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CALIBRATION OF A CARBON RESISTOR THERMOMETER BETWEEN 0,3°K AND 20°K

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ABSTRACT

Old Erie resistor were calibrated, and used as secondary thermometers in the region between $0,3^{o}K$ and $20^{o}K$. A calibration method is suggested which allows a precision better than 0,3% in all the interval, after cycling to room temperature, withouth recalibration against magnetic thermometers.

RESUMO

Antigos resistores "Erie" foram calibrados, e usados como termômetros secundários na região entre $0,3^{o}K$ e $20^{o}K$. Sugerimos um método de calibração que permite uma precisão melhor do que 0,3% em todo o intervalo, mesmo após reciclagens até temperatura ambiente, sem necessidade de recalibração contra termômetros magnéticos.

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INTRODUCTION

Carbon resistors have been currently used as secondary thermometers for low temperature measurements since 1952 due mainly to the careful work of Clement and Quinnell [1,2] with Allen Bradley resistors.The main drawback of these resistors remains in the fact that their resistance increases too fast with decreasing temperature beeing of the order of meg-ohms at a few tenths of a degree. This inconvenience is also found in LAB resistors [3] and in nearly all germanium thermometers [4,5], though recently many improvements were made which allow them to go down to at least $0,05^oK$, with good reproductibility [6]. Speer resistors [7], and IRC resistors [8], have a much smaller dR/dT but must be recalibrated after cycling to room temperature, for precise measurements (better than 1%).

We needed a thermometer to use in experiments of susceptibility measurements, down to $0,3^oK$ and eventually lower, and we had a lot of resistors to experiment with. Finally, after a few preliminary essays, we found a resistor whose dR/dT was quite low and whose mechanical properties were good enough to allow it to be cut and machined easily in the most convenient forms. It was made by ERIE,of England, around 1950, and its production was discontinued, but we had a lot of them*. D.H. Howling. F.J. Darrel and E. Mendoza [9] published a paper suggesting the use of ERIE re-

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* Although the production has been discontinued a long time ago, these resistors can still be found in Brasil even in some old stores of Electronics.

sistors as primary thermometers. They used a 100 ohm resistor and obtained a R × T curve down to $0,05^{o}K$. In the region of 0,3 to $1,2^{o}K$ their curve can be seen to be very similar to ours, suggesting that the carbon mixture of their resistor could be nearly the same as the one of the resistors we used.

It would be interesting if a correlation between compositions of different resistors could be made in order to make a more effective thermometer especially for the very low temperature region.

THE THERMOMETER

The resistors are uninsulated carbon cylinders of 150 ohms nominal value 1" long by 1/4" diameter. They follow the old body-head-dot code and have radial connections of copper wire, wound and tin-soldered in each end of the cylinder.

We cut the resistor in thin slabs, 1 × 3 × 20 mm to improve thermal contact with samples and provide them with electrical connections of manganin wire (30 ohms/ft) wound at each end previously painted with Silver Micropaint SC. 13 of "Micro Circuits CO". After winding, each end was again covered with the silver paint. The resistor thus prepared was then glued in a slot practiced on the wall of an Araldite cylinder, inside of which we placed the paramagnetic salts for the magnetic measurements. The cylinder was then introduced into the cryostat for measurements.

The resistance was measured with a Wheatstone bridge feeded through a lock-in amplifier working at 155 Hz

(Princeton Appl. Res. Co. Mod. HR-8), which also detected the umbalanced signal. A Graphyrack and an oscilloscope helped to vizualize the equilibrium of temperature. Power dissipation at the resistor was kept at about 10^{-9}W ; the resistance measured was rather insensitive to current variations if the power dissipated by the resistor was kept smaller than 10^{-8}W .

THE CALIBRATION

The calibration was made in the usual way , with hydrogen vapor pressure from 14°K to 20°K and He^4 vapor pressure from 1,4°K to 4,2°K . He^3 was used to obtain temperatures down to 0,3°K but we could not afford to measure its vapor pressure due to the small quantity of gas which we have. Nevertheless no significant discrepancy was observed below the He^4 lambda point which could effect calibration. Cerous Magnesium Nitrate (CMN) was used as the magnetic thermometer from 0,3 to 1,5°K , with its magnetic susceptibility extrapolated from a Curie-law adjusted in the He^4 vapor pressure interval. Manganous Ammonium Sulphate (MAS) was employed from 4,2°K to 14°K , due to its bigger magnetic susceptibility over CMN in this region, and its Curie-law was adjusted in both H_2 and He^4 vapor pressure regions.

Temperatures between 4,2°K and 14°K were obtained by heating the MAS by means of an eletric heater, which was made of manganin wire, wound around the Araldite Cylinder enclosing the salt, and the carbon thermometer, feeded through a D.C. variable power supply. After taking each set of measurements heating the sample from $4{,}2^{o}K$ to $14^{o}K$, we took another set of measurements cooling it from $14^{o}K$ to $4{,}2^{o}K$. A slight sistematic difference was observed among magnetic susceptibilities at temperatures above $7^{o}K$ when heating and cooling. Since we could not account well for this fact, we used the arithmetic media of the susceptibilities and this is the greatest source of error in our calibration in this region.

To account for magnetic susceptibilities in the materials used in the cryostat, a system was divised which allowed the removal of the salt from inside the coils of the mutual inductance bridge. We thus used the difference of susceptibilities obtained with and without sample as the correct result for the calibration.

The error of the temperatures obtained with each primary thermometer relative to the Kelvin temperature scale was estimated to be the order given in table I, with usual error propagation.

Insert Table I

Nearly 50 R × T curves obtained over a period of one year, the majority of them in He^4 and H_2 , and about 8 in He^3 , for a total of three resistors. All the resistors were obtained from the original 150 ohm carbon cylinders and when ready had nearly 1300 ohm each.

Insert Figure 1

A complete recalibration was expected to be necessary after each cycling to room temperature, and indeed , differences as big as 60 ohms were found, at a fixed temperature, between any two $R \times T$ curves, though in average they were less than 20 ohms. Nevertheless, it was found that if we obtained an $R \times T$ curve, for instance from $0,3^{o}K$ to $20^{o}K$, then heated the thermometer to room temperature and later took another set of $R \times T$ values, the difference ΔR given by each curve at any fixed temperature could be considered quite constant. This suggested that a good calibration procedure would be to draw a graph of $R \times T$, big enough to allow good interpolation, and then, for each subsequent run, take a few vapor pressure temperatures and find the difference ΔR between these points and those of the graph. We used a graph where $0,1^{o}K = 5$ mm and 10 ohm $= 5$ mm between $4^{o}K$ and $14^{o}K$, and with the above procedure we could get an error smaller than 0,6% on temperatures, relative to the Kelvin scale. This error can be reduced to less than 0,3%, if we need more accuracy, by using more points in the calibration with MAS, and establishing a better temperature equilibrium when heating and cooling it.

Insert Figure 2

For the region below $1,2^{o}K$ we constructed a graph where $0,01^{o}K = 5$ cm and 10 ohms $= 1$ cm. By the same method we would get an error of about 0,3% on the Kelvin scale, which we think will be very difficult to reduce, mainly because temperatures obtained from Curie's Law applied to CMN are precise to within $0,001^{o}K$ (Daniels and Robinson[10]) on the Kelvin thermodynamics scale, down to $0,006^{o}K$.

In the $1,4^{o}K$ to $4,2^{o}K$ interval and in the $14^{o}K$ to $20^{o}K$ interval it is obviously better to take the temperatures directly from the vapor pressure readings. The derivative $\frac{dR}{dT}$ at $0,3^{o}K$ is of the order of 2000 ohm/^{o}K (fig. 3) and it is quite smaller than those of most thermometers now in use. Due to this it is possible to use such resistors down to very low temperatures without the need to measure too high resistances, with the inherent error that usually results.

Insert Figure 3

DISCUSSION

The important point concerning this resistor is the fact that once calibrated, the recalibration with magnetic thermometers can be avoided even after cycling to room temperature. The precision obtained, better than 0,6% in all the interval of $0,3^{o}K$ to $20^{o}K$ can be considered quite good in comparison with other carbon thermometers, and it can be further improved to about 0,3% .

It is possible that the major part of ΔR comes from a variation of contact resistance between leads, silver paint and carbon. To check this we compared two resistors, the first with the same kind of contact as the one used in the calibration, and the second, in the form of a dumbbell, with the perimeter of the contact area at least twice as big. Due to the smallest resistivity in this region the effect of contact resistance should be smaller. Both were immersed in boiling He^4 and then removed and heated to room temperature. This procedure was repeated several times and we found that the reistor with bigger contacts varied about 0,2% while the other changed up to 0,6% . It thus seems obvious that better electric contacts are desirable. Another important factor which can be deduced from this test is that the resistors immersed directly in the liquid helium had a variation quite smaller than those of the calibration . This probably happened because the resistors used in the calibration were glued inside a slot on the wall of the Araldite tube , and thus were subjected to more mechanical stress than the other two. If ultimate precision is required,better electric contacts must be devised, perhaps by electroplating the ends of the carbon with copper. Besides this, the resistor must be mounted in a way as to avoid mechanical tensions, and a very careful calibration from $4^{o}K$ to $14^{o}K$ has to be done.

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TABLE I

TEMPERATURE INTERVAL			ERROR		
14°K	to	20°K	± 0,02°K		
4,2°K	to	14°K	± 0,02°K	to	±0,07°K
1,4°K	to	4,2°K	± 0,003°K	to	±0,005°K
0,3°K	to	1,3°K	± 0,0015°K	to	±0,0030°K

FIGURE CAPTIONS

Fig. 1 - A curve of $R \times T$ for our resistor in comparison with those of some other resistors obtained from ref. 3 .

Fig. 2 - Typical behaviour of the differences ΔR between any two $R \times T$ curves.

Fig. 3 - Sensitivy of our thermometer compared with those of Fig. 1 .

Fig 1

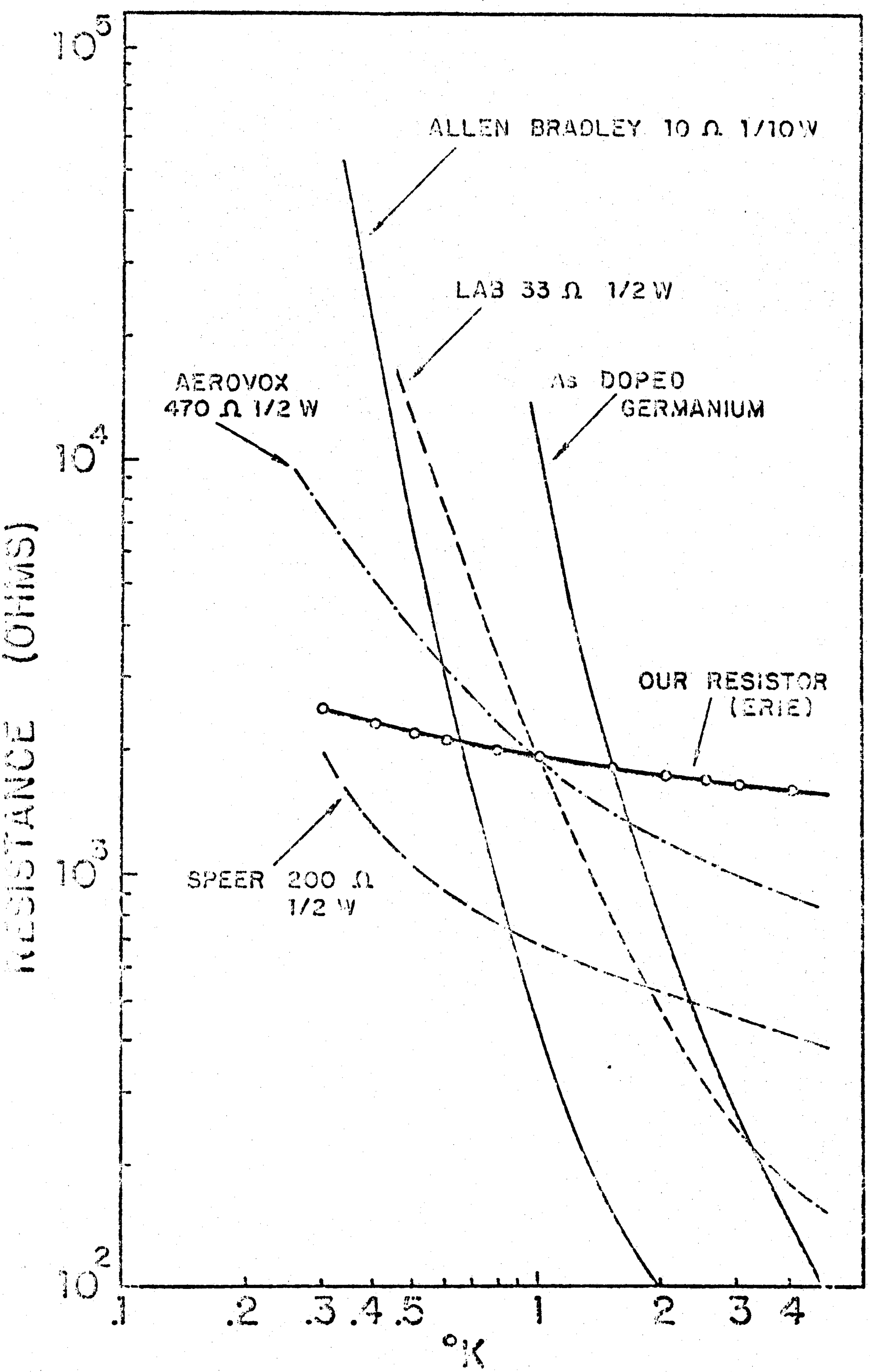
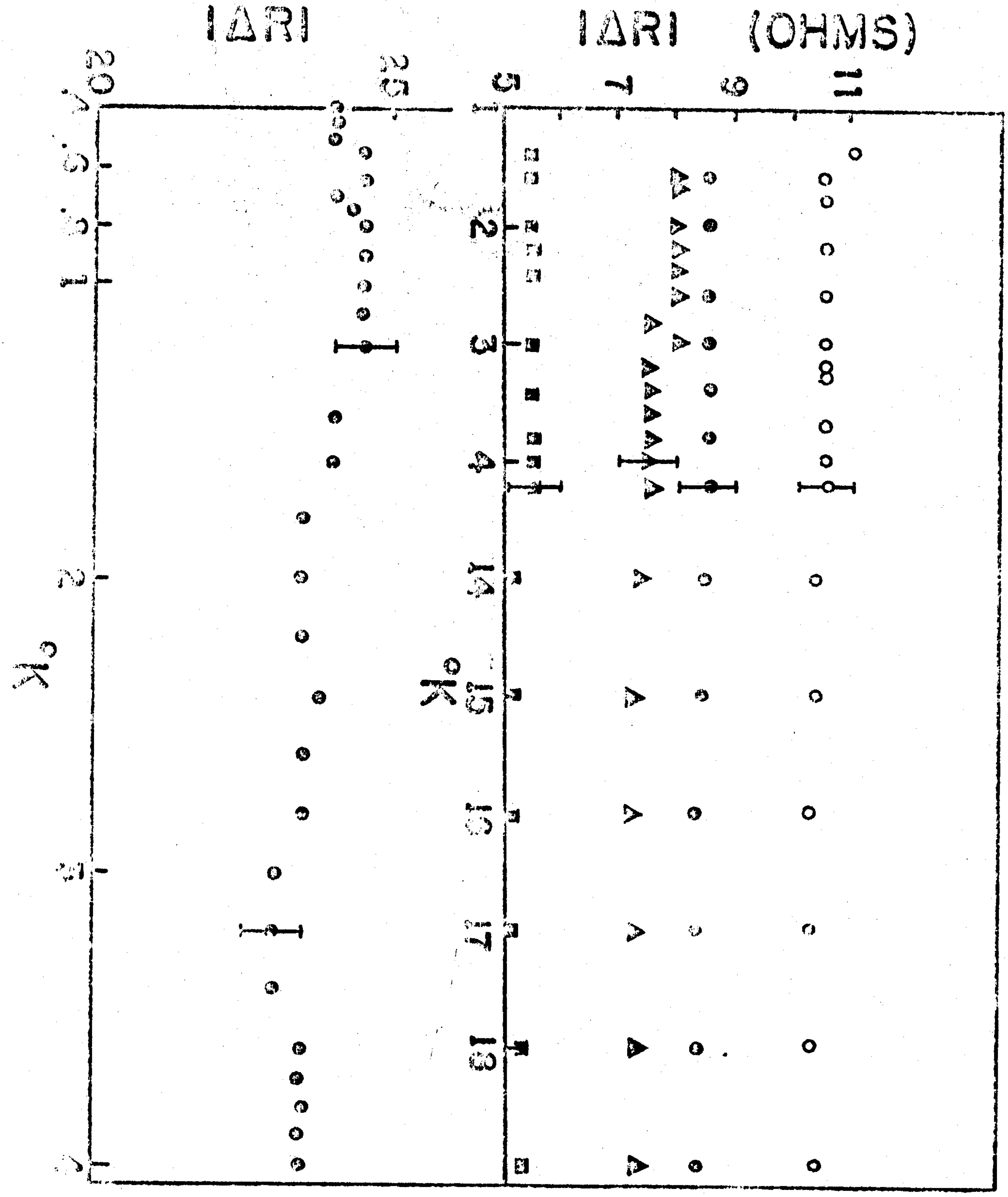
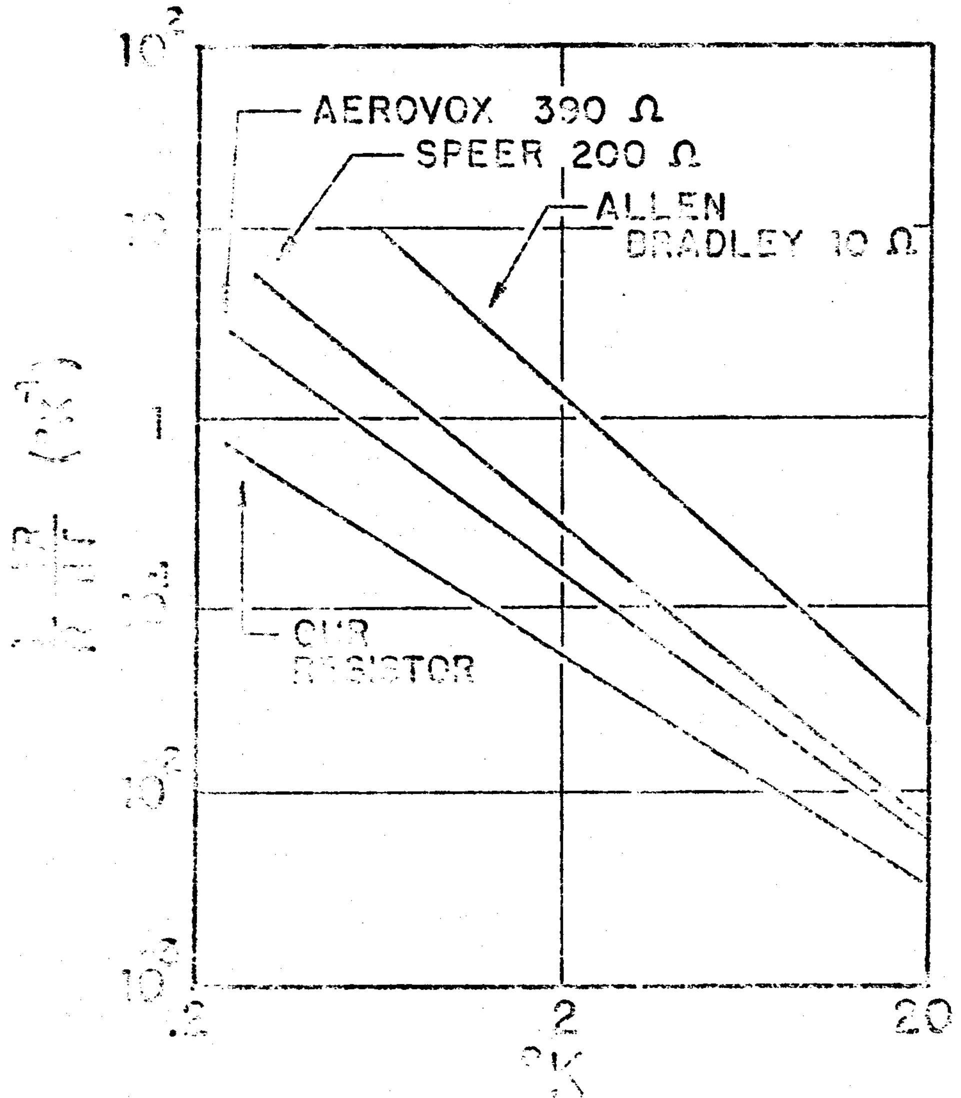

ALLEN BRADLEY 10 Ω 1/10 W
LAB 33 Ω 1/2 W
AEROVOX
470 Ω 1/2 W
As DOPED
GERMANIUM
OUR RESISTOR
(ERIE)
SPEER 200 Ω
1/2 W
RESISTANCE (OHMS)
10^5
10^4
10^3
10^2
.1
.2
.3
.4
.5
1
2
3
4
°K

fig. 2





AEROVOX 390 Ω
SPEER 200 Ω
ALLEN BRADLEY 10 Ω
OUR RESISTOR
1
2
20
°K