

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1645

River Sediment

This Standard Reference Material (SRM) is intended for use for the calibration of apparatus and the verification of methods used in the analysis of river sediments and material with a similar matrix.

Certified Values of Constituent Elements: The certified values for the constituent elements are shown in Table 1. The analytical techniques used and the names and affiliations of the analysts are shown in Table 3. Certified values are based on results obtained by reference methods of known accuracy and analyses performed by two or more analysts; or alternatively, from results obtained by two or more independent, reliable analytical methods. Noncertified values are given for information only in Table 2. All values are based on measurements made on a dried sample of at least 100 mg for all constituents except iron and chromium for which a 1-g sample was used.

Notice and Warnings to Users:

Expiration of Certification: This certification is invalid 5 years from the date of purchase.

Stability: This material has been freeze-dried and is essentially free of moisture. However, its stability has not been rigorously assessed. NBS will continue to monitor this material and if substantive changes in certification occur the purchasers will be notified. The material should be kept in its original bottle and stored at temperatures between 10-30 °C. The material should be dried without heat to a constant weight before using. Recommended procedures for drying are: (1) drying for 24 hours using a cold trap at or below -50 °C and a pressure not greater than 30 Pa (0.2 mm Hg); (2) drying for 24 hours in a desiccator over P₂O₅ or Mg (ClO₄)₂.

Use: Material of this kind is intrinsically heterogeneous. Consequently, the analyst should endeavor to minimize any segregation by thoroughly mixing the contents of the bottle by shaking and/or rolling before each use. In addition, when taking a portion for analysis, the analyst should strive to remove as representative a sample as possible.

Source and Preparation of Material: The material for this SRM was prepared from material dredged from the bottom of the Indiana Harbor Canal near Gary, Indiana. The material was screened to remove foreign objects, freeze-dried, and sieved to pass a No. 80 (180 μm) screen. The material was thoroughly mixed in a V-blender and bottled. The bulk material was radiation-sterilized to minimize alteration due to biological activity.

The collection, freeze-drying and homogenization of this SRM were performed under the supervision and direction of H.L. Rook, Gas and Particulate Science Division.

The overall direction and coordination of the technical measurements leading to certification were performed under the chairmanship of J.K. Taylor, Center for Analytical Chemistry.

The technical and support aspects involved in the preparation, current and previous certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T.E. Gills and W.P. Reed.

Supplemental Information

Note: The following values are not certified because they are not based on the results of either a reference method or of two or more independent methods. These values are included for information only.

Table 2. Noncertified Values for Constituent Elements

<u>Element</u>	<u>Content wt. Percent</u>	<u>Element</u>	<u>Content µg/g</u>
Calcium	(2.9)	Antimony	(51)
Fluorine	(0.09)	Arsenic	(66)
Sulfur	(1.1)	Lanthanum	(9)
		Scandium	(2)
		Selenium	(1.5)

Additional Information: The values listed below are based on measurements made in one laboratory and while no reason exists to suspect systematic bias in these numbers, no attempt was made to evaluate such bias attributable to either the method or the laboratory. The method used for each set of measurements is also listed. The indicated uncertainties are two times the standard deviation of the mean. These values are included for information only.

Table 3

<u>Constituent</u>	<u>Content wt. percent</u>
Kjeldahl Nitrogen	(0.0797% ± 0.0048)
Total Phosphorus	(0.051% ± 0.001) [1]
Loss on Ignition (800 °C)	(10.72% ± 0.28)
Oil and Grease (Freon)	(1.71% ± 0.26) [3]
Chemical Oxygen Demand (Dichromate)	(149,400 mg/kg ± 9,000)[2]

References

1. ASTM Method E-350
2. Standards Methods for the Examination of Water and Waste Water, 14th Edition (1975), Section 508, pp 550.
3. Ibid., Section 502, pp 518.

Homogeneity Assessment and Certification: The homogeneity of this material was established using a minimum sample size of 100 milligrams for all constituents except iron and chromium for which the sample size was 1.0 gram.

Randomly selected bottles were used for the analytical measurements. Each analyst examined at least 6 different bottles, some of them measuring replicate samples from each bottle. Accordingly, it is believed that all bottles of this SRM have substantially the same composition. Measurements and calibrations were made to reduce random and systematic errors to no more than one percent, relative.

Table 1. Certified Values of Constituent Elements

<u>Major Constituents</u>		<u>Minor Constituents</u>	
<u>Element</u>	<u>Content wt. percent^a</u>	<u>Element</u>	<u>Content wt. percent^a</u>
Aluminum ^b	2.26 ± 0.04	Magnesium ^b	0.74 ± 0.02
Chromium	2.96 ± 0.28	Sodium ^b	0.54 ± 0.01
Iron	11.3 ± 1.2	Zinc	0.172 ± 0.017
Potassium ^b	1.26 ± 0.05		

<u>Trace Constituents</u>			
<u>Element</u>	<u>Content μg/g^a</u>	<u>Element</u>	<u>Content μg/g^a</u>
Cadmium	10.2 ± 1.5	Nickel	45.8 ± 2.9
Copper	109 ± 19	Thallium	1.44 ± 0.07
Cobalt ^b	10.1 ± 0.6	Thorium	1.62 ± 0.22
Lead	714 ± 28	Uranium	1.11 ± 0.05
Manganese	785 ± 97	Vanadium	23.5 ± 6.9
Mercury	1.1 ± 0.5		

^aThe uncertainties of the certified values for the elements, except those noted by superscript "b," include those errors associated with both measurement and material variability. They represent the 95 percent tolerance limits for an individual sub-sample, i.e., 95 percent of the sub-samples from a unit of this SRM would be expected to have a composition within the indicated range of values 95 percent of the time.

^bThese elements are certified as a part of the NBS update certification program. For each element a "best value" is given based on all methods of measurement that were used as well as a *standard error* of this value. Both are based on considerations of variability both within and between analytical methods.

Analysts

NBS Center for Analytical Chemistry

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|--------------------|---------------------|
| 1. T. J. Brady | 10. R. R. Greenberg |
| 2. E. R. Deardorff | 11. S. H. Harrison |
| 3. L. P. Powell | 12. G. J. Lutz |
| 4. M. S. Epstein | 13. L. A. Machlan |
| 5. R. Filby | 14. E. J. Maienthal |
| 6. M. Gallorini | 15. T. C. Rains |
| 7. E. L. Garner | 16. H. L. Rook |
| 8. T. E. Gills | 17. T. A. Rush |
| 9. J. W. Gramlich | 18. W. P. Schmidt |

Cooperators

19. L. Kosta (Nuclear Chemistry Section, Josef Stefan Institute, Ljubljana, Yugoslavia)

Table 3A Methods and Analysts

Method/ Element	A	B	C	D	E	F
Aluminum	•		•		•	
Arsenic			•			
Antimony	•					
Cadmium			•	•		
Calcium			•			
Chromium		•	•			
Cobalt			•		•	
Copper		•	•			
Fluorine						•
Iron	•		•			
Lanthanum			•			
Lead		•		•		
Magnesium	•				•	
Manganese		•	•			
Mercury	•		•			
Nickel		•		•		
Potassium	•		•			
Scandium			•			
Selenium	•					
Sodium	•		•			
Sulfur						•
Thallium		•				
Thorium		•				
Uranium		•				
Vanadium	•		•			
Zinc	•			•		

Analytical Methods

- A. Atomic Absorption Spectrometry
- B. Isotope Dilution Mass Spectrometry
- C. Neutron Activation Analysis
- D. Polarography
- E. D. C. Plasmas Atomic Emission Spectrometry
- F. Ion Chromatography