# National Institute of Standards & Technology

# Certificate

## Standard Reference Material 741

### Tin

### Primary Freezing Point Standard

### 231.928 °C

### International Temperature Scale of 1990

The temperature given above is the value assigned to the freezing point of pure tin as one of the fixed points on the International Temperature Scale of 1990 [1]. Based on chemical analyses and freezing point experiments, the freezing point of SRM 741 is estimated to be:

231.928 ± 0.001 °C

SRM 741 is high-purity tin, with a total of all other elements that would affect the freezing point of about 1 ppm. The freezing points (with 25 percent in the solid phase) of eleven selected specimens of SRM 741, each of which weighed 1300 g, agreed within 0.1 mK. (For full details of the preparation and comparison of freezing points of tin specimens, see Proceedings of the 5th Symposium on Temperature, June 1971.)

In the freezing-point experiments, the furnace surrounding the sample-cell was maintained at 0.9 K below the tin point (where the total freezing time, typically about 14 hours, was dependent on the degree of super-cooling). During these experiments, the freezing points generally did not decrease by more than 0.1 mK over a period of seven hours. The standard deviation of the freezing temperatures of each specimen was ± 0.05 mK from freeze to freeze (with about 25 percent in the solid phase).

The tin metal for the preparation of SRM 741 was obtained from Cominco American, Inc., Spokane, Washington.

Temperature studies were performed by G. T. Furukawa, J. L. Riddle, and W. R. Bigge of the NBS Heat Division.

Evaluations of purity and homogeneity were performed in the NBS Cryogenics Division (Boulder, CO.) by V. A. Deason, A. F. Clark, and R. L. Powell; and in the NBS Analytical Chemistry Division by C. W. Mueller, P. J. Paulsen, H. L. Rook, and P. D. LaFleur.

This certificate is a revision of the certificate dated July 18, 1972. The changes consist primarily of the conversion of temperatures on the IPTS-68 to those on the ITS-90 by B.W. Mangum of the Chemical Process Metrology Division.

The technical and support aspects involved in the revision, update and issuance of this Standard Reference Material were coordinated through the Standard Reference Materials Program by J. C. Colbert. The original coordination of certification efforts was performed by R. E. Michaelis.

Gaithersburg, MD 20899
June 26, 1990
(Revision of certificate dated 7-18-72)

William P. Reed, Acting Chief
Standard Reference Materials Program

(over)

For further information on temperature scales and metal freezing points, see:

[1] Preston-Thomas, H., The International Temperature Scale of 1990, Metrologia 27, 3 (January 1990).

[2] McLaren, E. H., The Freezing Point of High-Purity Metals and Precision Temperature Standards, Temperature, its Measurements and Control in Science and Industry, Vol.3, Part 1, Rheinhold Publishing Corp., New York, N. Y. (1962).

## SUPPLEMENTARY INFORMATION

### Preparation, Testing and Handling of High-Purity Tin Freezing Point Material

PREPARATION: The tin was prepared to obtain material of the highest possible purity and homogeneity. The starting material was a carefully selected lot of refined tin (99.97%). Purification processes included electrolysis, zone refining, homogenization, and degasification. Close analytical control was maintained throughout the entire preparation. The final bars (about two feet long) were individually sealed in argon-filled polyethylene bags.

TESTING: Selected samples, representative of the entire lot, were tested. The tests were initially performed by optical emission and spark source mass spectroscopic procedures and by residual resistivity ratio measurements. As a result of these tests, the material was tentatively accepted both from the homogeneity and composition standpoints. Finally, freezing point studies of selected specimens were performed to determine the suitability of the material. Based on the specimens tested, the freezing point of the lot was estimated to be within 0.7 mK of that of 100 percent pure tin.

HANDLING: Any handling procedures on high-purity materials are apt to introduce contamination. The procedures described below were followed to minimize contamination, particularly with respect to the elements that could affect the freezing-point measurements.

1. The tin bars were cut dry with a carbide tipped cutter in such a way that only the carbide tip touched the tin metal. At no time were the tin bars touched by hand (polyethylene gloves were used), and at no time were the bars in contact with any part of the milling machine other than through the polyethylene cover.

2. The resulting specimens were acid cleaned by immersion in 40% HCl for one minute, and then in 40% HCl plus 10% $HNO_3$ for one minute. After acid cleaning, the bars were rinsed in distilled water, then in alcohol, and finally air dried.

3. Individual specimens were sealed in polyethylene bags.

SHOULD ANY CUTTING OF SRM 741 BE REQUIRED OR SHOULD ANY SURFACE CONTAMINATION BE SUSPECTED, IT IS RECOMMENDED THAT THE HANDLING AND CLEANING PROCEDURES DESCRIBED ABOVE BE EMPLOYED JUST PRIOR TO USE.