Standard Reference Materials:

Preparation and Use of Superconductive Fixed Point Devices, SRM 767

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PREPARATION AND USE OF SUPERCONDUCTIVE FIXED POINT DEVICES SRM 767

by

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The preparation, testing, and use of SRM 767 devices are described. These devices incorporate samples of lead, indium, aluminum, zinc, and cadmium within a mutual inductance coil pair. These elements become superconductive at temperatures near 7.2 K, 3.4 K, 1.2 K, 0.85 K and 0.5 K, respectively, and the transition midpoints, when attained by observing the sample magnetic susceptibilities in negligibly small magnetic fields, provide thermometric reference points which are reproducible to ± 1 mK.

Key words: Aluminum; cadmium; cryogenics; indium; lead; magnetic susceptibility; superconductive transition temperature; superconductivity; thermometric fixed points; zinc.

I. INTRODUCTION

In scientific and engineering work, temperature measurements below 20 K can be referred to a variety of thermometric fixed points and temperature scales. The International Practical Temperature Scale of 1968 [1] recognizes the triple point and two boiling points of equilibrium hydrogen as fixed points, and it defines temperatures from 13.81 K upwards in terms of the resistance of standard platinum

resistance thermometers. In addition, it recommends the 1958 ^{4}He and the 1962 ^{3}He vapor pressure scales for use between 0.2 K and 5.2 K. Furthermore, it appears likely that a temperature scale based on the velocity of sound ir pure gases [2] eventually will be recognized as a standarc from about 2 K to 30 K. Still other temperature scales, such as one based on the thermal noise in a resistance [3] and another involving the angular distribution of radiatic from oriented nuclei [4], appear to be quite promising at lower temperatures.

However, the realization of each of these fixed point and temperature scales is a challenging problem in the laboratory. With the exception of the ^{3}He and the ^{4}He vapor pressure scales, none has been used with any regulai ity in actual experimental cryostats. With the advent of the ^{3}He dilution refrigerator [5], moreover, the cooling l which determines the temperature of the experimental chamb is no longer a pure liquid, but rather is a variable mixtı of the two isotopes of helium, for which no ready vapor pressure -- temperature relation is at hand.

In order to provide reliable temperature information, various experimenters use semiconductor resistance, paramı netic susceptibility, thermocouple, nuclear resonance and other thermometers. These have the common feature that they must be calibrated so that the temperature-dependent observable can be related to an accepted temperature scale in many cases, this must be done for each experiment because the device is not reproducible after warming to roon temperature.

One means of providing a convenient *in situ* temperatı calibration involves the observation of the superconducti transitions in various metals. Since T_c values are often quite sensitive to impurities, it appeared resonable to examine the purest stocks available. Preliminary studies

indicated that superconductive transitions reproducible to ± 1 mK might be obtained for pure samples of lead ($T_c \sim 7.2$ K), indium ($T_c \sim 3.4$ K), aluminum ($T_c \sim 1.2$ K), zinc ($T_c \sim 0.8$ K), cadmium ($T_c \sim 0.5$ K), and iridium ($T_c \sim 0.1$ K). The superconductive transitions in higher - T_c alloys were quite broad, so that no further attempt has been made to use them in thermometric fixed point devices. A search for sources of pure, yet inexpensive supplies of the elements listed above indicated that only the first five were readily available. This report contains a discussion of the preparation and use of devices containing these elements.

II. SAMPLES

Bulk quantities of high-purity aluminum, zinc, and cadmium were available from the Office of Standard Reference Materials. Ingots of lead in indium were obtained commercially. The following table lists the assays available with these materials:

Table I. Assays of Bulk Starting Materials for Superconductive Thermometric Fixed Point Devices (ppm by weight)

ZINC	LEAD	CADMIUM	ALUMINUM	INDIUM
SRM 682	HPM 9284	SRM 746	HPM 5831	JK 762
Cl < .5	Cu .2	K < 4	Si 1.	Pb 3
O < .5	Cd .1	Na < 3	Cu .5	Tl 3
Si < .5	Fe .1	O < 2	Mg .5	Sn 1
Ca < .2	Si .1	Pb .8	Ca .2	Cd < 1
Na < .2	Tl .1	Ca < .6	Cr .2	Fe < 1
Ti < .2	Bi < .1	Cr .4	Ag .1	Cu nf
Cd .1	Ca < .1	C < .1		Ga nf

Table I (continued)

ZINC SRM 682	LEAD HPM 9284	CADMIUM SRM 746	INDIUM JK 762
Fe .1	Mg < .1	Mg .1	Ni n
K < .1	Ag < .1	Cl .2	Ag n
Mg < .1		Zn .1	
Ni < .1		As < .1	
		Rb < .1	

(Fe, Mn-not determined)

The preparation of fixed-point samples of Pb, In, Zn, and Cd was begun by casting the high-purity ingots, under high vacuum, into rods 1 cm in diameter and 25 cm long. These rods were cut into slugs 1.5 cm long and then etched with nitric acid to remove possible contamination. Subsequently, the slugs were handled with clean tweezers. Before the slugs were placed in pyrex molds for specimen casting, they were rinsed three times in distilled water and dried.

The casting of the slugs into rods 1.5 mm in diameter and 15 cm long was done under high vacuum while heating the pyrex molds to the melting point of the particular material being cast. In order to force the molten slug into the capillary, it was often necessary to isolate the vacuum pump from the mold and to introduce an atmosphere of helium. The helium was purified by passing it through a zeolite trap at 77 K.

With the exception of Zn, the glass molds were etched away from the rod with hydrofluoric acid. The Zn samples were not prepared this way because hydrofluoric acid attacks the metal; instead, they were extracted by breakin the glass mold with a wooden mallet. The specimens cut from the 1.5 mm rod were then filed, rounding the ends to reduce any effects on the measurement of the superconducti

transitions. The samples were etched in a solution of nitric acid and rinsed three times in distilled water. The Pb samples received an additional alcohol rinse, as this seemed to retard oxidation.

To avoid excessively broad (20-40 mK) transitions, it was necessary to anneal the Zn and Cd samples; however, it was not necessary for the Al, Pb, and In specimens.

The Zn specimens were annealed for 48 hours at 395-398 °C and the Cd samples were annealed for 48 hours at 308-311 °C. Both Zn and Cd samples were sealed in pyrex tubes with two-thirds of an atmosphere of purified helium to prevent sublimation.

The Al samples were made by tying six 3 cm long, 0.5 mm diameter wires of OSRM HPM 5831 Al into a bundle with nylon thread.

III. EXPERIMENTAL APPARATUS AND METHOD

A. Apparatus

The apparatus used in this work was a ^{3}He-^{4}He dilution refrigerator with a countercurrent capillary heat exchanger, in which a copper platform was provided for the simultaneous measurement of up to five germanium resistance thermometers and of as many as five sets of superconductive samples.

The single-heat-exchanger dilution refrigerator has been discussed in the literature, and its operating characteristics are reasonably reliable [5]. Design features of this cryostat which are particularly relevant for these experiments are shown in schematic form in Fig. 1.

In order to provide an environment with thermometric precision and reproducibility of one millikelvin at temperatures ranging from 0.5 to 20 K, it was necessary to employ a set of five germanium resistance thermometers monitored

by a set of the superconductive fixed point samples. The five resistance thermometers, five superconductive device and a resistive heater were mounted on the copper platfor shown in Fig. 1.

Individual samples were inserted into a copper stud, as shown in Fig. 2. The following procedure was develope for mounting to ensure thermal equilibrium and mechanical rigidity between the samples and the stud; stopcock greas was inserted into the mounting hole before the sample was placed therein, and an electrically conductive varnish-si powder mixture was brushed on afterward. It was found in early experiments that several samples could be included a single stud without noticeable interaction; in fact, hc for extra samples were drilled in the studs in the event that other materials might eventually prove useful for thermometric fixed points.

As indicated in Fig. 2, the transitions were observe by means of a mutual inductance coil pair. In these meas ments, the primary coils on each of the five devices were connected in series and the five secondary coils had one common lead; thus the measurement of twenty-five individu samples required but eight electrical leads.

The germanium thermometer resistances were obtained four-lead dc potentiometry, in which the voltage across a standard 1000 Ω resistor and the voltage across the one resistance thermometer electrically in series with it wer measured alternately. The voltage could be resolved to (μV and, for the measuring currents used, the resultant temperature resolution was $\leq$ 0.1 mK.

Superconductive fixed point devices were made part of a mutual inductance bridge circuit, which was monitore by a phase-sensitive detector. The circuit is shown in block form in Fig. 3, and is discussed in Appendix A. Th device coils were operated at 400 Hz with a magnetic fie

amplitude of a few hundred nT. The earth's magnetic field was reduced by external Helmholtz coils to less than 1 μT.

This method of observing the superconductive transitions was selected primarily because of its convenience. It has the added advantage, however, of avoiding the introduction of strains or impurities which might accompany the placing of electrical contacts on the sample for resistance measurements. Finally, the magnetic inductance method has been shown to produce values of T_c within one millikelvin of those obtained from resistivity and heat capacity measurements [7].

B. Experimental Procedure

Before attempting to stabilize the platform at a temperature point, the dilution chamber was first stabilized a few tens of millikelvins below that point. For temperatures below about 1 K, this procedure required operation of the dilution refrigerator at a reduced ^{3}He flow rate and the application of up to one milliwatt of heat to the dilution chamber. The thermal link to the dilution chamber then brought the platform to a temperature slightly below that desired with in a few minutes.

To reach the temperature of the appropriate superconductive fixed point, a manually variable direct current was then applied to a resistor mounted on the center of the platform while the progress of the superconductive transition was followed on the phase-sensitive mutual inductance bridge detector meter or on an X-Y recorder connected to it. Varying the heater current stabilized the platform at the temperature characterized by the midpoint of the superconductor's inductive transition. To avoid errors due to possible super-cooling effects, the transition midpoint was always approached from the low-temperature side, and the

earth's field compensation was often checked before proceeding with the resistance measurements [8].

One result of using the dilution chamber for rough temperature control was that less than 10 microwatts of power was generated in the platform resistor during most measurements; thus the possibility of thermal gradients due to large heat flow in the platform was reduced. A second advantage in using this method is that the electrical leads to the platform components pass through two nearly isotherma "anchors", considerably reducing heat flow to or from the components through their leads. For similar reasons, the temperatures of the pumped ^{4}He bath and of the ^{3}He evaporator were adjusted to consistent values each time measurements were made at a given platform temperature.

Once stability was obtained at a given platform temperature, the appropriate germanium resistors were measured with one or more current settings.

IV. ANALYSIS OF DATA AND TEMPERATURE MEASUREMENT

A. Data Analysis

Analysis of the individual superconductive transitions involved an evaluation of the width of the transition and of the temperature of its transition midpoint in relation to other samples of that kind. For these purposes, it was necessary to know dR/dT, the temperature dependence of the resistance of the appropriate germanium thermometers, at each of the superconductive transition temperatures.

In an early set of measurements, five devices were measured many times, while the effects of thermal cycling, resistor measuring current variations and mutual inductance bridge parameter variation were examined. The resistance values of the appropriate resistors were measured at the

midpoints of the superconductive transitions of each sample. From these measurements, an average resistance, R_{AVE}, of each resistor was obtained for each fixed point for which the resistor was a useful thermometer. The resulting values of R_{AVE} are listed in the last column in Table II. Resistors

Table II. Temperature Dependences of Germanium Thermometers (values in parentheses have changed during the experiments described in this report)

Thermometer Number	Temperature, K	$\frac{dR}{dT}$, $\frac{\Omega}{mK}$	R_{AVE}, Ω
1394	7.2 (lead)	0.08	(314.45)
1395	7.2	0.08	346.31
2412	7.2	0.08	(339.65)
1394	3.4 (indium)	0.8	1347.0
1395	3.4	0.8	1470.4
2412	3.4	0.8	(1580.3)
1394	1.2 (aluminum)	60.	20,015.
1395	1.2	70.	21,490.
2412	1.2	70.	(22,193.)
452	1.2	1.7	(1,104.0)
452	0.84 (zinc)	5.7	(2,129.3)
1403	0.84	0.25	123.21
452	0.5 (cadmium)	40.	(7,321.)
1403	0.5	0.4	206.65

1394 and 1395 were calibrated against the NBS 2-20 K acoustic temperature scale [2], readily yielding values of dR/dT at the lead and indium points. Resistor 452 was calibrated against T_{62} [9] from 0.95-2 K in a separate experiment, and the calibration was extended to 0.4 K by means of cerous magnesium nitrate paramagnetic salt thermometry [10]; these measurements yielded values of dR/dT for resistor 452 at the aluminum, zinc, and cadmium points. In order to obtain the remaining values of dR/dT, it was sufficient to apply small magnetic fields to the fixed-point samples, reducing the temperature of the superconductive transitions slightly and to measure the resistances of the appropriate resistors once again, obtaining the temperature shifts from values of $(dH_c/dT)_{T_c}$ in the literature.

Once the resistor temperature dependences shown in Table II were determined, it was possible to obtain values for deviations of individual sample T_c's from the average T_c for that element, and to evaluate the width of each transition as well. The former quantity, which was designated ΔT_c, was obtained from the relation

$$\Delta T_c = \Delta R[dR/dT]^{-1}$$

where ΔR was simply the difference between the resistance at the sample T_c and R_{AVE}. The width, W, was obtained from the X-Y recordings of the transitions by evaluating the resistances at which the superconductive-to-normal and the normal-to-superconductive transitions were 80-90% complete as shown in Fig. 3. Once again, if ΔR refers to the resistance difference thus obtained, a simple ratio

$$W = \Delta R[dR/dT]^{-1}$$

yields the desired quantity.

It should be clear from the method used to evaluate W that any hysteresis exhibited by the sample would be included in its measured width. However, such effects,

when present, rarely exceeded one millikelvin.

The analysis of the fixed-point devices has been complicated by changes occurring in the resistance thermometers; in fact, the existence of these changes has shown the usefulness of thermometric fixed points. In a typical case of this kind, the ΔT_c values of all the samples of one element would be relatively large and similar to each other, according to measurements made with one resistor. Because at least two resistors were in use at each fixed-point temperature, this problem was easily interpreted. Usually, the five sample ΔT_c values measured with all other resistors were less than about one-half millikelvin and the five values measured with the deviant resistor were clustered about a relatively large value; even though only two resistors might be in use at the fixed point, the fact that all five samples gave similar indications was convincing evidence that the anomalous results were due to a shift in the resistance-temperature relation of the corresponding resistor. For example, we have shown that resistor 1394 has shifted from time to time by as much as 2 mK at the lead point; the shift followed the warming of the apparatus to room temperature. (It may be worth noting, however, that the resistor itself had not been remounted, nor had its leads been knowingly disturbed, during these times). A larger shift has occurred once in resistor 452, referring all of the fixed-point data to new, evidently reproducible resistance values [11]. Resistor 2412 has suffered a drastic and apparently fatal change in its behavior, as its fixed-point values are no longer reproducible.

Recurring anomalies in the lead and aluminum ΔT_c values led to a modification in the procedure used to fasten the samples into the Cu stud. We found that ΔT_c values of perhaps one-fourth of over three dozen of these samples were greater than 1 mK, and poor thermal response often appeared

during the transition recording. Upon microscopic examination of the device, it was found that the solvent used the anchoring varnish apparently evaporated in such a way as to force most of the varnish out of the annular space between the sample and its mounting hole. When stopcock grease was substituted for the varnish, the problem disappeared, and no anomalous lead or aluminum ΔT_c has since been observed in over three dozen measurements on these samples. Inasmuch as the ΔT_c values observed in these experiments so far give a reasonable picture of the overall experimental uncertainty of the fixed-point measurements, summaries of these values are shown in Table III. The reduction in average ΔT_c values accompanying the change in mounting procedure can be seen readily. In fact, only two of eight dozen measurements made after the mounting procedure change have yielded ΔT_c values as high as 1 mK.

Table III. Summaries of ΔT_c Values, by Element, in mK, Before and After Adopting a Grease-Plus-Conductive-Varnish Mounting Technique

A. Varnish Only

Average	Pb(22)*	In(17)	Al(18)	Zn(17)	Cd(18
ΔT_c	.63	.28	.77	.27	.42
ΔT_c	+.56	-.14	-.28	0	+.35

B. Grease and Conductive Varnish

ΔT_c	.32(24)	.15(17)	.28(15)	.22(20)	.30
ΔT_c	+.24	-.06	-.09	+.21	+.27

*Numbers in parentheses are the numbers of specimens measured.

B. Temperature Measurement

Values of the various transition temperatures were derived from paramagnetic salt thermometry in conjunction with the T_{62} ^{3}He vapor pressure-temperature scale [9] or with the NBS 2-20 K acoustical temperature scale [2]. In the former case, the 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 which had been calibrated on the NBS 2-20 K scale. These derivations are indicated in Table IV.

A summary of the transition temperatures assigned to the superconductive fixed-point device samples by this procedure is given in the second column of Table IV. The uncertainties in assigning the appropriate temperature scale values to the respective fixed-point resistances are shown in the fourth column, and the experimental average magnitude of ΔT_c is listed in the last column.

Table IV. Assigned Transition Temperatures of Superconductive Samples, Derivations of the Assigned Values, Uncertainties in the Assignments, and Experimental Reproducibilities as Fixed Points.

Element	Assigned T_c(K)	Derivation	Assignment Uncertainty (mK)	Experime Reproducib (mK)
Lead	7.201	(NBS 2-20 K) + salt thermometry	2.5	0.32
Indium	3.416_7	(NBS 2-20 K) + salt thermometry	1.5	0.15
Aluminum	1.174_6	Direct Calib. - ^{3}He v.p. T_{62}	2.	0.28
Zinc	0.844	T_{62} + salt thermo-metry	1.5	0.22
Cadmium	0.515	T_{62} + salt thermo-metry	2.5	0.30

It should be emphasized that the limits of error shown in Column 4 of Table IV indicate primarily the difficulty of assigning temperature scale values to the appropriate germanium resistances, while Column 5 reflects the experi al reproducibilities of the various samples as thermometr fixed points. No attempt has been made to estimate the deviation of the two temperature scales from the thermodynamic scale; there is evidence that both scales deviate to some extent from thermodynamic temperatures, but of course, these experiments cannot disclose such deviations

The transition temperature assignment uncertainties in Table IV can be expected to decrease as the correspond T_c values continue to be measured on the defining scales; the uncertainties not shown in Table IV but present nonetheless due to uncertainties in the relevant temperature scales can be reduced by inclusion of the devices in expe

ments of the kind mentioned in the introduction to this report. It is the fond hope of the present authors that such experiments will soon take place.

V. USE OF SRM 767

SRM 767 as released by NBS Office of Standard Reference Materials consists of a set of five samples (Pb, In, Al, Zn, and Cd) varnished into a copper stud, as shown in Fig. 2, and enclosed by a bakelite cover and a mutual inductance coil set. Overall, the device is about 1.5 cm diameter by 4 cm long. An identifying serial number is located at the top of the protective cover.

The user should be particularly aware of three considerations in placing SRM 767 in service. First, the copper stud should be brought to thermal equilibrium with the experiment or with the thermometer which is to be calibrated. This can be accomplished by connecting both to a solid copper block. The SRM 767 copper mounting stud terminates in a 6-32 thread about 5 mm long, and a matching tapped hole should be provided in the sample block. A light coating of stopcock grease placed on the threads of the mounting stud and a light tightening pressure (simply with the fingers) will form an adequate thermal connection between the mounting stud and the sample block. In addition, the four electrical leads to the measuring coils should be varnished or greased to the sample block over a length of perhaps 5 cm to avoid heat influx to the device via that avenue.

A second consideration in using the device is control of the ambient magnetic field. In order to avoid depressing the transition temperatures of the superconductive samples and probably introducing hysteresis due to supercooling, the user should reduce the ambient magnetic field to 1 μT or

less. This can be accomplished by using three mutually orthogonal Helmholtz pairs outside of the apparatus, provid ing that no extensive ferromagnetic or superconductive layer intervenes. (An example of an interfering superconductive layer would be a vacuum jacket tinned with 50/50 lead-tin solder and enclosing the sample area. Such a cylinder will become superconductive slightly above 7 K, and will trap the magnetic flux inside it at the time of its transition. Subsequent adjustment of external earth's field compensation coils would not alter the trapped internal magnetic field.) Another method of removing the earth' field involves the use of ferromagnetic shielding using mumetal or its equivalent. Carefully made and carefully used such shielding can reduce the ambient magnetic field to values well below 1 μT, but it is well to use it only in a situation where the remanent field can be measured. If the ambient field must be tolerated in the experimental chamber its value must be measured, and the various fixed-point T_c values must be reduced by the appropriate $(dH_c/dT)_{Tc}$ values In addition to correcting the T_c, the user should note that supercooling of the fixed-point samples is likely to occur in the earth's magnetic field. If it does, then only the superconductive-to-normal transition midpoint will correspo to T_c, and therefore (as will be noted below) T_c must be reached in warming.

The third consideration relevant to the use of SRM 767 is that an adequate mutual inductance measuring circuit should be used. The circuit used in the present measurements is shown in block form in Fig. 3 and in more detail in Appendix A. It is not necessary to use a very sophisticated measuring circuit, but care should be taken to restrict the measuring field to 1 μT, (which implies a primary current of less than 20 μA) and to obtain a stable circuit output. 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 indeterminate length of time. It should be noted, however, that drift in the measuring circuit output can mislead the operator; indeed, a drift of one-half the transition width in the monitoring circuit can result in failure to maintain T_c at all.

In summary, SRM 767 is a four-lead mutual inductance device which can provide millikelvin reproducibility at each of five cryogenic temperatures, if proper care is taken to ensure good thermal contact between the device and an experiment or a thermometer, to cancel or to measure the earth's magnetic field, and to provide a suitable current amplitude and output stability in the measuring circuit.

VI. APPENDIX A
THE TRANSITION MEASURING CIRCUITS

The mutual inductance and thermometer resistance circuits are shown in Figs. 4 and 5, respectively, in more detail than in Fig. 3.

The mutual inductance measurement was based on the well-known Hartshorn's bridge circuit. The phase-sensitive detector supplied a 400 Hz signal to the bridge circuit through an isolation transformer, shown as A in Fig. 4. The standard inductor B was chosen so that the sample coil mutual inductance (about two millihenries) was within the useful range of the ratio arm transformer inductance, C. Switches D and E provided for reversal of the inductive and resistive components of the sample secondary coil voltage. Transformer F isolated the secondary circuit form the detector ground. Typically, the sample coil primary current was a few microamperes. The mutual inductance change which occurred when a particular sample became superconductive was about ten microhenries; in this circuit, the resulting detector signal was perhaps five microvolts.

It is quite feasible to observe the superconductive transitions with simpler mutual inductance equipment, and work is in progress to develop an economical but stable circuit.

The resistance measuring circuit used in these experiments is shown in some detail in Fig. 5. It is a reasonably simple dc potentiometric circuit of the "substitution" type in which the voltage across the sample resistor is compared with the voltage across a standard resistor. In this measurement scheme, slow drift of the potentiometer standard cell does not effect the accuracy of the measurement. Other refinements adopted here are the temperature stabilizing of the potentiometer working cell and of the resistor current

supply cell, the provision of continuous current drain through a 1-megohm resistor to electrically stabilize the latter cell, and a high-quality four-deck switch (S4) which simultaneously reverses the potentiometer current and the resistor current.

In use, the circuit involved the selection of a particular germanium thermometer and current with switches S3 and S1, respectively, and the sequential measurement of the standard resistor and germanium thermometer voltages. The use of reversing switch S4 during a voltage measurement enabled the operator to ignore thermal emfs generated in the voltage leads; since these would not change sign on reversal of the current, they simply resulted in a zero offset of the detector meter. Thermal emf's of one to three microvolts were commonly seen in measuring the germanium thermometer voltages. The ratio of thermometer voltage of standard resistor voltage provided the thermometer resistance in terms of the standard resistance. The standard resistor voltage drifted during a day's measurements by one or two parts in ten thousand, and it was assumed that this change represented drift in the resistor current.

Different current values were used for measuring the different germanium thermometers. These currents ranged from 0.1 μA to 10 μA, and in general we used the highest current setting consistent with the avoidance of noticeable heating of the germanium thermometer. This level was easily determined by measuring the thermometer resistance with several current values.

It is quite possible that the use of an ac resistance measurement technique would have yielded results as accurate as (or, perish the thought, more accurate than!) the dc technique described here; certainly the ac measurements are faster and more sensitive. However, the relative simplicity of the dc technique and its freedom from unknown sources of

error gave the present authors an extremely comforting sense of well-being.

VII. REFERENCES

[1] International Committee on Weights and Measures, Metr logia 5, 35 (1969).

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

[3] R. A. Kamper, paper M1 in Symposium on the Physics of Superconductive Devices Univ. of Virginia, 28-29 Apri 1967. See also R. A. Kamper and J. E. Zimmerman, J. Appl. Phys. 42, 132 (1971).

[4] P. M. Berglund, H. K. Collan, G. J. Enholm, R. G. Gyl and O. V. Lounasmaa, J. Low Temp. Phys. 6, 357 (1972)

[5] See, for example, A. C. Anderson, RSI 41, 1446 (1970)

[6] J. F. Schooley and R. J. Soulen, Jr., paper S-12, 5th Symposium on Temperature, 21-24 June 1971, Washington D. C.

[7] R. J. Soulen, Jr. and J. H. Colwell, J. Low Tem. Phys 5, 325 (1971).

[8] R. E. Fassnacht and J. R. Dillinger pointed out in Ph Rev. 164, 565 (1967) that one can establish a minimum field at the sample by varying the Helmholtz coil current until a maximum T_c is reached.

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

[10] J. M. Daniels and F. N. H. Robinson, Phil. Mag. 44, 630, (1953).

[11] It should be noted that a small shift in the fixed-point resistance did not render a resistor useless in these experiments. This is true, once again, because a group of fixed-point samples is present; in case a cluster of ΔT_c values larger than, say, 0.4 mK occurs with only one resistor, a new value of R_{AVE} can be adopted for that experiment such that the new ΔT_c values cluster near the average ΔT_c determined from the remaining thermometers. Then any single sample which shows an anomalous value of ΔT_c on the remaining resistors should show one of similar magnitude and sign on the corrected resistor.

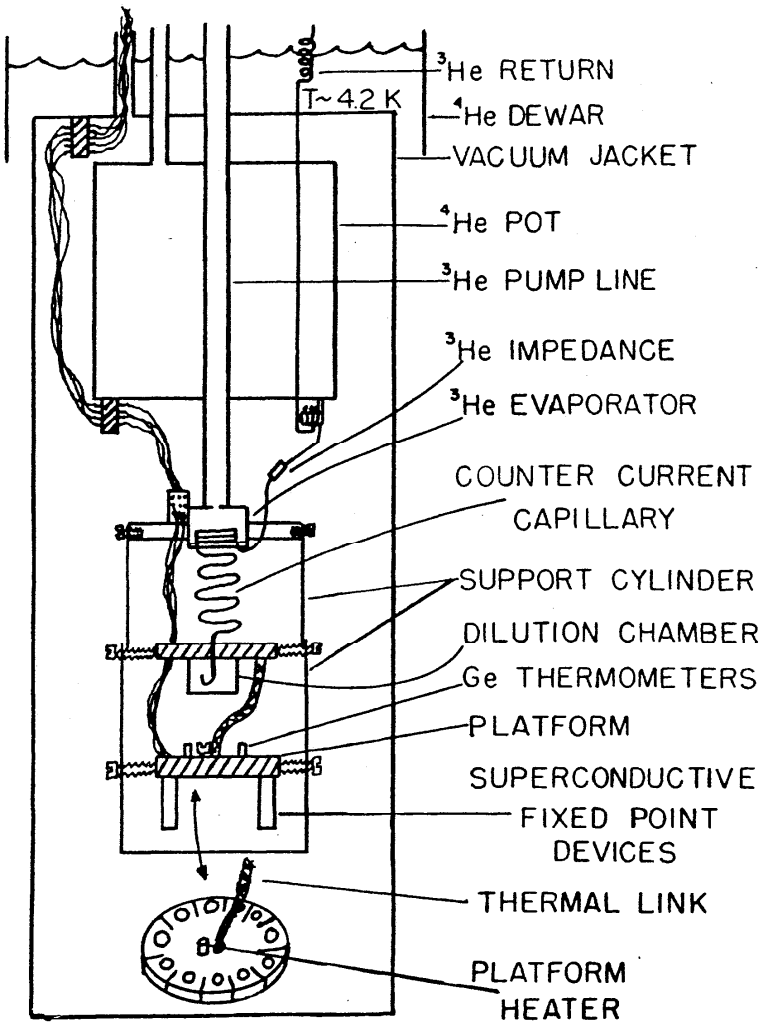

Figure 1. The experimental cryostat. The ^{3}He return flow is equilibrated at 4.2 K, at 1 K, and at the sintered Cu flow impedance at 0.6 - 0.8 K. The impedance permits a flow rate of about 0.2 cc of ^{4}He gas per minute with a 1-atm pressure differential. The heat exchanger is composed of one meter of concentric .010" ID - .02" OD and .040" ID - .050" OD stainless steel tubes. The ^{3}He evaporator and the dilution chamber volumes are each about 5 cc, permitting a wide range of operating conditions without displacing the phase boundary from the dilution chamber. The support cylinders are two layers of .005" mylar. The platform is a 6 cm dia., 1 cm thick block of OFHC copper, and it is thermally connected to the dilution chamber by 100 #38 copper wires. The .004" electrical lead wires are thermally anchored to the platform and to copper blocks on the dilution chamber, on the evaporator, on the ^{4}He pot, and on the vacuum jacket.

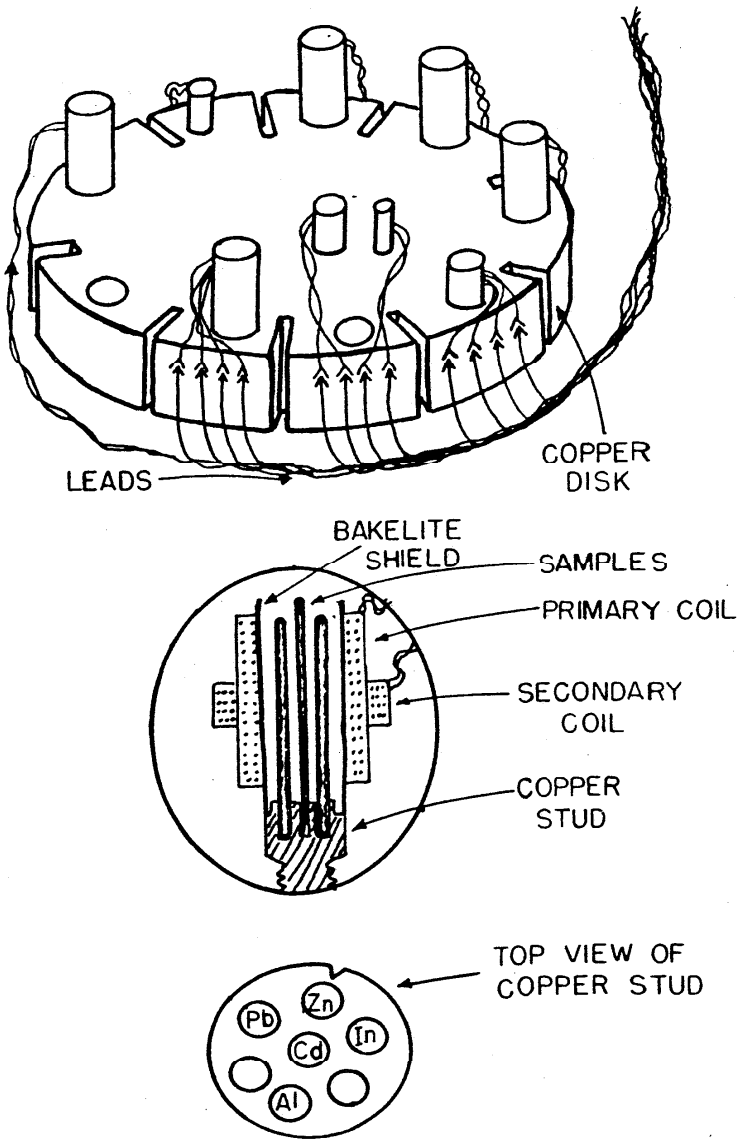

Figure 2. Schematic of the copper disk and sample mounting assembly. The mutual inductance coils are wound on bakelite formers. The primary coil contains 400 turns of #38 AWG copper wire and is 2.5 cm long, while the 1 cm long secondary coil contains 2000 turns of #40 AWG copper wire. The insert at the bottom shows the location of the individual samples relative to the indicator notch in the copper stud.

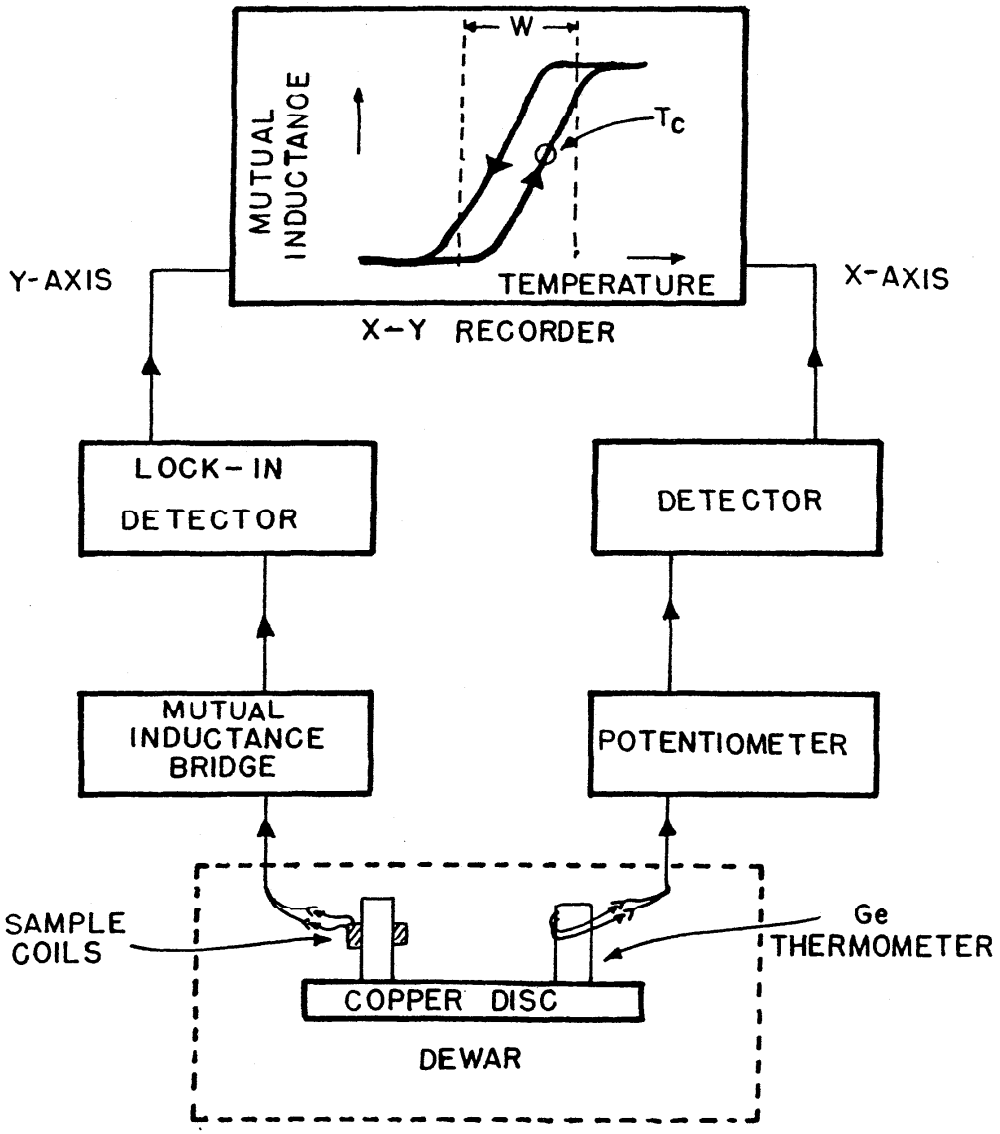

Figure 3. Block diagram of the transition measurement scheme, illustrating the definitions of the transition width, W. and of the transition temperature, T_c.

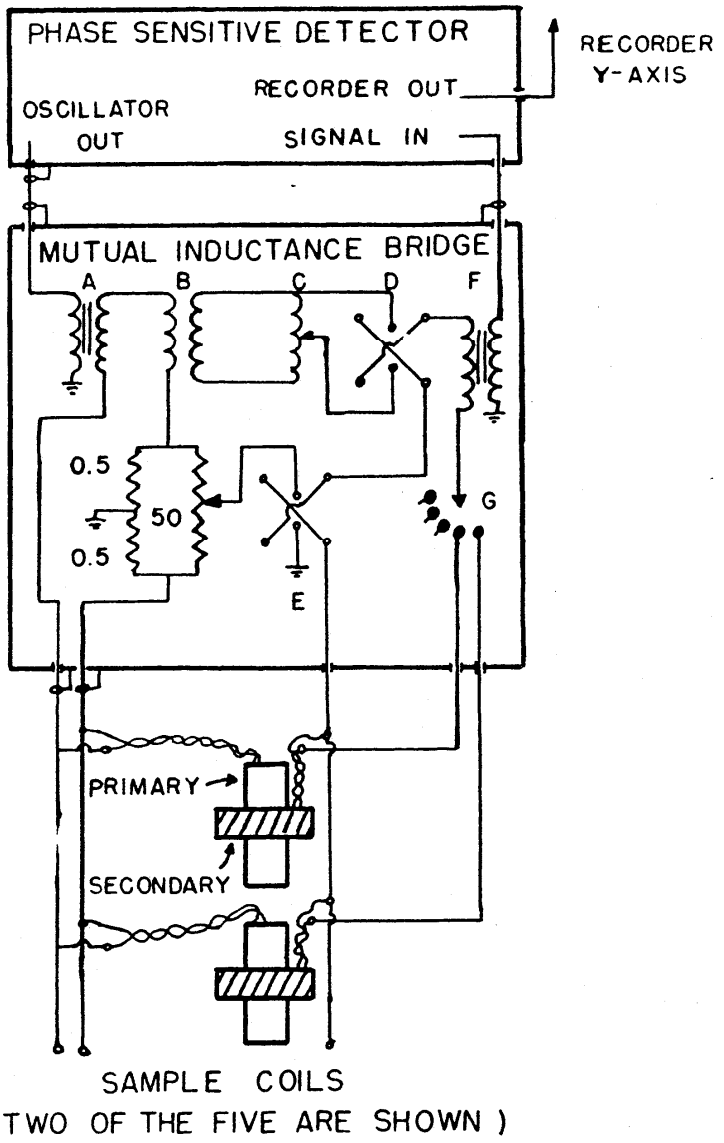

Figure 4. Schematic drawing of the mutual inductance circ
A and F are isolation transformers, B is a 50 mH standard
inductor, C is a ratio arm transformer, D reverses χ', the
inductive voltage component, and E reverses χ'', the resis-
tive voltage component. Switch G selects the appropriate
secondary coil.

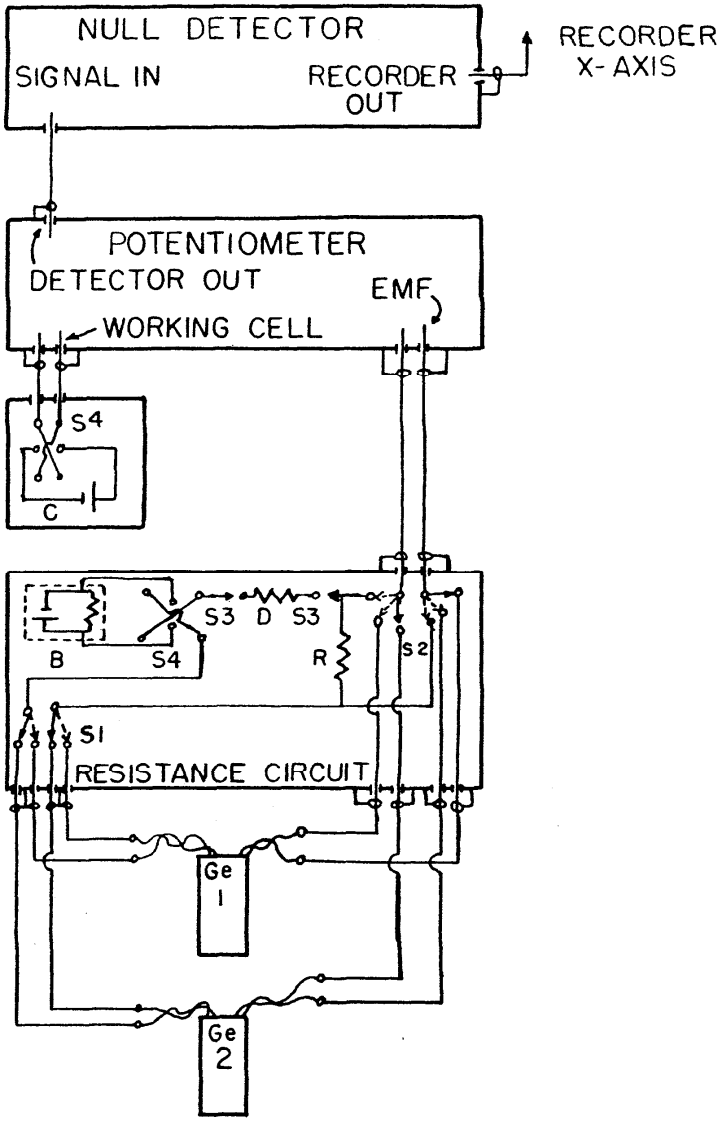

Figure 5. Schematic drawing of the resistance measurement circuit. B is an oil-bath-stabilized mercury cell with a continuous current draining resistor of 1 megohm, C is the potentiometer constant current supply, D is a set of resistors which allow various thermometer currents to be used, R is a 1000 Ω standard resistor, S1 switches the thermometer current from one germanium resistor to another, S2 switches the potentiometer emf terminals from one resistor to another, S3 is the current range switch, and S4 simultaneously reverses the potentiometer supply current and the resistor current.

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The preparation, testing, and use of SRM 767 devices are described. These devices incorporate samples of lead, indium, aluminum, zinc, and cadmium within a mutual inductance coil pair. These elements become superconductive at temperatures near 7.2 K, 3.4 K, 1.2 K, 0.85 K and 0.5 K, respectively, and the transition midpoints, when attained by observing the sample magnetic susceptibilities in negligible small magnetic fields, provide thermometric reference points which are reproducible to ± 1 mK.

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