National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1646a

Estuarine Sediment

This Standard Reference Material (SRM) is intended primarily for calibrating instrumentation and evaluating the reliability of analytical methods for the determination of major, minor, and trace elements in estuarine sediments and similar matrices. One unit of SRM 1646a contains 70 g of material.

Certified Values and Uncertainties: 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. The results of two or more independent analytical methods were weighted according to the algorithm of Paule and Mandel [1]. The expanded uncertainties, whose level of confidence is approximately 95 %, include random and systematic sources of uncertainty from within each analytical method, material variability, which was detected for lead, and a systematic component of uncertainty between analytical methods [2]. All values are based on a minimum sample size of 500 mg of the material dried as indicated under ("Instructions for Drying").

Noncertified Values: Noncertified values are given in Table 2. Noncertified values are provided for information only because only one independent method was used, or there was insufficient agreement between the methods.

NOTICE AND WARNING TO USERS

Expiration of Certification: The certification is valid for five years from the date of shipment from NIST. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NIST. Registration, see attached sheet, will facilitate notification.

Stability: This material is considered to be stable; however, its stability has not been rigorously assessed. NIST will monitor this material and will report any substantive changes in certification to the purchaser.

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

Instructions for Drying: Except for volatile elements (e.g., arsenic, mercury, and selenium), elements should be determined on samples that have been dried at 110 °C for 2 h.

Volatile elements should be determined on undried samples. However, because the certified values are reported on a dry weight basis, the volatile elements determined on undried samples will have to be adjusted for the difference in moisture content.

The overall direction and coordination of the technical measurements leading to certification were performed by R.R. Greenberg of the NIST Analytical Chemistry Division.

Statistical consultation was provided by S.B. Schiller of the NIST Statistical Engineering Division.

Willie E. May, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 20 February 2004
See Certificate Revision History on Last Page

John Rumble, Jr., Chief
Measurement Services Division

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by J.S. Kane and B.S. MacDonald of the NIST Measurement Services Division.

Source and Preparation of Material: The material for this SRM was collected under the direction of M. Unger, Virginia Institute of Marine Sciences, Gloucester Point, VA. It was dredged from the Chesapeake Bay at a location of 37° 11.1' min N, 76° 17.11' min W. The material was freeze-dried at Hanover Foods, Inc., Lancaster, PA, and transferred to the U.S. Geological Survey (USGS) in Denver, CO. The material was lightly deagglomerated and sieved through a 1 mm screen to remove coarse contaminants. The < 1 mm material was then ball milled to pass a (75 μm) 200 mesh and then blended in a single batch using a 10 ft^3 blender. The blended material was radiation sterilized at COBE Laboratories, Lakewood, CO, and then bottled.

Homogeneity Assessment: Prior to bottling, a preliminary evaluation of homogeneity was performed by the USGS using ten 50 g samples obtained from various locations in the blender. These homogeneity measurements were made by wavelength dispersive X-ray fluorescence (WDXRF) and inductively coupled plasma optical emission spectrometry (ICPOES) analyses. Final homogeneity evaluations of the bottled samples were made by WDXRF at the USGS, and by instrumental neutron activation analysis (INAA) at NIST. Except for lead, nickel, and antimony, a satisfactory level of homogeneity was observed for the certified elements, that is, no large sample-to-sample variations were observed over those expected from the analytical measurements. Some heterogeneity was observed for nickel and antimony, and because of the degree of heterogeneity observed for nickel and antimony, the concentrations of these elements have not been certified.

Table 1. Certified Values

Element	Mass Fraction, (%)	Element	Mass Fraction, (mg/kg)
Aluminum	2.297 ± 0.018	Arsenic	6.23 ± 0.21
Calcium	0.519 ± 0.020	Cadmium	0.148 ± 0.007
Iron	2.008 ± 0.039	Chromium	40.9 ± 1.9
Magnesium	0.388 ± 0.009	Copper	10.01 ± 0.34
Phosphorus	0.027 ± 0.001	Lead	11.7 ± 1.2
Potassium	0.864 ± 0.016	Manganese	234.5 ± 2.8
Silicon	40.00 ± 0.16	Selenium	0.193 ± 0.028
Sodium	0.741 ± 0.017	Vanadium	44.84 ± 0.76
Sulfur	0.352 ± 0.004	Zinc	48.9 ± 1.6
Titanium	0.456 ± 0.021		

Table 2. Noncertified Values

Element	Mass Fraction, (mg/kg)	Element	Mass Fraction, (mg/kg)
Antimony	0.3	Neodymium	15
Barium	210	Nickel	23
Beryllium	<1	Rubidium	38
Cerium	34	Scandium	5
Cobalt	5	Silver	<0.3
Gallium	5	Strontium	68
Lanthanum	17	Thallium	<0.5
Lithium	18	Thorium	5.8
Mercury	0.04	Tin	1
Molybdenum	1.8	Uranium	2.0

Participating NIST Analysts

E.S. Beary	P.J. Paulsen
D.A. Becker	M.S. Rearick
J.D. Fassett	T.A. Rush
K.M. Garrity	R. Saraswati
R.R. Greenberg	J.M. Smeller
W.R. Kelly	G.C. Turk
E.A. Mackey	R.D. Vocke
J.R. Moody	R.L. Watters, Jr.
K.E. Murphy	L.J. Wood

Cooperative Analyses for Certification were Performed in the Following Laboratories:

R. Presley; Department of Oceanography, Texas A & M, College Station, TX.
E. Crecelius; Battelle Pacific Northwest, Sequim, WA.
S.S. Berman, V. Boyko, J. Clancy, B. Lam, B. Methvan, J. McLaren, S. Willie; Institute for Environmental Chemistry, National Research Council of Canada, Ottawa, Ontario, Canada.
V.S. Zdanowicz; Northeast Fisheries Center, Sandy Hook Laboratory, Highlands, NJ.
P. Hanson and D. Evans;, Southeast Fisheries Center, Beaufort Laboratory, Beaufort, NC.
S. Wilson, P. Briggs, D. Siems, R. Knight and B. Arbogast; U.S. Geological Survey, Lakewood, CO.

Table 3. Analytical Methods Used for the Analysis of SRM 1646a

Element	Certification Methods*
Aluminum	**WDXRF;** INAA; FAAS; ICPOES
Antimony	INAA; RNAA; ICPMS
Arsenic	**HYDR; RNAA;** ICPOES; WDXRF; ETAAS
Barium	ICPOES
Beryllium	ICPMS; ICPOES
Bromine	EDXRF
Cadmium	**ID-TIMS; RNAA;** ETAAS; ICPOES
Calcium	**ICPOES; WDXRF**
Cerium	ICPOES
Chromium	**ICPMS**; **INAA**; FAAS; WDXRF; ETAAS; ICPOES
Cobalt	ICPOES; INAA
Copper	**ID-ICPMS**; **RNAA;** ETAAS; FAAS; WDXRF; ICPOES
Gallium	WDXRF; ICPOES
Iron	**XRF**; **INAA**; FAAS; ICPOES; EDXRF
Lanthanum	ICPOES
Lead	**ID-ICPMS**; ETAAS; WDXRF; ICPMS; ICOPES;
Lithium	ICPOES
Manganese	**WDXRF**; **ICPOES**; **INAA**; ETAAS; FAAS
Magnesium	**ICPOES**; **WDXRF**; **ED-ICPMS**
Mercury	CVAAS
Molybdenum	ID-ICPMS
Neodymium	ICPOES
Nickel	ID-ICPMS, ICPMS; FAAS; WDXRF; ETAAS; ICPOES; EDXRF
Phosphorus	**COLOR**; **ICPOES**; **WDXRF**
Potassium	**ICPOES**; **WDXRF**; **FES**
Rubidium	EDXRF
Scandium	ICPOES
Selenium	**RNAA**; **HYDR**; ICPMS
Silicon	**XRF**;**GRAV**
Silver	ETAAS, ICPOES
Sodium	INAA; **XRF**; ICPOES
Sulfur	**ID-TIMS**

Thallium	ETAAS; ICPMS
Thorium	ICPOES; DNAA
Tin	ETAAS; ICPMS; ICPOES
Titanium	**ICPOES**; **INAA**; **WDXRF**; ICPOES; EDXRF
Uranium	DNAA
Vanadium	**INAA**; **ICPOES**
Yttrium	INAA; ICPOES
Zinc	**ICPOES**; **INAA**; FAAS; WDXRF; ICPMS; EDXRF
Zirconium	EDXRF

*Methods in **bold** were used as certification methods; other methods listed were used to corroborate certification methods.

CVAAS - Cold Vapor Atomic Absorption Spectrometry
ETAAS - Electrothermal Atomic Absorption Spectrometry; Mixed Acid Digestion
FAAS - Flame Atomic Absorption Spectrometry; Mixed Acid Digestion
FES - Flame Emission Spectrometry; Mixed Acid Digestion
GRAV - Gravimetry; sodium carbonate fusion
HYDR - Hydride Generation Atomic Absorption Spectrometry; Mixed Acid Digestion
ICPOES - Inductively Coupled Plasma Optical Emission Spectrometry; Mixed Acid Digestion
ID-ICPMS - Isotope Dilution Inductively Coupled Plasma Mass Spectrometry; Mixed Acid Digestion
ID-TIMS - Isotope Dilution Thermal Ionization Mass Spectrometry; Mixed Acid Digestion
INAA - Instrumental Neutron Activation Analysis
RNAA - Radiochemical Neutron Activation Analysis; Mixed Acid Digestion
DNAA - Delayed Neutron Activation Analysis
WDXRF - Wavelength Dispersive X-ray Fluorescence on Fused Borate Discs
EDXRF - Energy Dispersive X-ray Fluorescence on Pressed Pellets

REFERENCES

[1] Paule, R.C. and Mandel, J., J. Res. Natl. Bur. Stds, 87:337 (1982).
[2] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9 1st ed. ISO, Geneva, Switzerland (1993).

Certificate Revision History: 20 February 2004 (Editorial changes); 02 September 1998 (Editorial revision of headings in Tables 1 and 2); 03 May 1998 (Addition of a noncertified value for silver); 26 January 1995 (Original certificate date).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.