

Certificate of Analysis

HPS Certified Reference Material

Coal A1

Lot # 032219

This Certified Reference Material is coal obtained from West Virginia. The certified values are based on at least two independent analytical techniques for major, minor, and trace elements after a total digestion procedure.

The material was collected from the field and stored in polyethylene bags in cardboard boxes (12 in. x 12 in. x 18 in.) and transported to the laboratory. During the drying period all foreign objects were removed by hand. The coal was then dried under infrared heat lamps, ground in a ballmill, and sieved. Coarse particles were removed and only particles that pass a 100 mesh sieve were saved. The coarse particles were reground and sieved. The saved material was transferred to a 55-gal polyethylene mixing-drum and blended for several hours. Then the material was bottled into 50-g units. Randomly selected bottles were taken for the final homogeneity testing.

Instructions for drying: Sample should be dried for 2 h at 110°C. Volatile elements (i.e., Hg) should be determined on samples received. Separate samples should be dried as previously described to obtain a correction factor for moisture. The weight loss on drying was determined to be in the range of 0.5 to 1.0 percent.

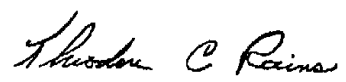
Preparation of Sample for Analysis:

Total Digestion Method: Transfer 1.000 g of the dried material to a clean 100 mL Teflon beaker. Add 5 mL of high-purity HNO₃ and 10 mL of HF, cover beaker with a Teflon lid and digest on a hot plate at 120°C for 6-8 h. Remove the lid and add 10 mL of HClO₄. (NOTE: If the sample has gone dry or contains less than 5 mL of acid, add an additional 5 mL of HNO₃). Continue the digestion on a hot plate at a 160°C or strong fumes of perchlorate until sample is dry. Rinse down the sides of the beaker with water and take sample to dryness. Continue to heat until all signs of acid fumes are removed. Add 5 mL of HNO₃ and 20 mL of water and heat to dissolve the residue. Filter off the insoluble residue using a quantitative filter paper. Save the filtrate and return the filter paper with residue to the beaker. Add 10 mL of HNO₃ to the beaker and heat to destroy the filter paper. Evaporate the solution to a low volume and add 10 mL of HCl and 5 mL of HNO₃. Evaporate the solution to approximately 5 mL. Add 10 mL of HF, and cover beaker with a Teflon lid. Heat the solution for 1 hour at a temperature of 120°C. Then remove the Teflon lid, rinse down the side

of the beaker, and add 10 mL of HClO₄. Heat to strong fumes of perchlorate. Continue to heat until sample is dry. Rinse down the sides of the beaker and continue to heat until all signs of acid fumes are removed. Add 5 mL of HNO₃ and 15 mL of water and heat to dissolve residue. (NOTE: The solution should be clear at this stage.) If so desired, the analyst can ignore any residue or repeat the addition of HF and HClO₄. Combine this solution with the filtration and dilute to a calibrated volume with water and proceed with the analysis of the analytes by AAS, ICP, or ICP/MS.

Certified Values for Coal A1 Total Digestion	
Element	Conc., %
*C	78.4
*S	0.90
*N	1.76
	Conc., mg/g
Al	4.14
Ca	0.785
Fe	2.92
K	0.255
Mg	0.147
Na	0.37
Ti	195
	Conc., µg/g
Ag	<0.005
As	(12)
Ba	24.8
Be	2.1
Cd	<0.005
Co	11.3
Cr	(6)
Cu	10.2
Li	(3)
Mn	3.6
Mo	<0.02
Ni	(15)
Pb	(4)
Sb	(1)
Se	(1.4)
Sn	<0.01
Sr	58.5
Tl	<0.02
V	(6)
Zn	(17)

*Analyzed by Combination: Elemental Analyzer


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