National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 330a

Copper Ore Mill Heads

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical and instrumental methods of analysis as well as evaluating the accuracy of "material balance" measurements in the copper mining and metallurgical industries. A unit of SRM 330a consists of one bottle containing approximately 90 g of powder.

Certified Mass Fraction Values: Certified values, expressed as mass fractions [1] for seven elements in SRM 330a 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 [2]. Certified values are based on the weighted means of analyses performed at NIST and collaborating laboratories [3]. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval and is calculated according to the methods in the ISO Guide and its Supplement 1 [4,5].

Reference Mass Fraction Values: Reference values for fourteen elements are provided in Table 2. 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. Reference values are based on the weighted means of analyses performed at NIST and collaborating laboratories [3]. The uncertainty listed with each value is an expanded uncertainty based on a 95 % confidence interval and is calculated according to the methods in the ISO Guide and its Supplement 1 [4,5].

Information Mass Fraction Values: The information values for seven elements provided in Table 3 are noncertified values with no reported uncertainties. A NIST information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to adequately the uncertainty associated with the value [2].

Expiration of Certification: The certification of **SRM 330a** is valid, within the measurement uncertainty specified, until **31 December 2020**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for 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) will facilitate notification.

The coordination of the technical measurements for certification of this SRM was under the direction of R.D. Vocke, Jr. of the NIST Analytical Chemistry Division.

Analytical measurements for certification of this SRM were performed by A.F. Marlow and J.R. Sieber of the NIST Analytical Chemistry Division. Additional measurements were provided by SGS Minerals Services, Lakefield, Ontario, Canada and the U.S. Geological Survey (USGS), Denver, CO under the direction of S.G. Wilson.

Statistical consultation for this SRM was provided by R.C Hagwood 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: 09 November 2010

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

INSTRUCTIONS FOR USE

To relate analytical determinations to the assigned values in this Certificate of Analysis, a minimum test portion 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 0.87 %.

Material Preparation and Analysis:[1] The material for SRM 330a was provided by C. Bucknam of Newmont Metallurgical Services, Englewood, CO. The material was ground, blended, and bottled at the USGS, Denver, CO under the supervision of S.G. Wilson. The material was characterized using the methods listed in Table 4.

Table 1. Certified Mass Fractions for SRM 330a Copper Ore Mill Heads

Constituent	Value[(a)] (%)	Expanded Uncertainty[(b)] (%)	Coverage Factor[(b)] (*k*)
Al	7.053	0.070	2
Ba	0.156	0.011	2
Ca	0.323	0.024	2
Cu	0.845	0.065	2
K	5.47	0.27	2
Mg	0.868	0.074	2
Na	0.657	0.021	2

[(a)] The assigned value is a weighted mean of the results from two sets of measurements from multiple independent analytical methods using the method of DerSimonian and Laird as described by Rukhin [3].

[(b)] The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor *k*, calculated by Monte-Carlo simulation of uncertainty components using methods from the ISO Guide and its Supplement 1 [4,5].

[1] Certain commercial equipment, instruments, or materials are identified in this certificate 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 2. Reference Mass Fractions for SRM 330a Copper Ore Mill Heads

Constituent	Value[a] (%)	Expanded Uncertainty[b] (%)	Coverage Factor[b] (k)
Fe	1.06	0.19	2
Si	33.4	1.3	2.47

Constituent	Value[a] (mg/kg)	Expanded Uncertainty[b] (mg/kg)	Coverage Factor[b] (k)
Cd	3.391	0.027	2
Ce	22.32	0.34	2
Co	4.542	0.069	2
Cr	77.0	4.0	2
Ga	17.4	0.18	2
Li	22.19	0.30	2
Ni	28.95	0.28	2
Sc	5.693	0.098	2
Sr	218.1	1.5	2
Y	20.01	0.92	2
Zn	94.9	3.4	2
Zr	80.5	2.2	2

[a] The assigned value is a weighted mean of the results from two sets of measurements from multiple independent analytical methods using the method of DerSimonian and Laird as described by Rukhin [3].

[b] The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor k, calculated by Monte-Carlo simulation of uncertainty components using methods from the ISO Guide and its Supplement 1 [4,5].

Table 3. Information Mass Fractions for SRM 330a Copper Ore Mill Heads

Constituent	Value (mg/kg)
Mo	4.5
Nb	5.7
P	326
Pb	27
Th	7.6
Ti	1 223
V	43

Table 4. Analytical Methods Used for Characterization of SRM 330a Copper Ore Mill Heads

Constituent	Methods
Al, Ba, Ca, Cu, Fe, K, Mg, Na, Ti	WDXRF, ICP-OES, ICP-MS
Si,	WDXRF
P	WDXRF, ICP-OES,
Cd, Ce, Co, Cr, Ga, Li, Mo, Nb, Ni, Pb, Sc, Sr, Th, Ti, V, Y, Zn, Zr	ICP-OES, ICP-MS

Key to Methods in Table 4:

WDXRF	Wavelength dispersive X-ray fluorescence analyses performed at NIST
ICP-OES	Inductively coupled plasma optical emission spectrometry performed at the collaborating laboratories
ICP-MS	Inductively coupled plasma mass spectrometry performed at the collaborating laboratories

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 http://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm (accessed Nov 2010).

[2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm (accessed Nov 2010).

[3] Rukhin, A.L. *Weighted means statistics in interlaboratory studies*; Metrologia, Vol. 46, pp. 323–331 (2009).

[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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Nov 2010); 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/physlab/pubs/index.cfm (accessed Nov 2010).

[5] JCGM 101:2008; *Evaluation of measurement data – Supplement 1 to the "Guide to Expression of Uncertainty in Measurement" – Propagation of Distributions Using a Monte Carlo Method*; Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Nov 2010).

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) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.