

National Bureau of Standards Certificate of Analysis Standard Reference Material 1632a Trace Elements in Coal (Bituminous)

This Standard Reference Material is intended for use in the calibration of apparatus and the evaluation of techniques employed in the trace element analysis of coal and similar materials. The material should be dried without heat to constant weight before use.

The recommended procedures for drying are either vacuum drying at ambient temperature for 24 hours, or freeze drying in which the drying chamber is kept at room temperature. When not in use, the material should be kept in a tightly sealed bottle and stored in a cool, dark place. 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, $\mu\text{g}/\text{g}^2$	Element ¹	Content, $\mu\text{g}/\text{g}^2$
Arsenic ^{a,b}	9.3 ± 1.0	Thorium ^{c,e}	4.5 ± 0.1
Cadmium ^{c,d}	0.17 ± 0.02	Uranium ^c	1.28 ± .02
Chromium ^{c,e}	34.4 ± 1.5	Vanadium ^{c,g}	44 ± 3
Copper ^{a,e}	16.5 ± 1.0	Zinc ^{c,d}	28 ± 2
Lead ^{c,d}	12.4 ± 0.6	Element	Wt. %
Manganese ^{a,e}	28 ± 2	Calcium ^{a,g,h}	0.23 ± 0.03
Mercury ^{a,e}	0.13 ± 0.03	Iron ^{c,d,e,f}	1.11 ± .02
Nickel ^{c,d}	19.4 ± 1.0	Potassium ^{c,g}	.42 ± .02
Selenium ^{a,e}	2.6 ± 0.7	Sulfur ^{i,j,k,l}	1.58 ± .04
Sodium ^{a,e}	840 ± 40		

1. Methods of Analysis:

- | | |
|---------------------------------------|---|
| a. Atomic Absorption Spectrometry | h. Inductively Coupled Plasma Emission Spectrometry |
| b. Photon Activation | i. Gravimetry |
| c. Isotope Dilution Mass Spectrometry | j. Ion Chromatography |
| d. Polarography | k. ANSI/ASTM Standard Test Method D1552-64 |
| e. Neutron Activation | l. Prompt-Gamma Activation Analysis |
| f. Spectrophotometry | |
| g. Flame Emission Spectrometry | |

2. The estimated uncertainty is based on judgment and represents 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.)

The overall direction and coordination of the analytical measurements leading to this certificate were performed in the Analytical Chemistry Division under the chairmanship of L. J. Moore.

Washington, D.C. 20234
 October 24, 1983
 (Revision of Certificate
 dated 1-23-78)

Stanley D. Rasberry, Chief
 Office of Standard Reference Materials

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PREPARATION, TESTING and ANALYSIS

This material was prepared from one lot of coal designated as Pennsylvania Seam Coal. It was prepared under the auspices of F. Walker and J. Doubruck of the U.S. Bureau of Mines, Pittsburgh, Pennsylvania. The crushed and ground coal was sieved through a 250 μm (No. 60) sieve and thoroughly blended in a V-type blender.

Samples for homogeneity testing were taken from the top, middle, and bottom of three bulk containers of coal and analyzed by neutron activation analysis for scandium, chromium, iron, cobalt, cerium, and thorium. Replicate analyses of 250-mg samples indicated the material variability for these elements to be within $\pm 2\%$ (relative). The homogeneity measurements were performed in the NBS Analytical Chemistry Division by R. R. Greenberg.

Certification analyses for the various elements were made in the NBS Analytical Chemistry Division by T. J. Brady, B. I. Diamondstone, L. P. Dunstan, M. S. Epstein, M. Gallorini, E. L. Garner, T. E. Gills, J. W. Gramlich, R. R. Greenberg, S. H. Harrison, G. M. Hyde, H. M. Kingston, R. M. Lindstrom, G. J. Lutz, L. A. Machlan, E. J. Maienthal, J. D. Messman, T. J. Murphy, T. C. Rains, and R. L. Watters, Jr.

The following values are *not certified* because they were 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</u> <u>($\mu\text{g/g}$)</u>
Antimony	(0.6)
Cerium	(30)
Cesium	(2.4)
Cobalt	(6.8)
Europium	(0.5)
Gallium	(8.5)
Hafnium	(1.6)
Rubidium	(31)
Scandium	(6.3)
	<u>(wt. %)</u>
Aluminum	(3.1)
Magnesium	(0.1)
Titanium	(0.18)