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DYNATECH CORPORATION

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SOME THERMOPHYSICAL PROPERTY MEASUREMENTS OF POROUS CERAMIC "GLASSROCK"

Submitted to:

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Langley Station
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Prepared by:

L. C. Hoagland
R. L. Wentworth
P. C. Dustin

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DYNATECH CORPORATION
17 Tudor Street
Cambridge, Massachusetts 02139

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TABLE OF CONTENTS

Section		Page
1	INTRODUCTION	1
2	THERMAL CONDUCTIVITY MEASUREMENTS	2
	2.1 Test Procedure	2
	2.2 Data Reduction	6
	2.3 Sample Calculation	7
	2.4 Test Results	8
	2.5 Discussion of Results	8
3	SPECIFIC HEAT MEASUREMENTS	10
	3.1 Test Procedure	10
	3.2 Data Reduction Method	13
	3.3 Sample Calculation	14
	3.4 Test Results	14
	3.5 Discussion of Results	18
4	DENSITY AND POROSITY MEASUREMENTS	19
	4.1 Summary	19
	4.2 Density	19
	4.3 Porosity	19
	4.4 Coefficient of Thermal Expansion	19
	4.5 Discussion	21

LIST OF ILLUSTRATIONS

Figure		Page
1	TCFC-R8 Series	3
2	Test Stack Assembly TCFC-R8 Series	4
3	Test Stack Arrangement	5
4	Graph of Thermal Conductivity vs. Temperature	9
5	QTA N-7-ER	11
6	Sample and Guard Arrangement, Adiabatic Calorimeter	12
7	Specific Heat vs. Temperature	17

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Section 1

INTRODUCTION

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This report describes the measurements performed on a sample of a porous ceramic described as "Glassrock". These tests were conducted by Dynatech Corporation for the NASA, Langley Research Center under Contract No. NAS 1-4758.

The thermal conductivity was measured from room temperature to 700°F by means of a comparative test. Over the same temperature range the variation of specific heat with temperature was measured.

A determination of the variation of density with temperature was performed using techniques which rely upon measuring the cubical expansion of the material.

The following sections of this report present the methods of testing and the procedures followed in this test program.

author

Section 2

THERMAL CONDUCTIVITY MEASUREMENTS

2.1 Test Procedure

The thermal conductivity was determined by the Comparative Method in which the thermal conductivity of the 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. Data was taken for eight points between room temperature and 700°F. The temperature points were run in the sequence starting from room temperature.

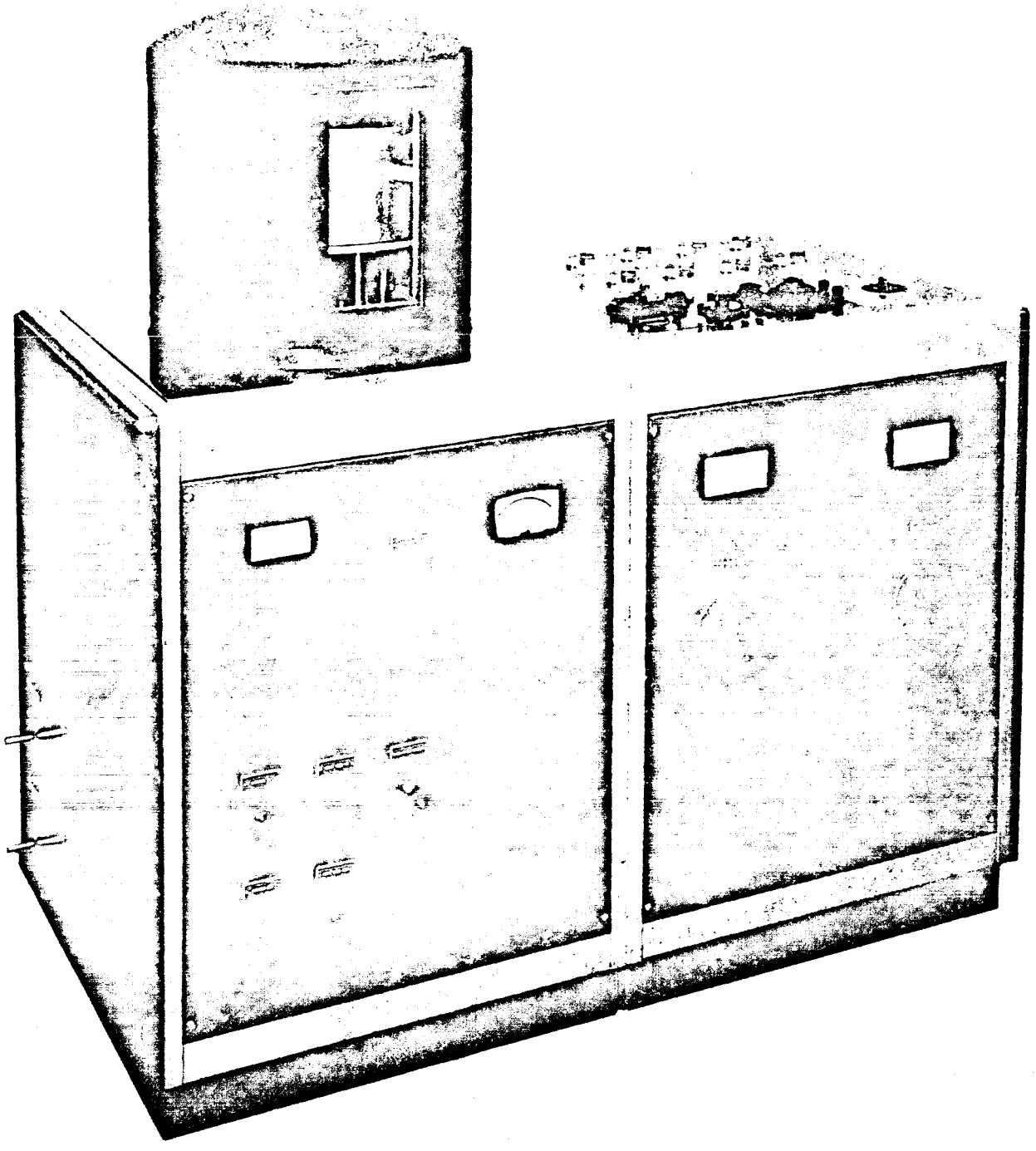
The test stack configuration is shown on figure three. The sample was provided with thermocouple grooves machined into the faces and was instrumented with chromel Alumel thermocouples. The heat meters were instrumented in a similar manner also with chromel alumel thermocouples.

The sample was sandwiched between two identical Pyroceram #9606 heat meters and a heat flux was directed through the stack by a heater placed at the top of the assembly. The temperature level of the stack was maintained by an auxiliary heater placed between the bottom heat meter and a cooled heat sink. Water was used as the coolant in the sink for all points except for the room temperature point where liquid nitrogen was used. Insulating paper was placed between the auxiliary heater and the heat sink in order to allow easy control of the auxiliary heater temperature. Aluminum plates, 1/8 inch thick, were inserted between the heater and the heat meters so as to provide an evenly distributed heat flux across the entire contact surface. To preclude side heat losses, the stack was surrounded by a cylindrical guard heater, the temperature of which was maintained close to the average sample temperature, and the stack assembly was covered with a low conductivity powdered insulation.

Figure 1 shows the conductivity instrument used for this test. Figure 2 shows a typical test stack being assembled; the outer guard heater appears to the left of the stack.

To minimize thermal contact resistances, all surfaces were coated with a thin layer of silicone grease. A load of approximately 5 pounds was applied to the top of the test stack to further ensure good thermal contact.

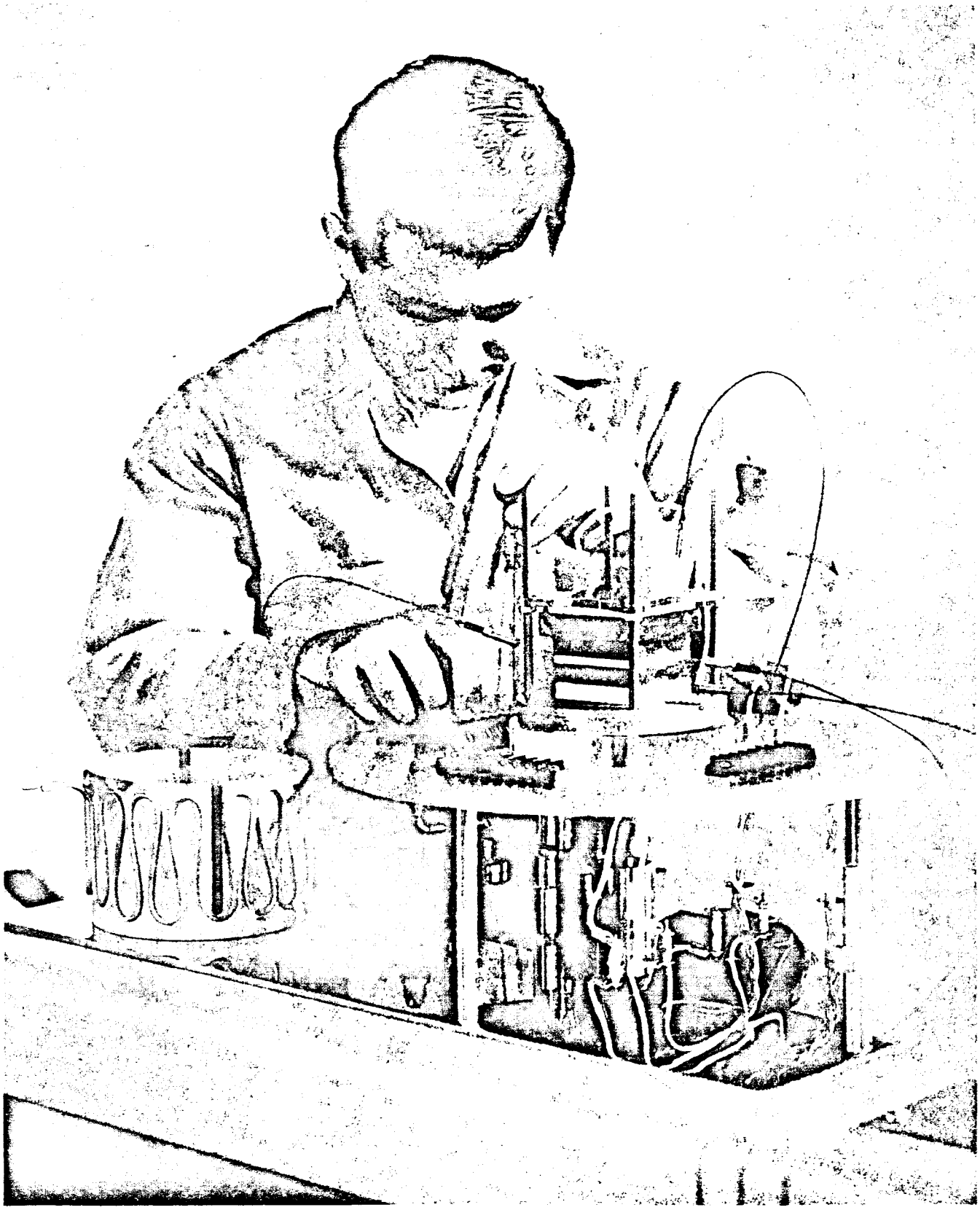
The tests were performed by heating the test stack to the required temperature level and allowing the stack to reach an equilibrium or steady state condition. Each test was concluded when it was established that this condition had been maintained for a minimum period of one hour.



DYNATECH
COMPARATIVE THERMAL CONDUCTIVITY APPARATUS
TCFC-R8 SERIES

DYNATECH CORPORATION
17 TUDOR STREET
CAMBRIDGE 39, MASSACHUSETTS

Telephone: 617-868-8050



Test Stack Assembly
TCFC-R8 Series
Comparative Thermal Conductivity Apparatus

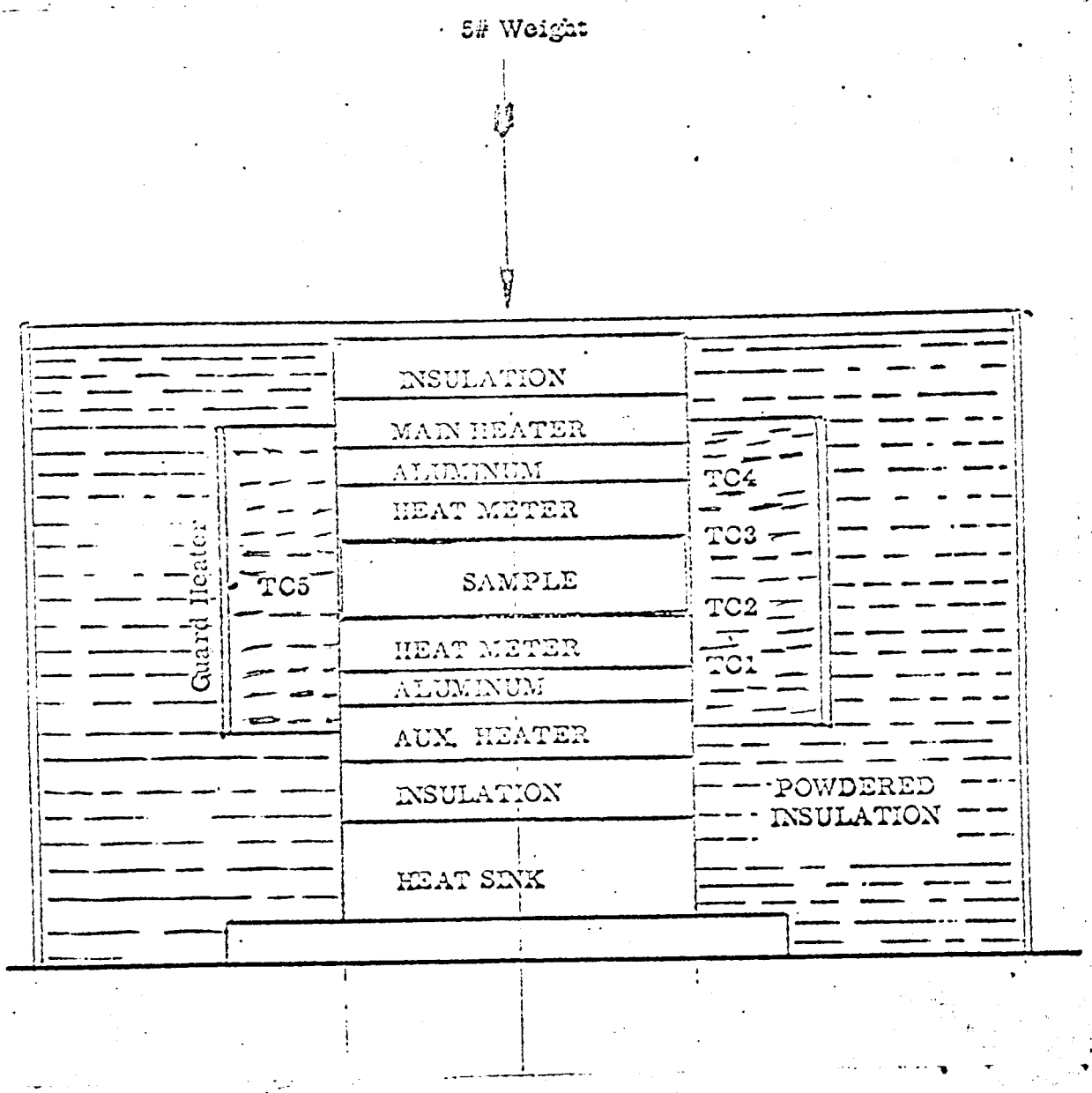


Figure 3. Test Stack Arrangement

The measurements were made under a bell jar which was evacuated by means of a mechanical vacuum pump. For all tests the vacuum was maintained in the range of 30 to 100 microns.

A separate determination was made at one temperature (approximately 125° F.) of the variation of thermal conductivity with absolute pressure. Three measurements were taken.

2.2 Data Reduction

By assuming a unidirectional heat flow under steady state conditions, the heat flux through both heat meters and the sample is equal, i.e.

$$\left.\frac{Q}{A}\right\}_{THM} = \left.\frac{Q}{A}\right\}_{Sample} = \left.\frac{Q}{A}\right\}_{BHM}$$

or

$$\left.\frac{k\Delta T}{\Delta X}\right\}_{Top\ HM} = \left.\frac{k\Delta T}{\Delta X}\right\}_{Sample} = \left.\frac{k\Delta T}{\Delta X}\right\}_{Bottom\ HM}$$

where

ΔX = thickness
ΔT = temperature difference
k = thermal conductivity

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

$$\left.k_s\right\}_T = k_{THM} \cdot \frac{\Delta X_s}{\Delta X_{THM}} \cdot \frac{\Delta T_{HM}}{\Delta T_s}$$

$$\left.k_s\right\}_B = k_{BHM} \cdot \frac{\Delta X_s}{\Delta X_{BHM}} \cdot \frac{\Delta T_{BHM}}{\Delta T_s}$$

$$k_s = \frac{k_{s_T} + k_{s_B}}{2}$$

2.3 Sample Calculation

A sample calculation is now presented to illustrate the data reduction procedure.

Test Data

Sample: Porous ceramic "Glassrock"
Temperature Point: 75°F
Thermocouples: Chromel Alumel
Temperature Differences - ΔT

Bottom heat meter:	5.7°F
Sample:	17.5
Top heat meter:	4.8

Thickness (ΔX) - distance between thermocouples

Bottom heat meter:	0.476 inch
Sample:	0.238
Top heat meter:	0.476

Conductivity of heat meters at mean temperature of meter

Heat meter	T_{mean}	"k" (Btu/ft-hr °F)
BHM	61 °F	2.33
THM	92	2.28

Heat meter material: Pyroceram #9606

$$k_s = k_{HM} \circ \frac{\Delta T_{HM}}{\Delta T_s} \circ \frac{\Delta X_s}{\Delta X_{HM}}$$

$$k_s\Big\}_{BHM} = 2.33 \times \frac{5.7}{17.5} \times \frac{.238}{.476}$$

$$= 0.38 \text{ Btu/hr-ft- F}$$

$$k_s\Big\}_{THM} = 2.28 \times \frac{4.8}{17.5} \times \frac{.238}{.476}$$

$$= 0.313 \text{ Btu/hr-ft- °F}$$

$$k_s\Big\}_{Avg} = \frac{0.38 + .313}{2}$$

$$= .346\Big\}_{\text{at } T_{mean} = 79°F}$$

2.4 Test Results

The thermal conductivity is listed in the following table. In addition the results are presented in a graphical form on figure 4 for the variation of k with temperature.

Mean Sample Temperature	"k" (Btu/hr-ft-°F)
79 °F	0.346
135	0.360
218	0.402
312	0.437
402	0.536
500	0.561
583	0.620
706	0.836

Upon completion of this phase of the program the variation of thermal conductivity with absolute pressure was measured for one temperature point.

Temperature (Mean sample):	130 °F
Pressure (Torr)	k: Btu/hr-ft-°F
760 mm	0.505
380	0.495
190	0.495

2.5 Discussion of Results

With the exception of the 400 °F testpoint all of the data lie on a smooth curve. This point is approximately 9 1/2 % high. The usual accuracy for this type of test is between ± 5 and ± 10%. It is felt that these results are within the 10% allowance.

The tabulated difference of thermal conductivity with absolute pressure for a constant temperature of 130 ° F shows little change. It seems also that within the same experimental error the conductivity is independent of vacuum.

Section 3

SPECIFIC HEAT MEASUREMENTS

3.1 Test Procedure

The specific heat was determined as a function of temperature over the range -75 °F to + 700 °F in the Dynatech Model QTA-N7 quantitative adiabatic calorimeter (Figure 5). In this instrument the specific heat of the sample is determined by measuring the temperature response of the sample to a known heat input. The sample is inserted into a sample heater assembly which is instrumented with thermocouples for temperature measurement and suspended inside an adiabatic jacket. (See Figure 6). The adiabatic jacket is constantly maintained at the same temperature as the sample assembly by means of a jacket or guard heater coupled to an automatic control system. Thus, there is very little heat transfer between the sample assembly and its environment. To reduce further the heat transfer between the same and its environment, a mechanical pump vacuum (1 to 100 microns) is provided between the sample assembly and the adiabatic jacket to eliminate convection and all surfaces are highly polished to reduce radiation.

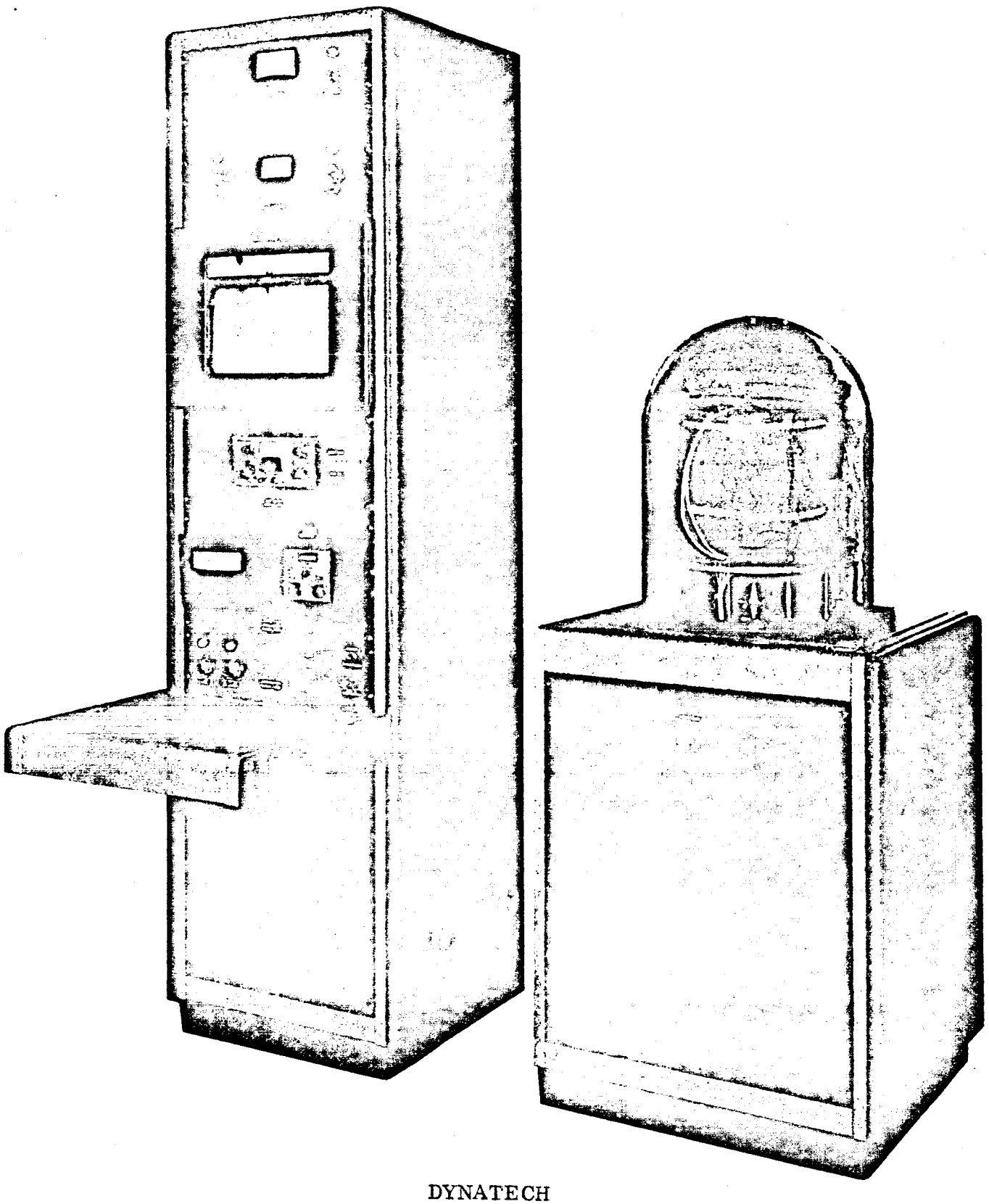
The mode of operation used for the tests described here consisted of operating the sample heater at a constant power input throughout the test and recording the sample temperature versus time response to this heat input over the temperature range of interest.

A continuous record of sample specific heat versus temperature is obtained by operating the instrument with a continuous, constant power input to the sample assembly and recording the sample temperature as a function of time throughout the temperature range of interest. The temperature response (and hence the $M C_p$ product) of the sample container is first determined by operating the instrument over the full temperature range without a sample. A sample of unknown specific heat is then inserted into the sample container and the unit is again operated over the desired temperature range. The specific heat of the sample is then calculated from the equation:

$$q = \left[(M C_p)_{\text{container}} + (M C_p)_{\text{sample}} \right] \frac{dT}{dt}$$

where

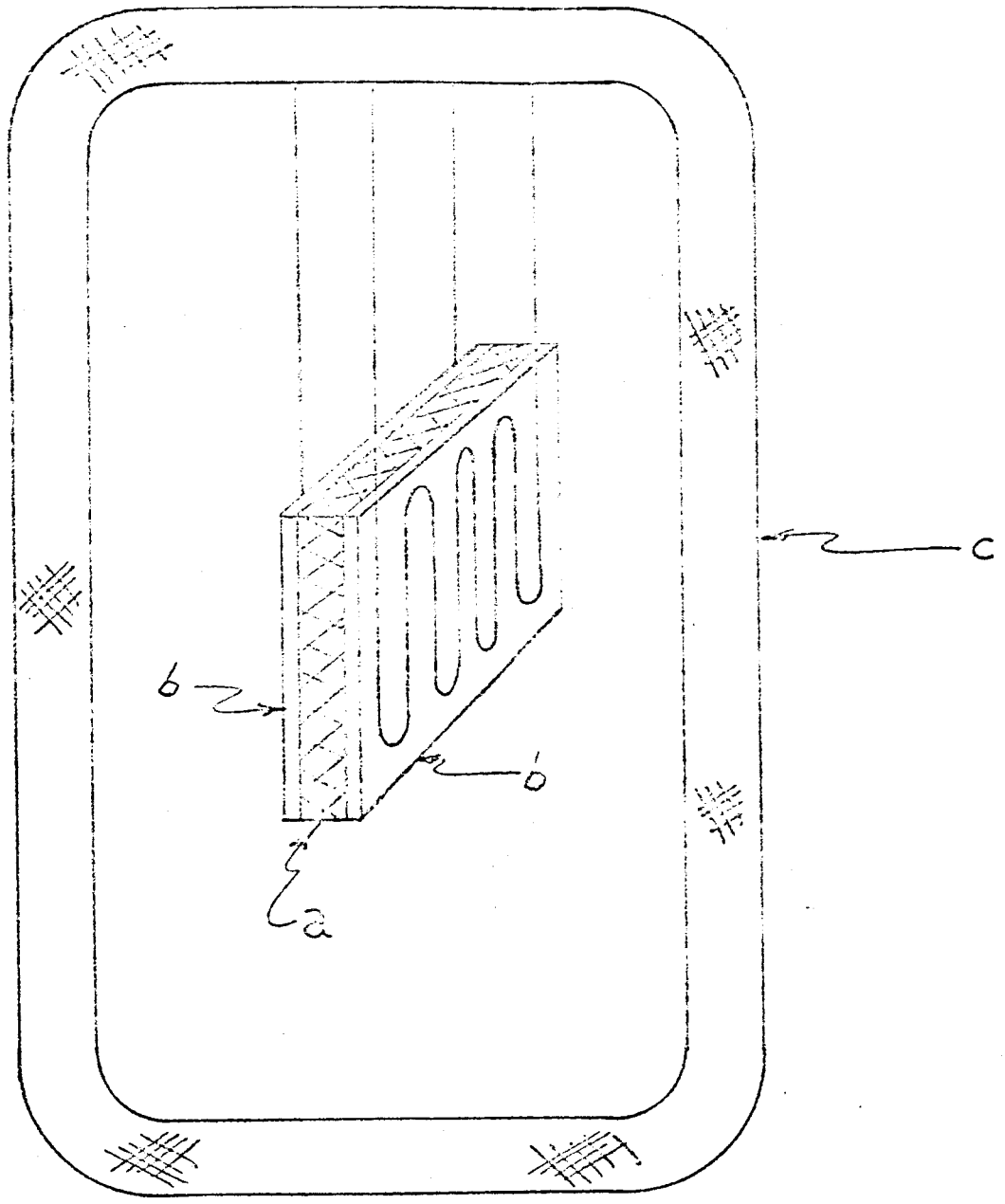
q	=	heat input to sample container
$M C_p$	=	mass-specific heat product
T	=	temperature
t	=	time



DYNATECH
QUANTITATIVE ADIABATIC CALORIMETER INSTRUMENT
QTA-N7-ER SERIES

DYNATECH CORPORATION
17 TUDOR STREET
CAMBRIDGE, MASSACHUSETTS 02139

Telephone: 617-868-8050



LEGEND: a- SPECIFIC HEAT SAMPLE
b- SAMPLE HEATER PLATES
C- COPPER GUARD & HEATER

SAMPLE & GUARD ARRANGEMENT
ADIABATIC CALORIMETER
FIG. 6

The sample container MC_p is determined from a container calibration run and dT/dt is determined by measuring the slope of the recorded temperature-time trace at a number of discrete points (temperatures).

3.2 Data Reduction

The specific heat apparatus was operated in a manner described in the previous section. The primary data obtained from the test run was:

1. A continuous recording of sample thermocouple emf versus time over the temperature range of the test,

2. Readings of sample heater power at 10 or 15 minute intervals thoughout (although the sample heater power was very nearly constant throughout each test, the periodic readings permit accounting for slight variations in power observed).

Data reduction was made in accordance with the following equation as indicated in the previous section.

$$q \text{ Btu/min} = EI \times \frac{3.4152}{60} = \left[(MC_p)_{\text{container}} + (MC_p)_{\text{sample}}\right]\frac{dT}{dt}$$

E = sample heater voltage - volts

I = sample heater current - amps

Solving for the sample specific heat:

$$C_{P\text{sample}} = \left[\frac{EI \times 3.4152/60}{dT/dt\ (°F/\text{min.})} - (MC_p)_{\text{container}}\right] \frac{1}{M_{\text{sample}}}$$

M = lb; C_p = Btu/lb-°F; dT/dt = °F/min.

The timewise temperature gradient dT/dt is determined from the recorded trace of thermocouple emf versus time as follows:

$$\frac{dT}{dt} = \frac{d(\text{emf})}{dt} \div \frac{d(\text{emf})}{dT}$$

The first quantity on the right d (emf)/dt is determined by measuring the slope of the recorded emf - time trace while the second term d (emf)/dT is evaluated as a function of temperature from the NBS thermocouple emf tables*.

Although a continuous recording of sample temperature versus time is obtained from this method of testing (thereby providing data for a continuous curve of C_p versus temperature), data reduction (calculation of C_p) is accomplished at a limited number of discrete points, only enough to define a smooth continuous curve for C_p versus temperature.

3.3 Sample Calculation

Since under this program only one specimen was tested the sample calculation shown presents the complete reduced data for the program.

3.4 Test Results

The following table is extracted from the sample calculation and summarizes the results. Values are given from the specific heat of the sample versus the sample temperature. These are the values shown in columns one and eight of the sample calculation.

The results are further presented in the form of a graph. From this the trend with increasing temperature can be readily observed.

* Reference Table for Thermocouples. Circular 561, National Bureau of Standards.

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TEST RESULTS

Sample:	Porous Ceramic - "Glassrock"
Run on:	April 19, 1965
Sample Weight:	46.948
Thermocouples:	Copper-Constantan

Temperature °F	Specific Heat Btu/LB-°F
120.5	0.196
162	.201
201	.210
239.5	.222
278.5	.228
313	.237
348	.241
382	.247
416	.249
449	.248
481.5	.250
513.5	.252
545	.254
576	.255
607	.259
637	.258
667	.264

SAMPLE CALCULATION AND DATA REDUCTION

(1) Temperature mv	(1) Temperature °F	(2) Slope of Recorder Trace $\frac{dmv}{dt}\left(\frac{mv}{min}\right)$	(3) Thermocouple Characteristic $\frac{dmv}{dT}\left(\frac{mv}{°F}\right)$	(4) $\frac{dT}{dt}\left(\frac{°F}{min}\right) = \frac{(2)}{(3)}$	(5) $q = \frac{3.4152}{60}$ E1 (Btu/min)	(6) $MC_{p_{sample}} + MC_{p_{heater}} = \frac{(5)}{(4)}$	(7) $MC_{p_{heater}}$	(8) $C_{p_{sample}} = \frac{(6)-(7)}{M_{sample}}\ \frac{Btu}{lb-°F}$
2.0	120.5	.0682	.02876	2.630	.1552	.0583	.038	.196
3	162	.0642	.02479	2.590	.1545	.0596	.0388	.201
4	201	.0645	.02570	2.510	.1535	.0611	.0393	.210
5	239.5	.0650	.02660	2.440	.1535	.0629	.0399	.222
6	276.5	.0657	.02738	2.400	.1535	.0639	.0404	.228
7	313	.0659	.02812	2.345	.1535	.0654	.0409	.237
8	348	.0663	.02870	2.320	.1535	.0661	.0412	.241
9	382	.0670	.02938	2.280	.1535	.0673	.0418	.247
10	416	.0675	.02995	2.255	.1535	.0680	.0423	.249
11	449	.0684	.03048	2.240	.1535	.0685	.0429	.248
12	481.5	.0688	.03100	2.220	.1535	.0691	.0433	.250
13	513.5	.0692	.03144	2.200	.1535	.0698	.0438	.252
14	545	.0700	.03190	2.190	.1535	.0701	.0438	.254
15	576	.0705	.03235	2.180	.1535	.0704	.0442	.255
16	607	.0705	.03279	2.150	.1535	.0713	.0445	.259
17	637	.0710	.03312	2.145	.1535	.0715	.0448	.258
18	667	.0712	.03351	2.125	.1535	.0724	.0450	.264

Test Run of: April 19, 1965
Sample Weight: 46.948 grams
(no weight loss during test)
Thermocouples: Copper constantan

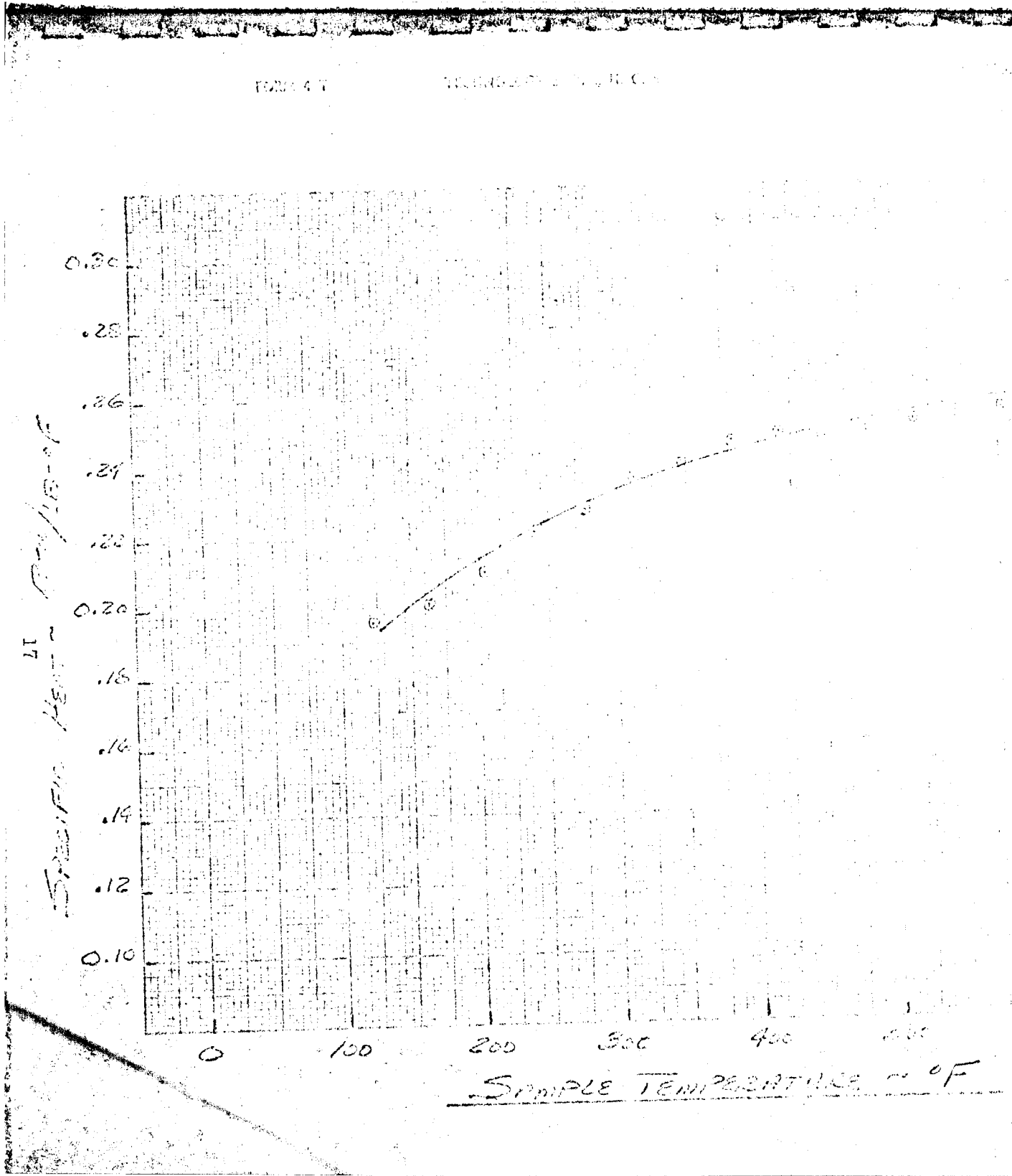

Fig. 7. Specific Heat vs. Temperature

3.5 Discussion of Results

The specific heat measurements were made with the sample under a vacuum of about 100 microns absolute pressure rather than 1 atmosphere air as desired. The following approximate calculation for the ratio of the heat capacity of the air occupying the voids (at 1 atmosphere pressure) to the heat capacity of the solid material shows that the contribution of the air is negligible in comparison.

$$\frac{M C_{p\ air}}{M C_{p\ solid}} = \frac{\rho_{air} C_{p\ air} P}{\rho_{solid} C_{p\ solid}} = \frac{.075\ LB/FT^3 \times .24\ BTU/LB\,^\circ F \times .18}{0.19\ BTU/LB\,^\circ F}$$

$$= .017$$

M = Mass

where

C_p = specific heat

ρ_{air} = density of air at 1 atmosphere, room temperature

ρ_{solid} = bulk density of solid

P = Porosity

Since this ratio is very small compared with unity the specific heat values for the sample measured under vacuum conditions are also valid for the sample when the pores are filled with gas at 1 atmosphere.

Section 4

DENSITY AND POROSITY MEASUREMENTS

4.1 Summary

The true density of the material at 75°F is 138.3 lb/ft^3. The porosity of the material is 18% on a total apparent volume basis. The coefficient of cubical expansion of the material is so small that the thermal expansion of the material cannot be distinguished over the temperature range 75° to 300°F in a dilatometer filled with mineral oil.

4.2 Density

The true density of the material was determined at room temperature by measuring the displacement of water by a sample of material in a Kimble No. 15123, 50 ml pycnometer. The following data were obtained:

Temperature:	28°C
Empty volume of pycnometer	32.90 cc
Weight of sample:	25.125 gram
Volume of water to fill pycnometer with sample present:	41.65 cc
Density:	2.210 g/cc = 138.3 lb/ft^3

4.3 Porosity

The porosity of thematerial was determined by measuring the proportion of water absorbed by a sample according to the procedure of Mottlau and Fisher (Analytical Chemistry 34 (6), 14 (May, 1962)). In this technique a comminated sample of the material is placed in a flask and water is added slowly from a microburette while stirring the sample. The end-point is determined when the surface of the particles remains wet and agglomeration of the particles persists in spite of continued stirring. The porosity is then calculated as:

$$\text{porosity} = \frac{\text{(volume of water absorbed)}}{\text{(volume water absorbed)} + \text{(true solid volume of sample}}$$

The data obtained by this technique were as follows:

	Trial A	Trial B
Weight of Material, g	2.479	2.778
Volume Water absorbed, cc	0.250	0.275
Porosity, %	18.8	18.0

4.4 Coefficient of Thermal Expansion

The volumetric expansion of a sample of material immersed in mineral oil was measured in a 10 ml dilatometer having a stem graduated in 0.01 ml. Temperatures were maintained by a well-stirred, mineral oil-filled bath in the range 75-300 °F. The procedure is that described by Beckedahl (J. Research National Bureau of Standards 42, 145 (1949)).

The dilatometer was found to contain 10.100 cc at 28 °C when filled to the zero mark with a weighed amount of water. The scale was found by measuring the volumes of water contained at various levels in the stem. The dilatometer was filled to an arbitrary level with mineral oil, and the volume registered on the stem was observed as the temperature was varied over the range 83 ° to 300 °F. The volume was found to change linearly with temperature. In another test a cylindrical sample of material weighing 12.487 grams and 4.239 grams of oil were placed in the dilatometer. Prior to taking readings the contents of the dilatometer were subjected to vacuum and were carried through a cycle of temperatures up to 300 °F. Following this the volume changes as a function of temperature over the range 86 ° to 300 °F were observed. The sample volumes computed as a result of these measurements (corrected for the thermal expansion of the Pyrex dilatometer) were:

Temperature, °F	Density, lb/ft^3
86.0	137.6
124.6	137.7
140.2	137.7
157.4	137.8
180.4	137.6
210.8	137.6
228.5	137.7
249.0	137.5
277.0	137.4
300.0	137.1

Discussion

The accuracy of the basic density determination is estimated to be better than 5 parts in 1000. The accuracy of the gravity measurements is estimated to be ± 1%. The results of the experiments to determine the volumetric expansion of the material do not indicate any systematic change in volume over the temperature range investigated. It was concluded that the coefficient of cubical expansion of the material is so small the contribution of the material to the volume change observed in the dilatometer was negligible. Consequently the determination of the coefficient of cubical expansion of the material cannot be determined within the precision of the test method employed. Since the mineral oil possesses a coefficient of cubical expansion of approximately 7×10^{-4} °c^{-1} above room temperature, the material is estimated to possess a coefficient of cubical expansion of 10^{-5} °c^{-1} or less.