National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2691

Coal Fly Ash

This Standard Reference Material (SRM) is intended for use in the evaluation of analytical methods used for the classification of coal fly ash and for the determination of constituent elements in coal fly ash or materials of a similar matrix. A unit of SRM 2691 consists of three 10 g hermetically sealed glass vials of fly ash pulverized to less than 150 μm particle size and blended to a high degree of homogeneity.

Certified Mass Fraction Values: The certified values for major and minor constituent elements 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 taken into account [1]. The uncertainty is expressed as two standard deviations of the unweighted mean across methods on which the certified value is based. Values reported on an as-received (not dry-mass) basis in mass fraction units [2].

Reference Mass Fraction Value: A reference value for residue on a 45 μm electroformed sieve is provided in Table 2. A NIST reference value is a non-certified value that is the best estimate of the true value; however, the value does not meet NIST criteria for certification and is provided with an associated uncertainty that may not include all sources of uncertainty [1].

Information Values: Information values are provided in Table 3. A NIST information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value; therefore, no uncertainty is provided [1]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM 2691** is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the 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.

Coordination of the technical measurements leading to the certification of this SRM was performed by J.R. DeVoe formerly of NIST.

The preparation of the SRM and the coordination of the collaborating laboratory's technical measurements leading to the certification were performed under the direction of H.M. Kanare with assistance from C.M. Wilk, both of Construction Technology Laboratories, Portland Cement Association (Skokie, IL).

Analytical measurements at NIST were performed by M.S. Epstein and S.E. Long of the NIST Chemical Sciences Division; and D.A. Becker, E.S. Beary and W.R. Kelly formerly of NIST. Analyses at Construction Technology Laboratories were performed by R. Crow, J.B. Delles, H.M. Kanare, H. Love, A.H. Malen, and C.M. Wilk.

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Original statistical analysis was performed by R.C. Paule and reanalysis was performed by S.D. Leigh both formerly of the NIST Statistical Engineering Division.

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling and Storage: The composition and specific surface area of the ash may change on exposure to moisture in the air. Therefore, the SRM should be used as soon as possible after opening. If not used immediately, it should be protected from atmospheric moisture by transferring the opened SRM to a tightly closed vial and storing in a desiccator.

Use: To open a vial containing the SRM, make a deep scratch with a file 0.6 cm (1/4 inch) from the bottom. Invert the vial and press a red-hot file point against the scratch to cause a circumferential crack to form. To be related to the certified values in this certificate, a minimum 500 mg test portion should be used for analytical determinations, following precautions to prevent absorption of moisture.

SOURCE, PREPARATION, AND ANALYSIS[(1)]

Source and Preparation of Materials: The fly ash used for preparation of this material was obtained from a coal-fired power plant (Iatan Power Station, Iatan, MO, by Kansas City Power and Light Co., Kansas City, MO) and is a product of sub-bituminous (low sulfur) coal that was mined in the Arco Black Thunder Mine, Powder River Basin, Gillette, WY. The fly ash is ASTM C618-85 Class C [3]. Typical portions of such coals include mass fractions of 27.6 % moisture, 4.8 % ash, 0.3 % sulfur, and an energy content of 20 470 kJ/kg (8,800 Btu/lb). The fly ash was size-classified using a Vortex C-13 air classifier and particles greater than 45 μm were removed for grinding. The coarse material consisted mostly of quartz and partially burned fragments. Once ground to pass a 150 μm (No. 100) sieve, this material was blended back into the rest of the fly ash, and the entire lot of material was homogenized in a ribbon blender, hermetically sealed in glass vials, and packaged. This process narrowed the particle size distribution so that the fly ash could be blended to a high degree of homogeneity and the tendency toward segregation would be minimized. The packaging operations were performed in a temperature-and-humidity controlled atmosphere to minimize moisture differences between samples.

Certification Analyses: The fly ash was analyzed by a number of techniques at NIST and by a collaborating laboratory. The certified values, except for that for Hg, are based on measurements using two or more independent analytical techniques and/or methods. The certified mass fraction for Hg is an unweighted mean value based on results from a single NIST method for which a complete evaluation of all sources of uncertainty has been performed. The uncertainty of the certified value is a two-sided 95 % confidence interval for the mean (coverage factor, $k = 2$).

A list of analytical methods used for value assignment is provided in Table 4. For user convenience, gravimetric multipliers of the constituent elements to oxides are given in Table 5.

The reference value for the residue retained on a No. 325 U.S. Standard Sieve is based on an ideal 45 μm sieve. The value was calculated from a least-squares straight line fit of mean values of measurements made using NIST-calibrated sieves with average sieve openings of 42.5 μm, 44.0 μm, 45.5 μm, 46.0 μm, 46.5 μm, and 47.0 μm. The residue data were obtained from tests performed in accordance with ASTM C430-83 [4], Standard Test Method for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve, as specified in ASTM C311-85 [5], Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete.

Homogeneity Testing: Material taken from stratified randomly selected vials was analyzed using X-ray fluorescence spectrometry (XRF). Twenty-five vials were opened and two test portions of 0.5 g were taken from each and fused into discs for XRF analyses. For the elements measured (Al, Ca, Fe, and Si), no evidence of sample heterogeneity was observed.

[(1)] Certain commercial equipment, instruments or materials are identified in this certificate 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.

Table 1. Certified Mass Fraction Values for SRM 2691 Coal Fly Ash

Constituent Element	Mass Fraction[a] (%)		
Aluminum (Al)	9.81	±	0.39
Calcium (Ca)	18.45	±	0.32
Iron (Fe)	4.42	±	0.03
Magnesium (Mg)	3.12	±	0.08
Phosphorus (P)	0.51	±	0.02
Potassium (K)	0.34	±	0.01
Silicon (Si)	16.83	±	0.12
Sodium (Na)	1.09	±	0.05
Sulfur (S)	0.83	±	0.05
Titanium (Ti)	0.90	±	0.02
Constituent Element	**Mass Fraction (mg/kg)**		
Mercury (Hg)	0.0578	±	0.0043

[a] The measurand is the total mass fraction for each element. The result, calculated as a mass fraction and expressed as a percentage, is traceable to NIST's realization of the SI unit of the kilogram [2].

Table 2. Reference Mass Fraction Value for SRM 2689 Coal Fly Ash

Property	Mass Fraction[a] (%)		
Residue on a 45 µm electroformed sieve (ASTM Standard Test Method C430-83) [4]	10.5	±	0.5

[a] The measurand is the total mass fraction based on the indicated test method. The result, calculated as a mass fraction and expressed as a percentage, is traceable to NIST's realization of the SI unit of the kilogram [2].

Table 3. Information Values for SRM 2691 Coal Fly Ash

Constituent Element	Mass Fraction (mg/kg)
Antimony (Sb)	3
Arsenic (As)	30
Barium (Ba)	5900
Beryllium (Be)	8
Cobalt (Co)	26
Chromium (Cr)	68
Cesium (Cs)	1
Europium (Eu)	2
Hafnium (Hf)	10
Lead (Pb)	29
Manganese (Mn)	200
Nickel (Ni)	53
Scandium (Sc)	24
Selenium (Se)	17
Strontium (Sr)	2700
Thorium (Th)	26
Zinc (Zn)	120
LOI 750 °C[a]	0.23
Moisture (110 °C)[a]	0.08

[a] Loss on Ignition (LOI) determined in accordance with ASTM Standard Test Method C311-85 [5].

Table 4. Analytical Methods Used for Value Assignment

Element	Methods
Aluminum	AAS, INAA, XRF
Antimony	AAS
Arsenic	AAS
Barium	AAS, INAA
Beryllium	AAS
Calcium	AAS, INAA, TITR, XRF
Cobalt	INAA
Chromium	AAS, INAA
Cesium	INAA
Europium	INAA
Hafnium	INAA
Iron	AAS, TITR, XRF
Lead	AAS
Magnesium	AAS, XRF
Manganese	DCP, INAA, XRF
Mercury	ID-CV-ICPMS
Nickel	AAS
Phosphorus	DCP, COLOR, XRF
Potassium	AAS, INAA, XRF
Scandium	INAA
Selenium	AAS
Silicon	AAS, DCP, GRAV, XRF
Sodium	AAS, INAA, XRF
Strontium	AAS, INAA, XRF
Sulfur	GRAV, COLOR, XRF
Thorium	INAA
Titanium	AAS, INAA, XRF
Zinc	INAA
LOI	GRAV
Moisture	GRAV
Sieve Residue	ASTM

Key to Analytical Methods

AAS	Atomic absorption spectrometry
DCP	Direct-current plasma optical emission spectrometry
GRAV	Gravimetry
ID-CV-ICPMS	Isotope dilution-cold vapor-inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
TITR	Titrimetry
COLOR	Colorimetry
XRF	X-ray fluorescence spectrometry
ASTM	ASTM Test Method C430-83 [4] for Fineness of Hydraulic Cement by the 45 μm (No. 325) Sieve as specified in ASTM C311-85 [5], Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete

Table 5. Multipliers for Element to Oxide Conversion [6]

Constituent Element	Oxide Sought	Multiplier
Aluminum (Al)	Al_2O_3	1.889
Barium (Ba)	BaO	1.117
Calcium (Ca)	CaO	1.399
Iron (Fe)	Fe_2O_3	1.430
Potassium (K)	K_2O	1.205
Magnesium (Mg)	MgO	1.658
Manganese (Mn)	MnO	1.291
Sodium (Na)	Na_2O	1.348
Phosphorus (P)	P_2O_5	2.291
Silicon (Si)	SiO_2	2.139
Strontium (Sr)	SrO	1.183
Titanium (Ti)	TiO_2	1.668

REFERENCES

[1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/publications.cfm (accessed July 2015).

[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 http://www.nist.gov/pml/pubs/sp811/indexfull.cfm (accessed July 2015).

[3] ASTM C618-85; *Standard Specifications for Fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Portland Cement Concrete*; Annual Book of ASTM Standards, Vol. 04.02 (1985).

[4] ASTM C430-83; *Test Method for Fineness of Hydraulic Cement by the 45-µm (No. 325) Sieve*; Annual Book of ASTM Standards, Vol. 04.01 (1984).

[5] ASTM C311-85; *Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use in Portland-Cement Concrete*; Annual Book of ASTM Standards, Vol. 04.02 (1985).

[6] Wieser, M.E; Holden, N.; Coplen, T.B; Böhlke, J.K.; Berglund, M.; Brand, W.A.; De Bièvre, P.; Gröning, M.; Loss, R.D.; Meija, J.; Hirata, T.; Prohaska, T.; Schoenberg, R.; O'Connor, G.; Walczyk, T.; Yoneda, S.; Zhu, X.; *Atomic Weights of the Elements* ; Pure Appl. Chem., Vol. 85, pp. 1047–1078 (2013).

Certificate Revision History: 22 July 2015 (Updated multipliers for element-to-oxide conversion; corrected typographical error in Table 5 for the strontium multiplier; updated particle size; editorial changes); 10 August 2012 (Removed cadmium information value; updated information value for mercury; editorial changes); 20 December 1993 (Revision of certificate); 01 October 1986 (Original certification 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) 948-3730; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.