U. S. Department of Commerce
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National Bureau of Standards
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National Bureau of Standards Certificate of Analysis

Standard Reference Materials 2689, 2690, and 2691

Coal Fly Ashes

These Standard Reference Materials (SRM's) are intended for use in the evaluation of analytical methods and techniques used for the classification of coal fly ash and for the determination of constituent elements in coal fly ash or materials with a similar matrix. Each SRM consists of 3 hermetically sealed glass vials of fly ash, which has been size classified and blended to a high degree of homogeneity. Each vial contains 10 grams of fly ash.

The three different fly ash SRM's were chosen to cover a wide range of chemical and mineralogical compositions. The certified values for the constituent elements and the sieve residue are given in Table 1. For user convenience, gravimetric multipliers for the conversion of the constituent elements to oxides are given in Table 2. The certified values, except for the sieve residue, are based on measurements using two or more independent reliable analytical techniques and/or methods. Noncertified values (in parentheses), also given in Table 1, are provided for information only.

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

Information on the sources of the fly ashes and a description of the coals from which they were derived is given in Table 3. A list of analytical techniques and methods that were used for the certification of these SRM's is given in Table 4.

Notice to Users: These SRM's are sold individually rather than in sets; however, only one Certificate of Analysis is provided. Therefore, the user must be careful to use the data specific to the SRM being used.

Use: Before certification, these fly ash SRM's were homogenized and hermetically sealed in glass vials to minimize any changes in chemical and physical properties. Therefore, the SRM's should be used as received.

To open a vial containing the SRM, make a deep scratch with a file 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.

The composition and specific surface area of the ash may change on being exposed to the 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 stoppered vial and storing in a desiccator.

The preparation of these SRM's and the coordination of cooperative technical measurements leading to certification were performed under the direction of Howard M. Kanare with assistance from Charles M. Wilk, both of Construction Technology Laboratories, Portland Cement Association, Skokie, Illinois.

The overall direction and coordination of the analytical measurements leading to certification were performed in the Inorganic Analytical Research Division, J.R. DeVoe, Chief.

The statistical analysis of the certification data was performed by R.C. Paule of the NBS National Measurement Laboratory.

The technical and support aspects involved in the preparation, certification, and issuance of these Standard Reference Materials were coordinated through the Office of Standard Reference Materials by T.E. Gills.

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Stanley D. Rasberry, Chief
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Table 1

Certified Concentrations of Constituent Elements in SRM's 2689, 2690, and 2691

Constituent Element	2689 (Weight Percent)	2690 (Weight Percent)	2691 (Weight Percent)
Aluminum	12.94 ± 0.21	12.35 ± 0.28	9.81 ± 0.39
Barium	(0.08)	(0.65)	(0.66)
Calcium	2.18 ± 0.06	5.71 ± 0.13	18.45 ± 0.32
Iron (Total)	9.32 ± 0.06	3.57 ± 0.06	4.42 ± 0.03
Potassium	2.20 ± 0.03	1.04 ± 0.04	0.34 ± 0.01
Magnesium	0.61 ± 0.05	1.53 ± 0.05*	3.12 ± 0.08
Manganese	(0.03)	(0.03)	(0.02)
Sodium	0.25 ± 0.03	0.24 ± 0.02	1.09 ± 0.05
Phosphorus	0.10 ± 0.01	0.52 ± 0.01	0.51 ± 0.02
Silicon	24.06 ± 0.08	25.85 ± 0.17	16.83 ± 0.12
Sulfur	-----	0.15 ± 0.01	0.83 ± 0.05
Strontium	(0.07)	(0.20)	(0.27)
Titanium	0.75 ± 0.01	0.52 ± 0.01	0.90 ± 0.02
LOI 750 °C **	(1.76)	(0.53)	(0.23)
Moisture (110 °C) **	(0.14)	(0.12)	(0.08)
Residue on a 45 μm electroformed sieve (ASTM Standard Test Method C430-83)	12.8 ± 1.2	8.0 ± 0.7	10.5 ± 0.5

The uncertainty is expressed as two standard deviations of the certified value unless otherwise noted. *This uncertainty is based on scientific judgement. It is meant to approximate ± two standard deviations of the certified value.

**In accordance with ASTM Standard Test Method C311-85.

Table 2

Multipliers For Element to Oxide Conversion.

Constituent Element	Oxide Sought	Multiplier
Aluminum	Al_2O_3	1.88946
Barium	BaO	1.11650
Calcium	CaO	1.39919
Iron	Fe_2O_3	1.42974
Potassium	K_2O	1.20459
Magnesium	MgO	1.65807
Manganese	MnO	1.29122
Manganese	Mn_2O_3	1.43684
Sodium	Na_2O	1.34798
Phosphorus	P_2O_5	2.29137
Silicon	SiO_2	2.13931
Strontium	SrO	1.18261
Sulfur	SO_3	2.49714
Titanium	TiO_2	1.66806

Source and Preparation of Materials: The fly ashes were obtained from three different coal-fired power plants and are products of western Kentucky, Colorado, and Wyoming coals. Each fly ash was size classified and particles greater than 45 micrometers were removed. These coarse particles, mostly quartz and partially burned fragments, were ground to pass a No. 100 (150 μm) 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. The packaging operations were performed in a temperature and humidity controlled atmosphere to minimize moisture differences between samples.

Homogeneity Testing: Stratified random selections of vials from each SRM were made and analyzed using x-ray fluorescence. Each vial was opened and two aliquots of 0.5 g were taken and fused into glass discs for XRF analyses. The duplicates of the individual vials made a total of 50 discs for each fly ash. For the elements measured (Al, Ca, Fe, and Si), no evidence of sample heterogeneity was observed.

TABLE 3

SUPPLEMENTAL INFORMATION ON THE SOURCE AND DESCRIPTION OF SRM's 2689, 2690, and 2691

	2689	2690	2691
Source:	Monier Resources, Inc. San Antonio, Texas 78216	Pozzolanic International Mercer Island, Wash.	Kansas City Power & Light Co. 98040 Kansas City, Mo. 64141
Power Plant:	Georgia Power Co. Plant Bowen Stilesboro, Ga.	Colorado-Ute Electric Association Craig Station Craig, Colo.	KCP and L Iatan Power Station Iatan, Mo.
ASTM C618 Class:	F	F	C
Coal Mine:	Western Kentucky Coal Districts 8 & 9	Trapper Mining, Inc. Craig, Colo.	Arco Black Thunder Mine Powder River Basin Gillette, Wyo.
Coal Type:	Bituminous (Moderate Sulfur)	Sub-bituminous (Low Sulfur)	Sub-bituminous (Low Sulfur)
Typical Properties of Coals Burned to Produce SRM's			
Btu/lb:	12,000	9,700	8,800
Moisture, wt. %:	6	16.5	27.6
Ash, wt. %:	12	5.3	4.8
Sulfur, wt. %	1.5	0.3	0.3

TABLE 4

Analytical Techniques and Methods Used in the Certification of SRM's 2689, 2690, and 2691

Method/ Element	A	B	C	D	E	F	G
Aluminum	*			*		*	
Barium	*			*			
Calcium	*			*	*	*	
Iron	*				*	*	
Magnesium	*					*	
Manganese		*		*		*	
Phosphorus		*			*	*	
Potassium	*			*		*	
Silicon	*	*	*			*	
Sodium	*			*		*	
Strontium	*			*		*	
Sulfur			*		*	*	
Titanium	*			*		*	
Loss on Ignition			*				
Moisture			*				
Sieve Residue (No.325 sieve)							*

ANALYTICAL TECHNIQUES AND METHODS

A. Atomic Absorption
B. Direct Current Plasma Emission Spectrometry
C. Gravimetry
D. Neutron Activation Analysis
E. Titrimetry (Colorimetry)
F. X-Ray Fluorescence Spectrometry
G. ASTM Test Method C430-83 for Fineness of Hydraulic Cement by the 45-micrometer (No.325). Sieve as specified in ASTM C311,Standard Methods of Sampling and Testing Fly Ash or Natural Pozzolans for Use as an Admixture in Portland Cement Concrete.

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