NATIONAL INSTITUTE OF
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U.S. DEPARTMENT OF COMMERCE

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Standard Reference Material® 2782

Industrial Sludge

CERTIFICATE OF ANALYSIS

Purpose: 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.

Description: A unit of SRM 2782 consists of approximately 70 g of industrial sludge that has been dried, pulverized, and sterilized.

Certified Mass Fraction Values: 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. Analytical methods used for the characterization of this SRM are given in Table C1. 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. The measurands are the total mass fractions of selected elements in industrial sludge. The certified values are metrologically traceable to the SI unit for mass.

Certified Mass Fractions for Selected Inorganic Constituents (Dry-Mass Basis)

Element	Mass Fraction (mg/kg)
Arsenic (As)	166 ± 20
Cadmium (Cd)	4.17 ± 0.09
Chromium (Cr)	109 ± 6.0
Copper (Cu)	2594 ± 52
Lead (Pb)	574 ± 11
Mercury (Hg)	1.10 ± 0.19
Molybdenum (Mo)	10.07 ± 0.20
Nickel (Ni)	154.1 ± 3.1
Selenium (Se)	0.44 ± 0.11
Zinc (Zn)	1254 ± 196

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/JCGM 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 the ISO/JCGM 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].

Non-Certified Values: Non-certified values are provided in Appendix A.

Period of Validity: The certified values delivered by **SRM 2782** are valid within the measurement uncertainty specified until **17 September 2034**. The certified values are nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

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Additional Information: Values of potential interest to users and additional information are provided in Appendices B and C.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

Storage: The original unopened bottles of SRM 2782 should be stored tightly sealed and away from sunlight and intense sources of radiation at room temperature (20 °C ± 5 °C). Although a tightly-sealed bottle should prevent absorption of moisture, storage at relative humidities above 60 % are not recommended. An open bottle can be reused until the material reaches its expiration date, provided that the open bottle is resealed and stored at room temperature (20 °C ± 5 °C).

Use: A minimum sample mass of 100 mg 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. 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 ºC. 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.]

REFERENCES

[1] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Dec 2024).
[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: https://www.nist.gov/pml/special-publication-811 (accessed Dec 2024).
[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 https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Dec 2024); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Dec 2024).
[5] Federal Register SW-846 (1995); available at https://www.epa.gov/hw-sw846/federal-register-notices-related-sw-846 (accessed Dec 2024).
[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: 03 December 2024 (Change of period of validity, format changes, editorial changes); 20 August 2015 (Change of expiration date; editorial changes); 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 Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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APPENDIX A

Non-Certified Values: A non-certified value is the present best estimate of the true value; however, the value does not meet NIST criteria for certification. Non-certified values are provided with associated uncertainties that may reflect only measurement reproducibility and may not include all sources of uncertainty or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1]. The non-certified values are the means of the results from two or more independent analytical methods. The uncertainty in the non-certified values is expressed as an expanded uncertainty, U, and is calculated according to the method described in reference 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 to the appropriate associated degrees of freedom and 95 % confidence for each element.

Table A1. Non-Certified Mass Fractions for Selected Inorganic Constituents (Dry-Mass Basis)

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (%)
Barium (Ba)	254 ± 24	Aluminum (Al)	1.37 ± 0.09
Cerium (Ce)	1240 ± 110	Calcium (Ca)	0.67 ± 0.06
Cobalt (Co)	66.3 ± 4.8	Iron (Fe)	26.9 ± 0.7
Gallium (Ga)	35 ± 10	Magnesium (Mg)	0.26 ± 0.02
Indium (In)	238 ± 70	Phosphorus (P)	0.50 ± 0.06
Lanthanum (La)	58.1 ± 2.4	Potassium (K)	0.32 ± 0.01
Silver (Ag)	30.6 ± 4.7	Sodium (Na)	1.30 ± 0.05
Titanium (Ti)	880 ± 90		
Vanadium (V)	80 ± 10		

Additional Non-Certified Values: Non-certified 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 non-certified values. The non-certified values given in Table A2 are not NIST certified but are provided as a reference for U.S. EPA 3050, 3051, and NJDEP 100 methods. The non-certified 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 A2. Non-Certified Mass Fraction Leach Values for SRM 2782

Element	Leachable Mass Fraction (mg/kg)	Leach Recovery(a) (%)
Aluminum (Al)(b,c)	1553 ± 82	11
Barium (Ba)(b,c)	152 ± 11	60
Calcium (Ca)(c,d)	4740 ± 220	71
Chromium (Cr)(b,c)	66.1 ± 9.3	61
Cobalt (Co)(b,c)	54.5 ± 4.6	82
Copper (Cu)(b,c)	2435 ± 47	94
Iron (Fe)(b,c)	254 000 ± 16 000	94
Lead (Pb)(b,c,e)	554 ± 36	97
Magnesium (Mg)(b,c)	484 ± 28	19
Manganese (Mn)(b,c)	258 ± 15	86
Nickel (Ni)(b,c)	95.9 ± 4.7	62
Potassium (K)(b,c)	87 ± 33	3
Sodium (Na)(b,c)	2620 ± 330	20
Zinc (Zn)(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).

Maintenance of Non-Certified Values: NIST will monitor this material to the end of its period of validity. If substantive technical changes occur that affect the non-certified values during this period, NIST will update this Reference Material Information Sheet and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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APPENDIX B

Values of Potential Interest: 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]. Values of potential interest cannot be used to establish metrological traceability.

Mass Fraction Values of Potential Interest for Selected Inorganic Constituents (Dry-Mass Basis)

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (%)
Antimony (Sb)	2.0	Carbon (C)	2.1
Europium (Eu)	0.34	Silicon (Si)	20.3
Gold (Au)	2.2	Sulfur (S)	0.2
Hafnium (Hf)	0.77		
Lithium (Li)	5.0		
Manganese (Mn)	300		
Rubidium (Rb)	23		
Samarium (Sm)	1.3		
Scandium (Sc)	3.4		
Tantalum (Ta)	0.73		
Terbium (Tb)	0.48		
Thorium (Th)	2.4		
Uranium (U)	8.3		
Ytterbium (Yb)	0.74		
Yttrium (Y)	10		

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APPENDIX C

Source and Preparation: 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: Analytical methods used for the characterization of this SRM are given in Table C1. 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.

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 non-certified 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 A2, 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].

Cooperating Analysts and Laboratories:
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Table C1. 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 non-certified values and values of potential interest 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

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