



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 2782

Industrial 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. A unit of SRM 2782 consists of 70 g of dried, pulverized, and radiation sterilized industrial sludge.

Certified Concentration Values: The certified values, expressed as mass fractions on a dry basis, for ten elements are provided in Table 1. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST [1]. The certified values in this SRM are based on either a single NIST primary method or on concordant results from NIST and two or more collaborating laboratories.

Reference Concentration Values: Reference values, expressed as mass fractions on a dry basis, for 16 additional elements are provided in Table 2. Reference values are noncertified values that are the best estimate of the true value; however, the values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty [1]. The reference values in this SRM are based on a combination of results from collaborating laboratories.

Information Concentration Values: Information values are listed in Table 3. These values may be of interest to the SRM user and represent results from one or more collaborating laboratories. Insufficient information is available to adequately assess the uncertainty associated with these values [1].

Analytical methods used for the characterization of this SRM are given in Table 4. All values are reported as mass fractions [2], on a dry mass basis and are based on measurements using a sample mass of at least 100 mg.

Leachable Mass Fractions: Reference values for 14 acid-leachable mass fractions using U.S. Environmental Protection Agency (EPA) 3050, 3051, and New Jersey Department of the Environment (NJDEP) 100 methods are contained in Appendix A. These values are the means of all results supplied by cooperating EPA and NJDEP laboratories for the different leach and measurement methods used.

Expiration of Certification: The certification of **SRM 2782** is valid, within the measurement uncertainty specified, until **1 October 2015**, provided the SRM is handled and stored in accordance with instructions given in this certificate (See "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

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 and G.C. Turk of the NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Robert L. Watters, Jr., Chief
Measurement Services Division

Gaithersburg, MD 20899
Certificate Issue Date: 09 September 2011
Certificate Revision History on Page 5

Source and Preparation of Material: The base material for SRM 2782 was obtained as effluence from an industrial site in northern New Jersey involved in pharmaceutical research and was collected before treatment with the assistance of the New Jersey Department of Environmental Protection. The base material was freeze dried at a commercial facility under contract to NIST, and transferred to the U.S. Geological Survey (USGS) in Denver, CO. At USGS the material was ball milled to pass a 75 μm (200 mesh) sieve and then blended into a single lot using a 0.2832 m^3 (10 ft^3) blender. The blended material was radiation sterilized and then bottled at USGS.

Analysis: The homogeneity was assessed at USGS on 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 with sample sizes of 100 mg or greater. No sample-to-sample variations in excess of those expected from the analytical measurements were detected.

Instructions for 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: The mass loss on drying at the time of certification was found to be in the range of 2.1 % to 3.2 % when using the recommended drying procedures.]

Certified Values and Uncertainties: The certified values for Cd, Cu, Pb, Mo and Ni are based on a primary NIST method. The certified values for the remaining elements are the results from two or more independent analytical methods as described by Schiller and Eberhardt [3].

The uncertainty in the values certified by a NIST primary method is expressed as an expanded uncertainty, U , and is calculated according to the method described in the ISO Guide [4]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with the measurement uncertainty and basis weight uncertainty. The coverage factor, k , is determined from the Student t -distribution corresponding to the appropriate associated degrees of freedom and 95 % confidence for each method.

The uncertainty in the values certified from combined independent method data is calculated as, $U = ku_c + B$. The quantity, u_c , is the combined standard uncertainty calculated according to ISO Guide [4], which accounts for the combined effect of the within variance for all participating laboratories at one standard deviation. The bias adjustment, B , is for the difference between methods, which is the maximum difference between the certified value and the method means [3].

Table 1. Certified Concentrations for Selected Inorganic Constituents (Dry-Mass Basis)

Element	Mass Fraction (mg/kg)		
Arsenic	166	±	20
Cadmium	4.17	±	0.09
Chromium	109	±	6.0
Copper	2594	±	52
Lead	574	±	11
Mercury	1.10	±	0.19
Molybdenum	10.07	±	0.20
Nickel	154.1	±	3.1
Selenium	0.44	±	0.11
Zinc	1254	±	196

Reference Values and Uncertainties: The reference values are the means of the results from two or more independent analytical methods. The uncertainty in the reference values is expressed as an expanded uncertainty, U , and is calculated according to the method described in the *ISO Guide to the Expression of Uncertainty in Measurement* [4]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of between-laboratory and within-laboratory components of uncertainty. The coverage factor, k , is determined from the Student t -distribution corresponding the appropriate associated degrees of freedom and 95 % confidence for each element. As the accuracy of the measurements could not be assessed from the data, the uncertainty associated with a reference value may fail to include all sources of uncertainties and may represent only a measure of the precision of the measurement methods.

Table 2. Reference Concentrations for Selected Inorganic Constituents (Dry-Mass Basis)

Element	Mass Fraction (mg/kg)			Element	Mass Fraction (%)		
Barium	254	±	24	Aluminum	1.37	±	0.09
Cerium	1240	±	110	Calcium	0.67	±	0.06
Cobalt	66.3	±	4.8	Iron	26.9	±	0.7
Gallium	35	±	10	Magnesium	0.26	±	0.02
Indium	238	±	70	Phosphorus	0.50	±	0.06
Lanthanum	58.1	±	2.4	Potassium	0.32	±	0.01
Silver	30.6	±	4.7	Sodium	1.30	±	0.05
Titanium	880	±	90				
Vanadium	80	±	10				

Information Values: Additional data from collaborating laboratories are provided as information to the SRM user. These values represent results from one laboratory, two or more laboratories that used non-independent methods, or from two or more laboratories using different methods with significant differences in their results [1].

Table 3. Information Concentrations for Selected Inorganic Constituents (Dry-Mass Basis)

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (%)
Antimony	2.0	Carbon	2.1
Europium	0.34	Silicon	20.3
Gold	2.2	Sulfur	0.2
Hafnium	0.77		
Lithium	5.0		
Manganese	300		
Rubidium	23		
Samarium	1.3		
Scandium	3.4		
Tantalum	0.73		
Terbium	0.48		
Thorium	2.4		
Uranium	8.3		
Ytterbium	0.74		
Yttrium	10		

Table 4. Methods Used for the Analysis of SRM 2782^(a)

Aluminum	ICP-AES, INAA, XRF	Mercury	CV-AAS, INAA
Antimony	INAA	Molybdenum	ICP-AES, ID-ICPMS , INAA, TXRF
Arsenic	HG-AAS, ICP-AES, INAA, TXRF	Nickel	ICP-AES, ID-ICPMS , INAA, TXRF
Barium	ICP-AES, INAA, TXRF	Phosphorus	ICP-AES, XRF
Cadmium	ID-ICPMS , INAA, TXRF	Potassium	ICP-AES, INAA, TXRF, XRF
Calcium	ICP-AES, TXRF, XRF	Rubidium	INAA, TXRF
Carbon	RF-CIR	Samarium	INAA
Cerium	ICP-AES, INAA, TXRF	Scandium	ICP-AES, INAA
Chromium	FAAS, ICP-AES, INAA, TXRF	Selenium	HG-AAS, INAA
Cobalt	ICP-AES, INAA	Silicon	XRF
Copper	ICP-AES, ID-ICPMS , INAA, TXRF	Silver	ICP-AES, INAA, TXRF
Europium	INAA	Sodium	ICP-AES, NAA, XRF
Gallium	ICP-AES, INAA	Sulfur	TXRF, RF-SIR
Gold	AAS, INAA	Tantalum	INAA
Hafnium	INAA	Terbium	INAA
Indium	INAA, TXRF	Thorium	INAA
Iron	ICP-AES, INAA, TXRF, XRF	Titanium	ICP-AES, XRF
Lanthanum	ICP-AES, INAA	Uranium	INAA
Lead	ICP-AES, ID-ICPMS , TXRF	Vanadium	ICP-AES, INAA
Lithium	ICP-AES	Yttrium	ICP-AES, TXRF
Magnesium	ICP-AES, XRF	Ytterbium	INAA
Manganese	ICP-AES, INAA, TXRF, XRF	Zinc	ICP-AES, ID-ICPMS, INAA, TXRF

^(a) Methods used for establishment of certified values are shown in bold-face type; methods used for reference and information values or to corroborate certified values are not in bold.

Methods

AAS	Atomic absorption spectrometry
FAAS	Flame atomic absorption spectrometry
RF-CIR	Resistance furnace with carbon infrared detector
RF-SIR	Resistance furnace with sulfur infrared detector
CV-AAS	Cold vapor atomic absorption spectrometry
HG-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
TXRF	Total reflectance x-ray fluorescence
XRF	Wavelength dispersive x-ray fluorescence

NIST Analysts

E.S. Beary
M.S. Epstein
R. Saraswati

Cooperating Analysts and Laboratories:

S. Landsberger and D. Wu; University of Illinois, Nuclear Engineering Department, Champaign, IL.
S.A. Wilson, D. Siems, and P. Briggs; USGS, Lakewood, CO.
A. Prange, U. Reus, and R. Neidergesäs; GKSS Research Center, Institute of Physical and Chemical Analytics, Geesthacht, Germany.

REFERENCES

- [1] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements; NIST Special Publication 260-136 (2000); available at <http://www.nist.gov/srm/publications.cfm> (accessed Sep 2011).
- [2] Thompson, A.; Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811; U.S. Government Printing Office: Washington, DC (2008); available at: <http://www.nist.gov/pml/pubs/> (accessed Sep 2011).
- [3] Schiller, S.B. and Eberhardt, K.R., *Combining Data from Independent Analysis Methods*; Spectrochim. Acta, Vol 46B, pp. 1607–1613 (1991).
- [4] JCGM 100:2008; *Evaluation of Measurement Data - Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2011); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297; U.S. Government Printing Office: Washington, DC (1994); available at <http://physics.nist.gov/Pubs/> (accessed Sep 2011).
- [5] Federal Register SW-846 (1995); available at <http://www.epa.gov/osw/hazard/testmethods/sw846/frn.htm> (accessed Sep 2011).
- [6] New Jersey Administrative Code, 1994. N.J.A.C. 7:14-4.
- [7] Kane, J.S.; *Leach Data vs Total: Which is Relevant for SRMs*, Fresenius' J. Anal. Chem. Vol. 352: pp 209-213, (1995).

Certificate Revision History: 09 September 2011 (Editorial changes); 10 December 1998 (Original certificate date).

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the SRM Program: Telephone (301) 975-2200, Fax (301) 926-4751, e-mail srminfo@nist.gov, or via the Internet at <http://www.nist.gov/srm>.

APPENDIX A

Leachable Mass Fractions Using U.S. EPA and NJDEP Methods

The U.S. EPA in its monitoring programs, has established a number of leach methods such as Methods [5] 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 for state use [6]. The NJDEP and the U.S. EPA prepared samples of SRM 2782 using the NJDEP 100 method and EPA Methods 3050 and 3051 and analyzed the resulting leachates by FAAS, flame emission spectrometry (FES), and ICP-AES. Based upon these results, method dependent reference values have been developed for EPA 3050, 3051, and NJDEP 100 methods.

SRM 2782 has been certified for total mass fractions of constituent elements and thereby requires complete sample decomposition. However, 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 or total recoverable results. However, reported acid labile or extractable mass fractions of elements are generally lower than total mass fractions (see Table A1, Percentage Leach Recovery). 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 [7].

Reference Values and Uncertainties: Reference values have been established for the acid-leachable mass fractions of fourteen elements in SRM 2782. These values are the means of all results from the different leach and measurement methods used. Please note that because EPA method 3051 did not provide useable results for silver, its results were excluded. In addition, variation in arsenic, cadmium, and vanadium data prevented their use as reference values. The reference values given in Table 1 are not NIST certified but are provided as a reference for U.S. EPA 3050, 3051, and NJDEP 100 methods. The reference values along with their uncertainties are based on 95 % confidence intervals of the means of results. The uncertainties include within-laboratory and between-laboratory differences, which were significant for some elements.

Table A1. Mass Fractions Reference Concentrations Leach Values for SRM 2782

Element	Leachable Mass fraction (mg/kg)			Leach Recovery ^(a) (%)
Aluminum ^(b,c)	1553	±	82	11
Barium ^(b,c)	152	±	11	60
Calcium ^(c,d)	4740	±	220	71
Chromium ^(b,c)	66.1	±	9.3	61
Cobalt ^(b,c)	54.5	±	4.6	82
Copper ^(b,c)	2435	±	47	94
Iron ^(b,c)	254 000	±	16 000	94
Lead ^(b,c,e)	554	±	36	97
Magnesium ^(b,c)	484	±	28	19
Manganese ^(b,c)	258	±	15	86
Nickel ^(b,c)	95.9	±	4.7	62
Potassium ^(b,c)	87	±	33	3
Sodium ^(b,c)	2620	±	330	20
Zinc ^(b,c)	1167	±	57	93

^(a) Percentage is calculated as leach value divided by certified (or non certified) value then multiplying it by one hundred.

^(b) FAAS.

^(c) ICP-AES.

^(d) FES.

^(e) Graphite furnace atomic absorption spectrometry (GFAAS).

Cooperating Analysts and Laboratories:

S.J. Nagourney, N. Tumillo; New Jersey Department of Environmental Protection, Trenton, NJ.

J. Birri, K. Peist; U.S. Environmental Protection Agency, Edison, NJ.