

Reproducibility of Germanium Resistance Thermometers at 4.2 °K

M. H. Edlow¹ and H. H. Plumb

Institute for Basic Standards, National Bureau of Standards, Washington, D.C., 20234

(June 13, 1966)

NBS has needed a set of very reproducible germanium resistors that would be capable of maintaining temperature scales. This paper describes our procedure for selecting such a set.

A group of germanium resistors from three commercial sources have been thermally cycled between 4.2 °K and room temperature. The resistance-temperature calibrations at 4.2 °K were made with reference to a liquid helium bath so that reproducible temperatures could be determined from liquid helium vapor pressure measurements. Seven resistors out of 25 demonstrated reproducibilities (of the 4.2 °K calibration) of about 0.001 °K after undergoing the multiple cyclings. Guided by these results, we procured twelve, new, similar resistors which were cycled in a comparable procedure. After 85 cycles, in which 14 resistance-temperature calibrations were performed at 4.2 °K for each resistor, 10 of the 12 resistors demonstrated reproducibilities of approximately 0.001 °K.

The resulting set of secondary thermometers have undergone calibrations in the temperature ranges, 2 to 5 °K and 2 to 20 °K; reports of this work will be published in the near future.

Key Words: Germanium resistors, germanium resistor reproducibility, low temperature thermometry, reproducibility of germanium resistors, thermal cycling at low temperature, thermometry.

1. Introduction

A major problem in low temperature work has been the lack of a highly reproducible, precisely calibrated secondary thermometer in the region of 1 to 20 °K. Carbon composition resistors found wide popularity after Clement et al. [1],² had reported their reproducibilities and calibrations in the temperature regions of liquid helium and liquid hydrogen. However, carbon composition resistors show limited reproducibilities after cycling from room temperatures to 4.2 °K [2,3], and exhibit an aging effect when maintained in a highly stabilized liquid helium bath for several weeks [4].

Encouraging results for resistance thermometry in the liquid helium and liquid hydrogen temperature ranges were reported by Estermann [5] for doped germanium and silicon; by Friedberg [6] for polycrystalline *p*-type germanium; and by the Bell Telephone Laboratories group [7, 8] using arsenic-doped germanium that was encapsulated in platinum cans. A very limited number of the B.T.L. specimens were distributed by the Calorimetry Conference to several laboratories for evaluation. Results from the several laboratories [9] indicated, in general, reproducibilities of 0.001 °K. Since that time, the properties of impurity doped germanium resistors have been reported

by several commercial sources [10,11,12]. This paper is concerned with the determination of calibration reproducibilities of commercial specimens³ at or near the normal boiling point of liquid helium. The objective of the work was to obtain a group of 10 or 12 similar resistors that exhibited reproducibilities of 1 mdeg or better. This group of resistors will serve as secondary thermometer standards for maintaining temperature scales below 20 °K. Subsequent papers will cover their precise resistance-temperature relationships in the region 2 to 5 °K and 2 to 20 °K.

2. Experimental Apparatus

Our determinations of resistance are based upon d-c potentiometric measurements and the attendant techniques—four leads to a resistor are required for the measurement. A dual-six-dial potentiometer is used in conjunction with a special reversing switch that affords a reversal of current in all circuits—internal potentiometer and external resistors (fig. 1)—to compensate for undesired thermal e.m.f.'s. The null detector, a millimicrovoltmeter, has a useful range of source resistance from 1 to 100,000 Ω with the capability of detecting changes as small as one part in sixty thousand for measured resistances of 3,000 Ω. The germanium resistors are connected in series with 1,000 and 10,000 Ω standard resistors [13] to the exter-

¹ M. H. Edlow is presently associated with the United States Patent Office.

² Figures in brackets indicate the literature references at the end of this paper.

³ At the time that the resistors were procured these were, to the best of our knowledge, the only products of American manufacture available.

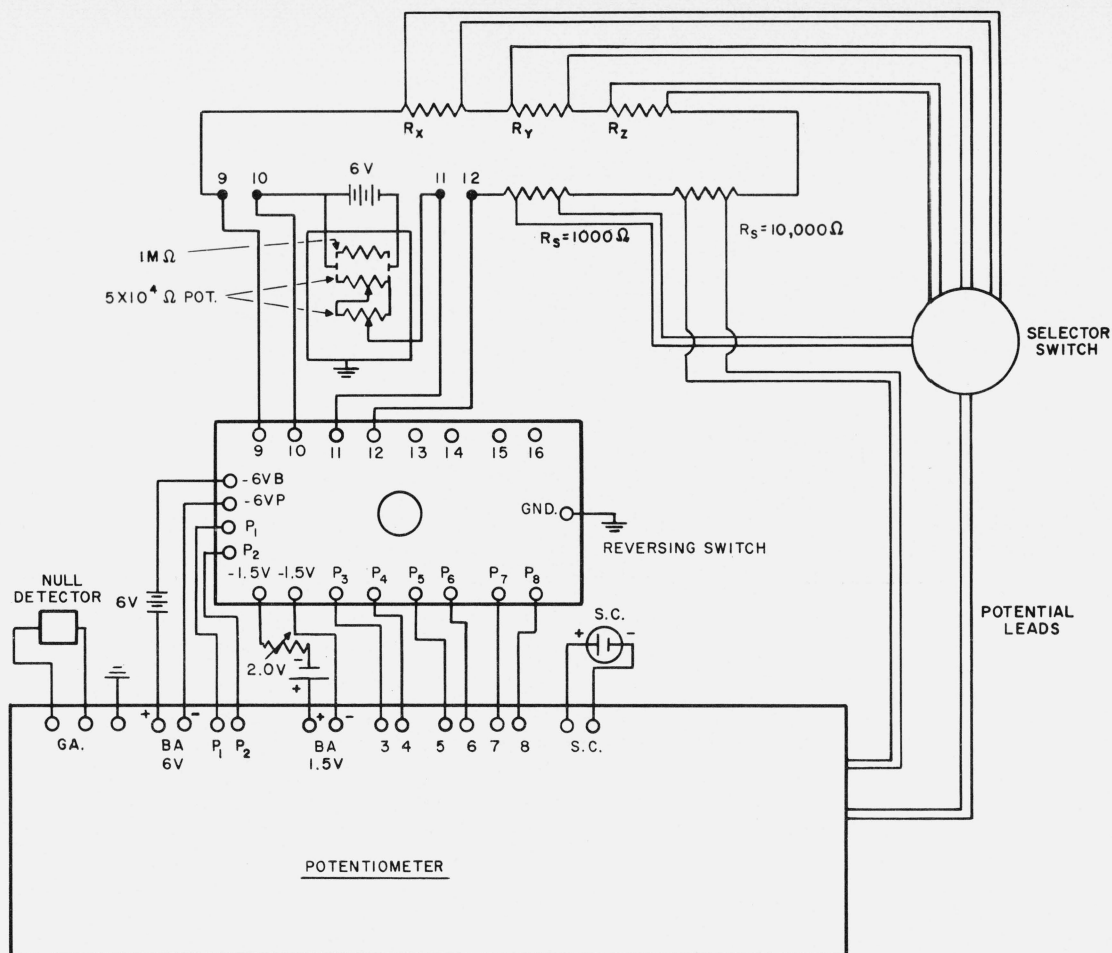


FIGURE 1. *D-c electrical circuit for measuring germanium resistors.*

R_s are standard resistors; R_x , R_y , and R_z , the resistors to be calibrated.

nal reversing switch; currents through this circuit are determined from potentials measured across the standard resistors. Measuring currents of the order 1 to $2 \mu\text{A}$, are obtained by using two $50,000 \Omega$ voltage dividers connected in parallel across the battery. A double-throw, double-pole toggle switch connects the current supply to either the germanium resistor circuit or a $1 \text{ M}\Omega$ "bleeder" resistor; the bleeder resistor allows the battery to deliver a small, continuous current when not in use with the germanium resistors. A two-volt, low discharge battery (series connected to a resistance decade box) supplies current to the middle internal potentiometer circuit while six-volt, low discharge batteries supply current for both the external series circuit containing the germanium resistors and the upper internal potentiometer circuit. The batteries are surrounded by thermal insulation to minimize temperature changes of the electrolyte that would produce variations in the discharge voltage. Current standardizations need only be performed a few times per day, although more frequent standardization checks were routinely performed.

The potentiometer, external reversing switch, batteries, null detector and standard resistors are supported on acrylic plates, which in turn rest on copper sheets that are interconnected by copper wires. A lead from one of the sheets is connected to a surrounding screen cage which is at earth potential. The double-wall, copper-screen cage completely encloses the d-c measuring apparatus and eliminates a-c joule heating that is caused by signals which emanate from nearby commercial transmitters [14].

The germanium resistors are immersed in a constant-temperature liquid helium bath contained in a 25-liter metal storage Dewar [15]. A vacuum tight Dewar cap has been designed to fit over the Dewar neck J (fig. 2) and permits routine thermal cycling of the resistors. A, B, C, and D are "quick couplings" and are hard soldered to the top E; E and a threaded brass cylinder G (internal component of quick coupling H) are soft soldered into the copper body F. The vapor from the helium bath passes through a side arm M that is connected to a 2.8 cm vapor-pressure pump-line.

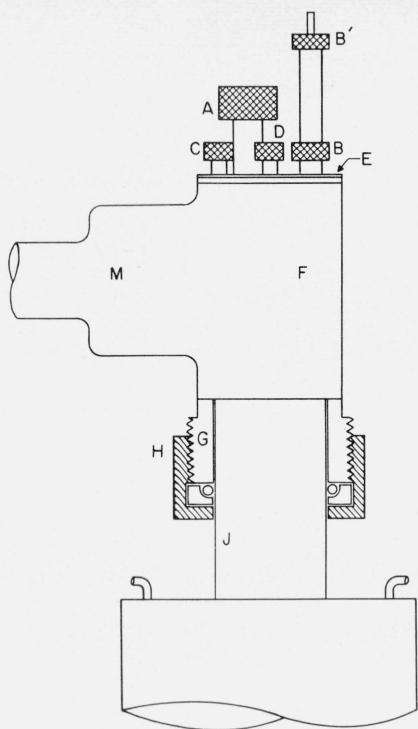


FIGURE 2. Dewar cap.

A stainless steel tube that passes through coupling C leads to a jacketed vapor pressure thermometer [16] that is positioned 0.3 cm from the bottom of the Dewar. A surface vapor pressure tube passes through coupling D and extends from about 10 cm above the coupling to about 8 cm below the neck of the Dewar. Both vapor pressure tubes consist of stainless steel, thin-wall (0.015 cm) tubing of 0.65 cm diam. A rubber line, when necessary, connects an external helium cylinder to the surface tube and permits helium gas to be bled into the Dewar. The gas flows out of coupling A during resistor thermal cycling and prevents frozen air from accumulating on the inside surface of the Dewar neck. The coupling B contains a 0.25 cm quick coupling B' that is closed by a metal rod; it will contain a He^3 vapor pressure thermometer in future work.

The component of the cap that is displaced during thermal cycling of the resistors is shown in figure 3. A hollow copper cylinder R, 1.9 cm o.d., slides through the coupling A. A multi-tube Kovar-glass seal at S is set in a small recess machined at the end of the cylinder R. Six pairs of #38 A.W.G. Formvar-coated copper wire, T, are strung through the capillary tubes; and the region of the seal, tubes and leads is covered with wax to achieve a vacuum seal. The copper leads are permanently maintained in a compact bundle within the Dewar by applying a cement to the taut wires. The resistors (positioned at the end of the leads) touch the Dewar bottom. Spun

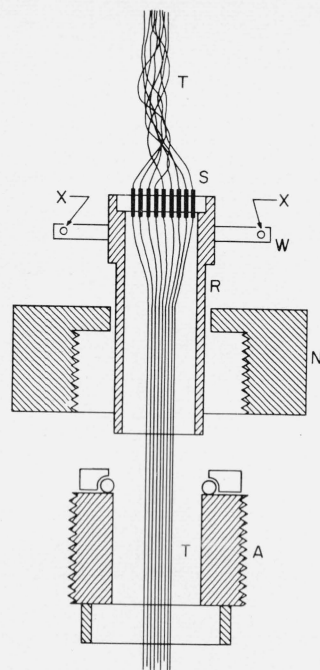


FIGURE 3. Dewar cap component that is moved during thermal cycling procedure.

glass spaghetti insulation covers the connections between resistor leads and permanent connecting wires. Thread is tied around the spaghetti-covered leads to achieve a compact bundle which easily passes through the 1.9 cm opening of A during thermal cycling. Copper wire strung through holes X of the arms W and wound tightly around the Dewar handles secures the cylinder R to the cap. The cap is secured to the Dewar handles in a similar manner. These precautions are necessary in the work from 4.2 to 5 °K where the helium vapor pressure is greater than atmospheric, and exerts sufficient force to dislodge the Dewar cap. A complete picture of the storage Dewar and cap is shown in figure 4.

Groups of three resistors are solder-connected to the lower extremities of the wires. (Experience with impurity-doped germanium resistors has shown that it is necessary to minimize the heat conducted to the specimen while soldering resistor leads to connecting wires. We have found that a reproducible calibration at 4.2 °K has been changed by several millidegrees when solder such as 50/50 lead-tin was used; consequently, Wood's metal is used to solder the resistor leads to the connecting wires. A pencil iron is used to supply the minimum heat required to melt the solder.) Each resistor has four leads—two for current and two for potential—that extend from within the Dewar, out through the seal at S (fig. 3) to ambient conditions. The current leads are series connected external to the Dewar so that in event of a

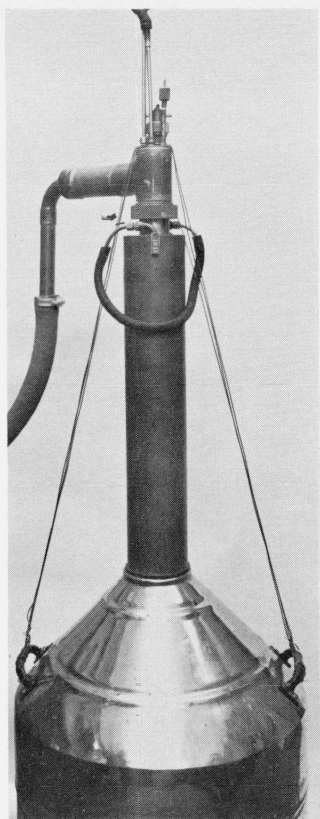


FIGURE 4. Complete picture of storage Dewar and cap.

resistor failure the faulty resistor can be quickly disconnected from the current circuit and measurements continued on the other two resistors. The potential leads emerging from the Kovar-glass seal and the 1,000 Ω standard resistor potential leads are appropriately soldered to terminals of a rotary selector switch. The rotary switch is mounted on the lid of a glass jar so that the switch is completely surrounded by a glass wall to provide a degree of thermal insulation from possible spurious temperature fluctuations. The common terminals of the switch are connected to one pair of emf terminals of the potentiometer by #38 A.W.G. double-nylon covered, Formvar-coated copper wire. The potential leads from the 10,000 Ω standard resistor are directly connected to the second pair of emf terminals on the potentiometer.

The manometer for measuring the helium vapor pressure in this work is a digital readout 152.4 cm mercury manometer [4]. Either the vapor pressure thermometer-bulb or the bath surface vapor pressure can be connected to the manometer by means of insulated 0.6 cm copper tubing (A, A', and G) and the vacuum bellows-type valves C and D (fig. 5). Valve F affords isolation of the manometer while the vacuum pumping line, through valve K, enables one

to evacuate any of the components that lead to the manifold E.

The gas filling system consists of a 1-liter storage can, L, and needle valve J through which helium gas can be metered into the system via rubber tubing H and vacuum valve I. Needle valve J' permits refilling the can from a nearby helium cylinder. The manifold of figure 5, the mercury manometer, manifold evacuating apparatus, and helium bath vacuum "pumping" source are all external to the screened room. Tubing ingress to the room is afforded by portholes that have been built into a wall of the screened room. Our experience has shown that effective shielding by the screened room is destroyed when an underground conductor is brought through the portholes; consequently, the Dewar pumping line has been appropriately grounded to the screened room. The space in porthole B (fig. 5), containing the two vapor pressure lines, is packed tightly with brass filament sponge and no evidence of R. F. signals is detected. The shielding of the screened room is tested at the beginning of every week using a battery operated F.M. receiver.

The well-known phenomenon of an oscillating helium gas column [18] was occasionally detected in the present setup. When this condition occurred in connection with the surface vapor-pressure tube, the liquid helium evaporation rate, as detected by a flow-meter, increased by a factor of 3 and oscillations of the manometer mercury meniscus were readily observed. A sheet of fine copper screening rolled into a tight bundle and inserted in the open end of the surface vapor-pressure tube for a length of 7 to 10 cm eliminated the troublesome oscillations in this instance.

3. Method of Cycling

The following procedure has been found satisfactory (damage to the resistor leads and freezing of the leads or resistor specimens to the Dewar neck have been avoided) for repetitive thermal cyclings of resistors from room temperature to 4.2 $^{\circ}\text{K}$. Rubber tubing connected a cylinder of helium gas to the surface vapor pressure tube and a slight overpressure of gas was accumulated in the Dewar. The nut N of coupling A (fig. 3) was loosened and the ensemble of cylinder, coupling nut N and portions of the connecting wires were withdrawn from the Dewar cap; the slight gas overpressure caused a stream of helium gas to continually pass through the opening of coupling A and thereby minimized any flow of air into the Dewar. After the resistors were raised to the vicinity of the Dewar cap, several layers of tissue paper (or raw cotton) were wrapped around the leads and positioned in the opening of the coupling A. The helium cylinder was then closed off. The resistors remained for three minutes within the Dewar cap, which is at approximately room temperature, and then were brought out into the room for approx-

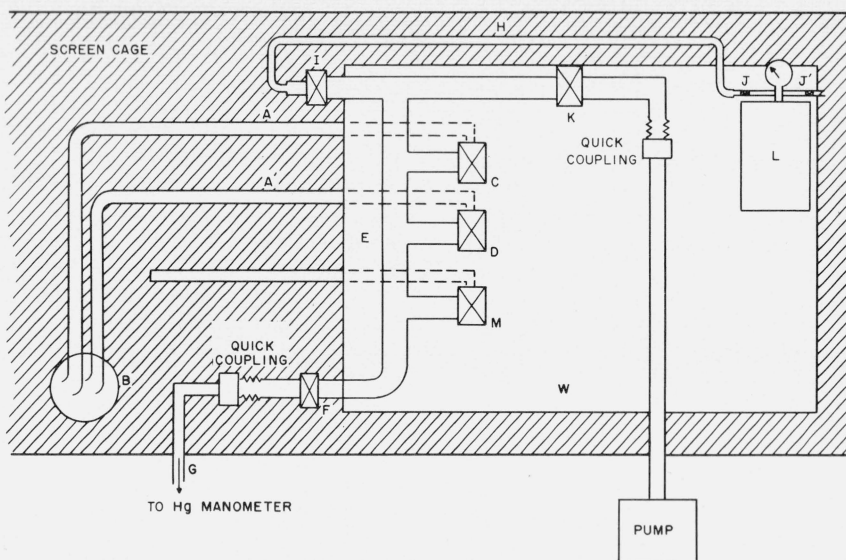


FIGURE 5. Vapor pressure manifold.

imately 1 min.⁴ (The resistors were not brought immediately out into the room in order to minimize the accumulation of condensed room moisture on the encapsulations. A sufficient quantity of moisture can lead to mechanical stressing of the resistor leads.)

Before the resistors were cycled back into the liquid bath, the helium cylinder valve was again opened to produce a steady stream of gas-flow through coupling A. As soon as the resistors approached the surface of the bath, the helium cylinder valve was closed since the then-increased evaporation rate of the bath was sufficient to maintain a slight overpressure. The entire process of lowering the resistors into the helium bath consumed only a minute and inflicted a thermal shock on the resistors.

The resistances of the specimens were measured soon after they were immersed in the liquid helium; at least 15 seconds were required to make measurements that would yield a value of resistance, which indicated an approximate condition of thermal equilibrium between the resistors and the surrounding liquid helium bath. However, the resistors were allowed to remain in the bath for about one minute prior to another cycle (see footnote 4).

3.1. Determination of a Calibration Temperature

When a well defined calibration point is desired, a meaningful thermal equilibrium must be achieved

within the liquid helium bath and between the bath and resistors. The vapor pressure of the bath is placed under control of the pressure regulator [17] and values of vapor pressure (from the vapor pressure thermometer) and resistance are monitored until a steady state condition results. This requires about 45 min and on many occasions we have ascertained that the thermal equilibrium attained in this period of time will not change more than a few tenths of a millidegree over periods of days or weeks.

In practice we do not experience difficulty in associating a temperature (near 4.2 °K) with a resistance value for a reproducible germanium resistor. The surface vapor pressure of the liquid helium bath is easily controlled, using our pressure regulator [17], within 0.05 mm of mercury—this corresponds to 0.00007 °K at 4.2 °K—so the reproduction of a bath surface vapor pressure does not constitute a problem. We have previously reported [16] the existence of a pressure gradient in the vicinity of the liquid helium surface for the type of bath we employ; in the bulk of the liquid helium the temperature is relatively constant. Since the surface gradient is dependent upon the Dewar evaporation rate, and perhaps the Dewar geometry also, it is essential that a helium vapor pressure bulb and the resistors be located within the liquid helium bulk. (From the vapor pressure determination and the “1958 He⁴ Scale of Temperature” [19], a temperature can be ascertained.) We have also found that the vapor pressure thermometer tube must be insulated so that it does not make thermal contact with the “colder” liquid helium surface through which the tube must pass. Vacuum-jacketing of the tube along the length that would make contact with the surface provides proper thermal insulation and avoids the effect of a “cold spot” [16, 20]. Radiant energy is prevented from reach-

⁴Our experience has always indicated that the germanium resistors, when warming to ambient conditions after a 4.2 °K exposure, require a lapse of only 2 or 3 min before attaining nominal ambient temperature. The reverse operation, cooling to a nominal 4.2 °K from ambient temperatures, is accomplished 10 or 15 sec after immersion of the resistor in liquid helium. Private communications from Mr. Herder (Cryocal) and Mr. Halverson (Radiation Research) reveal, for comparable cooling and heating treatment, very similar experiences.

ing the vapor pressure bulb by a radiation shield that is soldered within the tube and extends from 7.6 cm above the top of the jacket upward for 10.2 cm. When vapor pressure and temperature have been derived from a jacketed vapor pressure thermometer, and when an immersed germanium resistor has been stable, we have been able to associate resistance measurements with temperature to a constancy that is equivalent to 0.0003°K . However, if temperatures are referenced to surface vapor pressure measurements, the above constancy can decrease to an equivalent of 0.001°K . The decrease, in part, can be attributed to variations in the liquid helium evaporation rate and the helium level within the Dewar.

Within the bulk liquid helium a steady-state temperature can be routinely reproduced within 0.001°K . For most of the resistors being reported, the well-defined calibrations have been conducted at temperatures that generally were reproduced within 0.0003°K (with reference to jacketed vapor pressure thermometer measurements). A few of the resistors were calibrated at reference bath-temperatures which differed by as much as 0.001°K . We obtained dR/dT at approximately 4.2°K for the individual resistors by performing an additional calibration of resistance versus temperature (0.050°K or 0.100°K removed from the reference temperature for calibration after thermal cycling); this enabled us, through interpolation, to correct all of the nominal 4.2°K calibrations of a resistor to a particular temperature.

4. Description of the Resistors

The resistors were obtained from three manufacturers who have periodically supplied us with specimens for experimentation [21]. Some of the prototypes have been described [10, 11, 12] but for purposes of adequate comparison we shall briefly describe each. Resistors from one source (Minneapolis-Honeywell) are encapsulated in a platinum can that contains hermetically sealed helium gas. Four thin platinum wires from the resistor are brought through a glass header of the can and soldered to multistrand, plastic covered, copper leads. Mechanical support for the leads is achieved by "potting" an epoxy resin around the soldered platinum wire-copper lead connections. Resistors from a second source (Radiation Research) are also encapsulated in a platinum can which contains hermetically sealed helium gas. These resistors have four 0.025 cm platinum leads that are brought through a glass header for electrical measurements. Resistors from a third source (Texas Instruments) are in a soft, opaque glass envelope and have two 0.025 cm platinum leads for electrical measurements. (Envelopes or encapsulations may be perforated depending upon the user's requirements.) Later specimens have been encapsulated in a metal can with a transistor header. Various manufacturers have used arsenic and gallium as the major doping impurities; both the re-

sistivity and its temperature coefficient are effected by control of the impurity concentrations.

4.1. Thermal Properties

Joule heating, developed when current passes through a resistor, raises the temperature of the specimen to a value higher than that of its surroundings. Since the usefulness of the germanium resistance thermometer depends upon its representation of the correct temperature of the surrounding medium, this effect must be minimized. The results for two typical resistors follow. The resistance variation with current squared for a reproducible, hermetically sealed Minneapolis-Honeywell resistor in a stabilized liquid helium bath at 4.2°K is shown in figure 6. The abscissa is the square of the current which is approximately proportional to the power being dissipated; at the left, the ordinate is the fractional change of resistance caused by joule heating, while at the right it is the corresponding increase in temperature, ΔT , obtained from the temperature coefficient of resistance. The increase in temperature, ΔT , varies linearly with I^2 up to the value $I^2 \cong 1.3 \times 10^{-9}$ amperes² which is in reasonable agreement with Blakemore's result [11]. Tested with the above resistor was a Minneapolis-Honeywell resistor contained in a perforated encapsulation; its fractional change of resistance as well as the corresponding increase in temperature are also plotted as a function of I^2 in figure 6. Comparison of the two plots shows relatively little or no joule heating for the perforated encapsulation, indicating that the helium gas (in hermetically sealed specimens) is a relatively inefficient heat exchanger. A similar plot of $\Delta R/R$, ΔT versus I^2 for a Texas Instruments encapsulated specimen is shown in figure 7. The result indicates ΔT varying linearly through most of the I^2 range but exhibiting a noticeable deviation from linearity at $I^2 \cong 1.6 \times 10^{-9}$.

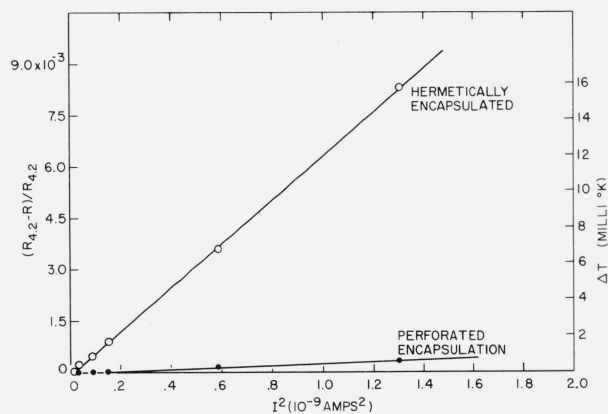


FIGURE 6. Joule heating for two Minneapolis-Honeywell resistors. $(R_{4.2} - R)/R_{4.2}$, the fractional change in resistance, and its temperature equivalent, ΔT , are plotted as a function of I^2 . The nominal 4.2°K resistance of the hermetically sealed and perforated encapsulations are $2800\ \Omega$ and $4800\ \Omega$, respectively.

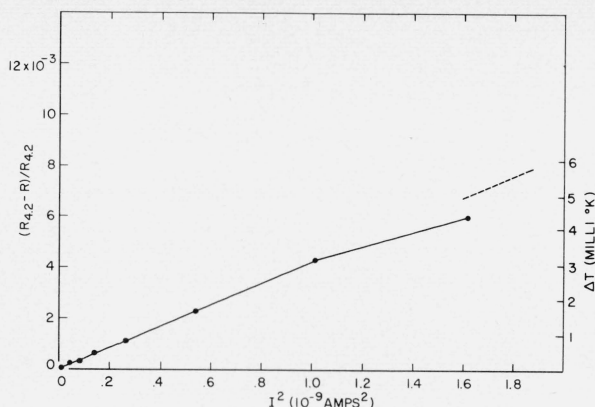


FIGURE 7. Joule heating for a Texas Instrument resistor. $(R_{4.2} - R)/R_{4.2}$, the fractional change in resistance, and its temperature equivalent, ΔT , are plotted as a function of I^2 . The nominal 4.2 °K resistance of the hermetically encapsulated resistor is 1800 Ω.

The lowest points of the curve for the encapsulated, hermetically sealed resistors (figs. 6 and 7) show a negligible change of temperature due to joule heating; consequently, a power dissipation of about 0.02 μW was taken as a reasonable maximum for joule heating and used as an upper limit for all subsequent measurements at 4.2 °K. From the linear portion of the curve in figure 6, we can determine the thermal resistance, $Q = dT/d(I^2R)$ (in degrees-per-microwatt), between the resistor element and its surroundings. For the Honeywell resistor $Q = 0.0045$ °K/μW; Blakemore obtained, for a similar resistor, $Q = 0.0023$ °K/μW [10]. From figure 7 we find for the Texas Instrument resistor $Q = 0.0017$ °K/μW while Low obtained a value $Q = 0.001$ °K/μW [12].

4.2. Reproducibility of Resistors

If a germanium resistor is thermally cycled n times from 300 °K to 4.2 °K and systematic calibrations are made and referenced to a particular temperature, i.e., 4.214 °K, we define the calibration reproducibility of the resistor to mean: the difference, ΔR (ohms), between the maximum and minimum values of resistance that were measured during the calibrations (properly referenced to 4.214 °K) made within the n cycles. From knowledge of the resistor's dR/dT at 4.2 °K, the ΔR can be more pertinently expressed in equivalent millidegrees—thus we refer to calibration reproducibilities at 4.2 °K in terms of millidegrees.

The results of thermal cycling between 300 °K and 4.2 °K for 25 resistors are listed in table 1. The resistors were obtained from three commercial sources and we were seeking information that might lead to the selection of a group of similar resistors that could be used as secondary standards in our laboratory. We established the requirement that to

be useful for our purposes resistors should have a 4.2 °K calibration reproducibility which was 0.001 °K or better. Resistors which failed mechanically during the thermal cycling have not been included in table 1. Some of the resistors (numbers 19, 22, 23, and 25) failed to meet our calibration reproducibility criterion rather early in the cycling procedure while others failed to perform satisfactorily after a greater number of cycles.⁵ In general, accurate 4.2 °K calibrations were performed for the first few cycles, and thereafter every 10 cycles. Seven of the twenty-five resistors were reproducible to 0.0011 °K and have subsequently been used rather extensively in our laboratory. For example, resistors A and B have been incorporated with the acoustical thermometer for several years and will be directly referred to in forthcoming publications.

TABLE 1. Reproducibility of germanium resistors from three commercial sources under thermal cycling from 4.2 °K to room temperature

Resistor serial #	Number of cycles	Average R (ohms) 4.2 °K	Reproducibility (milli- °K)
1	61	2829.0	1.3
2	123	2552.5	0.9
3	275	2628.0	.9
4	11	6260.0	.3
A	50	2868.3	1.0
B	40	3086.0	1.1
7	87	3945.4	1.3
8	50	1074.0	1.3
9 ^a	51	1764.2	1.5
10 ^a	83	2743.0	2.3
11	36	367.75	1.1
12	36	617.74	1.2
13	36	292.75	1.5
14	36	292.50	1.4
15	64	236.84	6.6
16	64	327	5.8
17	40	327	17.4
18	64	675.1	7.1
19	8	671.60	4.6
20	30	1890	0.7
21	30	720	9.0
22	14	23100	10.0 (estimated)
23	14	14500	10.0 (estimated)
24	23	1825	2.1
25	8	1940	3.8

^a Resistor encapsulation was perforated.

An exhaustive, accumulative cycling test was continued for resistor number 3. It was thermally cycled 275 times with equilibrium resistance measurements determined in 43 instances. The results are shown in figure 8. The first series of tests occurred in July 1963 and the resistor was then set aside. On January 7, 1964, the resistor was again connected and the cycling test continued during January–February 1964. The resistance measurements demonstrate a reproducibility of 0.9 mdeg at 4.2 °K.

Figure 9 shows the equilibrium resistance-accumulated cycles plot for resistor number 1. We first measured this resistance in July 1963 and, subse-

⁵ Resistor number 4 was needed for other experimentation so its thermal cycling was not continued.

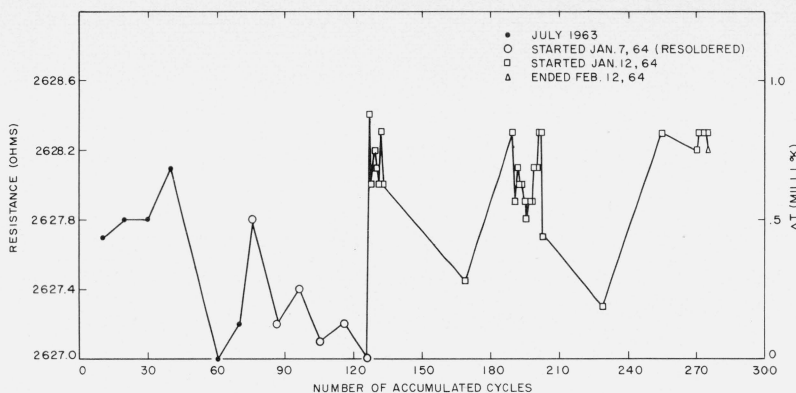


FIGURE 8. Equilibrium resistance as a function of the number of accumulated cycles for #3.
 $T = 4.2^\circ\text{K}$.

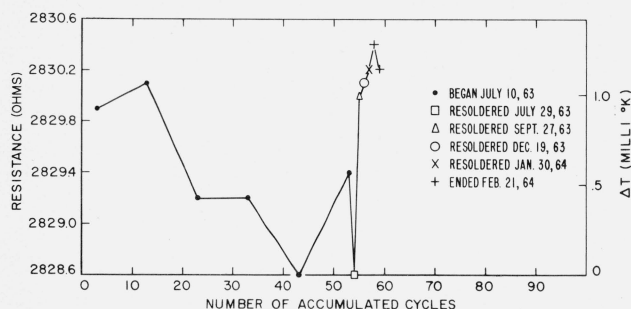


FIGURE 9. Equilibrium resistance as a function of the number of accumulated cycles for #1.
 $T = 4.2^\circ\text{K}$.

quently, made measurements after every 10 cycles until an accumulated total of 53 cycles was reached. The resistor leads were disconnected and subsequently resoldered to the connecting wires five times over a period of six months. Despite this treatment, the resistance shows a reproducibility of 0.0013°K . Between the 56th and 57th cycles, the resistor was inadvertently physically shocked. New connecting wires were soldered to number 1 and additional cycling demonstrated little change from the measurements made two and a half months earlier.

5. Select Resistor Group

After obtaining a group of resistors that offered promising calibration-reproducibilities, we adopted the following method as a criterion for determining resistance-temperature reproducibilities under thermal cycling. First, resistors were to be cycled three to five times from room temperature to 4.2°K prior to a first measurement of resistance. This was adopted when the accumulated data over a number

of resistors occasionally exhibited, for individual resistors, a 4.2°K resistance value for the first cycle that appreciably differed from the rest of the cycling data. This effect may be due to strains developed in the specimen. Naturally, the performance of several cycles prior to a first measurement will eliminate dependence upon this effect [22]. Second, resistors were to be cycled ten times with an equilibrium resistance measurement performed after each cycle. Third, if the data showed a reproducibility of approximately 1 mdeg (during the previous 10 cycles), resistors were to be cycled 75 more times with an equilibrium resistance measurement performed after every 25th cycle. A cycle in which a resistance measurement is not performed consists of retaining the resistor for 3 min at the top of the Dewar neck, 1 min in the room and returning it for one minute's exposure at the Dewar bottom. By this procedure, accumulative effects of cycling, if any, were to be detected. Thus, a total of some 85 cycles with 14 resistance-temperature calibrations were to be performed in this "search." The results, shown in table 2, demonstrate that 10 out of 12 resistors were reproducible to approximately 0.001°K ; four to 0.0007°K or better. The equilibrium resistances for resistors L, G, and I as a function of accumulated thermal cycles are shown in figures 10 to 12. All three resistors were cycled simultaneously and measured within several minutes of each other during the test. Resistors L and I appear to be good thermometers with reproducibilities of 0.0007°K and 0.0008°K while G exhibits a reproducibility of only 0.0019°K . Another group of three resistors D, J, and N were tested simultaneously and their equilibrium resistances versus accumulated cycles are displayed in figures 13 to 15. Resistors D, J, and N exhibit reproducibilities of 0.45, 0.66, and 0.7°mK , respectively.

TABLE 2. Reproducibility of a selected group of resistors under thermal cycling from 4.2 °K to room temperature

Resistor	Number of cycles	Average R (ohms) 4.2 °K	Reproducibility (milli- °K)
C	88	2800	1.1
D	86	2565	0.5
E	85	2890	1.0
F	88	2678	1.1
G	86	2561	1.9
H	87	2718	^a 1.2
I	86	2790	0.8
J	86	2593	.7
K	87	2620	1.1
L	86	2672	0.7
M	87	2659	.9
N	86	2769	.7

^a Resistor not cycled prior to a first measurement.

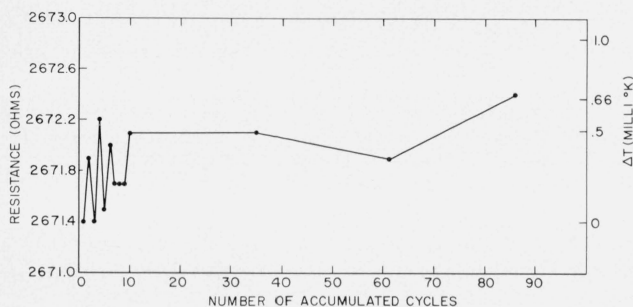


FIGURE 10. Equilibrium resistance as a function of the number of accumulated cycles for resistor L.
 $T = 4.2$ °K.

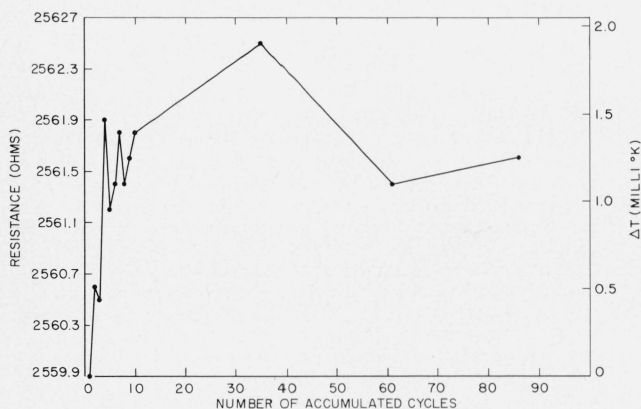


FIGURE 11. Equilibrium resistance as a function of the number of accumulated cycles for resistor G.
 $T = 4.2$ °K.

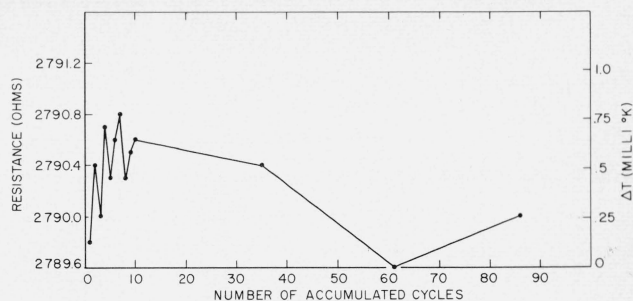


FIGURE 12. Equilibrium resistance as a function of the number of accumulated cycles for resistor I.
 $T = 4.2$ °K.

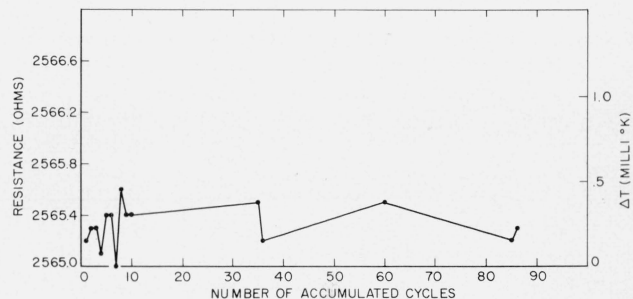


FIGURE 13. Equilibrium resistance as a function of the number of accumulated cycles for resistor D.
 $T = 4.2$ °K.

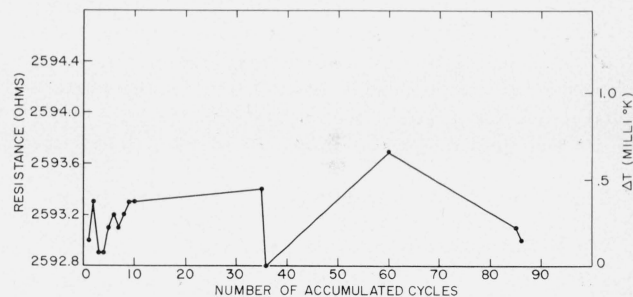


FIGURE 14. Equilibrium resistance as a function of the number of accumulated cycles for resistor J.
 $T = 4.2$ °K.

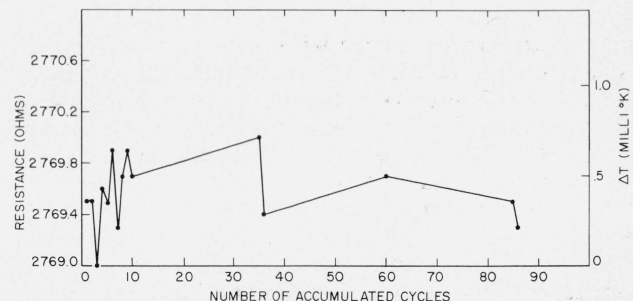


FIGURE 15. Equilibrium resistance as a function of the number of accumulated cycles for resistor N.
 $T = 4.2$ °K.

6. Measurement Errors

The measurement errors for the potential and current readings are estimated to be approximately 0.005 percent. At 4.2 °K the resistance-temperature coefficients ($\frac{1}{R} \frac{dR}{dT}$) are approximately 0.06 percent per m°K. The error in a resistance determination, therefore, is equivalent to 0.17 m°K. The estimated error in pressure measurements is 0.01 percent; since $\frac{1}{P} \frac{dP}{dT}$ for helium vapor pressures at 4.2 °K is close to 0.1 percent per m°K, the estimated error in pressure determinations is equivalent to 0.1 m°K. Spurious temperature fluctuations within the bulk liquid helium bath are estimated as 0.1 or 0.2 m°K. Thus the total measurement error in determining resistance-temperature calibration reproducibilities at 4.2 °K could become as large as a temperature indeterminacy of 0.4 or 0.5 m°K. Recognizing that the reproducibilities of column 4, table 2, are maximum calibration variations (over an average of 14 calibration determinations for each resistor), the experimental reproducibilities (0.7 m°K or less) for the best resistors are in satisfactory agreement with the estimated error (0.4 or 0.5 m°K).

7. Conclusions

The germanium resistors that have been reported in table 1 were, to our knowledge, not preferentially selected. Rather, they constitute representative resistors from three commercial sources. Our experiments have indicated that 14 of the 25 resistors reproduce a 4.2 °K resistance-temperature calibration within 2.0 m°K while only 7 of the group failed to reproduce within 5.0 m°K. This would seem to indicate a general availability of germanium resistors that are suitable for many low temperature purposes where the thermometry needs are not exacting.

The selection of resistors reported in table 2 was preferential (guidance was furnished by the results of investigations on the first group—table 1) and the reproducibilities listed in column 4 clearly indicate the general superiority of this selection—7 of the 12 resistors were reproducible within 1.0 m°K.

This investigation has fulfilled our original purpose—to obtain a group of germanium resistance thermometers that offered the promise of being suitable for use as secondary thermometers. Since the investigation was completed nearly 2 years ago, there has been ample time for employing the resistors as secondary thermometers in subsequent endeavors. Resistors A and B (table 1) have been used extensively in acoustical thermometry; C and D (table 2) have been calibrated with reference to the acoustical thermometer and are the basic reference for calibrations between 2 and 20 °K at the National Bureau of Standards.

Articles on analytical representations of germanium resistor calibration data are in preparation and will appear in the literature.

Since some of the germanium resistors listed in table 1 were obtained as long ago as 5 or 6 years, we have no opinions concerning the current availability of particular models. Without doubt some are now outdated due to advances in manufacturing technology. We have not had the opportunity to investigate groups of resistors which have been commercially available during the last 3 years.

8. References and Notes

- [1] J. R. Clement and E. H. Quinzel, *Rev. Sci. Instr.* **23**, 213 (1952).
- [2] J. R. Clement, *Temperature, Its Measurement and Control in Science and Industry* (Reinhold Publishing Corporation, New York, 1955), Vol. **2**, H. C. Wolfe, ed., p. 380.
- [3] P. Lindenfeld, *Temperature, Its Measurement and Control in Science and Industry* (Reinhold Publishing Corporation, New York, 1962), Vol. **3**, C. M. Herzfeld, ed., Part 1, F. G. Brickwedde, ed., p. 399.
- [4] M. H. Edlow and H. H. Plumb, *Advances in Cryogenic Engineering* (Plenum Press, New York, 1961) Vol. **6**, p. 542.
- [5] I. Estermann, *Phys. Rev.* **78**, 83 (1950).
- [6] S. W. Friedberg, *Phys. Rev.* **82**, 764 (1951).
- [7] T. H. Geballe, F. J. Morin, and J. P. Marta, *Conference de Physique des Basses Temperature*, Paris (1955) p. 87.
- [8] J. E. Kunzler, T. H. Geballe, and G. W. Hull, *Rev. Sci. Instr.* **28**, 96 (1957).
- [9] P. Lindenfeld, *Opt. Cit.*, p. 399.
- [10] J. S. Blakemore et al., *Rev. Sci. Instr.* **33**, 106 (1962).
- [11] J. S. Blakemore, *Rev. Sci. Instr.* **33**, 545 (1962).
- [12] F. J. Low, *Advances in Cryogenic Engineering*, (Plenum Press, New York, 1961) Vol. **7**, p. 514.
- [13] Standard Resistors are certified by the National Bureau of Standards.
- [14] E. Ambler and H. H. Plumb, *Rev. Sci. Instr.*, **31**, 636 (1960).
- [15] H. H. Plumb and M. H. Edlow, *Rev. Sci. Instr.* **30**, 376 (1959).
- [16] G. Cataland, M. H. Edlow and H. H. Plumb, *Temperature, Its Measurement and Control in Science and Industry* (Reinhold Publishing Corporation, New York, 1962), Vol. **3**, C. M. Herzfeld, ed., Pt. 1, F. G. Brickwedde, ed., p. 413.
- [17] G. Cataland, M. H. Edlow, and H. H. Plumb, *Rev. Sci. Instr.* **32**, 980 (1961).
- [18] D. Ditmars and G. Furukawa, *J. Res. NBS*, 69C (Engr. and Instr.) No. 1, 35 (1965); K. W. Taconis, J. J. Beenakker, A. O. C. Nier, and L. T. Aldrich, *Physica* **15**, 733 (1949); H. A. Kramers, *ibid*, 971 (1949).
- [19] F. G. Brickwedde, H. van Dijk, M. Durieux, J. R. Clement, and J. K. Logan, *NBS Monograph* 10 (1960); *J. Res. NBS*, 64A (Phys. and Chem.) No. 1, 1 (1960).
- [20] M. Durieux, thesis, University of Leiden (1960).
- [21] Our thanks to the following people who have supplied us with resistors for these experiments:
Mr. Tom Herder, formerly of Minneapolis-Honeywell Company, Semiconductor Division, Riviera Beach, Florida;
Mr. Gilbert Halverson, Radiation Research Corporation, West Palm Beach, Florida;
Mr. Jack Veasaw, Industrial Products Group, Texas Instruments, Inc., Houston, Texas.
- [22] During the period that Mr. Herder was associated with Minneapolis-Honeywell Company, he noted and reported similar effects in a private communication to us.

(Paper 70C4-235)