National Bureau of Standards

Certificate of Analysis

Standard Reference Material 682

High-Purity Zinc

This Standard Reference Material (SRM) is intended primarily for the calibration of instruments and the evaluation of chemical methods used in zinc analysis. SRM 682 is in the form of a semicircular bar segment, 57 mm diameter (2 1/4 inch), 25.4 mm (1 inch) deep at mid-diameter and 19 mm long (3/4 inch).

Element[1]	Recommended Value (ppm by wt.)	Range of Values Reported[2] (ppm by wt.)	Methods of Analysis[3]
Copper	0.042	[0.038 - 0.050]	a,b
Cadmium	(.1)[4]	---	c
Iron	(.1)	---	c
Silver	(.02)	---	c
Tin	(.02)	---	c

[1] Other elements were detected by spark-source mass spectroscopy and by neutron activation. These are listed below with an estimated conservative upper limit of concentration; all values are given in parts per million by weight.

Al (<0.03)	C (<0.5)	Cr (<0.06)	Li (<0.003)	N (<0.06)	O (<0.5)
B (<.01)	Ca (< .2)	F (< .03)	Mg (< .1)	Na (< .2)	Si (< .5)
Be (<.03)	Cl (< .5)	K (< .1)	Mn (< .03)	Ni (< .1)	Ti (< .2)

In addition, spark-source mass spectroscopy on some sample sizes of 20-50 mg showed definite evidence of gross inhomogeneity for Bi, Pb, and Tl. For Pb, this was partially confirmed by polarography using 1-g samples, which showed Pb heterogeneity to a lesser degree. Residual resistivity ratio measurements on samples about 10-15 g also indicated some limited variability for electrically active elements such as Bi, Pb, and Tl; however, the minimum ratio of 33,000 (R_{273K}/R_{4K}) would be inconsistent with any one of these elements exceeding 0.1 ppm (by weight) on a 10-15 g sample size.

No other elements were detected, and most elements have an estimated limit of detection by spark source mass spectrometry of 0.01-0.05 ppm. Direct interference by Zn on S precluded any reasonable estimate for this element. Slight interferences also occurred for Ba, Cs, Hg, and Pt, but none of these were detected at about the 0.2 to 1 ppm level.

Neutron activation analysis[3d], did not detect As, Ga, Sc, and W at about the 0.005 ppm level, nor Au at about the 0.02 ppb level.

[2] The range of values reported is that of the eight individual determinations made by the two analytical methods used. The recommended value is based on considerations of the estimated systematic bias of each of the methods. Six of the eight values reported were in the range from 0.040 to 0.044 ppm.

[3] a. Atomic Absorption Spectrometry (T.C. Rains)
b. Spectrophotometry (R.W. Burke)
c. Spark-Source Mass Spectrometry (P.J. Paulsen)
d. Neutron Activation Analysis (B.A. Thompson and D.A. Becker)

[4] Values in parentheses are not certified as only one method of analysis was used. They are provided for information only.

Gaithersburg, MD 20899
January 15, 1988
(Revision of certificates
dated 7-9-68 & 2-1-71)

Stanley D. Rasberry, Chief
Office of Standard Reference Materials

(over)

This reference material should be of interest to chemists, physicists, and materials engineers. Its very high purity makes it ideal as a starting material for the preparation of phosphors and other solid-state materials, where a knowledge of the purity of the material is important. It will also meet the needs of analysts working at trace level concentrations of elements in high-purity zinc. The material was prepared by Cominco American, Inc., from a special lot of high-grade electrolytic zinc that was further purified by vacuum distillation, zone-refining, and degasification. Each bar was etched, dried, and sealed in a polyethylene pouch to minimize contamination.

Homogeneity testing was performed at the NBS Gaithersburg and Boulder laboratories. Specimens were carefully chosen to represent the extreme variations that might be expected as a result of the preparation procedures. Although the spark-source mass spectrographic results indicated gross inhomogeneity for lead, thallium, and bismuth, the residual resistivity measurements indicate that this segregation should be minimized provided the specimen size is increased to ten grams or more and is representative of the full cross section. Activation results revealed some relatively minor inhomogeneity for sodium and antimony. Residual resistivity ratio (R_{273K}/R_{4K}) results varied from 33,000 to 38,000.

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.

CAUTION

Before use, it is recommended that possible surface contamination be removed by placing the specimens in high-purity nitric acid for about one minute, followed by rinsing in distilled water.