

Certificate of Analysis

Standard Reference Material® C1251a

Phosphorus Deoxidized Copper – Cu VIII

This Standard Reference Material (SRM) is intended primarily for use in evaluating chemical methods of analysis and in the calibration of instrumental methods for analysis of copper and its alloys. A unit of SRM C1251a consists of a directionally solidified, chill-cast block, approximately 32 mm square and 19 mm thick.

Certified Mass Fraction Values: Certified mass fraction values are provided in Table 1. Value assignment categories are based on the definitions of terms and modes used at NIST for certification of chemical reference materials [2]. 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. 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 test methods listed in Table 3.

Information Mass Fraction Values: Information mass fraction values are provided in Table 2. 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 [2]. Information values cannot be used to establish metrological traceability.

Expiration of Certification: The certification of **SRM C1251a** is valid indefinitely, within the measurement uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see "Instructions for Use"). Periodic recalibration or recertification of this SRM is not required. 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, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements for certification was accomplished under the direction of J.R. Sieber of the NIST Chemical Sciences Division.

Analytical measurements for certification of this SRM were performed by T.A. Butler, M.L. Salit, J.R. Sieber, and G.C. Turk of the NIST Chemical Sciences Division, and E.S. Beary, D.A. Becker, C. Blundell, K.A. Brletic, B.I. Diamondstone, M. Epstein, J.D. Fassett, E.L. Garner, J.W. Gramlich, R.R. Greenberg, W.R. Kelly, G.J. Lutz, L.A. Machlan, J.R. Moody, P.J. Paulsen, and T.C. Rains of the NBS Inorganic Analytical Research Division. Analyses for certification were also performed at the following cooperative laboratories from 1975 to 1985: Anaconda Company, Primary Metals Division, Raritan Copper Works, Perth Amboy, NJ; Kennecott Copper Corporation, Metals Mining Division, Salt Lake City, UT and Utah Copper Division, Magna, UT; Kennecott Refining Corporation, Baltimore, MD; Phelps Dodge Refining Corporation, El Paso Works, El Paso, TX; Reading Metals Refining Company, Carteret, NJ; U.S. Metals Refining Company, AMAX Copper Division, Carteret, NJ

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

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

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

INSTRUCTIONS FOR USE

The test surface is the side opposite to the labeled surface. The certified portion extends from the test surface to a depth of 13 mm. Each packaged block has been prepared by finishing the test surface using a milling machine. The user must determine the optimum surface preparation procedure for each analytical technique. For example, preparation for X-ray fluorescence measurements at NIST involved fly cutting to avoid smearing of soft metals. The user is cautioned to use care when either resurfacing the block or performing additional polishing as these processes may contaminate the surface.

PREPARATION AND ANALYSIS(1)

The base material for the preparation of Cu VIII was vacuum melted and cast into a single ingot at Canon Muskegon Corp., Muskegon, MI. The final base material for SRM C1251a, Cu VIII, was prepared by remelting and recasting portions of the original ingot sections on the NIST water-cooled, copper plate mold assembly at the Brass Foundry, American Cast Iron Pipe Co., Birmingham, AL. The preparation and homogeneity testing plan was similar to that described in NIST Special Publication 260-2, *Standard Reference Materials: Preparation of NBS Copper-Based Spectrochemical Standards* [4].

SRM C1251a Phosphorus Deoxidized Copper is one in a series of twelve different copper composition "Benchmark" materials. The series consists of Cu "O" through Cu "XI" that was prepared in a cooperative NBS-ASTM-Industry Program. About 25 elements were included in the aim composition, covering the concentration range of about 15 to 500 mg/kg. Extensive homogeneity studies were performed at NIST Boulder, by J.G. Hust using residual resistivity ratio measurements and at NIST Gaithersburg, by C.H. Brady using metallographic studies, and by G.J. Lutz using neutron activation analysis. The results of measurements indicated the maximum material variability to be less than 10 %.

Certified Mass Fraction Values: Certified values are listed in Table 1. The measurands are the mass fraction for each analyte, expressed in milligrams per kilogram or percent, and are metrologically traceable to the SI unit of mass. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [2]. The uncertainty listed with each value is an expanded uncertainty, with coverage factor 2, calculated by combining a between-method variance [5] with a pooled, within-method variance following the ISO/JCGM Guide [3].

Table 1. Certified Values for SRM C1251a Phosphorus Deoxidized Copper – Cu VIII

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Antimony (Sb) Arsenic (As) Bismuth Bi) Cobalt (Co) Gold (Au) Iron (Fe) Lead (Pb)	$ \begin{array}{r} 14.9 & \pm & 0.4 \\ 16 & \pm & 3 \\ 3.7 & \pm & 1.0 \\ 13.2 & \pm & 1.5 \\ 15.5 & \pm & 0.9 \\ 285 & \pm & 23 \\ 23.5 & \pm & 1.0 \\ \hline 4.6 & \pm & 0.0 \\ \end{array} $	Nickel (Ni) Phosphorus (P) Selenium (Se) Silver (Ag) Tellurium (Te) Tin (Sn) Zinc (Zn)	$\begin{array}{ccccc} 23.6 & \pm & 1.0 \\ 420 & \pm & 29 \\ 11 & \pm & 2 \\ 80 & \pm & 8 \\ 16 & \pm & 2 \\ 16 & \pm & 3 \\ 24 & \pm & 3 \end{array}$
Manganese (Mn)	4.6 ± 0.9	Copper (Cu)	% 99.89 + 0.16

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⁽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.

Information Mass Fraction Values: The information value for each element is an estimate obtained from one or more NIST or collaborator test methods. No uncertainty is provided because there is insufficient information available for its assessment.

Table 2. Information Values for SRM C1251a, Phosphorus Deoxidized Copper - Cu VIII

Element	Mass Fraction (mg/kg)	Element	Mass Fraction (mg/kg)
Aluminum (Al)	< 20	Magnesium (Mg)	< 20
Cadmium (Cd)	< 3	Silicon (Si)	< 50
Chromium (Cr)	3	Sulfur (S)	35

Table 3. Methods of Analysis for SRM C1251a

Element	Method ^(a)	Element	$Method^{(a)} \\$
Al	1, 5, 8	Mg	8
Sb	6, 8	Mn	5, 8
As	3, 6, 8	Ni	3, 4, 8
Bi	3, 8	P	5, 7, 8
Cd	1, 5, 8	Se	4, 6, 8
Co	6, 8	Si	1, 7, 8
Cu	8, 9, 10	Ag	2, 6, 8
Cr	6, 8	Ag S	8
Au	2, 8	Te	4, 8
Fe	5, 6, 8	Sn	1, 3, 8
Pb	4, 8	Zn	6, 8

(a) Key to Methods in Table 3:

- 1. Atomic Absorption Spectrophotometry
- 2. Fire Assay
- 3. Photon Activation Analysis
- 4. Isotope Dilution Mass Spectrometry5. Direct-Current Plasma Atomic Emission Spectrometry
- 6. Neutron Activation Analysis
- 7. Chemical Analysis
- 8. X-ray Fluorescence Spectrometry
- 9. Inductively Coupled Plasma Atomic Emission Spectrometry
- 10. Electrogravimetry

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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://www.nist.gov/pml/pubs/sp811/index.cfm (accessed Sep 2014).
- [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://www.nist.gov/srm/upload/SP260-136.PDF (accessed Sep 2014).
- [3] 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 http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2014); 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 Sep 2014).
- [4] Michaelis, R.E.; Wyman, L.L.; Flitsch, R.; Standard Reference Materials: Preparation of NBS Copper-Based Spectrochemical Standards, NBS Misc. Publ. 260-2, U.S. Government Printing Office, Washington, DC, p. 36 (1964).
- [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.; Zhang, N.F.; *An Approach to Combining Results from Multiple Methods Motivated by the ISO GUM*, J. Res. Natl. Inst. Stand. Technol.; Vol. 105, No. 4, p. 571 (2000).

Certificate Revision History: 02 October 2014 (Change of the expiration date, editorial changes); 23 July 2002 (Original certification 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.

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