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THE TEMPERATURE SCALE FOR NEODYMIUM ETHYLSULFATE BELOW 1° J

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J. Blok, D. A. Shirley, and N. J. Stone

August 1965

THE TEMPERATURE SCALE FOR NEODYMIUM ETHYLSULFATE BELOW $1^{\circ}K$*

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ABSTRACT

The thermodynamic method of γ-ray heating and the statistical mechanical method of nuclear orientation were used in a complementary fashion to determine the final temperature reached on demagnetizing a single crystal of neodymium ethylsulfate from magnetic fields up to 20 kG, applied along the c axis at $1^{\circ}K$. The temperature scale so established allows nuclear orientation data to be fitted much more satisfactorily than did the old scale, removing a troublesome "S" shaped deviation. Two independent checks were made of the validity of the new scale. The nuclear magnetic moment of Ce^{137m} is again corrected. The best value, obtained from orientation in both NES and CMN lattices is 0.69 ± 0.03 nm, using $\langle r^{-3} \rangle_{4f} = 4.44$ au.

I. INTRODUCTION

Neodymium ethylsulfate (NES) was first used for orientation studies on nuclei of trivalent rare earths by Bishop et al.[1] in 1955. Subsequently many experiments have used this cooling salt, which gives alignment relative to the trigonal axis through the interaction of the crystal field with the 4f electrons. Recent work has also indicated crystal-field polarization of the closed electron shells, the effect of which on the nuclear interaction is described in terms of antishielding factors.[2,3] These experiments generally involve correlation of the angular distributions of nuclear radiation with the absolute temperature T of the system. The latter is obtained from the magnetic susceptibility of the salt (and hence the magnetic temperature T^*), using the T-T^* relation determined by Meyer.[4]

In several cases nuclear alignment data have shown systematic deviations from the form of the temperature dependence required by the known nuclear spin Hamiltonian.[5,6,7,8] In all cases it can be seen that the discrepancy would be removed if the lowest temperatures reached on demagnetization were considerably lower than indicated by Meyer, and if the T-T^* relation were altered in a consistent way at higher temperatures. Accordingly we have redetermined the NES temperature scale using the conventional adiabatic demagnetization method, measuring magnetic susceptibility and heat capacity. In addition, angular distribution measurements of the gamma radiation of Ce^{137m} aligned in NES have also been used to determine absolute temperatures, particularly at the lower end of the accessible range. These two methods were discussed recently in a stury of the cerium magnesium nitrate (CMN) temperature scale[9] where their complementary interaction in giving the most accurate scale over the whole temperature range was demonstrated.

The T-T^* relation measurement is described in Sec. II and the nuclear orientation work in Sec. III. Results are given in Sec. IV and are discussed and applied in Sec. V.

II. THE T-T* RELATION MEASUREMENT

The magnetic temperature T^* is defined in terms of the magnetic susceptibility χ by assuming that Curie's law holds, viz

$$T^* \equiv C/\chi \tag{1}$$

where C is the Curie constant. At relatively high temperatures Curie's law is obeyed:

$$\chi = C/T \tag{1'}$$

and we have $T = T^*$.

The susceptibility, and hence T^*, depends upon the sample shape and the orientation of the crystal c axis relative to the measuring field. In these experiments the susceptibility was measured using a 20 cps ac mutual inductance bridge two arms of which were counterwound matched secondary coils mounted on the glass cryostat with its axis vertical. The crystal was mounted at the center of the lower coil with its c axis horizontal, parallel to the demagnetizing field (see Fig. 1). Thus the susceptibility was measured perpendicular to the c axis. For an arbitrary crystal shape we may write

$$\chi_\perp = \frac{C}{T^*_{S\perp} - (\frac{4\pi}{3} - N)\, C} = \frac{C}{T^*_\perp} \quad . \tag{2}$$

Here N is the demagnetization factor and T^*_S is the magnetic temperature for a spherical specimen (for which N has the value $4\pi/3$). As Curie's law is strictly obeyed only for a sphere, a spherical sample was used in this work, yielding T^*_S directly and avoiding the shape correction. Hereafter we write $T^*_{S\perp}$ as T^* for simplicity of notation.

A spherical single crystal was grown over a period of 6 weeks from a small seed placed inside a spherical glass bulb. The resulting 24 mm diameter crystal was completely translucent, and was spherical to better than 1 mm in any diameter. The c axis was readily determined to within 5° by rotating the crystal between crossed polaroids to find the axis of rotational invariance of transmitted intensity. The crystal mounting is shown in Fig. 1. A small volume of chromium potassium alum slurry and a disc of compressed manganous ammonium sulfate served to reduce the heat leak to the crystal. They were mounted midway between the two secondary susceptibility coils to give a negligible contribution to the unbalance signal in the bridge. The bridge was calibrated in the liquid helium vapor pressure temperature scale range between 4.2°K and 0.96°K.

The thermodynamic determination of the temperature scale derives T from the expression

$$T = \frac{dQ}{dS} = dQ/dT^{*} \Big/ dS/dT^{*} = C^{*}/R \Big/ d(S/R)/dT^{*} \qquad (3)$$

here C^{*} is the 'magnetic' specific heat and S the entropy of the crystal. The method used to determine the various quantities was quite standard. The relation between entropy and magnetic temperature was obtained by a series of adiabatic demagnetizations from various initial H_i/T_i values; H_i being the applied field and T_i the helium bath temperature. Hull and Hull[10] calculated the relationship between S/R and H_i/T_i for an ideal spin system for the spin-1/2 case corresponding to the neodymium ion in NES at these temperatures. Although the actual entropy of the crystal is not given solely by that of the ideal spin system, Meyer has shown that all other significant contributions are field-independent. Thus, provided that each demagnetization is made from the same initial temperature, the true entropy along each adiabat differs only by an additive constant from the spin entropy calculated by Hull and Hull, and we can write

$$\frac{dS_{ideal}}{dT^*} = \frac{dS}{dT^*} \tag{4}$$

The value of dS/dT^* is thus obtained from the slope of an S_{ideal} versus T^* plot (Fig. 2), T^* being given by bridge measurements extrapolated to the instant of demagnetization.

The magnetic specific heat is obtained as a function of T^* using the gamma ray heating method,[11] taking the approximation

$$C^* = \frac{dQ}{dT^*} \cong \frac{\Delta Q}{\Delta T^*}$$

for small intervals ΔT^*. Two Pb holders containing 100 mC sources of Co^{60} were placed on a line through the specimen. Movable Pb doors on the holders permitted the NES sphere to be radiated for measured time intervals. For fixed geometry the heating period is a measure of ΔQ in arbitrary units. Extrapolating the 'normal' warming curves before and after heating to the middle of the heating period gives a measure of the change, ΔT^*, produced. A typical heating curve is shown in Fig. 3. The resulting relation between C^* and T^* is plotted in Fig. 4. As C^* is not measured absolutely, the temperature T_{meas}, obtained from Eq. (3), is only proportional to the absolute temperature T. In Fig. 5, T_{meas}/T^* is plotted as a function of T^*. Since at high temperatures in the experimental range T^* approaches T the asymptotic limit of T_{meas}/T^* gives the normalization factor T_{meas}/T. The resulting relation between T and T^* is shown in Fig. 6, the indicated errors arising primarily from the C^* measurements. The experiment is summarized in Table I.

III. THE TEMPERATURE SCALE FROM NUCLEAR ALIGNMENT OF Ce^{137m}

For temperatures lower than $1/T^* = 30$, errors in both the slope of the S/R versus T^* curve and in C^* increase rapidly, as may be seen in Figs. 2 and 4. The main reason is that T^* is now changing very slowly and is no longer a sensitive thermometric parameter. However in this lower temperature region the anisotropic angular distribution of the 255 keV isomeric transition in aligned Ce^{137} nuclei may be used as a thermometer, as it still increases rapidly with decreasing temperature.

Use of nuclear orientation in this way requires a) that the form of the spin Hamiltonian for the aligned nuclear system be known throughout the temperature range of the experiment, and b) that the adjustable parameters in this Hamiltonian be fitted accurately in the temperature region inwhich the T-T^* relation is well known. The absolute temperature scale can then be extended into the lower temperature region using the calculated relation between anisotropy and absolute temperature. In practice the former condition is not generally satisfied a priori, but may be justified by successful application of the resulting temperature scale.

Nuclear alignment of Ce^{137m} in NES has been investigated previously,[4,5] and is described by the spin Hamiltonian[6]

$$\mathcal{H} = AS_zI_z + B(S_xI_x + S_yI_y) \quad .$$

If the values of $\langle 1/r^3 \rangle$ for the Ce^{3+} ion given by Judd and Lindgren[11] are used, we have $A = 0.074\ \mu_N/I\ cm^{-1}$, $B = 0.002\ \mu_N/I\ cm^{-1}$, and $I = 11/2$. The experimental data presented here were obtained in two experiments performed independently several months apart. The earlier work was reported in Ref. 7 in which a complete account of the experimental technique is given. The second experiment

was run to obtain a more complete set of data for certain temperature ranges. The active crystal was prepared and mounted as before. After demagnetization several 15-second counts were taken with NaI(Tl) detectors at 0° and 90° to the crystal c-axis which is the axis of alignment. The counting rate was normalized to isotropic distribution by warming the crystal to the helium bath temperature. The anisotropy ϵ is defined as

$$\epsilon = 1 - W(0^\circ)/W(90^\circ)$$

where $W(\theta)$ is the normalized intensity at angle θ from the c axis. The value of ϵ for each count was calculated and an extrapolation made to the time of demagnetization. As the warm-up time of the crystal to bath temperature was several hours, this extrapolation over the first minute increases the observed anisotropy only slightly. A series of demagnetizations were made from various values of H_i/T_i; the results were in complete agreement with those of Ref. 7. The two experiments have been analyzed together.

For final temperatures above $1/T = 40$ the magnetic susceptibility thermometer was used with the new T-T^* relation to give the absolute temperature. In this region a theoretical fit was made, yielding

$$A = 0.0109(7)\ \mathrm{cm}^{-1}, \text{ assuming } B = 0.03\ A,$$

where the error estimation includes uncertainty in the T-T^* relation as well as statistical errors in the anisotropy measurements. Using this value the theoretical curve shown in Fig. 7 was used to deduce temperatures for $1/T > 40$.

Figure 8 shows a plot of $W(0)$ versus $W(90)$, the theoretical curve being for a pure M4 transition corrected for detector solid angle. The temperatures to which given points correspond are indicated. This figure gives evidence of

the internal consistency of the nuclear orientation experiment. The experiment is summarized in Table II.

IV. THE RESULTING TEMPERATURE SCALE

The results of the T-T^* determination were shown as a plot of T vs T^* in Fig. 6, and are given as T vs H_i/T_i in Fig. 9. The results obtained by Meyer are given for comparison. Figure 9 also includes the result of the nuclear alignment experiment where temperatures are calculated from the observed angular distribution using the theoretical ϵ-vs-$1/T$ curve as described in the previous section. These results are most easily discussed by considering three separate temperature ranges.

A. $1/T < 40$

The thermodynamic T-T^* relation method can give values of $1/T$ to ± 2.5 ($1/T = 40$), ± 1.5 (25), and ± 1 (15). The experimental uncertainty arises largely from the normal warming curve correction in the C^* determination. As Fig. 6 shows, T^* tends to T at high temperatures as required, and there is also agreement between the present work and that of Meyer down to $1/T = 25$. Below this the experiments differ, and if the new relation is used the fit of the nuclear alignment data to the form of the theoretical curve is greatly improved. This is seen by comparing the $1/T < 40$ region of the anisotropy versus $1/T$ curve (Fig. 7) with that of Fig. 11, Ref. 7. Accordingly the value of the nuclear orientation parameter A determined by the fit in this region was adopted.

B. $40 < 1/T < 70$

As the temperature falls below $1/T = 40$, the uncertainty in temperatures derived from the T-T^* relation rises steadily, and those obtained from nuclear alignment become the more accurate. However, down to $1/T = 70$ ($T^* = 0.028$) reasonable T-T^* measurements can be made. The values of T from the two methods are in close agreement as is shown in the last two columns of Table II. Figure 9 also shows this agreement within experimental error. The deviation of Meyer's values appears to derive largely from the S/R versus T^* curve he obtained (Fig. 2). Presuming to correct his work by some empirical adjustment of the form

$$T^*_{\text{present work}} = T^*_{\text{Meyer}} - \Delta$$

one finds a consistent value of Δ equal to 4 mdeg throughout the temperature range. However, it is difficult to see how so large a correction could be justified in terms of a demagnetization factor for his quoted specimen shape. In a further attempt to discover the source of the discrepancy Dr. Maynard Michel of this laboratory kindly checked the Nd odd-A isotopic abundances in our sample. These were found within 1% of normal abundance, whereas to explain our low values of T^* on the basis of abnormally low nuclear heat capacity requires a depletion of 35%.

C. $1/T \gtrsim 70$

In this lowest region the T-T^* relation is of little value due to large uncertainties in C^* and in the slope of the S/R vs T^* curve. Thus temperatures have been derived solely from nuclear orientation. However measurements of T^*

can be made, and as a qualitative check we include in Fig. 6 the highest $1/T$ obtained by nuclear alignment plotted against the highest $1/T^*$ measured. This point lies on a reasonable extrapolation of the T-T^* relation at higher temperatures.

To summarize this section, in the region $1/T < 40$ the T-T^* relation has been taken to measure temperature, and here the nuclear alignment thermometer is 'calibrated'. From $1/T = 40$ to 70 nuclear alignment temperatures become the more accurate, but the T-T^* measurement gives satisfactory agreement. At lower temperatures the nuclear alignment method is the only measure of temperature used. A composite relation of $1/T_f$ and $1/T_f^*$ versus H_i/T_i based on all measurements, is given in Table III.

V. VALIDITY OF THE NEW SCALE

In the previous section the temperature scale was derived entirely from the present work. Here we consider other data which serve to test the new scale. In Sec. III the only "adjustable" parameter in calibrating the nuclear alignment thermometer was the magnetic dipole moment (μ) of Ce^{137m}. The first check of the scale is afforded by comparison of this value of μ with that obtained from nuclear orientation in CMN. In Table IV we give the magnetic moments for CMN for various values of $\langle 1/r^3 \rangle_{4f}$ (as discussed in Ref. 7). These have been corrected to the amended CMN temperature scale.[7] The magnetic moments from this work are also given in Table IV. The agreement of the two sets of values indicates a high degree of consistency between the two temperature scales. Inasmuch as the moment could have been regarded as a known quantity in this work, this is also evidence for the accuracy of the T-T^* relation, because the nuclear alignment and thermodynamic values of T agree well.

A second and independent check is to use the new temperature scale in analyzing a nuclear alignment experiment for which the nuclear Hamiltonian is very different from Eq. (5). The Lu^{3+} ion has a completely filled 4f shell, and hence no magnetic hyperfine interaction, so the spin Hamiltonian is

$$\mathcal{H} = P[I_z^2 - (1/3)\, I(I+1)] \quad .$$

This pure quadrupole interaction is characterized by a linear dependence of ϵ upon 1/T for small values of ϵ. This is true also for the related quantity W(90) - W(0). In Figure 10 we show the experimental W(90) - W(0) vs 1/T plot obtained for the 208 keV transition in Hf^{177} [15] using Meyer's T-T* relation; in Figure 11 the new temperature scale has been used. The improvement in agreement of the temperature dependence with theory is evident and striking, and provides further reason for confidence in the new scale.

VI. CONCLUSION

This work was undertaken to try to explain several anomalous temperature dependencies observed in nuclear orientation experiments using NES. The new temperature scale has given satisfactory resolution of the anomaly for Ce^{137m} in the region $1/T < 40$, and for Lu^{177} in the entire temperature range. This success indicates that the new scale is a definite improvement over that which was previously available, and further demonstrates the value of nuclear orientation as an accurate thermometric technique in the near-millidegree range.

FOOTNOTES AND REFERENCES

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Table I. Thermodynamic $T-T^*$ determination for NES.

H_i/T_i kG/°K	S_{ideal}/R	T^*	C^* arbitrary units[a]	$dS/R/dT^*$	T°K	$1/T^*$	1/T °K^{-1}
2750	.638	0.0953	.0296	0.965	.095	10.5	10.4
3070	.626	0.0843	.0332	1.22	.084	11.9	11.8
3510	.612	0.0740	.0372	1.64	.071	13.5	14.1
4000	.595	0.0650	.0413	2.17	.0595	15.4	16.8
4410	.574	0.0573	.0466	2.87	.051	17.5	19.7
4820	.551	0.0510	.0532	3.84	.043	19.6	23.1
5360	.527	0.0458	.0611	5.30	.036	21.9	27.8
5830	.501	0.0415	.0702	7.30	.030	24.1	33.3
6330	.475	0.0383	.0795	9.66	.026	26.2	38.9
6900	.445	0.0355	.0901	12.66	.022	28.2	45.0
7440	.416	0.0335	.100	15.8	.020	29.8	50.5
8120	.379	0.0315	.113	20.1	.0175	31.8	56.9
9080	.333	0.0295	.130	26.6	.015	33.9	65.4
9770	.303	0.0285	.141	31.3	.014	35.1	71.2

[a]The value of the normalization constant $C' = (T'_{meas})/(T_{abs})$ is 3.15 ± 0.2.

Table II. Angular distribution of the 255-keV γ ray from Ce^{137m} aligned in NES.

W(0)*	W(90)*	ϵ(%)	1/T*	1/T from T-T*	1/T from theor. curve taking A = 0.0109 cm^{-1}
.911 ± .016	1.031 ± .012	11.6	14.4	15	
.900	1.039	13.3	16.5	18	
.766	1.117	31.4	24.0	33	
.713	1.166	38.8	26.3	39 ± 2.5	
.653	1.190	45.2	28.7	(47)	44.5
.602	1.215	50.4	29.8	(50)	49
.597	1.247	52.1	30.7	(51)	50.5
.527	1.273	58.6	31.4	(55)	56
.489	1.291	62.2	32.1	(58)	60 ± 3.5
.457	1.298	64.8	32.8	(61)	62.5
.437	1.323	67.0	33.7	(65)	65
.391	1.351	71.1	35.6	(74)	70.5
.319	1.395	77.1	37		81
.271	1.421	80.9	38		89 ± 5

*The anisotropy data were taken with 3" × 3" NaI(Tl) detectors 10 cm from the source.

Table III. Temperatures reached on demagnetization of NES.[a]

H_i/T_i (kG/°K)	2	4	6	8	10	12	14	16	18	20
$1/T_f^*$	7	15.5	24.2	29.8	33.6	35.4	36.6	37.4	37.8	38.2
$1/T_f$	7	16.5	33.5	50	64	73.5	80.5	85.5	88.5	90.5

[a]Magnetization along c axis. Here T^* is the magnetic temperature of a spherical speciman taken perpendicular to the c axis. Accuracy in T is 6%.

[b]By extrapolation.

Table IV. Magnetic moment of Ce^{137m} obtained from nuclear alignment in NES and CMN, taking various values of $\langle r^{-3} \rangle_{4f}$ for Ce^{3+}.

$\langle r^{-3} \rangle$, au	μ_{137m}, nm CMN	NES
3.64 [12]	0.87 (5)	0.81 (5)
4.74 [13]	0.67 (4)	0.62 (4)
4.44 [14]	0.72 (4)	0.66 (4)
4.44	Avg = 0.69 (3)	

Fig. 1. The apparatus. The neodymium ethylsulfate crystal (NES) was suspended in a vacuum inside a glass cryostat at $0.96^{\circ}K$ by a glass rod. A chrome alum-glycerol slurry (CA) and a pressed manganous ammonium sulfate pill (MAS) also cooled in the stray field of the magnet pole tips (P). Magnetization occurred along the c axis of the NES crystal, denoted by arrow. Inductance coils (c) were used to determine T^*. Compensating coils and coolant baths are not shown.

Fig. 2. Magnetic spin entropy of the lowest doublet in neodymium ethylsulfate versus the magnetic temperature, reached on demagnetization, of a spherical single crystal, as determined perpendicular to the c axis. Meyer's results are denoted by squares.

Fig. 3. Heating curve, showing foredrifts and afterdrifts, for a typical heat capacity run.

Fig. 4. Variation of the "magnetic temperature" heat capacity with magnetic temperature, for a spherical single crystal of NES, with susceptibility measured perpendicular to the c axis (note that this affects both C^* and T^*). Here the C^* measurements are only relative. Vertical bar denotes that only measurements for $T^* > 0.028^{\circ}$ were used to determine the temperature scale.

Fig. 5. Ratio of absolute temperature, as determined by heating experiments, to T^*. This ratio, extrapolated to high temperatures, was used to determine the scale factor for the absolute temperature of NES. For clarity the ratio was normalized to unity in this figure.

Fig. 6. The T-T^* relation for NES, with susceptibility measured perpendicular to the c axis of a spherical specimen, as determined from heating experiments alone. At the lowest temperatures T^* becomes a very insensitive index of temperature. At $0.01^{\circ}K$, dT/dT^* is approximately 20. For a non-spherical specimen any errors in shape (demagnetizing factor) correction would be very harmful. Meyer's results are shown as squares.

Fig. 7. Anisotropy of the 255-keV γ ray of Ce^{137m} oriented in NES versus reciprocal absolute temperature. A theoretical curve was fitted to the data for $T > 0.025^{\circ}K$, using the new T-T^* relation, thus fixing the hyperfine structure parameter A. For $T < 0.025^{\circ}K$, the temperature scale was deduced from the anisotropy.

Fig. 8. Intensities of the 255-keV γ ray from Ce^{137m} oriented in NES at 0° and 90° from the c axis. Open circles represent data from Ref. 7. Filled circles are taken from the more recent run. Curve is the theoretical relationship for the Ce^{137m} decay sequence and the given spin Hamiltonian. Straight line represents the angular distribution given by $1 + A_2P_2(\cos\theta)$.

Fig. 9. Reciprocal temperature reached on demagnetization along crystalline c axis versus initial H/T, for NES. Open circles represent temperatures determined by nuclear orientation, closed circles those determined by the heating method. Triangles are Meyer's results. Meyer's data and ours deviate for $1/T > 30$, while for $1/T > 40$ our T-T^* data deviate somewhat from the orientation data, remaining in acceptable agreement with, but accompanied by larger errors than the latter to $1/T = 70$. For lower temperatures the T-T^* data are very unreliable and uncertain, but the orientation data have an error of only about 6% to $1/T = 90$.

Fig. 10. Temperature dependence of the function $W(90)-W(0)$ for the 208 keV γ ray following the decay of Lu^{177} oriented in NES, using the temperature scale due to Meyer. Systematic "S" deviation characteristic of several nuclear orientation experiments in NES is clearly shown.

Fig. 11. Same data shown in Fig. 10, but using the temperature scale developed in this work. Excellent agreement with theoretical curve is evident.

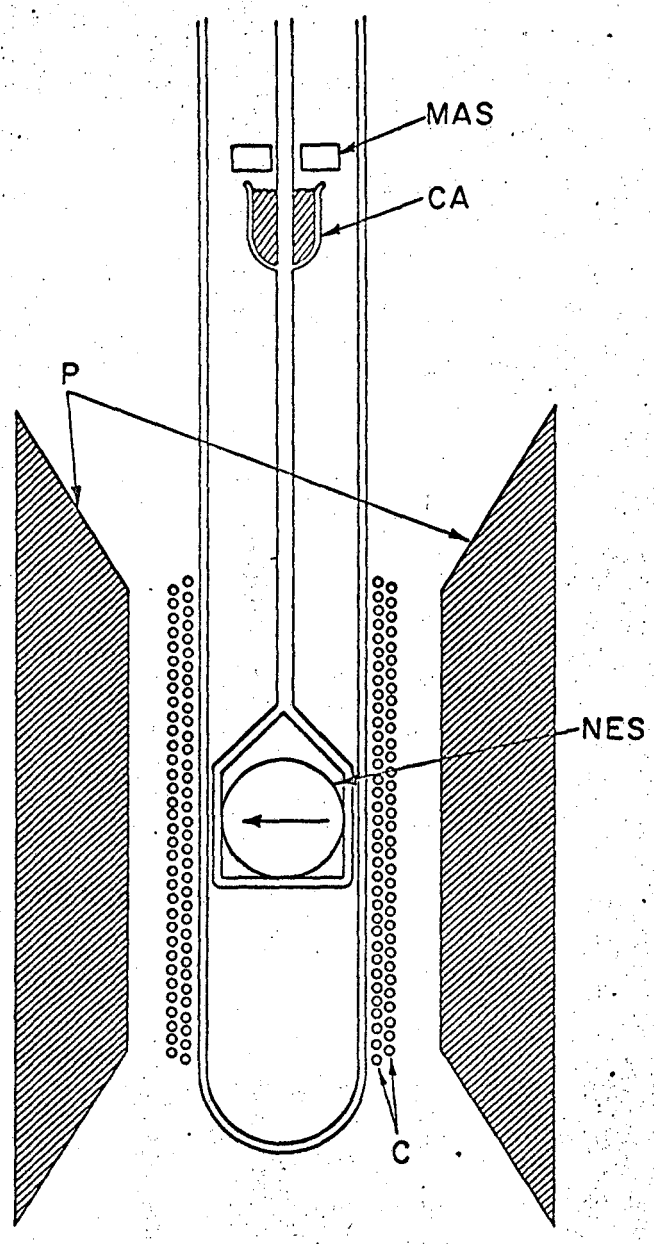

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Fig. 1

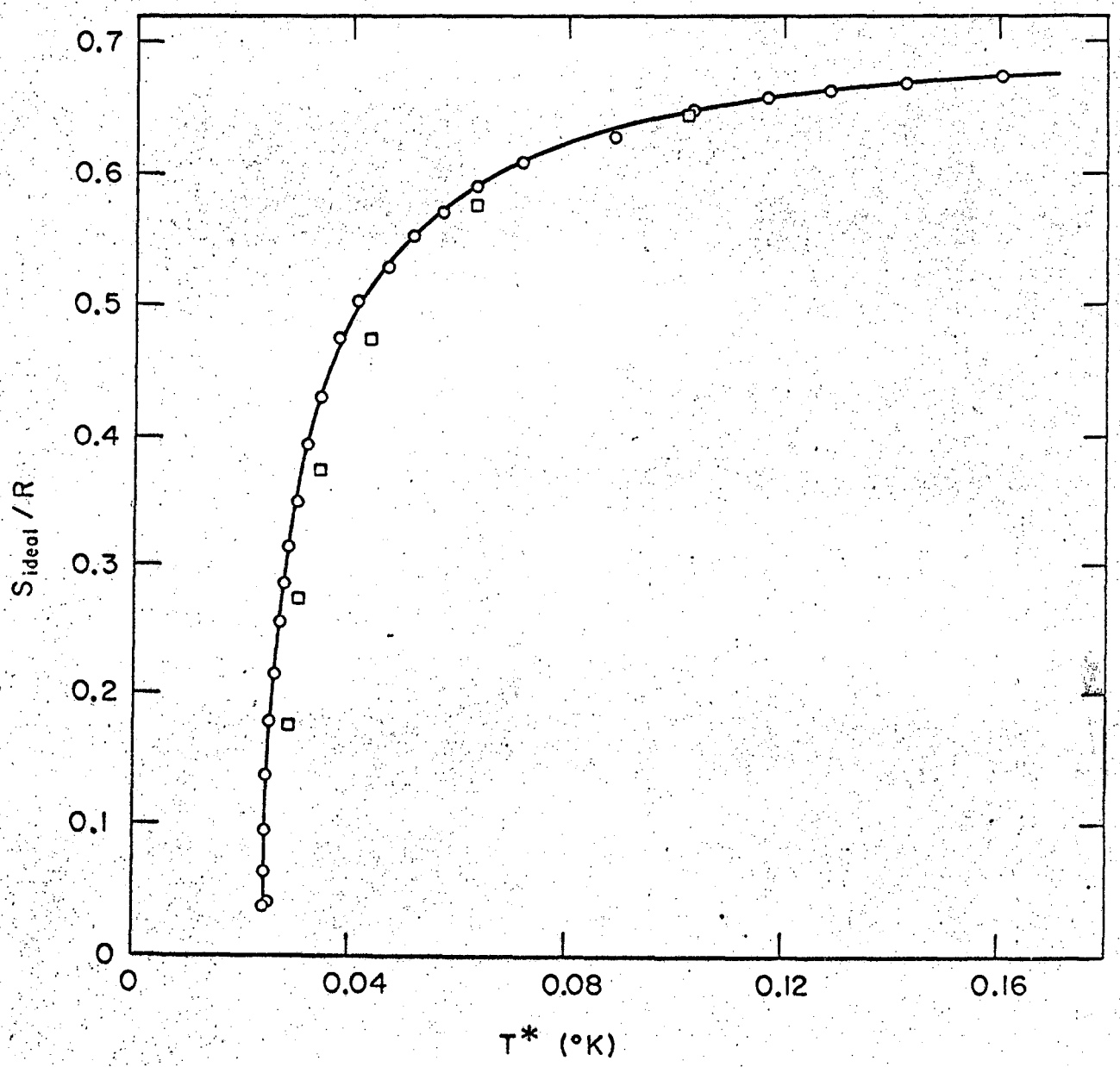

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Fig. 2

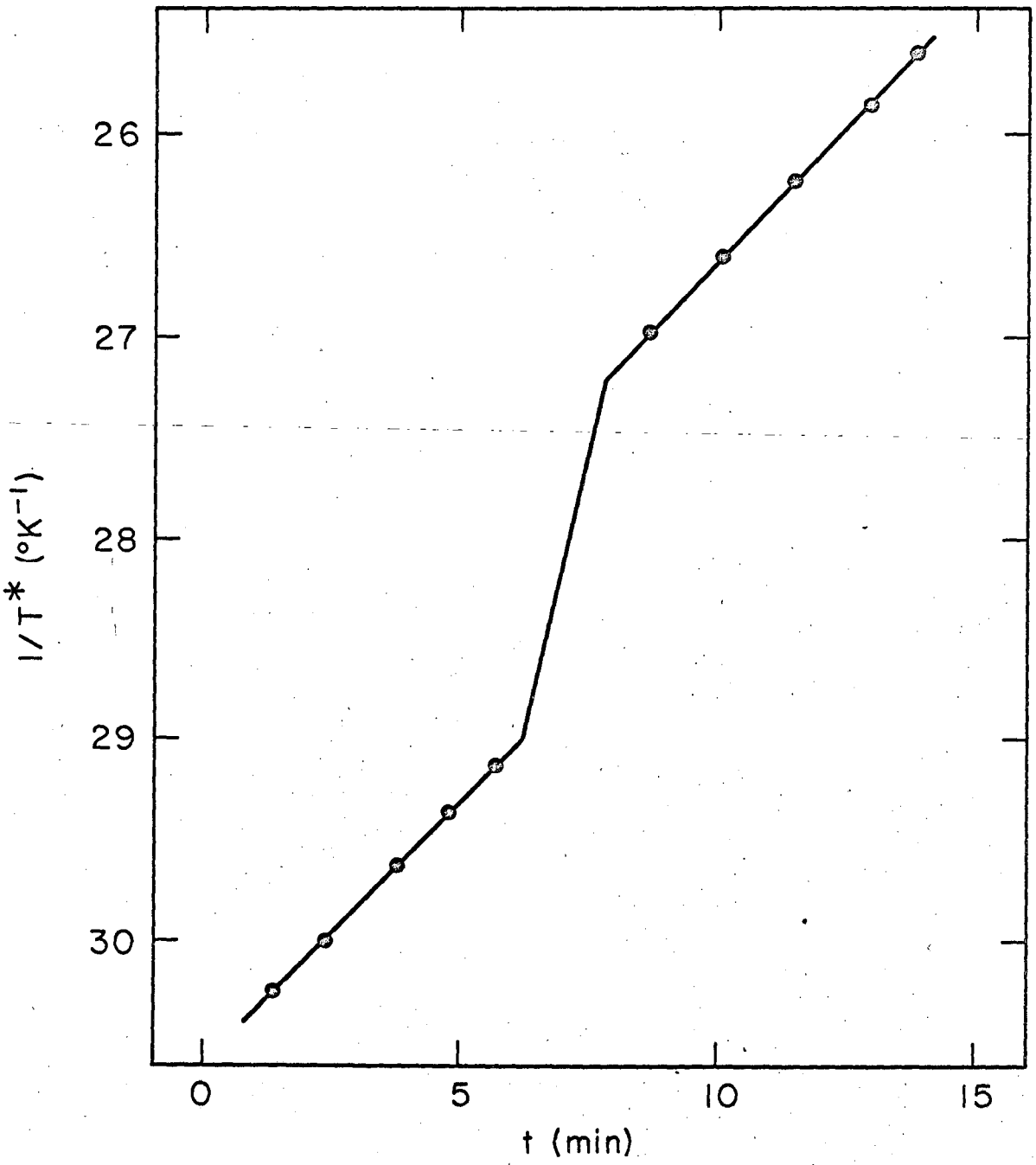

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Fig. 3

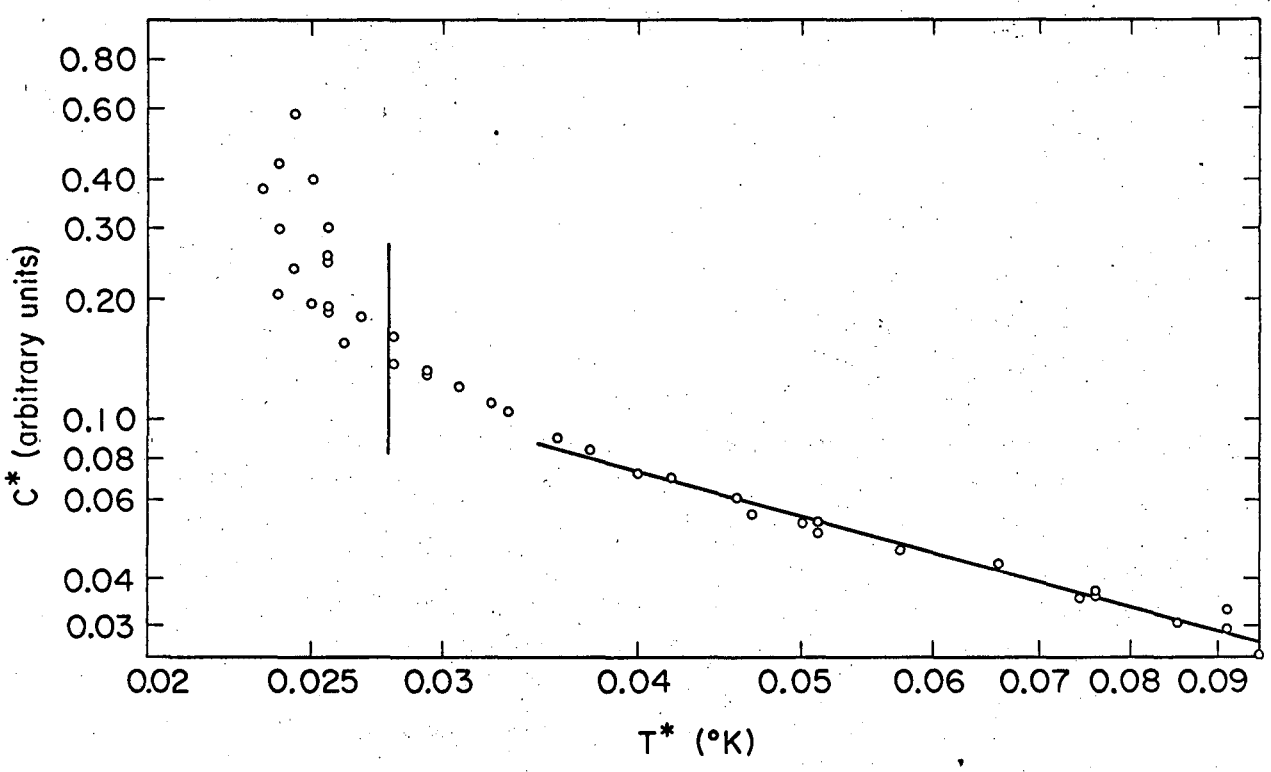

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Fig. 4

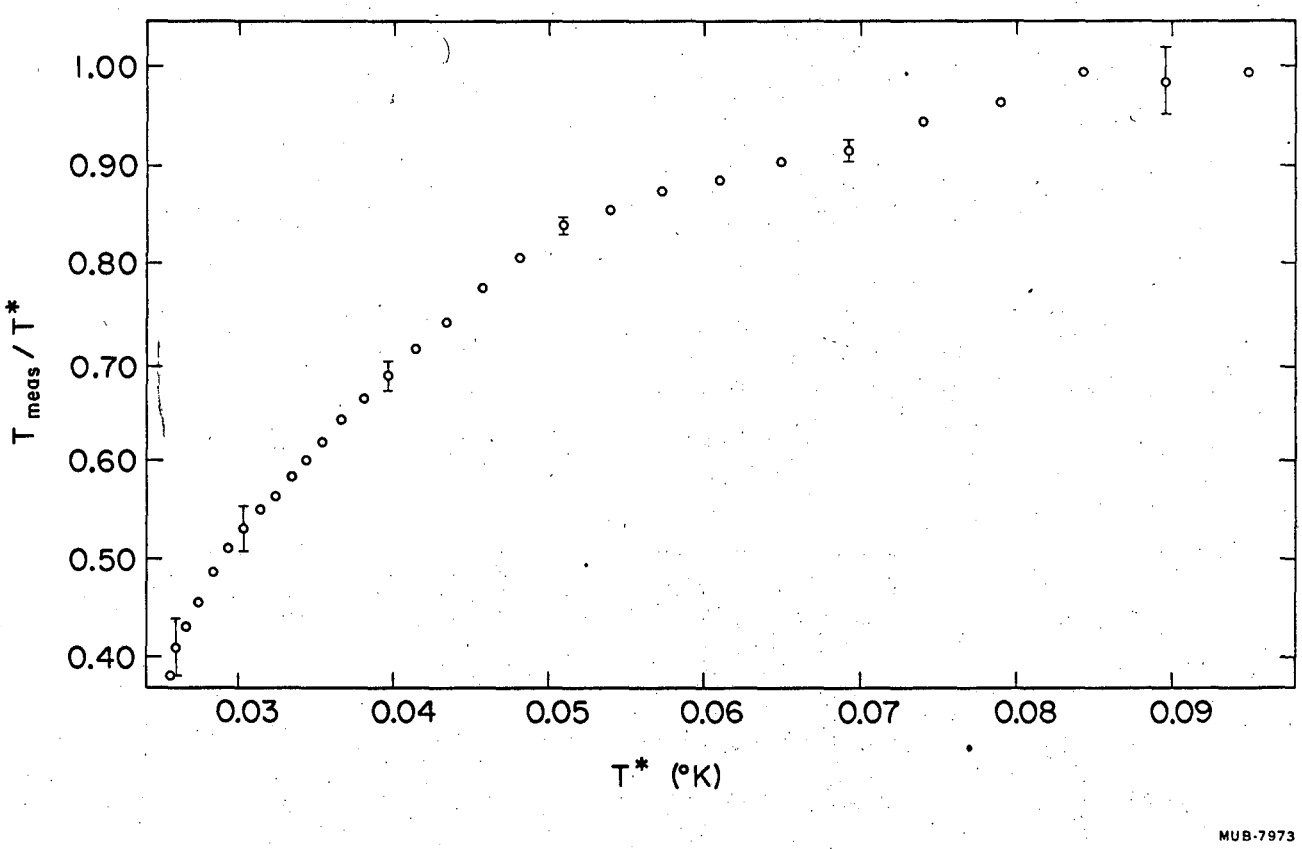
1.00
0.90
0.80
0.70
0.60
0.50
0.40
T meas / T*
0.03
0.04
0.05
0.06
0.07
0.08
0.09
T* (°K)
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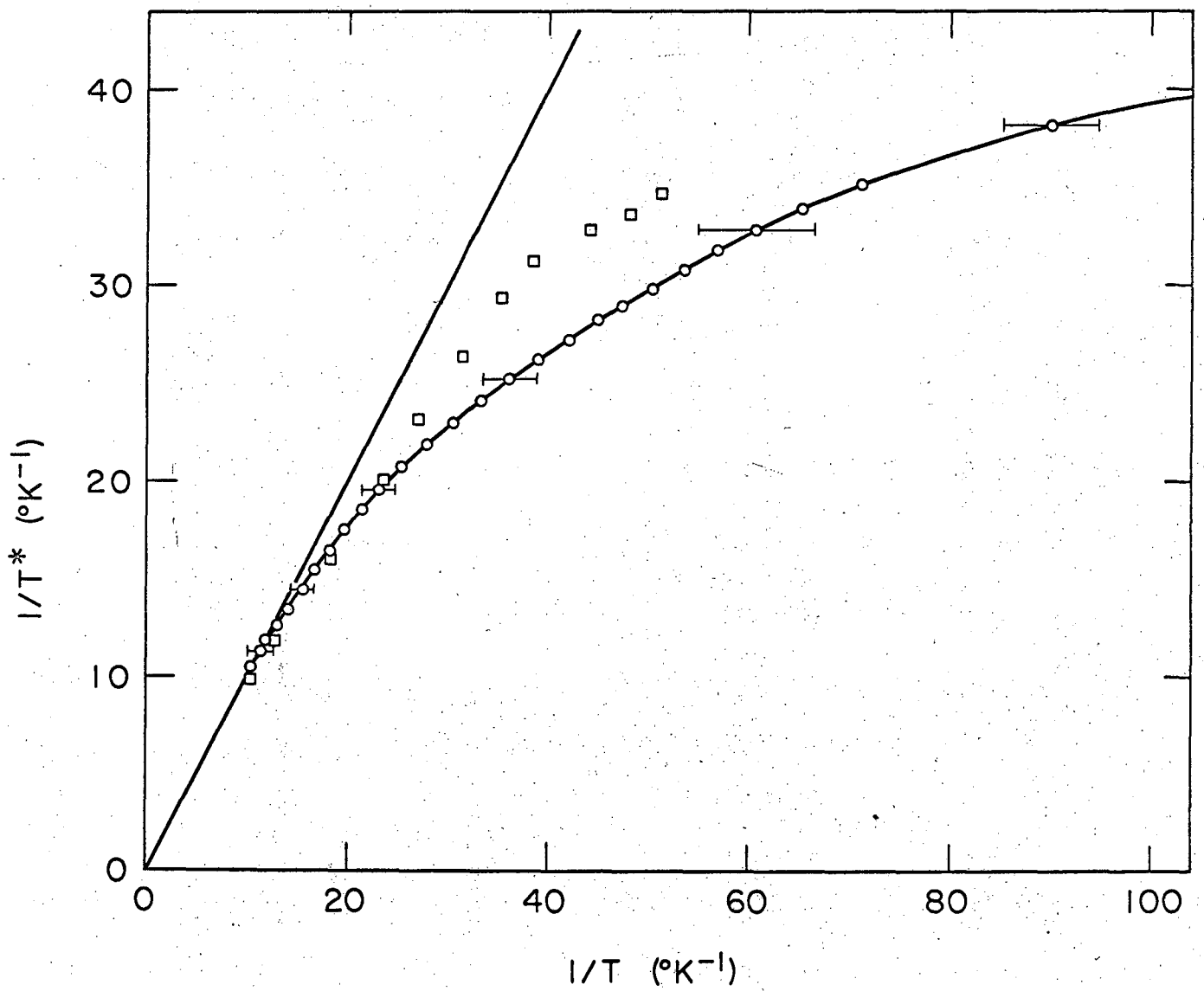
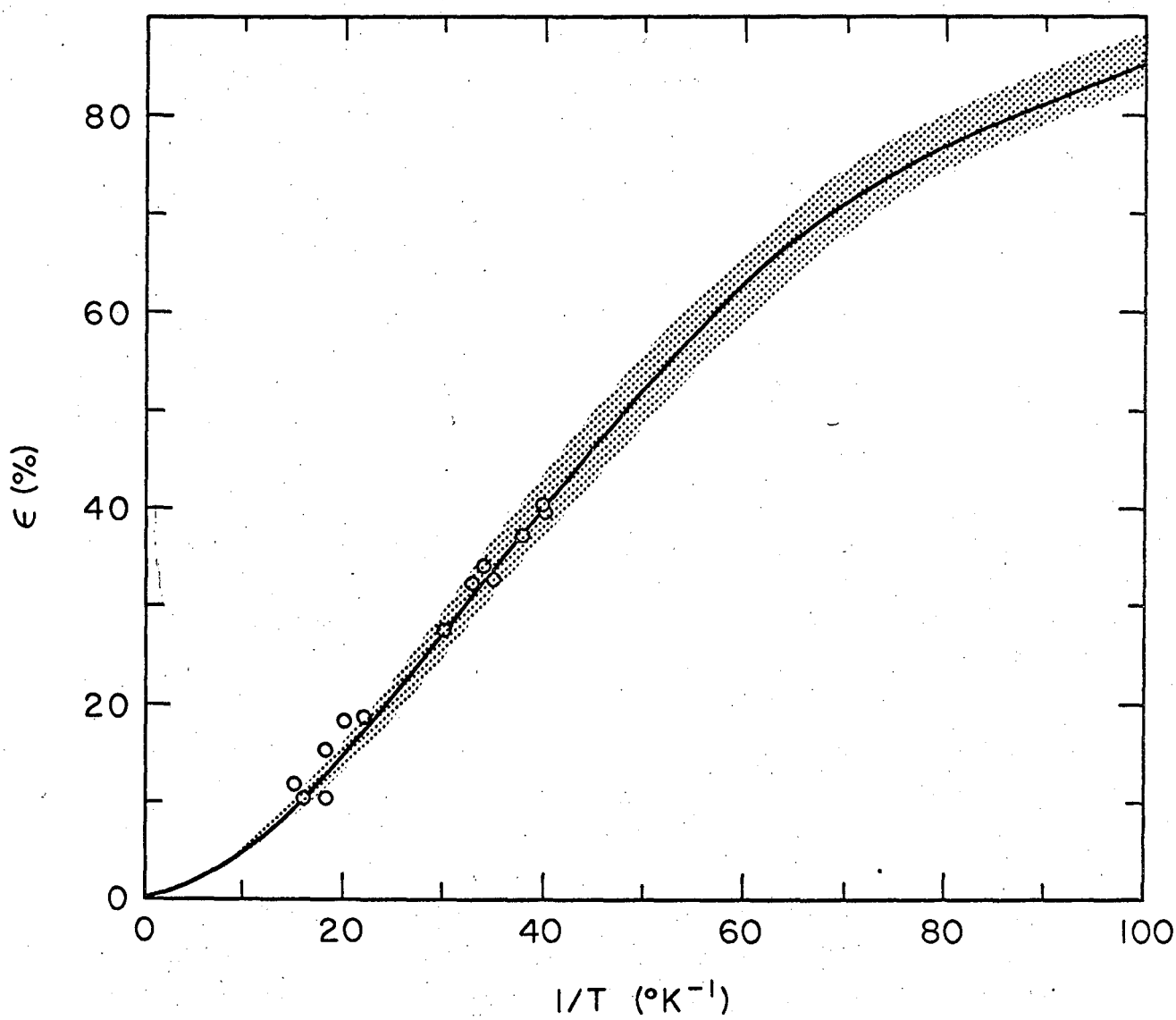
Fig. 5



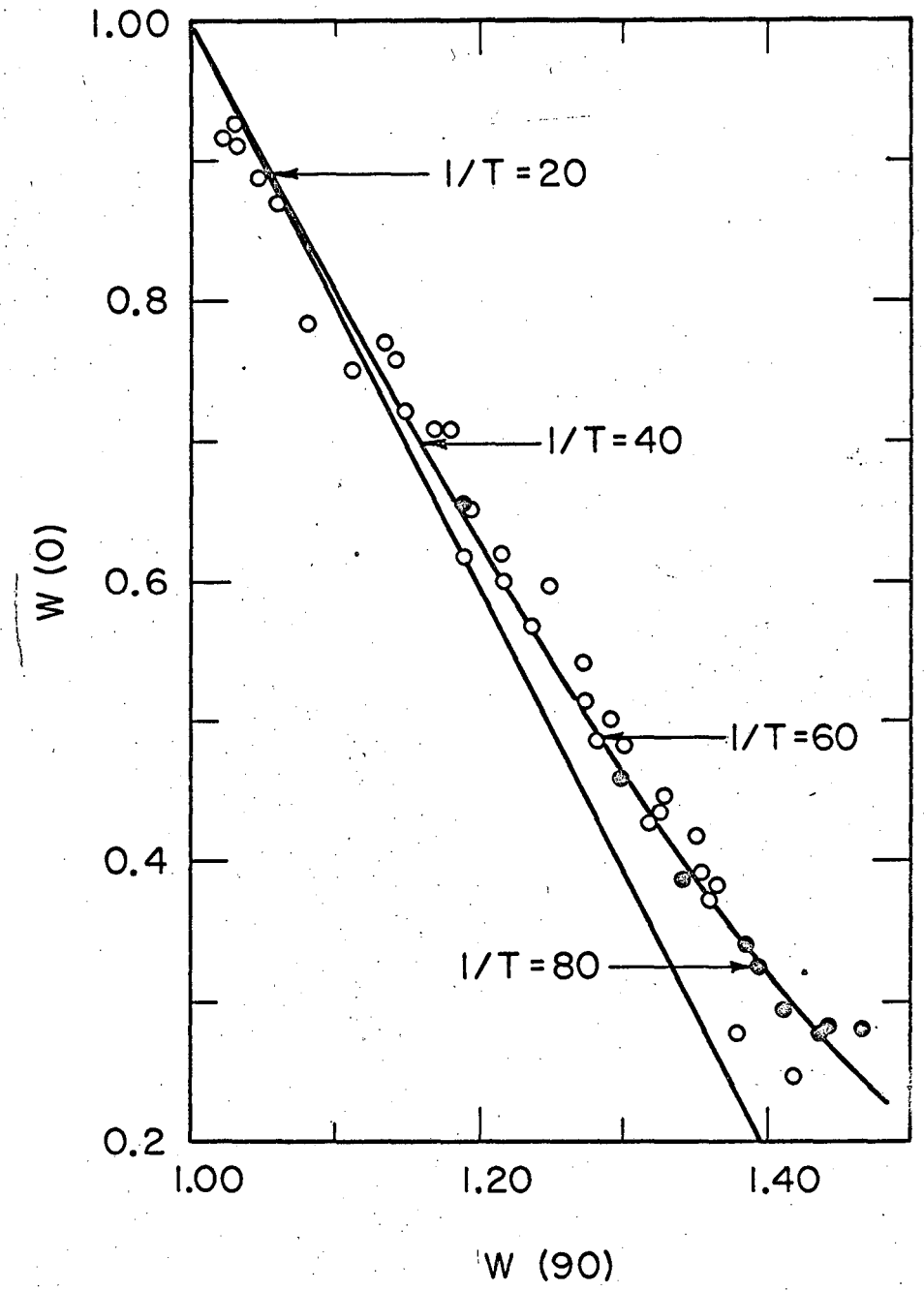
MU-36424

Fig. 6



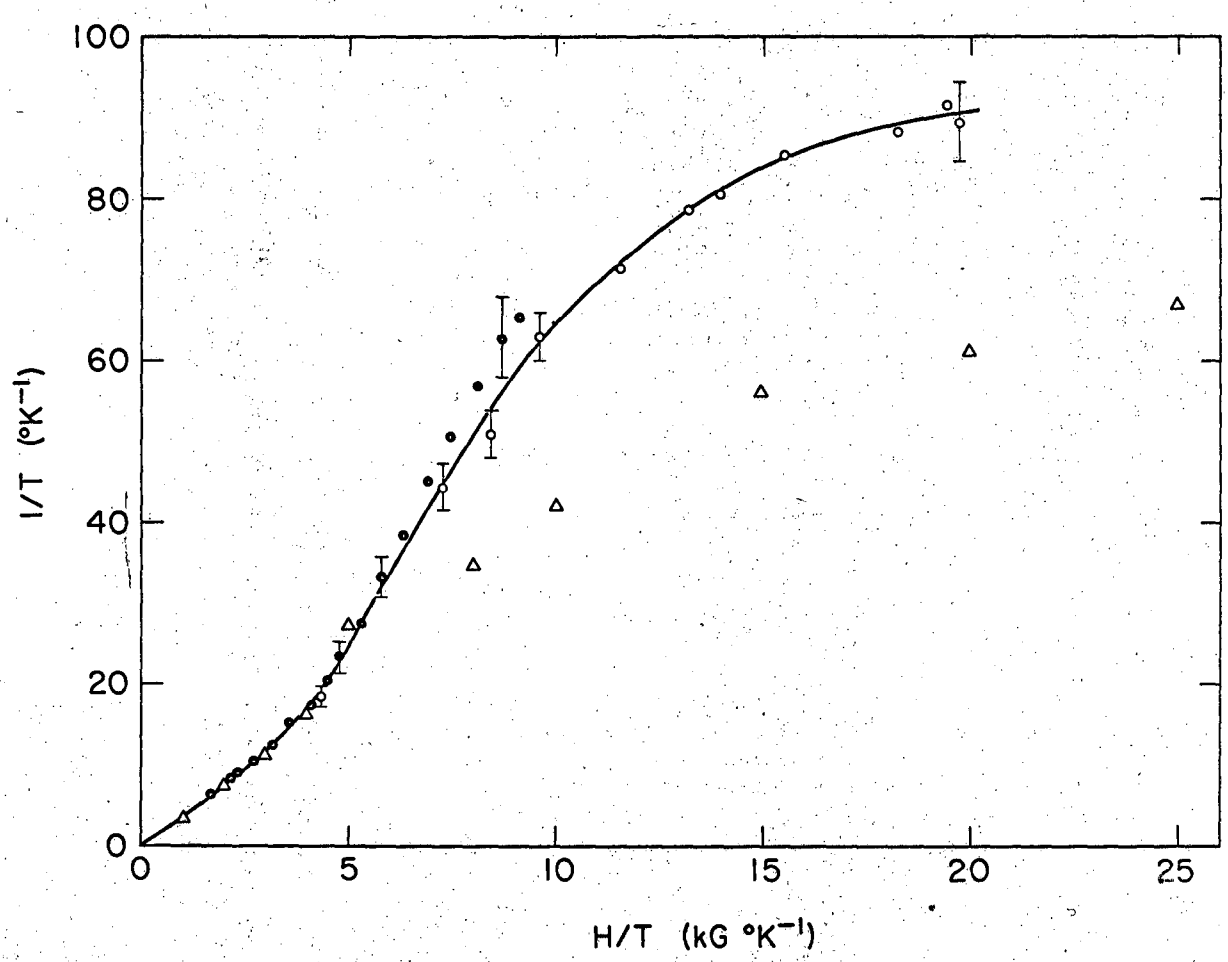
MU-36425

Fig. 7



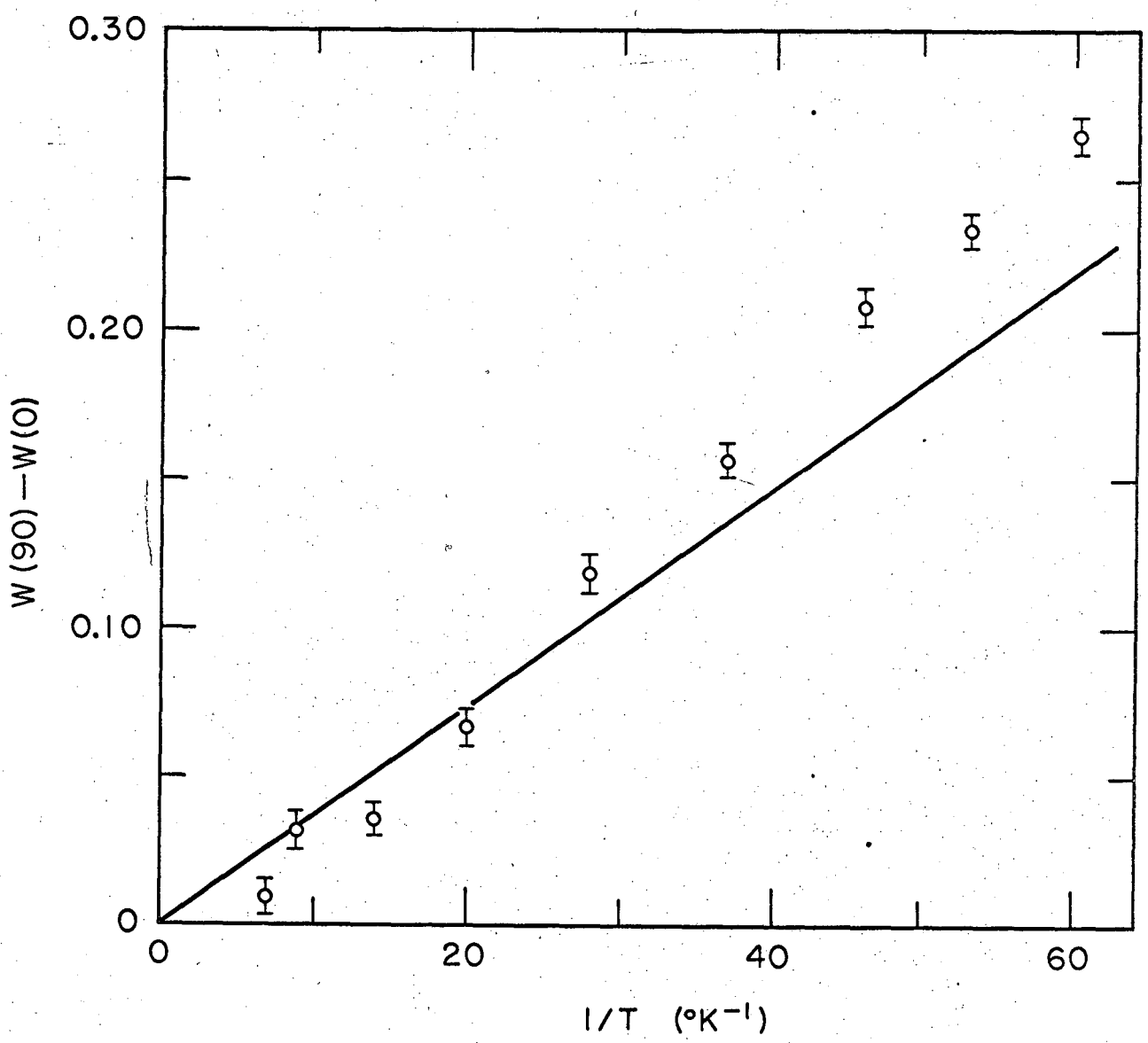
MUB-7974

Fig. 8



MUB-7975

Fig. 9



MUB-7976

Fig. 10

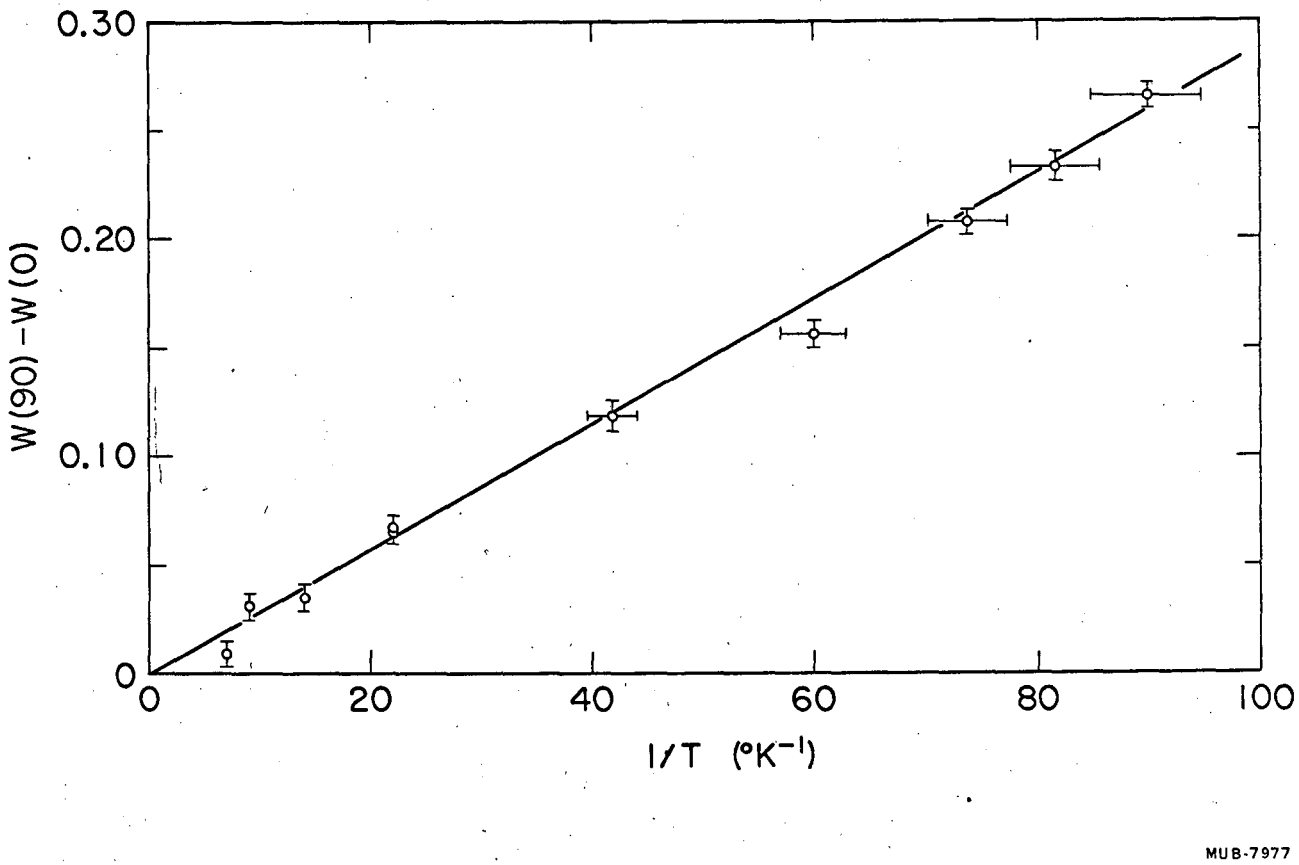


Fig. 11

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