U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1633a

Trace Elements in Coal Fly Ash

This Standard Reference Material (SRM) is intended for use in the evaluation of analytical methods for the determination of constituent elements in coal fly ash or materials with a similar matrix.

SRM 1633a is a fly ash that was sieved through a No. 170 sieve with a nominal sieve opening of 90 μm.

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the analysts are given in Table 3. The certified values are based on results obtained by reference methods of known accuracy or from two or more independent, reliable analytical methods. Noncertified values are given for information only in Table 2.

Notice and Warnings to Users: This certification is invalid 5 years from date of purchase of the SRM. The constituents certified or analyzed are reviewed periodically and may be updated to reflect improved measurement. Updated certificates will be made available upon request.

Use: This material should be dried to a constant weight before using. Recommended procedures for drying are: (1) Vacuum drying for 24 hours at ambient temperature using a cold trap at or below -50 ° C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying for 2 hours in an oven at 105 ° C; (3) drying in a dessicator over P_2O_5 or Mg_2ClO_4. Samples of the dried material weighing at least 250-mg should be used for analysis. When not in use the material should be kept in a tightly sealed bottle.

Source and Preparation of Material: The fly ash material was supplied by a coal fired power plant and is a product of Pennsylvania and West Virginia coals. It was selected as a typical fly ash and is not intended as a fly ash from a specific coal or combustion process. The material was sieved and blended for 2 hours in a Vee blender. The material was then removed and placed in a series of bulk containers from which specific samples were taken for homogeneity testing and certification analysis. Twelve bottles were selected for the homogeneity test. Samples from each bottle were analyzed for cobalt, chromium, europium, iron, scandium, and thorium using nondestructive neutron activation analysis. The observed standard deviations for both 50 and 250 mg sample sizes were consistent with counting statistics, indicating that the fly ash is homogeneous within $\pm$ 5% (relative) based on these elements. The homogeneity testing and certification analyses were performed in the NBS Center for Analytical Chemistry.

The overall direction and coordination of the analytical measurements leading to the initial certification were performed in the Center for Analytical Chemistry under the chairmanship of L.A. Machlan.

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

Gaithersburg, MD 20899
January 5, 1985
(Revision of certificate
dated April 18, 1979)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Table 1. Certified Values of Constituent Elements

Major Constituents	Content Wt. Percent	Minor Constituents	Content Wt. Percent
Aluminum	14.3 ±1.0[a]	Magnesium	0.455 ± 0.010
Iron	9.4 ±0.1	Sodium	0.17 ± 0.01
Potassium	1.88 ±0.06		
Silicon	22.8 ±0.8		
Calcium	1.11 ±0.01		

Trace Constituents

Element	Content µg/g	Element	Content µg/g
Antimony	6.8 ± 0.4	Rubidium	131 ± 2
Arsenic	145 ±15	Selenium	10.3 ± 0.6
Cadmium	1.00 ± 0.15	Strontium	830 ± 30
Chromium	196 ± 6	Thorium	24.7 ± 0.3
Copper	118 ± 3	Thallium	5.7 ± 0.2
Manganese	179 ± 8	Uranium	10.2 ± 0.1
Mercury	0.16 ± 0.01	Vanadium	297 ± 6
Nickel	127 ± 4	Zinc	220 ± 10
Lead	72.4 ± 0.4		

[a]The uncertainties of the certified values are based on judgment and represent 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).

Supplemental Information

Note: The following values are not certified because they are not based on the results of either a reference method or of two or more independent methods. These values are included for information only.

Table 2. Noncertified Values for Constituent Elements

Element	Content Wt. Percent	Element	Content µg/g
Barium	0.15	Beryllium	12
Titanium	0.8	Cerium	180
Sulfur	0.18	Cobalt	46
		Cesium	11
		Europium	4
		Gallium	58
		Hafnium	8
		Molybdenum	29
		Scandium	40

Table 3. Analytical Methods Used for Certified Constituent Elements

Method/ Element	A	B	C	D	E	F	G	H	I
Aluminum	•		•						•
Antimony			•				•		
Arsenic	•		•						
Cadmium		•	•	•			•		
Calcium	•	•			•				
Chromium	•	•	•						
Copper	•	•	•						
Iron	•	•	•						
Lead		•		•	•				
Magnesium	•	•							
Manganese	•		•						•
Mercury	•		•						
Nickel	•	•		•	•				
Potassium	•	•			•				
Rubidium	•	•	•		•				
Selenium	•		•				•		
Silicon					•			•	
Sodium	•		•						
Strontium	•				•	•			
Thallium		•					•		
Thorium		•	•						
Uranium		•							
Vanadium	•	•	•						
Zinc	•	•		•	•	•			

Analytical Methods

A. Atomic Absorption Spectrometry or Flame Emission Spectrometry
B. Isotope Dilution Mass Spectrometry
C. Neutron Activation Analysis
D. Polarography
E. X-ray Fluorescence Spectrometry
F. Inductively-Coupled Plasma Emission Spectrometry
G. Isotope Dilution Spark Source Mass Spectrometry
H. Gravimetry
I. Direct Coupled Plasma Emission Spectrometry

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