National Institute of Standards & Technology

Report of Investigation

Reference Materials 8406, 8407, and 8408

Mercury in Tennessee River Sediments

These Reference Materials (RM's) are intended primarily for use in the determination of mercury in sediments, soils or materials of a similar matrix. These RM's provide samples in order that investigators in different laboratories can be assured that they are analyzing the same homogeneous material.

The RM's consist of 25 gms each of three different Tennessee River sediments that were passed through a 100 mesh size screen. The three RM's differ in mercury content and should prove useful as control materials for the concentration range from 0.06 μg/g to 100 μg/g.

Background

Sediments as a matrix are among the most useful types of reference materials for inorganic elemental analysis. The complex composition of sediments coupled to the volatility of mercury makes great demands on methods of dissolution and analysis. It is very difficult to assess the accuracy of a method or technique for the determination of mercury without the availability of reference materials whose mercury content has been determined by employing reliable independent methods.

Material Source

RM's 8406, 8407, and 8408 were produced in collaboration with the Oak Ridge Y-12 Plant*, Martin Marietta Energy Systems, Inc., Oak Ridge, Tennessee, and with the Oak Ridge Associated Universities, Oak Ridge, Tennessee.

The base materials were obtained from the flood plain of East Fork Poplar Creek which passes through the city of Oak Ridge, Tennessee. The mercury present in RM's 8407 and 8408 is a result of mercury discharges from the Oak Ridge Y-12 plant during the 1950's and 1960's. RM 8406, "a background soil", was obtained from the flood plain of Big Creek which floods into Norris Lake near LaFollette, Tennessee. Norris Lake floods the sample area several months each year.

*Operated for the U.S. Department of Energy by Martin Marietta Energy Systems, Inc., under contract DE-AC05-840R21400.

Gaithersburg, MD 20899
June 15, 1990

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Preparation

All samples were collected in early November 1985. Each sample was individually forced-air dried at just above ambient temperature (30 °C) in a large laboratory oven. The average drying time was three days per sample after which the sediments were transferred into polyethylene bags where they were pulverized using rubber mallets in preparation for sieving. The sediments were sieved through 100 mesh size stainless steel screens. The less-than-100-mesh fraction was collected and blended in a V-shell type blender. A small sample "lot" was shipped to NIST for subsequent bottling and analysis.

Analysis

The recommended mercury values are given in Table 1.

The values are based on cold-vapor atomic absorption spectrometry (CVAAS) and instrumental neutron activation analysis (INAA) with SRM 2704, Buffalo River Sediment, used as a control. All values are reported on a dry weight basis.

A minimum sample weight of 1 gm should be used for any analytical determination to be related to the recommended values listed in Table 1. Due to the volatile nature of mercury, sample preparation should be to effect complete dissolution of the sediments without volatilization losses of mercury.

Table 1

Mercury in Tennessee River Sediments

RM	Recommended Value
8406	(0.06 μg/g)
8407	(50 ± 2 μg/g)
8408	(107 ± 2 μg/g)

The uncertainty of the recommended value is based on judgment and is intended to represent the range of results obtained on these materials using two different methods of analysis. The recommended value for RM 8406 is based on only one method (CVAAS) and therefore no range of results is provided.

The recommended mercury values and matrix characterization are based on analyses by T. A. Butler, T. A. Rush and R. L. Watters, Jr., of the NIST Inorganic Analytical Research Division; G. Gleason of the Oak Ridge Associated Universities, Oak Ridge, Tennessee; and L. E. White of the Oak Ridge Y-12 Plant, Martin Marietta Energy Systems, Inc., Oak Ridge, Tennessee.

Statistical consultation was provided by R. C. Paule of the National Measurement Laboratory, NIST.

The technical and support aspects involved in the preparation, and issuance of this Reference Material were coordinated through the Standard Reference Materials Program by T. E. Gills.

Supplemental Information

The information given in Table 2 is to provide additional information on each SRM matrix. These are not recommended values as they were determined using a single technique, instrumental neutron activation analysis, under highly controlled conditions afforded by a californium-252 neutron facility.

Table 2

Note: Results in μg/g unless otherwise noted.

Element	RM 8406	RM 8407	RM 8408
Ag	< 0.6	10	5
Al(wt.%)	5.5	4.4	6.1
As	10	7.8	7.3
Au	---	0.06	0.05
Ba	400	400	360
Br	1	5.0	6.5
Ce	---	86	92
Co	18	15	19
Cr	67	98	88
Cs	3.9	3.6	3.8
Dy	5.4	4.4	5.3
Eu	1.2	0.9	1.3
Fe(wt.%)	2.96	2.42	2.6
Ga	13	11	---
Hf	13	5	18
Ho	1.2	---	---
I	---	---	10
K(wt.%)	1.5	1.7	1.5
La	36	27	30
Lu	0.61	.48	---
Mn	430	970	1400
Na	1900	1200	1200
Nd	35	---	---
Pb	40	96	80
Rb	82	67	60
Sb	0.90	0.61	---
Sc	10.7	6.9	7.6
Se	< 1	---	---
Sm	6.6	7.2	7.0
Sr	80	74	60
Ta	0.84	0.9	---
Tb	1.1	---	---
Th	10	12	15
Ti(wt.%)	0.45	0.44	0.56
U	4.0	30	23
V	76	50	60
W	1	4.0	5.4
Yb	4.0	2.8	2.9
Zn	145	230	160
Zr	740	500	650