National Institute of Standards & Technology

Certificate

Standard Reference Material 767a

Superconductive Thermometric Fixed Point Device

Device No. PP

This Standard Reference Material (SRM) is a superconductive, thermometric fixed-point device containing six high-purity elements--niobium, lead, indium, aluminum, zinc, and cadmium--in the form of long, thin rods, enclosed in a measuring coil pair and mounted on a copper stud. It is intended to provide secondary fixed points on the International Temperature Scale of 1990 (ITS-90)[1] and to be used to calibrate germanium resistance and other interpolating thermometers.

The transition temperature and transition width measured for each of the elements used in this device are given below. The superconductive transition of each of the six elements is certified to be reproducible to ±0.3 mK when measured as recommended in this Certificate.

Elements	Measured Transition Temp. T_c (K)	Measured Transition Width W (mK)
Niobium	9.2488	23
Lead	7.19974	0.2
Indium	3.41483	0.5
Aluminum	1.18074	0.3
Zinc	0.85024	0.5
Cadmium	0.51981	1.7

The transition temperatures of the six elements were obtained with a Re-Fe [2] resistance thermometer using an automatic ac resistance bridge [3]. The thermometer was calibrated against the EPT-76 Scale in the NBS Low Temperature Scale Laboratory [4]. Two SRM 767a devices are maintained in the calibration apparatus as internal standards; the experimental variations of these reference-sample transition temperatures in the calibration system have not exceeded 0.2 mK over the calibration period.

Accompanying this certificate is a copy of the x-y plot of each transition as it was recorded during the calibration process. This plot can be employed to compare the NIST (formerly the National Bureau of Standards) determination of the transition width and the midpoint with the user's measurements.

The preparation, technical measurements, and characterizations of this Standard Reference Material were performed by J. F. Schooley and G. A. Evans, Jr., 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. K. Kirby.

The August 13, 1990 revision of this certificate consisted primarily of the conversion of temperature of the IPTS-68 to those on the ITS-90 by B. W. Mangum of the Chemical Process Metrology Division.

Gaithersburg, MD 20899
February 14, 1992
(Revision of certificate dated 8-13-90)

William P. Reed, Chief
Standard Reference Materials Program

USE OF SRM 767a

The accurate use of the superconductive thermometric fixed-point device SRM 767a requires consideration of the following items:

1) The user must provide good thermal contact between the fixed-point device and the thermometer(s) to be calibrated, and avoid stray sources of heat that might generate appreciable thermal gradients. The former requirement can be met by screwing the SRM 767a device into a tapped, lightly-greased hole in a solid, high-conductivity copper block to which the thermometers are attached in similar fashion. Stray heating can be minimized by tempering the SRM 767a coil leads and the thermometer leads to the copper block and to the primary cooling system. Temperature gradients within the copper block can be minimized by using the lowest possible power level on any heater attached to the block.

2) 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. During the NIST calibration process, the ambient field was reduced below 1 T by three mutually orthogonal sets of 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 depend strongly on their previous treatment. If a small ambient field must be tolerated, the transition temperatures quoted herein should be reduced by the following amounts: for niobium, 1.0 mK/G (Note: $1G = 10^{-4}T$); for lead, 4.9 mK/G; 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 [5].

3) The user must exercise care in the selection of suitable measurement techniques and circuitry. A stable mutual inductance bridge [6] should be used in conjunction with the device coils; maintaining the device temperature at a fixed point depends upon the stability of the bridge output. Electronic drift in the measuring circuit can mislead the user--a drift of one-half the transition width in the monitoring circuit can result in failure to maintain T_c. The device's primary coil current should not exceed $20 \mu A$, so that the primary coil field is less than $1 \mu T$. 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 user can reach T_c by warming the sample block a second time until the meter shows that the transition midpoint has been reached. By maintaining the heating rate at a suitable value, the user can maintain T_c for an extended length of time.

4) Under no circumstances should individual superconductive samples be removed from the device. The samples have been carefully annealed and mounted so as to minimize strains. Rough handling of the device or touching of the samples can easily lead to broadened superconductive transitions and consequent shifts in T_c.

REFERENCES

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

2. R. L. Rusby, p. 125 in Temperature Measurement, Conference Series No. 26, Institute of Physics, London (1975). Also see The Rhodium-Iron Resistance Thermometer: Ten Years On, R. L. Rusby, p. 829 in Temperature, Its Measurement and Control in Science and Industry, J. F. Schooley, Ed., Vol. 5, Part 2 (1982).

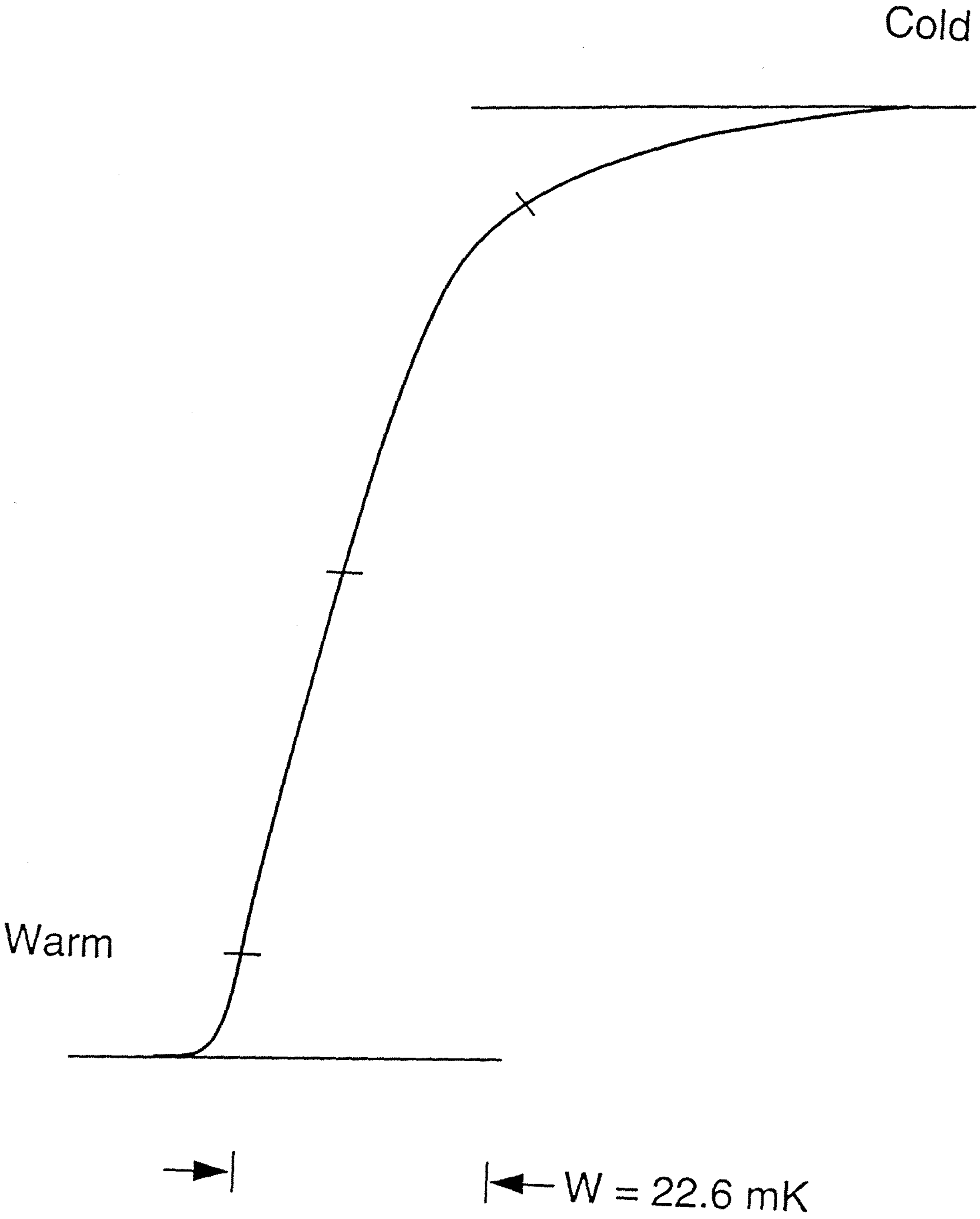
3. R. D. Cutkosky, IEEE Trans. Instr. and Meas. IM-29, 330 (1980). Also see Automatic Resistance Thermometers Bridges for New and Special Applications. R. D. Cutkosky, p. 711 in Temperature, Its Measurement and Control in Science and Industry, J. F. Schooley, Ed., Vol. 5, Part 2 (1982).

4. Realization of the 1976 Provisional 0.5 K to 30 K Temperature Scale at the National Bureau of Standards, E. R. Pfeiffer and R. S. Kaeser, p. 159 in Temperature, Its Measurement and Control in Science and Industry, J. F. Schooley, Ed., Vol. 5, Part 1 (1982).

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

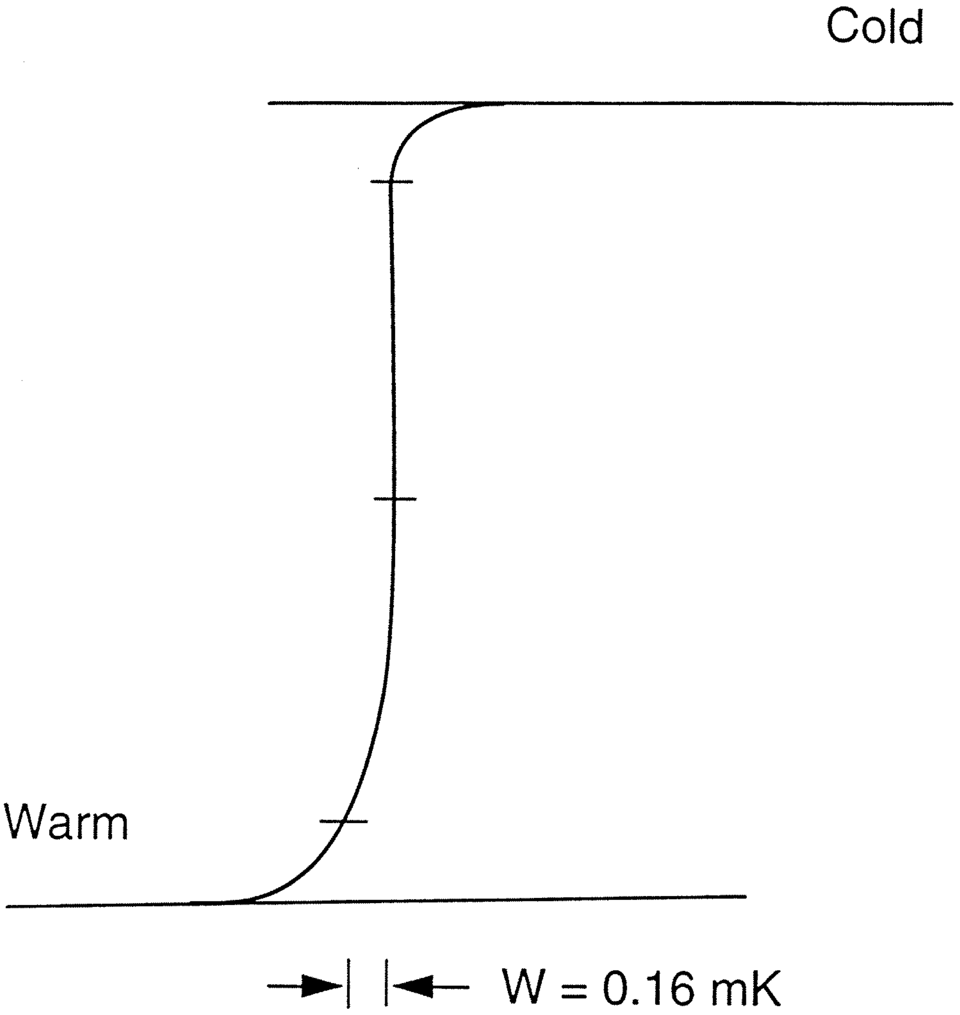
6. Superconductive Thermometric Fixed Points, J. F. Schooley and R. J. Soulen, Jr., p. 251 in Temperature, Its Measurement and Control in Science and Industry, J. F. Schooley, Ed., Vol. 5, Part 1 (1982).

SRM 767a
Nb Sample PP
H ≈ 0 GAUSS



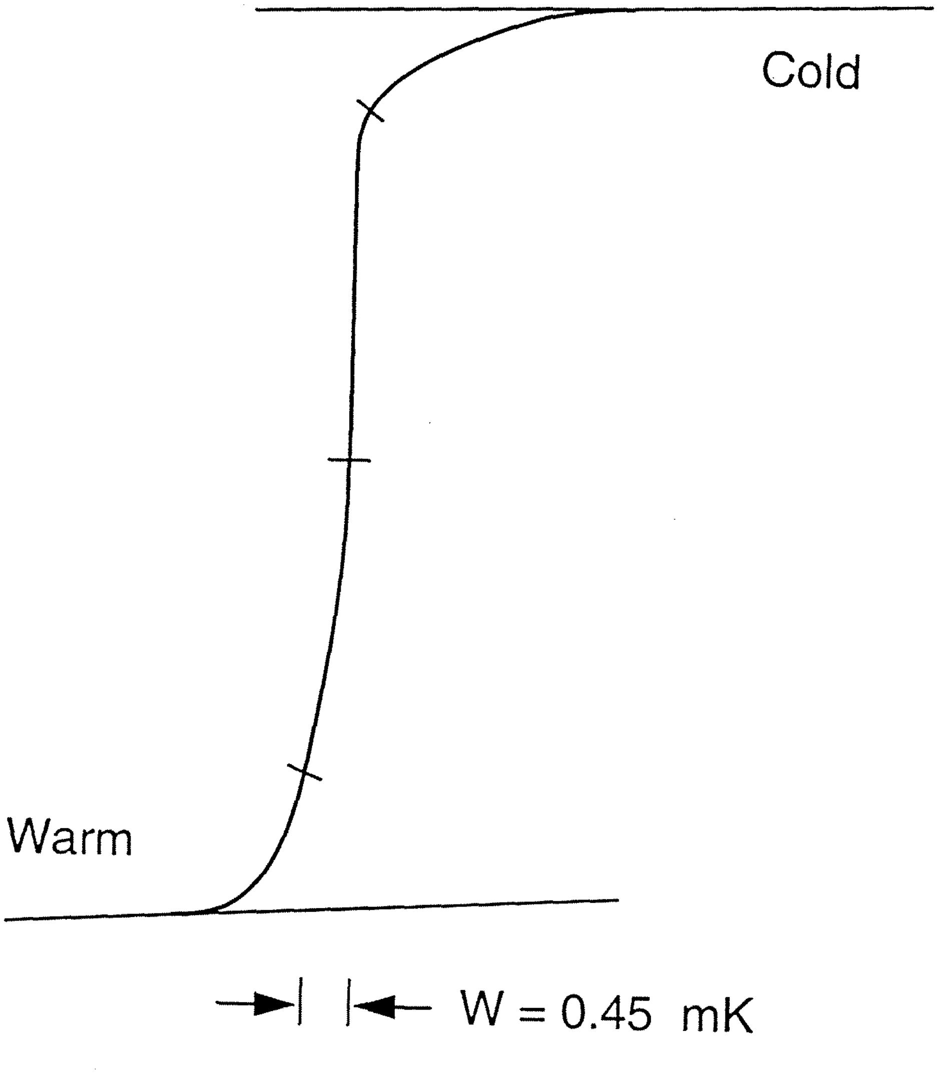
$R\,(T_C) = 8.062230\ (10)_{\Omega} \Rightarrow 9.24925\mathrm{K}$ (EPT-76)

SRM 767a
Pb Sample PP
$H \approx 0$ GAUSS



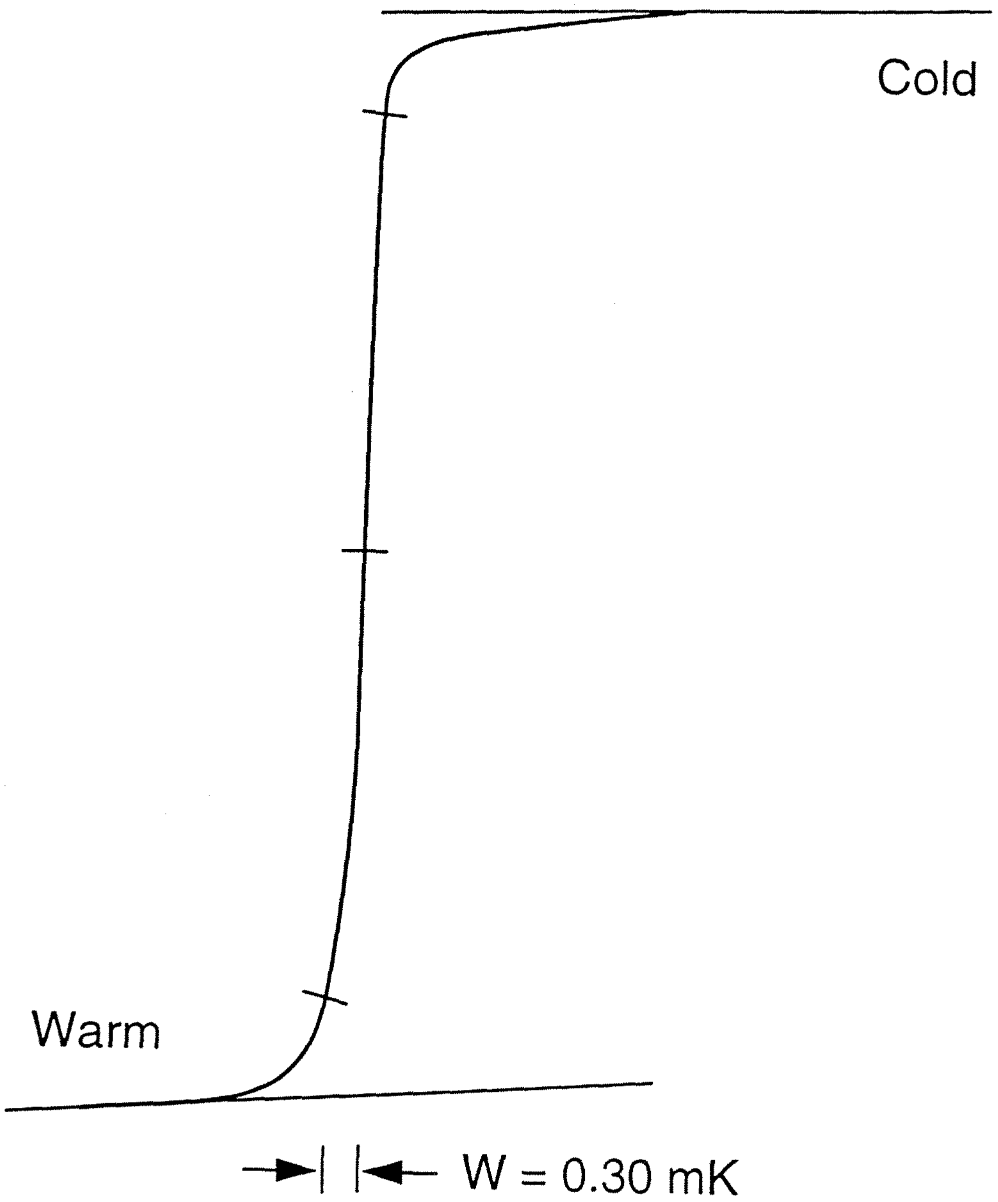
$$R\ (T_C) = 7.442412(10)_{\Omega} \Rightarrow 7.20004\text{K (EPT-76)}$$

SRM 767a
In Sample PP
H ≈ 0 GAUSS



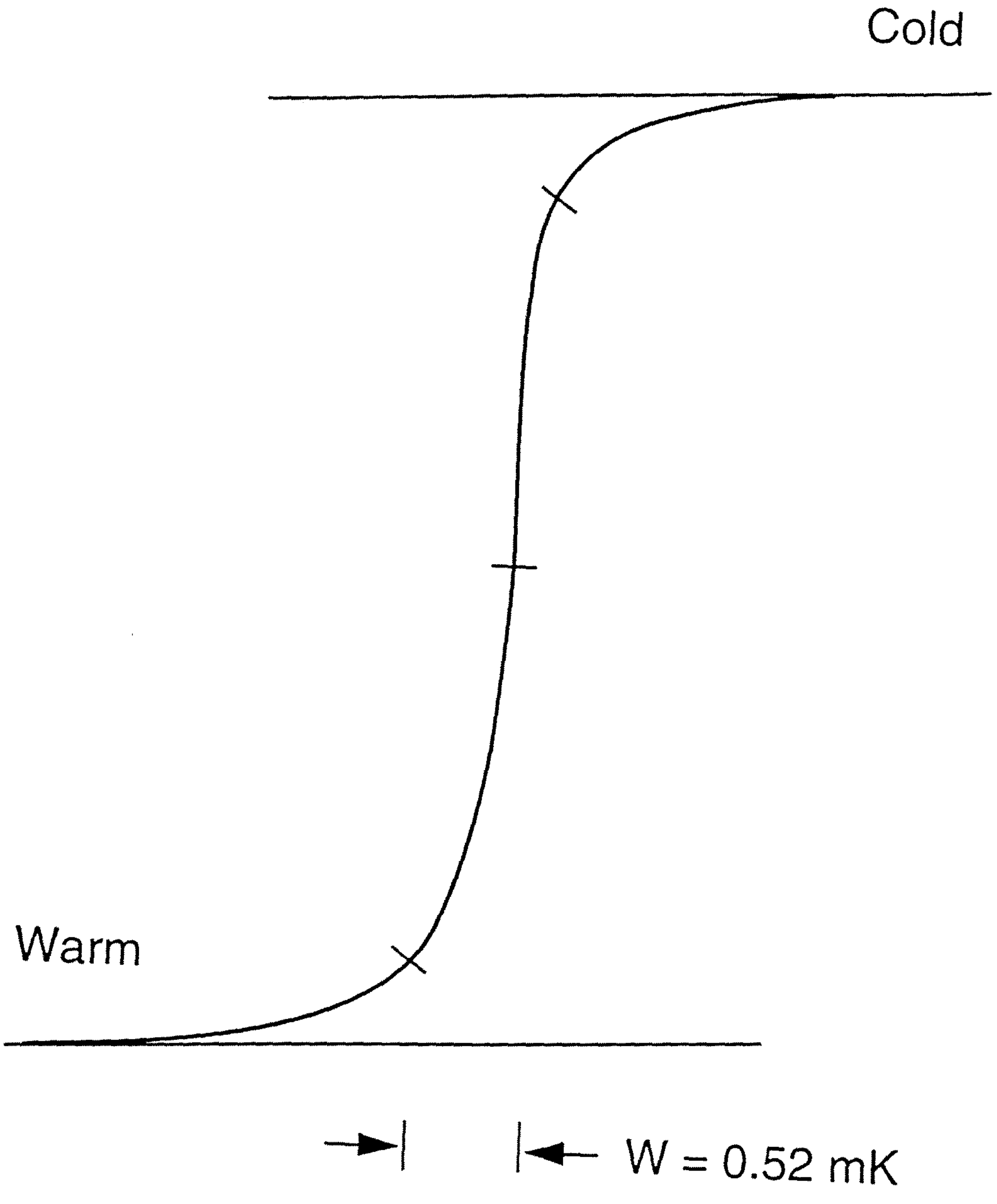
$R\ (T_c) = 5.947900(10)_\Omega \Rightarrow 3.41483\text{K (EPT-76)}$

SRM 767a
Al Sample PP
$H \approx 0$ GAUSS



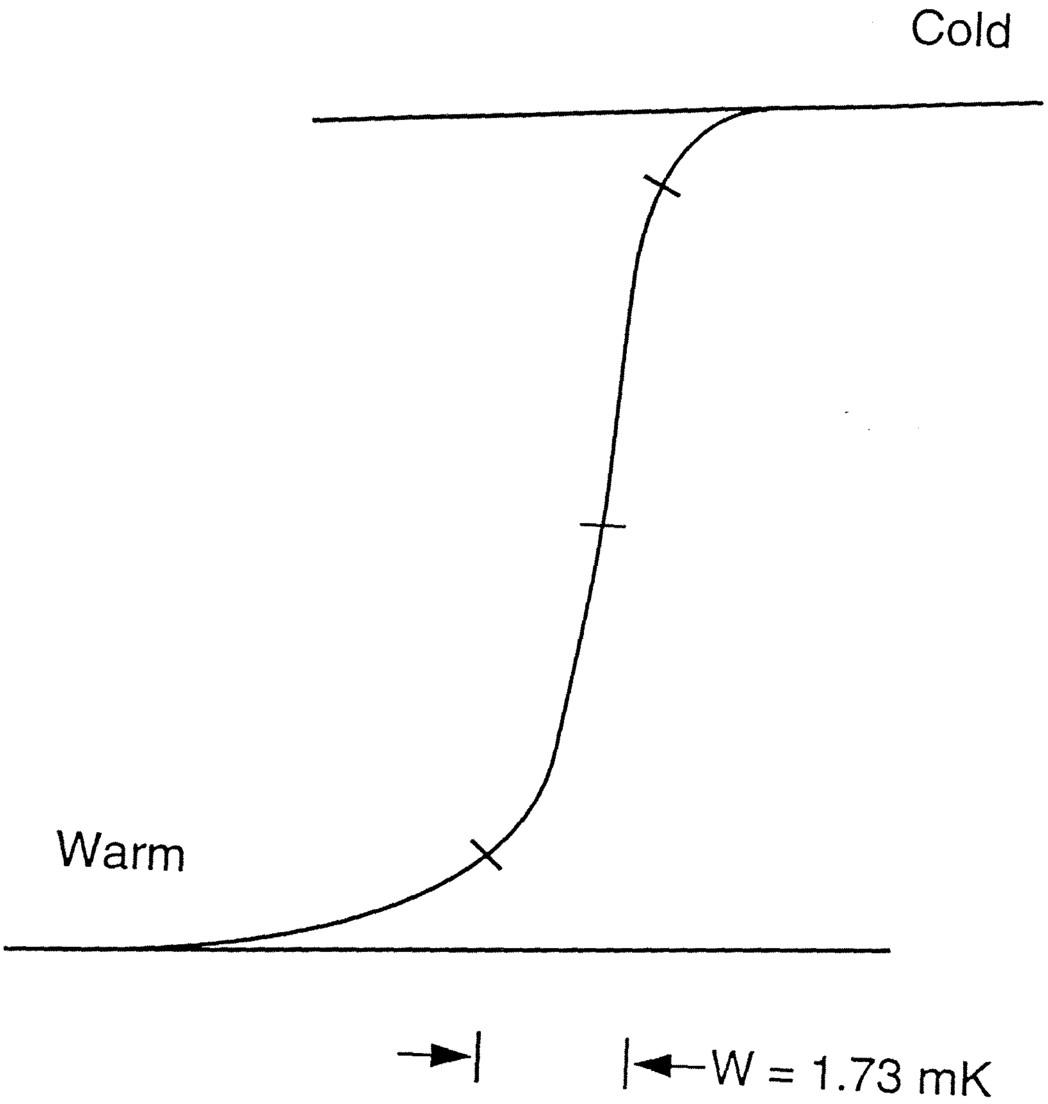
$R\,(T_c) = 4.784110(10)_\Omega \Rightarrow 1.18074\,\mathrm{K}$ (EPT-76)

SRM 767a
Zn Sample PP
$H \approx 0$ GAUSS



$R\ (T_c) = 4.595992(10)_\Omega \Rightarrow 0.85024\text{K}$ (EPT-76)

SRM 767a
Cd Sample PP
$H \approx 0$ GAUSS



$R(T_c) = 4.406386(10)_{\Omega} \Rightarrow 0.51981\text{K}$ (EPT-76)