



# National Institute of Standards & Technology

## Certificate of Analysis

### Standard Reference Material 498

#### Unalloyed Copper - Cu V

(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. 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.

| <u>Element</u>        | <u>Certified Value<sup>a</sup></u><br><u>mg/kg</u> | <u>Estimated Uncertainty<sup>b</sup></u> | <u>Element</u>         | <u>Certified Value<sup>a</sup></u><br><u>mg/kg</u> | <u>Estimated Uncertainty</u> |
|-----------------------|--|--|------------------------|--|------------------------------|
| Antimony <sup>c</sup> | 7.4  | 0.2                                      | Nickel <sup>d</sup>    | 7.0  | 0.2                          |
| Arsenic <sup>c</sup>  | 25   | 3  | Selenium <sup>c</sup>  | 17.5   | 0.8                          |
| Bismuth <sup>c</sup>  | 2.0  | 0.3                                      | Silver <sup>d</sup>    | 20.1   | 0.4                          |
| Cobalt <sup>c</sup>   | 2.7  | 0.2                                      | Tellurium <sup>c</sup> | 10.1   | 0.2                          |
| Iron <sup>d</sup>     | 11   | 2  | Tin <sup>c</sup>       | 5  | 1                            |
| Lead <sup>d</sup>     | 10   | 1  | Zinc <sup>c</sup>      | 25   | 3                            |

| <u>Element</u>              | <u>Certified Value<sup>a</sup></u><br><u>wt %<sup>*</sup></u> | <u>Estimated Uncertainty<sup>b</sup></u> |
|-----------------------------|---|--|
| Copper <sup>c</sup> , assay | 99.98   | 0.01                                     |

\*wt % = mg/kg x 10<sup>-4</sup>

<sup>a</sup>The value listed for an element is the *present best estimate* of the "true" value based on the results of the analytical program for certification.

<sup>b</sup>The 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.)

<sup>c</sup>Values are based on agreement of determinations at NIST and cooperating laboratories.

<sup>d</sup>Values are based on determinations at NIST by two or more of the following methods; atomic absorption and flame emission spectrometry, isotopic dilution mass spectrometry, neutron activation analysis, and spark source mass spectrometry.

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

The technical and support aspects involved in the original preparation, certification, and issuance of this SRM were coordinated through the Standard Reference Materials Program by R.E. Michaelis. Revision of this certificate was coordinated through the Standard Reference Materials Program by P.A. Lundberg.

Gaithersburg, MD 20899  
May 17, 1993  
(Revision of certificate dated 4-10-86)

Thomas E. Gills, Acting Chief  
Standard Reference Materials Program

(over)

*This Certificate of Analysis has undergone editorial revision to reflect program and organizational changes at NIST and at the Department of Commerce. No attempt was made to reevaluate the certificate values or any technical data presented on this certificate.*

## **PLANNING, PREPARATION, TESTING, ANALYSIS**

This material is one in a series of twelve different composition copper "Benchmark" materials, Cu "O" through Cu XI, that were prepared in a cooperative industry-ASTM/NIST Program.

Base materials for the preparation of Cu V were supplied by the Kennecott Copper Corp., Salt Lake City, UT, and Baltimore, MD. Melting and casting of Cu V were done at Iowa State University, Ames, IA. Some of the additions were provided by the Wolverine Tube Co., Decatur, AL, courtesy of R.E. Stanton.

Preliminary analyses, primarily by optical emission methods of analysis, were performed in the following laboratories:

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

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

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

Kennecott Refining Corp., Baltimore, MD, A.A. DiLeonardi.

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

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

The ingot was processed by the U.S. Bureau of Mines, Albany, OR, R.A. Beall, to provide material of the highest possible homogeneity, both in billet and rod forms. The ingot was approximately 25.4 cm (10 in) in diameter, 56 cm (22 in) long, and weighing about 272 kg (600 lb). About five percent of the total volume was cropped from the bottom end of the ingot and about fifteen percent from the top. A selected ingot section was forged to a 3.2 cm (1 1/4 in) octagon cross-section. This section was swaged (8 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 spectro-chemical analysis, A.A. DiLeonardi. Extensive homogeneity studies were made at NIST Boulder, CO, by residual resistivity ratio measurements, J.G. Hust, and at NIST Gaithersburg, MD by chemical analyses (see listing below). The results indicated the maximum gross material variability to be less than 5%.

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

Anglo American Corp. 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 Corp., Research Center, Salt Lake City, UT, A.P. Langheinrich and T.N. Andersen.

Kennecott Refining Corp., Baltimore, MD, A.A. DiLeonardi.

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

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

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 NIST 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/NIST Assistant Research Associate.

Certification of Cu V, rod material, was accompanied by the following procedures:

1. Chemical analyses at NIST made on chips representative of the ingot sections of Cu V 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, NIST Analytical Chemistry Division.

Although from the same original ingot, this material in rod form, SRM 498, differs somewhat from the material in chip form, SRM 398, 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). (The oxygen content of the chip material cannot be certified because of corrosion (oxidation) of the small particles.)

#### SUPPLEMENTAL INFORMATION

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.)

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</u><br><u>µg/g</u> |
|------------------------------|---|
| Aluminum                     | (<2)                                    |
| Cadmium                      | (<22)                                   |
| Chromium                     | (0.3)                                   |
| Gold                         | (0.1)                                   |
| Magnesium                    | (<1)                                    |
| Manganese                    | (0.3)                                   |
| Oxygen                       | (30)                                    |
| Sulfur                       | (11)                                    |
| Silicon                      | (<2)                                    |
| <u>Elements Not Detected</u> |   |
| Calcium                      | (<0.3)                                  |
| Titanium                     | (<1)                                    |