



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. Certified values are provided for elements. Reference values are provided for elements, synthetic polycyclic musks, and perfluorinated compounds (PFCs) as Perfluorinated Alkyl Acids (PFAAs) and Hexabromocyclododecanes (HBCDs). Information values are provided for PFAAs. SRM 2781 is a dried, pulverized domestic sludge. A unit of SRM 2781 consists of a bottle containing 40 g of dried, pulverized domestic sludge.

Certified Mass Fraction Values: The certified mass fraction values for elements are listed in Table 1; analytical methods are provided in Table 7. 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 taken into account [1]. The measurands are the certified mass fractions listed in Table 1 and are metrologically traceable to the SI unit for mass expressed as percentages or as milligrams per kilogram, as indicated in the table.

Reference Mass Fraction Values: Reference mass fraction values for elemental and organic constituents are provided in Tables 2 through Table 5; analytical methods are provided in at the end in Table 7. Reference values are noncertified values that are the best estimate of the true values based on available data. The values do not meet NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement reproducibility, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [1]. The measurands are the mass fractions listed in Tables 2 through 5 as determined by the methods indicated and are metrologically traceable to the SI unit for mass fraction expressed as percentages or as micrograms per kilogram, as indicated in the tables.

Information Mass Fraction Values: Information mass fraction values for organic constituents are provided in Table 6. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value or only a limited number of analyses were performed [1]. Information values may not be used to assess metrological traceability.

Expiration of Certification: The certification of **SRM 2781** is valid, within the measurement uncertainty specified, until **31 August 2025**, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Storage and 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 or register online) will facilitate notification.

Overall direction and coordination of technical measurements for the original characterization of this SRM was performed by J.D. Fassett of the NIST Chemical Sciences Division. Coordination and certification of additional elements and organic constituents were performed by J.L. Reiner of the NIST Chemical Sciences Division and D.J. O'Kelly formerly of NIST.

Statistical consultation was provided by L.M. Gill and N.A. Heckert of the NIST Statistical Engineering Division.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 15 September 2015
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Robert L. Watters, Jr, Director
Office of Reference Materials

Analytical measurements were performed at NIST by M.H. Ahsan, E.S. Beary, C.M. Beck II, D.A. Becker, M.S. Epstein, K. Garrity, R.R. Greenberg, R.M. Lindstrom, E.A. Mackey, J.L. Molloy, J.R. Moody, M.S. Nocun, B.R. Norman, D.J. O'Kelly, P.J. Paulsen, A. Peck, S.A. Rabb, M.S. Rearick, J.L. Reiner, T.A. Rush, R. Saraswati, J.M. Smeller, R.L. Watters, Jr., and L.J. Wood.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

INSTRUCTIONS FOR STORAGE AND USE

Storage: SRM 2781 must be stored in its original bottle at temperatures less than 30 °C.

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) and organic constituents 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.]

PREPARATION AND ANALYSIS⁽¹⁾

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 (Lakewood, CO) for processing. It was dried at ambient temperature in a forced air chamber, ground to pass a 74 µm (200 mesh) sieve, blended for 24 h to assure homogeneity of the pulverized material. 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 performed at USGS were detected.

Value Assignment: Analyses for value assignment were performed by NIST and collaborating laboratories where appropriate. Values were reported on a dry-mass basis in mass fraction units and are based on measurements using a sample mass of at least 100 mg. Analytical methods are provided in Table 7.

⁽¹⁾ Certain commercial equipment, instruments, or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 1. Certified Mass Fractions for Elements

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (%)
Arsenic (As)	7.81 ± 0.67 ^(a)	Nitrogen (N)	4.78 ± 0.16 ^(b)
Cadmium (Cd)	12.78 ± 0.63 ^(a)		
Chromium (Cr)	202 ± 14 ^(a)		
Copper (Cu)	627.8 ± 18.4 ^(b)		
Lead (Pb)	200.8 ± 4.2 ^(b)		
Mercury (Hg)	3.68 ± 0.14 ^(b)		
Molybdenum (Mo)	46.6 ± 5.4 ^(a)		
Nickel (Ni)	80.2 ± 1.8 ^(b)		
Selenium (Se)	16.0 ± 1.5 ^(a)		
Silver (Ag)	97.6 ± 6.5 ^(b)		
Zinc (Zn)	1273 ± 68 ^(a)		

^(a) The certified mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The uncertainty listed with each value is an expanded uncertainty about the mean [1,2], with coverage factor, k , based on the t -interval where the degrees of freedom is the number of methods minus one and calculated by combining a pooled within-method variance with a between-method variance [3] following the ISO/JCGM Guide [4,5]. The certified values are reported on a dry-mass basis. For certified values to be valid, the material must be dried according to the instructions provided above.

^(b) The certified mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The expanded uncertainty is the half-width of a symmetric 95 % parametric bootstrap confidence interval [6], which is consistent with the ISO/JCGM Guide [4,5]. The effective coverage factor, k , is 2. The certified values are reported on a dry-mass basis. For certified values to be valid, the material must be dried according to the instructions provided above.

Reference Mass Fraction Values: Each reference mass fraction value, expressed as a mass fraction on a dry-mass basis, is an equally weighted mean of results provided by NIST and/or collaborating laboratories.

Table 2. Reference Mass Fractions for Elements

Element	Mass Fraction (μg/kg)	Element	Mass Fraction (%)
Beryllium (Be)	613.3 ± 24.7 ^(a)	Aluminum (Al)	1.6 ± 0.1 ^(b)
		Calcium (Ca)	3.9 ± 0.1 ^(c)
		Iron (Fe)	2.8 ± 0.1 ^(c)
		Magnesium (Mg)	0.59 ± 0.02 ^(b)
		Phosphorus (P)	2.43 ± 0.04 ^(b)
		Potassium (K)	0.49 ± 0.03 ^(b)
		Silicon (Si)	5.1 ± 0.2 ^(a)
		Sodium (Na)	0.21 ± 0.01 ^(b)
		Titanium (Ti)	0.31 ± 0.01 ^(b)

^(a) The reference mass fraction value was calculated from a single measurement method. The expanded uncertainty is the half width of a 95 % Students t -confidence interval for μ . The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

^(b) The reference mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The reference values are reported on a dry-mass basis. The expanded uncertainty is the half-width of a symmetric 95 % parametric bootstrap confidence interval [6], which is consistent with the ISO/JCGM Guide [4,5]. The effective coverage factor, k , is 2. For reference values to be valid, the material must be dried according to the instructions provided above.

^(c) The reference mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The uncertainty listed with each value is an expanded uncertainty about the mean [1,2], with coverage factor, k , based on the t -interval where the degrees of freedom is the number of methods minus one and calculated by combining a pooled within-method variance with a between-method variance [3] following the ISO/JCGM Guide [4,5]. For reference values to be valid, the material must be dried according to the instructions provided above.

Table 3. Reference Mass Fractions for Polycyclic Musks

	Mass Fraction ($\mu\text{g/kg}$)
1,3,4,6,7,8-Hexahydro-4,6,6,7,8-hexa-methyl-cyclopenta-(γ)-2-benzopyran (HHCB)	9200 \pm 1220 ^(a)
7-Acetyl-1,1,3,4,4,6-hexamethyltetralin (AHTN)	19700 \pm 900 ^(a)
5-Acetyl-1,1,2,6-tetramethyl-3-isopropylindan (ATII)	2260 \pm 90 ^(a)
4-Acetyl-1,1-dimethyl-6-tert-butylindan (ADBI)	1140 \pm 60 ^(a)
6-Acetyl-1,1,2,3,3,5-hexamethylindan (AHMI)	142 \pm 6 ^(b)

^(a) The reference mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The uncertainty listed with each value is an expanded uncertainty about the mean [1,2], with coverage factor, $k = 2$, calculated by combining a pooled within-method variance with a between-method variance [3] following the ISO/JCGM Guide [4,5]. The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

^(b) The reference mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The expanded uncertainty is the half-width of a symmetric 95 % parametric bootstrap confidence interval [6], which is consistent with the ISO/JCGM Guide [4,5]. The effective coverage factor, k , is 2. The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

Table 4. Reference Mass Fractions for Selected Perfluorinated Alkyl Acids (PFAAs)

	Mass Fraction ^(a) ($\mu\text{g/kg}$)
Perfluorohexanoic Acid (PFHxA)	13.0 \pm 2.0
Perfluoroheptanoic Acid (PFHpA)	7.96 \pm 1.50
Perfluorooctanoic Acid (PFOA)	28.5 \pm 3.3
Perfluorohexanesulfonic Acid (PFHxS)	9.39 \pm 1.76
Perfluorooctanesulfonic Acid (PFOS)	225 \pm 41
Perfluorooctane Sulfonamide (PFOSA)	6.31 \pm 0.97

^(a) The reference mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The uncertainty listed with each value is an expanded uncertainty about the mean [1,2], with coverage factor, $k = 2$, calculated by combining a pooled within-method variance with a between-method variance [3] following the ISO/JCGM Guide [4,5]. The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

Table 5. Reference Mass Fractions for Hexabromocyclododecanes (HBCD)

	Mass Fraction ($\mu\text{g/kg}$)
α -hexabromocyclododecane (α -HBCD)	17.8 \pm 1.7 ^(a)
β -hexabromocyclododecane (β -HBCD)	1.65 \pm 0.39 ^(a)
γ -hexabromocyclododecane (γ -HBCD)	9.73 \pm 0.77 ^(b)

^(a) The reference mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The uncertainty listed with each value is an expanded uncertainty about the mean [1,2], with coverage factor, $k = 2$, calculated by combining a pooled within-method variance with a between-method variance [3] following the ISO/JCGM Guide [4,5]. The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

^(b) The reference mass fraction value is a weighted mean of the mass fractions determined by the methods indicated for each analyte [1]. The expanded uncertainty is the half-width of a symmetric 95 % parametric bootstrap confidence interval [6], which is consistent with the ISO/JCGM Guide [4,5]. The effective coverage factor, k , is 2. The reference values are reported on a dry-mass basis. For reference values to be valid, the material must be dried according to the instructions provided above.

Table 6. Information Mass Fraction Values for Selected Perfluorinated Alkyl Acids (PFAAs)

	Mass Fraction (µg/kg)
Perfluorononanoic Acid (PFNA)	5.09
Perfluorodecanoic Acid (PFDA)	4.76

Table 7. Methods used for the Analysis of SRM 2781

Analyte	Method ^(a)	Analyte	Method ^(a)
Aluminum	INAA, ICP-AES, XRF	Musks	GC-MS
Arsenic	RNAA, Hyd. AAS, INAA	Nickel	ICP-AES, ID-ICPMS, INAA, TXRF
Beryllium	ICP-MS	Nitrogen	PGAA
Cadmium	ID-ICPMS, PGAA, RNAA, INAA, TXRF	PFAAs	LC-MS/MS
Calcium	INAA, TXRF, ICP-AES, XRF	Phosphorus	Color, ICP-AES, XRF
Chromium	INAA, ICP-OES	Potassium	INAA, TXRF, ICP-AES
Copper	ID-ICPMS, RNAA, INAA, TXRF	Selenium	Hyd. AAS, RNAA, INAA, TXRF
HBCDs	LC-MS/MS	Silicon	XRF
Iron	INAA, TXRF, ICP-AES, XRF	Silver	INAA, ICP-OES
Lead	ICP-AES, ID-ICPMS, TXRF	Sodium	INAA, ICP-AES, XRF
Magnesium	INAA, ICP-AES, XRF	Titanium	INAA, TXRF, XRF
Mercury	FIA-CV-AAS, RNAA, INAA	Zinc	ICP-AES, ID-ICPMS, INAA, TXRF
Molybdenum	ID-ICPMS, ICP-AES, TXRF, INAA		

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

Methods Key:

INAA: Instrumental Neutron Activation Analysis

ICP-AES: Inductively Coupled Plasma-Atomic Emission Spectroscopy

XRF: X-Ray Fluorescence

RNAA: Radiochemical neutron activation analysis

Hyd AAS: Hydride Generation Atomic Absorption Spectroscopy

ICP-MS: Inductively Coupled plasma – mass spectrometry

ID-ICPMS: Isotope Dilution Inductively Coupled Plasma Mass Spectrometry

PGAA: Prompt Gamma Activation Analysis

TXRF: Total Reflection X-ray Fluorescence Spectrometry

ICP-OES: Inductively Coupled plasma

LC-MS/MS: Liquid chromatography – Mass Spectroscopy Mass Spectroscopy

FIA-CV-AAS: Cold Vapor Atomic Absorption Spectroscopy

GC-MS: Gas chromatography – Mass Spectroscopy

Color: Colorimetry

Cooperating Analysts and Laboratories:

University of Illinois, Nuclear Engineering Department, Champaign, IL, S. Landsberger and D. Wu.

USGS, Lakewood, CO, S.A. Wilson, D. Siems, and P. Briggs.

GKSS Research Center, Institute of Physical and Chemical Analytics, Geesthacht, Germany, A. Prange, U. Reus, and R. Neidergesäss.

3M Company; St. Paul, MN.

Colorado School of Mines.

US Environmental Protection Agency; Athens, GA.

Wageningen IMARES; Ijmuiden, The Netherlands.

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Certificate Revision History: 15 September 2015 (Change from reference to certified mass fractions and the addition of reference mass fractions for several inorganic and organic constituents; addendum changed to appendix; change of expiration date; editorial changes); 25 October 1996 (Addition of Addendum); 22 June 1995 (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) 948-3730; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.

APPENDIX A

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

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 [7].

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 [8,9] 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 [10]. 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 FAAS and 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 A1, 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: The reference values given in Table A1 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.

$$\text{Leach Recovery (\%)} = 100 \times \frac{\text{Leach Value}}{\text{Certified or Reference Value}}$$

Table A1. Reference Leach Values for SRM 2781

Element	Leachable Mass Fraction (mg/kg)		Leach Recovery (%)
Aluminum (Al)	8040	± 980	50
Barium (Ba)	570	± 65	-- ^(a)
Cadmium (Cd)	11	± 2	86
Calcium (Ca)	36440	± 1830	93
Chromium (Cr)	143	± 14	71
Copper (Cu)	601	± 16	96
Iron (Fe)	24300	± 2100	87
Lead (Pb)	183	± 15	91
Magnesium (Mg)	4850	± 290	82
Manganese (Mn)	745	± 33	--
Nickel (Ni)	72.3	± 6.3	90
Silver (Ag)	86.3	± 1.7	88
Vanadium (V)	81.9	± 3.8	--
Zinc (Zn)	1120	± 34	88

^(a) -- indicates that a certified or reference total mass fraction value was not available for the element.

Cooperating Analysts and Laboratories:

S.J. Nagourney; New Jersey Department of the Environment, Trenton, NJ.
J. Birri, K. Peist; U.S. Environmental Protection Agency, Edison, NJ.