U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts. Director

National Bureau of Standards

Certificate

Standard Reference Material 767

Superconductive Thermometric Fixed Point Device

J. F. Schooley, R. J. Soulen, Jr., and G. A. Evans, Jr.

This Standard Reference Material is a superconductive thermometric fixed-point device containing five high-purity elements--lead, indium, aluminum, zinc, and cadmium--in the form of long, thin cylinders.

The superconductive transition temperature of each of the five elements is certified to be reproducible to ± 1 mK, when used as stated in this Certificate.

The assigned transition temperatures and uncertainties for each lot of the elements used in this device as well as the derivations of these temperatures are given below.

Element	Assigned T_c (K)	Derivation	Experimental Reproducibility (mK)
Lead	7.201 ± 0.0025	(NBS 2–20 K) + magnetic thermometry	0.32
Indium	3.416 ± .0015	(NBS 2–20 K) + magnetic thermometry	.15
Aluminum	1.174_6 ± .002	Direct Calib. – ^{3}He v.p. T_{62}	.28
Zinc	0.844 ± .0015	T_{62} + magnetic thermometry	.22
Cadmium	0.515 ± .0025	T_{62} + magnetic thermometry	.30

The values of the transition temperatures were derived from paramagnetic salt thermometry in conjunction with the T_{62} ^{3}He vapor pressure-temperature scale [1] or with the NBS 2-20 K acoustical temperature scale. [2] For T_{62} a set of germanium resistors was calibrated at several temperatures against readings taken on a ^{3}He vapor pressure bulb, and the paramagnetic susceptibility of a cerous magnesium nitrate single crystal sphere was used to evaluate the Zn and Cd transition temperatures. The germanium resistances corresponding to the Al transition were evaluated directly against the ^{3}He bulb temperature. The Pb and In transition temperatures were obtained by paramagnetic salt thermometry interpolation between points of germanium resistors that had been calibrated on the NBS 2-20 K scale.

The experimental reproducibilities of the prepared devices were determined by measuring germanium thermometer resistances corresponding to the midpoints of the transitions occurring in about one hundred devices. Based on these measurements, the transition temperatures of these devices are certified to be reproducible to ±1 mK. It should be emphasized that the assigned T_c values may be expected to change with increased accuracy in assignment of temperature values to the respective germanium resistances and with increased accuracy of the temperature scales themselves. However, these devices should continue to provide reproducible thermometric reference points regardless of such variations in assigned T_c values.

The technical measurements and characterizations of this Standard Reference Material were performed by J. F. Schooley, R. J. Soulen, Jr., and G. A. Evans, Jr., of the National Bureau of Standards Heat 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. E. Michaelis.

Washington, D.C. 20234
June 5, 1974

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

USE OF SRM 767

To use SRM 767 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, avoiding stray sources of heat that might generate appreciable thermal gradients. The former principle can be met by screwing the SRM 767 device into a tapped, lightly-greased hole in a solid, high-conductivity copper block to which the thermometers are attached in similar fashion. The latter consideration can be satisfied by thermally tempering the SRM 767 coil leads and any thermometer leads to the copper block itself and to the primary cooling system. By maintaining the cooling system at a temperature only slightly lower than that of the copper block, the operator can provide two nearly isothermal points for removal of unwanted heat via the leads. Because of possible strain and contamination, the elements should not be removed from the device.

Second, the user must control the ambient magnetic field. The T_c values of the various superconductors are depressed by a magnetic field, and hysteresis (usually caused by supercooling) often accompanies this effect. At NBS the ambient field was reduced below 1 μT by Helmholtz coils mounted outside the cryostat. Other shielding, such as mu-metal or superconductive cans, should be used with considerable care, because the actual internal field in such devices depends strongly on their history. If a small ambient field must be tolerated, the T_c values should be reduced by the following factors: for lead, 4.9 mK/G (note: 1 G = 10^{-4}T); for indium, 6.4 mK/G; for aluminum, 5.6 mK/G; for zinc, 8.6 mK/G; and for cadmium, 10.6 mK/G. For ambient fields greater than 1 gauss, the linear approximations used above are inappropriate, and the corrections should be obtained from actual experimental critical field curves. [3]

Third, the user must exercise care in the selection of suitable measurement techniques and circuitry. A stable mutual-inductance bridge [4] should be used in conjunction with the device coils. The capability of maintaining the device temperature at a fixed point depends upon the stability of the bridge output. The device's primary-coil current should not exceed 20 μA, so that the primary coil field is less than 1 μT. 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 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 T_c at all.

A detailed discussion of the purity and preparation of the superconductive cylinders used in SRM 767; the experimental determination of the T_c values, uncertainties, and reproducibilities; and the use of SRM 767 is published in Special Publication 260-44, Preparation and Use of Superconductive Fixed Point Devices, SRM 767. SP 260-44 is available from the Superintendent of Documents, U. S. Government Printing Office, Washington, D. C. 20402; as SD Catalog No. C13.10: 260-44.

REFERENCES

1. Sherman, R. H., Sydoriak, S. G., and Roberts, T. R., J. Res. Nat. Bur. Stds. 68A, 579 (1964).

2. Plumb, H. H., and Cataland, G., Metrologia 2, 127 (1966).

3. A summary of critical field curves can be found on pp. 224 and 225 of "Superconductivity", by D. Shoenberg, Cambridge, University Press, England (1962).

4. A circuit for this purpose is described by R. J. Soulen, Jr., J. F. Schooley, and G. A. Evans, Jr. in Rev. Sci. Instr. 44,1537 (1973).