DYNATECH CORPORATION

THERMOPHYSICAL PROPERTIES MEASUREMENTS PERFORMED FOR NASA

Prepared by:

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Submitted to:

National Aeronautics and Space Administration
Langley Research Center
Langley Station
Hampton, Virginia

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Dynatech Project No. NAS-7

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Section 1

INTRODUCTION

This report summarizes the work done on the Thermophysical Properties Test Program performed by Dynatech Corporation for the National Aeronautics and Space Administration under Purchase Order L-48,876.

The test program consisted of thermal conductivity, specific heat, and density measurements over a range of 75 to 300°F. The test results, methods and procedures used, and a discussion of the data are presented in the following sections of this report.

Section 2

THERMAL CONDUCTIVITY MEASUREMENTS

2.1 The Method of Testing

The thermal conductivity of the sample was determined by the comparative method. By this method the thermal conductivity of a material is determined by comparing it to that of a known standard, referred to as a heat meter. This is accomplished by sandwiching the test sample between two identical heat meters and passing a unidirectional heat flow through the stack. With a unidirectional heat flow and at steady state conditions, the heat flux through the sample is equal to the heat flux through the heat meters, and is directly proportional to the temperature difference across the sample or heat meters.

$$\frac{Q}{A} = \frac{k \Delta T}{X}$$

where

$\dfrac{Q}{A}$ = heat flux, Btu/hr ft^2

k = thermal conductivity, Btu/hr ft $^{\circ}$F

ΔT = temperature difference, $^{\circ}$F

X = length of heat flow path, ft.

Since the heat flux is the same in both heat meters and in the sample, it is therefore only necessary to measure the temperature differences in the heat meters and sample to determine the thermal conductivity of the sample.

Based upon the sensitivity of the measuring instruments, the accuracy of the heat meter conductivity values and the actual deviation from unidirectional heat flow, the accuracy of this method of testing can be safely assumed to be within ±5%.

2.2 Test Procedure

The thermal conductivity tests were performed on the instrument shown as Figure 1. Figure 2 shows the test stack being assembled. The actual elements used in the test stack are shown schematically in Figure 3.

As shown in Figure 3, the sample was sandwiched between two identical fused silica heat meters. The heat flux through the stack was generated by a flat plate heater located at the top of the stack, and the temperature level of the stack was maintained by an auxiliary heater placed between the bottom heat meter and heat sink. Insulating material was placed between the auxiliary heater

Figure 1.

DYNATECH MODEL TCFC-R8 SERIES
COMPARATIVE TESTER

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17 TUDOR STREET
CAMBRIDGE 39, MASSACHUSETTS

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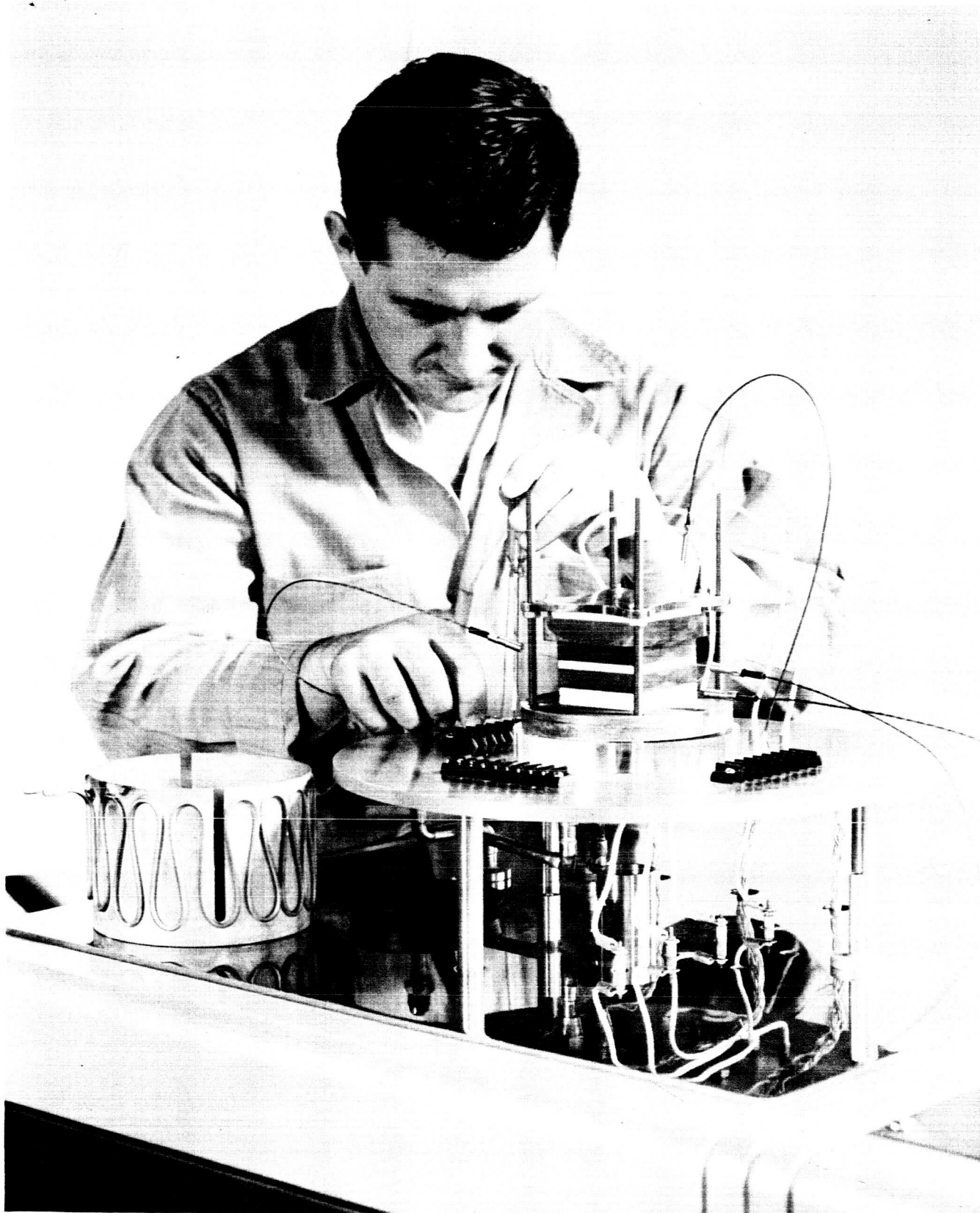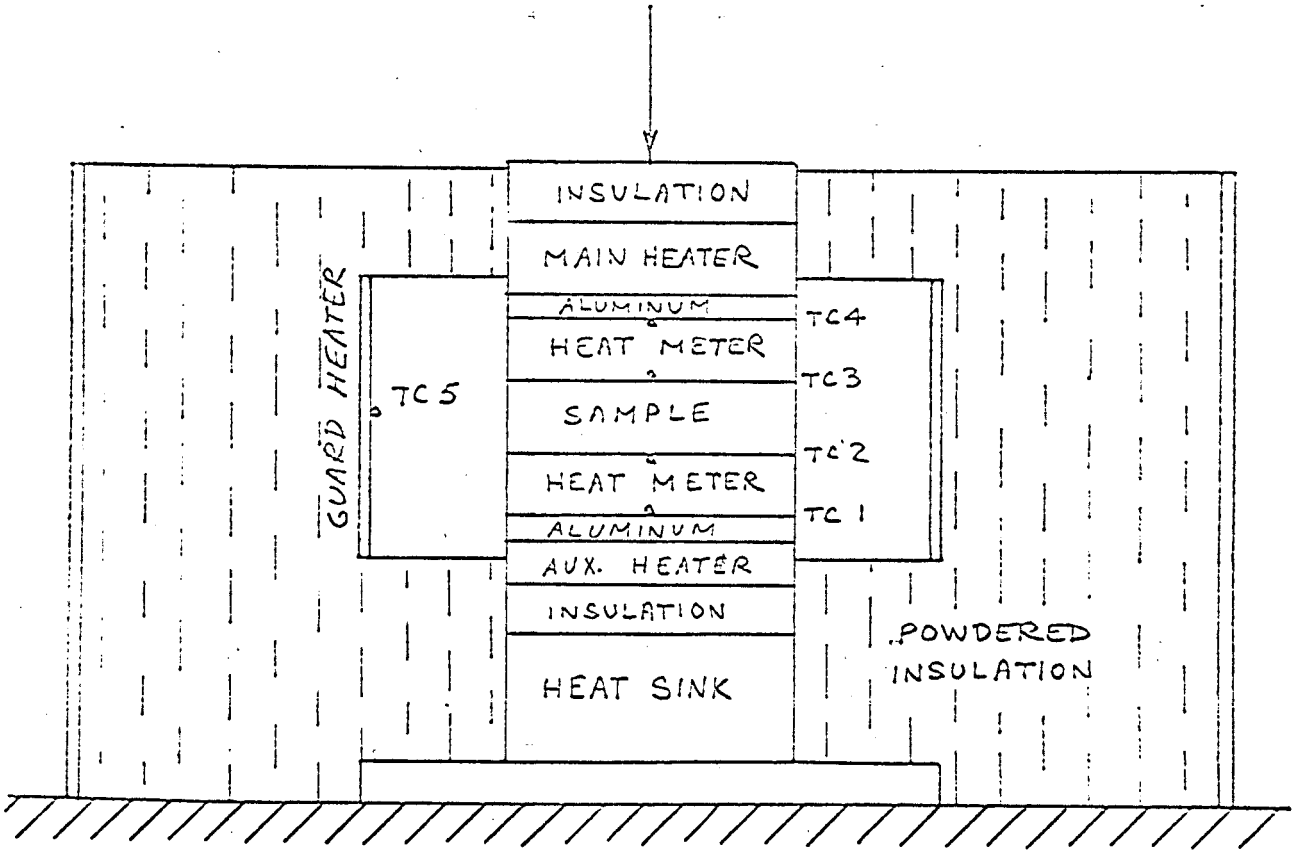Figure 2.

Test Stack Assembly
TCFC-R8 Series
Comparative Thermal Conductivity Apparatus

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5# WEIGHT

INSULATION	
MAIN HEATER	
ALUMINUM	TC4
HEAT METER	
SAMPLE	TC3
	TC2
HEAT METER	
ALUMINUM	TC1
AUX. HEATER	
INSULATION	
HEAT SINK	

GUARD HEATER

TC5

POWDERED INSULATION

TEST STACK
ARRANGEMENT

FIGURE 3

and the sink in order to provide a measure of isolation for the auxiliary heater. Aluminum plates, 1/8 inch thick, were placed between the heaters and heat meters so as to ensure an evenly distributed heat flow across the entire 2-1/2 inch square surface.

To preclude side heat losses, the stack was surrounded by a cylindrical guard heater, the temperature of which was maintained at the average sample temperature. In Figure 2 the outer guard can be seen to the left of the stack. The stack-guard heater assembly was then surrounded by a liquid-cooled shroud, and the entire test rig within the shroud was covered with a low conductivity powdered insulation.

The heat meters were instrumented with copper constantan thermocouples cemented into grooves cut across each of the two heat meter faces. For data reduction, the heat meter thickness used was the actual thickness measured between thermocouple junctions. Temperatures were measured with a Rubicon potentiometer using an electronic ice reference junction. Sample and heat meter thicknesses were measured with a micrometer to .001 inch.

To minimize thermal contact resistance, all contacting surfaces within the stack were coated with a thin layer of silicone grease. A load of approximately 5 pounds was applied to the test stack to further ensure good thermal contact and to prevent sample warping.

A water-cooled heat sink was used in these tests and liquid nitrogen was used as the coolant for the 75 $^\circ$F point. The test runs proceeded from low to high temperature.

All tests were performed by heating or cooling the test stack temperatures to the required levels and allowing the stack to reach an equilibrium or steady state condition. The test was considered acceptable if the conductivity of the sample did not vary more than 3% over the period of one hour.

2.3 Data Reduction

By assuming unidirectional heat flow under steady state conditions, the heat flux through both heat meters equals the heat flux through the sample, i.e.,

$$\frac{Q}{A}\bigg|_{\text{Top H.M.}} = \frac{Q}{A}\bigg|_{\text{Sample}} = \frac{Q}{A}\bigg|_{\text{Bottom H.M.}} \tag{1}$$

or

$$\left(\frac{k\Delta T}{X}\right)_{\text{Top H.M.}} = \left(\frac{k\Delta T}{X}\right)_{\text{Sample}} = \left(\frac{k\Delta T}{X}\right)_{\text{Bottom H.M.}} \tag{2}$$

The thermal conductivity of the sample is calculated by first using the top heat meter conditions, then the bottom heat meter conditions, and finally averaging these two values to arrive at a final conductivity value.

$$k_{s_{(Top)}} = k_{THM} \left(\frac{X_s}{X_{THM}} \right) \left(\frac{\Delta T_{THM}}{\Delta T_s} \right) \qquad (3)$$

$$k_{s_{(Bot.)}} = k_{BHM} \left(\frac{X_s}{X_{BHM}} \right) \left(\frac{\Delta T_{BHM}}{\Delta T_s} \right) \qquad (4)$$

and

$$k_s \text{ ave} = \frac{k_{s_{(Top)}} + k_{s_{(Bot.)}}}{2} \qquad (5)$$

The heat meter conductivity values are taken at the average heat meter temperatures.

2.4 Sample Calculation

A sample calculation detailing the data reduction procedure is now presented. The thermocouple numbers listed correspond numerically to those shown in Figure 3, and were recorded when the test stack had reached a steady state.

Test Point $175^{o}F$

T.C.	Temperature, emf	Temperature, ^{o}F
1	2.442	139.96
2	2.991	153.60
3	3.614	186.16
4	4.199	209.00

$$\Delta T_{BHM} = .549 \text{ mv}$$

$$Tave_{BHM} = 146.8^{o}F$$

From Figure 4, $k_{BHM} = 0.84$ Btu/hr ft ^{o}F

$$\Delta T_s = .623 \text{ mv}$$

$$\Delta T_{THM} = .585 \text{ mv}$$

$$Tave_{THM} = 197.6^{o}F$$

From Figure 4, k_{THM} $=$ 0.87 Btu/hr ft ^{o}F

X_s $=$ .246 inch

X_{HM} $=$ .361 inch

From 3: $k_{s(Top)}$ $=$ $(.87) \left(\dfrac{.246}{.361}\right) \left(\dfrac{.585}{.623}\right)$ $=$ .548

From 4: $k_{s(Bot.)}$ $=$ $(.84) \left(\dfrac{.246}{.361}\right) \left(\dfrac{.549}{.623}\right)$ $=$ .509

From 5: k_s ave $=$ $\dfrac{.548 + .509}{2}$

k_s ave $=$ .529 Btu/hr ft ^{o}F

T_s ave $=$ $\dfrac{153.60 + 186.16}{2}$ $=$ 170°F

A complete list of conductivity values is presented in the following section.

2.5 <u>Test Results</u>

Average Sample Temperature, ^{o}F	k, Btu/hr ft ^{o}F
73	.542
108	.545
133	.553
148	.562
170	.529
206	.533
251	.529
300	.524

These results are shown graphically on Figure 5.

Section 3

SPECIFIC HEAT MEASUREMENTS

3.1 Method of Testing

The specific heat of the material was determined by the continuous method. By supplying an accurately measured quantity of heat to the test specimen, the functional relationship between the temperature response of the sample and the quantity of added heat is obtained.

$$Q = MC_p \frac{dT}{dt}$$

where

Q = heat added, Btu

M = mass of sample, pounds

T = Temperature, ^{o}F

t = time, minutes

Since dT/dt represents the slope of the time-temperature response curve, C_p may be readily calculated .

The accuracy of the specific heat measurements can be safely assumed to be within 2% due to close controlling of the guard heater and the accuracy of the readout instruments.

3.2 Test Procedure

The sample was sandwiched between two identical flat plate heaters and this assembly, in turn, was suspended within a triple-walled chamber. The innermost chamber is a thick-walled copper guard heater. The guard temperature was accurately controlled by a null balance controller feeding a silicon control rectifier power supply, and was maintained equal to the sample temperature throughout the entire temperature range of the test. Any temperature imbalance was sensed by a differential thermocouple placed between the sample and the guard. Such stringent controlling is necessary to preclude radiative heat losses from the sample. Otherwise, only a portion of the total heat input to the sample will be used as sensible heat, and the resulting data would be erroneous. The instrument used in the specific heat measurements is shown as Figure 4.

The second chamber, enclosing the guard, is a stainless steel structure which serves as a radiation shield for the guard. It is also used as a liquid nitrogen container for low temperature testing. The outermost chamber is a vacuum chamber. This rather elaborate scheme is necessary to minimize radiative and convective heat losses. Conductive heat losses are minimized by using fine gage copper wire for heater leads.
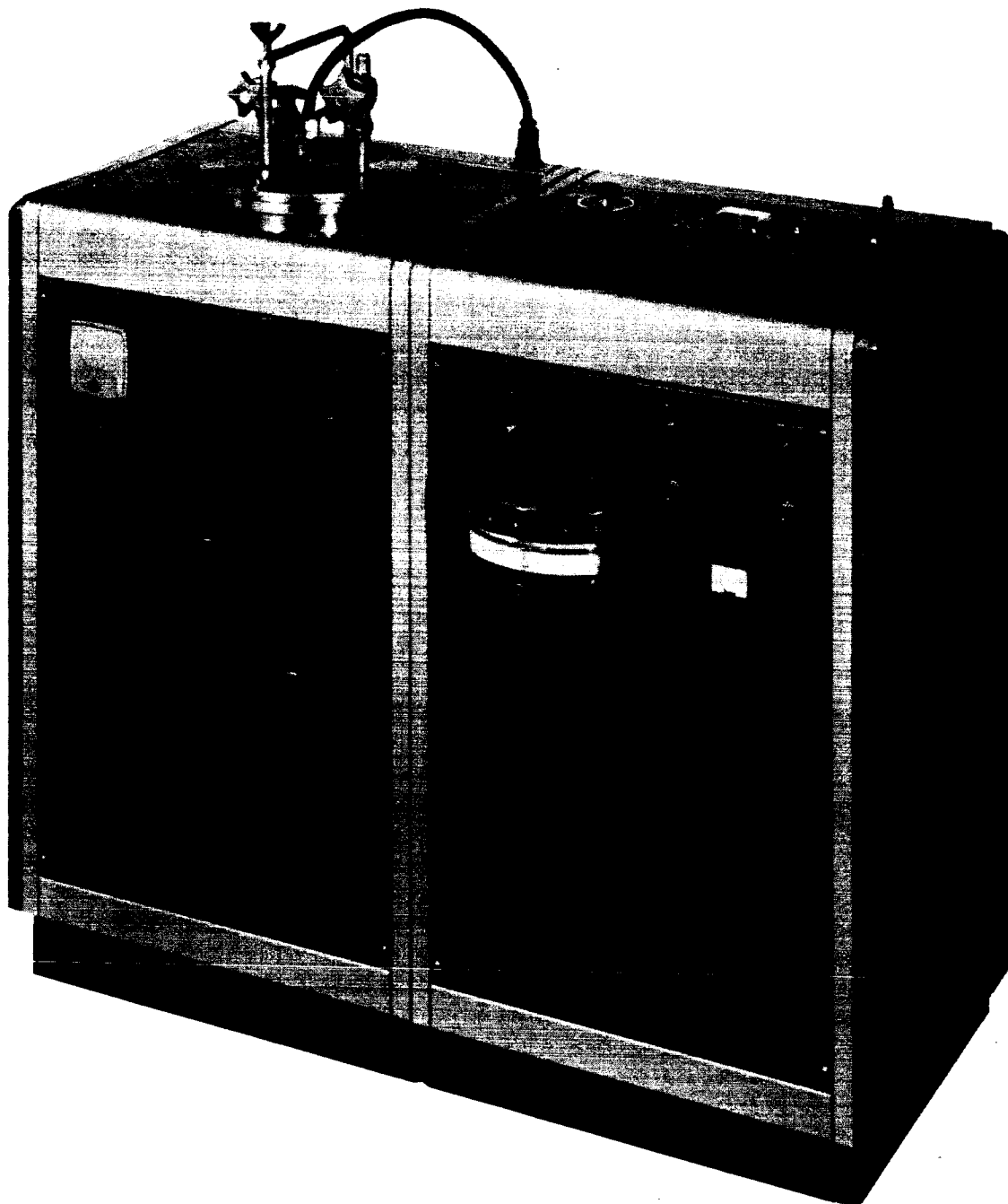
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Figure **4.**

DYNATECH
QUANTITATIVE ADIABATIC CALORIMETER
QTA SERIES

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17 TUDOR STREET
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A calibration run was first performed on the heaters alone to determine their MCp values over the temperature range. These values are subsequently subtracted from the MCp values obtained from the heater plus sample assembly, thus yielding the MCp values of the sample and, finally, the sample specific heat.

3.3 Data Reduction

The time temperature history was automatically recorded during the test on an extended range strip chart recorder, and sample heater power was monitored and recorded at intervals of 10 minutes during the course of the run. Knowing the speed of the chart and measuring local slopes of the curve, the data could be readily reduced.

$$\text{Since} \quad Q = MCp \; \frac{dT}{dt}$$

$$\text{or} \quad Q = MCp \; \frac{\frac{dmv}{dt}}{\frac{dmv}{dt}}$$

where mv = thermocouple emf in millivolts and dmv/dt is the slope of the time temperature curve; and dmv/dt is a physical constant for the thermocouple material used.

$$\text{Therefore,} \quad MCp = \frac{Q}{\frac{dT}{dt}}$$

and:

$$(MCp)_{Sample} = (MCp)_{Sample + Heater} - (MCp)_{Heater}$$

$$\text{or} \quad Cp_{Sample} = \frac{(MCp)_{Sample + Heater} - (MCp)_{Heater}}{M_S}$$

Since this is a dynamic test, points may be reduced anywhere along the temperature range. For ease, it may be noted that the particular points reduced were chosen at every half millivolt between one and six millivolts for chromel-alumel thermocouples which were used in this test.

3.4 Sample Calculation

At $208^{\circ}F$, the slope of the curve (dmv/dt) is 2.166. Since

$$\frac{dmv}{dT} = .02308,$$

$$\frac{dT}{dt} = 93.85$$

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The heat input to the sample was 8.283 Btu/hr. Therefore, the sample and heater

$$MCp = \frac{Q}{\frac{dT}{dt}} = \frac{8.283}{93.85} = .08826.$$

Since the heater MCp calibration value is .04588, the sample

$$MCp = .08826 - .04588 = .04238$$

and the sample weight, $m = .127$ pound.

Thus,

$$Cp_{Sample} = \frac{.04238}{.127} = .333 \text{ Btu/lb } ^oF$$

3.5   Test Results

Temperature, ^oF	Specific Heat, Btu/lb, ^oF
88	.325
99	.350
121	.365
154	.385
175	.387
187	.366
208	.333
251	.342
296	.340

Section 4

DENSITY

4.1 Method of Testing

The density of the plastic material was determined at room temperature by measuring the weight of water displaced by a sample of the plastic material when it was placed in water in a calibrated pycnometer. The variation in volume, with temperature, of the plastic material was determined by measuring the volume change occurring in a dilatometer filled with the sample and mineral oil. From these data the coefficient of cubical expansion of the plastic material was calculated. The densities at elevated temperatures were calculated from the room temperature density and the coefficient of cubical expansion.

4.2 Test Procedure

From the sample of plastic material submitted, strips were cut 0.25" x 0.25" x 2.5" in a milling machine. The density of the material was determined at room temperature by measuring the displacement of water in a Kimble 15123, 50 ml pycnometer. The density of a mineral oil was also measured in this pycnometer. The volumetric expansion and contraction of mineral oil, and mineral oil and a sample of plastic material was measured in a 10 ml dilatometer, having a stem graduated to 0.01 ml. Temperatures were maintained by a well-stirred, mineral oil-filled thermal bath in the range 75-303°F. From these measurements the coefficients of cubical thermal expansion of the oil and of the sample were determined. From these data and the basic density determined with the pycnometer, the densities at the required temperatures were calculated.

4.3 Data Reduction

The density, ρ_S, of the sample is:

$$\rho_S = \frac{\text{weight of sample}}{(\text{pycnometer volume}) - \left(\frac{(\text{weight water} + \text{sample}) - (\text{weight sample})}{\text{density of water}} \right)}$$

The coefficient of cubical expansion, α, is defined by the relationship:

$$V_2 = V_1 (1 + \alpha \Delta t)$$

where

V = volume, ml; V_1 at temperature 1 and V_2 at temperature 2

Δt = temperature change, °C

α = coefficient of cubical expansion, $°C^{-1}$

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When a sample of plastic material and a quantity of oil are placed together in a pycnometer the sum of their volumes, V_Σ, changes so as to define an apparent coefficient of cubical expansion, α_Σ. The sum of volumes, V_Σ, may be equated to the individual volumes, of oil, V_o, and the sample, V_S:

$$V_{\Sigma_2} = V_{\Sigma_1} (1 + \alpha_\Sigma \Delta t) = V_{o_1} (1 + \alpha_o \Delta t) + V_{S_1} (1 + \alpha_S \Delta t)$$

This permits the coefficient of cubical expansion of the sample to be calculated:

$$\alpha_S = \frac{V_{\Sigma_1} \alpha_\Sigma - V_{o_1} \alpha_N}{V_{S_1}}$$

The volumes of oil and sample placed in the dilatometer are known from their weights and densities. The term $V_{\Sigma_1} \alpha_\Sigma$ is determined from a plot of dilatometer volume vs temperature:

$$\frac{V_{\Sigma_2} - V_{\Sigma_1}}{\Delta t} = V_{\Sigma_1} \alpha_\Sigma$$

The thermal expansion of the glass dilatometer is accounted for by the fact that the true coefficient of expansion of a contained material is equal to the apparent coefficient plus the coefficient of expansion for the container material.

After determination of the base density and coefficient of cubical expansion of the sample the density at other temperatures is calculated through the relationship:

$$\rho_{S2} = \frac{\rho_{S1}}{1 + \alpha_S \Delta t}$$

4.4 Sample Calculation

A sample of the plastic material 2.50" x 0.249" x 0.246" weighed 5.674 gms, yielding a rough density of 139 lb/ft^3.

The volume of the pycnometer was found to be 53.5011 ml by determining the weight, (corrected to vacuum), of water required to fill the pycnometer at 23.6 ^oC. When a second sample of plastic material weighing 5.279 gms, (corrected to vacuum), was placed in the pycnometer, 51.2301 ml of water were required to fill it at 24.6 ^oC. From these data the density of the plastic material was calculated to be 2.324 g/ml or 145.1 lb/ft^3. By filling the pycnometer with 47.008 gms, (corrected to vacuum), of mineral oil at 24.6^oC the density of the

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oil was found to be 0.8786 g/ml. The dilatometer was found to contain 9.709 ml at 26.1°C when filled to the zero mark by calibration with weighed amounts of water. The scale was found to read high, requiring the correction

$$\text{(actual volume)} = 0.9882 \text{ (volume read)}$$

The dilatometer was filled to an arbitrary level with mineral oil and the volume registered on the scale was observed as temperature was varied. Within the precision of the experiment the volume was found to change linearly with temperature. In another test , a sample of plastic material was placed in the bulb and the dilatometer was filled again to an arbitrary level with mineral oil. Volume changes with temperature were again observed to be linear. A first trial of temperature changes was rejected because air was released from the pores of the sample during the heating period. The necessary data and the coefficients of cubical thermal expansion determined from them are:

	Filling	
	Oil Only	Oil and Sample
Weight of oil, g	8.534	6.509
Weight of plastic sample, g	——	5.279
Temperature, °C	26.5	25.1
Volume change, ml/°C	7.03×10^{-3}	5.75×10^{-3}
Coefficient of cubical thermal expansion, °C^{-1}	7.2×10^{-4}	2.1×10^{-4}

In carrying out these calculations the coefficient of cubical thermal expansion of the dilatometer glass (Pyrex) was taken to be 9.75×10^{-6} °C^{-1}.

These measurements were carried out by R. L. Wentworth and C. F. Bruce. Data are recorded on notebook pages 909-928.

4.5   Test Results

t, °F	density, lb/ft^3
75	145.1
100	144.7
125	144.3
150	143.8
175	143.4
200	143.0
250	142.2
300	141.4

NAS-7a

THERMAL CONDUCTIVITY vs. TEMPERATURE

FIG #5

THERMAL CONDUCTIVITY , BTU/HR.FT.°F
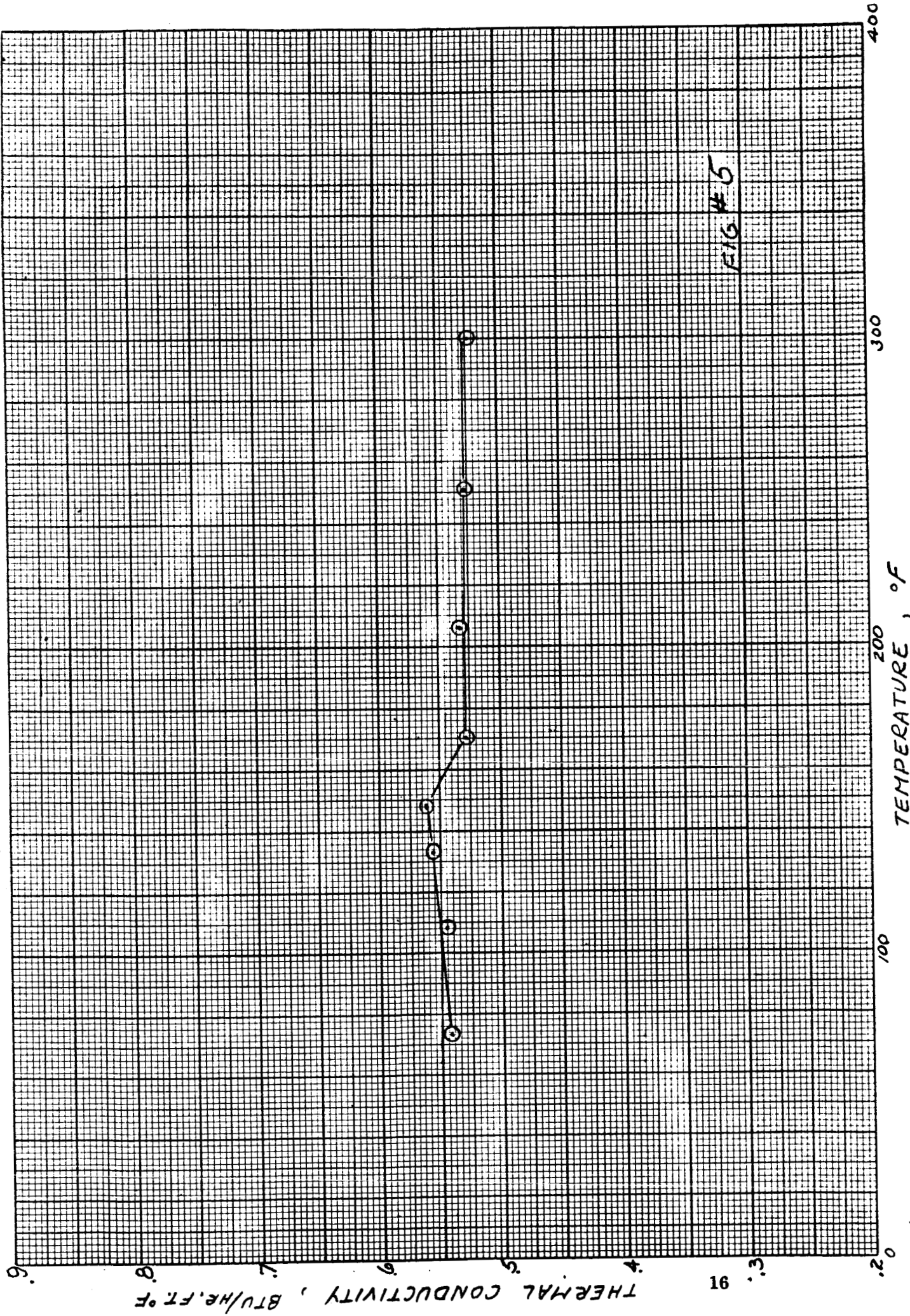
TEMPERATURE , °F

NAS-16

SPECIFIC HEAT VS. TEMPERATURE

FIG. #6

SPECIFIC HEAT, BTU/POUND, °F

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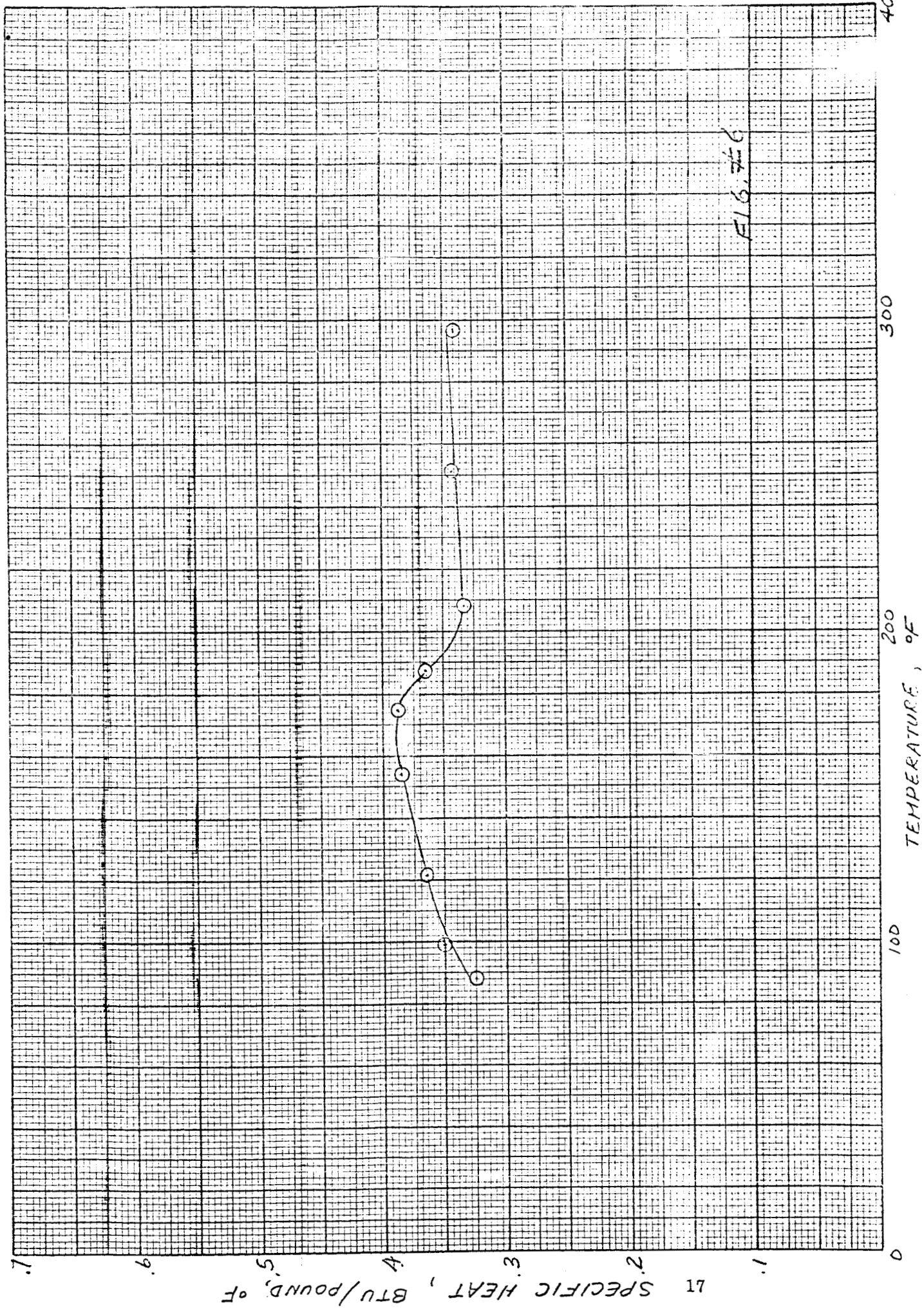
TEMPERATURE, °F

NAS-7C

DENSITY VS. TEMPERATURE
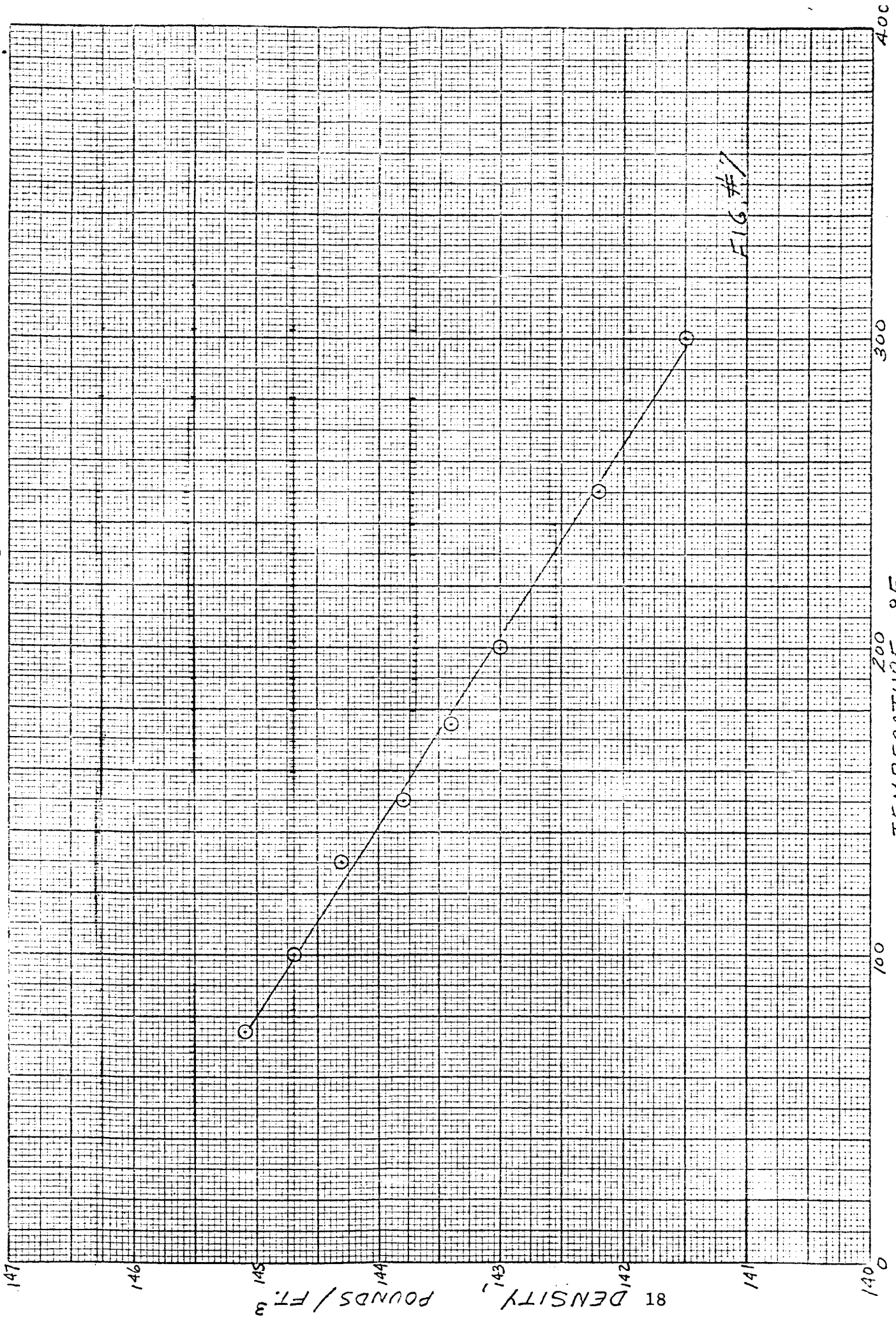

FIG. #1

TEMPERATURE, °F

DENSITY, POUNDS/FT.³ 18

Section 5

DISCUSSION OF RESULTS

The test results are plotted in Figures 5 through 7. In comparing the thermal conductivity and specific heat values, it would appear that a transition point is present at about 175°F, since each curve displays a discontinuity in slope at this temperature. In looking solely at the thermal conductivity curve, the difference in values at the transition point is 5.5%. If specific heat data were not taken, it would be reasonable to assume that the conductivity were a straight line having a very slight downward slope. The largest deviation of any point from this curve would be 3.5% for the 150°F point. This would be well within the test accuracy of 5%.

However, the specific heat curve follows the same shape as the thermal conductivity curve. It has a slowly increasing slope to about 165°F, falls until it reaches 200°F, and then slowly rises up again. This correlation between the two sets of data is quite consistent.

During the density measurements, outgassing from the sample occurred at 150°F. This was manifested by gas bubbling through the oil in the dilatometer. For this reason, the density test was rerun after it was felt that the sample had been completely degassed. Since the gas pockets within the material are random in size, number, and location, it is quite difficult to judge their effect on the conductivity and specific heat measurements. However, it is reasonable to consider this as a second or possibly third order effect.

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