U. S. Department of Commerce
Malcolm Baldrige
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1646

Estuarine Sediment

This Standard Reference Material is intended primarily for calibrating instrumentation and evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in sediments, and similar matrices.

Values of Constituent Elements: The *certified* values for the constituent elements are shown in Table 1. They are based on results obtained either by definitive methods or by two or more independent, reliable analytical methods. *Non-certified values,* which are given for information only, appear in Table 2. All values are based on a minimum sample size of 500 mg of the material dried as indicated under "Instructions for Drying".

Notice to Users:

Expiration of Certification: The certification of this SRM will be invalid 5 years after date of shipping.

Use: The material should be kept in its original bottle and shaken well before each use. A minimum sample of 500 mg of the dried material (see Instructions for Drying) should be used for any analytical determination to be related to a certified value of this certificate.

Statistical consultation was provided by K. R. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the technical measurements leading to certification were performed in the Inorganic Analytical Research Division, E. L. Garner, Chief.

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 R. Alvarez.

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(over)

George A. Uriano, Chief
Office of Standard Reference Materials

Table 1. Certified Concentration of Constituent Elements

Element	Concentration, weight %	Element	Concentration, weight %
Aluminum[2b,c;6]	6.25 ± 0.20	Magnesium[1c;2c]	1.09 ± 0.08
Calcium[2b,c;6]	0.83 ± 0.03	Phosphorus[2a;6]	0.054 ± 0.005
Iron[2c;4a;6]	3.35 ± 0.10		

Element	Concentration, μg/g	Element	Concentration, μg/g
Arsenic[1d;4b]	11.6 ± 1.3	Manganese[1c;2c]	375 ± 20
Cadmium[1b,3a,b;4b]	0.36 ± 0.07	Mercury[1a;4b]	0.063 ± 0.012
Chromium[1c;3b;4a]	76 ± 3	Nickel[1b;2c;5]	32 ± 3
Cobalt[1b;4a]	10.5 ± 1.3	Vanadium[2a,3a]	94 ± 1
Copper[1c;2c;4b]	18 ± 3	Zinc[1b,c;2c;3b;5]	138 ± 6
Lead[1b;3a;5]	28.2 ± 1.8		

1. Atomic absorption spectrometry
   a. cold vapor
   b. graphite furnace
   c. flame
   d. hydride generation
2. Atomic emission spectrometry
   a. dc plasma
   b. flame
   c. inductively coupled plasma
3. Isotope dilution mass spectrometry
   a. thermal ionization
   b. spark source
4. Neutron activation
   a. instrumental
   b. radiochemical
5. Polarography
6. X-ray fluorescence spectrometry

Notes: (1.) Analytical values are based on the "dry-weight" of material (see Instructions for Drying). Mercury should be determined on samples without drying and the results adjusted to a "dry-weight" basis by determining the moisture content of separate samples.

(2.) The estimated uncertainty for an element 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 500 mg or more.

Table 2. Non-certified Concentrations of Constituent Elements

Note: The values shown in this table are not certified because they are not based on the results of either a definitive method or two or more independent analytical methods. These values are included, for information only, to provide additional information on the composition.

Element	Concentration, Weight %	Element	Concentration, Weight %
Potassium	(1.4)	Sulfur	(0.96)
Silicon	(31)	Titanium	(0.51)
Sodium	(2.0)		

Element	Concentration, μg/g	Element	Concentration, μg/g
Antimony	(0.4)	Molybdenum	(2.0)
Beryllium	(1.5)	Rubidium	(87)
Cerium	(80)	Scandium	(10.8)
Cesium	(3.7)	Selenium	(0.6)
Europium	(1.5)	Tellurium	(0.5)
Germanium	(1.4)	Thallium	(0.5)
Lithium	(49)	Thorium	(10)

Analysts:

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Instructions for Drying: Except for mercury, elements should be determined on samples that have been dried at 110 °C for 2 hours.

Mercury should be determined on undried samples. However, because the certified concentration is reported on a "dry-weight" basis, the concentration determined on undried samples should be adjusted for the moisture content of the samples.

Source and Preparation of Material: The material for this SRM was supplied by R. Huggett, Virginia Institute of Marine Sciences, Gloucester Point, Va. It had been dredged from the Chesapeake Bay at a location: 37° 11.1′ N, 76° 17.1′ W. The material was freeze-dried at Eastern Freeze-Dry Corporation, Lancaster, Pa., and radiation sterilized at Neutron Products Inc., Dickerson, Md. At NBS, the sediment was sieved through a screen with openings of 1.00 mm (No. 18) to remove coarse contaminants; ball-milled to pass a sieve with openings of 150 μm (No. 100); thoroughly mixed in a V-blender; placed in polyethylene bags; and bottled.

Homogeneity Assessment: A preliminary evaluation of homogeneity was made by instrumental neutron activation using samples of approximately 250 mg taken from various locations of the bulk materials. The samples were irradiated and the activities from radionuclides of Ce, Co, Cr, Cs, Eu, Fe, Rb, Sc and Th were counted. Except for Ce and Th, the observed sample-to-sample variations for the elements were approximately the same as the counting statistics indicating satisfactory homogeneity for these elements within approximately 2%. The homogeneity of the material for As, Cd, Hg, N, and Zn was evaluated by various analytical techniques using samples weighting 250 to 300 mg and found to be satisfactory. The homogeneity of the remaining certified elements was determined using sample weights not exceeding one gram.

The uncertainties of the elemental concentrations in Table 1 take into account possible material inhomogeneity for samples weighing 500 mg.