CLM - R 47

CLM - R 47

United Kingdom Atomic Energy Authority

RESEARCH GROUP

Report

THE MEASUREMENT OF TEMPERATURE IN THE RANGE 3°K TO 80°K USING CARBON RESISTORS

D. H. J. GOODALL

Culham Laboratory,
Culham, Abingdon, Berkshire
1966
Available from H. M. Stationery Office
TWO SHILLINGS AND SIXPENCE NET

U.D.C.
536.531

UNCLASSIFIED
(Approved for Sale)

CLM-R 47

THE MEASUREMENT OF TEMPERATURE IN THE RANGE $3^{o}K$ TO $80^{o}K$ USING CARBON RESISTORS

by

D.H.J. GOODALL

ABSTRACT

The low temperature characteristics of some commercial carbon resistors are obtained using a vapour pressure thermometer as a standard. The temperature characteristics calculated from three resistance measurements near $4^{o}K$, $20^{o}K$ and $77^{o}K$ give a good approximation to the experimentally determined characteristics. The stability of the resistors improves with time and thermal cycling, and is better than ± 1% at $4.2^{o}K$ and $77^{o}K$.

U.K.A.E.A. Research Group,
Culham Laboratory,
Nr Abingdon,
Berks.

SEPTEMBER, 1965 (C/7 IMG)

CONTENTS

1. INTRODUCTION

Carbon resistors, especially those manufactured by the Allen Bradley Corporation(1), have been used successfully for low temperature measurements(2 - 4). Interest has however been centred mainly in the temperature range $1^{\circ} - 20^{\circ}$K and information about stability(5) is not necessarily related to conditions met in practice. Furthermore methods of manufacture change and new types of resistors may be unsuitable for use as thermometers. The present work is concerned therefore with the calibration of existing stocks of resistors and an investigation into their stability when thermally cycled over a period of time.

2. THE METHOD OF CALIBRATION

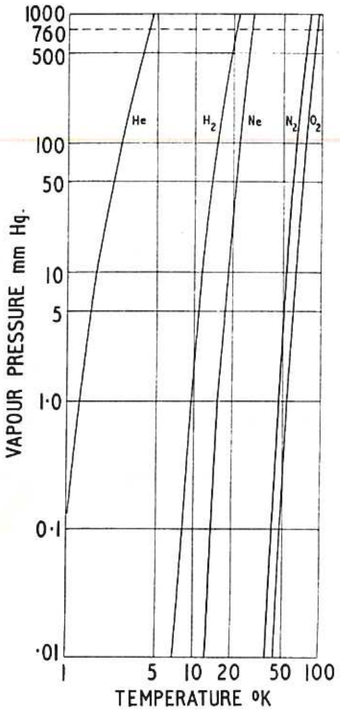

Fig.1 (CLM-R 47)
The vapour pressure curves of the gases used in the vapour pressure thermometer.

The 'Standard' thermometer against which the resistors were calibrated was a vapour pressure thermometer. This was chosen for the following reasons:

(1) Small changes in temperature produce large changes in the vapour pressure of a gas over its working range(6); for example, an 0.01°K change in the temperature of liquid helium near 4°K, produces a change in vapour pressure of 7 mm Hg.

(2) Pressures are fairly easy to measure accurately and by using a mercury manometer as a standard for calibrating the pressure indicating device, few sources of error are introduced.

(3) A vapour pressure thermometer is easy to use, and is robust and stable.

A disadvantage is that several gases must be used to cover a reasonable range of temperatures, and even then there are temperatures which cannot be measured. Fig.1 shows the vapour pressure curves(7) of the five gases used for calibration, and Fig.2 shows the gaps which cannot be measured with vapour pressure thermometers. The width of these gaps can be reduced by measuring vapour pressures below 1 mm Hg. but it

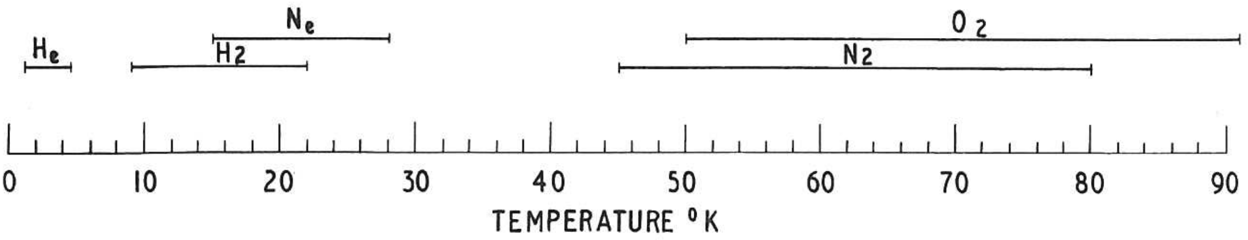

Fig.2 (CLM-R 47)
Working range of the gases used in the vapour pressure thermometer

is not easy to do this accurately. The purity of the gas also becomes more important in this region and all traces of gases more volatile than the one in use must be removed if large errors are to be avoided.

3. THE APPARATUS

A simplified diagram of the apparatus is shown in Fig.3. It consists of a double glass dewar in which is placed a vacuum jacketed copper block containing a vapour pressure thermometer bulb. The resistors to be calibrated are placed in pockets in the copper block. The voltage and current leads are 40 swg pure copper enamelled wire and they are taken from the resistors through the tube S to the measuring instruments.

The temperature of the block is changed by varying the distance of the block from the refrigerant surface by means of the sliding tube S. By pumping the inner dewar the boiling point of the refrigerant is lowered allowing further variation.

The pressure in the dewar and hence the refrigerant temperature is controlled by the bellows system shown. This consists of a flexible bellows, 4½" in diameter, inside a sealed container. When the pressure inside the bellows exceeds that of the surrounding container, the microswitch opens an electromagnetic valve in the pumping line. The pressure in the dewar is then reduced and the microswitch closes the valve when the pressure differential across the bellows falls to zero. The operating pressure as indicated by gauge C is set by valves 1 and 2. When liquid helium is used in the inner dewar, this vapour pressure control system maintains the pressure with an accuracy of ± 0.05cm~~m~~ Hg and the bath temperature is therefore constant within ± 0.001°K near 4°K.

The vapour pressure thermometer bulb is filled by allowing a known volume of gas to condense into the bulb when it is immersed in the refrigerant. This is done by filling the five litre flask to a predetermined pressure and observing the pressure change (gauge B) as the gas condenses. With the exception of helium, the gas is then solidified and pumped to remove any traces of more volatile gases. The solid gas is then allowed to melt to release any trapped impurity; the gas is then refrozen and pumped. This process is repeated several times.

The temperature of the copper block and therefore of the resistors is obtained from the reading of the pressure gauge 'A' and the appropriate vapour pressure curve. The gauge 'A' is a precision dial manometer(8) which is calibrated for pressures from 0 to 1200 mm Hg ± 1.0 mm Hg. (Measured with respect to its evacuated case.) The dial manometer is much more convenient to use than a mercury manometer and no appreciable errors were observed.

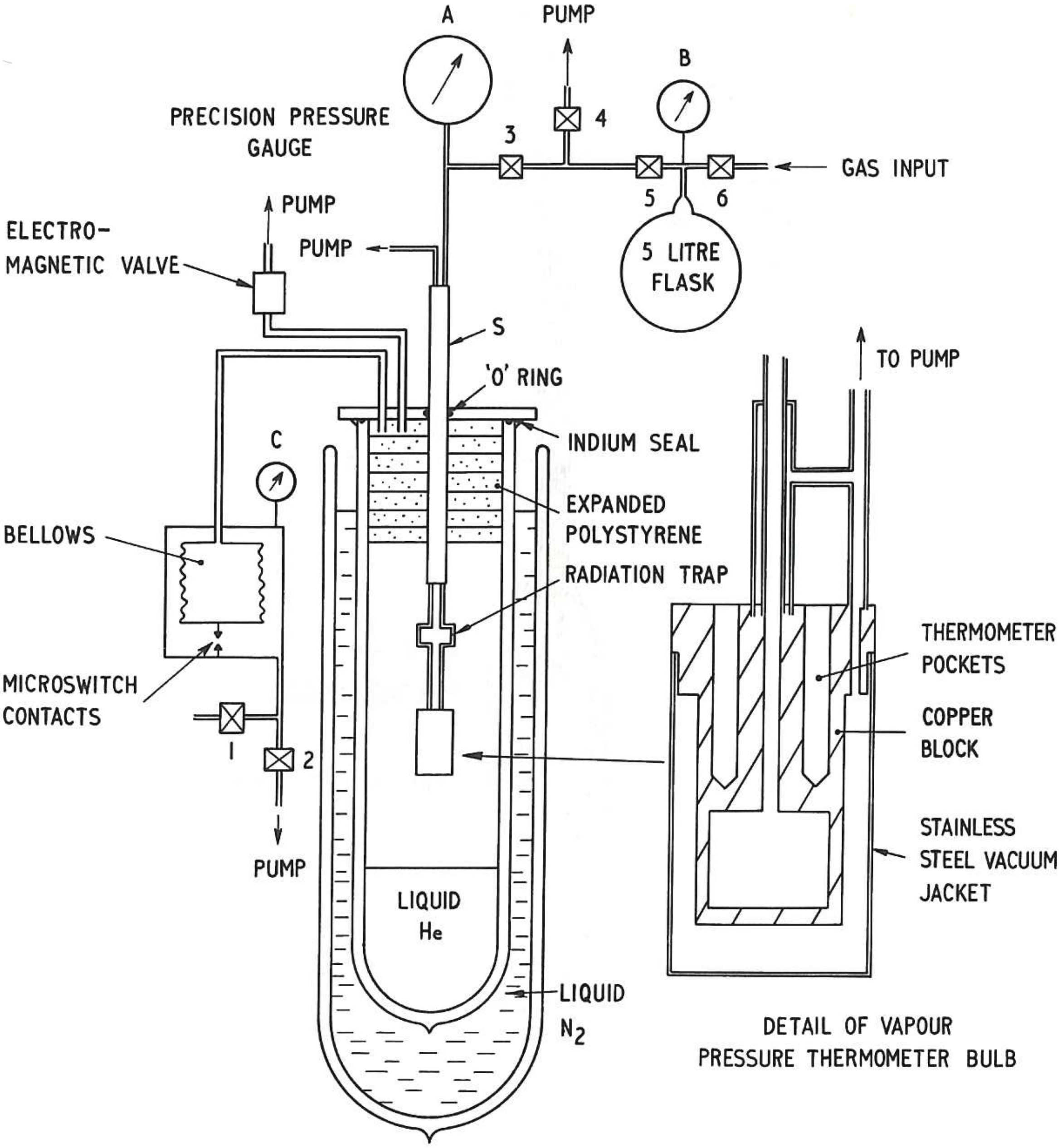

Fig.3 Schematic diagram of the apparatus (CLM-R47)

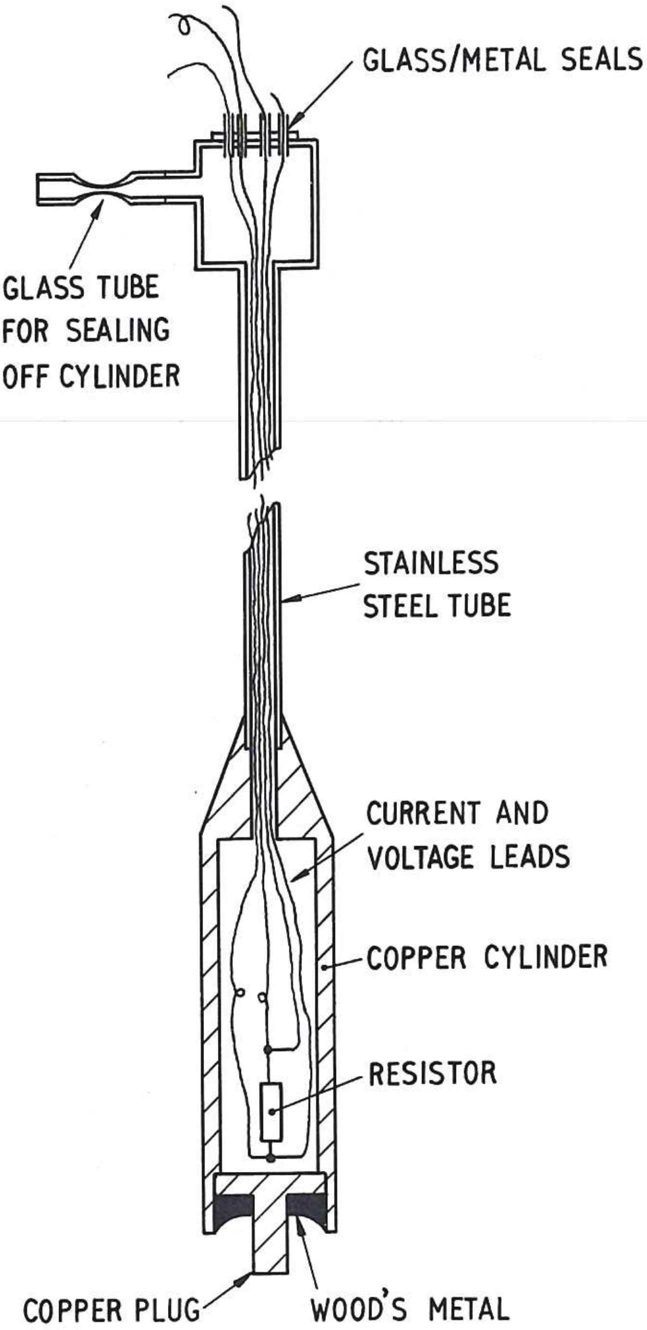

Fig.4 (CLM-R 47)
The apparatus used in a standard helim dewar for resistor stability measurements

By varying both the bath temperature and the height of the bulb above the liquid, a complete temperature calibration can be obtained over the working range of the gas.

The resistance of the thermometer to be calibrated is found by passing a known current through the resistor and by measuring the potential difference across it. The current is obtained from the potential difference across a standard resistance. Both potentials are measured with an accurate potentiometer(9).

An additional piece of apparatus for stability measurement at 4.2°K and 77°K is shown in Fig.4. This consists of a copper cylinder, 5/8" diameter which is small enough to pass down the neck of a standard liquid helium dewar. Resistors are placed inside the cylinder and the voltage and current leads are taken through the stainless steel tubing and the vacuum seal as shown. The cylinder itself is sealed by a plug soldered with Wood's metal. This enables the seal to be made without overheating the resistors. The apparatus is filled with helium to a pressure of 1200 mm Hg (absolute) which corresponds to a pressure of 100 mm Hg when the cylinder is immersed in liquid helium at 4.2°K. This apparatus is very successful when spot temperature checks are required, and it is very economical in the consumption of liquid helium.

4. EXPERIMENTAL RESULTS

Initially calibrations using 10 Ohm Allen Bradley resistors were attempted with the resistors inside a vapour pressure thermometer, i.e. in contact with the liquified or solidified gas. This technique was unsuccessful. In liquid hydrogen the calibration was not consistent with the calibrations in other gases and differed from later results obtained with the resistors mounted in the pocketed copper block. Solidified gas around the resistors also gave very erratic results; this was observed using solid hydrogen, neon and air, and is probably due to bad thermal contact between the resistor surface and the surrounding solid. Fig.5 shows a typical discontinuity in the temperature calibration for solid hydrogen. It is therefore important to ensure that the resistors are not in contact with solids or liquids and for the following results the resistors calibrated are surrounded by gas only.

Fig.6 shows the resistance vs temperature characteristic for a 10 Ohm ¼ watt Allen Bradley resistor, (Resistor A). This curve was obtained for a current of 500 μA using helium, hydrogen, neon, oxygen and nitrogen in the vapour pressure thermometer. It will be seen that there is good agreement between the various gases where their working range overlaps. Two other 10 Ohm resistors (B and C) from the same batch of resistors were

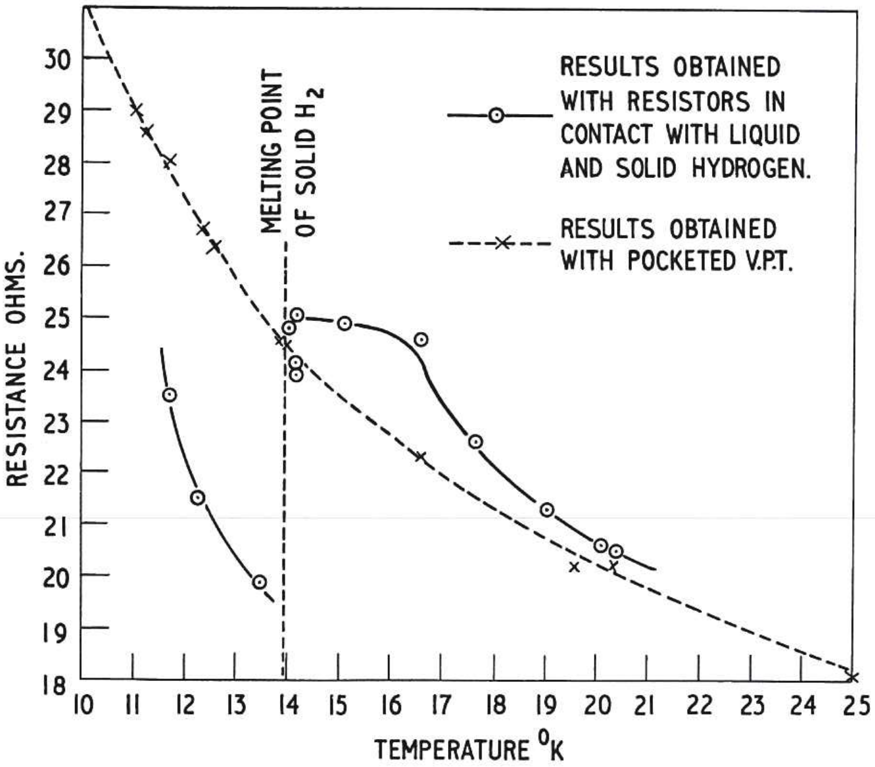

Fig.5 (CLM-R 47)

A typical example of the errors in temperature measurement which are possible when resistors are in contact with a liquified or solidified gas

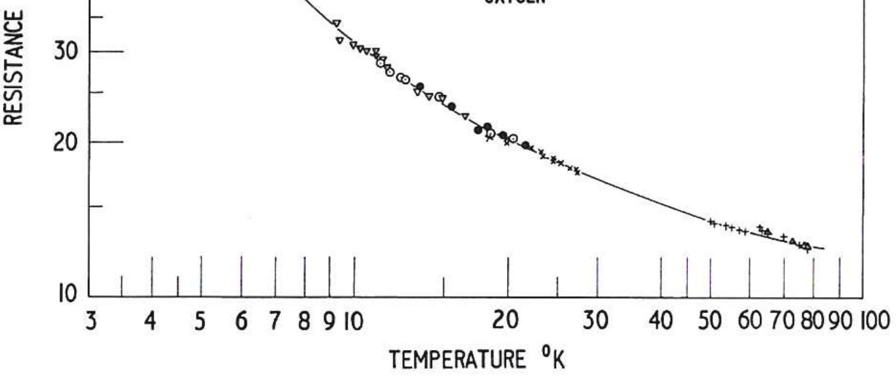

Fig.6 (CLM-R 47)

The temperature characteristic of a nominal 10 ohm ¼ watt Allen Bradley resistor

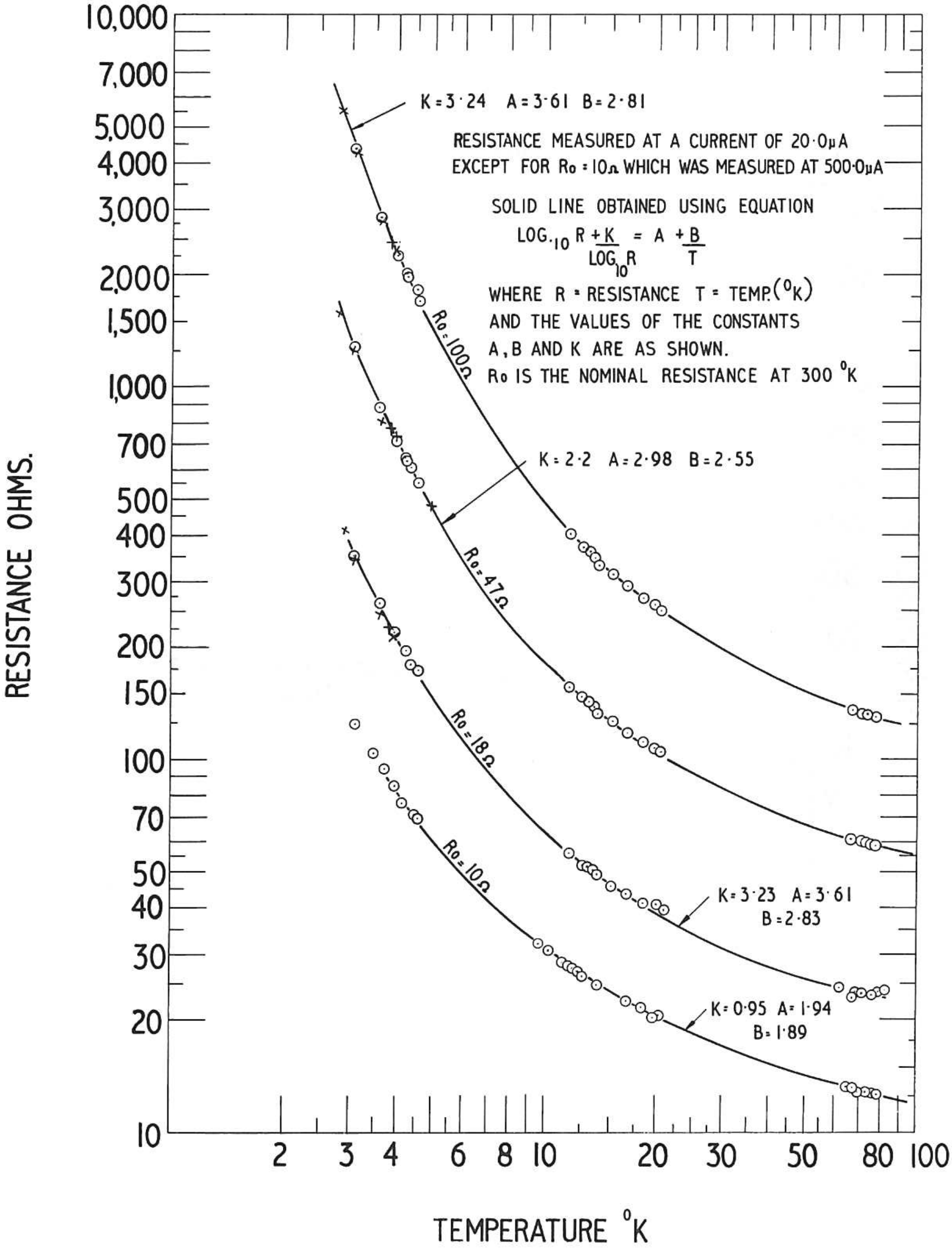

Fig.7 (CLM-R47)
The temperature characteristics of ¼ watt Allen Bradley resistors with nominal resistance values of 10, 18, 47 and 100 ohms

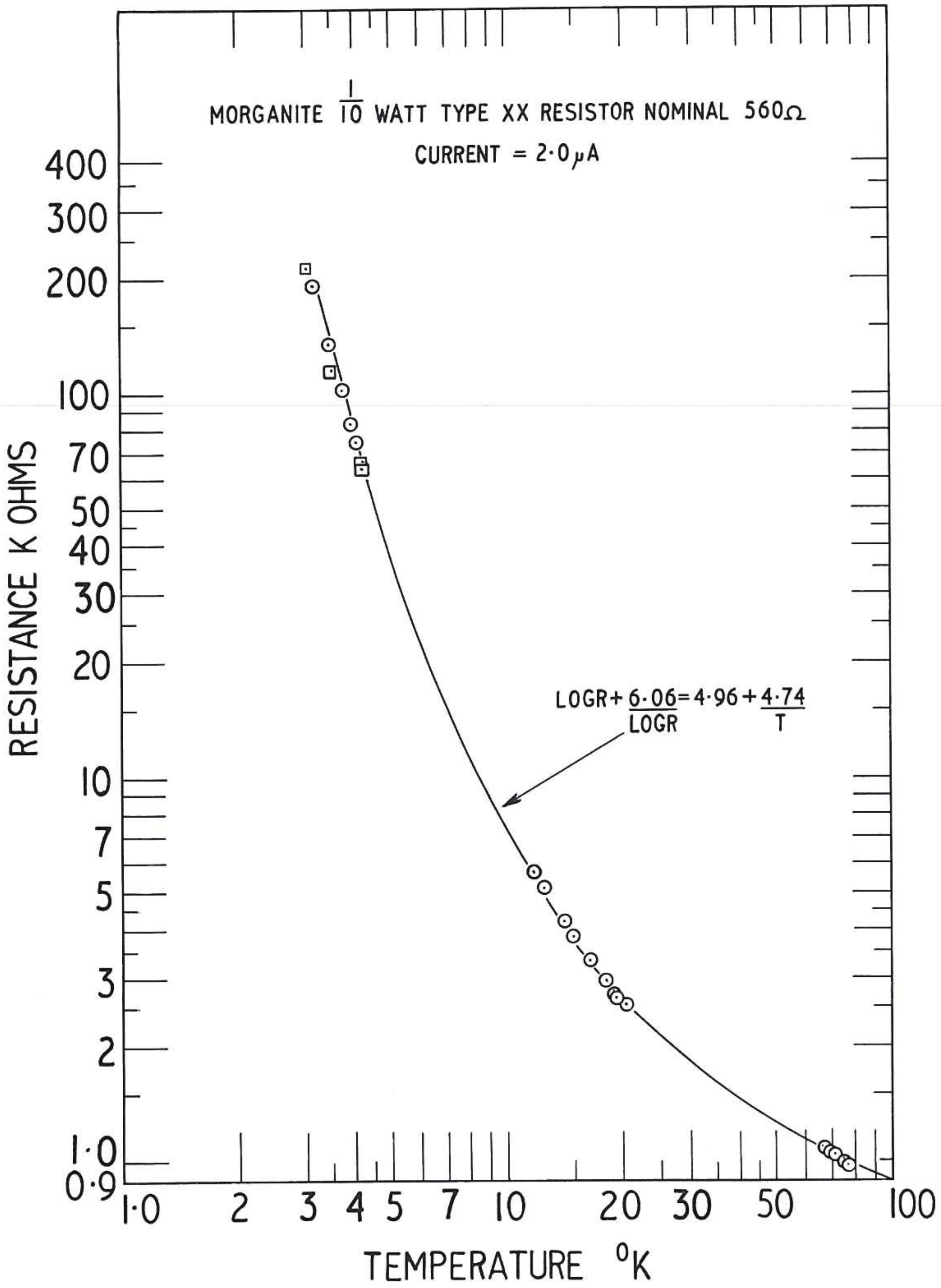

Fig.8 (CLM- R 47)
The temperature characteristic of a nominal 560 ohm 1/10 watt Morganite resistor

calibrated at the same time as resistor A and very similar results were obtained. Some of the results are given in Table 1 for comparison. In general it was found that resistors B and C differed from A by only 1% up to 30°K, and by 2% up to 90°K.

TABLE 1

Temperature	4.56	4.6	11.7	16.6	20.34	21.4	25.1	27.1	64.8	77.36	$^{\circ}$K
Resistor A	73.1	72.5	28.06	23.7	20.34	19.8	18.16	17.6	13.34	12.7	Ohms
Resistor B	72.3	71.2	27.62	23.7	20.07	19.56	17.9	17.45	13.19	12.5	Ohms
Resistor C	72.3	71.01	27.73	23.4	20.18	19.6	18.04	17.55	13.19	12.6	Ohms

The results for three other Allen Bradley ¼ watt resistors value, 18, 47 and 100 Ohms using a current of 20 μA are given in Fig.7. The solid lines drawn through the experimental points were calculated from the experimental results and will be discussed later. The results previously given for resistor A are also plotted for comparison.

Morganite(10) 1/10 watt, type XX carbon resistors were also calibrated. It was found that 10 and 100 Ohm resistors had a very small temperature coefficient and the resistance at 4°K was only 30% higher than the nominal resistance, and they were therefore unsuitable for use as thermometers. The 580 Ohm resistors, however, were found to be very sensitive and their resistance at 4°K was 140 times the nominal resistance. Three 560 Ohm resistors, (E,F,G) were then calibrated using a current of 2.0 μA, and the results obtained for resistor E are given in Fig.8.

Unlike the Allen Bradley resistors the Morganite resistors though taken from the same batch were not very consistent, and differences of up to 25% were observed at 3°K. Table 2 gives some of the results obtained.

TABLE 2

Temperature	3.19	3.48	4.3	12.6	15.0	20.39	$^{\circ}$K
Resistor E	191,950	133,650	65,075	5,103	3,825	2,581	Ohms
Resistor F	195,450	135,150	64,775	4,775	3,560	2,400	Ohms
Resistor G	141,800	101,000	51,825	4,185	3,559	2,435	Ohms

For both types of resistor the measuring current had no effect on the resistance measurements.

Four Allen Bradley resistors, value 10, 18, 47 and 100 Ohms, were placed in the small copper cylinder previously described (Fig.4) and were placed for periods of up to one hour in a liquid helium dewar. Resistance measurements were made at 20 and 50 μA. These

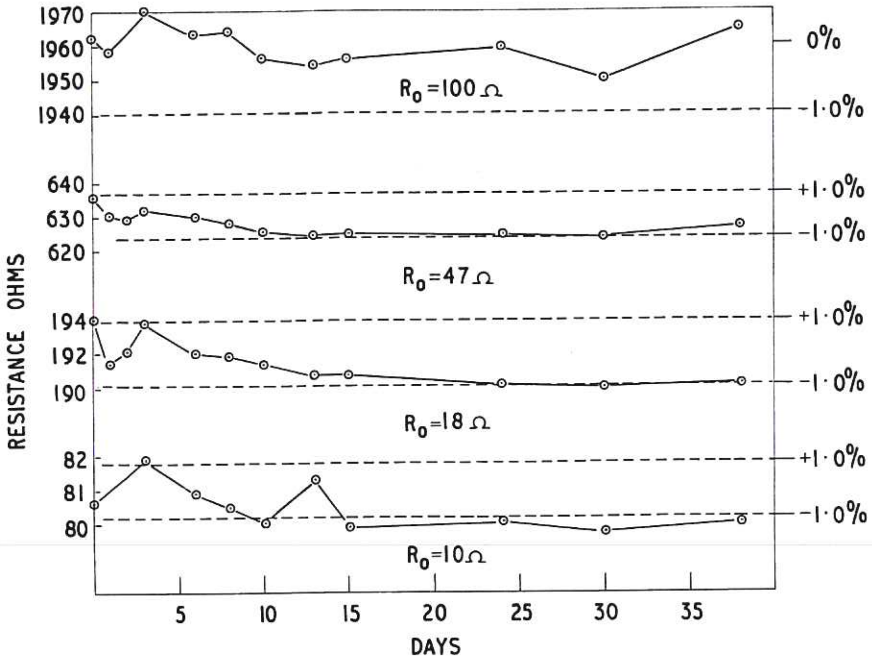

Fig.9 (CLM-R 47)
The stability of ¼ watt Allen Bradley resistors at 4.2°K

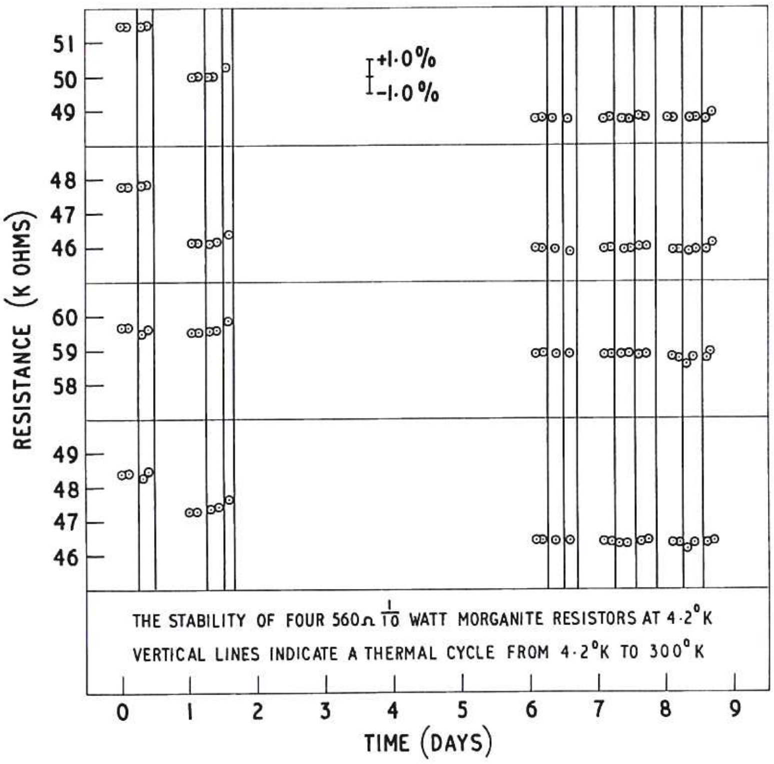

Fig.10 (CLM-R 47)
Stability of four 560 ohm, 1/10 watt Morganite resistors at 4.2°K.
Vertical lines indicate a thermal cycle from 4.2°K to 300°K

readings were repeated with the cylinder in pure liquid nitrogen. During the interval between immersions the cylinder remained at room temperature and thus the resistors received a thermal cycle for each reading taken. The results for these resistors in liquid helium are given in Fig.9. In liquid nitrogen all values gave constant resistance readings within ± 1.0%.

Four 560 Ohm Morganite resistors were then placed in a similar cylinder and resistance readings were taken in liquid helium and nitrogen. This time, however, some of the readings were not separated by thermal cycling. The liquid helium results for these resistors are given in Fig.10; here the vertical lines represent a thermal cycle to room temperature. Similar results were obtained for liquid nitrogen which also showed a decrease in resistance with time, stabilising at 97% of the initial value after 10 days of thermal cycling.

5. THERMAL CONTACT BETWEEN A RESISTOR IN VACUUM AND ITS ENVIRONMENT

There are many applications where it may be necessary to measure temperatures in a vacuum. In this case the heat transfer from the resistor to its surroundings takes place only by conduction and radiation, and near $4^{o}K$ the radiated heat is very small, since the heat radiated is proportional to T^4. Conduction therefore becomes the sole method of heat transfer and hence the efficiency of the thermal contact between the resistor and its surroundings is of prime importance.

Good thermal contact was achieved when a resistor was cemented into a close fitting hole in a copper block. The insulation of the resistor was first ground off and the exposed carbon was coated with a thin layer of cement which was allowed to dry. The resistor was then given a further coat and was pushed into the hole. Tensol perspex cement and dilute Durofix were both used successfully.

It was found that it was essential to provide an efficient thermal shunt for the 40 swg wires used for connections to the resistor, since any conducted heat produced a temperature differential between the resistor and the copper block. The shunt used consisted of a copper rod which was in contact with liquid helium and around which the fine wires connected to the resistor were wound and cemented.

6. THE EFFECT OF TEMPERATURES ABOVE 300°K

Situations may arise where it is desirable to heat apparatus containing resistance thermometers to temperatures several hundred degrees above ambient, e.g. vacuum bakeout. It is therefore important to know what effect this heating will have on the thermometer calibration.

Three 10 Ohm Allen Bradley resistors (H, I, J) were baked at temperatures up to 350°C and their subsequent resistance at room temperature and 4.2°K ($R_{4.2}$) was measured (Fig.11). When baked at temperatures below 150°C there was no appreciable change in $R_{4.2}$, but above 200°C their behaviour was erratic.

The room temperature measurements also showed a variation in resistance which followed the trend of the readings made at 4.2°K but with a smaller percentage change.

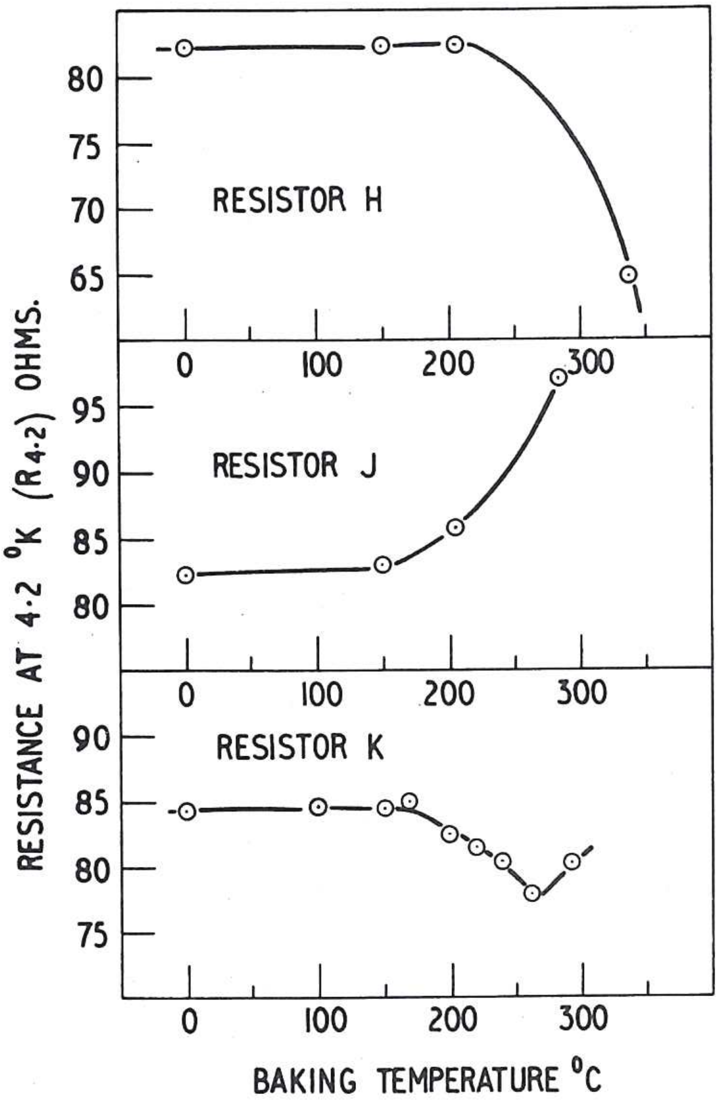

Fig.11 (CLM-R 47)
The resistance value of Allen Bradley resistors at 4.2°K after baking

Where the value of $R_{4.2}$ changed after baking, the new value was stable over a period of several days and thermal cycles. For example, after baking at 336°C the stability of resistor H, when thermally cycled five times over a period of fourteen days was equal to that of a normal 10 Ohm resistor, (Fig.9). Stable changes in resistor sensitivity have also been reported$^{(11)}$ for some unspecified carbon resistors baked at temperatures as high as 850°C.

7. DISCUSSION

It will be seen from Table 3 that the sensitivity of the Allen Bradley resistors increases with the room temperature resistance, R_0. The results of the stability test (Fig.9) also show that the stability increases with the nominal resistance. Therefore it is better to use the 100 Ohm resistors in preference to the lower values except where the final resistance reached is too high for the particular application. For example, a high impedance may be undesirable if the temperature is being displayed on a recorder.

TABLE 3

Temperature change		30° - 20°K	20° - 10°K	6° - 3°K	Resistance at 4°K
% Resistance	$R_0 = 100$	30.8%	92%	370%	22.5 R_0
change	$R_0 = 47$	23.8%	73%	280%	14.9 R_0
	$R_0 = 18$	22.2%	66%	230%	11.7 R_0
	$R_0 = 10$	20.2%	51%	175%	8.3 R_0

The Morganite 560 Ohm resistors are even more sensitive than the 100 Ohm Allen Bradley resistors (their resistance at 4°K is 143 R_0 compared with 22.5 R_0 for the 100 Ohm resistor at the same temperature). The stability of the 560 Ohm resistors is also very good, and these resistors are therefore suitable for use as low temperature thermometers. Again the high impedance at low temperatures may be a disadvantage, since the resistance is 200,000 Ohms at 3°K.

The solid lines through the experimental points in Figs.7 and 8 were obtained from the equation

$$\log_{10} R + \frac{K}{\log_{10} R} = A + \frac{B}{T} .$$

When A, B and K are constants, R is the resistance in Ohms and T the temperature in degrees absolute. The three constants were obtained from three values of R for T near 4.2°K, 20.4°K and 77.4°K which are the boiling points of helium, hydrogen and nitrogen respectively. (Considerable errors will result if the calibration points chosen are not widely spaced.) The calculated curves are in very good agreement with the experimental results and therefore spot point measurements are adequate to give a complete calibration curve.

Solving the equation is rather tedious and a computer programme has been written which evaluates the constants and produces a table of R against T for temperatures from 4^{0} to 300^{0}K. The three values of R and T are the only data required, but averaging of several experimental points is possible if the information is available.

8. CONCLUSIONS

The Allen Bradley (1/4 watt, type EB) resistors with a nominal resistance from 10 to 100 Ohms and 560 Ohms (type XX) Morganite resistors are suitable for use as thermometers in the temperature range 3^{0} - 80^{0}K. The Morganite resistors are the most sensitive and the sensitivty of the Allen Bradley resistors increases with nominal resistance. The stability of both types of resistor is good, especially after the initial thermal cycling. It is desirable therefore to 'condition' resistors by thermally cycling them between 4^{0} and 300^{0}K over a period of five days or more before calibration.

Contact between carbon resistance thermometers and solid gases will result in large errors in temperature measurement and should be avoided. Similar but smaller errors were observed with resistors in contact with liquid hydrogen.

Where temperatures are measured in a vacuum the resistor insulation should be replaced with a thin layer of insulating cement. It is also necessary to achieve intimate contact between the resistor and its environment and to thermally shunt the measurement leads.

The Allen Bradley resistors may be baked to temperatures not exceeding 150^{0}C without effecting their resistance value at 4.2^{0}K. Above this temperature the resistance changes in an unpredictable manner to a new stable value.

ACKNOWLEDGEMENTS

The author wishes to thank Mr T. Ellard for his assistance in obtaining the experimental results.

REFERENCES

1. Allen Bradley Corporation, Milwaukee, Wisconsin, U.S.A.

2. CLEMENT, J.R. and QUINNELL, E.H. The low temperature characteristics of carbon-composition thermometers. Rev. Sci. Instr., vol.23, no.5, May 1952. pp.213-216.

3. CLEMENT, J.R. and others. Carbon-composition thermometers at very low temperatures. Rev. Sci. Instr., vol.24, no.7, July 1953. pp.545-6.

4. BROWN, A., ZEMANSKY, M.W. and BOORSE, H.A. A sensitive and reproducible thermometer in the range 2°K to 20°K. Phys. Rev., vol.84, no.5, 1 December 1951. p.1050.

5. EDLOW, M.H. and PLUMB, H.H. Reproducibilities of carbon and germanium thermometers at 4.2°K. Adv. Cryog. Eng., vol.6, 1961. pp.542-547.

6. CLEMENT, J.R., LOGAN, J.K. and GAFFNEY, J. An examination of the 1948 liquid helium vapour pressure - temperature scale. U.S. Naval Research Lab., report no.NRL - 4542, April 1955. (Table reproduced in:- White, G.K. Experimental techniques in low-temperature physics. Oxford, Clarendon Press, 1959. pp.104-5.)

7. HONIG, R.E. and HOOK, H.O. Vapour pressure data for some common gases. R.C.A. Rev., vol.21, no.3, September 1960. p.360.

8. Precision dial manometer type FA - 145 manufactured by Wallace and Tiernan Ltd, Tonbridge, Kent.

9. Precision decade potentiometer and galvanometer pre-amplifier manufactured by Pye and Co. Ltd., Cambridge.

10. Resistors manufactured by Morganite Resistors Ltd., Jarrow, Co. Durham.

11. KALINKINA, I.N. Temperature dependence of the resistance of carbon thermometers. Cryogenics, vol.4, no.5, October 1964. p.327. (Trans. from Pribory i Tekhnika Eksper., no.3, 1963. p.204.)

Available from
HER MAJESTY'S STATIONERY OFFICE
49 High Holborn, London, W.C.1
423 Oxford Street, London W.1
13a Castle Street, Edinburgh 2
109 St. Mary Street, Cardiff
Brazennose Street, Manchester 2
50 Fairfax Street, Bristol 1
35 Smallbrook, Ringway, Birmingham 5
80 Chichester Street, Belfast

or through any bookseller.

Printed in England

S.O. Code No. 91-3-18-74