U.S. Department of Commerce
Elliot L. Richardson,
Secretary

National Bureau of Standards
est Ambler, Acting Director

National Bureau of Standards
Certificate of Analysis
Standard Reference Material 333
Molybdenum Concentrate

This material is in the form of fine powder intended for use both in checking chemical methods of analysis and in calibration with instrumental methods of analysis.

This SRM is one of a series of four SRM's issued primarily for use in evaluation of the critically important "material balance" in the copper mining and metallurgical industries. The other three are: SRM 330, Copper Ore, Mill Heads; SRM 331, Copper Ore, Mill Tails; and SRM 332, Copper Concentrate.

Constituent	Certified Value [a] [b]	Estimated Uncertainty[c]
	Percent by Weight	
Total Copper	1.038	0.010
Molybdenum	55.3	.1
Rhenium	0.087	.001

[a] Based on samples run "as received." **CAUTION:** The bottle should be kept tightly closed except when in direct use.

[b] The certified value is the *best estimate* of the "true" value.

[c] Estimated uncertainty includes both method imprecision and material variability with samples 0.25 g (or more) for total copper, molybdenum, and rhenium.

The following values indicate the results of the analytical tests made at NBS and the Magma Copper Company.

Constituent/Method[1]	Average	Standard Deviation[2]	Number of Determinations
	Percent by Weight		
Total Copper			
Isotopic dilution mass spectrometry[3] (0.25 g samples)	1.038	0.007	11
Polarographic (1.0 g samples)	1.034	.011	6
Atomic absorption spectrometry[4] (1.0 g samples)	1.033	.002	3
Molybdenum			
Isotopic dilution mass spectrometry[3] (0.25 g samples)	55.31	.02	10
α-Benzoinoxime gravimetric[5] (0.25 g samples)	55.43	.04	10
Rhenium			
Isotopic dilution mass spectrometry[3] (0.25 g samples)	0.0869	0.0003	17
Thermal neutron activation analysis[6] (0.15 g samples)	.085	.002	9

[1] Details of the methods used are given in a separate publication [1].

[2] Of single determinations.

[3] This method has been studied extensively and the data are considered free from systematic errors [2].

[4] Results from Magma Copper Company.

[5] Average of two independent analysts at NBS.

[6] Determinations made relative to an industrial molybdenum concentrate reference material containing 0.1140 wt. % of Re, (as determined by a number of industrial laboratories and by isotopic dilution mass spectrometry at NBS.)

Washington, D. C. 20234
January 20, 1977
(Revision of Provisional
Certificate of 2-20-73)

J. Paul Cali, Chief
Office of Standard Reference Materials

(Over)

NOTE: Recommendation is made that this material be analyzed in the "as received" condition. SRM 333 contains a small quantity (about 1%) of fuel oil introduced in the flotation process. Investigations at NBS indicate that the analytical results made on the "as received" samples may be calculated to the dry-weight basis by use of an acetone stripping procedure (includes fuel oil and moisture)[1]. (For information only, a calculated MoS_2 content thus would be 93.2±0.2%.)

ADDITIONAL INFORMATION ON THE COMPOSITION: Although certification is made only for total Cu, Mo, and Re, recommended values for Au and Ag are given below:

	Au	Ag
	ppm by weight	
Fire Assay, Magma (14.58 g samples)	8.9	25.0

Neutron activation values at NBS on 0.2 g portions (final blended material) gave values of 12.5 ppm Au and 22.6 ppm Ag. Definite evidence of inhomogeneity was observed for Au on these 0.2 g portions. Recommendation is made that sample sizes of 2.5 g (or more) be used to ensure homogeneity (<5% relative), regardless of the method used.

PLANNING, PREPARATION, TESTING, ANALYSIS: The material for this SRM was carefully selected and provided to NBS by Magma Copper Company, San Manuel, Arizona, through the courtesy of T. L. Young.

At NBS, highly specialized blending and mixing procedures were employed to obtain satisfactory homogeneity. Extensive chemical analyses performed by Magma both for total copper and for molybdenum sulfide demonstrated that homogeneity was not achieved until the total blending and mixing procedures were performed three separate times [1].

Final homogeneity testing was performed at NBS simultaneously with the analytical program for certification. The maximum variability of the accepted lot was determined to be ±0.010 percent for total copper, ±0.0010 percent for rhenium; and ±0.06 percent for molybdenum (all using 0.25 g samples).

Cooperative analyses were performed at the Magma Copper Company, San Manuel, Arizona, by Y. Arias, B. Cripe, R. L. Culver, A. B. Hall, D. A. Shah, J. T. Tadano, and M. Toelkes.

Analyses were performed in the NBS Analytical Chemistry Division by R. K. Bell, E. L. Garner, T. E. Gills, J. W. Gramlich, P. D. LaFleur, L. A. Machlan, E. J. Maienthal, J. R. Moody, L. J. Moore, and T. J. Murphy.

The overall direction and coordination of the technical measurements at NBS leading to certification were performed under the direction of W. R. Shields and 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.

REFERENCES

[1] J. R. Moody, I. L. Barnes, and R. E. Michaelis, Standard Reference Materials: Copper Ore, Mill Heads - SRM 330; Copper Ore, Mill Tails - SRM 331 Copper Concentrate - SRM 332, and Molybdenum Concentrate - SRM 333, Nat. Bur. Stand. Spec. Publ. 260-xx (in press).

[2] W. R. Shields, Editor, Nat. Bur. Stand. Tech. Note 546, (1970).