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CAT. NO. CRM-SA-A

Certificate of Analysis

HPS Certified Reference Material Sandy Soil A

Lot # <u>490228</u>

This Certified Reference Material is a pristine sandy soil obtained from Johns Island, Charleston County, South Carolina. The certified values are based on at least two different analytical techniques for major, minor, and trace elements after a total and EPA-Method 3050-A 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. The soil was transferred onto a 2 ft. x 4 ft. polyethylene-lined drying tray and air dried for three days with frequent mixing in direct sunlight during the month of July. During the drying period all foreign objects were removed by hand. The soil 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.

<u>Instructions for drying</u>: 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. (CAUTION: Samples used for Hg analyses should not be stored in a desiccator as this may lead to contamination.)



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Preparation of Sample for Analysis:

(A) Total Digestion Method: Transfer 2.000 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 or ICP.

(B) EPA 3050A Digestion: Proceed as described in EPA Method 3050A.

<u>Certified Values</u>: The certified values are based on the results of 5 to 30 determinations by two different analytical techniques. The estimated uncertainties at the 95% confidence level include those due to sample variation, possible method differences and errors of measurement.



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Certifie	d Values for Total Digestion	Sandy Soil A EPA 3050A HNO ₃ Digestion	
Element	Conc., mg/g	Conc., mg/g	
Al	21.7 ± 0.4	6.68 ± 0.26	
Са	3.27 ± 0.09	0.42 ± 0.02	
Fe	9.27 ± 0.30	4.38 ± 0.15	
K	7.07 ± 0.03	0.14 ± 0.02	
Mg	1.03 ± 0.03	0.22 ± 0.01	
Na	2.74 ± 0.05	0.012 ± 0.002	
S	(0.16)	/	
Ti	(4)	(0.04)	
Element	Conc., µg/g	Conc., µg/g	
Ag	(0.1)		
As	2.0 ± 0.4	< 0.005	
Ba	314 ± 26	10.1 ± 0.8	
Be	0.29 ± 0.04	0.14 ± 0.02	
Cd	(0.03)	(0.02)	
Со	1.3 ± 0.1	0.43 ± 0.04	
Cr	21.5 ± 0.4	6.9 ± 0.2	
Cu	3.2 ± 0.3	2.2 ± 0.2	
Li	(4)		
Mn	234 ± 30	17.9 ± 0.6	
Мо	(1)	(0.1)	
Ni	3.3 ± 0.3	1.53 ± 0.12	
Pb	15.2 ± 2.0	6.2 ± 0.6	
Sb	< 0.005	< 0.005	-
Se	0.40 ± 0.05	0.29 ± 0.04	
Sn		(0.4)	-
Sr		(3)	-
Tl	< 0.005	< 0.005	
V	(8)	(8)	
Zn	(11)	6.5 ± 0.4	



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Non certified values, shown in parentheses, are provided for information only. As additional data are obtained, the Certificate of Analysis will be revised to reflect any changes in concentration and its uncertainty.

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