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Use of Fusible Materials

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THERMAL CONTROL BY USE OF FUSIBLE MATERIAL

(NAS 8-11163)

FOURTH MONTHLY PROGRESS REPORT

8 July 1964

Summary

During this reporting period work has continued on the thermal analysis and on determination of thermophysical properties of the selected materials. A promising apparatus for measuring thermal conductivity of liquids is described.

Some interesting property data was obtained on normal paraffin and is discussed in this text.

Octadecane was received from the vendor on 1 July, test work was not performed on this material during this period.

Thermal Analysis

Work was continued on Parts 1 and 3 of the analysis as described in last month's report. Emphasis was placed on Part 1 which is essentially complete.

Part 1 Steady State Performance of Radiative Fin

The prediction of radiative fin's temperature distribution was checked by two analytical methods as presented in last month's report. The hand calculation analytical technique based on the article by J.W. Tatom, ARS Journal, January 1960, was selected in preference to the digital computer technique for this program because of the small number of fins required to analyze. The results of this analysis are presented in Figures 1 thru 4.

The fin steady state temperature distribution, Figure 3, and fin heat rejection, Figure 4, are the input parameters required for Part 3 of the analysis.

Figure 1 compares the selected fin thickness, .05 inch, with the theoretical minimal weight thickness for the three selected fin lengths (4", 8" and 12")

FIN THICKNESS SELECTION & COMPARISON
(FLAT PLATE RADIATING FROM ONE SIDE ONLY)

OPTIMUM WEIGHT CONFIGURATION
(REF. ASE TR-61-80, PART I)

ENVIRONMENTAL PARAMETER

$\frac{q_{in}}{q_{out}}$ = EXTERNAL RADIATION TO FIN / RADIATION FROM FIN

FIN EFFECTIVENESS

Ω = RADIATION FROM FIN / RADIATION FROM [illegible]

$q_{in} = 0$, Ω = .57, FOR MINIMUM WEIGHT [illegible]

$q_{in} = .5 q_{out}$, Ω = .31, FOR MINIMUM [illegible]

SELECTED FIN THICKNESS FOR STUDY OF RADIATOR MOUNTED HEAT SOURCES (ELECTRONIC EQUIPMENT)

= .05 INCHES

ϵ_{in} = .7

K_1 = 120 BTU/HR FT °F

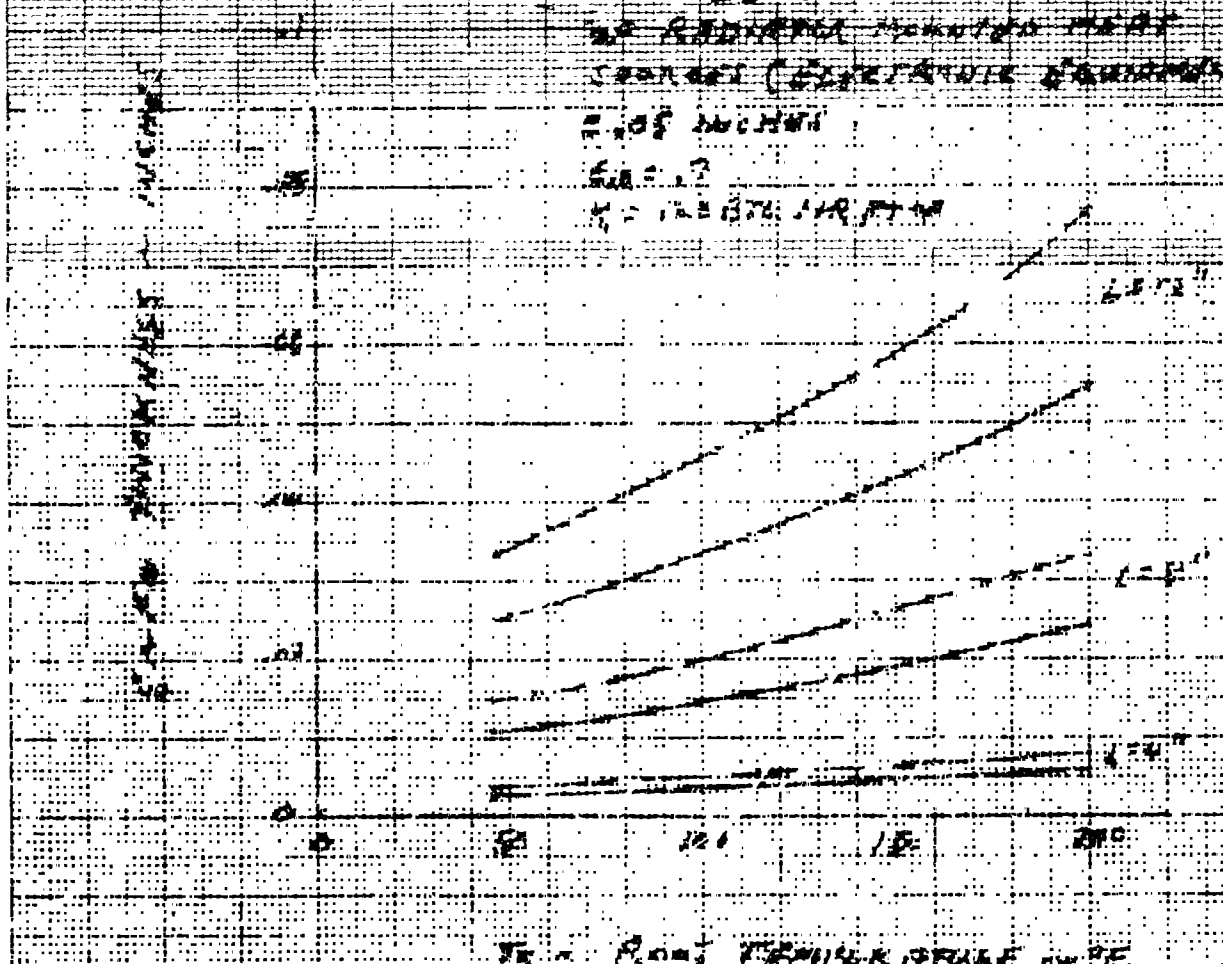

FIG. 1

K+E 10 X 10 TO THE CENTIMETER 46 1513
18 X 25 CM MADE IN U.S.A.
KEUFFEL & ESSER CO.

that were analyzed. The theoretical minimum weight thickness for the selected fin lengths are apparently below structural requirements for a spacecraft's exterior skin with electronic gear directly mounted and, therefore, the .05 inch thickness was selected. The assumed fin material is aluminum with a mean thermal conductivity of 120 BTU/HR-FT-°F and a surface coating having an infrared emissivity of 0.90. Both of these heat transfer properties are representative of spacecraft materials used.

Figure 2 presents the resulting radiating fin's tip temperature as a function of fin length and root temperature. The four root temperatures selected are the melting points of the selected fusible materials. By using these melt temperatures as a starting point for the transient analysis, both the steady state fin temperature distribution and heat rejection capability can be predicted. It should be noted that the highest root temperature analyzed is 115°F, which is the measured melt temperature of the purchased Camphene, rather than 122°F stated in the literature.

Figure 3-a, -b, -c and -d presents fin temperature distribution for fin lengths of 4, 8 and 12 inches with the above root temperatures. Including a fusible material under the fin will not significantly effect the longitudinal steady state temperature distribution with the fusible material below the melt point. This is because the ratio of thermal conductivity of aluminum to fusible material is:

$$\frac{K_{al}}{K_{f.mat.}} \cong \frac{100}{.1} = 10^3$$

and the thickness ratio will be less than:

$$\frac{al}{f.mat.} < \frac{.05}{.5} = .1$$

or the overall thermal resistance:

$$\frac{R_{al}}{R_{f.mat.}} \cong \frac{1}{100}$$

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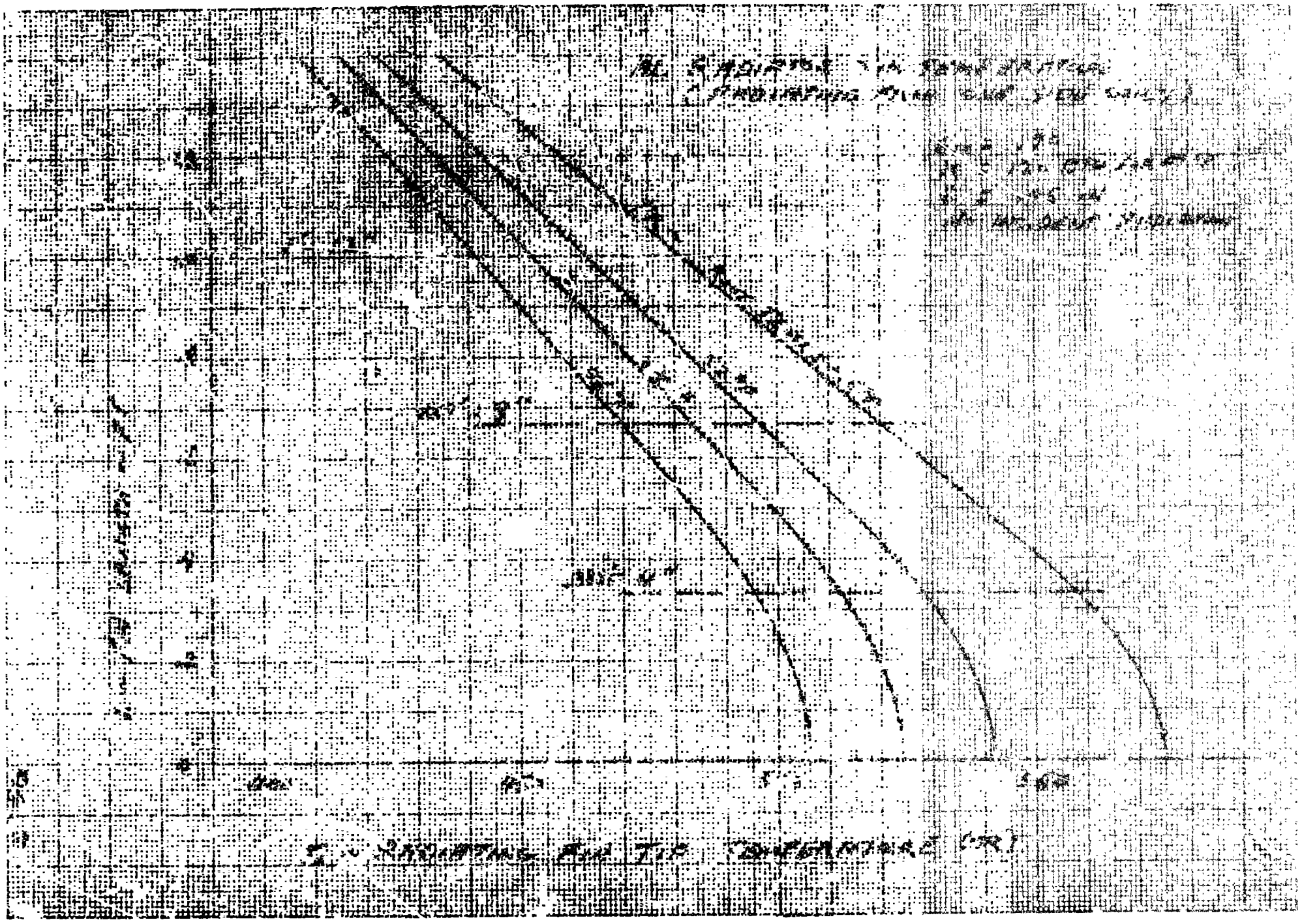

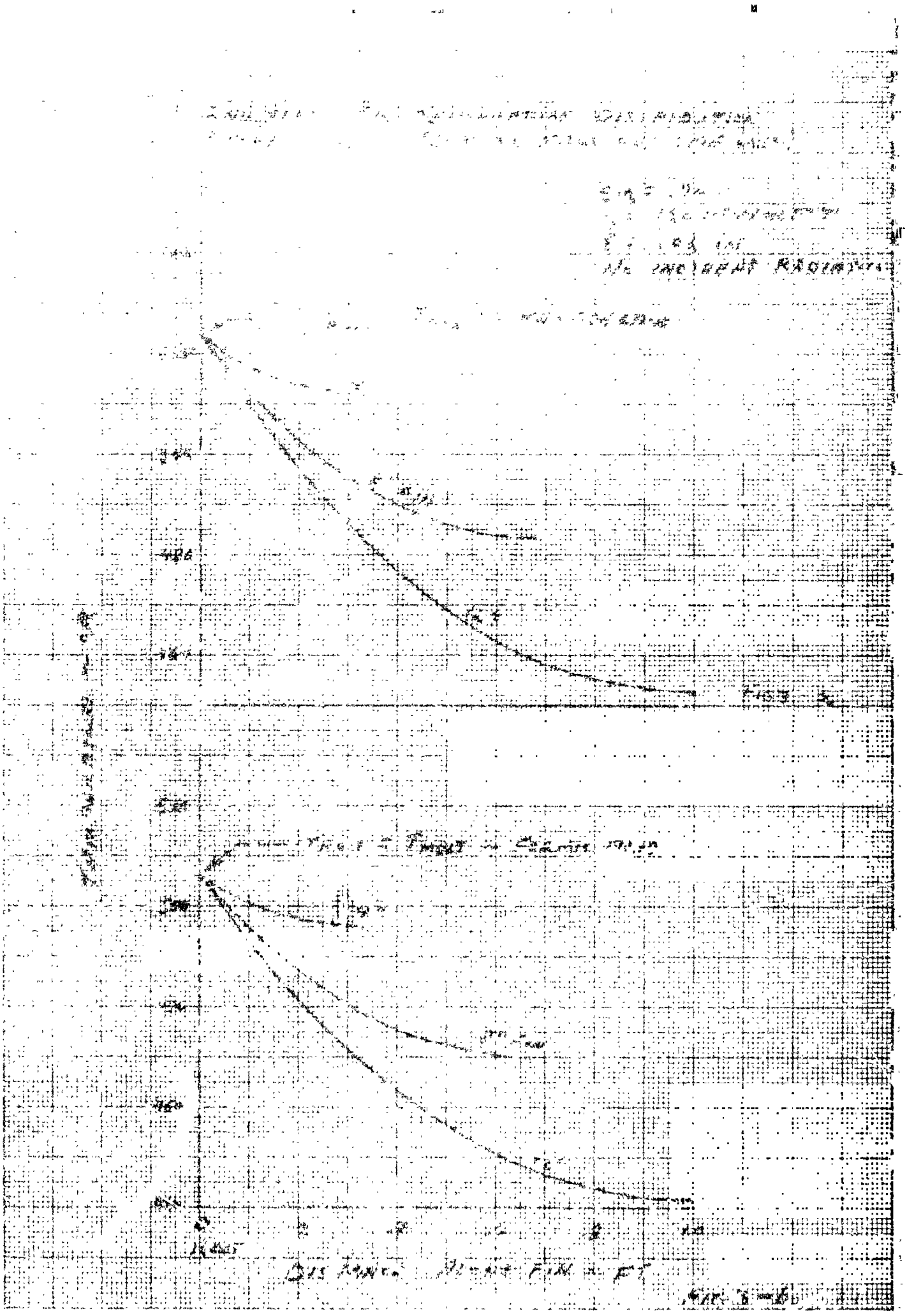

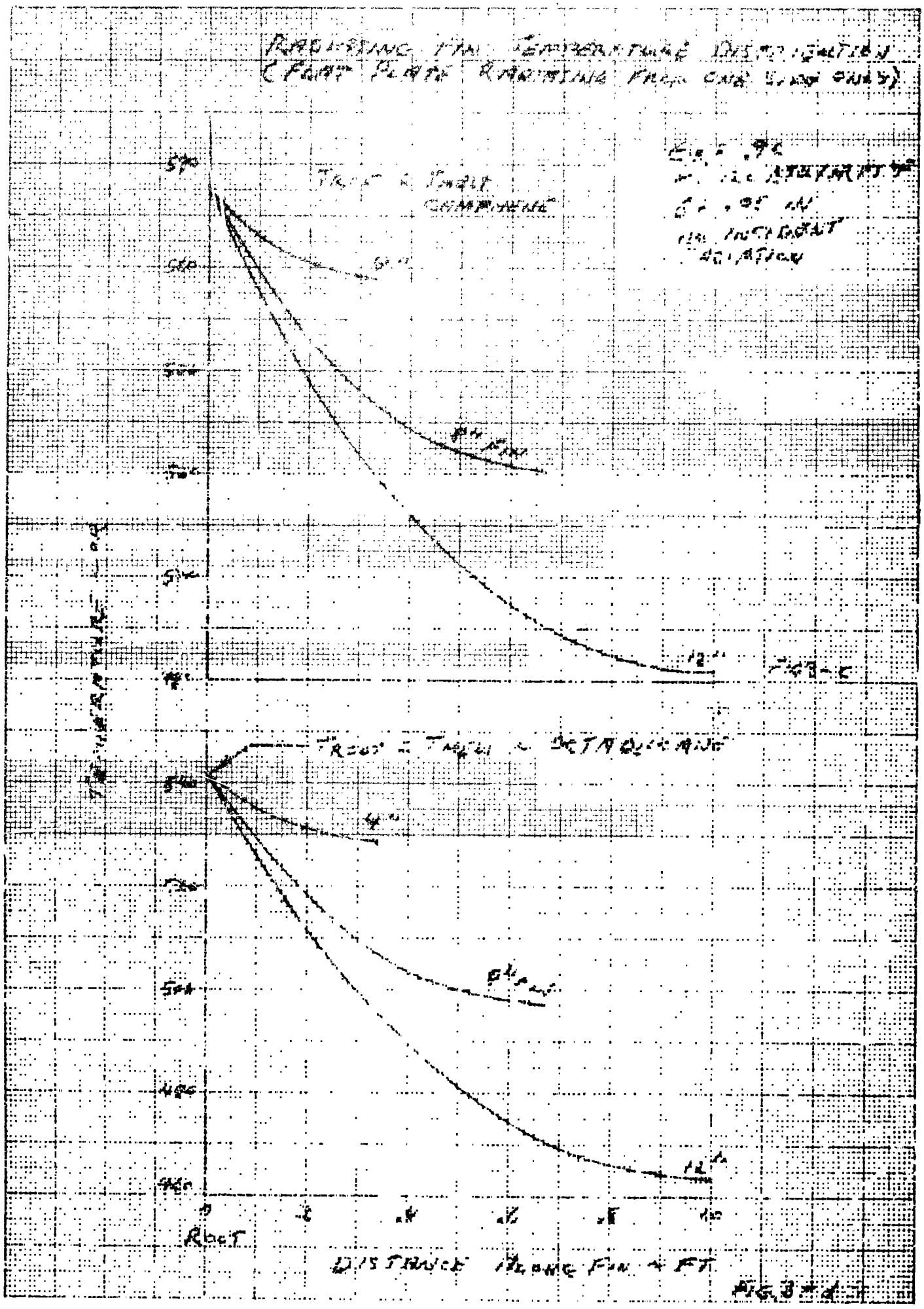

K+E 10 X 10 TO THE CM. 359-14 KEUFFEL & ESSER CO. MADE IN U.S.A.

Figure 4 presents the radiator fin performance for the selected configurations. The steady state heat rejection (q_R) of the radiating fin is plotted as a function of fin length and root temperature. The fin effectiveness is presented in the same manner.

Part 3 Analysis of a Heat Rejecting Fin With Attached Fusible Material

Debugging of the digital computer program for this analysis continued. It was found that the node array was too extensive to allow the required real time analysis to be performed with a reasonable amount of machine time. The program is being reworked to reduce machine time and still maintain a stable transient analysis. The revised parts of the program require additional corrections and checkout.

Thermophysical Properties

Normal Paraffins - A review of normal paraffins shows attractive properties for their use as thermal control fusible materials. These include high heats of fusion, selective melting temperatures and chemical inertness. The n-paraffins with an even number of carbon atoms from 14 to 30 cover the melt temperature range of interest, 40-150°F, Figure 5-a. These materials have a heat of fusion ranging from 98 lb. 109 BTU/lb., Figure 5-b. Two of the materials selected for this study are in this group, n-Hexadecane, $C_{16}H_{34}$, and n-Octadecane, $C_{18}H_{38}$. Tetradecane, $C_{14}H_{30}$, Eicosane, $C_{20}H_{42}$, and Octacosane, $C_{28}H_{38}$, are in this group and listed as available in Baker's Organic Laboratory Chemical Catalog. Camphene (Melt Pt 35-45°C), $2.20/500g was selected over Eicosane (Melt Pt 36-38°C), $17.85/100g, initially because of the relative price ratio of 1:40 and apparent equality in thermal properties. Both Camphene and Formic Acid have, on test, shown some undesirable properties for subject application as discussed under Experiments, Property Determination. Substitution for Camphene and/or Formic Acid by suitable n-paraffins is considered.

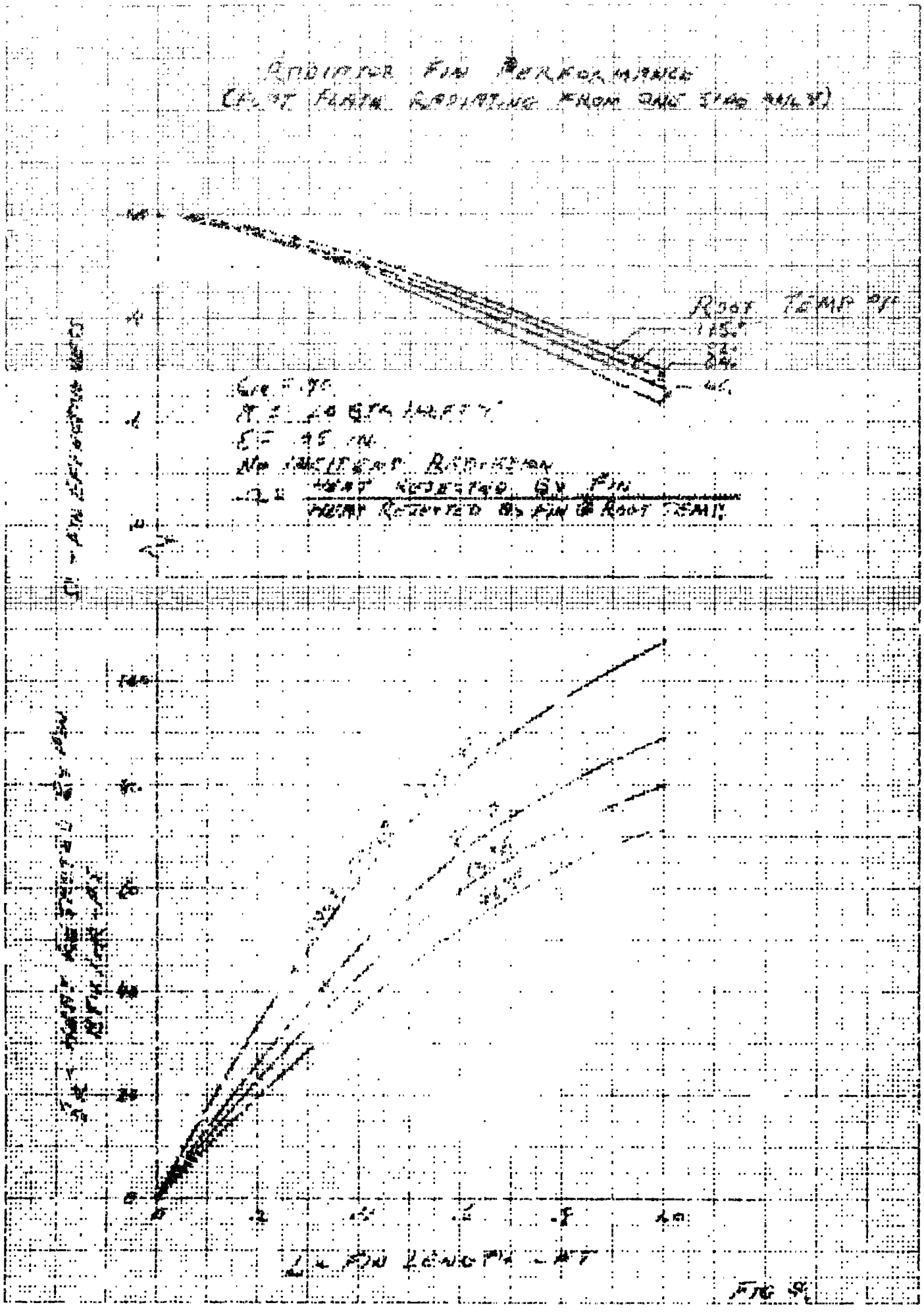

FIG 9

K+E 10 X 10 TO THE CENTIMETER 46 1513
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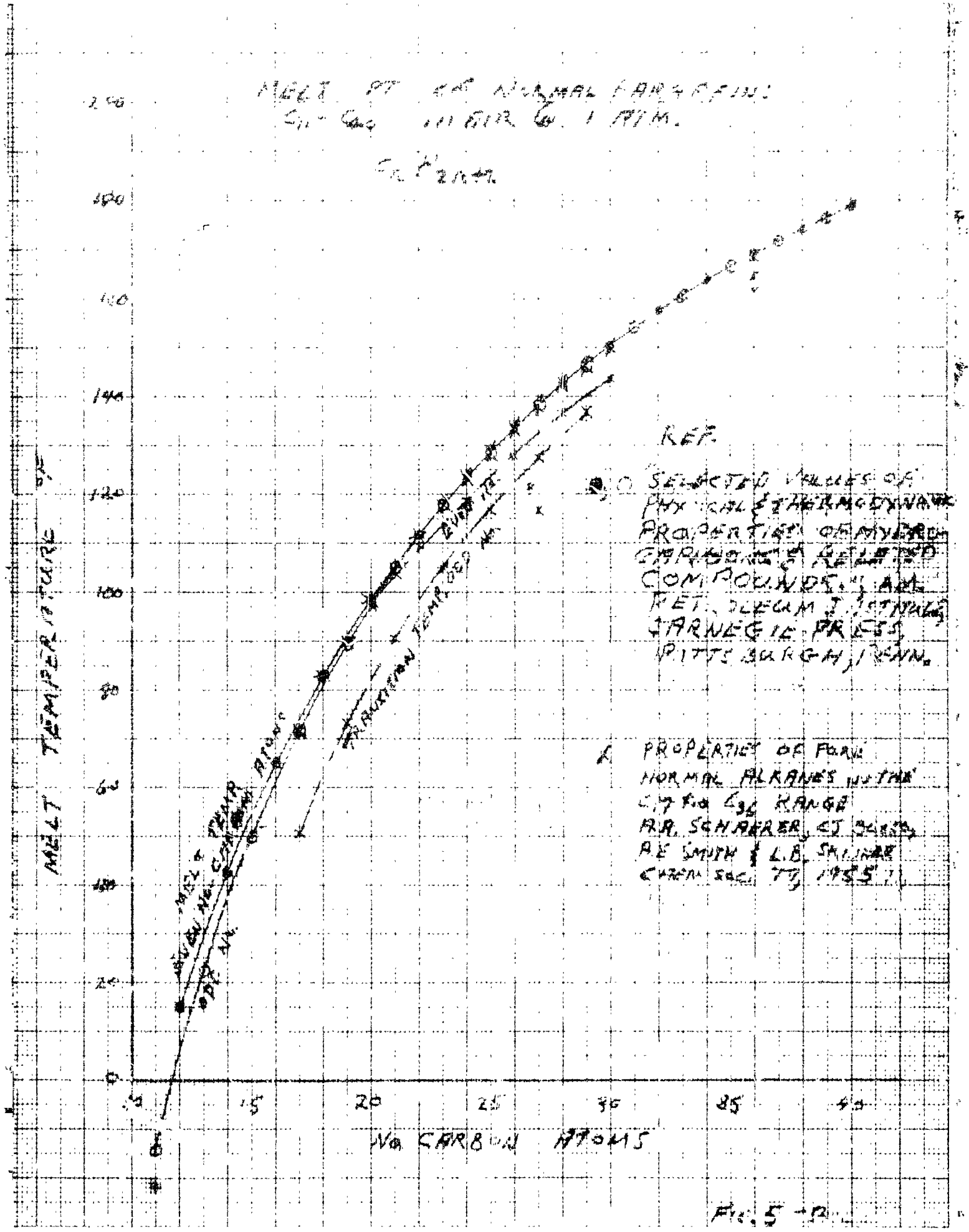
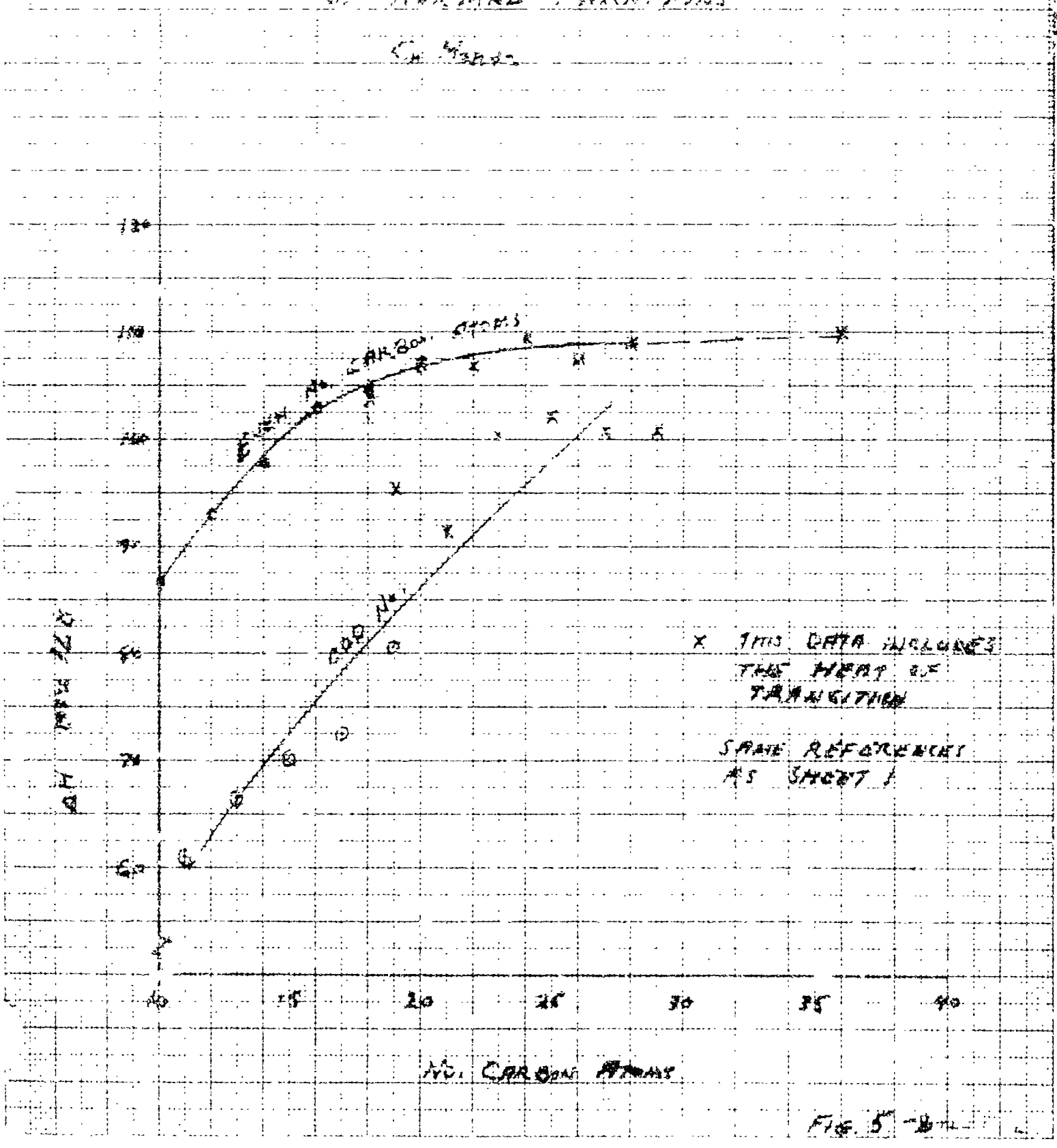

MELT PT OF NORMAL PARAFFINS
C_{11} - C_{36} IN AIR @ 1 ATM.
REF.
SELECTED VALUES OF PHYSICAL & THERMODYNAMIC PROPERTIES OF HYDROCARBONS & RELATED COMPOUNDS, AM. PETROLEUM INSTITUTE, CARNEGIE PRESS, PITTSBURGH, PENN.
PROPERTIES OF PURE NORMAL ALKANES IN THE C_{17} TO C_{36} RANGE
A.A. SCHAERER, CJ BOSTON, A.E. SMITH & L.B. SKINNER
CHEM. SOC. 77, 1955
MELT TEMPERATURE °F
NO CARBON ATOMS
TRANSITION TEMP. 000
MELT TEMP. EVEN NO. CARBON ATOMS
0
20
40
60
80
100
120
140
160
180
200
10
15
20
25
30
35
40

FIG. 5-B


HEAT OF FUSION & TRANSITION
OF NORMAL PARAFFINS
C_nH_{2n+2}
CARBON ATOMS
X THIS DATA INCLUDES THE HEAT OF TRANSITION
SAME REFERENCES AS SHEET 1
NO. CARBON ATOMS
FIG. 5

Additional data on the availability, cost and difficulty of producing the normal paraffins of interest will be obtained.

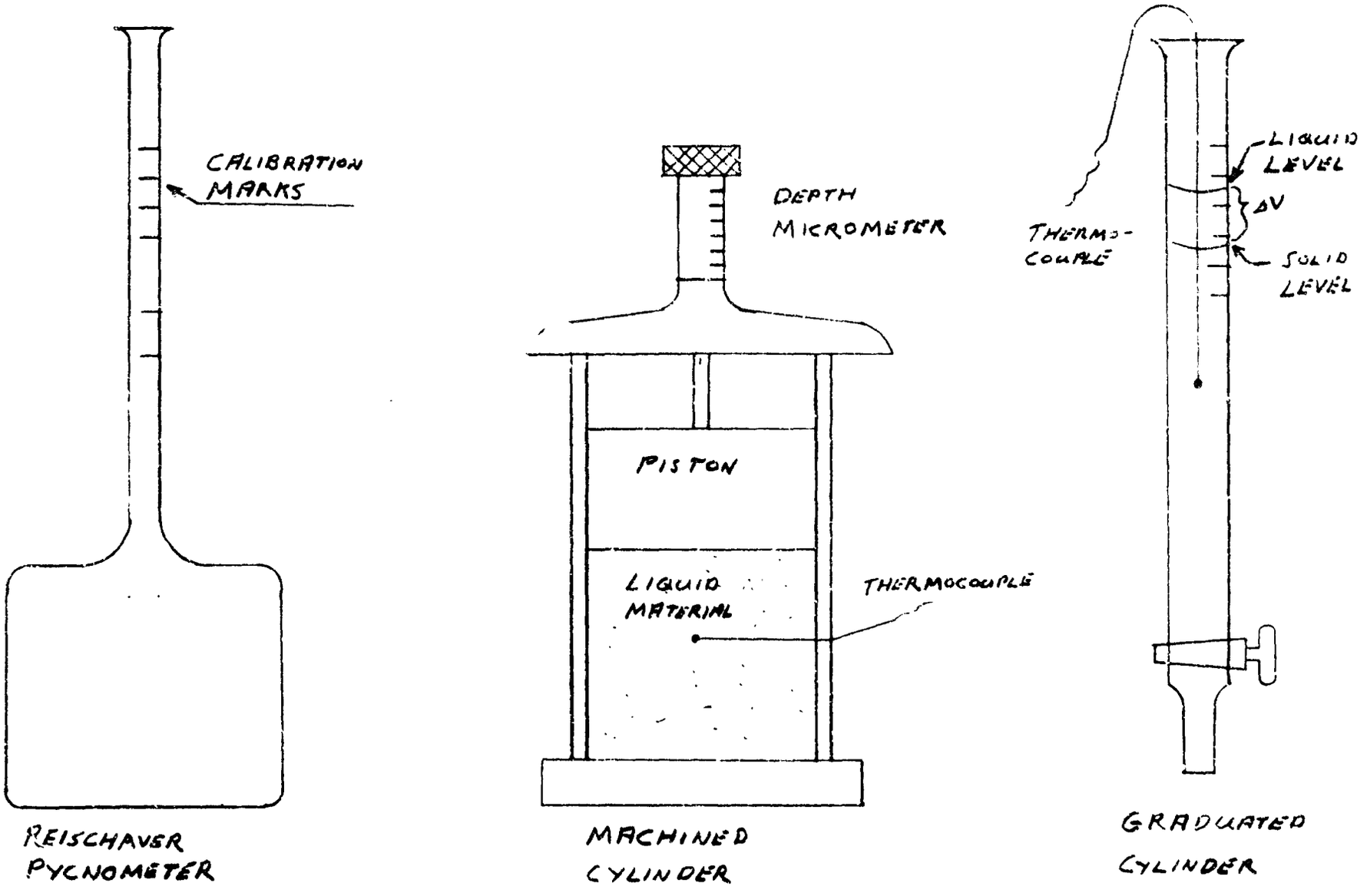
Experimental Property Determination - The volume occupied by the material at various temperatures both in the solid and liquid states as well as the rapid change of volume which takes place when changing phase at the melting point are of critical importance for this application. A literature search revealed that density data were available for all of the materials in the liquid state except Camphene, but literature on solid densities was rather scant. In addition to information on the solid densities, the variance of liquid density over the useful temperature range of the analysis is of importance for the change of thermal resistance between nodes of constant thermal mass. Once this density information could be developed experimentally, then the volume occupied by the selected material over a wide temperature range and in both the solid and liquid phase can be calculated. The following procedure was followed in developing this information on three materials so far obtained.

A. Determination of Density Variation with Temperature

An initial survey of methods available for determining the density of liquids indicated that a Reischauer pycnometer could be effectively used. A pycnometer, Figure 6, was obtained and its effective volume at various temperatures was determined by calibrating with known weights of doubly-deionized, distilled water.

1. Calibration of Pycnometer

The purified water was boiled to remove any dissolved gases and slowly cooled to the temperature at which the density measurement was to be made. This water was then introduced into the pycnometer until the calibration mark and the meniscus of the water coincided in the pycnometer neck. The filled pycnometer was held in a constant temperature bath until equilibrium was reached at the desired



DENSITY MEASUREMENT DEVICES

FIG 6

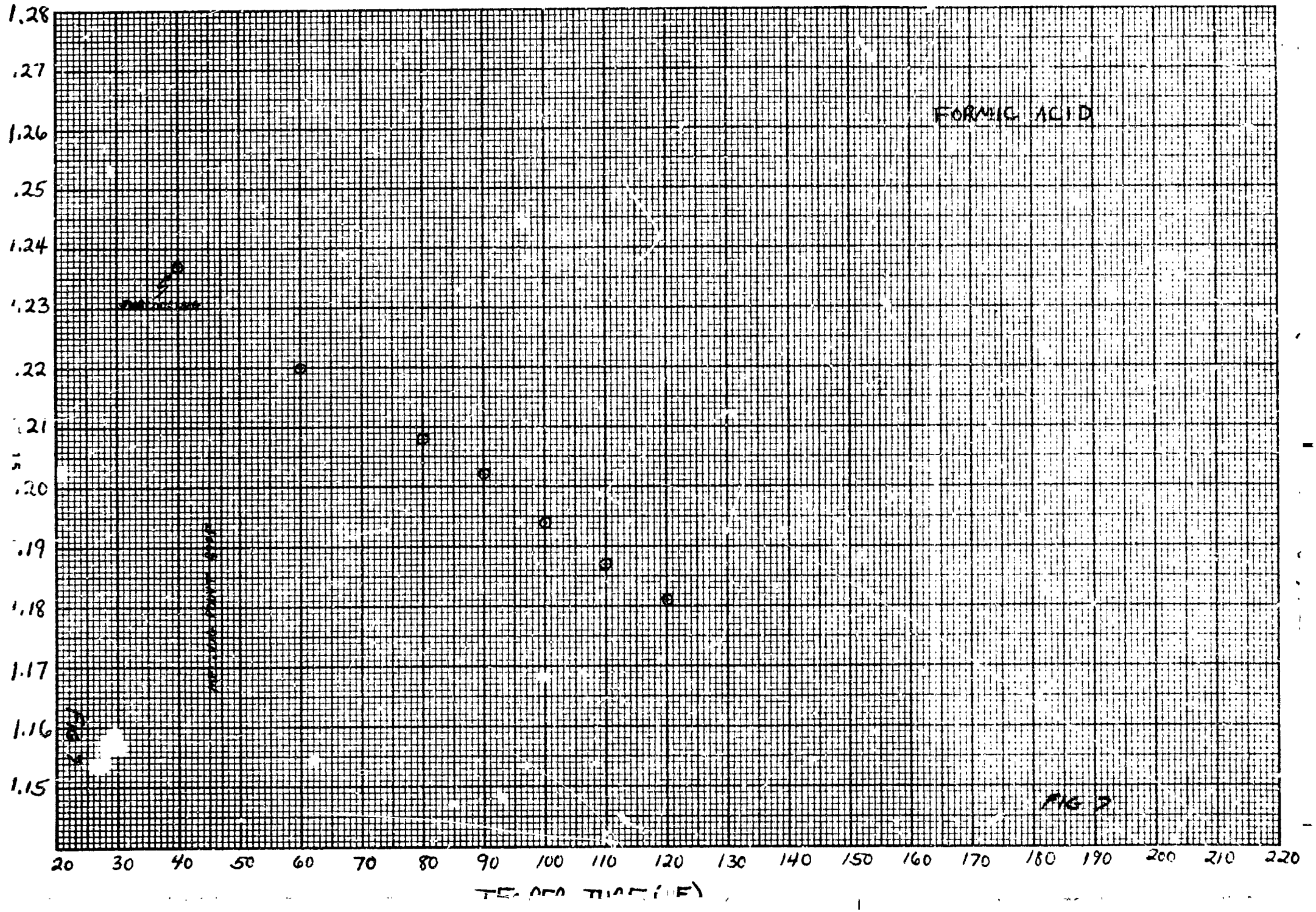
temperature then an adjustment was made in the water level to insure the level of the liquid was properly adjusted to the calibration mark. Once the water level had been adjusted to the mark at the desired temperature the pycnometer was removed from the constant temperature bath, carefully dried on the outside and weighed on a balance sensitive to 1/10 milligram. The water level in the pycnometer would usually change upon removal from the temperature bath, but this was of no consequence because we are now interested only in the weight of the water contained in the pycnometer and not the level in the pycnometer neck. The weight of the clean dry pycnometer was now subtracted from the weight of the filled pycnometer to determine the weight of water. This weight was converted to an appropriate volume of water by consulting water density tables in the literature. We had now determined the volume of the pycnometer to its calibration mark to the nearest 1/10 of a milliliter. We now used this volume to calculate a correction factor due to buoyancy of air corrected for temperature and barometric pressure at the time of the weighings. This buoyancy factor was added to the value obtained for the initial weighings, thus giving the true weight of water in the pycnometer. This adjusted weight was then used to compute the volume of the pycnometer to the nearest 1/1000 of a milliliter. This procedure was repeated at various temperatures to give a calibration chart for the pycnometer. This method minimizes errors introduced by expansion of the bottle portion of the pycnometer because it is calibrated at its expanded volume at the temperature. Since the pycnometer narrows down to a capillary-like neck the surface area exposed to the air is small compared to the overall volume of liquid contained, thus evaporation is kept at a minimum. It is estimated that density

values obtained by the Reischauer pycnometer method will have an error of less than $\pm$ 0.002.

2. Density Determination

The pycnometer was immersed in a constant temperature bath, at the desired temperature, and the liquid material introduced slowly until the meniscus of the material coincides with the calibration mark. The pycnometer is allowed to "soak" in the constant temperature bath until equilibrium is attained and any needed adjustment in the level at the calibration mark due to expansion or contraction of the contents is made. The pycnometer is removed from the bath, carefully dried and weighed to constant weight. The appropriate calculations and corrections are made to give the true weight of the material in the pycnometer. This true weight (in grams) is then divided by the volume (in milliliters) of the pycnometer at the temperature at which it was filled, to give the density of the material in grams per milliliter.

For solid density measurements, the material is added as a liquid as before, but only a small portion of the bulb of the pycnometer is immersed in the constant temperature bath at a temperature below the melting point. As the level of the liquid in the pycnometer is slowly raised by adding more material, the level of the pycnometer is lowered into the bath so as to only be cooling that portion of the bulb filled. This technique insures "good packing" of the solid and eliminates the tendency of "funneling" on solidification. The liquid is introduced until the calibration mark is reached, removed from the bath, dried and weighed to constant weight. This weight is then converted to a "true weight" and the density calculated as before. The temperature-density curves for Formic Acid, Camphene, and n-Hexadecane are shown in Figures 7 through 9, temperature-specific volume curves are shown in Figures 10 through 12.


FORMIC ACID
FIG 2
1.28
.27
1.26
.25
1.24
1.23
.22
.21
.20
.19
1.18
1.17
1.16
1.15
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220

K+E 10 X 10 TO THE ½ INCH 359-11
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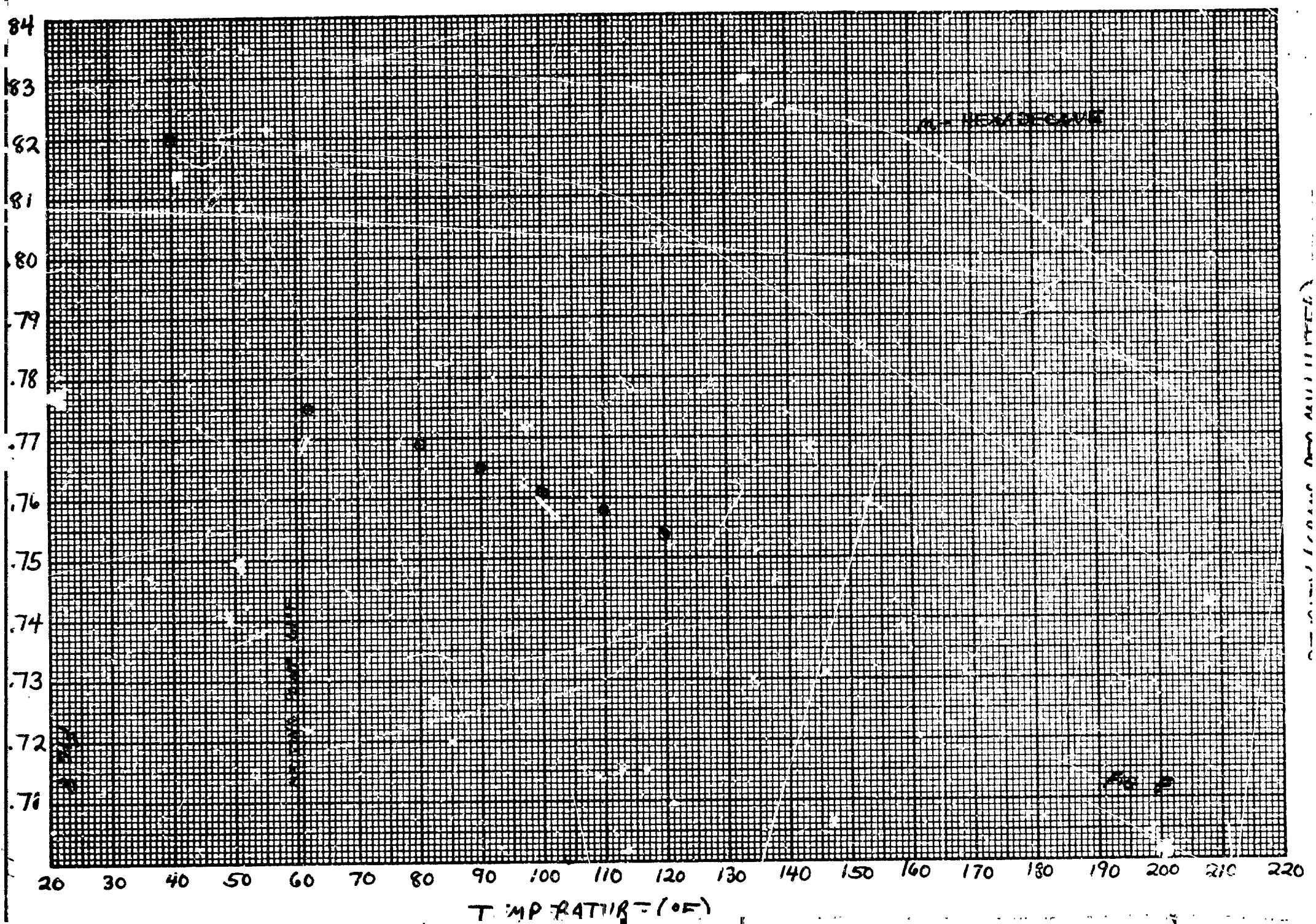

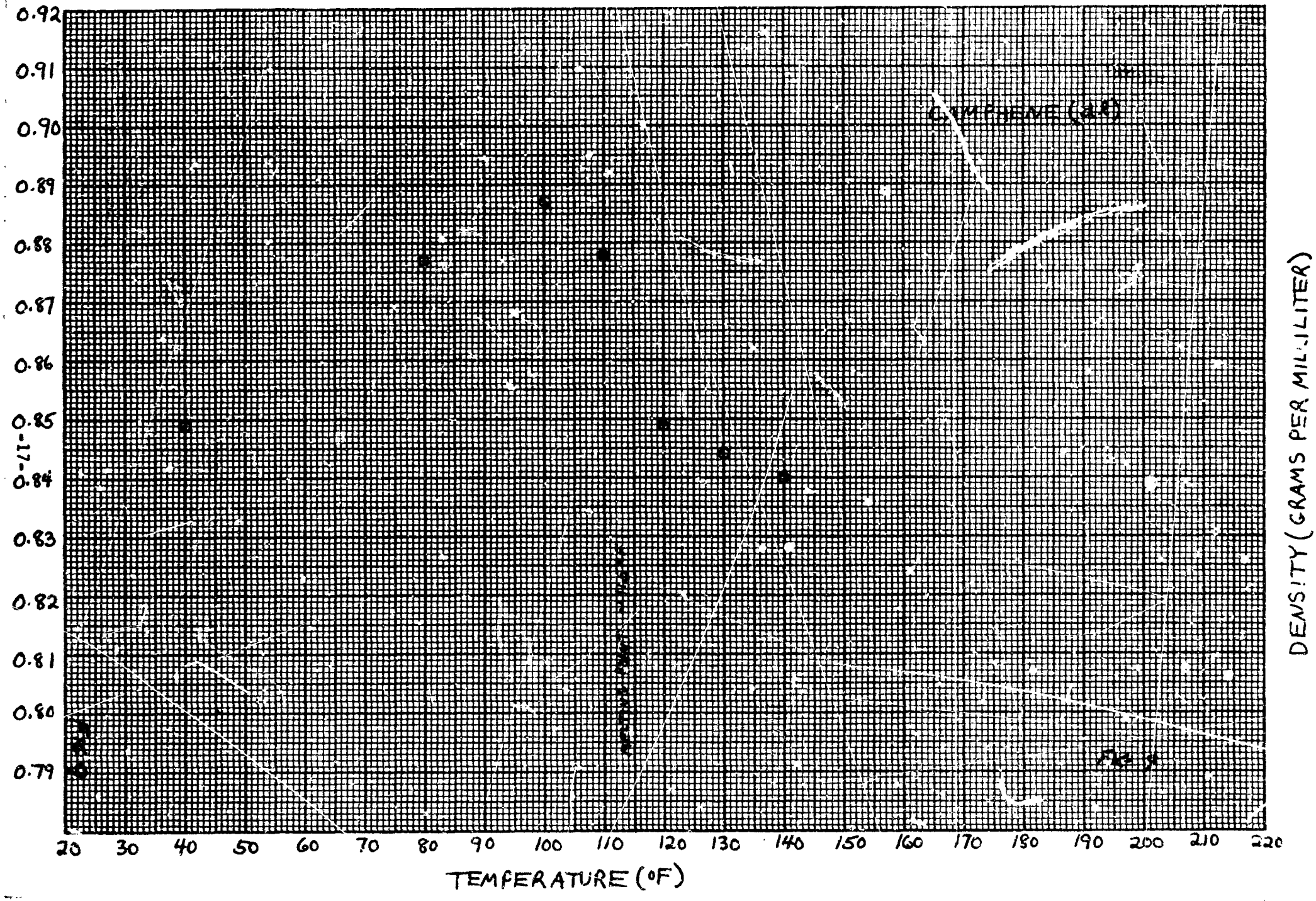

0.92
0.91
0.90
0.89
0.88
0.87
0.86
0.85
0.84
0.83
0.82
0.81
0.80
0.79
20
30
40
50
60
70
80
90
100
110
120
130
140
150
160
170
180
190
200
210
220
TEMPERATURE (°F)
DENSITY (GRAMS PER MILLILITER)

K+E 10 X 10 TO THE ½ INCH 359-11
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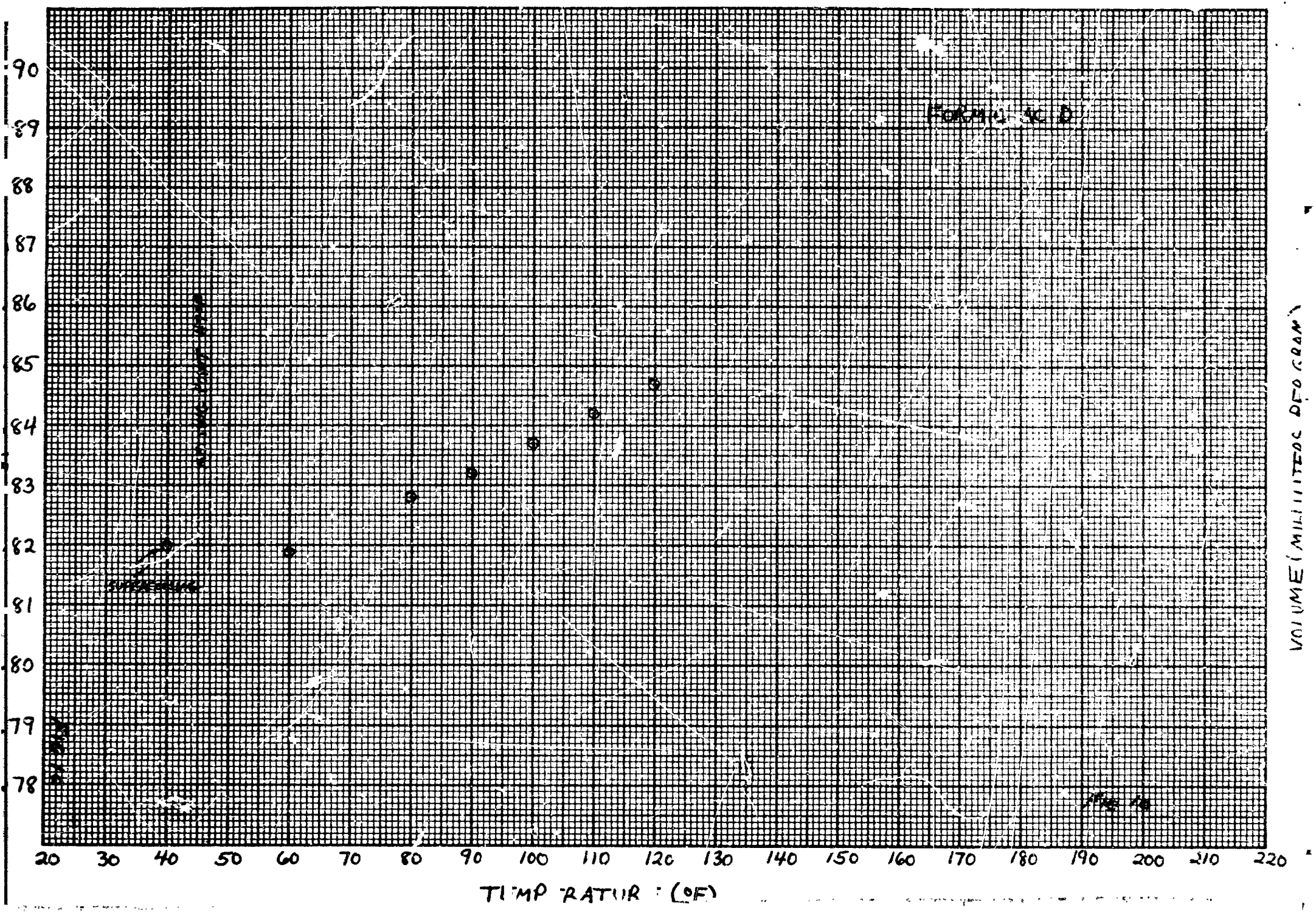

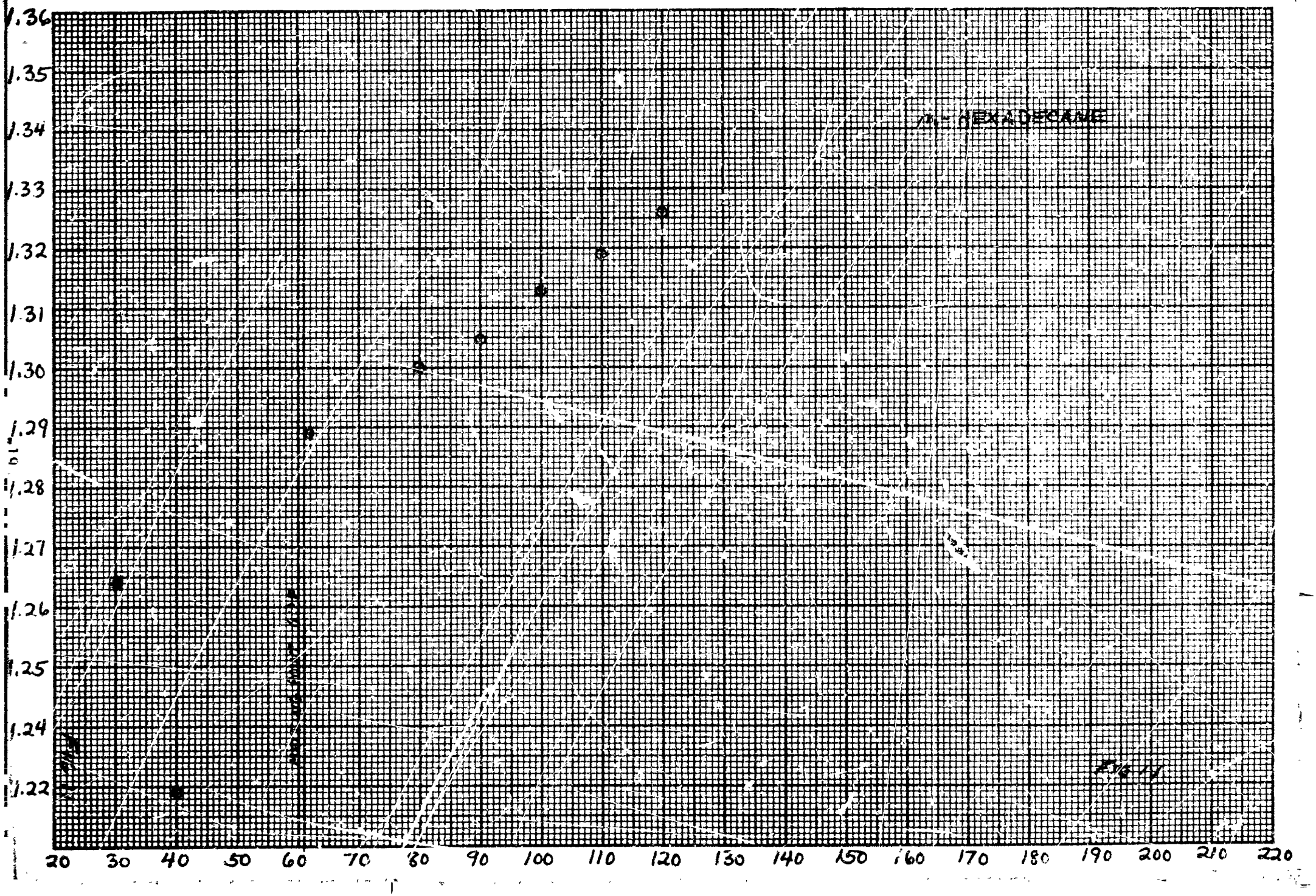
n - HEXADECANE
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.29
1.28
1.27
1.26
1.25
1.24
1.22
20
30
40
50
60
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220

K+E 10 X 10 TO THE ½ INCH 359-11
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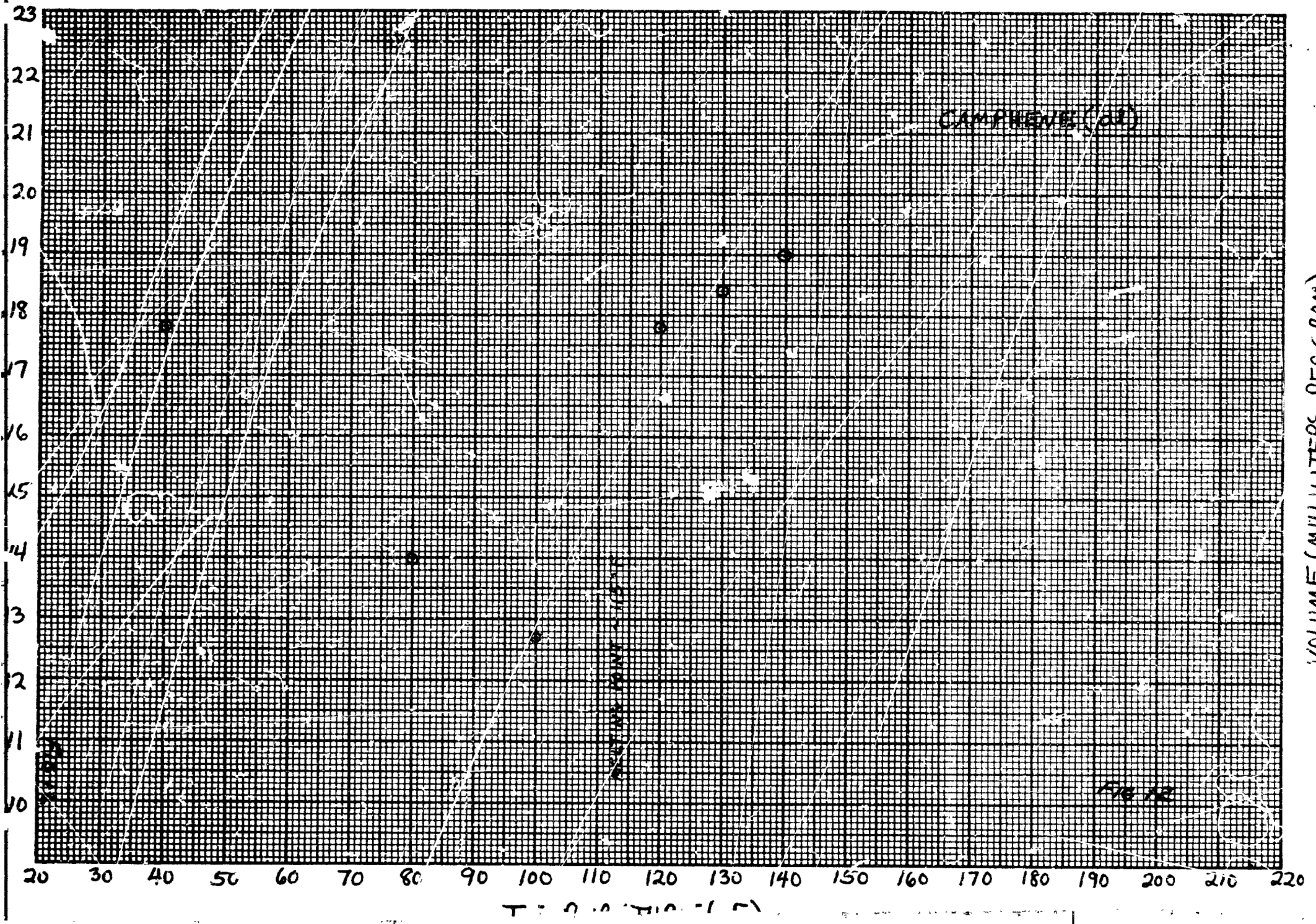


B. Determination of Volume Change Due to Phase Transition at Melting Point

Several methods were considered for determining this important property of the subject materials. One method was a machined cylinder, Figure 6, with a close fitting movable piston, the other a graduated glass cylinder in which the change of volume could be followed visually by means of the graduated markings.

The machined metal cylinder was first tried but was found to be unsatisfactory. The cylinder was filled with a known weight of liquid Camphene and immersed in a constant temperature bath at 120°F. The cylinder was fitted with a copper-constantan thermocouple positioned in such a manner so as to be as close to the center of the mass of added material as possible. In this manner it was thought that the temperature of the material being tested could be monitored with a potentiometer continuously throughout the phase transition. The piston was inserted into the machined cylinder and allowed to settle toward the upper surface of the Camphene liquid. It was thought that the piston would settle gradually displacing air until it rested in intimate contact with the Camphene surface. The settling was followed until no more movement was observed. The movement of the piston into the cylinder was easily followed to the nearest 1/1000 inch by means of a depth micrometer supported on the upper edge of the machined cylinder. By avoiding external support of this measuring device it was thought that errors due to movement of supports would be eliminated. Once the piston had stopped moving downward the temperature of the constant temperature bath was lowered at a rate of approximately 1°F every 15 minutes. This slow temperature change was used to allow the Camphene and the bath to be almost in thermal equilibrium. This equilibrium was monitored by alternately checking the bath temperature and the Camphene temperature with copper-constantan thermocouples. As the melting point of the Camphene was reached (approximately 115°F) the depth micrometer

showed erratic readings. It was concluded that this failure to achieve consistent results was due to trapped air between the Camphene surface and the piston. The piston behaved in a 'spongy' manner thus indicating that something compressible was beneath it. An attempt was made to go from solid Camphene to liquid Camphene by raising the temperature of the bath slowly above the melting point of Camphene. In this reverse procedure another inherent difficulty of the method became apparent as the liquid Camphene leaked around the piston rather than raising it. Due to these difficulties the machined cylinder method was abandoned.

It was decided to go the graduated glass cylinder method.

A known volume of the liquid materials was added to a reference mark on the glass cylinder, Figure 6. A copper constantan thermocouple was inserted into the liquid mass of material in the glass cylinder so the temperature could be monitored as desired. A correction was made for the displacement of liquid by the thermocouple wire and this value recorded. The glass cylinder was now cooled in a temperature bath to below the melting point of the material within the cylinder. It was found that the technique used with the pycnometer whereby the freezing is done in small increments from the bottom of the cylinder towards the top gave good "packing". Although the values obtained were not as accurate as those which would have been obtained had the machined cylinder worked (since the volume change could be measured only to 1/10 ml) they serve to show the order of magnitude which can be expected when these materials change volume in passing through their phase transition. It is estimated that the values obtained should have a precision of $\pm$ 0.5%.

C. Check of Melting Points

The measurement of the melting points of the candidate materials was conducted by inserting a copper-constantan thermocouple into the pycnometer

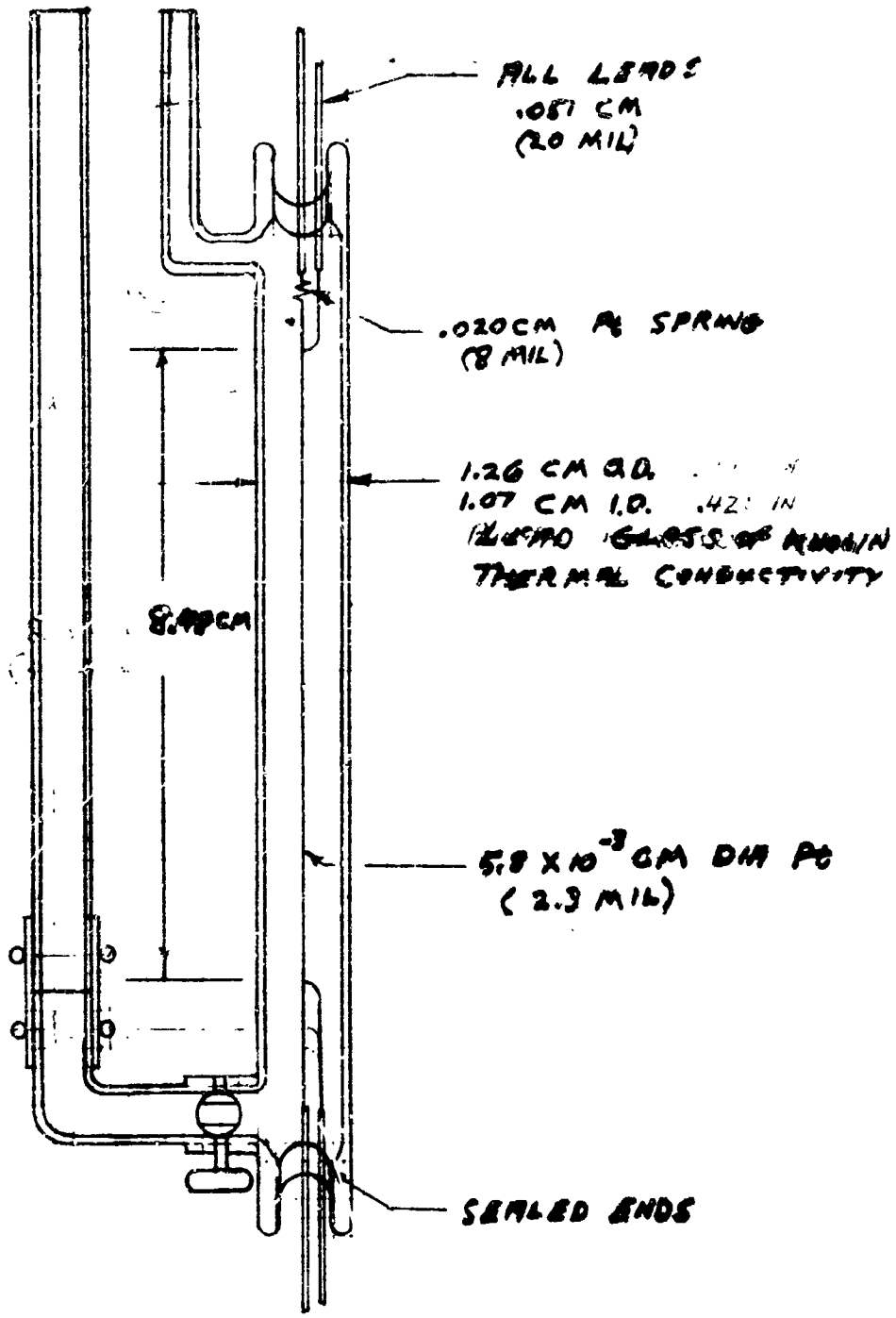
filled with the material to be tested. The thermocouple was referenced to ice made from deionized water at 32°F. Both n-Hexadecane and Camphene showed rather sharp melting points at 61°F and 115°F respectively. Formic Acid, however, exhibited a subcooling effect on solidification. To check Formic Acid it was cooled until the contents of the pycnometer had frozen solid. It was then allowed to warm up until the liquid-solid mixture showed a slight increase in temperature. This occurred just when the last remnants of the solid phase melted. This slight warming occurred at approximately 47°F. These values obtained are considered accurate within 1°F.

D. Subcooling of Formic Acid

In conducting the previous experiments it became apparent that Formic Acid, when slowly cooled remained in a liquid state at temperatures somewhat below its melting point. The phenomenon was investigated by cooling the acid in a graduated cylinder slowly below its melting point while continuously monitoring its temperature. It would be subcooled to a temperature range of 33°F - 35°F and the material solidified in several minutes. After solidification the solid material would cool in a linear fashion. No further work on how this sub-cooling effect can be avoided has been done up to the present time.

E. Measurement of Thermal Conductivity of Liquids

A tentatively selected apparatus for measuring the thermal conductivity of liquids is shown in Figure 13. This apparatus, its use and accuracy, is described in an article, "Thermal Conductivity of Some Organic Fluids" by O.B. Cecil and K.H. Munch, Industrial & Engineering Chemistry 48, 1956. Agreement between reported and measured values of thermal conductivity of some common liquids indicate that this method of determining thermal conductivities is reliable.



CELL FOR MEASURING THERMAL CONDUCTIVITY OF LIQUIDS (FULL SIZE)

FIG. 13

The method has further advantages of measuring speed and relative simplicity of apparatus and instrumentation. It is, therefore, considered as a good choice for experimental determination of liquid thermal conductivity, should such be required in the performance of the study.