National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material 1633b

Constituent 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 1633b is a bituminous coal fly ash that was sieved through a nominal sieve opening of 90 μm (170 mesh) and then blended to assure homogeneity. A unit of SRM 1633b consists of 75 g of powdered material.

The certified values for the constituent elements are given in Table 1. The values, except for Hg, are based on measurements using one definitive method or two or more independent and reliable analytical techniques. Noncertified values for a number of elements are given in Table 2 as additional information on the composition of the material. The noncertified values should not be used for calibration or quality control. Analytical methods used for the certification of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values are based on measurements using a dry sample weight of at least 250 mg.

NOTICE AND WARNING TO USERS

Expiration of Certification: This certification is valid for 5 years from the date of shipment from NIST. Should any of the certified values change before the expiration of the certification, the purchaser will be notified by NIST.

Stability: This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

Use: A minimum dry sample weight (see Instructions for Drying) of 250 mg should be used for analytical determinations to be related to the certified values on this Certificate of Analysis.

To obtain the certified values, sample preparation procedures should be designed to affect complete dissolution. If volatile elements (e.g., Hg, As, Se) are to be determined, precautions should be taken in the dissolution of SRM 1633b to avoid volatilization losses.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of R.R. Greenberg of the NIST Inorganic Analytical Research Division.

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

Gaithersburg, MD 20899
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Thomas E. Gills, Acting Chief
Standard Reference Materials Program

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Instructions for Drying: When non-volatile elements are being determined, this material should be dried to constant weight before using. Recommended procedures for drying are: 1) Vacuum drying for 24 h at ambient temperature using a cold trap at or below -50 °C and a pressure not greater than 0.2 mm Hg (30 Pa); 2) drying for 2 h in an oven of 105 °C. 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. Volatile elements should be determined on an as-received basis, and corrected to dry weight. Correction should be based on a separate determination of moisture, using one of the above drying procedures.

Source and Preparation of the Material: The fly ash was supplied by a coal fired power plant and is the product of Pennsylvania and West Virginia coals. It was selected as a typical bituminous coal fly ash and is not intended as a fly ash from a specific coal or combustion process. The material was air dried, sieved, and blended for 24 h, before being placed in a series of bulk containers. X-ray fluorescence and inductively coupled plasma atomic emission analyses were performed on ten grab samples taken from the bulk for a preliminary homogeneity assessment before proceeding with bottling the material in 75 g units.

Analysis: The homogeneity of the bottled material was assessed by X-ray fluorescence spectrometry and instrumental neutron activation analysis, using selected elements as indicators. In some cases, statistically significant differences between samples were seen, and the variance due to material inhomogeneity is included in the overall uncertainties of the certified values. The estimated relative standard deviation for material inhomogeneity is less than 1% for those elements for which homogeneity was assessed, except Th, for which material inhomogeneity was estimated to be 2%.

Certified Values and Uncertainties: The certified values are weighted means of results of two or more independent analytical methods, or the means of results from a single definitive method, except for mercury. Mercury certification is based on cold vapor atomic absorption spectrometry measurements performed at NIST. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385). The stated uncertainty includes allowances for measurement imprecision, material variability, and differences among analytical methods. Each uncertainty is the sum of the half-width of a 95% prediction interval, and includes an allowance for the systematic error among the methods used. In the absence of systematic error, a 95% prediction interval predicts where the true concentrations of 95% of the samples of this SRM lie.

Table 1. Certified Values

Element	wt %			Element	mg/kg		
Aluminum	15.05	±	0.27	Arsenic	136.2	±	2.6
Calcium	1.51	±	0.06	Barium	709	±	27
Iron	7.78	±	0.23	Cadmium	0.784	±	0.006
Magnesium	0.482	±	0.008	Chromium	198.2	±	4.7
Potassium	1.95	±	0.03	Copper	112.8	±	2.6
Silicon	23.02	±	0.08	Lead	68.2	±	1.1
Sodium	0.201	±	0.003	Manganese	131.8	±	1.7
Sulfur	0.2075	±	0.0011	Mercury	0.141	±	0.019
Titanium	0.791	±	0.014	Nickel	120.6	±	1.8
				Selenium	10.26	±	0.17
				Strontium	1041	±	14
				Thorium	25.7	±	1.3
				Uranium	8.79	±	0.36
				Vanadium	295.7	±	3.6

Table 2. Noncertified Values

Element	mg/kg	Element	mg/kg
Antimony	6	Phosphorus	2300
Bromine	2.9	Rubidium	140
Cerium	190	Scandium	41
Cobalt	50	Samarium	20
Cesium	11	Tantalum	1.8
Dysprosium	17	Terbium	2.6
Europium	4.1	Thallium	5.9
Gadolinum	13	Thulium	2.1
Hafnium	6.8	Tungsten	5.6
Holmium	3.5	Ytterbium	7.6
Lanthanum	94	Zinc	210
Lutetium	1.2		
Neodymium	85		

Table 3. Analytical Methods Used for Certification Analyses of SRM 1633b

Element	Certification Methods
Al	INAA, XRF
As	FIA-HAAS, INAA
Ba	ICP-MS, INAA
Ca	ICP, INAA, XRF
Cd	ETAAS, IDTIMS
Cr	FAAS, INAA
Cu	FAAS, ICP-MS
Fe	INAA, XRF
Hg	CVAAS
K	FAES, INAA, XRF
Mg	ICP, IDTIMS
Mn	FAAS, INAA
Na	FAES, INAA
Ni	ETAAS, ICP
Pb	ETAAS, ICP-MS
Rb	FAES, INAA
S	IDTIMS
Sb	ETAAS, INAA
Se	FIA-HAAS, INAA
Si	GRAV, XRF
Sr	FAES, INAA, IDTIMS
Th	ICP-MS, INAA
Ti	INAA, XRF
U	ICP-MS, INAA
V	ICP, INAA

ID-TIMS - Isotope dilution thermal ionization mass spectrometry, mixed acid digestion.
ICP-MS - Inductively coupled plasma mass spectrometry; mixed acid digestion.
INAA - Instrumental neutron activation analysis.
XRF - Wavelength dispersive X-ray fluorescence on fused borate discs.
ICP-AES - Inductively coupled plasma atomic emission spectrometry; mixed acid digestion.
ETAAS - Electrothermal atomic absorption spectrometry; mixed acid digestion.
CVAAS - Cold vapor atomic absorption spectrometry.
FIA-HAAS - Flow injection analyses - Hydride generation atomic absorption spectrometry.
FAAS - Flame atomic absorption spectrometry; mixed acid digestion except for Au, leached with $HBr\text{-}Br_2$.
GRAV - Gravimetry; sodium carbonate fusion.

Most information values were determined by INAA only; P was determined by ICP-AES and XRF, Tl was determined by ICP-MS, and Zn was determined by FAAS and ICP-AES.

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