National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1635

Trace Elements in Coal
(Subbituminous)

This Standard Reference Material (SRM) is intended for use in the calibration of apparatus and the evaluation of techniques employed in the analysis of trace elements in coal and similar materials. A unit of this SRM consists of 75 g of finely powdered coal ground to pass a 230 μm sieve (No. 65) and homogenized.

Certified Values: The certified values for SRM 1635 are given on a dry mass basis (see Drying Instructions) in Table 1. All values are reported as mass fractions [1], on a dry mass basis and are based on measurements using a sample mass of at least 250 mg. The certified values are based on agreement of results obtained using two or more independent methods for the analysis with the exception of sulfur which is based on a single NIST primary method, isotope dilution thermal ionization mass spectrometry (IDTIMS). Analytical methods used for the certification of this SRM are given in Table 3.

Uncertainties: The estimated uncertainty is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 250 mg or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the determination of most constituents.) The uncertainty for fluorine is based on a 95 % confidence interval for the mean [2]. The uncertainty in the certified value for sulfur is expressed as an expanded uncertainty, U, and is calculated according to the method in the ISO Guide [3]. The expanded uncertainty is calulated 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.

Information Values: Information values for additional elements are given in Table 2. A NIST information value is a value that may be of information and use to the SRM user, but insufficient information is available to assess its accuracy and associated uncertainty.

Expiration of Certification: The certification of SRM 1635 is valid, within the measurement uncertainties specified, until **30 October 2008**, provided the SRM is handled in accordance with the instructions given in this certificate. The certification is not valid if the SRM is damaged, contaminated, or modified.

Storage and Stability: When not in use, the material should be kept in a tightly sealed bottle and stored in a cool, dark place. Long-term stability of this SRM has not been rigorously established. NIST will continue to monitor this material and any substantive changes will be reported to purchasers.

The overall direction and coordination of the analytical measurements leading to certification were performed in the NIST Analytical Chemistry Division under the chairmanship of L.J. Moore. Coordination of fluorine measurements was performed by W.P. Huff, Chairman of Task Group D05.21.02, Ultimate Analysis on ASTM Committee D05 on Coal and Coke.

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

Willie E. May, Chief
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The homogeneity measurements were performed by R.R. Greenberg of the NIST Analytical Chemistry Division. Certification analyses for all elements except fluorine were made by T.J. Brady, B.I. Diamondstone, L.P. Dunstan, M.S. Epstein, M. Gallorini, E.L. Garner, T.E. Gills, J.W. Gramlich, R.R. Greenberg, S.H. Harrison, G.M. Hyde, G.J. Lutz, L.A. Machlan, E.J. Maienthal, J.D. Messman, T.J. Murphy, and T.C. Rains of the NIST Analytical Chemistry Division. The IDTIMS measurements for sulfur were performed by W.R. Kelly, J.C. Mann, and R.D. Vocke.

Fluorine measurements were performed by members of the ASTM Committee D05 Fluorine Task Group: Norfolk Testing Laboratories, W.P. Huff; Alberta Research Council, A. Iachelli; University of Kentucky, H. Francis, A.S. Wong, and J.D. Robertson; CANMET EMR, L. Janke and R. Dureau; CONSOL INC., L.W. Rosendale; Illinois State Geological Survey, C. Chaven.

Statistical analysis of the fluorine data was performed by S.B. Schiller of the NIST Statistical Engineering Division.

Source and Preparation of Material: This SRM was prepared from one lot of subbituminous coal from the Eagle Mine of the Imperial Coal Company, Erie, CO. This mine produces subbituminous coal with a sulfur content of approximately 0.3 % (dry mass basis). The material was ground and sieved through a 230 μm sieve (No. 65) by the Colorado School of Mines Research Institute. The material was then blended thoroughly in a V-type blender.

Samples for homogeneity testing were taken from the top, middle, and bottom of three bulk containers of blended coal, and analyzed using neutron activation analysis for sodium, scandium, chromium, iron, cobalt, lanthanum, cerium, and thorium. Replicate analyses of 250 mg samples indicated a homogeneity for these elements of ± 2.5 % (relative) except for chromium, which was homogeneous, within counting statistics, at ± 6 %.

Use: The bottle unit should be thoroughly mixed by rotating the bottle before sampling. A minimum sample mass of 250 mg (dry mass - see Instructions for Drying) should be used in order for analytical determinations to be related to the certified values provided.

Drying Instructions: The recommended procedures for drying are either vacuum drying at ambient temperature for 24 hours, freeze drying at room temperature or drying in a nitrogen atmosphere at 107 °C ± 3 °C to a constant mass. The moisture content of this material is approximately 17 %. Because of this moisture level, it is recommended that individual 250 mg samples be dried immediately before use or a separate 1 g sample, as received, be dried to obtain a correction factor for moisture. Drying of large samples may result in a violent discharge of water vapor and resultant loss of sample.

Table 1. Certified Mass Fractions for Selected Elements

Minor Constituents		Trace Elements	
Element	Mass Fractions (in %)	Element	Mass Fractions (in mg/kg)
Iron	0.239 ± 0.005	Arsenic	0.42 ± 0.15
Sulfur	0.3616 ± 0.0017	Cadmium	0.03 ± 0.01
		Chromium	2.5 ± 0.3
		Copper	3.6 ± 0.3
		Fluorine	25.9 ± 3.3
		Lead	1.9 ± 0.2
		Manganese	21.4 ± 1.5
		Nickel	1.74 ± 0.10
		Selenium	0.9 ± 0.3
		Thorium	0.62 ± 0.04
		Uranium	0.24 ± 0.02
		Vanadium	5.2 ± 0.5
		Zinc	4.7 ± 0.5

The noncertified values given in Table 2 are provided for information only as additional information on the matrix. These measurements did not meet NIST criteria for use as certified values. Therefore, they are not to be used for calibration or method evaluation purposes.

Table 2. Information Mass Fractions

Minor Constituents		Trace Elements	
Element	Mass Fraction (in %)	Element	Mass Fraction (in mg/kg)
Aluminum	0.32	Antimony	0.14
Sodium	0.24	Cerium	3.6
Titanium	0.02	Cobalt	0.65
		Europium	0.06
		Gallium	1.05
		Hafnium	0.29
		Mercury	0.02
		Scandium	0.63

Ash, Mass Fraction (in %) 4.6

Table 3. Methods Used for the Certification of SRM 1635

Element	Method
Arsenic	AAS, IPAA
Cadmium	IDTIMS, POLAR, INAA
Chromium	IDMS, INAA
Cobalt	AAS, IDTIMS, INAA
Copper	AAS, IDTIMS, INAA
Fluorine	BC-ISE, Fus-ISE, PI-GES, PYRO-IC-ISE
Iron	IDTIMS, POLAR, INAA, COLOR
Lead	IDTIMS, POLAR
Manganese	AAS, INAA
Nickel	IDTIMS, POLAR
Selenium	AAS, INAA
Sulfur	IDTIMS
Thorium	IDTIMS, INAA
Uranium	IDTIMS
Vanadium	IDTIMS, FES

Methods

AAS	Atomic absorption spectrometry
IPAA	Instrumental photon activation analysis
IDTIMS	Isotope dilution thermal ionization mass spectrometry
POLAR	Polarography
INAA	Instrumental neutron activation analysis
COLOR	Spectrophotometry/colorimetry
FES	Flame emission spectrometry
BC-ISE	Bomb combustion/ion selective electrode [2]
Fus-ISE	Fusion/ion selective electrode
PI-GES	Proton-induced gamma emission spectrometry
PYRO-IC-ISE	Pyrohydrolysis/ion chromagraphy/ion selective electrode

REFERENCES

[1] Taylor, B.N., "Guide for the Use of International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).

[2] ASTM D 3761-91, Test Method for Total Fluorine in Coal by the Oxygen Bomb Combustion/Ion Selective Electrode Method, ASTM Annual Book of Standards, **Vol.** 05.05, (1993).

[3] *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); available at http://physics.nist.gov/Pubs/.

Certificate Revision History: 9 May 2000 (Editorial change); 12 April 2000 (Certified sulfur value revised; ash information value and expiration date added); 24 October 1995 (Mercury info value added); 18 July 1995 (Certified fluorine added); 22 August 1979 (Certified iron and sulfur added); 23 January 1978 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (30l) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the internet: http://www.nist.gov/srm.