National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2780

Hard Rock Mine Waste

This Standard Reference Material (SRM) is intended for use in the evaluation of methods and for the calibration of apparatuses used to determine heavy metals and other elements in hard rock mine waste and materials of a similar matrix. SRM 2780 is composed of material collected from a waste pile of an abandoned mine site near Silverton, CO. A unit of SRM 2780 consists of approximately 50 g of material of which 90 % passes a 150 µm (No. 100) sieve.

Certified Mass Fraction Values: Certified mass fraction values for 12 elements are listed 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 taken into account. All values are reported as mass fractions [1], on a dry-mass basis (see "Instructions for Drying"), and are based on measurements using a sample mass of at least 250 mg.

Reference Mass Fraction Values: Reference mass fraction values for seven elements are provided in Table 2. A NIST reference value is a noncertified value that is the 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 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. Mass fraction values are on a dry-mass basis.

Information Mass Fraction Values: Information mass fraction values for 28 elements are provided in Table 3. An information value is a value that may be of use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value.

Expiration of Certification: The certification of **SRM 2780** is valid, within the measurement uncertainty specified, until **31 December 2014**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Use"). 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) will facilitate notification.

The overall direction and coordination of the technical measurements leading to the certification of this SRM were performed by G.C. Turk of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by C.M. Beck II, W.R. Kelly, S.E. Long, J.L. Mann, A.F. Marlow, J.R. Sieber, R.D. Vocke, Jr., and L.L. Yu of the NIST Chemical Sciences Division.

Statistical consultation was provided by S.D. Leigh and D.D. Leber of the NIST Statistical Engineering Division.

Mine waste sample collection, preparation of the SRM, and contributing chemical analyses were performed by the U.S. Geological Survey (USGS) under the direction of S.A. Wilson. Details are described [3] in USGS Open-File Report 99-370 and is available by contacting the USGS Earth Science Information Center: telephone (303) 202-4210; or e-mail infoservices@usgs.gov.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 11 October 2012
Certificate Revision History on Last Page

Robert L. Watters, Jr., Director
Office of Reference Materials

Partial financial support for the development of this SRM was provided by the U.S. Environmental Protection Agency (EPA) under the direction of project manager B.A. Schumacher of the EPA National Exposure Research Laboratory, Characterization Research Division, Las Vegas, NV.

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

INSTRUCTIONS FOR USE

To relate analytical determinations to the certified values on this certificate, a minimum sample mass of 250 mg should be used. 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. The sample should be dried accordingly (see "Instructions for Drying"). 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.

Instructions for Drying: Analyses should be performed on samples as received; separate samples should be weighed before and after drying to obtain a correction factor for moisture. Samples may be dried in an oven at 107 °C under clean, dry inert gas, or in a desiccator over fresh anhydrous $Mg(ClO_4)_2$, or by other equivalent method which reaches a constant dry weight.

Table 1. Certified Mass Fractions (Dry-Mass Basis)

Element	Mass Fraction (%)			*k*
Aluminum	8.87	±	0.33	2.3
Calcium	0.195	±	0.020	2.6
Iron	2.784	±	0.080	2.4
Lead	0.577	±	0.041	2.4
Magnesium	0.533	±	0.020	2.8
Potassium	3.38	±	0.26	2.8
Sodium	0.221	±	0.018	2.8
Sulfur	1.263	±	0.042	2.6
Zinc	0.257	±	0.016	2.6

Element	Mass Fraction (mg/kg)			*k*
Arsenic	48.8	±	3.3	2.0
Cadmium	12.10	±	0.24	2.8
Mercury	0.710	±	0.042	2.6

The certified values for mercury and sulfur are the means of results obtained by NIST using isotope dilution mass spectrometry. 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 standard uncertainty calculated according to the ISO Guide [1]. The coverage factor, *k*, is determined from the Student's *t*-distribution for the appropriate degrees of freedom to yield 95 % confidence. The certified values for the remaining elements are derived from the results of at least one analysis performed at NIST and independent results from one or more methods provided by the USGS using the approach for combining results for multiple methods [2]. Multiple method results from USGS were first combined to give a single value and uncertainty before being combined with the NIST results. The certified value is an unweighted mean of the results from NIST and USGS. The uncertainty listed with each value is an expanded uncertainty about the mean, $U = ku_c$, with a coverage factor, *k*, determined from the Student's *t*-distribution for the appropriate degrees of freedom to yield 95 % confidence. Each u_c is calculated by combining a between-method variance [2] with a pooled, within-method variance following the ISO Guide [1]. Analytical methods are listed in Table 4.

Table 2. Reference Mass Fractions (Dry-Mass Basis)

Element	Mass Fraction (%)	*k*
Titanium	0.699 ± 0.019	2.8

Element	Mass Fraction (mg/kg)	*k*
Barium	993 ± 71	2.1
Copper	215.5 ± 7.8	2.4
Manganese	462 ± 21	2.6
Phosphorus	427 ± 40	2.3
Strontium	217 ± 18	2.2
Vanadium	268 ± 13	2.4

The reference values are based on the results of a single NIST method and one or more USGS methods. Reference values were derived from multiple results in the same manner as was done for the certified values. Analytical methods are listed in Table 4.

Table 3. Information Mass Fractions (Dry-Mass Basis)

Element	Mass Fraction (%)
Silicon	31

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Antimony	160	Niobium	18
Cerium	64	Rubidium	175
Cesium	13	Scandium	23
Chromium	44	Selenium	5
Cobalt	2.2	Silver	27
Gallium	26	Tellurium	5
Gold	0.18	Terbium	0.58
Hafnium	4.4	Thallium	5
Holmium	0.84	Thorium	12
Lanthanum	38	Thulium	0.4
Lithium	18	Tungsten	24
Molybdenum	11	Uranium	4
Neodymium	28	Zirconium	176
Nickel	12		

An information value is a value that may be of use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value.

Table 4. Methods Used in Elemental Determinations

Method	Elements Determined
Cold Vapor Isotope Dilution Inductively Coupled Plasma Mass Spectrometry at NIST	Hg
Isotope Dilution Thermal Ionization Mass Spectrometry at NIST	S
Standard Additions Inductively Coupled Plasma Mass Spectrometry at NIST	As, Cd, Cu, Pb, Zn
Wavelength Dispersive X-ray Fluorescence (WDXRF)	Al, As, Ba, Ca, Fe, K, Mg, Mn
Spectrometry (WDXRF) at NIST	Na, P, Pb, Sr, Ti, V, Zn, Zr
Spectrometry (WDXRF) at USGS	Al, Ca, Fe, K, Mg, Ti
Inductively Coupled Plasma Atomic Emission (ICP-AES)	Al, Ba, Ca, Cd, Cu, Fe, K, Mg
Spectrometry (ICP-AES) at USGS	Mn, Na, P, Pb, Sr, V, Zn
Instrumental Neutron Activation Analysis at USGS	As, Ba, Cd, Fe, Na, Zn
Hydride Generation Atomic Absorption Spectrometry at USGS	As

REFERENCES

[1] 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 (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2012); 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://www.nist.gov/pml/pubs/index.cfm (accessed Oct 2012).

[2] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.-K.; Vangel, M.G.; Yen, J.H.; Zhang, N.F.; *An Approach to Combining Results From Multiple Methods Motivated by the ISO GUM*; J. Res. Natl. Inst. Stand. Technol., Vol. 105; pp. 571–579 (2000).

[3] Wilson, S.A.; Briggs, P.H.; Brown, Z.A.; Taggart, J.E.; Knight, R.; *Collection, Preparation and Testing of NIST Hard Rock Mine Waste Reference Material SRM 2780*; SGS Open-File Report 99-370, USGS, Denver, CO (1999).

Certificate Revision History: 11 October 2012 (Extension of certification period; editorial changes); 31 January 2003 (Original certificate date).