National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 331a

Copper Ore Mill Tails

(In cooperation with ASTM International)

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis. A unit of SRM 331a consists of one bottle containing approximately 40 grams of fine powder.

Certified values for eight elements in SRM 331a are listed in Table 1. Reference values for 16 elements are listed in Table 2. Information values for eight elements are listed in Table 3. For all elements, values are reported as mass fractions [1]. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [2].

Certified 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. A certified value is the present best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using the test methods listed in Table 4. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval [3] and is calculated according to the method in the ISO and NIST Guides [4].

Reference Value: Reference values are non-certified values that are the present best estimates of the true values. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval [3] and is calculated according to the method in the ISO and NIST Guides [4].

Information Values: 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.

Expiration of Certification: The certification of SRM 331a is valid, within the measurement uncertainty specified, until **31 March 2017**, provided the SRM is handled in accordance with the instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Maintenance of 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 coordination of the technical measurements for certification of this SRM was under the direction of J.R. Sieber of the NIST Analytical Chemistry Division.

Analytical measurements for certification of this SRM were performed by A.F. Marlow, J.R. Sieber, and L.J. Wood of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by S.D. Leigh of the NIST Statistical Engineering 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

Gaithersburg, MD 20899
Certificate Issue Date: 10 March 2008

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

INSTRUCTIONS FOR USE

To relate analytical determinations to the assigned values on this Certificate of Analysis, a minimum sample quantity of 100 mg is recommended on the basis of homogeneity testing performed at NIST using X-ray fluorescence spectrometry. The powder does not require preparation prior to weighing. The material should be stored in its original container, tightly capped, in a cool, dry location. Loss on drying was tested at 110 °C for 2 h and found to be approximately 0.7 %.

Table 1. Certified Values for SRM 331a Copper Ore Mill Tails

Constituent	Value [(a)] (mass fraction) (mg/kg)	Expanded Uncertainty (mass fraction) (mg/kg)	Coverage Factor (*k*)
Cr	13.9	2.7	3.2
Mn	497	15	2.0
Co	12.6	3.9	2.0
Cu	789	69	2.0
Zn[(b)]	71.8	4.9	2.0
Sr	252.8	9.3	2.0
Ba[(b)]	259	16	2.0
Hg[(c)]	0.00184	0.00017	2.26

[(a)] The assigned value is an unweighted mean from two to six sets of results from two to three analytical methods performed at as many as five laboratories. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k* calculated by combining a between-method variance with a pooled, within-method variance following the ISO and NIST Guides [4,5].

[(b)] The assigned value is a weighted mean from seven or eight sets of results from two to three analytical methods performed at as many as five laboratories. The uncertainty listed with each value is an expanded uncertainty about the mean, with a coverage factor *k* calculated by combining a between-source variance incorporating inter-method bias with a pooled within-source variance following the ISO and NIST Guides [4,6].

[(c)] The assigned value is based on analyses by a single primary method at NIST [2]. The expanded uncertainty is calculated as a 95 % confidence interval where $U = ku_c$. The quantity u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO and NIST Guides [4]. The coverage factor, $k = 2.26$, corresponds to a *t* factor obtained from the *t*-distribution for approximately 9.5 degrees of freedom.

Material Preparation[1]**:** The material for SRM 331a was provided by Newmont Metallurgical Services, Englewood, Colorado. The material was blended and bottled at the U.S. Geological Survey (USGS), Denver Colorado, under the supervision of S.G. Wilson.

Cooperating Laboratories: Analytical determinations for certification of this SRM were performed by the following laboratories:
ALS Chemex, 212 Brooksbank Avenue, N. Vancouver, B. C., V7J 2C1, B. Caughlin
LECO Corporation, 3000 Lakeview Avenue, St. Joseph MI 89822-0669, D. Lawrenz
Newmont Metallurgical Services, 10101 East Dry Creek Road, Englewood, CO 80112, C.H. Bucknam, M. Dietrich
SGS Minerals Services, 185 Concession Street, Lakefield, Ontario, Canada K0L 2H0, V. Murphy
U.S. Geological Survey, Denver, Colorado, S.G. Wilson

[1] Certain commercial organizations, equipment, instruments, or materials are identified in this certificate in order 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 services, materials or equipment identified are necessarily the best available for the purpose.

Table 2. Reference Values for SRM 331a Copper Ore Mill Tails

Constituent	Value (mass fraction) (%)	Expanded Uncertainty [a] (mass fraction) (%)	Coverage Factor (*k*)
Na	3.15	0.15	2.0
Mg	1.623	0.051	2.0
Al	7.92	0.26	2.0
K	0.967	0.032	3.2
Ca	1.552	0.018	2.1
Ti	0.228	0.051	2.0
Fe	4.207	0.086	2.1

Constituent	Value [a] (mass fraction) (mg/kg)	Expanded Uncertainty (mass fraction) (mg/kg)	Coverage Factor (*k*)
C	565	89	2.0
S	870	110	2.1
Sc	11.4	0.4	4.3
V	121	10	2.0
Ni	8.1	2.6	2.0
Ga	16.3	0.6	2.1
Mo	3.2	0.8	2.0
Ce	9.6	0.3	2.2
Au	0.121	0.014	2.8

[a] The assigned value is an unweighted mean of the results from two to six sets of results from one to three analytical methods performed at as many as five laboratories. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k*, calculated by combining a between-method variance with a pooled, within-method variance following the ISO and NIST Guides [4,5].

Table 3. Information Values for SRM 331a Copper Ore Mill Tails

Constituent	Value (mass fraction) (mg/kg)
Li	3
P	550
Rb	25
Y	8
Cd	0.1
Cs	1
La	4
Pb	6

Table 4. Analytical Methods

Constituent	Methods
Li	ICP-OES, ICP-MS
C	Combustion
Na	ICP-OES, ICP-MS, NAA
Mg	ICP-OES, ICP-MS
Al	ICP-OES, ICP-MS
P	ICP-OES, ICP-MS
S	Combustion
K	ICP-OES, ICP-MS, NAA
Ca	ICP-OES, ICP-MS
Sc	ICP-OES, ICP-MS, NAA
Ti	ICP-OES, ICP-MS
V	ICP-OES, ICP-MS
Cr	ICP-OES, ICP-MS, NAA
Mn	ICP-OES, ICP-MS
Fe	ICP-OES, ICP-MS, NAA
Co	ICP-OES, ICP-MS, NAA
Ni	ICP-OES, ICP-MS, NAA
Cu	ICP-OES, ICP-MS
Zn	ICP-OES, ICP-MS, NAA
Ga	ICP-OES, ICP-MS
Rb	ICP-MS, NAA
Sr	ICP-OES, ICP-MS, NAA
Y	ICP-OES, ICP-MS
Mo	ICP-OES, ICP-MS
Cd	ICP-MS
Cs	ICP-MS, NAA
Ba	ICP-OES, ICP-MS, NAA
La	ICP-OES, ICP-MS
Ce	ICP-MS, NAA
Au	ICP-MS, NAA, Fire Assay
Hg	ID-CV-ICP-MS
Pb	ICP-OES, ICP-MS

Methods Key: Combustion – combustion with infrared detection performed at the cooperating laboratories;
Fire Assay – fire assay preparation with measurement by atomic absorption spectrophotometry at one cooperating laboratory;
ICP-OES – inductively coupled plasma optical emission spectrometry performed at the cooperating laboratories and at NIST;
ICP-MS – inductively coupled plasma mass spectrometry performed at the cooperating laboratories;
ID-CV-ICPMS – isotope dilution cold vapor inductively coupled plasma mass spectrometry performed at NIST;
NAA – neutron activation analysis at one cooperating laboratory.

REFERENCES

[1] 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 (1995); available at http://www.physics.nist.gov/Pubs.

[2] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Spec. Pub. 260-136, U.S. Government Printing Office: Washington, DC, p. 16 (2000).

[3] Hahn, G. J., and Meeker, W. Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).

[4] ISO; G*uide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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/.

[5] Levenson, M.S.; Banks, D.L.; Eberhardt, K.R.; Gill, L.M.; Guthrie, W.F.; Liu, H.K.; Vangel, M.G.; Yen, J.H.; Zang, 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).

[6] Ruhkin, A.L.; Vangel, M.G.; Estimation of a Common Mean and Weighted Mean Statistics; J. Am. Statist. Assoc., Vol. 93, pp. 303-308 (1998).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.