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## Indium Resistance Thermometer; 4 to 300°K

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The possible advantages are discussed of a resistance thermometer made with a metallic element of low Debye characteristic temperature and very high purity, e.g., ratio of residual resistance to ice-point resistance of 10<sup>-4</sup>. Measurements on the electrical resistance of commercially available indium wire from 3 to 300°K suggest that it may be a useful thermometer as it remains temperature sensitive down to its superconducting transition at 3.41  $^{\circ}$ K, because of its high purity and  $\theta \sim 100 ^{\circ}$ K.

#### INTRODUCTION

HE problem of finding a reproducible electrical resistance thermometer which retains its sensitivity over the entire range from room temperature down to the temperature of liquid helium, is one of long standing (see, for example, Daunt<sup>1</sup> and Friedberg<sup>2</sup>).

The platinum resistance thermometer has served very well for may years for realization of the International Temperature Scale from -182,97°C to 630°C and will probably continue to do so; it combines the merits of being an isotropic noble metal, being ductile and obtainable in a state of moderately high purity with a highly reproducible electrical resistance which varies in a nearly linear fashion with temperature over this wide range. However high purity platinum wire, for which the ratio of the electrical resistance at 4.2°K  $(R_{4,2})$  to its ice-point resistance  $(R_{273})^3$  appears to be about 10<sup>-3</sup> at best, becomes relatively insensitive at low temperatures; for platinum there is a significant departure of the resistance-temperature relation from linearity below about 60°K, and below 15°K the residual resistance caused by impurity scattering becomes dominant. On the other hand, a metal of appreciably lower Debye characteristic temperature  $\theta_D$  retains approximate proportionality of resistance to temperature at lower temperatures, and if obtainable in a very pure form (for example, such that  $R_{4,2}$ /  $R_{273}\sim 10^{-4}$ ) could be a useful thermometric element down to liquid helium temperatures. Recently, while considering the results of an extended series of measurements on the electron transport properties of alkali metals and transition metals,4 we considered which metallic elements might have desirable thermometric

properties. Of those with a  $\theta_D \leq 100^{\circ}$ K (cf  $\theta_D \sim 225$  for Pt) one finds that (a) K, Cs, Rb exhibit hysteresis effects in their resistance, perhaps because of the effect of the constraint imposed by the glass moulds in which they are cast and used, and perhaps to the influence of transformations of the Martensitic type, (b) gallium is highly anisotropic and melts at 29.75°C and is consequently unsuitable, (c) thallium oxidizes rather rapidly at room temperature, (d) bismuth might be suitable but is difficult to obtain with a very low residual resistance and in the form of fine wires, (e) lead appears to offer many advantages and has been used in calorimetry as a thermometer, 5,6 but becomes superconducting somewhat above liquid helium temperatures, namely at 7.2°K. Finally however, indium, which is readily available in 99.999% pure form (e.g. Tadanac brand from Consolidated Mining & Smelting Company of Canada Ltd.), has a  $\theta_D \sim 100^{\circ}$ K, is comparatively unreactive chemically and has a superconducting transition at about 3.4°K. It has a slightly anisotropic crystal structure (see for example Hampel, for general properties of indium and other rare metals) being face centered tetragonal, and might therefore show some lack of reproducibility on thermal cycling.

#### EXPERIMENT

Finding that a sample cut from a sheet of Tadanac indium gave  $R_{4,2}/R_{295}\sim 9\times 10^{-5}$ , we extruded a few feet of wire of 0.016 in, diameter and made two rather crude resistance thermometers known as In 1 and In 2. The wire was wound bifilarly on a mica cross (In 1) and on a cylinder of Styrofoam (In 2), and then small protective glass tubes were slipped around these.

From each end of the wire, a  $\frac{1}{2}$ -in. length of indium was cut off and then cold-welded (by pressure of tweezers) to the wire to form a Y junction; fine copper leads were then hot-welded to the ends for current and potential leads.

The two "thermometers," In 1 and In 2, were then mounted in a cryostat8 of which the central experi-

<sup>&</sup>lt;sup>1</sup> J. G. Daunt in Temperature, Its Measurement and Control in Science and Industry, H. C. Wolfe, Editor (Reinhold Publishing Corporation, New York, 1955), p. 327.

<sup>&</sup>lt;sup>2</sup> S. A. Friedberg, reference, p. 359. <sup>3</sup> For simplicity we shall express temperatures in degrees Kelvin throughout this paper, and use  $R_{272}$  to signify the electrical resistance at 0°C or 273.15°K; the designation of  $R_0$  for ice-point resistance, used commonly in discussing thermometry at higher temperatures is apt to be confused in a low temperature discussion with the resistance at absolute zero or the residual impurity resistance.

<sup>&</sup>lt;sup>4</sup> For example, MacDonald, White and Woods, Proc. Roy. Soc. (London) A235, 358 (1956); G. K. White and S. B. Woods, Can. J. Phys. 35, 248 (1957); 35, 346 (1957); Kemp, Klemens, and White, Australian J. Phys. 9, 180 (1956).

A. A. Silvidi and J. G. Daunt, Phys. Rev. 77, 125 (1950).
 N. Pearlman and P. H. Keesom, Phys. Rev. 88, 398 (1952).
 C. A. Hampel, Rare Metals Handbook (Reinhold Publishing Corporation, New York, 1956).
 G. K. White and S. B. Woods Can. J. Phys. 33, 58 (1955).

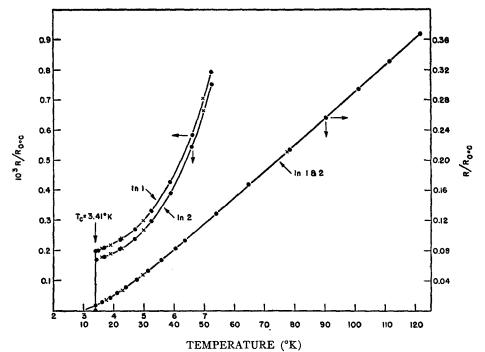


Fig. 1. Some values of the electrical resistance of indium as a function of a temperature:
• run 1; × run 2.

mental chamber is normally filled with a small pressure of helium exchange gas, and which can be temperature controlled at points from 1.2° to 300°K. Both a helium gas thermometer bulb connected to an external butyl phthalate manometer and a platinum resistance thermometer, T4, calibrated and kindly lent to us by Dr. J. A. Morrison of these Laboratories were also mounted in this central chamber.

Since the room temperature resistance of the two indium thermometers was only about 0.3 ohm in each case, and this resistance dropped to about 50 microohms at  $4^{\circ}$ K, measurements of their resistances were made with a direct reading galvanometer amplifier<sup>10</sup>; this has maximum sensitivity of about 10-cm deflection per microvolt, and gives a probable error in reading of  $\pm \frac{1}{4}\%$ .

#### RESULTS

Within the limits imposed by the accuracy of the galvanometer amplifier, resistance values were quite reproducible. The thermometers were cooled to  $77^{\circ}$ K and rewarmed to room temperature on a number of occasions, and two experiments (called Run 1 and Run 2) were made with liquid helium in each of which both thermometers were cooled to  $4.2^{\circ}$ K, then warmed to about  $45^{\circ}$ K while readings were taken, cooled again to  $3^{\circ}$ K, and later warmed to about  $20^{\circ}$ K before readings were completed. Some results are shown in Fig. 1. No serious attempt to find an analytic expression for  $R/R_{273}$  as a function of T has been made, but above

100°K an equation of the form  $A+BT+CT^2$  appears to fit the experimental values quite well; from 100°K down to about 40°K, the departure from a linear function  $R/R_{273}=A+BT$  is very small. As might be expected from the Bloch-Grüneisen theory, the resistance at temperatures below 10°K may be approximately represented by a formula of the type  $R=A+BT^5$ .

On the idealized Bloch-Grüneisen model of a free electron metal (see review by MacDonald<sup>11</sup>) we should expect the low- and high-temperature values of the electrical resistance (R and  $R_{\infty}$ , respectively) to be related by

$$\frac{R_i}{R_{\infty}} = \frac{R - R_r}{R_{\infty} - R_r} = 497.6 \left(\frac{T}{\theta}\right)^4 \frac{T}{T_{\infty}},$$

where  $T \ll \theta < T_{\infty}$ ; Here R is the "ideal" electrical resistance due to scattering of electrons by vibrations and  $R_r$  is the residual resistance due to scattering by imperfections. From our results for indium, a value of  $\theta \sim 85^{\circ}$ K is obtained.

It may be observed from the Fig. 1 that R is still quite temperature sensitive at about  $4^{\circ}$ K, i.e., the constant residual resistance is not yet completely dominant. The superconducting transition appeared to be quite sharp, the maximum width as observed with a measuring current of 10 ma being less than  $0.002^{\circ}$ K. The temperature of the transition, Tc, is close to  $3.412^{\circ}$ K, in good agreement with values

J. M. Los and J. A. Morrison Can. J. Phys. 29, 142 (1951).
 D. K. C. MacDonald, J. Sci. Instr. 24, 232 (1947).

<sup>&</sup>lt;sup>11</sup> D. K. C. MacDonald, *Handb. der Physik* Vol 14, p. 137. (Springer-Verlag, Berlin, 1956).

TABLE I. Z functions	for indium ar	nd platinum w	here $Z = (R -$	$(R_{4,9})/(R_{273}-R_{4,9})$

Indium Platinum		um		Indi	um	Platinum			
T°K	Z	$\Delta T/\Delta Z$	Z	$\Delta T/\Delta Z$	T°K	Z	$\Delta T/\Delta Z$	z	$\Delta T/\Delta Z$
3.5	-0.000035 $-0.000012$	217 000			30 35	0.0488 0.0651	307	0.016658 0.027589	457.4
4.5 5.0 6.0 7 8	+0.000016 0.000062 0.000214 0.000468 0.000860	17 850 11 000 6500 4000 2525 1695			40 45 50 60 70	0.0813 0.0980 0.1157 0.1505 0.1855	316 299 283 287 286 290	0.041258 0.057201 0.074942 0.114094 0.15606	365.8 313.6 281.8 255.4 238.3 232.3
9 10 11	0.00145 0.00220 0.00314	1333 1064			80 90 100	0.2200 0.2555 0.2908	282 283	0.19911 0.24259 0.28592	230.0 230.8
12 13	0.00425 0.00562	900 730 653	0.0005082 0.0006918	5447 4357	120 140	0.3620 0.437	280 267 260	0.371626 0.456220	233.4 236.4 239.2
14 15	0.00715 0.00888	578 532	0.0009213 0.0012074	3495 2862	160 180	0.514 0.593	253.1 243.9	0.539842	239.2
16 18	0.01076	472 417	0.0015568 2171 0.0024777 1580	2171	200 220 240	0.675	235.2 232.5		
20 22 24	0.0198 0.0250 0.0305	385 364	0.0037432 0.0054061 0.0075078	1203 951.6	260 280	0.846 0.937 1.032	219.7 210.5		
25	0.0333	357 322	0.008732	816.3 630.8	300 (273.15)	1.128 (1.000)	208.3	(1.000)	

obtained by Tuyn and Kamerlingh-Onnes<sup>12</sup> on rather less pure specimens.

With the possible use of indium as a low-temperature resistance thermometer in mind, we have tabulated values of the reduced electrical resistance  $Z=(R-R_{4,2})/(R_{273}-R_{4,2})$  as a function of temperature (Table I). The values of Z for the two specimens agreed to within the limits of experimental accuracy, and might be expected to represent values for other specimens equally well provided that their purity—as evidenced by the ratio  $R_{4,2}/R_{273}$ —is not vastly different from these. The general validity of such a Z function depends on the extent to which Matthiessen's rule is obeyed, i.e., the validity of the rule that the "ideal" resistance (caused solely by thermal vibrations) and impurity resistance (caused solely by imperfections and therefore constant) are strictly additive.

For purposes of comparison, values of Z and  $\Delta T/\Delta Z$  for the platinum resistance thermometer,  $T_4$  are also given in the table. This thermometer<sup>9</sup> has an ice-point

resistance of 24.3107 ohms and a resistance of  $0.0110_2$  ohms at 4.20°K. Values of Z were calculated from a calibration table kindly supplied by Dr. Morrison, covering the range 11.350°K to  $174.612_5$ °K.

The electrical resistivity at room temperature (22°C) of a sample of the Tadanac indium wire is approximately  $8.8 \times 10^{-6}$  ohm cm.

Our results on indium seem to be in quite good agreement with those of Swenson<sup>13</sup> who measured the electrical resistance from  $4^{\circ}$  to  $273^{\circ}$ K of a sample of indium as part of a wider investigation of the mechanical properties of indium and thallium at low temperatures. The major difference in results is that Swensor reported a discontinuity of about 10% in the slope of the resistance-temperature curve at about  $210^{\circ}$ K; he found R(T) to be linear above  $210^{\circ}$ K and again from  $210^{\circ}$ K down to  $160^{\circ}$ K, whereas our values in this region lie on a smooth curve such that they coincide with Swenson's at about  $60^{\circ}$ K,  $210^{\circ}$ K, and  $280^{\circ}$ K but lie a little lower (up to 2%) at intermediate temperatures. It seems possible that this difference or the discontinuity

<sup>&</sup>lt;sup>12</sup> W. Tuyn and H. Kamerlingh-Onnes, Leiden Communications 167a (1923).

<sup>&</sup>lt;sup>13</sup> C. A. Swenson, Phys. Rev. 100, 1607 (1955).

in Swenson's results is due to the somewhat lower purity of his indium  $(R_{4.2}/R_{273}=10^{-3})$ .

It is felt that indium, both because of its low Debye temperature and its availability in a very pure state might be a useful resistance thermometer over the entire temperature range from room temperature down to liquid helium temperatures. So far as we have been able to ascertain its resistance is quite reproducible but further measurements made with an accurate potentiometer or bridge (on a thermometer of higher total resistance) are required to confirm this. The metal is extremely soft at normal temperatures and anneals at room temperature. It should be possible to

extrude wire of less than 0.010 in. diameter and wind a resistance thermometer of  $R_{273}\sim10$  ohm without great difficulty. A thermometer made by winding fine indium wire on an insulated former of indium (rod or tube) might have practical advantages in cryogenic applications, but may also show small hysteresis effects due to small differences in expansion and contraction of the wire and its former, resulting from slight anisotropy in crystal structure. However its possible usefulness as a practical alternative to a combination of a platinum or copper thermometer (15–300°K) and a semiconducting or carbon thermometer (4–20°K) seems well worth considering.

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# All Metal Apparatus for the Determination of the Density of Liquefied Gases\*

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An all metal apparatus designed for density measurements on liquefied gases is described. The metal construction enabled its use with corrosive materials, and also allowed measurements to be made to moderate pressures. It consists of a pycnometer, temperature control device, and gas handling system. While this apparatus was particularly designed for density work it was used for vapor pressure determinations with slight modification. The design and construction is detailed and its use with liquid fluorine outlined.

#### INTRODUCTION

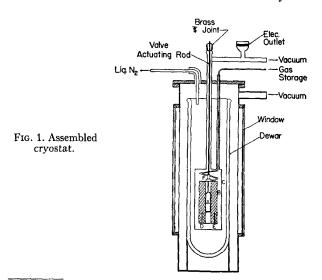
THE conventional method for measuring the density of a liquefied gas is by means of a dilatometer. In this type of apparatus the expansion coefficient of the liquid is the function actually measured. As these are made of glass so that the rise of the liquid can be observed they do not adapt themselves for work with materials that attack glass. In addition, they cannot be used conventionally at high pressures.

In our work with liquid fluorine<sup>1</sup> an apparatus was devised to overcome these deficiencies. This all metal apparatus acted as an ordinary pycnometer, in having a constant volume, while allowing a continuous range of measurements as with a dilatometer.

## DESCRIPTION OF APPARATUS

The apparatus consists of two general parts; the pycnometer contained in a cryostat and a volumetric system for measuring the quantity of material condensed into the pycnometer.<sup>2</sup> Figure 1 shows the assembled

cryostat diagrammatically. The cryostat is similar to those in general use in the various low temperature laboratories in this country. The pycnometer A, was made of  $\frac{3}{4}$ -in. nickel pipe with silver soldered ends. Valve  $F^3$  was connected to A and to the outside system



Ohio State University [H. L. Johnston, et al., Phys. Rev. 79, 235 (1950)].

<sup>\*</sup> This paper is the result of work done under Contract No. 18(600)761, supported by the United States Air Force through the Air Force Office of Scientific Research of the Air Research and Development Command.

<sup>&</sup>lt;sup>1</sup> R. L. Jarry and H. C. Miller, J. Am. Chem. Soc. 78, 1552 (1956).

<sup>&</sup>lt;sup>2</sup> Following the design and operation of this apparatus Dr. A. S. Friedman of the National Bureau of Standards pointed out the similarity of our apparatus to a P-V-T apparatus developed at The

<sup>&</sup>lt;sup>3</sup> Hoke, Inc., Style 1103, obtained through the AEC, Oak Ridge, Tennessee. This valve is packless, the bonnet seal being made through a brass bellows.