

Measurement of the Thermal Properties
of Carbonaceous Materials

By

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ABSTRACT

Three parameters of interest for thermal processing of carbonaceous materials have been studied. The mean specific heat capacity from room temperature to temperatures of 500° to 900°F have been shown to range from 0.54 to 0.41 for coal from the Pittsburgh Seam and 0.28 to 0.31 for its LTC char. These measurements were made in a simple high temperature calorimeter. The thermal conductivity of coal-char-pitch briquets was found to follow a pattern similar to coal being coked, exhibiting a 20-fold increase in value from room temperature to hot coke temperature. The conductivities were measured by a very direct method using a compensated conductometer. An estimation of the surface film heat transfer coefficient for fluidized solid heating of an immersed sphere was carried out experimentally using the unsteady state heating curve. Values for h of the order of 30 Btu/hr ft² F° were obtained which are consistent with the range generally found in the literature for wall and tube coefficients.

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INTRODUCTION

Many of the processes for the treatment of coal, either in present day use or under development, involve thermal conversion. One such process, the forming of coal-char briquets, is discussed in a companion paper. It is obviously desirable, in the development of such processes, to have available thermal data on the materials being handled. The work described here was designed to supply such thermal data for carbonaceous materials of interest. Heat capacity measurements have been made on a bituminous coal from the Pittsburgh Seam and its low temperature char. Thermal conductivity measurements have been carried out on a coal-char-pitch briquet. Finally, an estimate has been made of the heat transfer film coefficient from a fluidized heating medium to a spherical briquet. This latter work was carried out so that the thermal parameters could be applied to the calculation of heating rates for carbonaceous briquets, an application which is described in another paper.

Literature Survey

The heat capacity data for coal recorded in the literature have been reviewed by Clendenin, et al.³ In this review a correlation equation for the specific heat capacity of moisture-free coal is presented, based on data for 23 American bituminous coals. The equation relates heat capacity to temperature and volatile matter content as follows:

$$C = 0.200 + 0.0015 \text{ V.M.} + 0.00088 t$$

where V.M. = percentage volatile matter on a moisture-free basis
t = temperature, °C

This equation cannot be expected to hold into the temperature range in which coal becomes plastic. In fact the very limited data included in the correlation for temperatures above 100°C make its use above about 150°C an extrapolation that may not be justified.

The heat capacity of a bituminous coal within its plastic range may be a somewhat variable quantity depending upon the definition of the quantity and the method of measurement. The literature contains numerous papers devoted to the measurement of two heat quantities. One, the total "heat of carbonization", is defined to include all the sensible and reaction heats involved in carbonization; the other, often called the "heat of decomposition", refers to the reaction heats only after the sensible heats have been removed by calculation or by the use of a differential calorimetric method. The most notable of the data in the literature

are those of Terres and co-workers¹¹⁻¹⁵, Davis, et al.⁴⁻⁶, Weyman¹⁶, and Burke and Parry². Work has also been reported upon the heat capacity of chars produced from coal by thermal treatment¹³. In general, the data found in the literature fall short of providing applicable and consistent data for materials typical of Pittsburgh Seam bituminous coal.

Published information on the thermal conductivity applicable to materials with which we deal is also very meager. By the time our work had progressed to conductivity measurements, we were primarily interested in compactions of coal, char and pitch. Data on coals and cokes are summarized by Clendenin, et al.³, but again these data are principally low temperature measurements below 100°C. Some data by Terres¹¹ cover the whole coking range, but since the values are reported as "mean" conductivities they probably represent average values over a wide temperature range. Millard⁶ has published conductivity data for beds of granulated coal (and coke, subsequently produced) over a 100-900°C temperature range. While the experimental unit used by Millard was adequate for measurements on packed beds, it was not applicable to solid compactions. Work by Birch and Clark¹ on the conductivity of rock samples up to 900°F provided the basic design for the conductometer used in our work.

No work has been found in the literature which predicts directly the surface film heat transfer coefficient between fluidized solids and small spheres immersed in the bed. Numerous investigations and correlations are available for heat transfer coefficients between fluidized solids and the walls of the containing vessel and some work is reported for internal tubes. No effort has been made to critically evaluate all this information, but the range of values for h thought to be applicable is from 20 to 50 Btu/hr ft² F°.

Heat Capacity Measurements

Method

A variation of the standard calorimetric method of mixtures was used in this work. The bituminous coal samples under study were known to undergo irreversible changes upon thermal treatment in the temperature range of interest; for this reason, it was essential that a high temperature calorimeter be used in which the sample is heated and the calorimeter cooled. This procedure is exactly the opposite of the one generally used in which a hot sample is cooled in a cold calorimeter. The method adapted measured the total heat required to raise the sample from room temperature to the temperature investigated; sensible and reaction heats are thus lumped together. This is the important quantity, however, for most practical applications.

The use of a high temperature calorimeter made it impractical to reduce the thermal losses to a negligible level. Instead, it was necessary to control and standardize the losses at a reasonably high level. In an effort to keep the unit as simple as possible, an unsteady state method was used, in which the effect of an added sample on the normal cooling curve of a heated metal block was observed. This method proved adequate, and allowed the use of simple equipment; however, considerable care and extensive calculation were needed to produce these satisfactory results.

The heart of the calorimeter was an aluminum block 1-1/2" o.d. by 2-7/8" long with an axially bored hole 3/4" in diameter and 2-1/2" deep. Six holes were drilled on a 1-1/8" circle in the top of the block; two of these holes were tapped

for support rods, and the other four were used as thermocouple wells 1/2", 1", 1-1/2" and 2" deep, respectively. This aluminum block was wound with a resistance wire heater and suspended by small iron rods from a transite disc. The block was hung down into a glass Dewar flask, which in turn was placed in a tube furnace. The furnace temperature was controlled at a level near the operating temperature of the block to reduce the heat loss. A "seal-ring" of transite, slightly smaller than the inside diameter of the Dewar flask, was placed between the block and its supporting disc. This "seal-ring", which contained an electric heater powered from a constant voltage source, was provided to reduce the end losses upward from the block and to improve the temperature symmetry of the system. A long glass tube was used to provide an inlet path for the test samples. A nitrogen purge was metered in near the bottom of the Dewar flask to insure an inert atmosphere. The calorimeter is shown assembled in Figure 1.

The thermocouple circuits used to get a sensitive measure of the temperatures of the system consisted of two 4-element thermo-piles made of iron-constantan. The four hot junctions of one thermo-pile were placed in the holes drilled in the aluminum block, and because of their spacing in the block yielded a good average temperature directly. These junctions had to be insulated electrically from the block, and this was done with a bead of aluminum cement to which a little water-glass had been added. The hot junctions of the second thermo-pile were held about 1/16" away from the inner wall of the Dewar flask, spaced at 90° around the block at a level near the middle of the block. The sensitivity of each of these thermo-piles, between 500° and 900°F, is of the order of 0.12 millivolt per degree Fahrenheit, so that by measuring each output to the nearest 0.01 millivolt with a Leeds and Northrup portable potentiometer the temperatures within the block and external to it could be determined to the nearest 0.1°F.

In the normal experimental procedure, the outer furnace and the seal ring heater were used to establish an elevated "steady state" temperature in the calorimeter block. From this level the block heater was used to raise the block temperature to a level about 20°F higher. After conditions were steady at this upper temperature level, the block heater was switched off, and the block allowed to cool towards its lower steady state temperature. After about three minutes had elapsed a powdered solid sample, held in a small aluminum foil cylinder about 5/8" diameter and 1-1/2" long, was dropped into the block. The block and external temperatures were read alternately, each on a 30-second schedule, until the cooling rate became relatively small so that less frequent readings were sufficient.

The calculation of the effect of the added sample required that a "normal cooling curve be determined for each temperature level without any sample addition or other external stimulus. By graphical differentiation of the block temperature curve during "normal" cooling, the block cooling rate was plotted as a function of the instantaneous temperature difference between the block and its surroundings. At each level a good straight line relation existed, as predicted by the simple Newtonian cooling law; a typical plot is shown in Figure 2. The slope of this line depends on the properties of the system, but the intercept depends, principally, on the input to the seal ring heater. When used as a correction of the observed cooling after a sample has been added to the calorimeter block, this Newtonian plot must be extrapolated into the heating domain where $\frac{dt_{\text{block}}}{dt}$ is positive and

$(t_{\text{block}} - t_{\text{external}})$ is negative. To prove that this extrapolation was reasonably accurate, a dry ice pellet was added at one temperature level to force the block temperature below the external temperature; the observed heating rate for the block was just that predicted by extrapolation.

A typical pair of temperature curves for a run in which a solid sample was added is shown in Figure 3. For selected values of time the temperature driving force, ($t_{\text{block}} - t_{\text{external}}$), was noted and the normal cooling rate determined from the Newtonian plot. The integral expression, $\int \left(\frac{dt_{\text{block}}}{d\theta} \right)_{\text{normal}} d\theta$, was

determined graphically for each run, and served as a correction for the observed temperature drop of the block. The corrected temperature effect of the sample was the algebraic difference of the observed temperature drop and this integral normal temperature drop: The corrected temperature effect should approach a steady final value after a reasonable period of time, as shown in a typical curve in Figure 4. In some cases in which no final steady value was approached, it was necessary to shift the Newtonian cooling plot, holding its slope constant, until the total effect was forced to level off. This forced balance was equivalent to assuming that the seal ring heater input varied while the insulating properties of the system did not. This assumption appeared reasonable since the corrections were random and no aging trend was noticed during the period spent on measurements at a given temperature level.

After the total corrected effect of a sample on the calorimeter block was found, the remaining calculations were familiar. Powdered Al_2O_3 , a substance of known heat capacity, was run to establish a calorimeter constant. The heat capacities of other materials were determined by using this calibration as a reference.

Results

The heat capacity of a Pittsburgh seam coal (38.6% V.M. and 7.0% ash, from the Arkwright mine in West Virginia) was determined to a maximum temperature of 800°F. Swelling and frothing of the coal made it impossible to use higher temperatures. A char produced from this coal by fluidized carbonization at 950°F was used for measurements to 890°F. This Arkwright char contained 13.2% V.M. Magnesium oxide was used as a secondary standard to check the accuracy of the method, by comparison between observed and literature values for heat capacity from 650° to 800°F. The results for these three materials is shown in Figure 5; the mean specific heat capacities are, of course, averaged from room temperature to the final temperature plotted.

Most points on the curves shown in Figure 5 are the average of two or more determinations. The variability of individual values from the mean was generally less than 2% for the heat stable materials, but rose as high as 5% in one instance for the coal sample in the middle of the plastic temperature range. The agreement between the observed and literature values for MgO adds confidence in the method.

For comparison with data found in the literature, two other curves are shown in Figure 5. One represents the values that would be predicted by applying the equation derived by Clendenin, et al.³; the other curve is from data by Porter and Taylor⁹, these data being the highest temperature values included in the correlation of Clendenin. No comparable data are available for char.

The data obtained here by use of a high temperature calorimeter apply to the particular case in which the sample is heated very rapidly and the products of carbonization are carried out of the system. This corresponds quite well to the pattern in fluidized low temperature carbonization of coal, the application for which these data were originally determined, but may differ substantially from the heating regime in some other processes. Reaction effects, particularly from extensive secondary reaction of the tar products, may cause some variance in total specific heat capacity under other treatment conditions.

Thermal Conductivity Measurements

Method

Absolute thermal conductivities of a material are best determined by direct measurement of the temperature gradient which exists for a known rate of heat transfer across a unit area of material under steady state conditions. From the Fourier heat transfer equation

$$\frac{dq}{d\theta} = k A \frac{dt}{dx}$$

the thermal conductivity can be calculated directly. The thermal conductometer used in this work was modeled after one described by Birch and Clark¹, and allowed us to measure these needed factors for compactions made of char, coal and pitch.

A cross-sectional view of the conductometer is shown in Figure 6. The location of the lead-in wires has been omitted. The apparatus was symmetrically square about the vertical axis. The specimen was a flat slab approximately 2" square and 1/2" thick, cut from a single large briquet, or formed of close fitting rectangular sections from small briquets. It was placed between the heater block and cooling plate, both of which are made of copper to assure an even temperature distribution. A 30-gauge nichrome wire heating element in the heater block held its temperature about 10°C above that of the cooling plate when a steady state was reached. Measurement of this temperature difference across the specimen was made with thermocouples placed at positions indicated by the heavy dots. Provided that there is no heat transfer from the heater block other than through the specimen, the conductivity is simply calculated. The condition of no extraneous heat loss from the block was approached as closely as possible by use of a thermal guard dome of copper which covered the heater block as shown. The guard dome contained a heating element by which the dome's temperature was adjusted to equal the temperature of the heater block. Heat losses to the surroundings then came from the guard dome and not from the heater block. Lateral heat losses from the specimen were minimized by the guard ring of insulating material (transite) that surrounds the specimen. Small ceramic spacers prevented accidental metal-to-metal contact between the dome and heater block.

The entire conductometer was enclosed in a steel box which was purged with nitrogen to prevent oxidation of the copper or the specimen at high temperatures. The entire unit was immersed in a fluidized sand bath to provide the controlled elevated ambient temperatures.

Stable power for the heater block was provided by two six-volt storage batteries in series. The current was manually controlled with rheostats to give the desired temperature differential across the specimen. With currents of 100-200 milliamperes in the block heater, there was no discernible drift in the current during the course of a measurement. The less critical power for the guard dome was provided by a Variac with a rheostat for fine control. A type K Leeds and Northrup potentiometer was used for accurate measurement of the thermocouple potentials and the voltage and current of the heater block. Voltage measurements across a standard one-ohm resistor in the heater circuit gave direct measurements of the heater current.

Results

Thermal conductivities were determined for sections of briquets made from coal, char and pitch. The composition was 25% coal (Pittsburgh Seam from Montour

mine in Western Pennsylvania), 63.5% low temperature char produced from this coal by fluidized carbonization at 950°F and 11.5% pitch. These briquets were pre-coked at several temperature levels, their conductivity measured at that temperature, and then also at lower temperatures. This procedure separately demonstrates both the effect of coking on conductivity at a particular temperature and the temperature dependence of conductivity of specifically coked briquets. These data are plotted in Figure 7.

Trustworthy results could not be obtained at temperatures above 1600°F on the fully coked briquets because adequate fluidized sand furnace temperature control was not realized. However, the values obtained at other temperatures on this fully coked material allow a reasonable extrapolation of the curve to 1800°F.

Figure 8 shows a comparative plot of the present data with those presented by Millard⁸ for coal. Millard made no attempt to explain the difference in the conductivities of the two coals, which came from different mines. It is readily seen from the plot that there is no radical difference in the briquet conductivity as compared to the coals. Both materials show a very large increase in conductivity with temperature and a marked increase with degree of coking.

Estimation of Surface Film Coefficient for Heating With Fluidized Solids

The application of the thermal parameters reported above very often requires the estimation of a surface film heat transfer coefficient pertinent to the thermal system to be used. One of the thermal arrangements of interest for the carbonization of briquets is the heating with circulating, hot fluidized solids. Considerable work has been done by the authors and their associates on the treatment of carbonaceous briquets in this type of system. In an effort to bring order and predictability to some of the results noted, and further to check the validity of assumed values of the heat transfer film coefficient used in machine calculations made on this type system (reported in a companion paper), an experimental method for the estimation of the film coefficient was tested. This experimental method is simple, especially so under the special conditions in which it has been applied here. The results obtained represent only a rather cursory examination of the transfer coefficient, but it is felt that the method deserves description.

The method used was to observe the temperature rise of spherical objects after immersion in a constant temperature, fluidized sand bath. Although, generally speaking, the use of an unsteady state system leads to a relatively low degree of precision, it is also true that these measurements are rapid and may be made quite well in a very simple system, much simpler than would be needed for steady state measurements.

Examination of mathematical expressions for this system showed that in order to relate these results to the film coefficient it was necessary to restrict our attention to materials and temperature ranges for which the thermal diffusivity may be treated as a constant. In these cases, the Fourier equation can be solved analytically. A solution which has been published may then be applied to this problem^{7,10}. This solution reduced further to a very simple form for the case of objects with a high thermal conductivity such as aluminum or copper ($Nu \ll 1$).

Under these specific conditions the temperatures of the center and surface, respectively, of a sphere of radius r_0 at initial temperature T_1 which is suddenly surrounded by a medium at a constant temperature T_0 are given by the equations

$$Y_{r_0} = \left(\frac{T_c - T}{T_0 - T_1} \right)_{r_0} = e^{-3Nu\theta}$$

$$Y_{r=r_0} = \left(\frac{T_0 - T}{T_0 - T_1} \right)_{r=r_0} = \sqrt{3Nu} e^{-3Nu\theta}$$

The Nusselt number, Nu , is defined as

$$Nu = \frac{hr_0}{k}$$

and θ is a dimensionless time = $\frac{\alpha t}{r_0^2}$ or $\frac{k t}{\rho c_p r_0^2}$

The Nusselt number can be very simply determined in this case by plotting experimental values of $\ln Y$ versus θ . A straight line should be obtained whose slope is $-3 Nu$. This is true for both the center and surface temperatures.

Experimentally, all measurements were made by plunging an aluminum sphere into an 8" diameter fluidized sand bath. An aluminum sphere was chosen so that the simplified form of the Fourier solution equation could be applied; i.e., the thermal conductivity of aluminum is high and almost exactly independent of temperature, and the thermal diffusivity is relatively constant. It was assumed that the nature of the sphere's surface was unimportant to the result, so that values of h calculated here would be applicable to carbonaceous briquets.

Two thermocouples were used to sense the temperatures of interest in the sphere, one attached to the surface and one placed in a small hole bored to the specimen center. Small diameter thermocouple wire (28 gauge) was used to minimize error caused by heat conduction down the wire. A third thermocouple in the fluidized sand bath was used to sense and control its temperature. All three temperatures were recorded on a multi-point strip chart recorder. All the tests used a nominal fluidized sand temperature of 1000°F.

Five tests were run using a 2" sphere. Four of these tests were made using a fluidization velocity (superficial) of 0.6-0.7 feet per second; one test used a reduced linear velocity of 0.34 fps. Single tests were made with a 1" and a 3" sphere. A typical plot of the temperature history recorded by the thermocouple placed at the center of the sphere is shown in Figure 9.

A plot of the temperature approach data used for the estimation of some of the film coefficients, h , is shown in Figure 10. Straight lines are obtained as predicted by the special solution for the basic Fourier equation. Similar results may be obtained when the surface temperature is treated in a similar manner, but the surface temperature is more difficult to measure with precision.

The lines in Figure 10 do not show the unit value of y -intercept as predicted by theory. Part of the cause of this discrepancy is thought to be a thermocouple error caused by conduction of heat down the thermocouple wire, thus producing high temperature readings. It can easily be shown that the total heat conducted down the wire is a negligibly small fraction of that conducted through the aluminum. Further, it can be shown that if the thermocouple error is proportional to the temperature difference between the surroundings and the center of the sphere (a logical assumption), then only the intercept of the Fourier solution equation is shifted and its slope is unaffected.

The calculated values of the film coefficient, h , are listed in Table I. The precision of the replicate tests on the 2" inch sphere is not the best, but the results are consistent and reasonable. The test made at a reduced fluidizing velocity shows a somewhat lower value for h , which is also reasonable.

Table I

Experimental Values of Heat Transfer Coefficient
Between Fluidized Sand and Aluminum Spheres

Thermal Parameters for Calculations: $k = 117 \text{ Btu/hr. ft. } ^\circ\text{F}$
 $a = 3.33 \text{ ft.}^2/\text{hr.}$

Run No.	Sphere Diameter	Fluidization Velocity	Fluidized Sand Temperature	Calculated Heat Transfer Coefficient, h
Al-1	3"	0.72 ft/sec	950°F	38 Btu/hr ft ² F°
Al-2	2"	0.72	1000°F	31
Al-3	1"	0.72	1050°F	27
Al-4	2"	0.61	985°F	36
Al-5	2"	0.61	990°F	34
Al-6	2"	0.34	980°F	23
Al-7	2"	0.61	950°F	30

The values measured here might be expected to lie in the upper range of values reported because of the high density of the fluidized sand; on the other hand, the fluidization velocities used were rather low, especially for a dense solid, and this factor would lower the value of h . The average value of h measured here is about 30 Btu/hr. ft.² F°, and thus well within the range of 20-50 Btu/hr. ft.² F° chosen for the calculations of heating rates of carbonaceous briquets in fluidized solids systems.

SUMMARY

The various thermal data reported here are not entirely comparable in terms of materials and temperature ranges. However, they all bear directly on the problem of heat transfer to carbonaceous briquets containing coal, char and pitch. The heat capacity data measured for coal and char may be combined with values in the literature for pitch to provide composite values applicable to briquets. The thermal conductivity data were obtained directly on briquet samples. The heat transfer film coefficient, h , was estimated for a metal of high thermal conductivity (aluminum) for simplicity, but no reason is apparent why this external factor cannot be applied directly to the heating of carbonaceous shapes of much lower conductivity. These data have been used for machine computations of briquet heating rates as reported in a companion paper.

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FIGURE 1
CALORIMETER

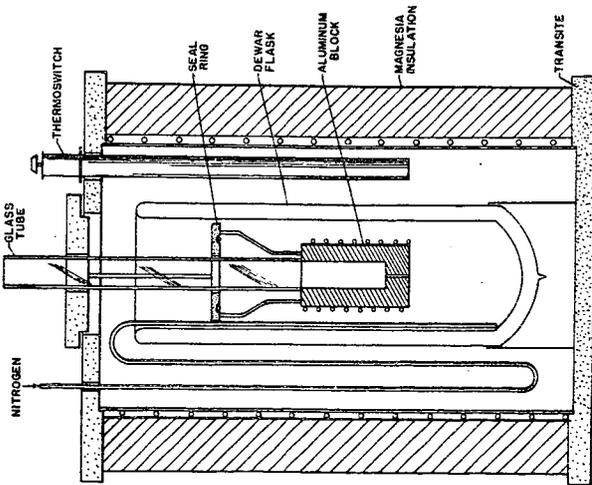


FIGURE 2
'NORMAL' COOLING CORRECTION LINE

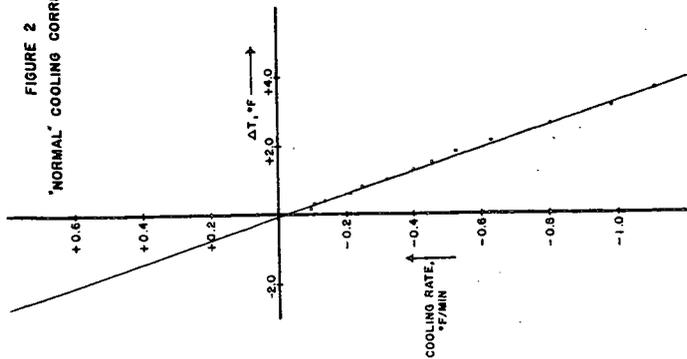


FIGURE 3
OBSERVED DATA FOR A CALIBRATION TEST

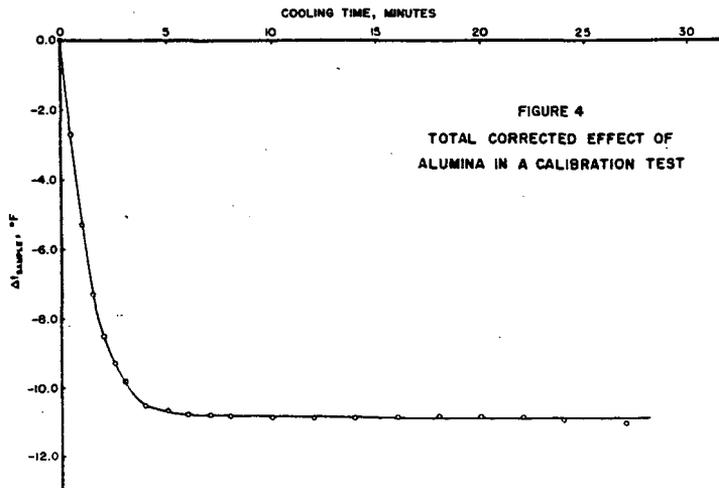
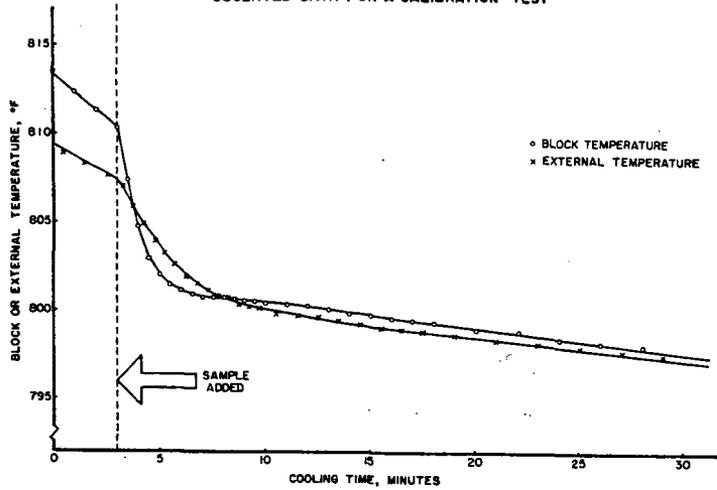


FIGURE 4
TOTAL CORRECTED EFFECT OF
ALUMINA IN A CALIBRATION TEST

FIGURE 5
MEAN SPECIFIC HEAT CAPACITIES OF COAL AND CHAR

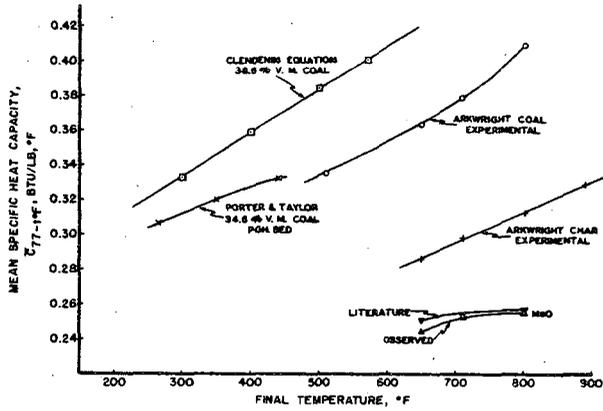


FIGURE 6
THERMAL CONDUCTOMETER

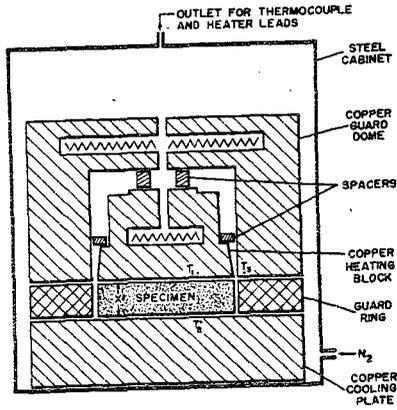


FIGURE 8
COMPARATIVE CONDUCTIVITIES
OF COAL AND CHAR-COAL-PITCH BRIQUETS

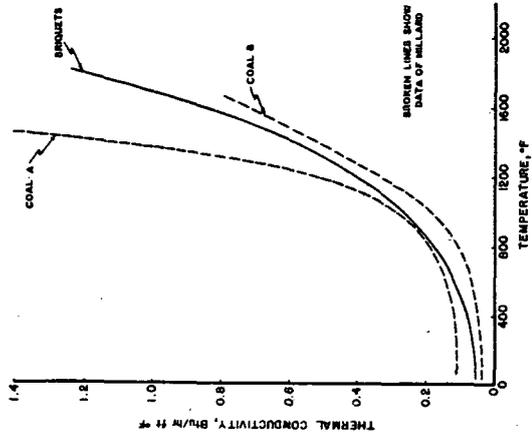


FIGURE 7
THERMAL CONDUCTIVITY OF
CHAR-COAL-PITCH BRIQUETS

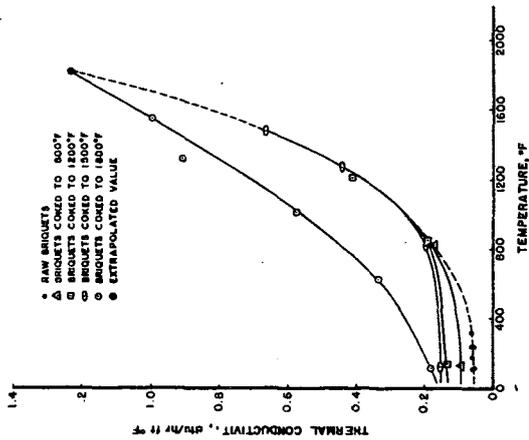


FIGURE 9
 CENTER TEMPERATURE OF ALUMINUM SPHERE
 HEATED BY FLUIDIZED SAND BED

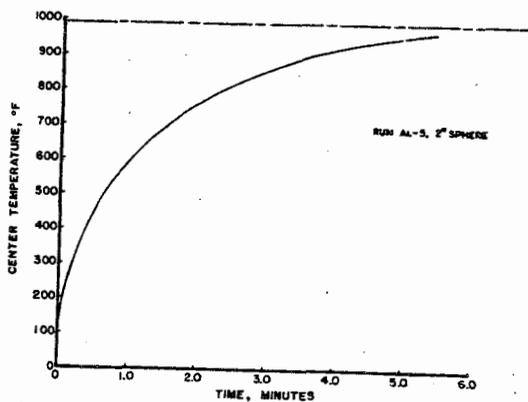


FIGURE 10
 TEMPERATURE APPROACH PLOT FOR
 DETERMINATION OF THE FILM COEFFICIENT
 IN FLUIDIZED BED

