U. S. Department of Commerce
Maurice H. Stans
Secretary

National Bureau of Standards
L. M. Branscomb, Director

Certificate of Analysis

STANDARD REFERENCE MATERIAL 682

High-Purity Zinc[1]

This standard of very high-purity zinc metal is issued as a special research material to further both chemical and physical methods of characterization. Two other zinc metal standards of a lesser degree of purity are also available: SRM 683 in the form of semicircular bar segments and SRM 728 in shot form. The same starting material was used for all three standards; however, this high-purity zinc material was further purified by vapor distillation, zone-refining, and degasification.

Element[2]	Recommended Value (ppm by wt.)	Range of Values Reported[3] (ppm by wt.)	Methods of Analysis[4]
Copper	0.042	(0.038 - 0.050)	AAS,SPPH
Cadmium	(.1)[5]	- - - - -	SSMS
Iron	(.1)	- - - - -	SSMS
Silver	(.02)	- - - - -	SSMS
Tin	(.02)	- - - - -	SSMS

1. The material is in the form of semicircular bar segments about 2 1/4 inches in diameter, 1 inch deep at mid-diameter, and 3/4 inch long.

2. In the course of analyses by spark-source mass spectroscopy and by neutron activation, other elements were detected as being present. 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

Spark-source mass spectrographic results on some sample sizes of 20 to 50 mg showed definite evidence of gross inhomogeneity for the elements Bi, Pb, and Tl. This was partially confirmed for Pb as determined by polarography using 1 g samples, which showed Pb heterogeneity but to a lesser degree. Residual resistivity ratio measurements made on about 10 to 15 g samples also indicated some limited variability for the 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 the three elements exceeding 0.1 ppm (by weight) on a 10-15 g sample size.

No other elements were detected, with most elements having an estimated limit of detection by spark source mass spectrographic analyses of 0.01-0.05 ppm. Direct interference by Zn on S precluded any reasonable estimate for this element. Slight interference also occurred for Ba, Cs, Hg, and Pt, but each was not detected at about the 0.2 to 1 ppm level.

By neutron activation analysis[4], the elements As, Ga, Sc, and W were not detected at about the 0.005 ppm level and Au was not detected at about the 0.02 ppb level.

3. 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.

4. AAS – Atomic Absorption Spectrometry (T. C. Rains)
SPPH – Spectrophotometry (R. W. Burke)
SSMS – Spark-Source Mass Spectrometry (P. Paulsen)
NAA – Neutron Activation Analysis (B. A. Thompson and D. A. Becker)

5. Values in parentheses are not certified since only one method of analysis was used, but are provided for additional information on the composition.

Washington, D. C. 20234
July 9, 1968
(Revised February 1, 1971)

J. Paul Cali, Acting Chief
Office of Standard Reference Materials

(over)

This standard is intended as a research material which should be of interest to the chemist, physicist, and materials engineer. Its very high purity makes it ideal as a starting material for the preparation of phosphors and as a solid-state matrix, where a knowledge of the purity of the material is important. It will also meet the urgent needs of analysts working at trace level concentrations of elements in high-purity zinc. The material should serve for the development of new or improved methods and techniques in extending the sensitivity of detection in the determination of trace constituents by chemical, optical emission and spark source mass spectrochemical, activation, and resistivity methods. The material was prepared by Cominco American, Inc. from a special lot of high-grade electrolytic zinc which 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 by NBS Washington and NBS Boulder with samples 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 sample 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 sample in high-purity dilute nitric acid for about one minute, followed by rinsing in distilled water.