National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2718

Green Petroleum Coke

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 green (raw) petroleum coke and other materials with a similar matrix. SRM 2718 consists of 50 g of green petroleum coke ground to pass a 250 µm (60 mesh) sieve, homogenized, and bottled under an argon atmosphere.

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

Reference Values: The reference values, expressed as a mass fraction on a dry basis, are provided in Table 2. The reference values for Co and Na are based on a single NIST method. The values for calcium (C), hydrogen (H), and nitrogen (N) are based on a single method performed by LECO Corporation (St. Joseph, MI) and corroborated by results from the interlaboratory analysis study administrated on behalf of NIST by Laboratory Quality Services International. 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.

Information Values: Information values are reported in Table 3. The silicon, ash content, volatile matter, and gross caloric values are based on an interlaboratory analysis study for this SRM, administrated on behalf of NIST by Laboratory Quality Services International. Information values are provided for information purposes only.

Expiration of Certification: The certification of **SRM 2718** is valid, within the measurement uncertainty specified, until **31 December 2020**, 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) will facilitate notification.

The overall direction and coordination of the technical measurements leading to certification were performed by J.D. Fassett and R.L. Watters, Jr. of the NIST Analytical Chemistry Division.

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

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 30 August 2010
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

INSTRUCTIONS FOR HANDLING, STORAGE, AND 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 reference values for cobalt and sodium. To relate analytical determinations to carbon, hydrogen, and nitrogen reference values, a minimum sample mass of 90 mg should be use. 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 reference 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° to a constant mass. Air is also an acceptable carrier gas for drying this material. The average mass loss measured at NIST for SRM 2718 was 0.40 % (standard deviation = 0.03 %, n = 6).

PREPARATION, HOMOGENIETY AND ANALYSIS

Source and Preparation of Material:[1] The green petroleum coke for this SRM was donated by Lake Charles Carbon, a division of Reynolds Metals Company (Lake Charles, LA). The collection of the approximately 230 kg of green petroleum coke was under the direction of R. Jones and R. Morris of Lake Charles Carbon.

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.

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

Table 1. Certified Values (Dry basis)

Elements	Mass Fraction (mg/kg)		
Aluminum	16.5	±	1.8
Calcium	174	±	15
Iron	290	±	14
Nickel	139.1	±	5.8
Sulfur	47 030	±	79
Vanadium	302	±	10

Certified Values and Uncertainties: Certification analyses for Al, Ca, Fe, Ni, V, and S were performed by the NIST Analytical Chemistry Division. The certified values for Al, Ca, Fe, Ni, and V are the equally weighted mean of two independent analytical methods. Al and V values are based on inductively coupled plasma optical emission spectrometry (ICP-OES) performed by L.J. Wood, and instrumental neutron activation analysis (INAA) performed by L. Tandon and D.A. Becker. The Ca value is based on ICP-OES, performed by L.J. Wood and M.S. Epstein, and INAA, performed by L. Tandon and D.A. Becker. Fe and Ni values are based on ICP-OES performed by L.J. Wood and M.S. Epstein, and INAA performed by R. Demiralp. The certified value for sulfur is based on a single NIST primary method, isotope dilution thermal ionization mass spectrometry (ID-TIMS) [2] performed by W.R. Kelly and R.D. Vocke.

The uncertainty in the concentrations certified by two NIST methods is calculated as $U = ku_c + B$, as described by Schiller and Eberhardt [3]. The quantity u_c, is the combined standard uncertainty calculated according to ISO Guide [4], 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

[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.

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 [3].

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 Guide. 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 uncertainty and material inhomogeneity, and *k* is a coverage factor corresponding to 95 % confidence.

Table 2. Reference Values (Dry basis)

Elements	Mass Fraction		
Carbon [5]	88.99 %	±	0.53 %
Cobalt	5.79 mg/kg	±	0.24 mg/kg
Hydrogen [5]	3.47 %	±	0.08 %
Nitrogen [5]	1.23 %	±	0.04 %
Sodium	88.6 mg/kg	±	2.8 mg/kg

Reference Value and Uncertainty: The reference value for Co was determined from (INAA) performed by R. Demiralp. The reference value for Na is based on INAA performed by D.A. Becker. The uncertainty in the cobalt and sodium reference values is expressed as an expanded uncertainty, *U*, and is calculated according to the method described in the ISO Guide [4]. 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* [5]. These results were corroborated by the SRM 2718 interlaboratory analysis study. The uncertainty in 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 Guide [4] 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 %.

Information Values: The information values given in Table 3 for silicon, ash, volatile matter and gross caloric value are based on an interlaboratory analysis study for SRM 2718, administrated on behalf of NIST by Laboratory Quality Services International. Information values are provided without uncertainty for information purposes only

Table 3. Information Values (Dry basis)

Silicon	63.00 mg/kg
Ash [6,7]	0.18 %
Volatile Matter [7,8]	10.6 %
Gross Calorific Value [9,10]	35.76 $Mj \bullet kg^{-1}$ (15 376 $Btu_{th} \bullet lb^{-1}$)

REFERENCES

[1] 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 http://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm (accessed Aug 2010).

[2] 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 (1994).

[3] Schiller, S.B.; Eberhardt, K.R.; *Combining Data from Independent Analysis Methods*; Spectrochemical Acta, Vol. 46B No. 12, pp. 1607–1613 (1991).

[4] 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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Aug 2010); 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 http://www.nist.gov/physlab/pubs/index.cfm (accessed Aug 2010).

[5] 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).

[6] 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).

[7] 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).

[8] 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).

[9] ASTM D 2015-93; *Test Method for Gross Calorific Value of Coal and Coke by Adiabatic Bomb*; Annual Book of ASTM Standards, Vol. 05.05 (1993).

[10] ASTM D 3286-91a; *Test Method for Gross Calorific Value of Coal and Coke by the Isoperibol Bomb Calorimeter*; Annual Book of ASTM Standards, Vol. 05.05, pp. 317–325 (1996).

Certificate Revision History: 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); **15 July 1999** (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; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.