

DESIGN AND OPERATION OF A VACUUM FURNACE CALORIMETER  
TO OBTAIN THE SPECIFIC HEATS OF SOLIDS

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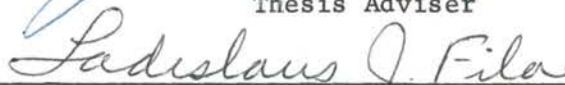
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## CHAPTER I

### INTRODUCTION

The demands of science today on the field of heat transfer are ever increasing as men of science delve further into the world of hypersonic flight, atomic power, rockets and satellites. Advanced research is almost always concerned either directly or indirectly with some form of heat transfer.

Because of this seemingly inseparable relationship between advanced technology and heat transfer problems, it becomes a necessity to know the various thermal properties of materials as soon as they are developed in order that the materials may be utilized most advantageously in new designs.

Heat measurements have always been the most difficult of all laboratory problems. This difficulty causes considerable delay in the determination of thermal constants which in turn hinders the scientist or engineer in his choice of a material for a particular assignment.

This investigation was undertaken to study a method for obtaining thermal data of materials rapidly; this would indeed be an extremely valuable tool for engineers and scientists.

A high temperature vacuum furnace was designed, constructed, and operated in the Mechanical Engineering Laboratory of Oklahoma State University. The prime purpose was to develop a method of rapidly

obtaining the specific heat data of metals of sufficient accuracy to be usable in critical design problems.

The theory of the comparative calorimeter method is presented in order that the reader may understand the reasoning behind the design of the apparatus. A detailed description of the design is given along with adequate mention of all auxiliary equipment used in the investigation. Also presented is a chapter on the operating procedures followed in the investigation.

This investigation, while not a complete solution to the problem, is thought to have laid the groundwork for a more critical evaluation of the method and to have paved the way for continued research. Perhaps with further development, temperatures much higher than the 1500°F maximum temperature investigated can be attained with improved accuracy in the experimental results.

One may envision the practical use of an apparatus of this type in a metallurgy research laboratory where it may be possible to determine the specific heat data for new metals in a matter of hours after they are developed, thereby allowing their most efficient usage in critical design.



## CHAPTER II

### PREVIOUS INVESTIGATIONS

A survey of the history of calorimetry reveals that most of the methods in use today were devised early in the development of the science of thermodynamics. Before 1800 Black (1) was credited with discovering a "method of mixtures." His method consisted of mixing equal amounts of different substances at different temperatures and allowing them to reach an equilibrium temperature. The characteristic of the substance which determined the equilibrium temperature was known as specific heat. Regnault (2) developed Black's method and thus attained a higher degree of accuracy.

The method of mixtures is the most widely used method. As used today, this method consists of dropping the substance under investigation from a furnace with measured and controlled temperature into a calorimeter operating at or near room temperature. The change in enthalpy between the furnace temperature and the calorimeter temperature is thus determined. The specific heats may then be calculated since

$$C_P = \left( \frac{\partial H}{\partial T} \right)_P$$

Three advantages of the method of mixtures are as follows:

1. It is direct and reasonably rapid.
2. The results are precise within the limits of reproducibility

in behavior of the substance.

3. It depends upon no extraneous assumptions.

Two disadvantages of the method of mixtures as given by Kelly (3) are:

1. The method is not always adequate for obtaining heat effects at transition points, especially if the heat of transition is small.

2. It may happen that the substance under investigation undergoes a change in state on heating and does not revert to its original state on cooling in the calorimeter.

Another general type of calorimetry, the method of cooling, was devised by Dulong and Petit (4). This method involves comparing the cooling rate of the experimental substance with the corresponding rate for a substance of known specific heat under conditions presumed to be the same.

Black extended his method of mixtures with the earliest form of ice calorimeter. It consisted merely of a block of pure ice, free from bubbles, in which a cavity was hollowed out. The mouth of this cavity was covered over by another slab of ice, so that a chamber was obtained, which was enclosed on all sides by ice at the melting point. A known weight of the substance under examination was heated to some definite temperature and dropped into the cavity. After equilibrium was established, the block was dried out and the mass of ice melted then determined by weighing. The specific heat of the substance could then be calculated.

Lavoisier and Laplace modified Black's ice calorimeter in such a

way as to avoid the necessity of large blocks of ice.

With the advent of Bunsen's (5) ice calorimeter very good accuracy was obtainable with the "phase-change" calorimeters. His instrument determined the amount of melting by volume rather than by weight. The apparatus consisted of a test tube which was fused into a large glass bulb partly filled with pure boiled water and partly with pure boiled mercury. A mercury column extended from the bottom and up through an adjustable connection to a calibrated glass tube.

To use the instrument, some of the water in the large bulb was caused to freeze on the side of the test tube by passing cold alcohol through the test tube. After freezing the layer of ice on the test tube the whole apparatus, except the scale and opening to the test tube, was submerged in snow and allowed to reach equilibrium. The body whose specific heat was to be measured was dropped into the test tube and equilibrium again allowed to establish. Since ice upon melting contracts, the mercury column receded in the calibrated scale. The change of volume was then determined from the physical dimensions of the capillary. Knowing the mass of ice melted, the heat which was given up by the body was calculated.

The ice calorimeter is the most successful of all devices in eliminating thermal leakage, since the heat transfer takes place in the interior of a mass of ice whose outer temperature, accurately zero, is the same as that of other ice which surrounds it.

A major development in the advancement of furnace calorimetry was the advent of electrical heating. Until the electrical equivalent of

heat was accurately determined, workers were tied to the specific heat of water as a standard for measurement of heat quantities. Since this property varied with temperature, confusion reigned, with workers using various values depending upon their experimental temperature and personal preference. Nerst (6) and Eucken (7) were the earliest users of electrical heating.

Another major development in the advancement of furnace calorimetry was the development of adiabatic calorimetry by Richards. (8). This consists simply of maintaining the temperature of the surroundings as nearly equal to the calorimeter temperature as possible, thus eliminating leakage. The value of this method is realized since the determination of thermal leakage is the greatest source of error in calorimetry. (9).

In 1845 Joule proposed using "twin calorimeters" to eliminate the effect of thermal leakage by compensating for it. Two calorimeters were used, which were both heated by the same amount. In Pfaundler's modification, the one most used, a differential thermoelement was used between the two calorimeters. When the calorimeters cooled rapidly this temperature difference was as stationary as the temperature in the adiabatic method. The chief advantage was in reducing the determination of heat capacity to purely comparative measurements which could be accomplished by relatively simple differential apparatus. (9).

Numerous methods of directly obtaining true specific heats have been tried. They are similar in that they involve measurement of heat required to raise the temperature of the substance by a relatively small amount, ranging from a fraction of a degree to a few degrees, depending upon the method and equipment. Most attempts to use these methods beyond moderately high temperature have failed as far as

accuracy is concerned, usually because it was impossible to evaluate satisfactorily the corrections for heat interchange with the surroundings. One of the more successful attempts on record in this line is that of Seekamp. (10).

Many others, too numerous to mention, have done work in the field of calorimetry. In fact, entire books have been devoted to the subject. Only a general background has been presented here to acquaint one with a few of the basic principles used in the field of calorimetry.

### CHAPTER III

#### THEORY OF THE COMPARATIVE METHOD OF CALORIMETRY

The comparative method, actually a refinement of the "twin calorimeters" of Pfaundler, has been applied by Smith (11) and Edmonds (12) for the determination of specific heats.

Only one calorimeter is used by first calibrating it by determining its rate of heating when empty and then with a sample of known specific heat. The standard sample should undergo no transformation and accurate specific heat data should be known over the temperature range to be investigated.

This method consists in placing the specimen with its thermocouple in a refractory container of low thermal conductivity and placing this in a furnace the temperature of which is maintained a constant amount above or below the specimen temperature. A differential thermocouple with one junction on the inside and one junction outside of the container may be used to maintain a constant temperature difference across the container walls. Since the thermal conductivity of the refractory remains constant at any given temperature, the heat flow through it will be constant, and so the heat received or lost by the sample in a given time will be constant. Therefore, the time taken for the sample to move through a given temperature interval will be proportional to the heat absorbed or evolved.

The rate of heat flow through the container is equal to the product

of its conductivity and a shape factor, multiplied by the temperature difference between its inner and outer surfaces. For a given container the first two factors are constant for a given temperature. A constant heat flow may then be attained by maintaining a constant temperature difference experimentally.

The heat flow may be used to supply the specific heat of the sample, to furnish the specific heat of the container, or to provide the latent heat of any transformations in the sample. This may be written as:

$$Q\Delta t_s = W_s C_s \Delta T_s + W_s L_s + W_e C_e \Delta T_s \quad (1)$$

where,  $Q$  = heat flow/unit time

$C_e$  = specific heat of the empty calorimeter

$C_s$  = specific heat of the standard sample

$W_e$  = mass of effective part of the empty container

$W_s$  = mass of the standard sample

$\Delta T$  = change of temperature during time  $\Delta t$

$L_s$  = latent heat of transformation that may occur within the temperature range  $\Delta T$ .

Writing equation (1) for an empty run, the standard sample run, and a run using some sample of unknown specific heat characteristics respectively, (if no transformations occur in either sample) results in the following:

$$Q\Delta t_e = W_e C_e \Delta T_e \quad (2)$$

$$Q\Delta t_s = W_s C_s \Delta T_s + W_e C_e \Delta T_s \quad (3)$$

$$Q\Delta t_u = W_u C_u \Delta T_u + W_e C_e \Delta T_u \quad (4)$$

Using  $W_e C_e = Q \frac{\Delta t_e}{\Delta T_e}$  from equation (2) and rearranging,

equations (3) and (4) become:

$$W_s C_s = Q \left( \frac{\Delta t_s}{\Delta T_s} - \frac{\Delta t_e}{\Delta T_e} \right) \quad (5)$$

$$W_u C_u = Q \left( \frac{\Delta t_u}{\Delta T_u} - \frac{\Delta t_e}{\Delta T_e} \right) \quad (6)$$

Dividing (6) by (5):

$$C_u = C_s \frac{W_s}{W_u} \frac{\left( \frac{\Delta t_u}{\Delta T_u} - \frac{\Delta t_e}{\Delta T_e} \right)}{\left( \frac{\Delta t_s}{\Delta T_s} - \frac{\Delta t_e}{\Delta T_e} \right)} \quad (7)$$

The only unknown in equation (7) is  $C_u$  since the slopes of the time-temperature curves are determined experimentally, the specific heat data of the standard are known, and the weights are easily determined.

The experimental results of this investigation were made possible by the utilization of the above relationship.



## CHAPTER IV

### DESCRIPTION OF THE APPARATUS

#### The Heater

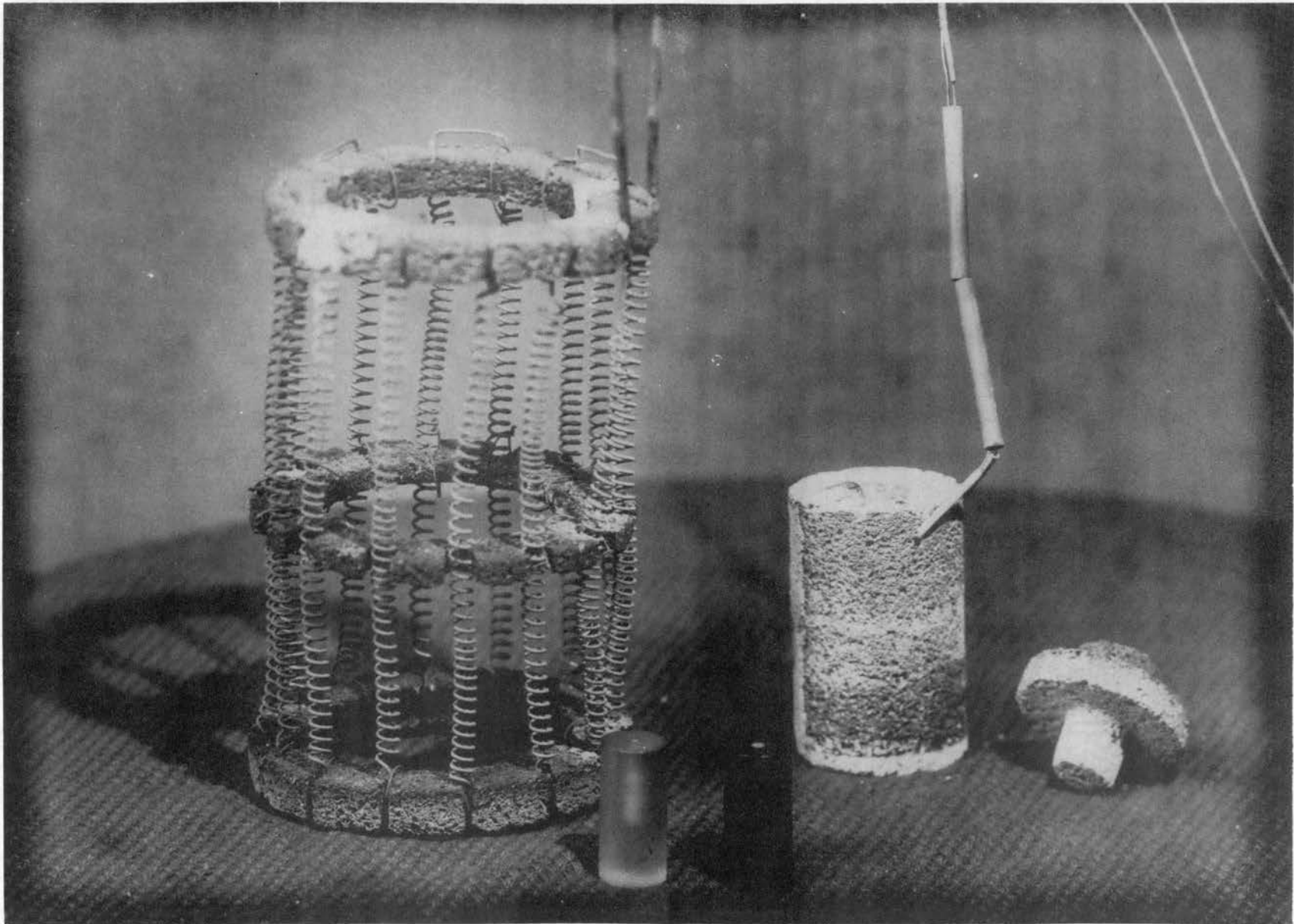
Previous investigators have indicated that a heater element should be designed so that the total mass is a minimum to reduce the thermal lag, and the expansion of the element should not cause short circuits.

DeBarr and Roberts (13) describe a heater element which should have a very low thermal lag and will lengthen to compensate for the heater wire expansion thereby preventing buckling and short circuits. This design also seems advantageous in that only a very small portion of the heater wires is confined. Thus the problem of hot spots and eventual burnouts, which has plagued previous investigators, could possibly be eliminated.

The heater element is shown in Plate I. Armstrong A-20 firebrick, which will withstand temperatures of 2000°F and will machine easily, was used to construct the three ceramic rings. The top and bottom rings are 1/2 x 4 1/2-in. diameter. The holes are 3-in. diameter and the circle diameters of the wire slots are 3 7/8-in. The inner ring is 1/4-in. x 3-in. diameter. The center hole is 2 1/2-in. and the slots are 1/4-in. wide. The centers of the semi-circular bottoms in the slots are on a 3 7/8-in. diameter.

Nichrome V wire, an alloy of 80% nickel and 20% chromium, was

PLATE I. THE HEATER ELEMENT, CRUCIBLE AND SAMPLES



chosen as the resistor wire because of its ability to withstand high temperatures for long durations. The creep rate of Nichrome V is only 1% in 10,000 hours at 2000°F and a load of 200 psi. (14). This small creep rate is particularly important in an element of this design since any great elongation could eventually ruin the element.

Information from the Driver Harris Catalog R-58 was used in the design of the heater wires. B. & S. No. 18 gauge wire wound in 1/4-in. O.D. coils requires 11 amperes to attain a wire temperature of 1500°F in ambient air while in a vacuum this temperature should be considerably higher due to a decrease in heat loss. It was desired to attain a sample temperature of 1500°F which would require a wire temperature somewhat higher. To have ample capacity the element was designed to handle 1.5 KW. The watt density chosen was 50 watts/sq. in. which was within the range specified. The length, L, of the wire was determined as follows:

$$\frac{1500 \text{ watts}}{\pi(0.040 \text{ in. Dia.})(L)} = 50 \text{ watts/sq. in.}$$

$$L = 239\text{-in.}$$

$$R = \frac{W}{I^2} = \frac{1500}{(11)^2} = 12.4 \Omega$$

$$\text{Closed coil length} = \frac{12.4 \Omega}{0.559 \Omega/\text{in.}} = 22.2\text{-in.}$$

It is recommended to stretch the closed coil to 1 1/2 to 4 times its closed length. Using a stretch factor of 4 results in an open coil length of

$$4(22.2) = 88.8\text{-in.}$$

The length of the element was chosen to be 6-in. which would require 14 passes.

The coils were made on an engine lathe by coiling the wire around a 0.170-in. diameter mandrel. For the first attempt at assembling the wire on the ceramic rings, a plaster form was cast in a wax mold. This form had the same cross section as the ceramic rings and was cut in two parts and placed between these three rings to hold them in their proper positions. The coils were then stretched one at a time and placed in their respective slots in the rings. After breaking a few of the ceramic supports it was discovered that the most feasible way to assemble the element was to stretch all of the coils first and put the proper bends between them. Then the assembly procedure was just a matter of feeding the coils onto the rings without utilizing a plaster support. B. & S. No. 22 gauge alumel wire was used to tie the coils in each slot. A 2-in. length of 1/8-in. diameter copper tubing was crimped on each end of the Nichrome wire to form the terminals.

#### The Crucible and Crucible Base

The crucible used in this investigation was the one used by Beakley (15) in his investigations. Its purpose was twofold. It served as a positive positioning device for the sample, and it also contained the differential thermocouples which were used in controlling the temperature difference across the crucible wall. Armstrong (16) A-20 insulating firebrick was used as the crucible material. This material had a thermal conductivity of  $1.536 \text{ Btu in. ft.}^{-2} \text{ hr.}^{-1} \text{ }^{\circ}\text{F}^{-1}$ .

The crucible was a 2-in. diameter cylinder 3-in. long having a 3/4-in. diameter hole 2 1/4-in. deep drilled axially to accommodate the sample.

A-20 brick had also been used for the crucible lid which had a main body 2-in. in diameter. One end was 1-in. diameter and the other end was turned to a 3/4-in. diameter in order that it might fit in the sample cavity. In this way the sample was completely covered by the A-20 brick. A 1/16-in. diameter hole was drilled through the center of the lid to allow insertion of the sample thermocouple.

Four holes to accommodate the differential thermocouples had been drilled through the cylinder. As can be seen in Fig. 1 the holes had been spaced at 90° intervals. Also the holes alternated in position from the outside to the inside of the crucible with a radial distance of 3/8-in. separating the outside and inside locations.

The differential thermocouples were composed of B. & S. No. 18 gauge Chromel (90% Ni, 10% Cr) and Alumel (95% Ni + Al + Si + Mn) wires. Figure 1 also shows the position of the junctions in the crucible.

A butt welding mechanism on a Model ML Do-All band saw had been used to form the thermocouple junctions. This method produced junctions of constant cross sectional area which simplified the threading of the wires through the holes.

As observed from the figure, two hot junctions and two cold junctions were utilized which resulted in doubling the electromotive force (emf) produced and averaging the differential.

The samples, also used by Beakley, were in the form of cylinders

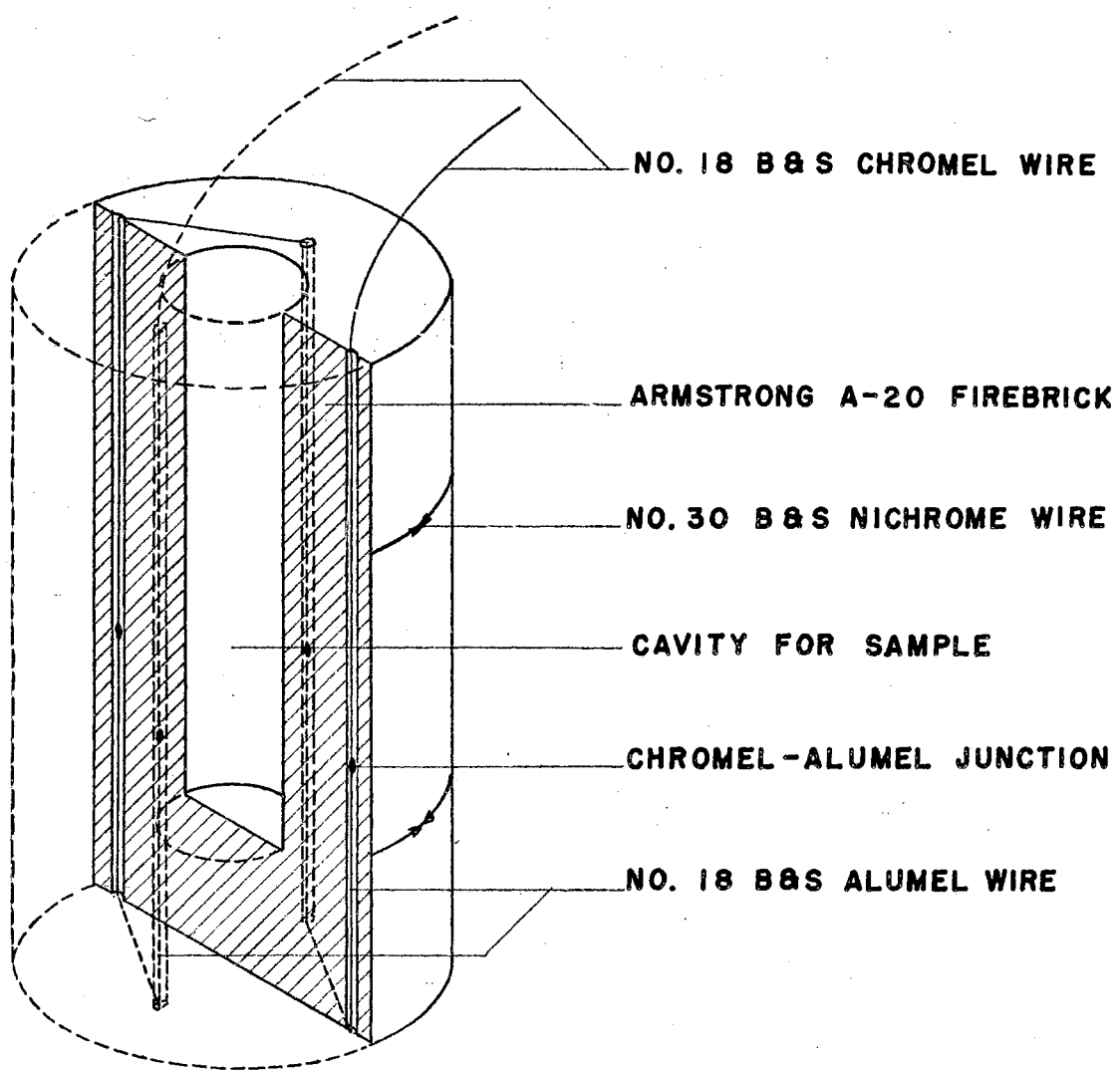


Figure 1. Schematic of Crucible

3/4-in. in diameter and 1 1/2-in. long. A 1/16-in. diameter hole had been drilled axially to a depth of 3/4-in. in one end to accommodate the sample thermocouple.

A sample of synthetic sapphire ( $\text{Al}_2\text{O}_3$ ) was chosen as the standard because of its known specific heat data and chemical stability up to its melting point (about 2300°K).

Originally a base of steel was used. The high thermal conductivity of this material would cause a high heat flow across the base and result in a temperature on the bottom of the crucible approximately equal to the outside crucible wall temperature. This would be a definite advantage, but it was later discovered that the steel base was causing an increase in the thermal lag characteristics of the furnace. Also, due to the relatively high specific heat of steel, more energy was required to attain a given sample temperature than with a base of ceramic.

These effects prompted a change to a ceramic base. This base, also used by Beakley, was in the form of a thin wall cylinder of A-20 firebrick supported by three equally spaced ceramic legs. These legs were supported by a block of firebrick which was positively positioned in the bottom of the calorimeter by three bolts which remained from the previous steel base supports.

#### The Inner Enclosure

The body of the inner enclosure was a 6 1/8-in. O. D. x 9-in. long copper tube having a 1/8-in. wall thickness. Twenty-two turns of 1/4-in. diameter copper tubing were evenly spaced around the outside of the tube

and soldered in place to form the cooling water circuit. The bottom of the enclosure was fabricated out of a series of three 6 1/8-in. diameter x 1/8-in. thick stainless steel radiation shields held together by three 1/8-in. diameter x 1 7/8-in. long stove bolts equally spaced so as to serve as legs for the enclosure. Two nuts placed between the shields on each bolt served as spacers to separate the shields approximately 1/4-in. The copper enclosure was permanently affixed to the bottom series of radiation shields by soldering around the entire periphery thus forming an excellent path for heat flow from the hot shield to the cooling water coils. A similar design was used for the top of the enclosure except that three equally spaced lugs were utilized to position the series of shields. Holes were drilled through the top shields to facilitate the thermocouples and power leads as can be seen in Plate II.

Four 1/4-in. diameter x 1 3/4-in. long bolts, equally spaced around the circumference, served as a method of supporting and positioning of the heater element. Two of the bolts, on opposite sides 1 5/8-in. from the top, supported the upper ring of the heater element. The other two bolts, 1 1/2-in. from the top, were gently screwed against the ceramic ring to prevent any lateral movement while the lower part of the element was allowed to hang freely.

A sheet of copper was fabricated into a cylinder for a radiation shield inside the enclosure. This 5 3/8-in. O. D. x 6-in. long cylinder was held off the bottom of the inner enclosure by three small pieces of A-20 firebrick.



PLATE II. THE INNER ENCLOSURE

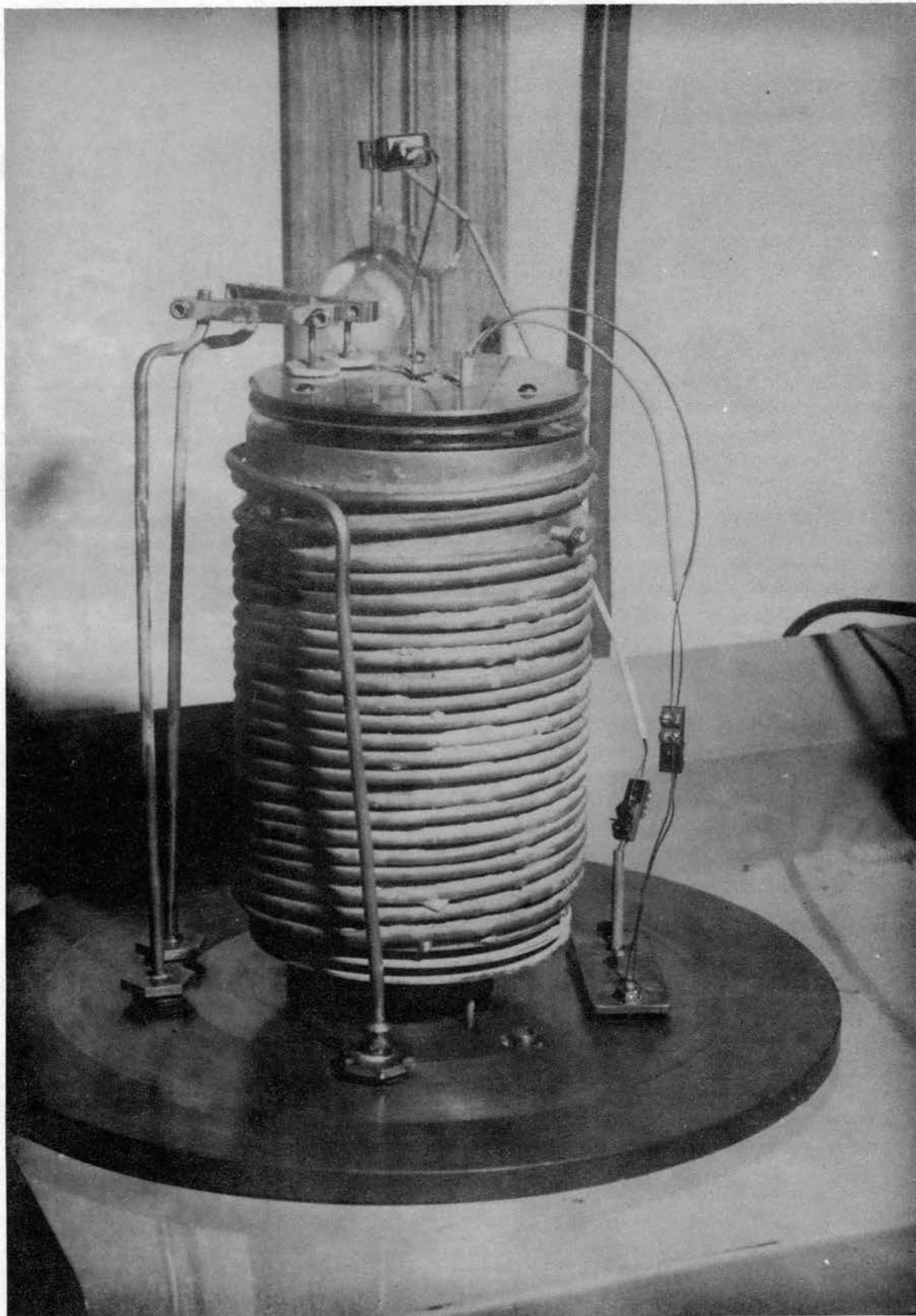
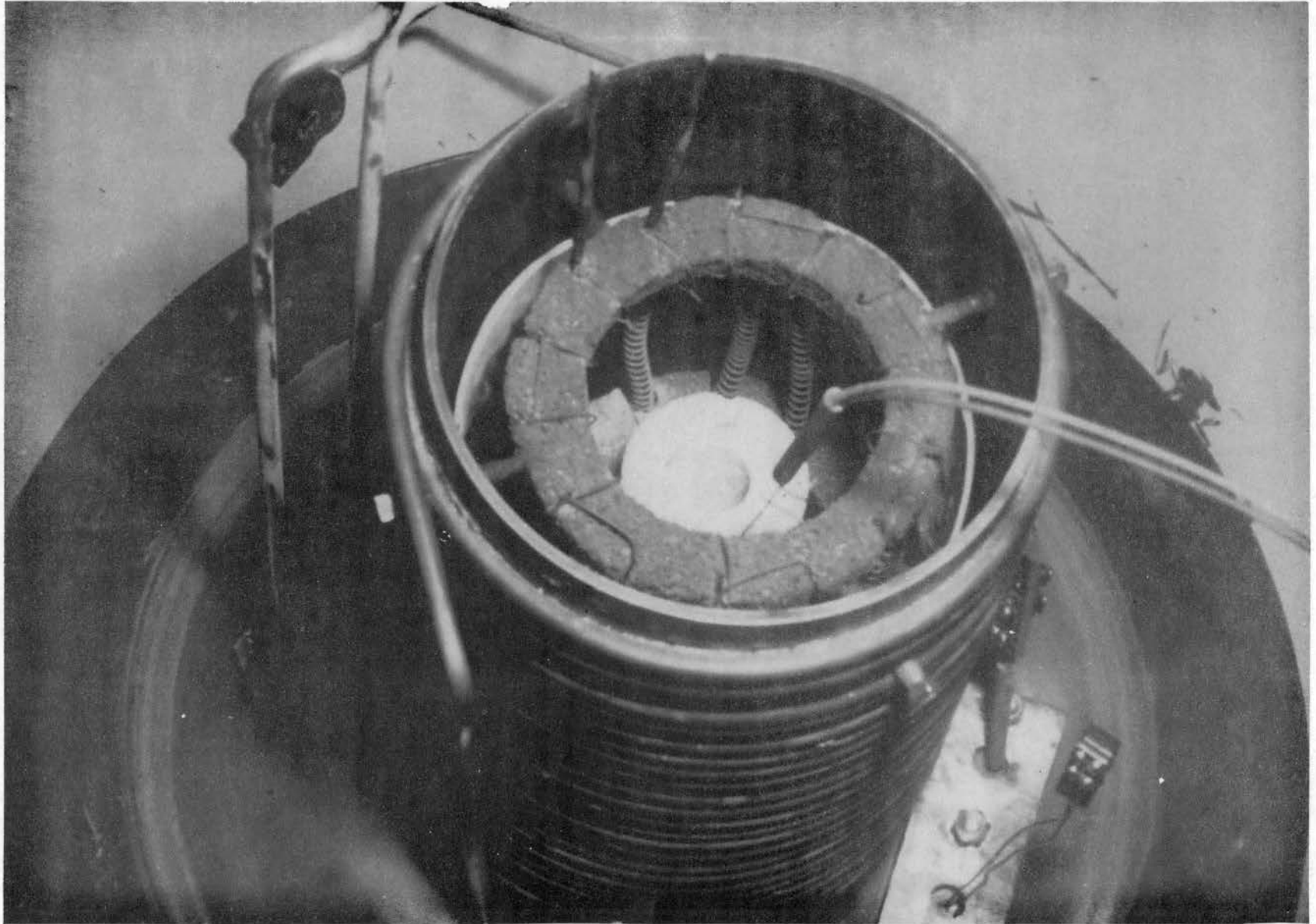


PLATE III. ASSEMBLY OF HEATER AND CRUCIBLE



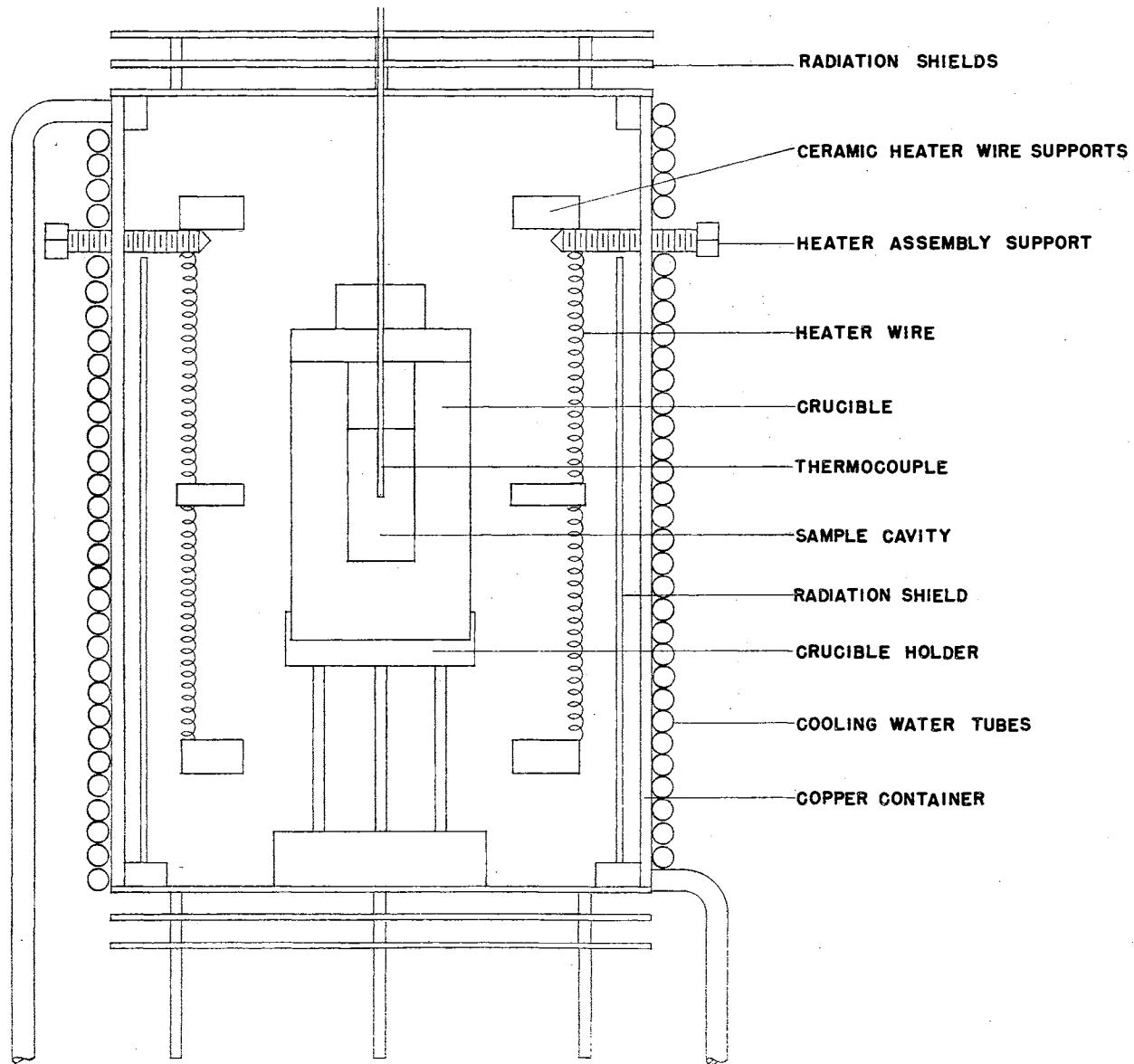


Figure 2. Schematic of Inner Enclosure Assembly

### The Vacuum System

A vacuum was chosen as the most desirable atmosphere under which to operate the furnace. First, the heat loss was limited essentially to radiation which in turn was controlled by shielding. Secondly, a vacuum prevented oxidation and contamination of the sample and heater wires.

The main components of the vacuum system were an oil diffusion pump, a mechanical pump, and a bell jar with base plate.

A 16-in. diameter plate was cut out of 5/8-in. thick hot rolled steel for the base plate. One surface was turned down on a lathe to insure a good seal with the bell jar. The inlet to the diffusion pump was a 4-in. diameter hole through the center of the base plate.

Holes tapped with 1/2-in. pipe thread were fabricated in the base plate to accommodate the water, power, thermocouple and vacuum gauge inlets. Originally the vacuum connectors used were type CGB connectors supplied by the Consolidated Electrodynamics Corporation, Rochester, New York. These connectors proved to be unsatisfactory and were replaced with the exception of the connector on the vacuum gauge inlet. One type of the replacement connectors is shown in Fig. 3. It was necessary to drill the threads out of the holes in the base plate before assembling these connectors. Upon tightening these connectors in the base plate, the phenolic bushings forced the rubber into the hole forming an excellent seal. The connector shown in the figure is a power lead. A piece of 1/4-in. diameter copper tubing was crimped on the end

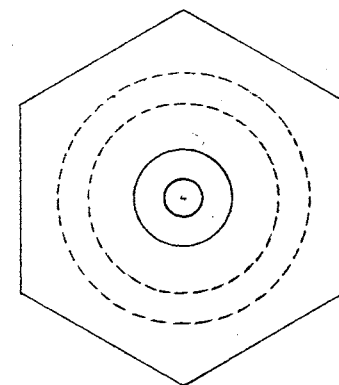
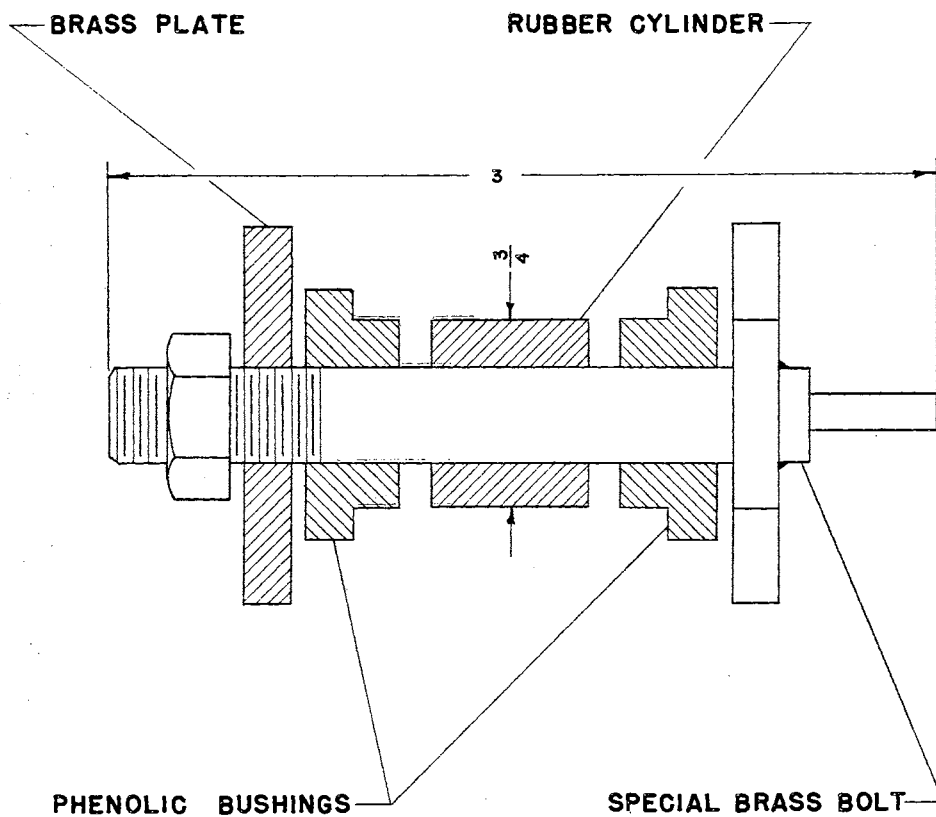


Figure 3. A Power Connector

of the connectors and led to the top where heavy copper bars, clamped on by means of Allen head bolts, connected them to the 1/8-in. diameter leads on the heater element as shown in Plate II. The cooling water connectors were similar except the brass rods were hollow, and one end of each was machined to accept 1/4-in. Swagelok fittings while the cooling water tubes were soldered in the other ends. A slightly different method was used for the thermocouple leads. Rubber cylinders, having two small holes to accommodate the wires, were cut to fit the base plate holes. Phenolic bushings with lugs to fit the base plate holes were used on each end of the rubber cylinders. These bushings were forced against the rubber cylinders by means of flat steel plates, mounted with stud bolts, on either side of the base plate.

A 12 1/2-in. diameter x 18-in. high Pyrex bell jar supplied by Consolidated Electrodynamics Corporation with a rubber boot gasket was used on the base plate to form the vacuum chamber. At all times when the vacuum system was in operation a sheet metal shield was placed over the bell jar to protect nearby persons from injury in case of fracture of the bell jar.

An oil diffusion pump and a mechanical pump were operated in series in the system. Consolidated Electrodynamics Corporation supplied both pumps. The type MC, Three-Stage, 4-in. Casing oil diffusion pump had an operating range of  $3 \times 10^{-6}$  to  $2 \times 10^{-3}$  mm of mercury. Its maximum speed was 205 liters/second. An oil diffusion pump is very simple in operation. Its only moving part is the fluid continuously cycling between the fluid and gaseous phases. The pump fluid, in the

form of hot vapor, rises from a boiler through a chimney from which it is projected downward through a series of annular openings at supersonic speed. These streams of vapor trap the gas molecules from the chamber being evacuated and compress them to a pressure at which the forepump can draw them from the outlet. The vapor then condenses on the cooled wall of the pump and returns to the boiler to repeat the cycle. (17). A special oil having very low vapor pressure is used for this type of pump.

Between the diffusion pump and vacuum chamber a type BW 40 water-cooled baffle was used. Baffles are essentially cooled mechanical obstructions which condense any back-streaming vapors and return the condensate to the pump, thus preventing the vapors from entering the vacuum chamber. This baffle was also supplied by Consolidated Electrodynamics Corporation.

A model 1402B, two stage, rotary, oil-sealed pump manufactured by the W. M. Welch Company, was used as the forepump. Its operating pressure range was  $1 \times 10^{-4}$  to 760 mm of mercury.

The diffusion pump and the mechanical pump were connected by an LC2-73 12-in. flexible connector. This connector was dual purpose in that it also damped out vibrations from the mechanical pump. The diffusion pump end was a bolted flange with an O-ring seal while the other end was 1 1/2-in. National Pipe Thread.

To seal effectively the bolted flanges connecting the base plate, baffle, diffusion pump, and flexible connector, "Celvacene" high vacuum grease was utilized on the O-ring seals.

A 1 1/2-in. x 3/4-in. ell connected the flexible connector to a modified connector into the mechanical pump. A special fitting was used to seal the ell on the flexible connector. To make this fitting a 1/4-in. ring was cut from the end of a 1 1/2-in. fitting. One face of this and its mating face on the ell were turned on a lathe to accept a rubber gasket. To assemble, the ring was tightened on the connector followed by the rubber gasket. Then the ell was tightened onto the connector. To effect the seal, the ring was then tightened against the face of the ell forcing the rubber gasket against the threads on the connector. Several coats of Glyptal enamel were painted over this connection to seal the pores in the cast iron ell and further seal the mechanical connections.

A 1/4-in. brass fitting was brazed into a hole in the side of the ell to facilitate connecting a short length of 1/4-in. copper tubing and a tee. The sensing element of a Pirani vacuum gauge, to be explained later, was connected to one side of the tee, while on the other side was a small rubber hose provided with a clamp to serve as the air bleed valve.

The entire vacuum system was mounted in a steel cabinet. Bulkhead connectors were used to lead the cooling water circuits through the side of the cabinet to the water supply and drain. One circuit was the cooling circuit for the inner enclosure. Another was made up of the baffle, the diffusion pump, and the forepump line coolers in series. A third circuit was the boiler cooler. A drain valve was installed in the boiler cooler circuit to drain the water from it before the boiler was put in



operation. All of the lines were assembled with 1/4-in. diameter polyethelene tubing and Swagelok fittings except the boiler drain line which was aluminum since polyethelene would melt at the boiler temperatures. The complete vacuum system is shown in Plate IV.

#### Instrumentation and Power Supply

The power supply was a 110 volt A. C. outlet which was controlled by a General Radio Corporation 20 ampere Variac, series V-20. A German made transformer, having a voltage ratio of 380:220 and a current ratio of 15.2:26.2, was used to increase the voltage from the Variac in order to obtain the necessary power in the heater element. To measure the current in the primary circuit, a General Electric, type AK-1, clip on volt ammeter was used.

A type 116 Variac manufactured by the Superior Electric Company was in the diffusion pump boiler circuit. During this investigation, however, it remained at its maximum setting.

Measurement of the temperature difference across the crucible wall was facilitated by the differential thermocouples in the crucible wall in series with a Cenco standard resistance box and a galvanometer. This galvanometer, manufactured by the Leeds and Northrup Company, Philadelphia, Pennsylvania, had a sensitivity of  $0.45 \mu\text{v}/\text{mm}$ . The purpose of the resistance box will be explained in Chapter V.

The sample temperature was measured with a chromel-alumel thermocouple element in a 1/16-in. diameter stainless steel sheath in conjunction with a No. 8662 Leeds and Northrup Portable Precision Potentiometer and standard Dewar flask ice junction. The thermocouple, furnished



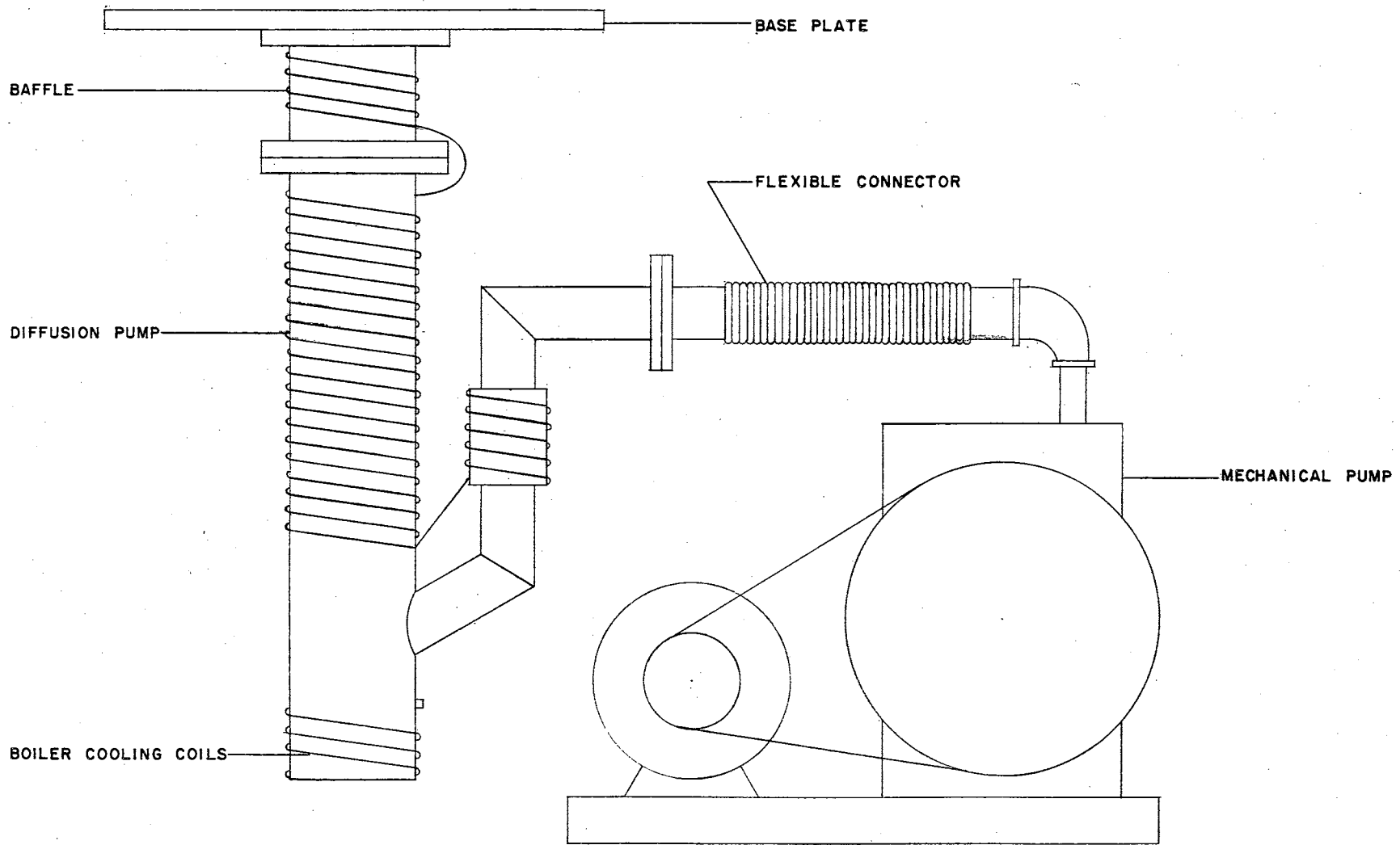


Figure 4. Schematic of Vacuum System

by Minneapolis-Honeywell Regulator Company, was equipped with a quick connect plug for ease of assembly and disassembly.

A continuous timer was used simultaneously with the potentiometer and galvanometer to obtain the desired readings which will be discussed in the next chapter.

The sensing element of a type GP-110 Pirani vacuum gauge was connected to the entrance line of the forepump as mentioned previously. A Pirani gauge uses a Wheatstone bridge circuit to balance the resistance of a tungsten filament sealed off in high vacuum against that of a tungsten filament which can lose heat by conduction to the gas being measured. A change of pressure causes a change in the filament temperature and, consequently, of the wire resistance, thus unbalancing the bridge. In this gauge the unbalance is measured and indicated on a microammeter calibrated in units of dry air pressure. (18).

The pressure in the vacuum chamber itself was measured by a McLeod Gauge. This type of vacuum gauge operates on the principle of compressing a known volume of low-pressure gas to a higher pressure and measuring the resultant volume. (19). It is the only type of gauge which is capable of measuring low pressures directly and is considered to be the most reproducible and reliable; consequently, it is used as a standard for the calibration of other types of vacuum measuring instruments. The range of the McLeod gauge used in this investigation was 0.1 to 400 microns.

The high vacuum attained in this investigation was sufficient to lower the thermal conductivity of the furnace atmosphere to a negligible value which was the ultimate goal of using a vacuum.

## CHAPTER V

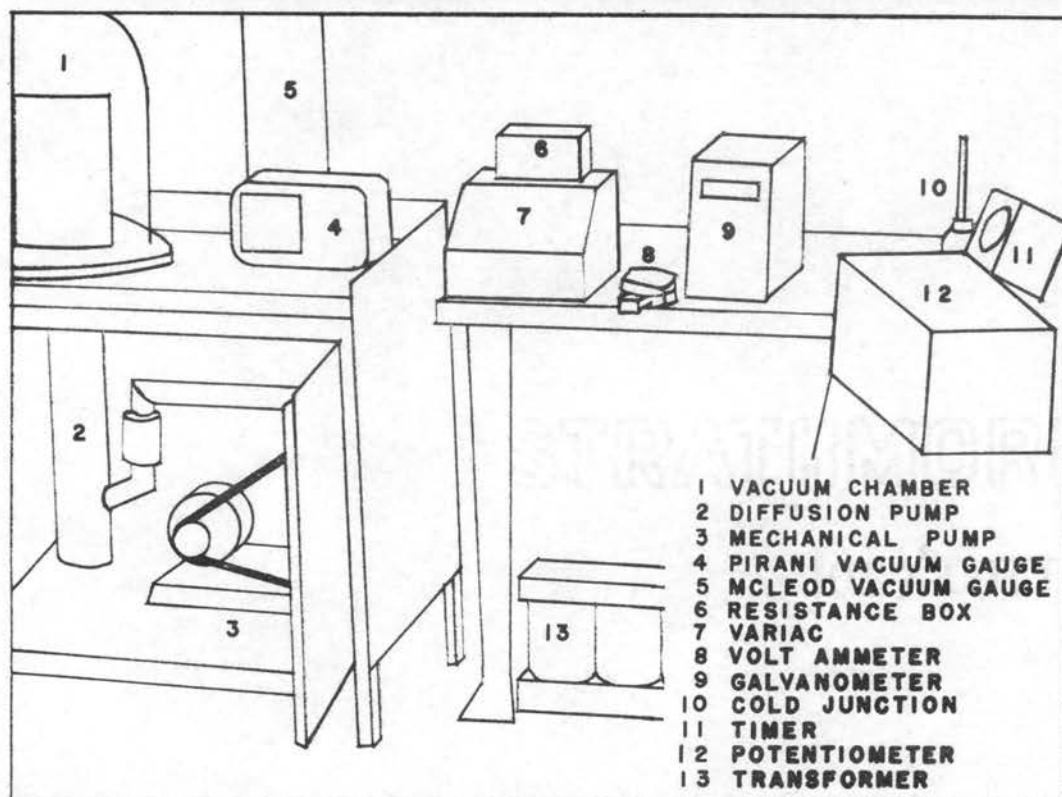
### EXPERIMENTAL PROCEDURES AND RESULTS

As explained in the chapter on the comparative method, it is required to have the slopes of the time-temperature curves,  $\left(\frac{\Delta t}{\Delta T}\right)$ , throughout the temperature range of investigation for the standard sample, an empty crucible, and the unknown sample in order to determine the specific heat of the unknown over this temperature range.

The three crucible wall temperature differentials used in this investigation were 75°F, 100°F, and 125°F. Prior to each run the differential circuit was set such that the galvanometer was at mid-scale for the desired temperature differential. A type PM-1 potentiometer manufactured by the Thwing-Albert Instrument Company, which will supply an emf equivalent to the output of a thermocouple, was series connected in the differential circuit. Provided there was no temperature differential at the time, the output emf corresponding to twice the desired temperature differential was supplied to the circuit. By utilizing the variable resistance box, the resistance in the circuit was changed to balance the galvanometer at mid-scale. The emf corresponding to twice the desired differential was required since there were two thermocouples in series in the circuit. Plate V shows the equipment layout.

Approximately twenty minutes were required to evacuate the system to a sufficiently high vacuum. The procedure for starting the pumping system consisted of first starting the mechanical pump and allowing it

PLATE V. EQUIPMENT LAYOUT



to reduce the pressure to the operating range of the diffusion pump, after which the diffusion pump boiler and its cooling water were started. Oxidation of the diffusion pump oil would result if the pump were operated at higher pressures than specified. The flow rate for the cooling water circuit was 0.1 GPM (90 milliliters per 15 seconds) which was set approximately using a watch and graduated cylinder.

A critical evaluation of the heat loss from the heater to the cooling water of the inner enclosure was not made; however at a flow rate of approximately 0.2 GPM the temperature of the water was not sufficiently high to be uncomfortable to the hand at the maximum furnace temperatures used.

The actual taking of the data was not complicated, but it tended to become tiring and monotonous for the operator due to the constant attention required and the length of the runs which ranged from one to four hours.

To start the heating, the power was turned on to the heater element and controlled by the Variac until the required crucible wall temperature differential was attained to bring the galvanometer to mid-scale. During the run a slight but continuous increase of the Variac setting was necessary to keep the galvanometer at this mid-scale position. Once the swing of the galvanometer had stopped at the proper position it was fairly easy to control it throughout the run.

The continuous timer was read and recorded at various millivolt intervals, usually 0.5 or 0.25 mv, throughout the run which lasted to 1500°F. At the completion of a run the sample was allowed to cool to

a sufficiently low temperature to prevent its rapid oxidation upon releasing of the vacuum.

The procedure for stopping the vacuum pumps was the reverse of the starting procedure. After turning off the diffusion pump boiler, it was allowed to cool by means of its cooling water until it was warm to the hand before the mechanical pump was turned off. This again was necessary to protect the diffusion pump oil from oxidation. Once the pumps were off, the air-bleed valve was opened to release the vacuum after which the bell jar could be removed to gain access to the inner assembly.

From the original data the slope of the time-temperature curve,  $\left(\frac{\Delta t}{\Delta T}\right)$ , and the average temperature were determined for each time interval. These were plotted and an average curve was drawn through the points to give a smoothed slope-temperature curve. Values from this smoothed curve were read at each 100°F and used in the calculation of the specific heat-temperature curve for the unknown sample. Slope-temperature curves are shown in Figs. 5, 6, 7, and 8. The specific heat results for these runs are shown in Figs. 9 and 10. The specific heat data for the synthetic sapphire ( $\text{Al}_2\text{O}_3$ ) were determined by Ginnings and Furukawa (20) of the National Bureau of Standards and are believed to have an accuracy of 0.2 per cent.

The first ten runs were taken with the steel crucible base in the system. Twice during this period a chromel lead wire broke off adjacent to the crucible. The first time a thermocouple welder was used to repair the break. The operation of this welder consisted essentially of making electrical contact between the twisted ends of the broken



wires and some oil submerged mercury. The resulting arc fused the wires back together, but, unfortunately, this type of repair was not durable enough for this installation and soon broke again. A more satisfactory repair was later made by fusing the twisted wires together with an acetylene torch.

The accuracy of the specific heat data obtained from the first ten runs was very poor. In an attempt to improve the results the steel crucible base was replaced by the one made of Armstrong A-20 firebrick.

After run 18 the results were evaluated once again and were still in great error when compared with the accepted results of previous investigators. To check for the magnitude of any induced emf in the sample thermocouple leads, since the leads were in close proximity of the Variac, the Variac was set at its maximum setting and then switched on and off. During this procedure the potentiometer remained balanced indicating that any induced emf was negligible.

Another check was made to determine if either of the thermocouple wires were shorted on the stainless steel sheath. A hot soldering iron was slowly moved along the surface of the sheath. At one point close to the connector the potentiometer indicated a drop in temperature which in turn indicated that one of the wires was shorted against the sheath. (21). This shorting in the thermocouple could cause large errors in the readings; therefore, it was replaced by the Minneapolis-Honeywell thermocouple described in Chapter IV. Also the crucible was replaced because the original one was slightly cracked, and the lead wires appeared as if they might break again in a different location.

No major difficulties were experienced with the heating element. This design apparently alleviated the problem of burnouts reported by other investigators. After the first run and throughout the remainder of the investigation the heater wires were a dark green color. Also a slight amount of plating continually reappeared on the inside of the inner enclosure. Apparently the green was the result of a phenomenon called "green rot" which is a form of attack sometimes encountered in 80/20 nickel-chromium alloys. The trouble arises from carbon being picked up from the atmosphere, forming chromium carbides which are precipitated mainly at grain boundaries until the matrix is practically devoid of chromium. (22). One would wonder how carbon could be picked up when the heater was operated in a vacuum. It is thought that even though a baffle was used between the diffusion pump and vacuum chamber a small amount of oil vapor found its way into the chamber thus providing the carbon for the "green rot" phenomenon. This could be verified by the fact that a thin coating of oil was noticed on the inner surface of the bell jar after a few runs. The "green rot" apparently was not extensive enough to damage the heater wires; however, over a much longer time some deterioration would probably become noticeable.

A total of 41 runs were made during this investigation. Of these, only the three series of wall differentials, 75°F, 100°F, and 125°F, will be included. These will be referred to as series 75, series 100, and series 125, respectively. Each series consisted of the standard run of sapphire, the empty run, and two unknown runs using chromium and an alloy of titanium. The titanium (Ti C-130AM) was supplied by Rem-Cru

Titanium, Inc. The Vanadium Corporation of America, Cambridge, Ohio, furnished the 99.1% pure chromium sample. A chemical analysis of these samples is included in the appendix. Table I shows the average heating rates for the runs in each series.

TABLE I

AVERAGE HEATING RATES ( $^{\circ}\text{F/hr}$ )

SAMPLE	SERIES 75	SERIES 100	SERIES 125
$\text{Al}_2\text{O}_3$	330	407	560
Empty	980	1178	1550
Ti C-130AM	408	485	716
Chromium	380	500	619

It can be seen from Figs. 5, 6, 7, and 8 that increasing the temperature differential tends to flatten the curves.

The procedure used to determine the slopes for runs 30, and 31 was slightly different than that for the other runs. The new method was necessary because of an extreme amount of scattering of the slopes due to interpolation in the tables to determine the temperature. The temperature does not enter into the calculation of the slope in this method. The first step consisted of calculating the time per some convenient mv interval throughout the run after which they were plotted against their corresponding temperatures. An average curve was then drawn through these points and values read from it at every  $100^{\circ}\text{F}$ . The time-temperature slope, as in the first method, was obtained by multiplying each of these values by the ratio of the mv interval to

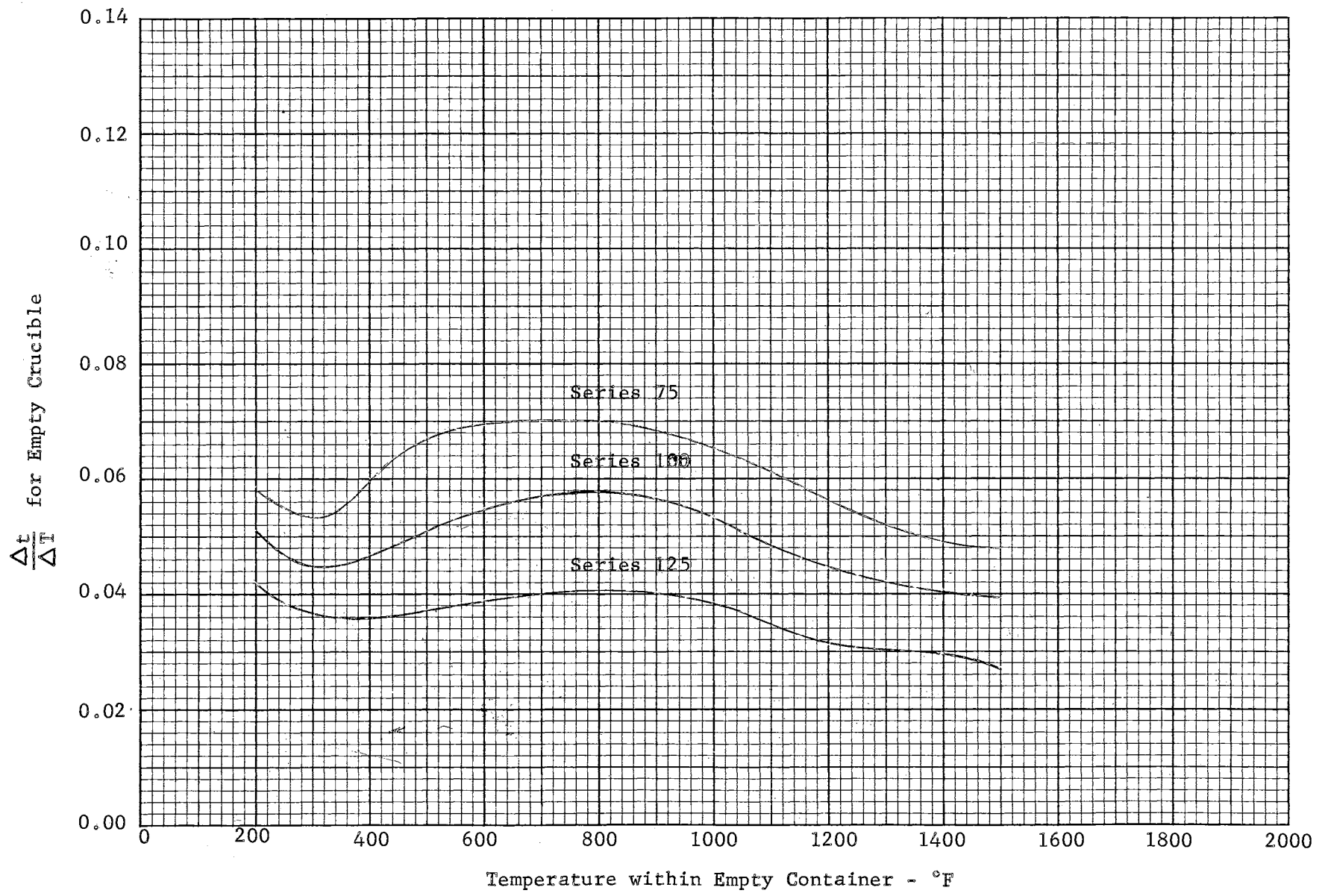


Figure 5. Smoother Time-Temperature Slopes for Empty Runs

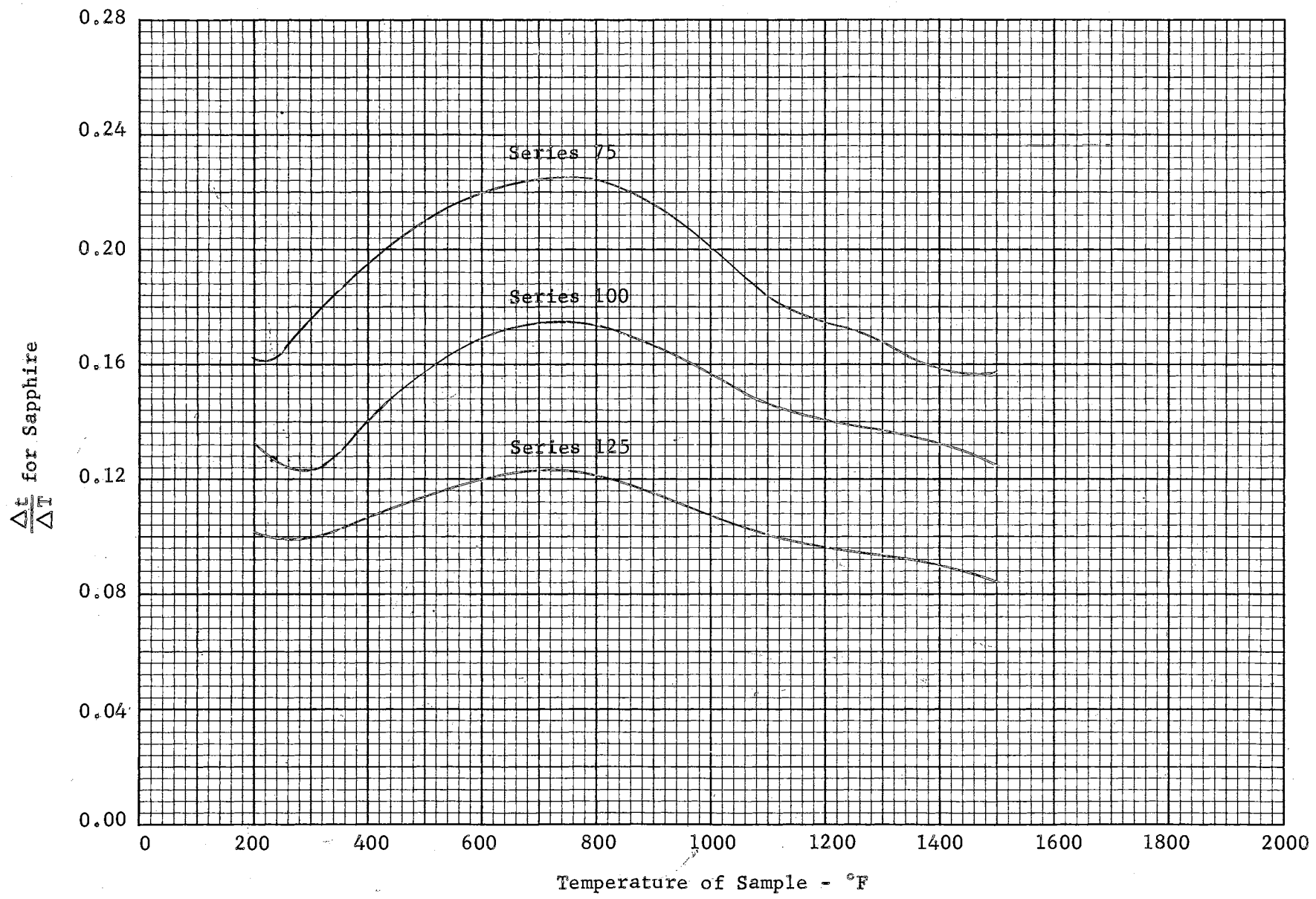


Figure 6. Smoother Time-Temperature Slopes for Sapphire

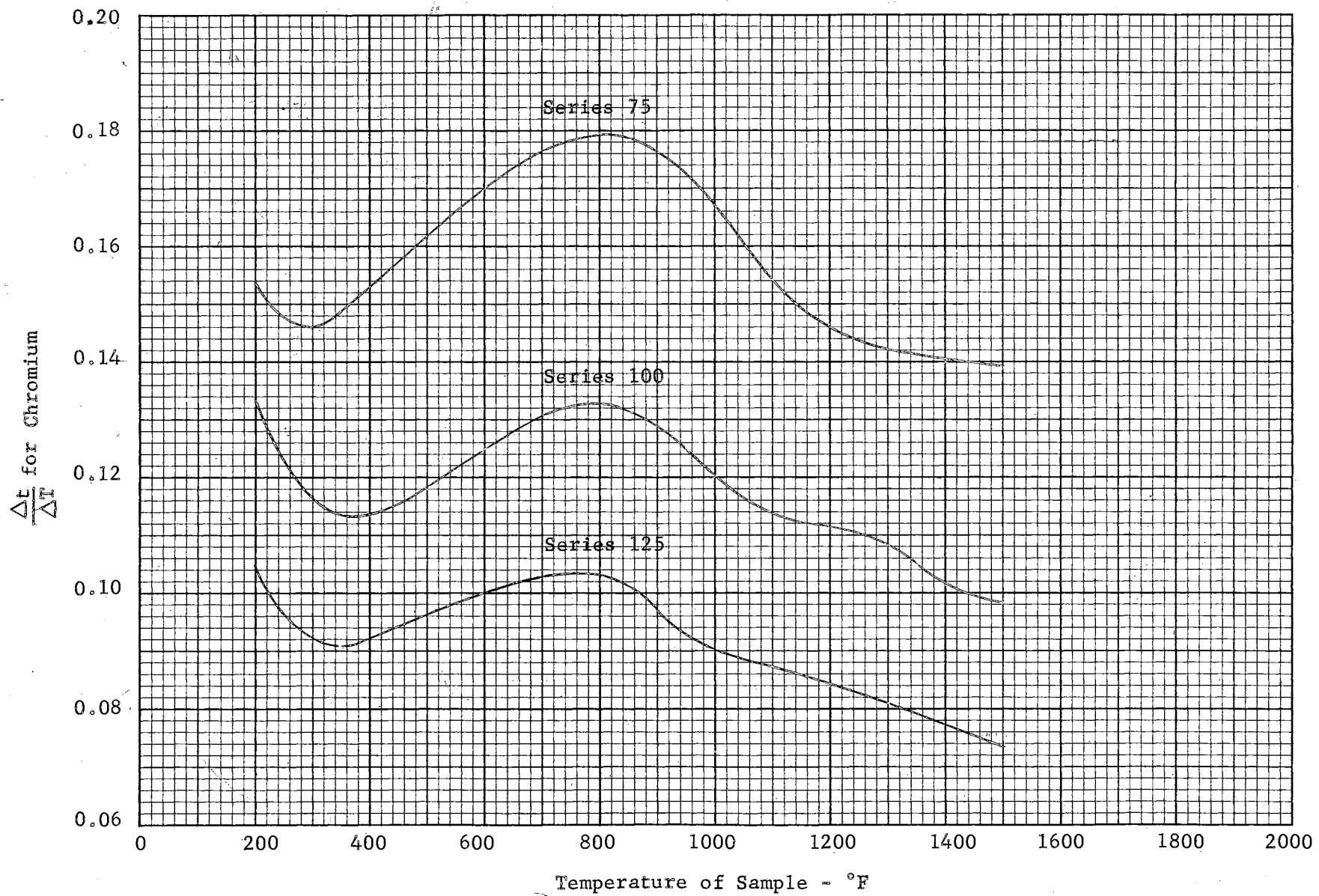


Figure 7. Smoothed Time-Temperature Slopes for Chromium

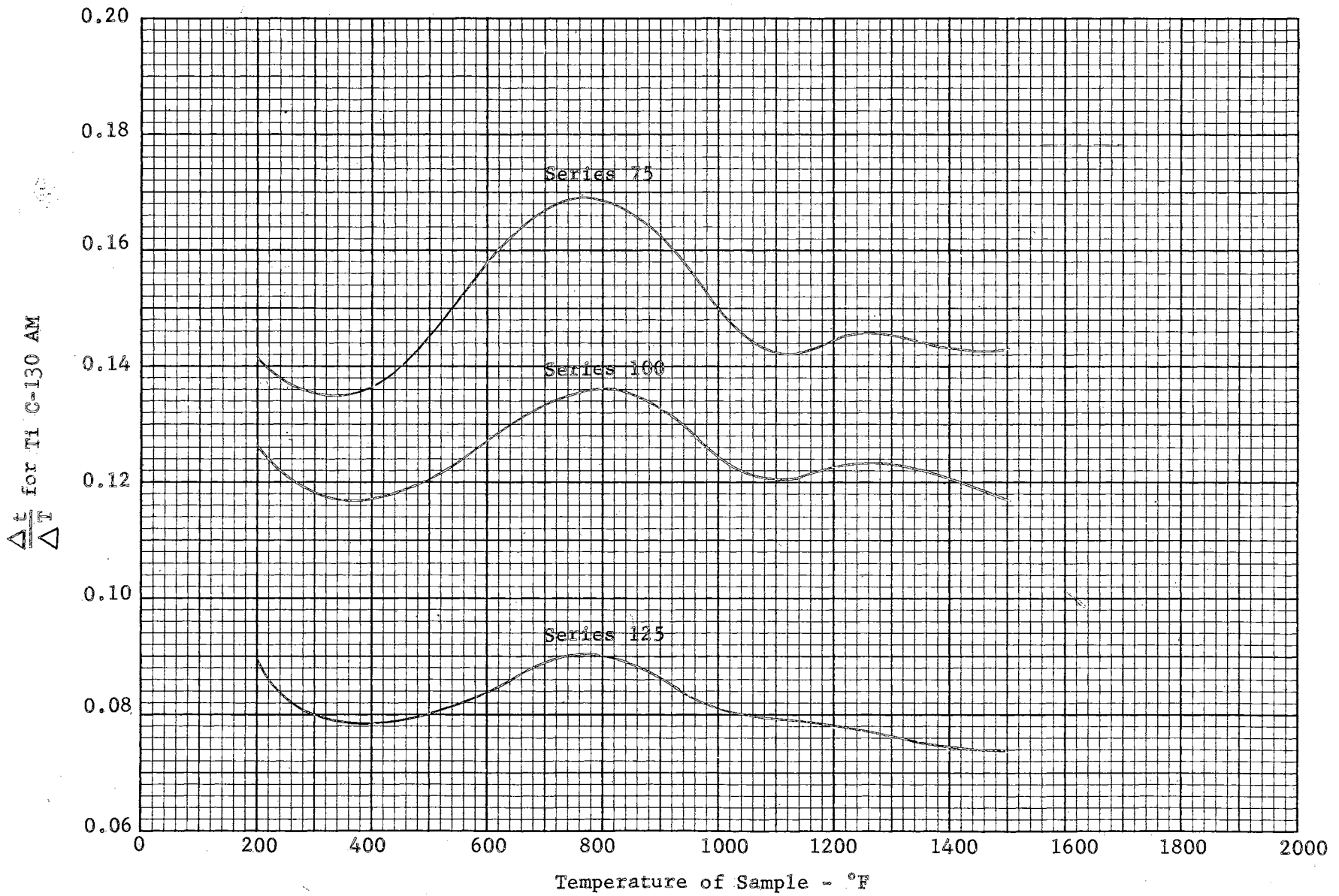


Figure 8. Smoothed Time-Temperature Slopes for Ti C-130 AM

its corresponding temperature difference.

$$\frac{\Delta t}{\Delta_{mv}} \times \frac{\Delta_{mv}}{\Delta T} = \frac{\Delta t}{\Delta T}$$

This method resulted in a very smooth slope-temperature curve, and was considerably faster provided equal intervals had been taken during the run.

From the specific heat curves, Figs. 9 and 10, one notices that inconsistency reigns since no one series is best for both samples. The results are compared with those given by Battelle Memorial Institute. (23). One would assume that the series 75 would give the best results since with a lower heating rate the sample would more nearly approach isothermal conditions than with a high heating rate. From Fig. 9, the specific heat data for chromium, it is seen that the results contradict this assumption because the series 125 is definitely the best; however, it is not the best in Fig. 10 for titanium.

A sine wave effect is inherent in all of the calculated specific heat results. Beakley (15), in his investigations also had this effect, but evidently this is not a characteristic of the calorimeter as he thought. In this investigation as well as Beakley's, the emf was held constant across the crucible wall. Therefore, the differential was not one of constant temperature as assumed since the mv/°F-temperature curve for chromel-alumel thermocouples is not linear. Inspection of this curve, Fig. 11, shows a very apparent sine wave. An average value was used in setting the differential galvanometer; thus during the course of the run, the temperature differential actually varied in a sine wave



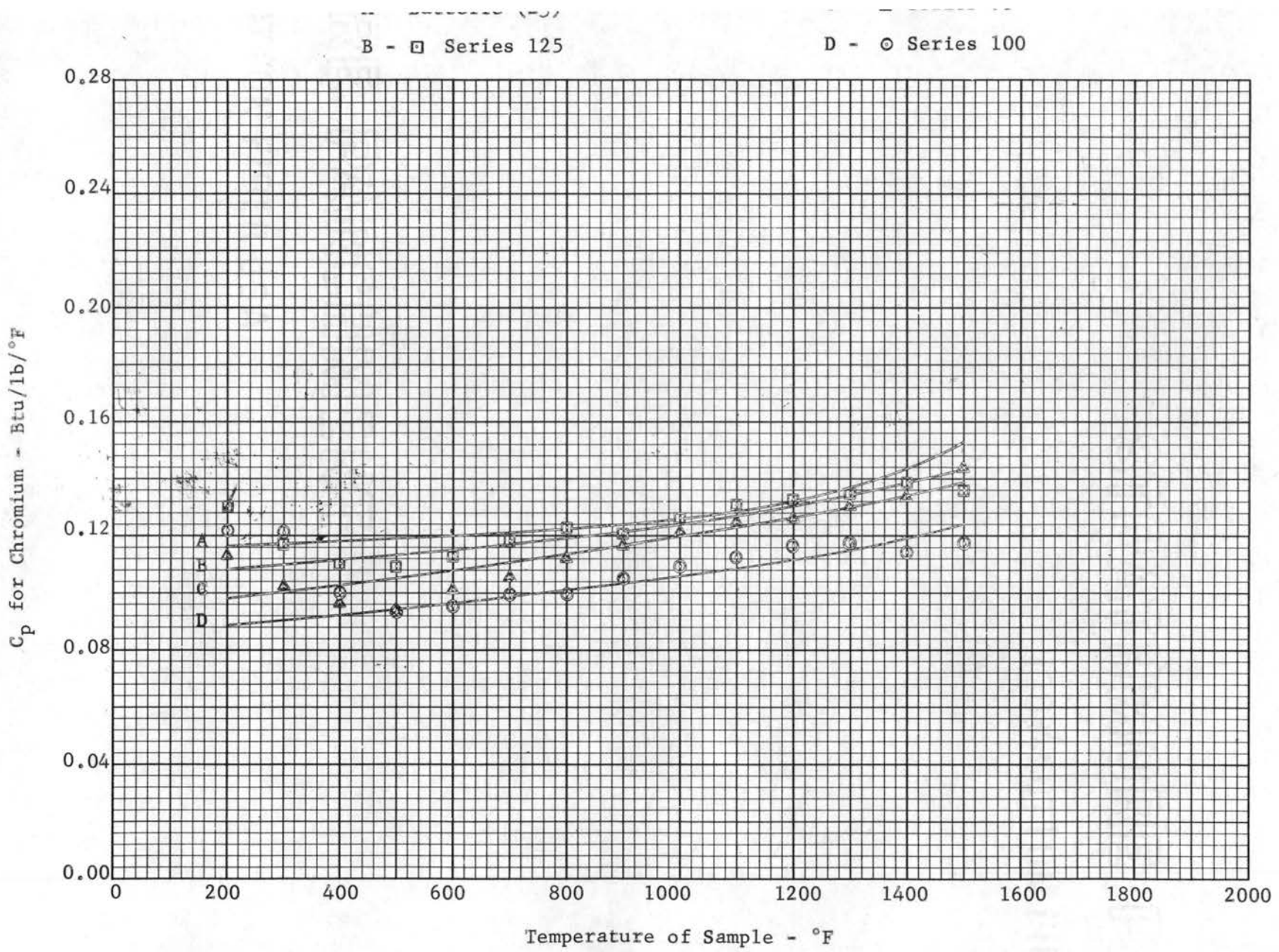


Figure 9. Specific Heat Data for Chromium

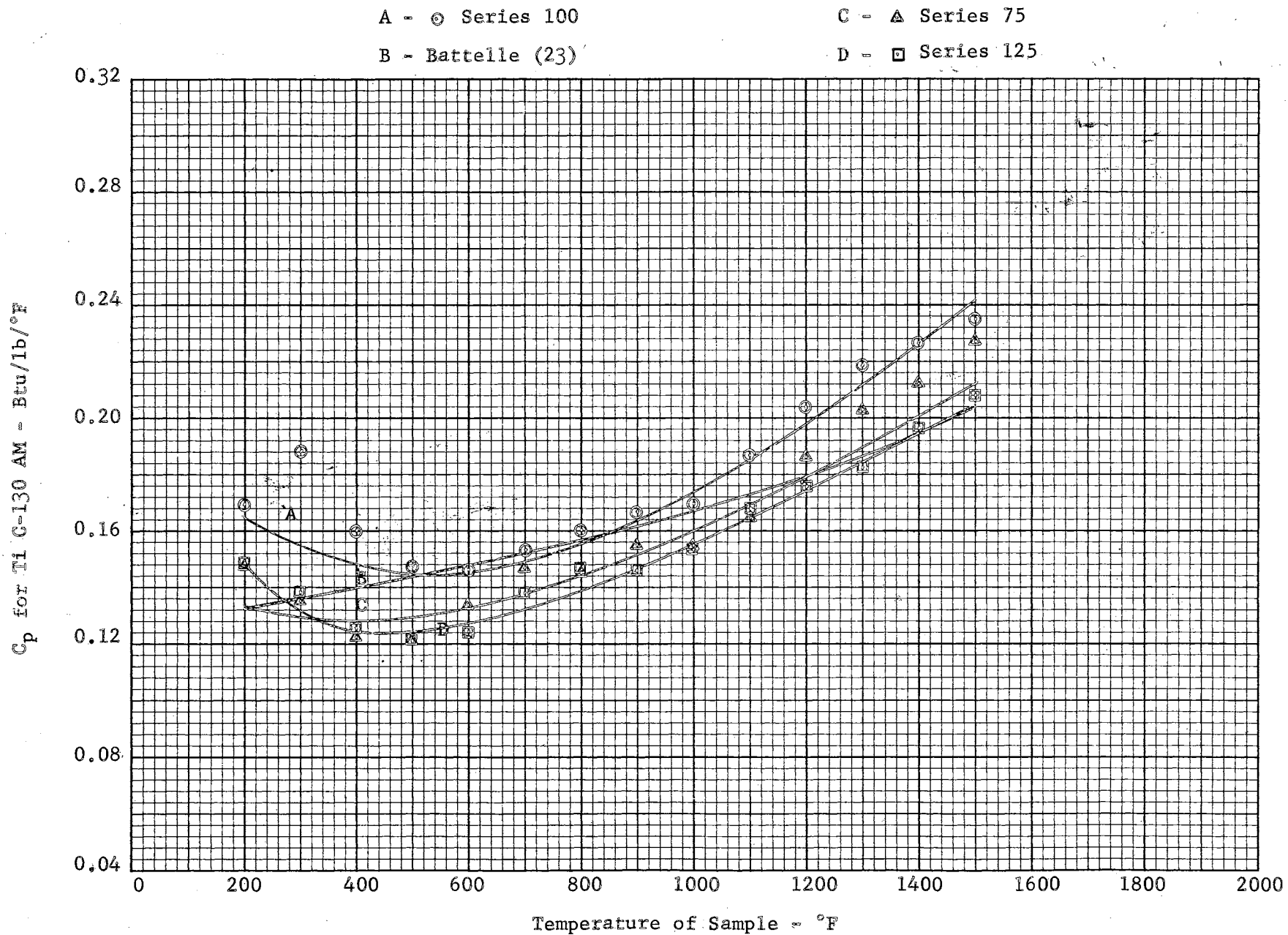


Figure 10. Specific Heat Data for Ti C-130 AM

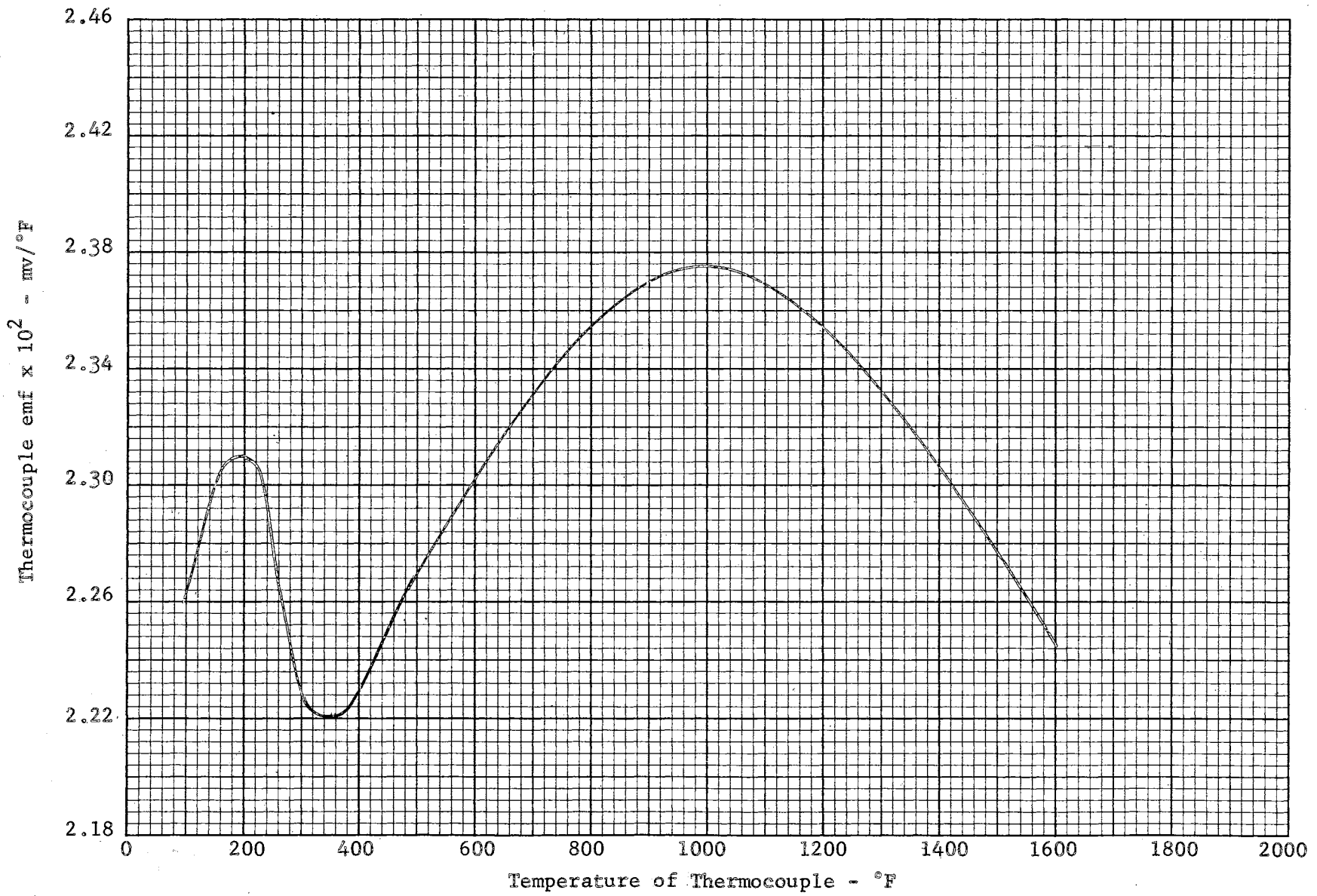


Figure 11.  $\text{mv}/^{\circ}\text{F}$  vs. Temperature for Chromel-Alumel Thermocouples

effect as the true  $mv/^{\circ}F$  curve rose and fell above and below the average value. It is thought that this is the cause of the sine wave variations. Further comparison between the  $mv/^{\circ}F$ -temperature curve and the slope curves shows a definite similarity in their characteristic shapes.

## CHAPTER VI

### CONCLUSIONS AND RECOMMENDATIONS

Probably one of the most outstanding achievements of this investigation was the design, construction, and very satisfactory performance of the heater element. Its low thermal lag characteristics, ease of construction, and apparent resistance to burnout make it a worthy choice in a laboratory furnace of this type.

The final design of the vacuum system proved to be practically airtight.

Determination of specific heats using this apparatus was very rapid, but more experimentation is necessary to attain a higher degree of accuracy in the experimental results.

After a careful study of the intriguing results of this investigation one is prompted to consider the feasibility of continuing the research to a much higher temperature. Before an attempt is made to continue the project to higher temperatures, however, it is recommended that the following suggestions be considered.

a. Investigate the effect of differences in emissivities of sample surfaces and the possibility of coating them with some common coating.

b. Secure a suitable controller to maintain a constant crucible wall temperature differential.

c. Use differential thermocouples that have a smooth mv/°F-temperature curve to determine if this will correct the sine wave effect in the

experimental results.

d. Investigate the possibility of irregular temperature distributions in the crucible walls due to the close proximity of the heater wires.

e. Analyze the temperature distributions in the sample using various heating rates and samples.

f. Examine critically the theory of the empty run to determine the best method of its temperature measurement.

g. Investigate the use of a metal radiation crucible.

h. Analyze a Russian paper by E. U. Nekhendzi (24) to determine the value of a parameter  $\beta_i = \frac{\alpha R}{\lambda}$  where

$\alpha$  = emissivity,

R = size factor,

$\lambda$  = thermal conductivity.

According to Nekhendzi this parameter can be controlled to optimize determination of thermal constants.

In addition to the above recommendations it is believed that some slight modifications could be made in the instrumentation of the system enabling the determination of thermal conductivities of metals in conjunction with their specific heats. (24). This should be of definite value and would increase the flexibility of the system.

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APPENDIXES

APPENDIX A

TIME-TEMPERATURE SLOPE ANALYSIS

TABLE II

TIME-TEMPERATURE SLOPE ANALYSIS FOR CHROMIUM

Test Run 25, Series 125

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ (°F)	Average Sample Temperature °F	Slope $\frac{\Delta t}{\Delta T}$
1.5	1.02	4.4	101.5	0.232
1.6	0.96	4.3	105.8	0.224
1.7	0.80	4.5	110.2	0.178
1.8	0.79	4.5	114.8	0.176
1.9	0.76	4.0	119.0	0.190
2.0	1.00	6.7	124.4	0.149
2.15	0.96	6.6	131.0	0.146
2.30	0.98	6.4	137.5	0.153
2.45	0.85	6.6	144.0	0.129
2.6	0.88	6.7	150.6	0.131
2.75	1.08	8.5	158.2	0.127
2.95	0.75	6.5	165.8	0.115
3.10	0.84	6.5	172.5	0.129
3.25	1.20	10.8	180.9	0.111

TABLE II (Cont'd)

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ (°F)	Average Sample Temperature °F	Slope $\frac{\Delta t}{\Delta T}$
3.5	1.19	10.7	191.6	0.111
3.75	1.15	10.6	202.3	0.109
4.00	1.09	10.9	213.0	0.100
4.25	1.06	11.0	224.0	0.096
4.50	1.09	10.8	234.9	0.101
4.75	1.08	11.0	245.8	0.098
5.00	1.03	11.2	256.9	0.092
5.25	1.03	11.0	268.0	0.094
5.50	1.08	11.2	279.1	0.096
5.75	1.03	11.3	290.4	0.091
6.00	1.06	11.0	301.5	0.096
6.25	1.02	11.5	312.8	0.089
6.50	1.01	11.2	324.1	0.090
6.75	1.06	11.3	335.4	0.094
7.00	1.03	11.5	346.8	0.090
7.25	1.05	11.0	358.0	0.095
7.50	1.01	11.5	369.2	0.088
7.75	1.06	11.0	380.5	0.096
8.00	1.06	11.3	391.6	0.094
8.25	1.06	11.2	402.9	0.095

TABLE II (Cont'd)

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ (°F)	Average Sample Temperature °F	Slope $\frac{\Delta t}{\Delta T}$
8.50				
	1.03	11.0	414.0	0.094
8.75				
	1.05	11.5	425.2	0.091
9.00				
	1.05	11.0	436.5	0.095
9.25				
	1.05	11.0	447.5	0.095
9.50				
	1.05	11.0	458.5	0.095
9.75				
	1.07	11.0	469.5	0.097
10.00				
	1.04	11.0	480.5	0.094
10.25				
	1.05	11.0	491.5	0.095
10.50				
	1.06	11.0	502.5	0.096
10.75				
	1.04	11.0	513.5	0.095
11.00				
	1.09	11.0	524.5	0.099
11.25				
	1.05	11.0	535.5	0.095
11.50				
	1.04	11.0	546.5	0.095
11.75				
	1.14	10.6	557.3	0.108
12.00				
	1.01	10.9	568.0	0.093
12.25				
	1.11	11.0	579.0	0.101
12.50				
	1.07	11.0	590.0	0.097
12.75				
	1.11	10.5	600.8	0.106
13.00				
	1.12	11.0	611.5	0.102
13.25				
	1.06	11.0	622.5	0.096

TABLE II (Cont'd)

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ (°F)	Average Sample Temperature °F	Slope $\frac{\Delta t}{\Delta T}$
13.50				
	1.11	10.5	633.2	0.106
13.75				
	1.07	10.8	643.9	0.099
14.00				
	1.12	10.7	654.6	0.105
14.25				
	1.10	11.0	665.5	0.100
14.50				
	1.10	10.5	676.2	0.105
14.75				
	1.13	10.8	686.9	0.105
15.00				
	1.09	10.7	697.6	0.102
15.25				
	1.13	10.7	708.4	0.106
15.50				
	1.10	10.8	719.5	0.102
15.75				
	1.10	10.5	729.8	0.105
16.00				
	1.10	10.7	790.4	0.103
16.25				
	1.12	10.6	751.3	0.106
16.50				
	1.08	10.7	761.6	0.101
16.75				
	1.13	10.5	772.2	0.107
17.00				
	1.07	10.8	782.9	0.099
17.25				
	1.14	10.7	793.6	0.107
17.50				
	1.06	10.5	804.2	0.101
17.75				
	1.12	10.5	814.8	0.107
18.00				
	1.12	11.0	825.5	0.102
18.25				
	1.10	10.5	836.2	0.105

TABLE II (Cont'd)

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ ( $^{\circ}F$ )	Average Sample Temperature $^{\circ}F$	Slope $\frac{\Delta t}{\Delta T}$
18.50				
	1.09	10.5	846.8	0.104
18.75				
	1.08	10.5	857.2	0.103
19.00				
	1.08	10.5	867.8	0.103
19.25				
	1.07	10.7	878.4	0.100
19.50				
	1.09	10.3	888.8	0.106
19.75				
	1.02	10.7	899.4	0.095
20.00				
	1.04	10.6	910.0	0.098
20.25				
	1.00	10.7	920.6	0.094
20.50				
	1.03	10.6	931.3	0.097
20.75				
	1.00	10.5	941.8	0.095
21.00				
	1.00	10.5	952.2	0.095
21.25				
	0.94	10.5	962.8	0.090
21.50				
	0.95	10.7	973.4	0.089
21.75				
	0.97	10.6	984.0	0.092
22.00				
	0.93	10.4	994.5	0.089
22.25				
	0.95	10.6	1005.0	0.0896
22.50				
	0.92	10.7	1015.6	0.086
22.75				
	0.96	10.5	1026.2	0.091
23.00				
	0.94	10.5	1036.8	0.090
23.25				
	0.95	10.5	1047.2	0.090

TABLE II (Cont'd)

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ ( $^{\circ}\text{F}$ )	Average Sample Temperature $^{\circ}\text{F}$	Slope $\frac{\Delta t}{\Delta T}$
23.50				
	0.92	10.5	1057.8	0.099
23.75				
	0.91	10.5	1068.2	0.087
24.00				
	0.94	10.5	1078.8	0.090
24.25				
	0.91	10.5	1089.2	0.087
24.50				
	0.93	10.8	1099.9	0.086
24.75				
	0.90	10.4	1110.5	0.086
25.00				
	0.92	10.6	1121.0	0.087
25.25				
	0.90	10.7	1131.6	0.084
25.50				
	0.90	10.5	1142.2	0.086
25.75				
	0.90	10.5	1152.8	0.086
26.00				
	0.90	10.5	1163.2	0.086
26.25				
	0.93	10.8	1173.9	0.086
26.50				
	0.89	10.7	1184.6	0.083
26.75				
	0.85	10.7	1195.4	0.079
27.00				
	0.90	10.6	1206.0	0.085
27.25				
	0.89	10.7	1216.6	0.083
27.50				
	0.90	10.7	1227.4	0.084
27.75				
	0.90	10.8	1238.1	0.083
28.00				
	0.90	10.5	1248.8	0.086
28.25				
	0.89	11.0	1259.5	0.081

TABLE II (Cont'd)

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ ( $^{\circ}F$ )	Average Sample Temperature $^{\circ}F$	Slope $\frac{\Delta t}{\Delta T}$
28.50				
	0.89	10.5	1270.2	0.085
28.75				
	0.87	10.5	1280.8	0.083
29.00				
	0.87	11.0	1291.5	0.079
29.25				
	0.88	10.5	1302.2	0.084
29.50				
	0.88	10.8	1312.9	0.082
29.75				
	0.86	10.7	1323.6	0.080
30.00				
	0.86	11.0	1334.5	0.078
30.25				
	0.87	10.5	1345.2	0.083
30.50				
	0.87	10.8	1355.9	0.081
30.75				
	0.87	10.7	1366.6	0.081
31.00				
	0.85	11.0	1377.5	0.077
31.25				
	0.83	10.7	1388.4	0.078
31.50				
	0.81	10.8	1399.1	0.075
31.75				
	0.85	10.8	1409.9	0.079
32.00				
	0.82	10.7	1420.6	0.077
32.25				
	0.84	11.0	1431.5	0.076
32.50				
	0.83	11.0	1442.5	0.076
32.75				
	0.83	11.0	1453.5	0.076
33.00				
	0.84	11.0	1464.5	0.076



TABLE II (Cont'd)

Potentiometer Reading (Millivolts)	$\Delta t$ (minutes)	$\Delta T$ ( $^{\circ}F$ )	Average Sample Temperature $^{\circ}F$	Slope $\frac{\Delta t}{\Delta T}$
33.25	0.84	11.0	1475.5	0.076
33.50	0.82	11.0	1486.5	0.075
33.75	0.82	11.0	1497.5	0.075
34.00	0.84	11.0	1508.5	0.076
34.25				

APPENDIX B

DETERMINATION OF SPECIFIC HEATS

TABLE III

DETERMINATION OF SPECIFIC HEATS FOR CHROMIUM

Sample Temperature °F	A $\frac{\Delta t}{\Delta T}$ Chromium	B $\frac{\Delta t}{\Delta T}$ Al <sub>2</sub> O <sub>3</sub>	C $\frac{\Delta t}{\Delta T}$ Empty	D A - C	E B + C	F $\frac{D}{E}$	G C <sub>p</sub> Al <sub>2</sub> O <sub>3</sub>	H $\frac{W_s}{W_u}$ $\frac{43.26}{74.49}$	I F <sub>x</sub> G <sub>x</sub> H C <sub>p</sub> Chromium
100	0.1830	0.1390	0.0429	0.1401	0.0961	1.583	0.191	0.581	0.1619
200	0.1060	0.1023	0.0373	0.0687	0.0650	1.066	0.213		0.1308
300	0.0923	0.100	0.0381	0.0542	0.0619	0.880	0.231		0.1175
400	0.0923	0.1073	0.0386	0.0537	0.0687	0.789	0.244		0.1110
500	0.0960	0.1156	0.0395	0.0565	0.0761	0.750	0.254		0.1096
600	0.1000	0.1209	0.0400	0.0600	0.0809	0.745	0.262		0.1130
700	0.1030	0.1228	0.0403	0.0627	0.0825	0.751	0.268		0.1184
800	0.1036	0.1218	0.0404	0.0632	0.0814	0.776	0.274		0.1236
900	0.0978	0.1172	0.0396	0.0582	0.0776	0.747	0.278		0.1210
1000	0.0904	0.1063	0.0368	0.0536	0.0695	0.765	0.282		0.1265
1100	0.0868	0.1009	0.0335	0.0533	0.0674	0.788	0.286		0.1314
1200	0.0842	0.0978	0.0315	0.0527	0.0663	0.794	0.288		0.1330
1300	0.0812	0.938	0.0301	0.0511	0.0637	0.802	0.291		0.1356
1400	0.0788	0.0891	0.0282	0.0496	0.0609	0.810	0.293		0.1389
1500	0.0742	0.0859	0.0291	0.0451	0.0568	0.801	0.295		0.1360

APPENDIX C

CHEMICAL ANALYSIS OF SAMPLES

TABLE IV

CHEMICAL ANALYSIS OF SAMPLES

Chromium Sample

Chromium	99.19%
Iron	0.11%
Carbon	0.01%
Oxygen	0.34%
Nitrogen	0.028%
Copper	0.005%
Sulphur	0.005%
Lead	0.001%

Rem-Cru Ti C-130 AM Sample

Titanium	92%
Aluminum	4%
Manganese	4%

VITA

MUREL EUGENE SCHLAPBACH

Candidate for the Degree of  
Master of Science

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TO OBTAIN THE SPECIFIC HEATS OF SOLIDS

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