National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2780a

Hard Rock Mine Waste

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis used to determine elements in hard rock mine waste and materials of a similar matrix. It can be used to validate value assignment of in-house reference materials. SRM 2780a is composed of material collected from waste piles of abandoned mine sites in the mountains of central Colorado. A unit of SRM 2780a consists of approximately 50 g of material of which 90 % passes a 150 µm (No. 100) sieve.

Certified Mass Fraction Values: Certified values for constituents in SRM 2780a are listed in Table 1 as mass fractions of the total amounts of the elements in rock powder [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 taken into account [2]. A certified value is the present best estimate of the true value. The certified values are metrologically traceable to the SI derived unit of mass fraction expressed as either percent or milligrams per kilogram. The expanded uncertainty intervals are expressed at a coverage level of approximately 95 %.

Expiration of Certification: The certification of **SRM 2780a** is valid, within the measurement uncertainty specified, until **31 December 2028**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage and Use"). Reference values are expected also to remain valid within this period. Periodic recalibration or recertification of this SRM is not required. This 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.

Coordination of technical measurements for the certification of this SRM was performed by J.R. Sieber of the NIST Chemical Sciences Division. Coordination of measurements by international collaborators was performed by M.A. Cabral de Goes of the Center for Mineral Technology (CETEM), Rio de Janeiro, Brazil.

Statistical consultation for this SRM was provided by A.L. Pintar of the NIST Statistical Engineering Division.

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

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 12 September 2017

Steven J. Choquette, Director
Office of Reference Materials

Table 1. Certified Mass Fraction Values in SRM 2780a Hard Rock Mine Waste

Element	Mass Fraction (%)	Uncertainty Interval (%)		
Aluminum (Al)	8.43	8.16	to	8.68
Barium (Ba)	0.093	0.078	to	0.138
Calcium (Ca)	0.247	0.227	to	0.305
Iron (Fe)	8.75	8.29	to	9.10
Lead (Pb)	0.665	0.628	to	0.729
Magnesium (Mg)	0.465	0.434	to	0.489
Potassium (K)	3.99	3.88	to	4.34
Silicon (Si)	24.1	22.3	to	28.1
Sodium (Na)	0.108	0.097	to	0.133
Sulfur (S)	8.85	8.02	to	9.08
Titanium (Ti)	0.643	0.595	to	0.732
Zinc (Zn)	0.102	0.096	to	0.106

Element	Mass Fraction (mg/kg)	Uncertainty Interval (mg/kg)		
Antimony (Sb)	18.3	15.5	to	19.9
Arsenic (As)	65.9	57.5	to	78.1
Cerium (Ce)	67.7	59.7	to	73.8
Cesium (Cs)	8.3	7.4	to	9.3
Chromium (Cr)	205	194	to	233
Cobalt (Co)	16.5	13.8	to	19.1
Copper (Cu)	240	234	to	253
Gold (Au)	6.6	5.3	to	8.4
Lanthanum (La)	34.4	26.5	to	38.1
Manganese (Mn)	490	460	to	530
Molybdenum (Mo)	25.0	24.2	to	28.3
Neodymium (Nd)	28.3	25.1	to	29.4
Nickel (Ni)	95	75	to	138
Phosphorus (P)	286	262	to	317
Rubidium (Rb)	220	212	to	231
Samarium (Sm)	4.7	3.8	to	5.1
Scandium (Sc)	15.6	13.9	to	17.0
Silver (Ag)	72.5	66.9	to	81.0
Strontium (Sr)	121	107	to	129
Thorium (Th)	12.0	10.3	to	13.3
Uranium (U)	4.0	3.3	to	4.4
Vanadium (V)	152	146	to	168
Zirconium (Zr)	206	177	to	231

INSTRUCTIONS FOR STORAGE, HANDLING AND USE

The values listed for this SRM were determined on the as-received basis. No drying is necessary. Sampling and sample preparation procedures should be designed to avoid material segregation on the basis of particle size. It is recommended to mix the contents of the bottle prior to sampling by turning the bottle end over end for two minutes. If particle size reduction is performed, it is the user's responsibility to prevent contamination or loss of material. This SRM must be stored in an air-conditioned or similar cool and dry environment away from light and fumes. A value for loss on ignition (LOI) at 950 °C is provided to enable the user to check for changes to stored material by determining the LOI in the user's laboratory and comparing the result to the value in Table 2 in this certificate.

To relate analytical determinations to the values in this Certificate of Analysis, a minimum sample quantity of 250 mg is recommended based on the quantities used for most test methods. From testing using microscale X-ray fluorescence spectrometry, a specimen mass as low as 5 mg is appropriate for the elements Al, As, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, P, Pb, S, Si, Ti, V, and Zn.

To use the uncertainty intervals given in this certificate in comparisons and calculations, the user must be aware that most intervals are not symmetric about the assigned values. The use of the intervals depends on the context, and instructions for two common uses follow.

Direct comparison to a result of a user determination: For this case, directly compare a coverage interval calculated by the user for their result to the appropriate coverage interval provided in this certificate.

Propagation of uncertainty when using SRM 2780a as a calibration standard: When a value provided in this certificate is used to calibrate a measurement process, the uncertainty associated with that value should be appropriately propagated into the user's uncertainty calculation. The asymmetric intervals in Table 1 and Table 2 cannot be interpreted as (value ± ku_c), where k is an expansion factor, and u_c is a combined standard uncertainty representative of both sides of the interval around the value. The user may choose one of the following two options for their uncertainty calculations. A first option is to use an approximation to u_c equal to one fourth of the width of the coverage interval given in this certificate (see note 8.1 in reference 3). A second and better option is to use a Monte Carlo method for propagation of probability distributions. This can be done using the NIST Uncertainty Machine at https://uncertainty.nist.gov. The user would enter his or her measurement equation and standard uncertainty estimates or probability distributions describing uncertainty for the terms of the equation. For the estimate of uncertainty of the calibration value, the user would choose the "Sample values" option and provide a file containing the actual distribution of the NIST analytical results of certification analyses. Plain ASCII files for the constituents may be found at https://www-s.nist.gov/srmors/view_detail.cfm?srm=2780A.

PREPARATION AND ANALYSIS[(1)]

Collection and preparation of mine waste for SRM 2780a were performed by the U.S. Geological Survey (USGS) under the direction of S.A. Wilson, who collected materials from several sites in the mountains of central Colorado. Grinding, blending, and bottling were performed at USGS in Denver, Colorado. Homogeneity testing was performed at NIST using X-ray fluorescence spectrometry and microbeam X-ray fluorescence spectrometry to measure numerous elements believed to be representative of the constituents given in this Certificate of Analysis. Suitability of this material for testing using low milligram quantities was evaluated using microbeam X-ray fluorescence spectrometry [5,6] to evaluate the elements Al, As, Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, P, Pb, S, Si, Ti, V, and Zn.

For each certified and reference value, the estimate was derived by fitting a statistical model to the results of measurements using the test methods listed in Table 4. That is, a posterior probability distribution, describing our knowledge of the measurand, was developed for each constituent. Its median was taken as the estimate of the measurand, and the 2.5^{th} and 97.5^{th} percentiles form the 95 % coverage interval. The Bayesian paradigm was used for statistical inference [7]. The probability distribution combines all recognized sources of uncertainty. This procedure is consistent with the ISO/JCGM Guide [8,9].

Measurements for value assignment of SRM 2780a were performed by A.F. Marlow, J.L. Molloy, R.L. Paul, and J.R. Sieber of the NIST Chemical Sciences Division. Additional analyses were performed by collaborating laboratories, including C. Bucknam, H. Corsi, J. Huddle, and J. Lorango (Newmont Metallurgical Services, Englewood, Colorado, United States); V. Kuch, E. Matthews and B. White (SGS Canada Minerals Lakefield, Lakefield, Ontario, Canada); M. Adams and S.A. Wilson, (United States Geological Service, Denver, Colorado, United States); J.A. Begum and R. Cardinall (AGAT Laboratories, Mississauga, Ontario, Canada); E. Miller (ALS Geochemistry Vancouver, North Vancouver, British Columbia, Canada); D. Demianiuk and J. Moore (Accurassay Laboratories, Thunder Bay, Ontario, Canada); M. Eseme and E. Hoffman (Activation Laboratories Ltd., Ancaster, Ontario, Canada); C. Chiang and L. Sison (SGS Canada Minerals Burnaby, Burnaby, British Columbia, Canada); T. Ferguson (Bureau Veritas Commodities Canada, Vancouver, British Columbia, Canada); S. Kenny, (ALS Geochemistry Brisbane, Stafford, Queensland, Australia); A. Gasiorowski and G. Petrakis (Intertek Laboratory Services, Wingfield, South Australia, Australia); P. Whittle, (HRL Testing Pty. Ltd., Brisbane, Queensland, Australia); D. Burridge and P. Riles (Newcrest Laboratory Services, Orange, New South Wales, Australia); A. Daly and M. Trutwein (Labwest Minerals Pty. Ltd., Malag, Western Australia, Australia); L. Clarke and A. Tairov (ALS Minerals Loughrea, Galway, Ireland); B. Lkhagvasuren and A. Purvee (Central Geological Laboratory of Mongolia, Ulaanbaatar, Mongolia); I.E. Vasileva and E.V. Shabanova (Vinogradov Institute of Geochemistry, Irkutsk, Russia); A. Raycheva (Eurotest-Control EAD, Sofia, Bulgaria); and A. Hlahla (MINTEK, Randburg, South Africa).

NOTICE TO USERS

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

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of this SRM as a primary benchmark for the quality and accuracy of the user's in-house reference materials and working standards. As such, the SRM should be used to validate the more routinely used reference materials in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

ADDITIONAL CONSTITUENTS: Noncertified values are provided for the following additional constituents in SRM 2780a.

Reference Mass Fraction Values: Reference values for SRM 2780a are reported in Table 2 as mass fractions of the constituents in rock powder. A reference value is a non-certified value that is the present best estimate of the true value based on available data; however, the value does not meet the NIST criteria for certification and is provided with an associated uncertainty that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods [2]. The measurands are listed in Table 2 as determined by the methods indicated. The reference mass fraction values are metrologically traceable to the derived SI unit for mass fraction expressed as either percent or milligram per kilogram.

Table 2. Reference Mass Fraction Values in SRM 2780a Hard Rock Mine Waste

Element	Mass Fraction (mg/kg)	Uncertainty Interval (mg/kg)
Beryllium (Be)	1.1	1.0 to 1.2
Bismuth (Bi)	45	43 to 47
Cadmium (Cd)	4.8	4.5 to 5.4
Dysprosium (Dy)	3.1	1.5 to 4.1
Erbium (Er)	2.0	1.0 to 2.5
Europium (Eu)	0.9	0.6 to 1.0
Gallium (Ga)	21	20 to 24
Gadolinium (Gd)	3.2	2.1 to 4.0
Hafnium (Hf)	5.5	5.3 to 5.8
Holmium (Ho)	0.7	0.2 to 0.9
Indium (In)	1.65	1.56 to 1.73
Lithium (Li)	14	11 to 26
Lutetium (Lu)	0.33	0.31 to 0.35
Niobium (Nb)	20	19 to 22
Praseodymium (Pr)	8	6 to 9
Tantalum (Ta)	1.2	1.1 to 1.6
Tellurium (Te)	22	21 to 24
Terbium (Tb)	0.5	0.3 to 0.6
Thallium (Tl)	5.5	5.2 to 5.8
Thulium (Tm)	0.31	0.15 to 0.43
Tin (Sn)	7.2	6.7 to 7.7
Tungsten (W)	17.4	16.9 to 18.1
Yttrium (Y)	18	8 to 26
Ytterbium (Yb)	2	1 to 3
Constituent	**Mass Fraction (%)**	**Uncertainty Interval (%)**
Carbon (C)	0.19	0.18 to 0.20
Loss on Ignition (950 ºC)	11.1	10.3 to 11.9

Information Mass Fraction Values: Information values for SRM 2780a are reported as mass fractions in Table 3. An information value is a value that may be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value [2]. Information values cannot be used to establish metrological traceability. The information values were determined using atomic emission, atomic absorption, and mass spectrometric test methods at NIST and collaborating laboratories as shown in Table 4.

Table 3. Information Mass Fraction Values in SRM 2780a Hard Rock Mine Waste

Element	Mass Fraction (mg/kg)
Boron (B)	27
Germanium (Ge)	<6
Mercury (Hg)	0.2
Rhenium (Re)	0.003
Selenium (Se)	6

Table 4. Methods Used in Elemental Analyses of SRM 2780a Hard Rock Mine Waste

Method	Constituents Determined
Wavelength Dispersive X-ray Fluorescence (WDXRF) Spectrometry at NIST and Collaborating Laboratories	Al, As, Ba, Ca, Ce, Cr, Cu, Fe, K, Mg, Mn, Na, Ni, P, Pb, Rb, S, Si, Sr, Ti, V, Zn, Zr
Energy Dispersive X-Ray Fluorescence (EDXRF) at Collaborating Laboratories	Fe, Mn, K, P, Si
Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) at Collaborating Laboratories	Ag, Al, As, Ba, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Hf, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Nb, Nd, Ni, P, Pb, Pr, Rb, S, Sb, Sc, Si, Sm, Sn, Ta, Tb, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn, Zr
Inductively Coupled Plasma Mass Spectrometry (ICP-MS) at Collaborating Laboratories	Al, Ag, As, Au, B, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs, Cu, Dy, Er, Eu, Fe, Ga, Gd, Ge, Hf, Hg, Ho, In, K, La, Li, Lu, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Pr, Rb, Re, Sb, Sc, Se, Sm, Sn, Sr, Ta, Tb, Te, Th, Ti, Tl, Tm, U, V, W, Y, Yb, Zn, Zr
Arc-Spark Optical Emission Spectrometry (AS-OES) at Collaborating Laboratories	Al, As, B, Bi, Cr, Cu, Fe, Ga, Ge, Mn, Mo, Nb, Sn, W, Y, Zn, Zr
Instrumental Neutron Activation Analysis (INAA) at NIST	Ag, As, Au, Ce, Co, Cs, La, Mo, Nd, Sb, Sc, Sm, Th, U
Thermogravimetric Analysis (TGA) at NIST and one Collaborating Laboratory	Loss on Ignition at 950 ºC in air
Combustion with Infrared Detection at Collaborating Laboratories	C, S
Fire Assay at one Collaborating Laboratory	Ag, Au
Direct Mercury Analyzer at NIST	Hg
Gravimetry at one Collaborating Laboratory	S

REFERENCES

[1] 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/physical-measurement-laboratory/special-publication-811 (accessed Sep 2017).
[2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136; U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF (accessed Sep 2017).
[3] Possolo, A.; *Simple Guide for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1900; U.S. Department of Commerce: Washington, DC (1994); available at http://nvlpubs.nist.gov/nistpubs/TechnicalNotes/NIST.TN.1900.pdf (accessed Sep 2017).
[4] Lafarge, T. and Possolo, A.; *The NIST Uncertainty Machine*; NCSLI Measure, 2015, Vol. 10, pp. 20–27.
[5] Molloy, J.L.; Sieber, J.R.; *Classification of Microheterogeneity in Solid Samples using μXRF*; Anal. and Bioanal. Chem., Vol. 392, pp. 995–1001 (2008).
[6] Molloy, J.L.; Sieber, J.R.; *Assessing Microscale Heterogeneity in Batches of Reference Materials Using Microbeam XRF*; X-Ray Spectrom., Vol. 40, pp. 306–314 (2011).
[7] Gelman, A.; Carlin, J.B.; Stern, H.S.; Dunson, D.B.; Vehtari, A.; Rubin, D.B.; *Bayesian Data Analysis*; 3rd ed., CRC Press (Boca Raton, FL) (2013).
[8] JCGM 100:2008; Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement (GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2017); 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 Sep 2017).
[9] JCGM 101:2008; Evaluation of Measurement Data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method; JCGM (2008); available at www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Sep 2017).

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 the Internet at https://www.nist.gov/srm.