



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2781

Domestic Sludge

This Standard Reference Material (SRM) is intended primarily for use in the evaluation of methods used for the analysis of sludges and other materials of a similar matrix. SRM 2781 is a dried and pulverized domestic sludge. A unit of SRM 2781 consists of 40 g of the dried material.

Certified Values: The certified values for 10 elements are listed in Table 1 and are based on concordant results from two or more analytical methods.

Analytical methods used for the characterization of this SRM are given in Table 3. All values are reported as mass fractions [1], on a dry mass basis and are based on measurements using a sample mass of at least 100 mg.

Noncertified Values: Noncertified values for eleven additional elements are listed in Table 2 and were derived from a combination of results from NIST and collaborating laboratories. These results may have sources of bias yet to be investigated and do not meet NIST criteria for certification.

Expiration of Certification: This certification is valid for five years from the date of shipment from NIST. However, the certification will be nullified if the SRM is contaminated or modified. Should any of the values change before the expiration of the certification, purchasers will be notified by NIST. Return of the attached registration card will facilitate notification.

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

Use: A minimum sample mass of 100 mg (dry mass - see Instructions for Drying) should be used and sample preparation procedures should be designed to effect complete dissolution for analytical determinations to be related to the certified values provided.

Instructions for Drying: When nonvolatile elements are to be determined, samples should be vacuum dried at room temperature for 24 h or oven dried for 2 h at 110 °C. Volatile elements (e.g., arsenic, mercury, and selenium) should be determined on samples as received; separate samples should be dried according to these instructions to obtain a correction factor for moisture. Moisture corrections are then made to measurement values before comparing them to the certified values. [Note that the mass loss on drying at the time of certification was found to be in the range of 4.7 % to 6.6 % when using the recommended drying procedures.]

Statistical consultation was provided by L.M. Gill of the NIST Statistical Engineering Division.

The overall direction and coordination of the analyses were under the chairmanship of J.D. Fassett of the NIST Analytical Chemistry Division.

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by J.S. Kane. Revision of this certificate was coordinated through the Standard Reference Materials Program by B.S. MacDonald.

Gaithersburg, MD 20899
Certificate Issue Date: October 25, 1996
(Revision of certificate dated 6-22-95 to include addendum)

Thomas E. Gills, Chief
Standard Reference Materials Program

Source and Preparation of Material: The U.S. Geological Survey (USGS), under contract to NIST, obtained partially dehydrated sewage cake material from the Metropolitan Denver Sewage Disposal District No. 1. The material (approximately 182 kg) was placed in plastic-lined drums and transported to the USGS facilities in Lakewood, CO for processing. It was dried at ambient temperature in a forced air chamber, ground to pass a 74 m. (200 mesh) sieve, blended for 24 h to assure homogeneity of the pulverized material after which test samples were taken from the blender for preliminary homogeneity analyses. The material was then radiation sterilized. The sterilized material was shipped in bulk to NIST, where the material was bottled in 40 g units after reblending for 4 h.

Analysis: The homogeneity was assessed at USGS on 10 replicate samples of bulk material for over 40 elements using x-ray fluorescence (XRF) and/or inductively coupled plasma atomic emission spectrometry (ICP-AES). Homogeneity was further assessed during certification analysis; at sample sizes of 100 mg or greater. No sample-to-sample variations in excess of those expected from the analytical measurements were detected.

Certified Values and Uncertainties: The certified values are the means of the results from two or more independent analytical methods as described by Schiller and Eberhardt [2]. The uncertainty is based on a 95 % confidence interval for the true value, and includes an allowance for differences between the analytical methods used [3].

Table 1. Certified Mass Fractions

Element	Mass Fraction (in mg/kg)	Element	Mass Fraction (in %)
Arsenic	7.82 ± 0.28	Nitrogen	4.78 ± 0.11
Cadmium	12.78 ± 0.72		
Copper	627.4 ± 13.5		
Lead	202.1 ± 6.5		
Mercury	3.64 ± 0.25		
Molybdenum	46.7 ± 3.2		
Nickel	80.2 ± 2.3		
Selenium	16.0 ± 1.6		
Zinc	1273 ± 53		

Noncertified Values and Uncertainties: The noncertified values are the means of the results from two or more independent analytical methods as described by Schiller and Eberhardt [2]. The uncertainty is based on a 95 % confidence interval for the mean, and includes an allowance for differences between the analytical methods used [3]. As the accuracy of the measurements could not be assessed from the data, the uncertainty associated with a noncertified value may fail to include all sources of uncertainties and may represent only a measure of the precision of the measurement methods.

Table 2. Noncertified Mass Fractions

Element	Mass Fraction (in mg/kg)	Element	Mass Fraction (in %)
Chromium	202 ± 9	Aluminum	1.6 ± 0.1
Silver	98 ± 8	Calcium	3.9 ± 0.1
		Iron	2.8 ± 0.1
		Magnesium	0.59 ± 0.04
		Phosphorus	2.42 ± 0.09
		Potassium	0.49 ± 0.03
		Silicon	5.1 ± 0.2
		Sodium	0.21 ± 0.02
		Titanium	0.32 ± 0.03

Table 3. Methods used for the Analysis of SRM 2781*

Aluminum	INAA, ICP-AES, XRF
Arsenic	RNAA, Hyd. AAS, INAA
Cadmium	ID-ICPMS, PGAA, RNAA, INAA, TXRF
Calcium	INAA, TXRF, ICP-AES, XRF
Chromium	INAA, TXRF, ICP-AES
Copper	ID-ICPMS, RNAA, INAA, TXRF
Iron	INAA, TXRF, ICP-AES, XRF
Lead	ICP-AES, ID-ICPMS, TXRF
Magnesium	INAA, ICP-AES, XRF
Mercury	FIA-CV-AAS, RNAA, INAA
Molybdenum	ID-ICPMS, ICP-AES, TXRF, INAA
Nickel	ICP-AES, ID-ICPMS, INAA, TXRF
Nitrogen	Kjeldahl, PGAA
Phosphorus	Color, ICP-AES, XRF
Potassium	INAA, TXRF, ICP-AES
Selenium	Hyd. AAS, RNAA, INAA, TXRF
Silicon	XRF
Silver	RNAA, INAA, TXRF
Sodium	INAA, ICP-AES, XRF
Titanium	INAA, TXRF, XRF
Zinc	ICP-AES, ID-ICPMS, INAA, TXRF

*Methods used for establishment of certified values are shown in bold-face type; methods used for information only values or to corroborate certified values are not in bold.

Methods

FIA-CV-AAS	Flow injection analysis cold vapor atomic absorption spectrometry
Hyd. AAS	Hydride generation atomic absorption spectrometry
ICP-AES	Inductively coupled plasma atomic emission spectrometry
ID-ICPMS	Isotope dilution inductively coupled plasma mass spectrometry
INAA	Instrumental neutron activation analysis
PGAA	Prompt gamma activation analysis
RNAA	Radiochemical neutron activation analysis
TXRF	Total reflectance x-ray fluorescence
XRF	Wavelength dispersive x-ray fluorescence

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REFERENCES

- [1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).
- [2] Schiller, S.B. and Eberhardt, K.R., Combining Data from Independent Chemical Analysis Methods, *Spectrochimical Acta*, **46B** (12), pp 1607-1613, (1991).
- [3] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, 1st Ed. ISO, Geneva, Switzerland, (1993): see also Taylor, B.N. and Kuyatt, C.E., "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results," NIST Technical Note, 1297, U.S. Government Printing Office, Washington D.C., (1994).

Addendum to Standard Reference Material[®] 2781

Domestic Sludge

Leachable Mass Fractions Using U.S. EPA and NJDEP Methods for Flame Atomic Absorption Spectrometry and Inductively Coupled Plasma Atomic Emission Spectrometry

The certified concentration values of constituent elements in this certificate are given as total mass fractions. To obtain total mass fractions, either subsamples of the SRM must be completely decomposed, or the sample must be analyzed directly in its solid form. For mixed acid dissolution, hydrofluoric acid must be included in the acid mixture to totally dissolve siliceous material present in sludge.

For a number of environmental monitoring purposes, acid extractable mass fractions of elements are often used rather than total mass fractions. Acid extractable methods do not necessarily result in total decomposition of the sludge. It should be noted that results obtained using acid leach conditions are often depicted in reports as total results. However, reported acid labile or extractable mass fractions of elements are generally lower than total mass fractions. Results are often presented as measured mass fractions in the leachate in comparison to the total or certified mass fractions. The recovery of an element as a percent of total is a function of several factors such as the mode of occurrence in the sample, leach medium, leach time, temperature conditions, and pH of the sample-leach medium mixture [1].

In its monitoring programs, the U.S. Environmental Protection Agency (U.S. EPA) has established a number of leach methods, such as Methods 3015, 3050, and 3051 [2,3] for the determination of acid labile or extractable mass fractions of elements. The New Jersey Department of the Environment (NJDEP) has developed its own leach method, NJDEP 100 for state use [4]. The NJDEP and the U.S. EPA prepared samples of SRM 2781 using the NJDEP 100 method and EPA Methods 3050 and 3051 and analyzed the resulting leachates by flame atomic absorption spectrometry (FAAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES).

Reference values have been established for the acid-leachable mass fractions of several elements in SRM 2781. These values are the means of all results from the different leach measurement methods and combinations used. The reference values are listed in Table 1, along with their uncertainties which are based on 95 % confidence intervals of the means of results. For some of the elements (copper, iron, silver, vanadium), no statistically significant differences were found among results from the two laboratories using three or four combinations of sample preparation and instrumental measurement techniques (NJDEP 100 - FAAS; NJDEP 100 - ICP-AES; EPA 3050 - ICP-AES; EPA 3051 - ICP-AES). For all other elements, statistically significant between-laboratory differences were identified and are included in the stated uncertainties. These differences are small in comparison to control limits for many environmental monitoring programs. Therefore, the reference values are meaningful, despite the between-laboratory differences found.

Reference Values and Uncertainties: The reference values given in Table 1 are not NIST certified but are provided as a reference for U.S. EPA 3050 and 3051, and NJDEP 100 methods. The uncertainties are based on a 95 % confidence interval for the mean and include an allowance for differences between the analytical methods used.

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Table 1. Reference Leach Values for SRM 2781

Element	Leachable mass fraction (in mg/kg)		% Leach Recovery*
Aluminum	8 040	± 980	50
Barium	570	± 65	---
Cadmium	11	± 2	86
Calcium	36 440	± 1 830	93
Chromium	143	± 14	71
Copper	601	± 16	96
Iron	24 300	± 2 100	87
Lead	183	± 15	91
Magnesium	4 850	± 290	82
Manganese	745	± 33	---
Nickel	72.3	± 6.3	90
Silver	86.3	± 1.7	88
Vanadium	81.9	± 3.8	---
Zinc	1 120	± 34	88

$$* \% \text{ Leach Recovery} = 100 \times \frac{\text{Leach Value}}{\text{Certified or Noncertified Value}}$$

Cooperating Analysts and Laboratories:

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REFERENCES

- [1] Kane, J.S., Leach Data vs Total: Which is Relevant for SRMs, Fresenius J. Anal. Chem. 352: pp 209-213, (1995).
- [2] U.S. EPA 1991 Code of Federal Regulations, Title 40, Part 136, Paragraph 33.
- [3] Federal Register 1-13-95 SW-846 update #2, Final.
- [4] New Jersey Administrative Code, 1994. N.J.A.C. 7:14-4.