

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1633

Trace Elements in Coal Fly Ash

This Standard Reference Material is intended for use in the calibration of apparatus and methods used in analyses of coal fly ash and other materials with similar matrices for trace elements. This material should be dried to a constant weight before using. Recommended procedures for drying are: (1) drying for 24 hours using a cold trap at or below -50°C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying in a desiccator over P_2O_5 or $\text{Mg}(\text{ClO}_4)_2$. When not in use, the material should be kept in a tightly sealed bottle. Long term (>1 year) stability of this SRM has not been rigorously established. NBS will continue to monitor this material and any substantive change will be reported to purchasers.

The certified values given below are based on at least a 250-mg sample of the dried material, the minimum amount that should be used for analysis.

Element ¹	Content ² $\mu\text{g/g}$	Element ¹	Content ² $\mu\text{g/g}$
Manganese ^{a,e,g}	493 \pm 7	Nickel ^{a,c,i}	98 \pm 3
Zinc ^{a,e}	210 \pm 20	Arsenic ^{e,h}	61 \pm 6
Vanadium ^{b,e,g}	214 \pm 8	Uranium ^{e,f}	11.6 \pm 0.2
Lead ^{c,i}	70 \pm 4	Selenium ^{d,e}	9.4 \pm 0.5
Chromium ^{a,c,e,i}	131 \pm 2	Cadmium ^{a d i,e}	1.45 \pm 0.06
Copper ^{a,d,j}	128 \pm 5	Mercury ^{a e}	0.14 \pm 0.01

1. Methods of Analyses:

- | | |
|--|----------------------------|
| a. Atomic Absorption Spectrophotometry | f. Nuclear Track Technique |
| b. Flame Emission Spectrometry | g. Photometric |
| c. Isotope Dilution Mass Spectrometry | h. Photon Activation |
| d. Isotope Dilution Spark Source Mass Spectrometry | i. Polarography |
| e. Neutron Activation | j. Colorimetry |

2. The values are based on the results of 4 to 28 determinations by each of at least two analytical techniques. The estimated uncertainties include sample variations, possible method differences, and errors of measurement (but in no case less than the 95% confidence limits computed for the analyses).

The overall direction and coordination of the analytical measurements leading to certification were performed in the Analytical Chemistry Division under the chairmanship of P. D. LaFleur.

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 C. L. Stanley.

Washington, D. C. 20234
 March 7, 1975

J. Paul Cali, Chief
 Office of Standard Reference Materials

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PREPARATION, TESTING, and ANALYSIS

This material is a blend of coal fly ashes supplied by five electric power plants: Tennessee Valley Authority, Stevenson, Alabama; Commonwealth Edison, Chicago, Illinois; Baltimore Gas and Electric Co., Baltimore, Maryland; Carolina Light and Power Co., Roxboro, North Carolina; and Potomac Electric Power Co., Washington, D. C. These plants were specially selected to provide fly ashes that covered a broad spectrum of the fly ashes from the coal mining industry. Five of the fly ashes were collected by electrostatic precipitators and one by a mechanical collector. The fly ashes were sieved and the portion passing through a 170 mesh sieve was taken (88 micrometers diameter or less). After sieving, the fly ashes were blended in a double-coned blender. After 1 hour the material was immediately removed and bottled.

A random scheme for sample selection was designed and a statistical analysis of the homogeneity data was performed by J. Mandel of the NBS Institute for Materials Research. Fifteen of 500 bottles were selected for homogeneity tests. These samples were analyzed for manganese by non-destructive neutron activation analysis. Replicate analyses on 250 mg samples indicated homogeneity within $\pm 5\%$ (relative) based upon this element. X-ray fluorescence analyses on bulk samples before bottling supports this conclusion with values for calcium, titanium, iron, nickel, zinc, strontium, and barium. The homogeneity analyses were performed in the NBS Analytical Chemistry Division by T. E. Gills and S. D. Rasberry. Analyses for the various elements were made in the NBS Analytical Chemistry Division by the following analyst: R. K. Bell, B. S. Carpenter, L. P. Dunstan, M. S. Epstein, E. L. Garner, T. E. Gills, E. S. Gladney, J. W. Gramlich, G. J. Lutz, L. A. Machlan, E. J. Maienthal, L. T. McClendon, T. J. Murphy, E. Orvini, P. J. Paulsen, T. C. Rains, T. A. Rush, and K. M. Sappenfield.

The following values are not certified because they are based on a non-reference method, or were not determined by two or more independent methods. They are included for information only.

<u>Element</u>	<u>Content ($\mu\text{g/g}$)</u>
Potassium	(1.72%)
Strontium	(1380)
Rubidium	(112)
Cobalt	(38)
Thorium	(24)
Beryllium	(12)
Thallium	(4)