

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 494

Unalloyed Copper – Cu I

(In Cooperation with the American Society for Testing and Materials)

This Standard Reference Material (SRM) is in the form of a rod 6.4 mm (1/4 in) in diameter and 103 mm (4 in) long.* This SRM is intended for use in trace analysis of copper materials. It is designed for all techniques applicable to compositional analysis of unalloyed copper and it is particularly well suited for calibration with optical emission methods of analysis.

| Element | Certified Value ^a μg/g | Estimated Uncertainty ^b | Element | Certified Value ^a μg/g | Estimated Uncertainty ^b |
|-----------------------------|--------------------------------------|------------------------------------|------------------------------------|--------------------------------------|------------------------------------|
| Antimony ^c | 4.5 | 0.3 | Nickel ^d | 11.7 | 0.2 |
| Arsenic ^c | 2.6 | 0.3 | Selenium ^c | 2.00 | 0.15 |
| Bismuth ^c | 0.35 | 0.05 | Silver ^d | 50 | 1 |
| Chromium ^c | 2.0 | 0.2 | Sulfur ^c | 15 | 3 |
| Cobalt ^c | 0.5 | 0.1 | Tellurium ^c | 0.58 | 0.03 |
| Lead ^d | 26.5 | 0.4 | Tin ^c | 70 | 5 |
| Manganese ^c | 3.7 | 0.7 | Zinc ^c | 400 | 30 |
| Element | Certified Value ^a | | Estimated Uncertainty ^b | | |
| Copper ^c , assay | 99.91 | | 0.01 | | |

^aThe value listed for an element is the *present best estimate* of the "true" value based on the results of the analytical program for certification.

^bThe estimated uncertainty listed for a constituent is based on judgment and represents an evaluation of the combined effects of method imprecision, possible systematic errors among methods, and material variability for samples of 1.0 g or more. (No attempt was made to derive exact statistical measures of imprecision because several methods were involved in the analysis of most constituents.)

^cValues for Sb, As, Cr, Co, Mn, Se, Te, Sn, Zn, and Cu are based on agreement of determinations at NBS and cooperating laboratories; values for Bi and S are based on agreement of determinations at cooperating laboratories.

^dValues for Pb, Ni, and Ag are based on determinations at NBS by two or more of the following methods: atomic absorption and flame emission spectrometry, isotopic dilution mass spectrometry, neutron activation analysis, polarography, and spark source mass spectrometry.

*Material from the same ingot was processed to the form of small chips, designated SRM 394.

Gaithersburg, MD 20899
 April 10, 1986
 (Revision of Certificate
 dated 1-20-78)

Stanley D. Rasberry, Chief
 Office of Standard Reference Materials

(Over)

PLANNING, PREPARATION, TESTING, ANALYSIS: This material is one in a series of twelve different composition copper "Benchmark" materials, Cu "O" through Cu XI, that was prepared in a cooperative Industry-ASTM-NBS Program.

Base material for the preparation of Cu I, was supplied by Esco Corporation, Portland, Ore. Melting and casting were done at the Esco Corporation, Portland, Ore.

Preliminary results, primarily by optical emission methods of analysis, indicated a useful concentration level was present for most of the desired impurity elements and the ingot was accepted. The preliminary analyses were performed in the analytical laboratories of:

Anaconda Company, Primary Metals Division, Raritan Copper Works, Perth Amboy, N.J., P.F. Stryker and A.J. Simon.

Hecla Mining Company, Lakeshore Project, Casa Grande, Ariz., N. Ciani.

Kennecott Copper Corporation, Ledge mont Laboratory, Lexington, Mass., F.D. Leipziger.

Kennecott Copper Corporation, Kennecott Research Center, Salt Lake City, Utah, A.P. Langheinrich.

Kennecott Copper Corporation, Utah Copper Division, Refinery Plant, Magna, Utah, N.N. Linde.

Kennecott Refining Corporation, Baltimore, Md., A.A. DiLeonardi.

Magma Copper Company, San Manuel Division, San Manuel, Ariz., T.L. Young and S.K. Young.

Phelps Dodge Refining Corporation, El Paso Works, El Paso, Tex., A.L. Cardinal.

U.S. Metals Refining Company, AMAX Copper Division, Carteret, N.J., R.M. Kennedy.

The ingot was processed by the U.S. Bureau of Mines, Albany, Ore., R.A. Beall, to provide material of the highest possible homogeneity, both in billet and rod forms. The ingot was approximately 24 cm (9 1/2 in) in diameter, 81 cm (32 in) long, weighing about 318 kg (700 lb). The ingot was forged to produce a bar 15 cm (6 in) square. Five percent of the total volume was cropped from the end of the bar representative of the bottom of the original ingot and fifteen percent from the top. The bar was then cut into equal lengths of approximately 46 cm (18 in) to form three billets. One billet, selected for the rod material, was cut lengthwise forming six outer sections 3.8 cm (1 1/2 in) x 7.5 cm (3 in) with the middle section 7.5 cm (3 in) square discarded. Two of the six sections were upset forged to produce small bars 13 mm (1/2 in) square and these were swaged (12 passes) and cold drawn to the final size of 6.35 mm (0.250 in) in diameter. Following each fabrication step the material was annealed and centerless ground; all cracks and/or folds were removed prior to proceeding with the next fabrication operation.

Cooperative homogeneity studies were made at Kennecott Refining Corp., Baltimore, Md., by optical emission spectrochemical analysis, A.A. DiLeonardi. Extensive homogeneity studies were made at NBS Boulder, by residual resistivity ratio measurements, J.G. Hust, and at NBS Washington, by chemical analyses (see listing below). The results indicated the maximum gross material variability to be less than 5%.

Cooperative chemical analyses for certification were made on composite samples in the following analytical laboratories:

Anaconda American Brass Company, Research and Technical Center, Waterbury, Conn., J.T. McCrackan and V.M. Horrigan.

Anglo American Corporation of South Africa Limited, Johannesburg, Republic of South Africa, R. Murray-Smith.

Bridgeport Brass Company, Bridgeport, Conn., A.W. Young.

Carpenter Technology Corporation, Research and Development Center, Reading, Pa., C.T. Polinko.

Council for Scientific and Industrial Research, National Physical Research Laboratory, Pretoria, Republic of South Africa, L.R.P. Butler, D.B. deVilliers, and J.H. Wepener.

International Nickel Company of Canada, Limited, J. Roy Gordon Research Laboratory, Sheridan Park, Mississauga, Ontario, Canada, St. J.H. Blakeley.

Kennecott Copper Corporation, Ledge-mont Laboratory, Lexington, Mass., F.D. Leipziger.

Kennecott Copper Corporation, Kennecott Research Center, Salt Lake City, Utah, A.P. Langheinrich.

Kennecott Copper Corporation, Utah Copper Division, Refinery Plant, Magna, Utah, N.N. Linde.

Kennecott Refining Corporation, Baltimore, Md., A.A. DiLeonardi.

National Research Council of Canada, Division of Chemistry, Ottawa, Canada, D.S. Russell.

Phelps Dodge, Copper Products Company, Elizabeth, N.J., J.R. Conniff.

Phelps Dodge Refining Corporation, El Paso Works, El Paso, Tex., A.L. Cardinal.

Phelps Dodge Refining Corporation, Laurel Hill Works, Maspeth, N.Y., W.D. Charles and R. Kretschmann.

Revere Copper and Brass Incorporated, Rome, N.Y., S. Glessner.

Scovill, Metals Division, Waterbury, Conn., E.D. Wade.

South African Bureau of Standards, Physical Chemistry Division, Pretoria, Republic of South Africa, H.P. Beyers and P.G. Odendaal.

Southwire Company, Copper Division, Carrollton, Ga., G.S. Bowers.

Analyses were performed in the NBS Analytical Chemistry Division by the following: I.L. Barnes, D.A. Becker, R.K. Bell, R.W. Burke, B.I. Diamondstone, L.J. Powell, M.S. Epstein, E.L. Garner, T.E. Gills, J.W. Gramlich, E.F. Heald, G.J. Lutz, L.A. Machlan, E.J. Maienthal, T.J. Murphy, P.J. Paulsen, T.C. Rains, H.L. Rook, T.A. Rush, and M.J. Seward.

Certification of Cu I, rod material, was accomplished by the following procedures:

1. Chemical analyses at NBS made on chips representative of the billets of Cu I material. Analysts are listed above.
2. Comparative spectrochemical analyses made on the chip SRM 394, vs. chips representative of the leading and trailing ends of the rod SRM 494, by two laboratories:

Kennecott Refining Corporation, Baltimore, Md., A.A. DiLeonardi.

Magma Copper Company, San Manuel Division, San Manuel, Ariz., S.K. Young.

3. Comparative neutron activation analyses (same as indicated in 2 above) for determination of selected elements: Sb, Co, Cr, Se, Ag, and Zn, by G.J. Lutz and H.L. Rook, NBS Analytical Chemistry Division.

Although from the same original ingot, this material in rod form, SRM 494, differs somewhat from the material in chip form, SRM 394, both in composition and in the estimated uncertainties associated with the certified values. For example, the inhomogeneity of the iron in the rod material precluded certification of this element. In general, the estimated uncertainties associated with the certified values for this rod material are greater than the companion chip material because of small differences in composition that exist over the rod length. (These differences were minimized by virtue of the blending operation of the chip material.) Ultimately, the oxygen content of this rod material is expected to be certified. (The oxygen content of the chip material cannot be certified because of corrosion (oxidation) of the small particles.)

The overall direction and coordination of the preparation and fabrication of this material were performed by J.G. Hust, M.B. Kasen, and R.P. Reed, NBS, Boulder, Colorado.

The overall coordination of the NBS analytical measurements leading to certification was under the direction of I.L. Barnes.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by R.E. Michaelis.

ADDITIONAL INFORMATION: Details concerning the justification, preparation, and analysis of this material is found in the ASTM Special Technical Publication 831, "Copper Standard Reference Materials," Barnes, I.L., Gills, T.E., and Reed, W.P., "Sampling and Analysis of Copper Cathodes," Tuddenham, W.M., and Hibbeln, R.S., Eds. American Society for Testing and Materials 1984.

Prior to using this rod SRM (especially after any machining, cutting, or drilling operations) the specimen should be etched to remove any contaminated material. (Suggested etch: use a 1:1 solution of nitric acid, follow with a 1:1 solution of hydrochloric acid, rinse with distilled water, and dry in air on filter paper.)

Elements other than those certified may be present in this material as indicated below. These are *not certified* but are given as additional information on the composition.

| Elements Detected | Information Value, $\mu\text{g/g}$ |
|------------------------------|------------------------------------|
| Aluminum | (<2) |
| Cadmium | (0.5) |
| Gold | (0.07) |
| Iron | (~155) |
| Magnesium | (<1) |
| Oxygen | (230) |
| Silicon | (<2) |
| <u>Elements Not Detected</u> | |
| Calcium | (<0.3) |
| Titanium | (<0.3) |