

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 499

Unalloyed Copper -Cu VI

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

This Standard Reference Material (SRM) is in the form of rods 6.4 mm (1/4 in) in diameter and 103 mm (4 in) long.* The 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	30	5	Lead ^d	114	5
Arsenic ^c	47	4	Nickel ^d	504	10
Bismuth ^c	10.5	0.5	Selenium ^d	95	3
Cobalt ^c	0.5	0.1	Silver ^d	114	3
Iron ^d	21	2	Tellurium ^d	50	1
			Zinc ^c	41	6
Element	Certified Value ^a		Estimated Uncertainty ^b		
Copper ^c , assay	99.79		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, Co, Zn, and Cu are based on agreement of determinations at NBS and cooperating laboratories; values for Bi are based on agreement of determinations at cooperating laboratories.

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

*Material from the same original ingot was processed to the form of small chips, designated SRM 399.

Gaithersburg, MD 20899
March 24, 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 are being prepared in a cooperative Industry-ASTM-NBS Program.

Base materials for the preparation of Cu VI were supplied by the Anaconda Copper Company, Perth Amboy, N.J.; Hecla Mining Co., Casa Grande, Ariz.; International Nickel Company of Canada Limited, Ontario, Canada; Kennecott Refining Corporation, Baltimore, Md.; and Nassau Smelting and Refining Co., Inc., Staten Island, N.Y. Melting and casting of Cu VI were done at the Esco Corporation, Portland, Ore. Some of the additions were provided by the Wolverine Tube Co., Decatur, Ala., courtesy of R.E. Stanton.

Preliminary analyses, primarily by optical emission methods of analysis, 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.

Iowa State University, Ames, Iowa, R. Smitt.

Kennecott Copper Corporation, Kennecott Research Center, Salt Lake City, Utah, A.P. Langheinrich and T.N. Andersen.

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

Reading Metals Refining Corp., Reading, Pa., W.P. Darrow.

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, and 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 Gaithersburg, by chemical analyses (see listing below). The results indicated the maximum gross material variability to be less than 5%.

Cooperative chemical analyses of Cu VI, chip material, were made on composite samples in the following analytical laboratories:

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

Kennecott Copper Corporation, Research Center, Salt Lake City, Utah, A.P. Langheinrich and T.N. Andersen.

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

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

Analyses were performed in the NBS Analytical Chemistry Division by the following: I.L. Barnes, R.W. Burke, B.I. Diamondstone, M.G. Diaz, L.J. Powell, E.L. Garner, L.A. Geldner, J.W. Gramlich, G.J. Lutz, L.A. Machlan, T.J. Murphy, P.J. Paulsen, P.A. Sleeth, and R.K. Bell, ASTM-NBS Assistant Research Associate.

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

- 1. Chemical analyses at NBS made on chips representative of the billet sections of Cu VI material. Analysts are listed above.**
- 2. Comparative neutron activation analyses between rod and chip materials for determination of selected elements: Sb, Co, Cr, Se, Ag, and Zn, by G.J. Lutz, NBS Analytical Chemistry Division.**

Although from the same original ingot, this material in rod form, SRM 499, differs somewhat from the material in chip form, SRM 399, both in composition and in the estimated uncertainties associated with the certified values. 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, 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 planning, preparation, testing, and analysis of this material and other copper "Benchmark" materials are to be published in an NBS Special Publication (260 Series). Information that should be of immediate interest to the user laboratories follows:

Prior to using this rod SRM (especially after any machining, cutting, or drilling operations) the specimen should be etched to remove any contaminated materials. (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.)

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

<u>Elements Detected</u>	<u>Information Value, $\mu\text{g/g}$</u>
Aluminum	(<2)
Cadmium	(<1)
Chromium	(0.5)
Gold	(4)
Magnesium	(<1)
Manganese	(0.3)
Oxygen	(950)
Sulfur	(10)
Silicon	(<2)
Tin	(~90)
<u>Elements Not Detected</u>	
Calcium	(<0.3)
Titanium	(<1)