HIGH-PURITY STANDARDS
www.highpuritystandards.com

P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

CAT. NO. CRM-MS-S

Certificate of Analysis

HPS Certified Reference Material

Marine Sediment

Lot No. 830702

This Certified Reference Material is marine sediment obtained from Charleston, South Carolina, harbor. The certified values are based on at least two independent analytical techniques for major, minor, and trace elements after a total HNO_3, HF and $HClO_4$ procedure.

The material was collected at low tide near the Charleston Marina in plastic buckets and transported to the laboratory. The sediment was allowed to settle and water removed. The sediment was transferred onto a 2 ft. X 4 ft. polyethylene – lined drying tray and air dried for several days in direct sunlight. The sediment was 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 as 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:

(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 HNO_3 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 $HClO_4$. (NOTE: If the sample has gone dry or contains less than 5 mL of acid, add an additional 5 mL of HNO_3). Continue the digestion on a hot plate at 160°C to strong fumes of perchlorate; continue until sample is moist dry. Rinse down the sides of the beaker with water and take sample to moist dryness. Add 5 mL of HNO_3 and 20 mL of H_2O 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 HNO_3 to the beaker and heat to destroy the filter paper. 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 sides of the beaker, and add 10 mL of $HClO_4$. Heat to strong fumes of perchlorate. Rinse down the sides of the beaker and continue to heat until the sample is moist dry. Add 5 mL of HNO_3 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 $HClO_4$. Combine this solution with the filtrate and dilute to a calibrated volume with water and proceed with the analysis of the analytes by AAS, ICP, or ICP/MS.

(B) EPA 3050B(HNO3/HCl) Digestion Method: Transfer 2.000 g of the dried material to a clean Teflon beaker and proceed as described in the EPA procedure.

HIGH-PURITY STANDARDS
www.highpuritystandards.com
P.O. Box 41727
Charleston, SC 29423
Phone (843) 767-7900
Fax (843) 767-7906

Certified Values: The certified values are based on the results of 5 to 30 determinations by two independent analytical techniques. The estimated uncertainties at the 95-percent confidence limits include those due to sample variation, possible method differences and errors of measurement.

Major Elements

Element	Total Digestion Concentration, mg/g	3050B (HNO_3/HCl) Digestion Concentration mg/g
Al	66.8 ± 2.6	23.3 ± 0.9
Ca	40.0 ± 1.4	36.3 ± 0.5
Fe	39.2 ± 1.7	29.1 ± 0.3
K	9.76 ± 1.08	2.89 ± 0.18
Mg	10.4 ± 0.3	8.94 ± 0.28
Na	19.4 ± 1.2	17.2 ± 0.6
P	1.24 ± 0.12	(1.2)
Ti	4.06 ± 0.21	(0.2)
*S	10.0 ± 0.2	(10)
*C	44.8 ± 0.3	
*N	3.6 ± 0.8	

Trace Elements

Element	Concentration, µg/g	Concentration µg/g
Ag	(0.1)	<0.005
As	14.4 ± 1.5	12.8 ± 2.4
Ba	196 ± 15	31.0 ± 2.7
Be	(1)	(1)
Cd	(0.3)	0.2
Co	9.69 ± 2.02	6.31 ± 0.54
Cr	128 ± 15	47.1 ± 7.8
Cu	35.5 ± 6.0	34.4 ± 1.4
Li	64.8 ± 6.3	38.2 ± 4.8
Mn	448 ± 30	352 ± 30
Mo	<0.05	<0.05
Ni	35.2 ± 4.8	16.4 ± 2.1
Pb	77.2 ±12.0	70.2 ± 4.2
Sb	(0.9)	<0.005
Se	(0.7)	<0.005
Sn	10.2 ± 1.8	6.3 ± 1.5
Sr	190 ± 18	149 ± 12
Tl	<0.005	<0.005
V	101 ± 12	47.6 ± 4.5
Zn	157 ± 20	134 ± 10

Values in parenthesis are given for information purposes only.
*By combustion: Elemental Analyzer

Theodore C. Rains, Ph. D.
President