Department of Commerce
Juanita M. Kreps
Secretary

National Bureau of Standards
Ernest Ambler, Director

National Bureau of Standards
Certificate

Standard Reference Material 768

Superconductive Thermometric Fixed Point Device
(0.015 K - 0.208 K)

R.J. Soulen, Jr., and R.B. Dove

This Standard Reference Material is a superconductive thermometric fixed-point device containing five high-purity materials--$AuIn_2$, $AuAl_2$, Ir, Be, and W.

The superconductive transition temperature (T_c) of each of the five materials is certified to be reproducible to ±0.2 mK, when used as stated in this certificate. There is sufficient variation between the T_c values for different units that a separate calibration certificate accompanies each unit.

The values of the transition temperatures assigned to each unit are derived from a temperature scale established and maintained at the National Bureau of Standards. It was obtained by intercomparing Co-60 gamma-ray aniosotropy [1] and noise [2] thermometers between 0.015 K and 0.05 K [3]. From 0.05 K to 0.5 K the noise thermometer was used alone. Furthermore, the temperature scale obtained from the comparison was "smoothed" with an interpolation thermometer, a spherical single crystal of cerium magnesium nitrate [4]. This smoothed temperature scale (dubbed NBS-CTS-1) is maintained at NBS via one or more germanium resistance thermometers that were calibrated at the same time as the gamma-ray, noise and CMN thermometers; it is also maintained by at least two units of SRM 768.

The experimental reproducibilities of the devices were determined by measuring germanium thermometer resistances corresponding to the midpoints of the transitions occurring in 8 devices that were cycled to cryogenic temperatures 10 times. Based on these measurements, the transition temperature of a given sample is certified to be within ±0.2 mK of the value assigned it on the calibration certificate. It should be emphasized that the temperature scale used to assign values to the five transitions in SRM 768 may eventually be replaced by improved versions, in which case the user of SRM 768 will be notified of the reassignments through publication and directly by mail. These possible changes notwithstanding, each unit will always provide five reference temperatures with an uncertainty not exceeding ±0.2 mK.

The technical measurements and characterizations of this Standard Reference Material were performed by R. J. Soulen, Jr., and R. B. Dove of the NBS Temperature Measurements and Standards Division.

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. K. Kirby.

Washington, D.C. 20234
December 13, 1978

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

Use of SRM 768

To use SRM 768 effectively, three particular features of the device must be considered.

First: The user must provide good thermal contact between the fixed-point device and the thermometer to be calibrated. This may be achieved by screwing the 6-32 threaded gold-plated stud of the SRM 768 unit into a mating gold-plated tapped hole in a solid, high-conductivity copper block to which the other thermometers are similarly attached. (Caution: The 6-32 stud on the unit is annealed copper and can be sheared off if too much torque is applied; "finger-tight" is sufficient.) It is also necessary to thermally temper the leads for the mutual inductance coils and thermometers to reduce heat leaks. Because of possible strain, contamination, or degradation of thermal contact between the five high-purity materials and the 6-32 stud, these materials should not be removed from the device.

Second: The user must reduce the ambient magnetic field experienced by the unit to the level of $1 \mu T$ (0.01 gauss) in order to prevent depression of T_c and supercooling. At NBS, fields of this level were obtained either by cancelling the local earth field with Helmholtz coils mounted outside the cryostat or by two mu-metal shields (one at 4 K and the other outside the cryostat).

Third: The user must exercise care in the selection of suitable measurement techniques and circuitry. A stable mutual inductance bridge [5] should be used in conjunction with the device coils. We have found that a peak-to-peak current (at 400 Hz) in the primary coil of 140 μA produces a measurable signal for the observation of the W transition yet generates only 1.1 nW of heat. No thermal gradients have been observed at NBS using these parameters. The other four transitions are stronger, requiring a measurement current of only 29 μA that generates 0.13 nW. In most applications, a given T_c can be reached by warming the sample block while observing the mutual inductance bridge imbalance on a meter. Having ascertained the meter readings corresponding to the limits of the transition, the operator can maintain T_c by warming the sample block a second time until the meter shows that the transition midpoint has been reached. The heating rate should be sufficiently slow (about 5 minutes) that the superconductive-to-normal transition is not completed. By maintaining the heating rate at a suitable value, the operator can maintain T_c for an extended length of time. Note: Electronic drift in the measuring circuit can mislead the operator--a drift of one-half the transition width in the monitoring circuit can result in failure to maintain the transition temperature.

A detailed discussion of the purity and preparation of the superconductive samples used in SRM 768; the experimental determination of the T_c values, uncertainties, and reproducibilities; and the use of SRM 768 is published in NBS Special Publication 260-62; "SRM 768: Temperature Reference Standard for Use Below 0.5 K."

References

1. T. L. Thorp, B. B. Triplett, W. D. Brewer, N. E. Phillips, J. E. Templeton, T. W. Start, and P. H. Schmidt, J. Low Temp. Phys. **3,** 589 (1970).
2. R. A. Kamper and J. E. Zimmerman, J. Appl. Phys. **42,** 132 (1971).
3. The temperature scale is based in part on the results reported by H. Marshak and R. J. Soulen, Jr., Journal de Physique, **39,** C6-1162 (1978).
4. J. M. Daniels and F. N. Robinson, Phil. Mag. **44,** 630 (1953). See also R. P. Hudson, H. Marshak, R. J. Soulen, Jr., and D. B. Utton, J. Low Temp. Phys. **20,** 1 (1975).
5. R. J. Soulen, Jr., J. F. Schooley, and G. A. Evans, Jr., Rev. Sci. Instru. **44,** 1537 (1973).