

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 2704

Buffalo River Sediment

This Standard Reference Material (SRM) is intended primarily for use in the analysis of sediments, soils, or materials of a similar matrix. SRM 2704 is a freeze-dried river sediment that was sieved and blended to achieve a high degree of homogeneity.

The certified elements for SRM 2704 are given in Table 1. The values are based on measurements using two or more independent and reliable analytical methods. Noncertified values for a number of elements are given in Table 2 as additional information on the composition. The noncertified values should not be used for calibration or quality control. Analytical methods used for the characterization of this SRM are given in Table 3 along with analysts and cooperating laboratories. All values (except for carbon) are based on measurements using a sample weight of at least 250 mg. Carbon measurements are based on 100 mg samples.

Notice and Warnings to Users: This certification is valid for 5 years from the shipping date. Should any of the certified values change before the expiration of the certification, purchasers will be notified by NBS.

Stability: This material was radiation sterilized to reduce the rate of any biodegradation. However, its stability has not been rigorously assessed. NBS will monitor this material and will report any substantive changes in certification to the purchaser.

Use: A minimum sample weight of 250 mg (dry weight - see Instructions for Drying) should be used for analytical determinations relating to the certified values on this certificate.

Sample preparation procedures should be designed to effect complete dissolution. If volatile elements (i.e., Hg, As, Se) are to be determined, precautions should be taken in the dissolution of SRM 2704 to avoid volatilization losses.

Statistical consultation was provided by S.B. Schiller and K.R. Eberhardt of the Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of M.S. Epstein and B.I. Diamondstone of the Inorganic Analytical Research Division.

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 T.E. Gills.

Gaithersburg, MD 20899
June 1, 1988

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

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Instructions for Drying: When nonvolatile elements are to be determined, samples should be dried for 2 hours at 110 °C. Volatile elements (i.e., Hg, As, Se) should be determined on samples as received; separate samples should be dried as previously described to obtain a correction factor for moisture. Correction for moisture is to be made to the data for volatile elements before comparing to the certified values. This procedure, which was used for the certification of volatile elements, ensures that these elements are not lost during drying. The approximate weight loss on drying has been found to be 0.8%.

Source and Preparation of Material: The river sediment for this SRM was collected from the Buffalo River in the area of the Ohio Street Bridge, Buffalo, N.Y. The U.S. Army Corps of Engineers, under contract to the NBS, collected and screened approximately 908 kg of river sediment and placed it in six 55 gallon Teflon lined drums. The drums were loaded onto a refrigerated truck and transported to the Technimed Corporation for freeze-drying. The freeze-dried sediment was shipped to an NBS contractor's laboratory where it was screened and passed through a 100 mesh sieve (nominal sieve opening of 150 μm) and retained on a 400 mesh sieve (nominal sieve opening of 38 μm). The sieved sediment was returned to NBS, radiation sterilized, blended, and bottled into 50-g units.

Analysis: The homogeneity of the bottled units was assessed using x-ray fluorescence spectrometry. Duplicate one gram samples from 8 randomly selected bottles were analyzed for the following elements: Al, Si, K, Ca, Ti, Fe, Zn, Sr, P, Mn, Rb, and Zr. No statistically significant differences in the composition of samples within or between bottles were observed relative to the uncertainty of the XRF measurements, which is less than 0.4%. Sample inhomogeneity of about 4% for lead was observed in measurements on 250 mg samples by thermal-ionization isotope dilution mass spectrometry. Sample inhomogeneity for lead is reflected in the uncertainty limits placed on the certified value for lead.

Table 1. Certified Values

<u>Element</u>	<u>Wt. %</u>	<u>Element</u>	<u>Wt. %</u>
Aluminum	6.11 $\pm$ 0.16	Phosphorus	0.0998 $\pm$ 0.0028
Calcium	2.60 $\pm$ 0.03	Potassium	2.00 $\pm$ 0.04
Carbon	3.348 $\pm$ 0.016	Silicon	29.08 $\pm$ 0.13
Iron	4.11 $\pm$ 0.10	Sodium	0.547 $\pm$ 0.014
Magnesium	1.20 $\pm$ 0.02	Titanium	0.457 $\pm$ 0.018

<u>Element,</u>	<u>$\mu\text{g/g}$</u>	<u>Element,</u>	<u>$\mu\text{g/g}$</u>
Antimony	3.79 $\pm$ 0.15	Lead	161 $\pm$ 17
Arsenic	23.4 $\pm$ 0.8	Manganese	555 $\pm$ 19
Barium	414. $\pm$ 12	Mercury	1.44 $\pm$ 0.07
Cadmium	3.45 $\pm$ 0.22	Nickel	44.1 $\pm$ 3.0
Chromium	135 $\pm$ 5	Thallium	1.2 $\pm$ 0.2
Cobalt	14.0 $\pm$ 0.6	Uranium	3.13 $\pm$ 0.13
Copper	98.6 $\pm$ 5.0	Vanadium	95 $\pm$ 4
		Zinc	438 $\pm$ 12

Certified Values and Uncertainty: The certified values are weighted means of results from two or more analytical techniques. The weights for the weighted means were computed according to the iterative procedure of Paule and Mandel (NBS Journal of Research 87, 1982, pp. 377-385). Each uncertainty is the sum, in quadrature, of the half-width of a 95% expected tolerance interval and an allowance for systematic error among the methods used. The interval, the endpoints of which are the certified value minus and plus the uncertainty, respectively, will cover the concentration in a minimum sample weight of 250 mg of this SRM for at least 95% of the samples with 95% confidence.

Table 2. Noncertified Values

<u>Element</u>	<u>Content, Wt. %</u>	<u>Element</u>	<u>Content $\mu\text{g/g}$</u>
Chlorine	(<0.01)	Bromine	(7)
Sulfur	(0.4)	Cerium	(72)
		Cesium	(6)
		Dysprosium	(6)
		Europium	(1.3)
		Gallium	(15)
		Hafnium	(8)
		Iodine	(2)
		Lanthanum	(29)
		Lithium	(50)
		Lutetium	(0.6)
		Rubidium	(100)
		Scandium	(12)
		Selenium	(1.1)
		Samarium	(6.7)
		Strontium	(130)
		Tin	(9.5)
		Thorium	(9.2)
		Ytterbium	(2.8)
		Zirconium	(300)

Noncertified Values: Noncertified values are provided for information only. An element concentration value may not be certified, if a bias is suspected in one or more of the methods used for certification, or if two independent methods are not available. Certified values for some of these elements will eventually be provided in a revised certificate when more data is available.

Table 3.

Analytical Methods Used for the Analysis of SRM 2704

<u>Laboratory^a</u>	<u>Method^b</u>	<u>Sample Preparation</u>	<u>Elements Determined</u>
LANL	IENAA	Direct - No preparation	As, Br, I, Ga
	INAA		Co, Cr, Eu, Fe, Rb, Sc, Th, Yb
MCL/PSU	COLOR	Closed Teflon bomb digestion with HNO ₃ , HF, and HClO ₄ for 18 hrs at 110 °C.	P
	DCP		Li
	DCP	Lithium metaborate fusion.	Ti, Fe, Mg, Ca, Mn, Sr, Ba, Na, K
NBS	COUL	Direct - No preparation	C
	IGF	Direct - No preparation	C, S
	CVAAS	Reflux acid digestion	Hg
	GFAAS	Open beaker acid digestion with HF, HNO ₃ , and HClO ₄ .	Cd
	HGAAS	Reflux acid digestion with HNO ₃ , H ₂ SO ₄ , and HClO ₄ .	As, Se
	IC	High-pressure calorimetric oxygen bomb.	Cl, S
	TI-IDMS	Open beaker acid digestion with HF, HClO ₄ , and HNO ₃ with subsequent ion exchange separation.	Pb, Ti, U
	ICP DCP	Open beaker acid digestion with HF, HClO ₄ , and HNO ₃ with ignition and acid digestion of residue.	Al, Ca, Cu, Mg, P, V Co, Na, Ni, Sr
	DCP FES FAAS	Lithium metaborate fusion in platinum crucibles with dissolution in 4% HNO ₃ .	Al, Ba, Ca, Cr, Fe, Mg, Si, Ti, V Al, K Mn, Zn
	INAA	Direct - No preparation	As, Cs, Cr, Co, Fe, K, Sb, U, Zn
	LEI GFAAS	Open beaker acid digestion with HF, HClO ₄ , and HNO ₃ with subsequent Chelex-100 separation.	Mn, Ni, Pb Cd
	GRAV XRF	Sodium carbonate fusion. Lithium tetraborate fusion.	Si Al, Si, P, K, Ca, Ti, Mn, Fe, Zn, Sr, Rb, Zr

	POLAR	Open beaker acid digestion with HF, HClO ₄ , and HNO ₃ with subsequent liquid-liquid extraction.	Cu, Pb, Zn
NRCC	CVAAS	Reflux acid digestion of sample in 250-mL quartz Erlenmeyer flasks with a mixture of HNO ₃ and HClO ₄ .	Hg
	ICP-IDMS	Microwave acid digestion	Ni, Cu, Zn, Pb, Cd, Sb, Sn, Ti, U
	GFAAS	Microwave acid digestion	Cd, Pb, Co, Cu, Ni, As, Sb
	XRF	Mixed tetraborate/carbonate fusion.	S
ORAU	INAA	Direct - No preparation	Al, Ca, Fe, K, Na, Ti As, Ba, Ce, Co, Cr, Cs, Dy, Eu, Hf, Lu Mn, Rb, Sb, Sc, Sm, Th, U, V, Yb, Zn
	CVAAS	Reflux acid digestion	Hg

^aLaboratory abbreviations:

LANL	=	Los Alamos National Laboratory, Los Alamos, NM 87545
MCL/PSU	=	Mineral Composition Laboratory, The Pennsylvania State University, University Park, PA 16802
NBS	=	Center for Analytical Chemistry, National Bureau of Standards, Gaithersburg, MD 20899
NRCC	=	Analytical Chemistry Division, National Research Council of Canada, Ottawa, Canada K1A0R6
ORAU	=	Oak Ridge Associate Universities, Oak Ridge, TN 37831-0117

^bMethod abbreviations:

COLOR	=	Colorimetry, Photometry, Spectrophotometry
COUL	=	Coulometry
CVAAS	=	Cold-Vapor Atomic Absorption Spectrometry
DCP	=	Direct-Current Plasma Emission Spectrometry
FAAS	=	Flame Atomic Absorption Spectrometry
FES	=	Flame Emission Spectrometry
GFAAS	=	Graphite Furnace Atomic Absorption Spectrometry
GRAV	=	Gravimetry
HGAAS	=	Hydride-Generation Atomic Absorption Spectrometry
IC	=	Ion Chromatography
ICP	=	Inductively-Coupled Plasma Emission Spectrometry
ICP-IDMS	=	Isotope-dilution Inductively-Coupled Plasma Mass Spectrometry
IGF	=	Inert Gas Fusion with Infrared Detection
INAA	=	Instrumental Neutron Activation Analysis
IENAA	=	Instrumental Epi-Thermal Neutron Activation Analysis
LEI	=	Laser-Enhanced Ionization Spectrometry (acid digestion/separation)
POLAR	=	Polarography
TI-IDMS	=	Thermal-Ionization Isotope Dilution Mass Spectrometry
XRF	=	X-Ray Fluorescence Spectrometry

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