	0.54 %
Gross Calorific Value[(d)]	32.90 $Mj \cdot kg^{-1}$ (14 146 $Btu_{th} \cdot lb^{-1}$)

[(a)] Measurements are based on test methods found in references 7 and 8.
[(b)] Measurements are based on test methods found in references 8 and 9.
[(c)] Samples having a thermal history above 600 °C, such as SRM 2719, are excluded from the scope of ASTM D 4421-89 [9]. The volatile matter reported may include a loss of mass associated with sample oxidation.
[(d)] Measurements are based on test methods found in references 10 and 11.

The values of potential interest given in Table B1 for silicon, ash, volatile matter, and gross caloric value are based on an interlaboratory analysis study for SRM 2719, administrated on behalf of NIST by Laboratory Quality Services International. Values of potential interest are provided without uncertainty for information purposes only.

Source and Preparation of Material: The calcined petroleum coke for this SRM was donated by VENCO (Moundsville, WV). The collection of the approximately 240 kg of calcined petroleum coke was under the direction of R.B. Murzyn, Environmental Coordinator, VENCO.

The gross sample was jaw crushed and subsequently pulverized using ceramic plates to pass a 250 µm (60 mesh) screen. The entire lot was then divided using the spinning riffle technique into 48 portions. Sixteen portions were subdivided by the spinning riffle technique into bottles, which were subsequently sealed under an argon atmosphere. The balance of the lot is being retained in long term storage at NIST under an argon atmosphere.

Overall direction and coordination of the technical measurements leading to certification were performed by J.D. Fassett formerly of the NIST Materials Measurement Science Division and R.L. Watters, Jr., of the NIST Office of Reference Materials.

INAA was performed by D.A. Becker and R. Demiralp, formerly of NIST. ID-TIMS [3] was performed by J.L. Mann and R.D. Vocke of the NIST Chemical Sciences Division, and W.R. Kelly, formerly of NIST.

Statistical analysis of the certification data was performed by D.D. Leber of the NIST Statistical Engineering Division and L.M. Gill, formerly of NIST.

Homogeneity: No evidence of inhomogeneity was noted during certification from replicate measurements using the minimum sample size.

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