A COMPARATIVE METHOD OF

SPECIFIC HEAT DETERMINATION

By

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CHAPTER I

INTRODUCTION

Heat measurements have long been of interest to scientists since most physical and chemical processes involve heat generation, its conversion into other forms of energy, or its ultimate transfer from one body to another. However, it has been only during recent years that engineers have also become vitally concerned with these phenomena. The sound barrier has relented before the onslaught of advancing technology, but in its place has appeared still another obstacle——the heat barrier. Researchers engaged in guided missile design, jet propulsion improvements, and techniques of inter-planetary space travel are constantly grasping for new materials which can survive higher and higher temperatures.

Today's research is further complicated by the ever increasing demand for techniques of rapid analysis. No longer can the engineer afford to delay a design several months while he awaits the precise determination of such design criteria as specific heats, thermal conductivities, and radiant emissivities of new materials that appear to be appropriate for his particular application. Since the engineer can ill-afford to produce a design which is based on chance, he must utilize the materials for which such heat characteristics have already been determined. In many cases this technique does not result in the most efficient design.

Calorimetry is the theory and practice of making measurements of quantities of heat. It has heretofore been considered axiomatic that the use of such techniques should be confined to the scientific laboratory under the careful supervision of a trained physicist or physical chemist. (1)

The thesis is taken here that certain principles of calorimetry can be adapted for use by the engineer in such manner as to eliminate the time delay which now intercedes and prevents immediate utilization of new materials.

The specific heat of a particular substance is the quantity of heat which must be added per unit mass of a substance to obtain a unit change in temperature. Mathematically, this may be expressed as follows:

$$C = \frac{Q}{m \bigtriangleup t}$$
 (I-1)

where

C = specific heat of the substance in B.T.U./lb/°F

Q = quantity of heat in B.T.U.

m = mass of the substance in 1bs.

 Δt = resultant temperature rise in °F

Approximate specific heat vs. temperature characteristics of a material must be known before transient heat flow problems can be solved. For this reason, a rapid method of specific heat determination would be of much importance to the design engineer of today. More particularly, to be of utmost value, such a method should: (A) be able to produce final results within a 24 hour period, (B) be reli-

able to within 10% of precise measurements, (2) (C) be adaptable to the high temperature range, (D) involve an inexpensive apparatus of elementary design, and (E) be so designed that a well-trained technician can perform the required operations.

Although some of the most prominent researchers in the field have been reluctant to advocate methods which depend upon heating or cooling rates, such a method of calorimetry was chosen as the type best suited to satisfy the above requirements. (3)

The advantages and disadvantages of this type and other types of calorimetry will be discussed in subsequent chapters.

CHAPTER II

A BRIEF HISTORICAL REVIEW OF CALORIMETRY

As suggested earlier, specific heat is the thermal capacity of unit mass of a given substance. Early researchers quickly recognized that water has the highest specific heat of all common substances. For this reason water was taken as a standard and assigned a specific heat of one. Later, investigators found that water, like other substances, has a specific heat which varies with the temperature. (4) (5) (6)

The specific heat of a substance may be determined either at constant pressure or constant volume. Roberts (7) gives an empirical expression which enables one to relate C_p and C_v . The relationship which he has given is:

$$C_{p} - C_{v} = \frac{0.0051}{T_{s}} C_{p}^{2} T$$
 (II-1)

where

 C_p = specific heat at constant pressure C_v = specific heat at constant volume T = absolute temperature of the solid T_s = melting point of the solid

Since the investigation posed by this thesis will be limited to an analysis of the specific heats of solids, the discussion in this chapter will not include constant volume determinations. Prior to 1800 Black (8) had discovered that when equal amounts of different substances at different temperatures were mixed, the equilibrium temperature which was finally reached by the mixture depended upon a characteristic of the particular substances. This characteristic has been defined as "specific heat".

Although these early experiments made use of rather crude apparatus, results of one percent accuracy were not unusual. (9) In the mid-eighteenth century Regnault improved upon Black's original design and thus obtained a higher degree of accuracy. (10) The essential items which were used in these investigations are shown in Figure 1 below.



Figure 1. Early Calorimetric Device

The specific heat of the sample in question could be calculated

by setting up the relationship

Heat Lost by Heat Gained by Heat Gained by (II-2) Hot Sample Cold Substance Calorimeter

Thus

 $m_h C_h (T_h - T_f) = (m_c C_c \neq m_k C_k) (T_f - T_c)$ (II-3) where the subscript "h" refers to the hot substance, "c" refers to the cold substance, "f" refers to the final temperature of the mixture, and "k" refers to the calorimeter.

The calorimeter may consist of any body into which heat can be introduced and measured. For example, it may take the form of a water type (9) or of a metal block type. (11) (12) (13) However, regardless of the form taken, this "method of mixtures" utilizes the basic concepts developed by Black. As used today, this method involves the determination of the change in enthalpy of a substance between the temperature of the furnace and that of the calorimeter. Then, by processes of differentiation, heat contents may be calculated from the relationship

$$C_{\mathbf{p}} = \left(\frac{\partial \mathbf{H}}{\partial \mathbf{T}}\right)_{\mathbf{p}} \tag{II-4}$$

White (14) has given the following two disadvantages of any "method of mixtures".

- The procedure is not always adequate for obtaining heat effects at transition points, especially if the heats of transition are small.
- (2) Many substances change state on heating and (for conditions of rapid cooling) they do not always

revert to their original state.

Kelley (3) gives three advantages of the "method of mixtures" as follows:

- (1) The method is direct and reasonably rapid.
- (2) Results are precise within the limits of reproducibility in behavior of the substance.
- (3) The method depends upon no extraneous assumptions.

The earliest uses of electrical heating in calorimetry were made by Nernst (15), Eucken (16), and Simon and Lange (17). Figure 2 represents a modification of Nernst's equipment as made by Simon and Lange.



Figure 2. An Early Adiabatic Calorimeter

Actually this calorimetric process (known as adiabatic vacuum

calorimetry) is a modification of the "method of mixtures" developed earlier by Black. Although it has been used mostly for low temperature analyses, Sucksmith and Potter (18) have done some work in the higher temperature ranges.

As shown in Figure 2, the substance to be investigated is placed in a thin-walled copper vessel. Inside this vessel are also located a heating coil and a resistance thermometer. Surrounding the vessel is a thermostat of brass which is equipped with a separate heating coil. Junctions of a differential thermocouple are placed in contact with the copper and brass. By adjusting the heating currents to make the thermocouples indicate zero, it is possible to prevent heat loss.

The power supplied to the internal heating coil is EI, and the temperature, T, can be measured as a function of time, t. If a graph is drawn with time as the ordinate and temperature as the abscissa, then the slope of the curve at any point is \underline{dt}_{T} . And, as previously \underline{dT}_{T}

$$C_{p} = \left(\frac{\partial_{H}}{\partial T}\right)_{p} = -\frac{EI}{J} - \frac{dt}{dT}$$
 (II-5)

where

J _ 4.185 Joules/Cal = 778.3 Ft-Lb/B.T.U.

From these relationships C_p can be determined at any desired temperature.

Bunsen (19), a famous German chemist, developed an ice calorimeter which, when skillfully used, has proved to be highly successful in measuring very small quantities of heat or in determining the specific heats of very small specimens. (20) Ginnings and Corruci-

ni (21) (22) have improved the technique and made modifications of the original method to the extent that their procedure is possibly the most accurate calorimetric process for high temperature measurements of specific heats. In Bunsen's instrument, Figure 3, the amount of melting was determined by volume rather than by weight.



Figure 3. Bunsen's Ice Calorimeter

A test-tube, heating chamber was sealed into a glass bulb which was filled partly with boiled, air-free water at 0°C, and partly with boiled mercury. The mercury column extended through an adjustable connection and finally ended in a capillary tube which was calibrated along a horizontal scale. The whole apparatus, with the exception of the capillary tube and the mouth of the test tube, was immersed in an ice bath or clean snow to minimize heat flow to or from the surroundings. A mantle of ice was made to form on the

outside of the test tube. This was accomplished either by bubbling ether inside the tube or by some other technique. Just prior to use, the capillary tube was adjusted in the sliding connection in such manner as to cause the mercury to stand near the extreme right end of the scale.

The body whose specific heat was to be measured was dropped into the test tube. It will be remembered that ice, on fusion, contracts. Hence, when equilibrium was finally reached, there had been a consequent decrease in volume due to the melting of some of the ice. This change in volume registered on the previously calibrated horizontal scale. Knowing the diameter of the capillary, the distance the mercury meniscus receded, and the density of the ice, it was possible to determine the exact mass of ice which had been melted and the heat which must have been given up by the body under examination.

The ice calorimeter is the most successful of all calorimetric devices in eliminating thermal leakage. (9) This is due to the fact that the heat transfer takes place within the interior of a mass of ice whose outer temperature, accurately zero, is the same (ideally) as that of other ice which surrounds it. In spite of these apparent advantages several difficulties are encountered in using such a method.

For example, in practice the ice encrusted on the test tube does not always have the same freezing point as the ice surrounding it. (23) This results in a drifting effect of the mercury in the capillary tube. Still another difficulty is encountered in maintaining a constant density of ice in the mantle which forms around the test tube.

Any variations (i.e. from calibration run to actual test) will result in errors in the specific heat determinations.

Although it is agreed that such methods of calorimetry will give excellent results when properly administered, the elaborate equipment and meticulous handling of the test procedure seem to point toward the inadvisability of utilizing such processes to accomplish the objectives outlined in Chapter I. For example, the time of test alone would banish this method from further consideration.

No attempt has been made here to cover the complete history of calorimetry. Willis (10), Fife (24) and others have given considerable attention to this aspect. In fact, entire books have been written on the subject. (9) However, an effort has been made to present a general background of calorimetry together with certain applications as applied by outstanding researchers in the field. Such a background is necessary since basic concepts as discussed here have been relied upon in the planning of this particular research. Techniques relating to the comparative method of calorimetry will be discussed in the following chapter.

CHAPTER III

THEORY OF THE COMPARATIVE METHOD OF CALORIMETRY

The first approach to comparative calorimetry was made between 1869 and 1891 by Pfaundler. His apparatus was characterized by the use of two calorimeters (twin calorimeters) which were constructed as much alike as possible. These calorimeters were heated by the same electrical current, with minute differences in their temperatures being measured by a sensitive thermoelement. Relatively high precision could be attained with this apparatus. (25) However, it has been most successfully applied to liquids---or'to the comparison of two samples which are nearly alike.

"Twin calorimetry" is limited since the method does not approach its maximum usefulness unless the two calorimeters are exactly alike. White (26) points out that

in a calibrated calorimeter practically all the errors come in temperature measurement, and most, though not the greatest of these, come in the "cooling correction"; that is, the determination of the effect of the thermal leakage between the calorimeter and the environment.

In recent years Smith (27) and Edmunds (28) have combined the concept of the comparative calorimeter with other basic calorimetric techniques (see Chapter II). The results of their investigations seem to indicate that a similarly designed apparatus might be devised which would satisfy the objectives of this thesis as previously outlined. According to Smith (27), the comparative method seems to

have utility in the determination of specific heats with fair accuracy and great ease, employing only apparatus available in every metallurgical or physical laboratory.

Although the previous investigators obtained satisfactory results, it should be recognized here that the maximum temperature attained in their investigations did not exceed 900 $^{\circ}$ F. As pointed out earlier in this thesis (p.3), there is need today for specific heat analyses in the higher temperature range (above 1000 $^{\circ}$ F).

The comparative method involves the electrical heating of a cylindrical sample in such manner that heat flows in a controlled manner into the sample. The specimen is placed in a refractory crucible of low thermal conductivity and, with the aid of differential thermocouples, the temperature gradient through the crucible wall is closely controlled. Although the thermal conductivity of the crucible varies with temperature, at any given temperature the heat flow will remain constant for any sample under test. This makes possible constant heat flow to any given test sample. Therefore, the heat received by any test sample (for a given temperature) will depend upon the elapsed time and consequently on the specific heat of the sample at the given temperature.

The heat supplied may be used for giving sensible heat to the sample, for transmitting sensible heat to the calorimeter, or for furnishing the latent heat necessary for any transformations within the sample. From this analysis the following heat balance results:

$$Q \Delta t_{s} = W_{s} C_{s} \Delta T_{s} + W_{e} C_{e} \Delta T_{s} + W_{s} L_{s}$$
(III-1)

where

Q = heat flow/unit time C_e = specific heat of the empty calorimeter C_s = specific heat of some "standard" specimen W_e = mass of the effective part of the empty calorimeter W_s = mass of the "standard" specimen ΔT = change in temperature during time Δt L_s = latent heat of any transformation that may occur within the temperature range ΔT

Now it is necessary to write Equation III-1 for calorimetry tests of (A) an empty run, (B) a test run utilizing a "standard" test specimen of known specific heat characteristics, i.e. synthetic sapphire, and (C) a test run where some test specimen of unknown specific heat characteristics is utilized.

(Empty) $Q \bigtriangleup t_e = W_e C_e \bigtriangleup T_e$ III-2

(Standard)
$$Q \bigtriangleup t_s = W_s C_s \bigtriangleup T_s + W_s C_e \bigtriangleup T_s$$
 III-3

(Unknown) $Q \Delta t_u = W_u C_u \Delta T_u + W_e C_e \Delta T_u$ III-4

Equation III-2
$$W_e C_e = Q \left(\Delta t_e \right) \qquad \text{III-5}$$

Substituting Equation III-5 into Equations III-3 and III-4 and rearranging:

$$W_{s} C_{s} = Q \left(\frac{\Delta^{t} s}{\Delta^{T} s} - \frac{\Delta^{t} e}{\Delta^{T} e} \right)$$
 III-6

and

From

$$u_{u} c_{u} = Q \left(\frac{\Delta t_{u}}{\Delta T_{u}} - \frac{\Delta t_{e}}{\Delta T_{e}} \right)$$
 III-7

Dividing Equation III-7 by III-6 and rearranging:

$$C_{u} = \frac{W_{s} C_{s}}{W_{u}} \begin{pmatrix} \frac{\Delta t_{u}}{\Delta T_{u}} & \frac{\Delta t_{e}}{\Delta T_{e}} \\ \frac{\Delta t_{s}}{\Delta T_{s}} & \frac{\Delta t_{e}}{\Delta T_{e}} \end{pmatrix} III-8$$

From Equation III-8, one can see that the specific heat of any substance (for a given temperature) may be obtained since the weight of the samples can be accurately determined, the specific heat characteristics of the standard material are known, and the time-temperature relationships Δ^{t} existing at the given temperature may be ob- $\overline{\Delta^{T}}$ tained by calorimetric testing.

Due to the higher temperatures experienced in this research considerable adjustments were necessary in applying the above relationships. However, these procedures will be discussed in a later chapter.

CHAPTER IV

DESCRIPTION OF THE TEST APPARATUS

In its final form the comparative calorimeter consisted of five main components: (A) the crucible and crucible base, (B) the heater, (C) the firebrick shield and evacuation chamber, (D) the standard and test cylinders, and (E) the temperature control and measuring apparatus.

Each of these constituents is briefly described in the following sections.

A. The Crucible And Crucible Base

The purpose of the crucible was two-fold. It served as a positive positioning device for the sample, and it also contained the differential thermocouples which were used in controlling the temperature gradient across the crucible wall. Since furnace temperatures of 2000 ^oF were anticipated, Armstrong (29) A-20 insulating fire brick was selected as a suitable crucible material. This heat resistant -2 -1 o -1material had a low thermal conductivity (1.536 Btu in.ft hr F) and its resistance to spalling was good.

Using an engine lathe, a two-inch diameter cylinder, three inches long, was first turned from the A-20 brick. Then, a 3/4-inch diameter hole $2\frac{1}{4}$ inches deep was drilled axially into the cylinder from one end. No tolerance was allowed to accomodate a 3/4-inch diameter sample since a tight fit between crucible and sample was desirable and since the

cavity could be reamed to fit by carefully using a test cylinder as a reamer.

A crucible lid (see Plate I) was also turned from the A-20 brick. Its geometric shape was formed of three cylinders of different diameters. The main body of the crucible lid was two inches in diameter, one end was one inch in diameter, and the other end was turned to a 3/4-inch diameter in order that it might be fitted into the axially drilled crucible. In this manner the test cylinder could be completely covered by the A-20 brick. Due to the thickness of brick at both ends of the test cylinders, heat flow was generally confined to flow through the lateral surface.

The differential thermocouples were composed of B.& S. Gauge No.18 Chromel (90 Ni, 10 Cr) and Alumel (95 Ni + Al + Si + Mn) wires. Figure 4 indicates the location of these different thermocouples with relation to the crucible proper.



Figure 4 The Crucible



THE CRUCIBLE, CRUCIBLE LID AND CRUCIBLE BASE

Prior to installation of the differential thermocouple wires, four accomodation holes were drilled through the three-inch long cylinder using a No. 52 twist drill. As can be seen in Figure 4, the accomodation holes were alternately placed at 90° in order to avoid excessive stress concentrations which might have resulted in failure of the crucible. Also, the holes alternated in position from the outside to the inside of the crucible wall with a radial distance of 3/8-inch separating the outside and inside locations.

Since the accomodation holes were not much larger than the wire size, it was desirable to form the four Chromel-Alumel thermocouple junctions without allowing any change in cross sectional area. Utilizing the butt-welding mechanism of a Model ML Do-All band saw, thermocouple junctions of constant cross sectional area were formed. This technique also increased the sensitivity of the thermocouples.(30)

By utilizing two sets of differential thermocouples in series, the resultant E.M.F. produced was magnified. Also, the averaging effect thus obtained was desirable.

Chromel leads were utilized in order that the Alumel wire, which under certain conditions is subject to embrittlement (31) (32), would be contained within the body of the crucible. The thermocouple leads were extended through hermetic seals in the walls of the evacuation chamber and thence to a ballistic galvanometer.

A crucible base in the form of a thin wall cylinder was constructed to hold the crucible, and it was so designed that the crucible was positively positioned. The supporting base (see Plate I) rested on

three equally spaced ceramic legs which were themselves secured to the crucible base by means of Sauereisen Cement No. 6. This type of arrangement allowed for easy removal of the crucible without thereby disturbing the positive positioning feature. Armstrong A-20 brick was also used in the construction of this support.

As a safeguard, in the case of possible crucible fracture, two bands of B. & S. No. 30 Nichrome wire were placed around the top and bottom of the crucible.

B. The Heater

The component of the apparatus which was most difficult to construct was the heater. Previous investigators (2), (27) had indicated that improvement was needed with regard to the furnace lag characteristics. For this reason, the two separate heating circuits were suspended from eight equally spaced support units. These thin, heating-wire supports were individually cut from an A-20 brick (see Plates II and III). In this way the effective mass of the heater was kept at a minimum.

Two heating circuits were designed and installed, although later test runs indicated that one circuit would have been adequate for sample temperatures of 1800 °F. Chromel heater wire, B. & S. No. 18, was used as the resistance material in both heating circuits. Each heating circuit was made on an engine lathe by coiling the wire around a 1/16-inch diameter mandrel. The electrical resistance was ten ohms for the inside heating circuit and eleven ohms for the outside heating circuit. In general, the heating-circuit design procedures as outlined

PLATE II THE CALORILETER HEATER







PLATE III THE COMPARATIVE CALORIMETER by the Driver-Harris Co. (33) were followed. By experience it was found that the heating wires tend to burn apart at the terminals. This was remedied by crimping a four-inch length of a 1/8-inch diameter copper tubing to each end of the coiled heater-circuits. (34)

The heater was mounted securely on a two inch thick base which was constructed of A-20 brick. An inner furnace lid, also made of A-20 brick, completed the furnace unit.

In the case of fracture (of the A-20 brick heating wire supports) the fault was repaired using Sauereisen Cement No. 6 which is electrically non-conductive to temperatures of 3000 ^OF. (35)

C. The Firebrick Shield and Evacuation Chamber

The heater was surrounded completely by a laminated, cylindrical, insulating shield. This unit was composed largely of a two-inch thick cylinder of A-20 brick which had a 1/4-inch thick interior coating of Armstrong Insulating Cement No. 166. The exterior of this shield was surrounded with 1/8 inch of fiberglass insulation, and the entire unit was then wrapped in four concentric layers of Reynolds aluminum foil (see Plate IV).

The vacuum chamber was made of a piece of steel pipe two feet long. One end was sealed. The chamber had an inside diameter of thirteen inches and a wall thickness of 3/8 inches. A two-inch wide flange was welded to the top end for purposes of holding a gum rubber gasket and to receive a 3/8-inch thick steel lid complete with twelve 3/4-inch diameter retaining bolts.

This system was evacuated to 29 inches of mercury using a Cenco

PLATE IV COMPONENTS OF THE COMPARATIVE CALORIMETER



Hyvac vacuum pump. A surge tank was located between the vacuum pump and the evacuation chamber as a safety device to catch any vacuum pump oil which might leak back through the line between consecutive test runs. After evacuation, the evacuation chamber was filled with argon to a gage pressure of one pound per square inch.

Since commercially manufactured hermetic seals were not readily available, brass tube fittings were altered for the purpose of bringing the thermocouple wires and electrical leads through the side of the evacuation chamber. This was accomplished by adapting several half-unions and accompanying flare nuts to receive Micarta insulating bushings. Prior to tightening the nut onto each fitting, a piece of Eberhart Faber kneaded eraser was inserted into the cavity which had been machined into the untapered end of the half-union. A pressuretight seal was formed by tightening the nut.

The evacuation chamber was equipped with a manually operated relief valve and a pressure gage. The gage was calibrated from 30 inches of (mercury) vacuum to 15 pounds per square inch pressure.

D. The Standard and Test Cylinders

Since the success of this research was largely dependent upon securing a dependable standard of known specific heat characteristics, a 3/4-inch diameter by 1 1/2-inch long machined cylinder of synthetic sapphire (Al₂0₃) was donated to the Oklahoma Institute of Technology by the Linde Air Products Company, New York, New York, for use in this investigation. This material was known to possess chemical stability in air up to its melting point (about 2300 ^oK). It was also commer-


cially available with a very high purity, its specific heat characteristics were accurately known, it was non-volatile, non-hygroscopic, and it apparently had no solid-solid transitions. (36) Figure 5 indicates the specific heat characteristics of synthetic sapphire (Al₂O₃) as determined by Ginnings and Furukawa (36) of the National Bureau of Standards. Drop calorimetric techniques were used with a Bunsen ice calorimeter and the accuracy obtained was believed to be in the range of 0.2 per cent. (37)

Each test sample was machined to a diameter of 0.750 ± 0.005 inch and to a length of 1.500 ± 0.005 inch. A 1/16 inch hole 3/4 inch deep (for thermocouple accomodation) was drilled axially into the sample from one end.

Altogether over twenty different samples were investigated. These included specimen of copper, brass, vanadium, nickel, zirconium, iodide titanium, commercially pure titanium, and various alloys of titanium. Table I gives a tabulation of the chemical impurities present in each of the materials used.

Plate V shows the synthetic sapphire (Al_2O_3) test standard and the other test cylinders.

E. The Temperature Measuring and Control Apparatus

A thermocouple unit made by the Thermo Electric Co., Saddlebrook, N. J., was utilized to measure the temperature of the test samples. It was made from B. & S. No. 30 Chromel-Alumel wire, and it was sheathed with incomel for protection against corresive atmospheres. The small diameter wire was selected to increase the speed of response and to

TABLE I

CHEMICAL ANALYSES OF TEST SAMPLES

Supplier	Material	% Impurities																		
		Al	С	Co	Cr	Cu	Fe	H	Hf	Mg	Mn	N	Ni	0	РЪ	Si	Ti	W	v	Zn
Rem-Cru Titanium, Inc.	Unalloyed Ti A-55																			
Rem-Cru Titanium, Inc.	TI C-110M										8									
Rem-Cru Titanium	Ti C-130AM	4									4									
Titanium Metals Corp. of Am.	Unalloyed Ti 55A		.10				12	.015				.05								
Titanium Metals Corp. of Am.	Unalloyed Ti 100A		.10				20	.015				.08				.04		.02		
Battelle Memorial Institute	Lodide Titanium		.03					*				.008		.030						
Cramet, Inc.	Commercially Pure 1	Fi	.048				24	.004		.08		.046		.112						
Mallory-Sharon Titanium Corp.	Commercially Pure	ſi	.10			•	45	.015				.04								
Republic Steel Corp.	Commercially Pure 1	Fi	.079				26	.013				.034								
Metals Research Laboratories	Commercially Pure	Fi	.04					.009				.007		.05						
Metals Research Laboratories	Vanadium		.071					.008				.097		.083						
Metals Research Laboratories	Zirconium	.01	.06		.01	.(01		02	.06	.01	.01	.007				.005		.005	
International Nickel Co.	Commercial Nickel	"A"		.01																
International Nickel Co.	"Cobalt Free" Nickel	1																		
American Brass Co.	99.94% Pure Copper																			
American Brass Co.	Brass (Alloy 271)					61.5								3	3.25				3	5.2
Linde Air Products Co.	Synthetic Sapphire	(A12	03)																	

* 630 parts per million of hydrogen

PLATE V TEST CYLINDERS



decrease errors due to radiation and stem-conduction. (30) Chromel-Alumel thermocouples were also known to be particularly useful in the temperature range 1400-2200 ^OF. Lead wires of cheaper material (iron alloy No. 125) with thermoelectric characteristics similar to those of Chromel-Alumel were used. Calibration data for the thermocouple, used to determine the test sample temperature, are included in Appendix A. Plate VI shows the order of assembly of this thermocouple into the test sample and crucible.

The Chromel differential thermocouple leads (which were attached to the crucible at one end) were connected to a Leeds & Northrup, ballistic, type P, galvanometer (sensitivity of $0.011 \,\mu c/mm$). The external critical damping was 52 ohms and the period was 25.55 seconds. To obtain a suitable temperature gradient across the crucible wall, it was necessary to insert a 620-ohm resistance of constantan wire.

An Argus filmstrip projector was used to project a beam of light onto the galvanometer mirror and thence into a light barrier where the position of the image thus formed was controlled closely.

The voltage input to the heating circuit was controlled manually with the aid of a General Radio Co. Variac, Series V-20.

Test sample temperatures were recorded initially with the aid of a Leeds and Northrup Micromax Recorder, Model S. However, this was later discontinued in favor of a Leeds and Northrup Portable Potentiometer, No. 8662 and a continuous timer. An external cold junction was used in conjunction with this latter configuration.

PLATE VI ASSEMBLY OF THE TEMPERATURE FEASURING THERMCCOUPLE



CHAPTER V

EXPERIMENTAL PROCEDURES AND DATA

The arrangement of experimental equipment is shown by Plate VII. All components were in close propinquity to each other in order that one operator could manipulate the temperature controls and other measuring apparatus. It was found that once the equipment had been set up, a single operator could record quite easily the necessary calorimetric test data.

Prior to the actual test run, the calorimeter was loaded with a test cylinder. Then the chamber was evacuated and filled with argon as described previously in Chapter IV. As the run progressed and as the temperature within the evacuation chamber increased, it was necessary to adjust the manually controlled relief valve so that a constant pressure within the calorimeter might be maintained. The practicability of this procedure will be discussed in the following chapter.

The actual testing procedure was not complicated. Once the calorimeter was contained within a suitable environment, and the potentiometer balanced, the research technique became somewhat routine. Following the aforementioned steps, electrical current was allowed to flow through one heating circuit and the temperature gradient within the crucible wall was thereby brought to operating conditions.

PLATE VII ARRANGEMENT OF EXPERIMENTAL EQUIPMENT





At the beginning of the test when no current flow existed in the differential thermocouple circuit, the image which was reflected from the galvanometer mirror was located at a null-point position. However, as the temperature on the outside of the crucible increased, the differential thermocouples in the crucible wall would sense this condition and the E.M.F. thus produced would cause the galvanometer mirror (and image) to deflect. Since the magnitude of the arc traversed by the mirror image was a direct indication of the difference in temperature across the crucible wall, any desired temperature differential could be maintained by causing the mirror image to remain motionless since the E.M.F./degree vs. temperature curve was practically a horizontal line. This was accomplished by a slight but continuous manual adjustment of the Variac.

A series of tests were run on synthetic sapphire (Al_2O_3) , copper, unalloyed titanium, and two alloys of titanium while utilizing a temperature differential across the crucible wall of approximately 113 °F. These data are referred to in this thesis as "Test Series A". During the progress of the research it was necessary to replace the furnace base and to make some slight adjustment in the precise location of the crucible within the calorimeter. After making these adjustments, test data were obtained for vanadium, zirconium, nickel, iodide titanium, and unalloyed titanium samples. These latter data, referred to as "Test Series B", were obtained while utilizing a temperature differential across the crucible wall of approximately 109 °F.



Figure 6. Typical Variac Voltage Settings

The duration of the calorimetric test depended upon the specific heat characteristics of the sample under consideration. Hence, the total testing time for the materials investigated varied from a minimum of five hours to a maximum of eight hours.

To prevent the adjusting procedure (which was necessary in the controlling of the Variac voltage) from becoming a tedious task, a series of voltage-temperature relationships were plotted. (Figure 6) The data for these curves were taken from trial runs, and they served to aid the operator in estimating the voltage setting which would be necessary for any given sample temperature. Using this technique, the temperature differential across the crucible wall never varied more than $\frac{1}{2}$ or F.

The theory of comparative calorimetry as herein applied necessitates that the researcher know the time-temperature slope relationships (at any given temperature) for the standard, the empty calorimeter, and the unknown sample. (see Equation III-8)

Tables III, IV, V, VI, VII, and VIII of Appendix B include abridged tables of data which were recorded for Test Series A. In general, the potentiometer and time readings were recorded every 0.50 millivolt as the temperature increased.

For comparative calorimetry to be successful, it is necessary that the equipment (calorimeter) give reproducible results. Figures 7 and 8 show that the time-temperature slopes for the standard (sapphire) and the empty container in Runs No. 46 and 47 were duplicated later in the research by Runs No. 53 and 52. The solid





Figure 8. Smoothed Time-Temperature Slopes for Empty Container

lines which are drawn through these data indicate a smoothed curve whose position was determined by the method of moving averages. Factors relating to the use of this curve fitting technique will be discussed in Chapter VI.

The first analysis in the research was concerned with the specific heat characteristics of a sample of unalloyed titanium which was furnished by Rem-Cru Titanium, Inc., Midland, Pennsylvania. This particular material was chosen because known specific heat data of one per cent accuracy were available for purposes of comparison. (38) These data had been supplied to Rem-Cru by the Battelle Memorial Institute, Columbus, Ohio, and it was known that an ice calorimeter had been used in determining these data. Figure 9 gives the timetemperature slopes for this material as determined by comparative calorimetry.

Figures 10, 11, 12, and 13 include other Test Series A timetemperature slope relationships as were determined by the comparative calorimeter for copper, Rem-Cru Ti C-110M, Rem-Cru Ti C-130AM, and Titanium Metals Corporation of America Ti 55A.

As previously indicated, Test Series A and Test Series B were not operated under the same differential temperature characteristics. It was therefore anticipated that, prior to running Test Series B, a new time-temperature slope relationship would be needed for the standard (sapphire). Figure 14 shows the new slope curve which was obtained for use in Test Series B. A comparison of the results of the two test series is also indicated on this graph.







Figure 10. Smoothed Time-Temperature Slopes for Copper

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Figure 11. Smoothed Time-Temperature Slopes for Rem-Cru Titanium C-110M

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Figure 12. Smoothed Time-Temperature Slopes for Rem-Cru Titanium C-130 AM

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Figure 13. Smoothed Time-Temperature Slopes for Titanium Metals Corporation Ti 55A

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Figure 14. Smoothed Time-Temperature Slopes for Sapphire (A1203)

Figures 15, 16, 17, 18, and 19 indicate the time-temperature slope relationships of Test Series B for the materials, vanadium, Republic Steel (Cleveland, Ohio) unalloyed titanium, iodide titanium, and nickel. As will be described later, these data were utilized to determine experimental specific heat vs. temperature curves for the materials.



Figure 15. Smoothed Time-Temperature Slopes for Vanadium









Figure 17. Smoothed Time-Temperature Slopes for Iodide Titanium



Figure 18. Smoothed Time-Temperature Slopes for Zirconium



Figure 19. Smoothed Time-Temperature Slopes for Nickel (Cobalt Free)

CHAPTER VI

DISCUSSION OF EXPERIMENTAL TECHNIQUES AND RESULTS

This research generally proceeded according to the theory of comparative calorimetry as summarized in Chapter III. However, certain basic modifications were made during the progress of the investigation which, it is believed, tend to increase the usefulness of the technique.

As noted previously, the first material investigated was Rem-Cru Ti A-55, and the time-temperature slope data given in Figure 9 was used to calculate the specific heat characteristics of the material. Figure 20 is a graphical representation of these results, while Table X (Appendix D) gives a tabular synopsis of the data. As directed by Equation III-8, the "test-run" empty curve (Figure 8) was used in the determination of these specific heat characteristics.

On analyzing the results obtained, it was determined that the per cent error obtained from the test results was under 6 per cent for temperatures up to 1200 °F. Above this temperature range the error increased, and at 1400 °F the per cent error was almost 14 per cent. This seemed to indicate that some change was taking place within the calorimeter at approximately 1200 °F, and indications were that the error was magnified as the temperature increased.

It was suspected that this difficulty was being caused by radiation losses which occurred during the empty run. (2) Since





such radiation would vary with the fourth power of the absolute temperature, it was decided to completely eliminate the "actual" empty run.

An examination of Equation III-8 revealed that if the time-temperature slope for the empty container were solved for, the resulting mathematical expression would be:

$$\frac{\Delta t_{e}}{\Delta T_{e}} = \frac{\left[\underbrace{W_{u} \ C_{u}} \left(\underbrace{\Delta^{t}_{s}}{\Delta T_{s}} \right) \right]_{-} \left[\underbrace{W_{s} \ C_{s}} \left(\underbrace{\Delta^{t}_{u}}{\Delta T_{u}} \right) \right]}{W_{u} \ C_{u} - W_{s} \ C_{s}}$$
 VI-1

Note: let "u" represent a second standard instead of an unknown.

This analysis indicated that if <u>two</u> standards were available, it would be possible to solve for a "theoretical." empty time-temperature slope curve. This resultant curve would represent an empty curve which, if it had existed in actuality, would have given 0 per cent error if one of the two materials used had been serving as a "known" and the other had been serving an an "unknown." Apparently radiation losses and other difficulties due to unknown aspects would thereby be largely eliminated.

Figure 21 and Table IX (Appendix C) indicate the results of a mathematical analysis which was obtained by using sapphire (Al_2O_3) and Rem-Cru Ti A-55 as the two necessary standards in Equation VI-1. These two particular materials were chosen because accurate specific heat characteristics of both materials were known, and because the time-temperature slopes of the two materials (for a given temperature) were not in proximity.

The first sample which was analyzed using this new method of analysis was 99.9 per cent pure copper. Figure 22 indicates the









experimental results of this test. Kelley's data (3) are shown by the dashed line. He believed his data to be accurate to within 0.5 per cent. Using Kelley's results as a basis, the experimental test results varied from 1.0-2.3 per cent error.

As can be seen by examining the plotted values, all experimental tests showed a tendency for the data to assume the characteristics of a sine wave. This differed from the random type distribution which had been expected. No physical phenomena were discovered which would account for this consistent behavior. This factor apparently did not detract greatly from the precision of the investigation, but rather served as an annoyance during curve fitting procedures. On the graphs included here, the solid lines denote smoothed or averaged paths through the test data while the dashed lines indicate the appropriate data to which the research is compared.

Other materials which were investigated in Test Series A were Rem-Cru Ti C-110M, Rem-Cru Ti C-130AM, and unalloyed titanium furnished by the Titanium Metals Corporation of America. Of these materials, only the results of the two Rem-Cru alloys could be compared with previously determined specific heat characteristics. In the case of the C-110M, the per cent error was consistently less than 7 per cent. For C-130AM, the per cent error varied from 1.5 per cent at low temperatures to 17 per cent in the region of the transition phase. Since no other appropriate research results were available for comparison with the unalloyed titanium, the dashed line in Figure 25 represents specific heat data for 99.9 per cent



Figure 23. Specific Heat Data for Rem-Cru Titanium C-110M



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To/dI/uf8 ni MAOE1-0 muinstiT ur0-mes rol q0



Figure 25. Specific Heat Data for Titanium Metals Corporation Ti 55A

pure (iodide) titanium. The specific heat curve of the unalloyed titanium apparently follows the same general trend as that of iodide titanium except that it is generally some 15 per cent higher.

Test Series B involved the specific heat determinations of five materials: vanadium, nickel, iodide titanium, zirconium, and a sample of unalloyed titanium which was manufactured by Republic Steel. Although it was necessary to use the revised time-temperature slope curve for sapphire (Figure 14), the use of the theoretical (calculated) empty curve was continued. (Figure 21)

Kelley (3) reported that he believed the specific heat data for vanadium to be accurate to within 3 per cent. Compared to Kelley's data, the test specific heat values varied from 1.7-7.5 per cent error.

The data used for comparative purposes for nickel were believed to be accurate to within 0.5 per cent. (3) Here, the test data did not exceed a 7 per cent error and location of the transition point was missed by only one degree Fahrenheit.

Although the precision of the data available for comparison with iodide titanium (39) was not known, it was believed to be accurate to within one per cent. Test results corresponded closely with these reference data. Except in the transition range, the maximum error did not exceed 7 per cent. The phase change took place more quickly than did that material described in the literature. (39)

As can be seen from Table I, the zirconium sample had many impurities in it. For this reason, it was not known which of the






Figure 27. Specific Heat Data for Nickel (Cobalt Free)









references listed (40) (41) most closely resembled the test specimen in chemical analysis. However, it is interesting to note that the test results fell almost mid-way between the data published by the other investigators. Unfortunately, neither of the previous researchers recorded test data in the vicinity of the transition point.

The last test sample analyzed was an additional sample of unalloyed titanium (Figure 30). Comparison was once again made to iodide titanium.

It is interesting to observe that the best apparent results with this apparatus were obtained for materials whose specific heat characteristics had been determined previously with the greatest precision. Thus, it is probable that specific heat values determined by this research are actually closer to the true values than has been evidenced by the graphical comparisons.

Certain improvements in design can be made which undoubtedly would improve the performance of the comparative calorimeter. These suggestions will be discussed in detail in Chapter VII. However, the technique of applying experimental applications to Equation VI-1 demands that the investigator have an understanding of the inherent weakness in this procedure. For example, both numerator and denominator of the equation involve differences, and this type of calculation inevitably leads to difficulty--especially where the two quantities involved are of similar magnitude.

Since the determination of time-temperature slopes plays a major role in the success or failure of comparative calorimetry, it is





Figure 30. Specific Heat Data for Republic Steel Commercially Pure Titanium

important that the investigator have some analytical basis for use in the technique of curve fitting--particularly in the case of the standard materials. The method of "moving averages" was used in this investigation for the purpose of establishing the "slope" curve for synthetic sapphire (Al_2O_3) and Rem-Cru Ti A-55. This method might also be applied to the slope data of the other test specimen.

The method of "moving averages" is a statistical process of smoothing out the fluctuations which normally exist in raw data. In general, it consists of a series of successive averages which are individually secured from the series of readings by dropping the first item in each group averaged and including the next in the series--thus obtaining the next average. (42) This method has advantages over other methods in that: (A) Computations are simple, (B) computations are not time consuming, and (C) computations may be used to present data which otherwise would involve the fitting of complex mathematical expressions. Disadvantages of this curve fitting technique are: (A) Since the moving average is computed by the use of the arithmetic mean, it is greatly affected by extreme values. Because of this fact, the moving average will be pulled out of line by invalid points. (B) If the moving average is utilized where the trend is that of a concave curve, the resulting calculations will assume a form higher than the true trend at all points. If the trend is convex, the resulting calculations will be correspondingly lower. (C) The method is not purely objective since the number of successive data to be averaged in each group depends upon the judgment

of the researcher. (D) The moving average method cannot be utilized to the extent of the data since the last point in the trend must occur several readings prior to the end of the recorded data. In spite of these objections, however, it is believed that the technique described is the most appropriate for purposes of comparative calorimetry.

It is possible that the test procedure could have been simplified by allowing the pressure within the calorimeter to increase with the temperature instead of "bleeding-off" the pressure manually as it increased. Even though the process is supposed to occur at constant pressure, for all practical purposes the specific heat of solids is insensitive to changes in pressure. (1) This alteration might also be desirable from the standpoint that it would allow for an atmosphere of constant density during calorimetric testing.

Possible sources of error might be attributed to the differences which existed in sample emissivities and to the sample oxidation which resulted from using impure argon.

CHAPTER VII

CONCLUSIONS AND RECOMMENDATIONS

A comparative calorimeter has been designed, constructed, and tested in such manner that (A) it can serve as a reliable means of specific heat determination, (B) it is relatively inexpensive to build, (C) results can be produced within an eight hour period, and (D) one operator can handle all test procedures satisfactorily.

Although improvements in the apparatus can be made, the preponderance of data obtained from this research clearly shows that processes of comparative calorimetry can give the engineer desired specific heat vs. temperature characteristics of homogenous solid materials. These data are well within $\frac{4}{2}$ 10 per cent of the selected standards, which makes them suitable for design criteria. In fact, except for temperature ranges involving solid-solid transitions, the majority of the results were nearer to a per cent error of $\frac{4}{2}$ 6 per cent.

Alterations which might improve this technique include (A) the coating of all samples with a common material to equalize the differences in emissivities, (B) improving the furnace and calorimeter design, (C) designing a more effective evacuation system, (D) obtaining an oxygen-free test atmosphere, and (E) securing several "standards" for use in improving the calibration of the comparative calorimeter.

Two possible courses of action, with regard to the emissivity problem, which might be considered include the application of an evaporated chrome coating to all test cylinders, and the utilization of an inorganic coating (such as the 3M Sealer EC-1137, which is manufactured by the Minnesota Mining and Manufacturing Co., Detroit, Michigan.) Inorganic coatings are limited, at the present, to temperatures of 2000 ^oF although improved products may soon be available. The chrome coating may not be the complete answer either since diffusion rates become more rapid at elevated temperatures. (43) This would indicate that metallic coating of samples might be of limited value.

It is believed that the furnace design could be improved by reducing the size of the apparatus. White (9), for example, believes this to be desirable.

Improving the efficiency of the evacuation system is purely a mechanical problem. For example, an "O Ring" seal might be desirable to replace the conventional gasket and bolt arrangement.

Oxygen can be removed from the argon by first passing this gas over titanium chips or turnings which have been heated to $650 \,^{\circ}C$ (44). Another possibility would be to replace the argon with helium, which has given quite satisfactory results in previous research of this nature. (2)

Before much improvement can be made over present investigations, it will be necessary to secure several standards for use in conjunction with the synthetic sapphire (Al₂O₃). Vanadium and chromium might be suitable for this purpose. However, it is very important that specific heat characteristics for these materials be known as precisely as for that of the sapphire.

Recommendations for future research in comparative calorimetry include (A) adaption of this type apparatus to the 2000 $^{\circ}$ F - 3000 $^{\circ}$ F temperature range, (B) investigation into the cause, and elimination, of the "sine wave" effect which is present in the calculated specific heat data, and (C) to study the results of using different combinations of materials in arriving at the most reliable "theoretical" empty curve.

This thesis called for an investigation of specific heat determinations for temperatures up to 1800 $^{\circ}$ F, which is double the temperature range previously investigated by comparative calorimetry. However, due to the creation of the "theoretical" empty curve, the comparative calorimeter now seems to be an excellent tool for even higher temperature investigations.

The "sine wave" effect is evidentally a characteristic inherent within the calorimeter. This should be eliminated for higher temperature investigations.

Although the use of synthetic sapphire (Al₂O₃) and Rem-Cru unalloyed titanium served quite well for the determination of a "theoretical" empty curve, it would be of much value to know what criteria are necessary and desirable for such use. Possibly other combinations would give more precise results.

It is hoped that other investigators will solve these and other related problems connected with this improved calorimetric technique.

SELECTED BIBLIOGRAPHY

- Zemansky, Mark W., <u>Heat and Thermodynamics</u> (McGraw-Hill: New York, 1937), p. 86.
- Rea, Joseph A., "The Construction of a Furnace Calorimeter and the Evaluation of a Method of Thermal Analysis for Obtaining the Specific Heats of Solids at High Temperatures" (unpub. thesis, Oklahoma A.& M. College, 1956), p. 4.
- 3. Kelley, K. K., <u>Contributions to the Data on Theoretical Metal-</u> <u>lurgy</u> (Bureau of Mines Bul. 476, 1949) p. 2.
- 4. Callendar, Phil. Trans., A. <u>212</u>: 30 (1912).
- 5. Jaeger and Steinwehr, Ann. Phys. Lpz., 64: 305 (1921).
- 6. Osborne, Nathan S. et al., J. Res. Bur. Stand., 23: 197 (1939).
- 7. Roberts, J. K. and Miller, A. R., <u>Heat and Thermodynamics</u> (Interscience Pub.: New York, 1954), p. 210.
- 8. Black, J., quoted from Roberts, J. K. and Miller, A. R., <u>Heat and</u> <u>Thermodynamics</u>, (Interscience Pub.: New York, 1954), p. 45.
- 9. White, Walter, P., <u>The Modern Calorimeter</u> (Chemical Catalogue Company: New York), 1928, p. 17.
- 10. Willis, William R., "A New Furnace Calorimeter" (unpub. Ph. D. dissertation, Oklahoma A. & M. College, 1950), p. 6.
- 11. Haeger, F. M. and Rosenbohm, E., Proc. Acad. Sci. (Amsterdam), 30: 905 (1927).
- 12. Southard, J. C., J. Am. Chem. Soc., 63: 3142 (1941).
- 13. Kelley, K. K. et al., <u>The Thermodynamic Properties of Manganese</u> (Bureau of Mines Tech. Paper 686, 1946), p. 34.
- 14. White, W. P. J. Phys. Chem., <u>34</u>: 1121 (1930).
- 15. Nernst, W., Ann. Physik (4) 36: 395 (1911).
- 16. Eucken, A. E., Physik. A. 10: 586 (1909).
- 17. Simon and Lange, quoted from Weber, Robert L., <u>Heat and Temperature</u> <u>Measurement</u> (Prentice Hall: New York, 1950), p. 179.

- Sucksmith, W. and Potter, H. N., Proc. Roy. Soc. (London), <u>112</u>: 157 (1926).
- 19. Bunsen, R. W., Phil. Mag. (4) 41: 161 (1871)
- 20. Weld, LeRoy D., <u>A Textbook of Heat</u>, (The Macmillan Co.: New York, 1948), p. 14.
- 21. Ginnings, D. C. and Corrucini, R. J., J. Res. Bur. Stand., <u>38</u>: 583, 593 (1947).
- 22. Ginnings, D. C. et al., J. Res. Bur. Stand., 45: 23 (1950).
- Worthig, Archie and Halliday, David, <u>Heat</u>, (John Wiley: New York, 1948), p. 144.
- 24. Fife, Wynn B., "A Study of the Factors Affecting Operation of a Radiation Calorimeter" (unpub. Ph. D. dissertation, Oklahoma A. & M. College, 1953).
- 25. White, Walter P., J. Am. Chem. Soc., Pt. 2, 40: 1887 (1918).
- 26. White, Walter P., J. Am. Chem. Soc., Pt. 2, 40: 1872 (1918).
- 27. Smith, Cyril S., Trans. A.I.M.E., <u>137</u>: 236 (1940).
- 28. Edmunds, G., Trans. A.I.M.E., <u>137</u>: 244 (1940).
- 29. <u>Armstrong's Industrial Insulations</u>, (Armstrong Cork Co.: Lancaster, Pa., 1951).
- 30. Sweeney, R. J. <u>Measurement Techniques in Mechanical Engineering</u>, (John Wiley: New York, 1953), p. 153.
- 31. Holzbock, Werner G., <u>Instruments for Measurement and Control</u>, (Reinhold Pub.: 1955), p. 33.
- 32. Uhlig, Hervert H., Corrosion Handbook, (John Wiley: 1948), p. 679.
- Driver Harris Catalogue No. R-54, <u>Design of Small Furnaces</u>, (Driver Harris Co.: Harrison, N. J., 1954).
- 34. Strong, John, <u>Procedures in Experimental Physics</u>, (Prentice Hall: New York, 1938).
- 35. <u>Sauereisen Engineer's Production Manual</u>, (Sauereisen Cements Co.: Pittsburgh, Pa., 1951).

- Ginnings, D. C. and Furukawa, George T., J. Am. Chem. Soc., <u>75</u>: 524 (1953).
- 37. Ginnings, D. C. et al., J. Res. Bur. Stand., 45: 23 (1950).
- 38. Finlay, Walter L., Rem-Cru Titanium, Inc., "Titanium Base Materials for Specific Heat Investigation," (private communication by letter dated Jan. 13, 1956).
- 39. Metals Progress, 66 (No. 1-A): 87 (July, 1954).
- 40. Squire, C. F. and Kaufmann, A. R., J. Chem. Phys., 9: 673 (1941).
- 41. Coughlin, J. P. and King, E. G., J. Am. Chem. Soc., 72: 2262 (1950).
- 42. Arkin, Herbert and Colton, Raymond R., <u>Statistical Methods</u>, (Barnes and Noble, 1953), p. 46.
- 43. Kubaschewski, O. and Hopkins, B. E., <u>Oxidation of Metals and</u> <u>Alloys</u>, (Butterworth Scientific Pub.: London, 1953).
- 44. Perkins, R. A., Metals Research Laboratories, Niagara Falls, New York, (private communication by letter dated March 14, 1956).

APPENDIX

THERMOCOUPLE CALIBRATION

APPENDIX A





Figure 31. Millivolt Correction Curve for Thermocouple

TABLE II

CHROMEL-ALUMEL THERMOCOUPLE CALIBRATION DATA

Potentiometer Reading (Millivolts	Thermocouple Correction (Millivolts)	Corrected Potentiometer Reading (Millivolts)	Corresponding Temperature OF
1.5	0.0010	1.5010	99.21
2.0	0.0015	2.0015	121.46
2.5	0.0025	2.5025	143.32
3.0	0.0030	3.0030	165.00
3.5	0.0040	3.5040	186.09
4.0	0.0047	4.0047	208.12
4.5	0.0052	4.5052	229.71
5.0	0.0060	5.0060	251.46
5.5	0.0070	5.5070	273.77
6.0	0.0078	6.0078	296.27
6.5	0.0082	6.5082	319.00
7.0	0.0090	7.0090	341.32
7.5	0.0100	7.5100	364.09
8.0	0.0105	8.0105	385.95
8.5	0.0112	8.5112	409.14
9.0	0.0120	9.0120	431.46
9.5	0.0130	9.5130	453.45
10.0	0.0138	10.0138	476.00
10.5	0.0145	10.5145	498.17

Potentiometer Reading (Millivolts)	Thermocouple Correction (Millivolts)	Corrected Potentiometer Reading (Millivolts)	Corresponding Temperature ^O F
11.0	0.0160	11.0160	519.83
11.5	0.0180	11.5180	542.18
12.0	0.0200	12.0200	564.09
12.5	0.0220	12.5220	585.93
13.0	0.0240	13.0240	607.67
13.2	0.0250	13.2250	616.46
13.4	0.0260	13.4260	624.83
13.6	0.0265	13.6265	633.62
13.8	0.0270	13.8270	642.14
14.0	0.0280	14.0280	650.82
14.2	0.0285	14.2285	659.54
14.4	0.0295	14.4295	667.92
14.6	0.0300	14.6300	676.36
14.8	0.0310	14.8310	685.05
15.0	0.0315	15.0315	693.83
15.2	0.0325	15.2325	702.41
15.4	0.0330	15.4330	711.05
15.6	0.0340	15.6340	719.75
15.8	0.0350	15.8350	728.12
16.0	0.0355	16.0355	736.64
16.2	0.0365	16.2365	745.30
16.4	0.0370	16.4370	753.63

TABLE II (Continued)

Potentiometer Reading (Millivolts)	Thermocouple Correction (Millivolts)	Corrected Potentiometer Reading (Millivolts)	Corresponding Temperature F
16.5	0.0375	16.5375	758.09
17.0	0.0395	17.0395	779.59
17.5	0.0415	17.5415	801.00
18.0	0.0435	18.0435	821.55
18.5	0.0450	18.5450	843.54
19.0	0.0470	19.0470	864.87
19.5	0.0490	19.5490	886.21
20.0	0.0510	20.0510	907.12
20.5	0.0530	20.5530	928.46
21.0	0.0550	21.0550	949.37
21.5	0.0570	21.5570	970.29
22.0	0.0590	22.0590	992.04
22.5	0.0605	22.5605	1013.23
23.0	0.0625	23.0625	1034.29
23.5	0.0645	23.5645	1055.50
24.0	0.0665	24.0665	1076.96
24.5	0.0685	24.5685	1097.88
25.0	0.0700	25.0700	1119.17
25.5	0.0720	25.5720	1139.92
26.0	0.0740	26.0740	1161.42
26.5	0.0760	26.5760	1182.75
27.0	0.0780	27.0780	1204.08

TABLE II (Continued)

Potentiometer Reading (Millivolts)	Thermocouple Correction (Millivolts)	Corrected Potentiometer Reading (Millivolts)	Corresponding Temperature O _F
27.5	0.0800	27.5800	1225.45
28.0	0.0820	28.0820	1246.91
28.5	0.0840	28.5840	1268.46
29.0	0.0855	29.0855	1289.82
29.5	0.0875	29.5875	1131.17
30.0	0.0890	30.0890	1332.87
30.5	0.0915	30.5915	1354.25
31.0	0.0930	31.0930	1375.49
31.5	0.0950	31.5950	1397.50
32.0	0.0970	32.0970	1419.36
32.2	0.0980	32.2980	1428.09
32.4	0.0985	32.4985	1436.77
32.6	0.0995	32.6995	1445.46
32.8	0.1000	32.9000	1454.17
33.0	0.1010	33.1010	1462.96
33.2	0.1020	33.3570	1470.03
33.4	0.1025	33.5025	1480.59
33.6	0.1030	33.7030	1489.68
33.8	0.1040	33,9040	1498.36
34.0	0.1050	34.1050	1507.04
34.2	0.1055	34.3055	1515.73

TABLE	II	(Continued)	

	TABLE II (CONCI	inded)		
Potentiometer Reading (Millivolts)	Thermocouple Correction (Millivolts)	Corrected Potentiometer Reading (Millivolts)	Corresponding Temperature °F	
34.4	0.1062	34.5062	1524.82	
34.6	0.1070	34.7070	1533.63	
34.8	0.1080	34.9080	1542.42	
35.0	0.1085	35.1085	1551.32	
35.2	0.1095	35.3095	1560.42	
35.4	0.1100	35.5100	1569.09	
35.6	0.1110	35.7110	1578.23	
35.8	0.1120	35.9120	1586.91	
36.0	0.1125	36.1125	1596.05	
36.2	0.1130	36.3130	1605.14	
36.4	0.1140	36.5140	1613.92	
36.6	0.1150	36.7150	1622.96	
36.8	0.1155	36.9155	1631.87	
37.0	0.1165	37.1165	1640.72	
37.2	0.1170	37.3170	1649.87	
37.4	0.1180	37.5180	1659.00	
37.6	0.1185	37.7185	1668.14	
37.8	0.1190	37.9190	1676.77	
38.0	0.1200	38.1200	1685.91	
38.5	0.1220	38.6220	1708.73	
39.0	0.1240	39.1240	1731.55	

		And the second se
TABLE	II	(Continued)

Potentiometer Reading (Millivolts)	Thermocouple Correction (Millivolts)	Corrected Potentiometer Reading (Millivolts)	Corresponding Temperature F	
39.5	0.1260	39.6260	1754.35	
40.0	0.1280	40.1280	1777.63	
40.5	0.1300	40.6300	1800.45	
42.0	0.1320	41.1320	1823.73	

APPENDIX B

TIME-TEMPERATURE SLOPE ANALYSIS

FOR TEST SERIES A

TABLE III

TIME-TEMPERATURE SLOPE ANALYSIS FOR SAPPHIRE (A1203)

Test Run #53 Weight of Sample = 43.255 grams Pressure of Chamber = 1 psig of Argon

Potentiometer	Δt	ΔT	Average Temp. of Sample	$\begin{array}{c} \text{Slope} \\ \Delta \text{t} \end{array}$
(Millivolts)	(minutes)	(°F)	(°F)	T
1.5		1.1.1.1		1.2.2
2.0	7.04	22.25	111	0.316
2.0	6.82	21.86	132	0.312
2.5	6.95	21.68	154	0.320
3.0				0.000
3.5	7.08	21.09	176	0.336
	7.07	22.03	197	0.320
4.0	6.97	21.59	219	0.323
4.5	(00	01.00	0/7	0 210
5.0	6.92	21.75	241	0.518
F F	7.22	22.31	263	0.324
2.2	7.35	22.50	285	0.327
6.0	7 25	22 73	308	0.319
6.5	1.27	~~~15	500	
7.0	7.25	22.32	330	0.325
7.0	7.20	22.77	353	0.316
7.5	7 22	21.86	375	0.330
8.0	/ • ~~	~1.00	515	
8 5	7.10	23.19	398	0.307
5.9	7.27	22.32	420	0.326
9.0	7.72	22.00	442	0.351
9.5				
10.0	7.40	22.54	465	0.328
10.0	7.33	22.17	487	0.331

TABLE III (Continued)

Potentiometer Reading	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	ΔT
10.5				
11.0	7.38	21.66	509	0.340
11.5	7.30	22.35	531	0.327
12.0	7.35	21.91	553	0.336
12.0	7.13	21.84	575	0.326
12.5	7.20	21.74	597	0.331
13.0	7.62	21.33	618	0 357
13.5	6.00	21.00	640	0.07
14.0	0.90	21.07	640	0.316
14.5	6.97	21.21	661	0.328
15.0	7.17	21.76	683	0.330
15 5	7.07	21.74	705	0.325
19.9	7.07	21.06	726	0.335
10.0	6.95	21.45	747	0.324
16.5	7.02	21.50	769	0.326
17.0	6.98	21.11	790	0.326
17.5	6.40	~1.41	7,50 (7,7)	0.200
18.0	ره.ه	21.00	118	0.508
18.5	6.47	21.99	833	0.295
19.0	6.33	21.33	854	0.297
10.5	6.57	21.34	876	0.308
19.5	6.42	20.91	897	0.307
20.0	6.22	21.34	918	0.292
20.5	6.33	20.91	939	0.303

TABLE III (Continued)

Potentiometer	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(^o F)	of Sample (°F)	$\frac{\Delta t}{\Delta T}$
21.0				
21.5	6.22	20.92	960	0.297
22.0	6.02	21.75	981	0.277
22.0	5.78	21.19	1003	0.273
22.5	5.72	21.06	1024	0.271
23.0	F 1 F			
23.5	5.45	21.11	1045	0.258
24.0	5.48	21.46	1066	0.255
24.0	5.53	20.92	1087	0.264
24.5	5 28	21 29	1109	0.2/8
25.0	2.20	~~~/	110)	0.240
25.5	5.12	20.75	1130	0.247
26.0	5.08	21.50	1151	0.236
20.0	5.15	21.33	1172	0.241
26.5	/ 07	27 22	1102	0 222
27.0	4.71	21.))	1195	0.233
27 5	5.03	21.37	1215	0.236
~1.5	4.85	21.46	1236	0.226
28.0	4.90	21.55	1258	0.227
28.5	4.75	07.04	2.070	0.001
29.0	4.72	2L. 30	1279	0.221
20.0	4.73	21.70	1322	0.218
50.0	4.53	21.38	1344	0.212
30.5	1.55	21 2/	1365	0 214
31.0	4.))	K-1 0 K44	1,0,	C. CIA
31.5	4.73	22.01	1387	0.214
2=07	4.47	21.86	1408	0.204

TABLE III (Continued)

Potentiometer	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	$\frac{\Delta t}{\Delta T}$
32.0				
32.5	4.58	21.85	1430	0,210
33 0	4.32	21.75	1452	0.199
	4.43	22.18	1474	0.200
33.5	4.37	21,90	1496	0.200
34.0	1 25	00.10	1510	0.100
34.5	4.2)	22.42	1918	0.189
35.0	4.28	21.86	1540	0.196
35 5	4.28	22.72	1563	0.189
55.5	4.25	22.01	1585	0.193
36.0	4.38	22.09	1607	0.198
36.5	/ 17	22 58	1629	0 185
37.0	4.1/	22.00	1029	0.18)
37.5	4.10	22.83	1652	0,179
38 0	4.05	22.36	1675	0.181
20.0	4.15	22.82	1697	0.182
38.5	3.95	22.82	1720	0.173
39.0	4.03	22.80	1743	0.177
39.5	3.02	22.20	2745	0.160
40.0	2.92	23.30	1,00	0.100
40.5	4.10	22.82	1789	0.179
(1.0	3.83	23.28	1812	0.164
4.2.0				

TABLE IV

TIME-TEMPERATURE SLOPE ANALYSIS FOR THE EMPTY CONTAINER

Test Run #52 Pressure of Chamber = 1 psig of Argon

Potentiometer	$\triangle t$	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	$\overline{\Delta T}$
1.5				
2.0	2.37	22.25	111	0.106
	2.12	21.86	132	0.096
2.5	2.04	21.68	154	0.094
3.0	2.34	21.09	176	0.111
3.5	2.20	22.03	107	0.100
4.0	2.20	22.00	197	0.100
1.5	2.30	21.59	219	0.106
4.2	2.37	21.75	241	0.109
5.0	2.33	22.31	263	0.104
5.5	2.43	22.50	285	0.108
6.0	0.21	22 43	304	0 103
6.5	2.94	22.15	308	0.105
P 0	2.28	22.32	330	0.102
7.0	2.32	22.77	353	0.102
7.5	2.25	21.86	375	0.103
8.0	2 35	23 19	398	0.102
8.5	2.000	~>•=>	570	0.201
9.0	2.33	22.32	420	0.104
2.0	2.33	22.00	442	0.106
9.5	2.30	22.54	4.65	0.102

TABLE IV (Continued)

Potentiometer	Δ_t	ΔT	Average Temp. of Sample	Slope At
(Millivolts)	(minutes)	(°F)	(°F)	$\overline{\Delta T}$
10.0				
10.5	2.35	22.17	487	0,106
11.0	2.42	21.66	509	0.112
11.5	2.32	22.35	531	0.104
12.0	2.30	21.91	553	0.105
12.0	2.30	21.84	575	0.105
12.5	2.42	21.74	597	0.111
13.0	2.23	21.33	618	0.105
13.5	2.37	21.87	640	0 108
14.0	2.12	21 21	640	0.11/
14.5	K.4K	21.21	100	0.114
15.0	2.57	21.76	683	0.118
15.5	2.43	21.74	705	0.112
16.0	2.37	21.06	726	0,113
16.5	2.33	21.45	747	0.109
10.9	2.40	21.50	769	0.112
17.0	2.32	21.42	790	0.108
17.5	2.37	21.55	811	0.110
18.0	2.37	21.99	833	0.108
18.5	2 30	21, 33	85/	801.0
19.0	2.00	21.2/	974	0.104
19.5	2.22	×1.54	070	0.104
20.0	2.37	20.91	897	0.113
	2.27	21.34	918	0.106

TABLE IV (Continued)

Potentiometer	Δt	Δ_{T}	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	$\frac{\Delta t}{\Delta T}$
20.5				
21.0	2.23	20.91	939	0.107
21.5	2.18	20.92	960	0.104
22.0	2.20	21.75	981	0.101
22.0	2.12	21.19	1003	0.100
22.5	2.17	21.06	1024	0.103
23.0	2.05	21.11	1025	0.097
23.5	2 03	27 /6	1066	0.005
24.0	2.05	21.40	1080	0.095
24.5	2.02	20.92	1087	0.097
25.0	1.83	21.29	1109	0.086
25.5	1.87	20.75	1130	0.090
25:0	1.83	21.50	1151	0.085
20.0	1.78	21.33	1172	0.084
26.5	1.82	21.33	1193	0.085
27.0	1.70	21.37	1215	0.080
27.5	1 67	21 .6	1236	0.078
28.0	1.07	07.55	1250	0,070
28.5	1.70	21.00	1238	0.079
29.0	1.62	21.36	1279	0.076
29.5	1.65	21.35	1300	0.077
20.0	1.63	21.70	1322	0.075
50.0	1.60	21.38	1344	0.075
30.5	1.55	21.24	1365	0.073

TABLE IV (Continued)

Potentiometer Reading	$\triangle t$	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	or Sample (°F)	$\frac{\Delta t}{\Delta T}$
31.0				
27 6	1.57	22.01	1387	0.071
51.5	1.53	21.86	1408	0.070
32.0	1.50	21.85	1/30	0.069
32.5	1 50	27.05		0.000
33.0	1.57	21.75	1452	0.072
33.5	1.42	22.18	1474	0.064
34.0	1.50	21.90	1496	0.069
34.0	1.45	22.42	1518	0.065
34.5	1.45	21.86	1540	0.066
35.0	1 / 2	20 60		0.0(0
35.5	1.45	22012	1203	0.063
36.0	1.45	22.01	1585	0.066
26 5	1.38	22.09	1607	0.063
J0.J	1.42	22.58	1629	0.063
37.0	1,37	22,83	1652	0,060
37.5	1.22	00.04	16775	0.060
38.0	2.00	22.30	1075	0.000
38.5	1.38	22.82	1697	0.060
30.0	1.32	22.82	1720	0.058
59.0	1.33	22.80	1743	0.058
39.5	1.47	23.30	1766	0.063
40.0	1 22	22 82	1780	0.058
40.5	رر ۲۰	22.02	1/07	0.000
41.0	1.37	23.28	1812	0.059

TABLE V

TIME-TEMPERATURE SLOPE ANALYSIS FOR COPPER

Test Run #48 Weight of Sample = 96.32 grams Pressure in Chambers = 1 psig of Argon

Potentiometer	Δt	ΔT	Average Temp. of Sample	Slope At
(Millivolts)	(minutes)	(°F)	(°F)	$\overline{\Delta T}$
1.6				
2.0	5.90	17.72	112	0.333
2.5	6.95	21.86	132	0.318
2.7	8.45	25.97	154	0.326
3.10	6.43	19.04	176	0.336
3.5	5.02	10.60	107	0 301
4.0	5.95	17.07	197	U.JUI
4.5	6.80	21.59	219	0.316
5.0	7.08	21.75	241	0.326
2.0	6.60	22.31	263	0.296
5.5	6.82	22.50	285	0.303
6.0	6.02	22 72	308	0 305
6.5	0.95	22.1)	500	0.909
7.0	6.67	22.32	330	0.299
FI 5	6.80	22.77	353	0.299
7.5	6.92	21.86	375	0.317
8.0	6.77	23.19	398	0.293
8.5	6 57	22 32	120	0.29/
9.0	0.97	KR. JR	420	0.274
9.5	6.83	22.00	442	0.310
	6.85	22.54	465	0.304

TABLE V (Continued)

Potentiometer	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	of Sample (°F)	Δt
10.0				
10.5	6.62	22.17	487	0.299
11.0	6.78	21.66	509	0.313
12.0	6.68	22.35	531	0.299
11.9	8.23	27.15	553	0.303
12.125	5.28	16.31	575	0.324
12.5	6.12	21.7/	597	0.296
13.0	6 80	21 22	470	0.210
13.5	6.80	~1.))	619	0.319
14.0	6.53	21.87	640	0.298
14.5	6.43	21.21	661	0.303
15.0	6.32	21.76	683	0.291
15.0	6.06	21.74	705	0.279
15.5	6.35	21.06	726	0.301
16.0	6.20	21.45	747	0.289
16.5	6.20	21 50	769	0.289
17.0	6.00	21 /1	700	0.200
17.5	8.00	~1.41	790	0.200
18.0	6.00	21.55	811	0.279
18.5	6.00	21.99	833	0.273
19.0	6.07	21.33	854	0.284
19.0	5.97	21.34	876	0.280
19.5	5.68	20.91	897	0.272
20.0	5.34	21.34	918	0.250

TABLE	V	(Continued)

Potentiometer	Δt	$\Delta \mathbf{T}$	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	ΔT
20.5				
21 0	5.57	20.91	939	0.267
	5.47	20.92	960	0.262
21.5	5.40	21.75	981	0.249
22.0	5.12	21.19	1003	0.242
22.5	5 08	27.06	102/	0.0/1
23.0	5.08	21.00	1024	0.241
23.5	5.02	21.11	1045	0.237
24.0	5.05	21.46	1066	0.235
24.0	4.58	20.92	1087	0.219
24.5	4.68	21.29	1109	0.220
25.0	4.78	20.75	1130	0.230
25.5	/ 50	21 50	1151	0 200
26.0	4.50	21.90		0.207
26.5	4.67	21.33	1172	0.219
27.0	4.48	21.33	1193	0.210
27.0	4.35	21.37	1215	0.204
21.5	4.28	21.46	1236	0.199
28.0	4.40	21.55	1258	0.204
28.5	5 18	26 59	1 270	0 195
29.125	9.10	20.97	12/)	0.175
29.5	3.10	15.77	1300	0.197
30.0	4.17	21.70	1322	0.192
30 5	4.03	21.38	1344	0.189
J U. J	4.17	21.24	1365	0.196

TABLE	V	(Continued)

Carl Street .	TADLE	(Continu	lea)	
Potentiometer Reading	Δt	ΔT	Average Temp. of Sample	Slope At
(Millivolts)	(minutes)	(°F)	(°F)	$\overline{\Delta T}$
31.0				
31.5	4.07	22.01	1387	0.185
32 0	3.82	21.86	1408	0.175
52.0	3.97	21.85	1430	0.183
32.5	4.02	21.75	1452	0.185
33.0	/ 18	22.18	1/7/	0 199
33.5	4.10	22.10	14/4	0.100
34.0	3.73	21.90	1496	0.170
31. 5	3.43	22.42	1518	0.153
54.5	3.63	21.86	1540	0.166
35.0	3.75	22.72	1563	0.165
35.5	3.60	22.01	1585	0.163
36.0	2 (0	22.02	1/07	0.1((
36.5	3.08	22.09	1607	0.166
37.0	3.70	22.58	1629	0.164
57.0	3.65	22.83	1652	0.160
51.5				
TABLE VI

TIME-TEMPERATURE SLOPE ANALYSIS FOR REM-CRU TI A55

Test Run #49 Weight of Sample = 49.040 grams Pressure in Chamber = 1 psig of Argon

Potentiometer Reading	Δt	ΔT	Average Temp. of Sample	Slope At
(Millivolts)	(minutes)	(°F)	$(^{\circ}F)$	$\overline{\Delta^{\mathrm{T}}}$
1.5	(0.000
2.0	6.17	22.25	111	0.211
2.0	5.50	21.86	132	0.251
2.5	5.25	21.68	154	0.242
3.0			->+	0.050
2 5	5.88	21.09	176	0.279
5.5	5.53	22.03	197	0.251
4.0	5 13	27 50	219	0 252
4.5	5.45	£1.)7	~~)	0.2.72
4.5	5.23	21.75	241	0.241
5.0	5.47	22.31	263	0.245
5.5	r 20	22 50	205	0 237
6.0	2.22	22.30	20)	0.201
0.0	5.50	22.73	308	0.242
6.5	5 37	22 32	330	0.240
7.0	1.51	~~	<u> </u>	
	5.47	22.77	353	0.241
7.5	5.38	21.86	375	0.246
8.0				
	5.45	23.19	398	0.235
8.5	5.42	22.32	420	0.243
9.0				0.015
0.5	5.40	22.00	442	0.245
9.5	5.63	22.54	465	0.250

TABLE	VI	(Continued)	

and the state	TABLE V	1 (Contin	nued)	
Potentiometer Readings	Δt	$\triangle T$	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	$\Delta \overline{T}$
10.0	E 10	77 00	105	
10.5	2.40	22.17	487	0.247
11.0	5.53	21.66	509	0.256
11.5	5.37	22.35	531	0.240
12.0	5.57	21.91	553	0.254
12.5	5.38	21.84	575	0.246
13.0	5.35	21.74	597	0.246
13.5	5.50	21.33	618	0.258
11.0	5.48	21.87	640	0.251
14.0	5.38	21.21	661	0.254
14.5	5.47	21.76	863	0.252
15.0	5.30	21.74	705	0.244
15.5	5.43	21.06	726	0.258
16.0	5.42	21.45	747	0.252
16.5	5.37	21.50	769	0.250
17.0	5.35	21.41	790	0.249
17.5	5 17	21.55	811	0.240
18.0	5 35	21 00	833	0.2/3
18.5	5.00	21.22	85/	0.025
19.0	5.02	رو. ۲۷	074	0.000
19.5	4.98	21.34	870	0.234
20.0	4.78	20.91	897	0.229
20 E 334	4.82	21.34	918	0.226

Potentiometer	Δt	Δt	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	Δt ΔT
20.5				
21.0	4.82	20.91	939	0.230
21 5	4.80	20.92	960	0.229
21.9	4.63	21.75	981	0.213
22.0	4.47	21.19	1003	0.211
22.5	4.50	21.06	1024	0.21/
23.0	4.50	~1.00	2015	0.010
23.5	4.50	21.11	1045	0.213
24.0	4.15	21.46	1066	0.193
~4.0	4.27	20.92	1087	0.204
24.5	4.13	21.29	1109	0.194
25.0	4 07	20 75	1130	0.180
25.5	4.07	20.15		0.100
26.0	3.87	21.50	1151	0,187
26 5	3.97	21.33	1172	0.186
20.7	3.92	21.33	1193	0.185
27.0	3.85	21.37	1215	0.181
27.5	3 73	21 /6	1236	0 17/
28.0	5.75	21.40	1250	0.174
28.5	3.75	21.55	1258	0.174
20 0	3.63	21.36	1279	0.170
27.0	3.72	21.35	1300	0.175
29.5	4.40	26.20	1325	0.168
30.125	3 16	21 30	13//	0.163
30.5	5.40	×-1. JU	±244	0.10)
	3.37	21.24	1365	0.158

Potentiometer	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	Δ^{T}
31.0	2 20	22.01	1200	0.163
31.5	2.31	22.01	1967	0.155
32.0	3.30	21.86	1408	0.151
20.5	3.316	21.85	1430	0.151
32.5	3.20	21.75	1452	0.147
33.0	3.28	22.18	1474	0.148
33.5	3 20	21 00	1/96	0.1/6
34.0	5.20	21.90	1490	0.140
34.5	3.58	22.42	1518	0.159
35.0	3.68	21.86	1540	0.168
25.5	4.55	22.72	1563	0.200
35.5	5.52	22.01	1585	0.251
36.0	6.15	22.09	1607	0.279
36.5	5 23	22 58	1629	0 232
37.0	5.25	22.90	1027	0.2/0
37.5	3.85	22.83	1652	0.168
38,125	3.40	27.86	1675	0.122
90.IL)	1.83	16.98	1699	0.108
38.5	2.30	22.82	1720	0.100
39.0	2.20	22.80	1743	0.096
39.5	2 1 5	22.20	1766	0 002
40.0	2.1)	~J. ~O	1,00	0.072
40.5	3.00	32.90	1782	0.091

TABLE VII

TIME-TEMPERATURE SLOPE ANALYSIS FOR REM-CRU TI C-110M

Test Run #51 Pressure in Chamber = 1 psig of Argon

Potentiometer Reading	∆t	ΔT	Average Temp. of Sample	Slope At
(Millivolts)	(minutes)	(°F)	(°F)	$\overline{\Delta T}$
1.5		1000		
2.0	5.94	22.25	111	0.267
	5.82	21.86	132	0.266
2.5	5.37	21.68	154	0.248
3.0	1 97	21 09	176	0.236
3.5	4.77	21.07	1/0	0.250
4.0	5.87	22.03	197	0.200
410	5.83	21.59	219	0.270
4.5	5.77	21.75	241	0.265
5.0	5.50	22.31	263	0.246
5.5	6.00	22 50	285	0 266
6.0	0.00	22. 90	20)	0.200
6 5	5.67	22.73	308	0.250
0.9	5.67	22.32	330	0.254
7.0	5.48	22.77	353	0.241
7.5	5.40	07.00	2015	0.212
8.0	5.40	21.86	375	0.241
D F	5.78	23.19	398	0.250
8.2	5.55	22.32	420	0.249
9.0	5.62	22.00	442	0.256
9.5	5.00	00.51	145	0 220
	5.38	22.54	405	0.239

TABLE	VII	(Continued)

Potentiometer	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	or Sample (°F)	$\frac{\Delta t}{\Delta T}$
30.5	1.50			
31.0	4.53	21.24	1365	0.213
31.5	4.27	22.01	1387	0.194
32 0	4.17	21.86	1408	0.191
22.0	1.58	8.73	1424	0.181
32.2	1.67	8.68	1432	0.193
32.4	1.57	8.69	1441	0.181
32.6	1.65	8.71	1450	0.189
32.8	1.55	8 79	1729	0 176
33.0	1.00	0.17	1457	0.173
33.3	1.22	7.07	1409	0.171
33.4	1.68	10.56	1477	0.159
33.6	1.40	9.09	1483	0.154
33.8	1.33	8.78	1492	0.152
ه،رر	1.33	8.68	1503	0.153
34.0	1.28	8.69	1511	0.148
34.2	1.25	9.09	1521	0.138
34.4	1.21	8,81	1527	0.137
34.6		8 70	1536	0.13/
34.8	1.10	0.79		0.194
35.0	1.23	8.90	1546	0.138
35.2	1.13	9.10	1556	0.124
35 /	1.15	8.67	1565	0.133
22.4	1.15	9.14	1574	0.126

Potentiometer	$\triangle t$	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	
20.0	1.00	01 04	010	
20.5	4.80	21.34	918	0.225
21.0	4.60	20.91	939	0.220
21.5	4.58	20.92	960	0.219
22.0	4.52	21.75	981	0.208
22.0	4.60	21.19	1003	0.218
22.5	5.03	21.06	1024	0.239
23.0	4.22	21.11	1045	0.200
23.4	4.45	21.46	1066	0.207
24.0	1 55	20 92	1087	0.218
24.5	4.77	20.72	1007	0.210
25.0	4.13	21.29	1109	0.222
25.5	4.95	20.75	1130	0.239
26.0	4.23	21.50	1151	0.197
26.5	4.43	21.33	1172	0.208
20.9	4.47	21.33	1193	0.209
27.0	4.47	21.37	1215	0.209
27.5	4.70	21.46	1236	0.219
28.0	4.67	21.55	1258	0.217
28.5	4.50	21 36	1270	0 211
29.0	4.00	21.00	12/7	0.010
29.5	4.41	21.35	1300	0.210
30.0	4.45	21.70	1322	0.205
	4.47	21.38	1344	0.210

	Potentiometer	$ riangle \mathbf{t}$	$\triangle T$	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	of Sample (°F)	$\frac{\Delta t}{\Delta T}$	
	10.0				
	10.5	5.33	22.17	487	0.241
	11.0	5.02	21.66	509	0.231
	11.6	5.57	22.35	531	0.250
	11.9	5.15	21.91	553	0.235
	12.0	5.47	21.84	574	0.250
	12.5	5.72	21.74	597	0.263
	13.0	5.12	21,33	618	0.25/
	13.5	5 20	01 dr	610	0.215
	14.0	2.27	21.0/	640	0.245
	14.5	5.48	21.21	661	0.258
	15.0	5.38	21.76	683	0.248
	15.5	5.38	21.74	705	0.248
	16.0	5.37	21.06	726	0.255
	10.0	5.83	21.45	747	0.272
	16.5	4.95	21.50	769	0.230
	17.0	5.45	21.41	790	0.254
	17.5	5.25	21.55	811	0.211
	18.0	5 26	27.00	625	0.2//
	18.5	5.50	21.99	200	0.244
	19.0	5.31	21.33	854	0.249
	19.5	5.05	21.34	876	0.236
		4.97	19.55	886	0.254

Potentiometer	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	$\frac{\Delta t}{\Delta T}$
35.6	7 00	0.10	1580	0.304
35.8	T.08	8.68	1282	0.124
36.0	1.10	9.14	1591	0.120
26. 2	1.10	9.09	1601	0.121
50.2	1.08	8.78	1610	0.123
36.4	1.07	9.04	1618	0.118
36.6	1.00	8.91	1627	0.112
36.8	1.05	0.05	1626	0 110
37.0	1.09	0.0)	1090	0.119
37.2	0.97	9.15	1645	0.106
37.4	1.03	9.13	1654	0.113
277 6	1.02	9.14	1664	0.112
57.0	0.95	8.63	1672	0.110
37.8	1.03	9.14	1681	0.113
38.0	2.35	22.82	1697	0.103
38.5	0.20	22 82	1720	0 102
39.0	4.74	ZZ.OZ	1720	0.102
39.5	2.22	22.80	1743	0.097
40.0	2.10	23.28	1766	0.090
40.0	2.08	22.82	1789	0.091
40.5	2.00	23.28	1812	0.086
41.0				

TABLE VIII

TIME-TEMPERATURE SLOPE ANALYSIS FOR REM-CRU TI C-130AM

Test Run #50 Pressure of Chamber = 1 psig of Argon

Potentiometer Reading	Δt	ΔT	Average Temp. of Sample	Slope At
(Millivolts)	(minutes)	(°F)	(°F)	ΔT
1.5				
2.0	6.92	22.25	111	0.311
2.5	5.72	21.86	132	0.262
2.7	6.17	21.68	154	0.285
3.0	5.10	21.09	176	0.242
3.5	5 73	22 03	107	0 260
4.0	5.15	22.05	177	0.200
4.5	5.62	21.59	219	0.260
5-0	5.62	21.75	241	0.258
F F	5.57	22.31	263	0.250
2.2	5.65	22.50	285	0.251
6.0	5.52	22.73	308	0.243
6.5	5 58	22 32	330	0 250
7.0		22.52	220	0.200
7.5	5.42	22.11	253	0.248
8.0	5.55	21.86	375	0.254
0.0	5.50	23.19	398	0.248
8.5	5.60	22.32	420	0.251
9.0	5.42	22.00	1.1.2	0.246
9.5	5 10	22 51	165	0.211
	9.40	KK. 94	405	0.244

Potentiometer	$\triangle t$	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	ΔT
10.0	6.10	12 20		
10.5	5.47	22.17	487	0.247
11.0	5.63	21.66	509	0.260
11 5	5.63	22.35	531	0.252
11.9	5.47	21.91	553	0.250
12.0	5.52	21.84	575	0.252
12.5	5.68	21.74	597	0.262
13.0	5.12	21.33	618	0.240
13.5	5.00	01 00	610	0.044
14.0	2.02	21.07	640	0.200
14.5	5.55	21.21	661	0.262
15.0	5.43	21.76	683	0250
15.5	5.47	21.74	705	0.252
1).)	5.52	21.06	726	0.262
16.0	5.52	21.45	747	0.257
16.5	5.47	21.50	769	0.254
17.0	6.60	26.95	790	0.245
17.6	6.05	15 01	811	0.270
18.0	5.00	27.00	d32	0.228
18.5	2.22	21.99	600	0.200
19.0	5.23	21.33	854	0.245
19.5	5.05	21.34	876	0.237
20.0	5.73	20.91	897	0.274
20.0	4.44	21.34	918	0.208

Potentiometer Reading	∆t	ΔT	Average Temp.	Slope	
(Millivolts)	(minutes)	(^o f)	(°F)	$\frac{\Delta t}{\Delta T}$	
20.5					
21.0	4.82	20.91	939	0.230	
21.5	4.88	20.92	960	0.234	
22.0	4.87	21.75	981	0.224	
22.0	4.40	21.19	1003	0.208	
22.5	4.85	21.06	1024	0.230	
23.0	1. 67	21 11	10/5	0.001	
23.5	4.07	~1,11	104)	0.221	
24.0	4.50	21.46	1066	0.210	
24.5	4.50	20.92	1087	0.215	
25.0	4.42	21.29	1109	0.208	
23.0	4.80	20.75	1130	0.232	
25.5	4.02	21.50	1151	0.187	
26.0	4.40	21.33	1172	0.206	
26.5	1 277	21.22	1102	0.205	
27.0	4.51	رو.12	1195	0.205	
27.5	4.25	21.37	1215	0.199	
28-0	4.28	21.46	1236	0.199	
2010	4.25	21.55	1258	0.197	
20.7	4.18	21.36	1279	0.196	
29.0	4.18	21.35	1300	0.196	
29.5	4.05	21.70	1322	0.186	
30.0	4.00	21 20	12//	0 189	
30.5	4.02	~1.30	1044	0.100	
	3.92	21.24	1365	0.185	

Potentiometer	Δt	ΔT	Average Temp.	Slope
(Millivolts)	(minutes)	(°F)	(°F)	$\frac{\Delta t}{\Delta T}$
31.0				
31.5	3.87	22.01	1387	0.176
32.0	3.83	21.86	1408	0.175
52.0	3.83	21.85	1430	0.175
32.5	1.85	10.87	1447	0.170
32.8	1 00	10 00		0.100
33.0	1.92	10.00	1458	0.177
33.2	1.15	6.68	1466	0.172
33.5	2.68	15.50	1477	0.173
,,,,	1.20	6.65	1488	0.181
33.7	1.13	6.57	1495	0.172
33.8	1.58	8 68	1503	0 100
34.0	1.90	0.00	1909	0.102
34.2	1.13	6.50	1510	0.174
34.5	2.63	15.92	1522	0.165
24.2	1.10	6.32	1533	0.174
34.7	1.15	8.64	1540	0.133
34.8	1.55	6.90	1548	0.225
35.0	7 077	10 77	1557	0 17/
35.2	1.07	10.77	1997	0.1/4
36.0	5.63	33.94	1579	0.166
36.3	1.83	11.36	1602	0.161
	1.75	10.73	1613	0.163
36.5	1.03	7.09	1621	0.145
36.7	1.00	6.64	1629	0.151

∆t	ΔT	Average Temp.	Slope
(minutes)	(°F)	(°F)	$\frac{\Delta t}{\Delta T}$
1.50	8.85	1636	0.170
1.65	13.42	1647	0.123
1 67	9 /1	1659	0 177
1.07	7.41	10))	0.177
0.92	7.32	1667	0.125
0.90	5.90	1674	0.153
1 18	9 1/	1681	0 129
1.10	7.14	1001	0.12/
1.18	9.14	1690	0.129
1.63	13.68	1702	0.119
2 63	22 82	1720	0 115
2.05	22.02	1/20	0.11)
2.38	22.80	1743	0.104
2.27	23.30	1766	0.097
2 13	22 82	1780	0 093
2.17	22.02	1/07	0.075
0.85	10.51	1806	0.081
	<pre> ∆t (minutes) 1.50 1.65 1.67 0.92 0.90 1.18 1.18 1.63 2.63 2.38 2.27 2.13 0.85 </pre>	$\begin{array}{c c} \Delta t & \Delta T \\ (minutes) & (^{\circ}F) \\ \hline 1.50 & 8.85 \\ 1.65 & 13.42 \\ 1.65 & 13.42 \\ 1.67 & 9.41 \\ 0.92 & 7.32 \\ 0.90 & 5.90 \\ 1.18 & 9.14 \\ 1.18 & 9.14 \\ 1.18 & 9.14 \\ 1.63 & 13.68 \\ 2.63 & 22.82 \\ 2.38 & 22.80 \\ 2.27 & 23.30 \\ 2.13 & 22.82 \\ 0.85 & 10.51 \\ \end{array}$	$\begin{array}{c c c c c c c c c c c c c c c c c c c $

APPENDIX C

DETERMINATION OF A THEORETICAL EMPTY CURVE

CAL	CULATION O	F A THEOR	ETICAL	EMPTY CU	RVE UT	ILIZING	SAPPHI	RE (Al2	03) AND	REM-CR	U TITAN	IUM A-5	5
	ı	2	3	4	5	6	7	8	9	10	11	12	13
Sample Temp. (°F)	C _{pu} Rem-Cru Ti-A55	W _u Rem-Cru Ti-A55	1 x 2	$\frac{\Delta t_s}{\Delta T_s}$ Al203	3 x 4	C _{ps} Al2 ⁰ 3	^W s ^{Al} 2 ⁰ 3	6 x 7	$\frac{\Delta t_u}{\Delta T_u}$ Ti-A55	8 x 9	3 - 8	5 - 10 - 12	∆t _e ∆Te 2 ÷ 1]
100	0.128	49.04	6.27	0.320	2.01	0.191	43.26	8.27	0.260	2.15	-2,00	-0.14	0.070
200	0.130	1	6.37	0.322	2.05	0.213	1	9.23	0.250	2.31	-2.86	-0.26	0.09]
300	0.133		6.52	0.320	2.09	0.231		10.00	0.240	2.42	-3.48	-0.33	0.09
400	0.136		6.67	0.324	2.16	0.244		10.55	0.243	2.57	-3.88	-0.41	0.106
500	0.139	H	6.81	0.332	2.26	0.254	4	11.00	0.249	2.74	-4.19	-0.48	0.11!
600	0.143	V	7.01	0.333	2.33	0.262	Y	11.31	0.256	2.90	-4.30	-0.57	0.13
700	0.147		7.20	0.327	2.35	0.268		11.60	0.257	2.98	-4.40	-0.63	0.14
800	0.151		7.40	0.316	2.34	0.274		11.82	0.249	2.95	-4.42	-0.61	0.138
900	0.156		7.65	0.300	2.29	0.278		12.04	0.233	2.81	-4.39	-0.52	0.118
1000	0.160		7.84	0.279	2.19	0.282		12.22	0,216	2.64	-4.38	-0.45	0.10:
1100	0.165		8.09	0.251	2.03	0.286		12.39	0.200	2.48	-4.30	-0.45	0.105
1200	0.171		8.38	0.234	1.96	0.288		12.46	0.183	2.28	-4.08	-0.36	0.088
1300	0.176		8.63	0.222	1.92	0.291		12.58	0.169	2.13	-3.95	-0.215	0.055
1400	0.182		8.93	0,207	1.85	0.293		12.70	0.154	1.95	-3.77	-0.102	0.027

TABLE IX

ЦЗ

APPENDIX D

DETERMINATION OF SPECIFIC HEATS FOR TEST SERIES A

TABLE X

DETERMINATION OF SPECIFIC HEATS OF REM-CRU TITANIUM A-55

	A .	В	C A t	D	E	F	G	H W-	
Sample Temperature ^O F	<u>∆t</u> ∆T Ti A-55		$\frac{\Delta t}{\Delta T}$ Actual Empty	A – C	B – C	D E	бр А1 ₂ 03	Wu 43.26 49.04	Ti A-55 FxGxH
100	0.260	0.320	0.109	0.151	0.211	0.716	0.191	0.882	0.121
200	0.250	0.322	0.105	0.145	0.217	0.668	0.213	1	0.126
300	0.242	0.320	0.104	0.138	0.216	0.638	0.231		0.130
400	0.243	0.324	0.106	0.137	0.218	0.629	0.244		0.135
500	0.249	0.332	0.110	0.139	0.222	0.626	0.254		0.140
600	0.256	0.333	0.112	0.144	0.221	0.651	0.262	1	0.150
700	0.257	0.327	0.112	0.145	0.215	0.674	0.268	V	0.159
800	0.249	0.316	0.112	0.137	0.204	0.671	0.274		0.162
900	0.233	0.300	0.108	0.125	0.192	0.651	0.278		0.160
1000	0.216	0.279	0.101	0.115	0.178	0.646	0.282		0.161
1100	0.200	0.251	0.091	0.109	0.160	0.681	0.286		0.172
1200	0.183	0.234	0.082	0.101	0.152	0.664	0.288		0.169
1300	0.169	0.222	0.075	0.094	0.147	0.639	0.291		0.164
1400	0.154	0.207	0.070	0.084	0.137	0.613	0.293		0.159
1500	0.159	0.198	0.065	0.094	0.133	0.706	0.295		0.184
1550	0.210	0.196	0.064	0.146	0.132	1.105	0.296		0.290

TABLE XI

DETERMINATION OF SPECIFIC HEATS OF COPPER UTILIZING A THEORETICAL EMPTY CURVE

Sample Temperature ^O F	A <u> </u>	$ \frac{\Delta t}{\Delta T} Al203 $	$\begin{array}{c} C\\ \underline{\Delta t}\\ \overline{\Delta T}\\ \end{array}$ Theoretical	D A – C	E B - C	F D E	G C _p	$\frac{W_{s}}{W_{u}}$	I C _p Copper
			Empty				A1203	96.32	FxGxH
100	0.334	0.320	0.104	0.230	0.216	1.065	0.191	0.449	0.092
200	0.316	0.322	0.099	0.217	0.223	0.974	0.213	Ē.	0.093
300	0.306	0.320	0.100	0.206	0.220	0.937	0.231		0.097
400	0.302	0.324	0.105	0.197	0.219	0.900	0.244		0.099
500	0.303	0.332	0.116	0.187	0.216	0.865	0.254		0.099
600	0.305	0.333	0.133	0.172	0.200	0.860	0.262	V	0.101
700	0.301	0.327	0.144	0.157	0.183	0.857	0.268	Y	0.103
800	0.289	0.316	0.139	0.150	0.177	0.847	0.2735		0.104
900	0.271	0.300	0.120	0.151	0.180	0.839	0.2782		0.105
1000	0.248	0.279	0.103	0.145	0.176	0.824	0.2824		0.105
1100	0.225	0.251	0.104	0.121	0.147	0.823	0.286		0.106
1200	0.208	0.234	0.082	0.126	0.152	0.829	0.288		0.107
1300	0.194	0.222	0.054	0.140	0.168	0.834	0.291		0.109
1400	0.184	0.207	0.026	0.158	0.181	0.872	0.293		0.115
1500	0.174	0.198	0.001	0.173	0.197	0.878	0.295		0.116
1600	0.165	0.191	-0.02	0.185	0.211	0.876	0.297		0.117

TABLE XII

DETERMINATION OF SPECIFIC HEATS OF REM-CRU TITANIUM C-110M UTILIZING A THEORETICAL EMPTY CURVE

120.20	A	В	C	D	E	F	G	H	I
Sample Temperature F			$\frac{\Delta t}{\Delta T}$ Theoretical Empty	A – C	B – C	D E	^C p Al ₂ 0 ₃	Wu 4 <u>3.26</u> 50.22	С _р ТіС-110М F х G х H
100	0.274	0.320	0.104	0.170	0.216	0.787	0.191	0.861	0.130
200	0.264	0.322	0.099	0.165	0.223	0.740	0.213	1	0.136
300	0.256	0.320	0.100	0.156	0.220	0.710	0.231		0.141
400	0.247	0.324	0.105	0.142	0.219	0.649	0.244		0.136
500	0.246	0.332	0.116	0.130	0.216	0.602	0.254	t	0.132
600	0.253	0.333	0.133	0.120	0.200	0.600	0.262	N N	0.135
700	0.257	0.327	0.144	0.113	0.183	0.617	0.268		0.143
800	0.254	0.316	0.139	0.115	0.177	0.650	0.274		0.153
900	0.240	0.300	0.120	0.120	0.180	0.667	0.278		0.160
1000	0.215	0.279	0.103	0.112	0.176	0.637	0.282		0.155
1100	0.212	0.251	0.104	0.108	0.147	0.735	0.286		0.181
1200	0.213	0.234	0.082	0.131	0.152	0.862	0.288		0.214
1300	0.211	0.222	0.054	0.157	0.168	0.938	0.291		0.234
1400	0.197	0.207	0.026	0.181	0.181	1.000	0.293		0.252
1450	0.180	0.201	0.013	0.193	0.188	1.026	0.294		0.260
1500	0.155	0.198	0.001	0.156	0.199	0.783	0.295		0.199
1550	0.134	0.196	-0.010	0.124	0.186	0.667	0.296		0.170
1650	0.113	0.184	-0.030	0.083	0.154	0.539	0.298		0.138
1750	0.095	0.173	-0.048	0.047	0.125	0.376	0.300		0.097
1800	0.088	0.167	-0.054	0.034	0.113	0.301	0.301		0.078

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TABLE XIII

DETERMINATION OF SPECIFIC HEATS OF REM-CRU TITANIUM C-130AM UTILIZING A THEORETICAL EMPTY CURVE

Semple		A A +	B ∧+	C A t	D	E	F	G	H W_	I C
Temperature ^o F	Ti	<u>∆T</u> C-130AM	Al203	△T Theoretical Empty	A - C	B – C	D E	°p Al ₂ 03	Wu 43.26	Ti C-130AM FxGxH
100		0.266	0 320	0.10/	0 162	0 216	0 750	0 191	47.02	0 125
200		0.256	0.322	0.000	0.157	0.223	0.70	0.213	0.072	0.131
200		0.250	0.322	0.099	0.157	0,220	0.704	0.21)		0.120
300		0.250	0.320	0.100	0.150	0.220	0.002	0.231	- 19 A	0.138
400		0.248	0.324	0.105	0.143	0.219	0.654	0.244		0.139
500		0.252	0.332	0.116	0.136	0.216	0.630	0.254		0.140
600		0.263	0.333	0.133	0.130	0.200	0.650	0.262		0.148
700		0.263	0.327	0.144	0.119	0.183	0.650	0.268	7	0.152
800		0.251	0.316	0.139	0.112	0.177	0.633	0.274	Y	0.151
900		0.238	0.300	0.120	0.118	0.180	0.655	0.278		0.159
1000		0.226	0.279	0.103	0.123	0.176	0.699	0.282		0.172
1100		0.215	0.251	0.104	0.111	0.147	0.755	0.286		0.189
1200		0.202	0.234	0.082	0.120	0.152	0.789	0.288		0.198
1300		0.191	0.222	0.054	0.137	0.168	0.816	0.291		0.207
1400		0.180	0.207	0.026	0.154	0.181	0.850	0.293		0.217
1500		0.171	0.198	0.001	0.170	0.199	0.854	0.295		0.220
1600		0.155	0.191	-0.020	0.175	0.171	1.021	0.297		0.265
1650		0.140	0.184	-0.030	0.170	0.154	1.105	0.298		0.287
1700		0.121	0.179	-0.040	0.161	0.139	1.159	0.299		0.302
1800		0.089	0.167	-0.054	0.143	0.113	1.267	0.301		0.332

TABLE XIV

DETERMINATION OF SPECIFIC HEATS OF TITANIUM METALS CORPORATION TITANIUM 55A

Sample Temperature OF	A △t △T Ti 55A	B At Al ₂ 03	C <u>At</u> AT Theoretical Empty	$\frac{\Delta t_u}{\Delta t_u} - \frac{\Delta t_e}{\Delta t_e}$ $A - C$		F D E	G Cp Al2 ⁰ 3	H W <u>s</u> Wu 43.26 48.84	I C _p =FxGxH
100	0.265	0.346	0.104	0.161	0.242	0.666	0.191	0.887	0.113
200	0.259	0.345	0.099	0.160	0.246	0.650	0.213	1	0.123
300	0.2535	0.345	0.100	0.1535	0.245	0.627	0.231		0.128
400	0.249	0.348	0.105	0.145	0.243	0.597	0.244		0.129
500	0.247	0.353	0.116	0.131	0.237	0.553	0.254		0.125
600	0.246	0.354	0.133	0.113	0.221	0.512	0.262	1	0.120
700	0.246	0.349	0.144	0.102	0.205	0.497	0.268	17	0.118
800	0.243	0.342	0.139	0.104	0.203	0.513	0.274	Y	0.125
900	0.228	0.332	0.120	0.108	0.212	0.510	0.278		0.126
1000	0.211	0.318	0.103	0.108	0.215	0.502	0.282		0.126
1100	0.196	0.300	0.104	0.092	0.196	0.469	0.286		0.119
1200	0.181	0.283	0.082	0.098	0.201	0.487	0.288		0.125
1300	0.169	0.268	0.054	0.115	0.214	0.533	0.291		0.137
1400	0.159	0.254	0.026	0.133	0.228	0.583	0.293		0.152
1500	0.151	0.241	0.001	0.150	0.240	0.625	0.295		0.164

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APPENDIX E

DETERMINATION OF SPECIFIC HEATS FOR TEST SERIES B

TABLE XV

DETERMINATION OF SPECIFIC HEATS OF VANADIUM UTILIZING A THEORETICAL EMPTY CURVE

	A	В	С	D	E	F	G	н	I
Sample Temperature F	$rac{\Delta t}{\Delta T}$ Vanadium	$\frac{\Delta t}{\Delta T}$ Al ₂ 0 ₃	$\frac{\Delta t}{\Delta T}$ Theoretical Empty	A – C	B – C	D C	с _р А1 ₂ 03	Ws Wu <u>43.26</u> 65.9	Ср Vanadium FхGхH
100	0.320	0.346	0.104	0.216	0.242	0.892	0.191	0.657	0.112
200	0.310	0.345	0.099	0.211	0.246	0.857	0.213		0.120
300	0.304	0.345	0.100	0.204	0.245	0.834	0.231		0.126
400	0.301	0.348	0.105	0.196	0.243	0.807	0.244		0.129
500	0.299	0.353	0.116	0.183	0.237	0.773	0.254		0.129
600	0.298	0.354	0.133	0.165	0.221	0.747	0.262		0.129
700	0.295	0.349	0.144	0.151	0.205	0.736	0.268	4	0.130
800	0.289	0.342	0.139	0.150	0.203	0.739	0.274	V	0.133
900	0.280	0.332	0.120	0.160	0.212	0.755	0.278	ż	0.138
1000	0.267	Q.318	0.103	0.164	0.215	0.763	0.282		0.142
1100	0.252	0.300	0.104	0.148	0.196	0.755	0.286		0.142
1200	0.236	0.283	0.082	0.154	0.201	0.766	0.288		0.145
1300	0.224	0.268	0.054	0.170	0.214	0.794	0.291		0.152
1400	0.215	0.254	0.026	0.189	0.228	0.829	0.293		0.160
1500	0.205	0.241	0.001	0.204	0.240	0.849	0.295		0.165
1600	0.192	0.228	-0.020	0.212	0.248	0.855	0.297		0.167
1700	0.177	0.216	-0.040	0.217	0.256	0.848	0.299		0.166
1800	0.160	0.204	-0.054	0.214	0.258	0.830	0.301		0.164

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TABLE XVI

TERMINATION OF SPECIFIC HEATS OF REPUBLIC STEEL UNALLOYED TITANIUM UTILIZING A THEORETICAL EMPTY CURVE

Sample	A ∆t	B ∆t	C ∆t	D	E	F	G C	H Wa	I C
Temperature ^O F	<u>⊼T</u> Ti 55		T Theoretical Empty	A – C	B – C	D E	^{°p} Al ₂ 03	$\frac{8}{W_{u}}$ $\frac{43.26}{47.95}$	ті 55 FxGxH
100	0.259	0.346	0.104	0.155	0.242	0.640	0.191	0.902	0.110
200	0.253	0.345	0.099	0.154	0.246	0.626	0.213		0.120
300	0.251	0.345	0.100	0.151	0.245	0.617	0.231		0.128
400	0.250	0.348	0.105	0.145	0.243	0.597	0.244		0.131
500	0.250	0.353	0.116	0.134	0.237	0.565	0.254		0.130
600	0.250	0.354	0.133	0.117	0.221	0.530	0.262	М	0.125
700	0.250	0.349	0.144	0.106	0.205	0.517	0.268	V	0.125
800	0.249	0.342	0.139	0.110	0.203	0.542	0.274	1	0.134
900	0.245	0.332	0.120	0.125	0.212	0.590	0.278		0.148
1000	0.232	0.318	0.103	0.129	0.215	0.600	0.282		0.153
1100	0.219	0.300	0.104	0.115	0.196	0.587	0.286		0.152
1200	0.202	0.283	0.082	0.120	0.201	0.597	0.288		0.155
1300	0.187	0.268	0.054	0.133	0.214	0.622	0.291		0.163
1400	0.171	0.254	0.026	0.145	0.228	0.636	0.293		0.168
1500	0.170	0.241	0.001	0.169	0.240	0.705	0.295		0.187
1550	0.200	0.234	-0.010	0.210	0.244	0.860	0.296		0.229
1620	0.262	0.226	-0.025	0.287	0.251	1.140	0.298		0.307
1650	0.210	0.222	-0.030	0.240	0.252	0.952	0.298		0.256
1700	0.150	0.216	-0.040	0.190	0.256	0.743	0.299		0.200
1800	0.093	0.204	-0.054	0.147	0.258	0.570	0.301		0.155

TABLE XVII

DETERMINATION OF SPECIFIC HEATS OF IODIDE TITANIUM UTILIZING A THEORETICAL EMPTY CURVE

Sample	A A+	B A+	C A+	D	E	F	G	H W_	I
Temperature ^o F	<u>∆T</u> Iodide Ti		<u>⊡</u> ∆T Theoretical Empty	A – C	B - C	D C	°p A1 ₂ 0 ₃	$\frac{-8}{W_{u}}$ $\frac{43.26}{49.02}$	^o p Iodide Ti FxGxH
100	0.278	0.346	0.104	0.174	0.242	0.719	0.191	0.882	0.121
200	0.268	0.345	0.099	0.169	0.246	0.687	0.213	1	0.129
300	0.262	0.345	0.100	0.162	0.245	0.661	0.231		0.135
400	0.257	0.348	0.105	0.152	0.243	0.625	0.244		0.135
500	0.255	0.353	0.116	0.139	0.237	0.586	0.254	1.0	0.131
600	0.255	0.354	0.133	0.122	0.221	0.552	0.262	Ħ	0.128
700	0.255	0.349	0.144	0.111	0.205	0.542	0.268	V	0.128
800	0.254	0.342	0.139	0.115	0.203	0.567	0.274	1	0.137
900	0.250	0.332	0.120	0.130	0.212	0.613	0.278		0.150
1000	0.238	0.318	0.103	0.135	0.215	0.627	0.282		0.156
1100	0.220	0.300	0.104	0.116	0.196	0.592	0.286		0.149
1200	0.202	0.283	0.082	0.120	0.201	0.597	0.288		0.152
1300	0.183	0.268	0.054	0.129	0.214	0.603	0.291		0.155
1400	0.168	0.254	0.026	0.142	0.228	0.623	0.293		0.161
1500	0.155	0.241	0.001	0.154	0.240	0.642	0.295		0.167
1550	0.155	0.234	-0.010	0.165	0.244	0.676	0.296		0.177
1600	0.661	0.228	-0.020	0.682	0.248	2.750	0.297		0.720
1650	0.120	0.222	-0.031	0.151	0.253	0.597	0.298		0.157
1700	0.111	0.216	-0.040	0.151	0.256	0.590	0.299		0.156
1800	0.092	0.204	-0.054	0.146	0.258	0.566	0.301		0.150

TABLE XVIII

DETERMINATION OF SPECIFIC HEATS OF ZIRCONIUM

	A	В	C	D	E	F	G	H	I
Sample Temperature ^O F	$\frac{\Delta t}{\Delta T}$ Zirconium	$\frac{\Delta t}{\Delta T}$ Al ₂ 0 ₃	$\frac{\Delta t}{\Delta T}$ Theoretical	$\frac{\Delta tu}{\Delta Tu} \frac{\Delta te}{\Delta Te}$ (A)-(C)	$\frac{\Delta ts}{\Delta Ts} \frac{\Delta te}{\Delta Te}$ (B)-(C)	(D) (E)	Cp Al ₂ 03	<u>Ws</u> Wu 43.26	Cp Z irconium (F)(G)(H)
			Employ					10.95	
100	0.240	0.346	0.104	0.136	0.242	0.562	0.191	0.6097	0.066
200	0.230	0.345	0.099	0.131	0.246	0.533	0.213	Ĩ.	0.069
300	0.225	0.345	0.100	0.125	0.245	0.511	0.231		0.072
400	0.227	0.348	0.105	0.122	0.243	0.502	0.244		0.075
500	0.234	0.353	0.116	0.118	0.237	0.497	0.254		0.077
600	0.238	0.354	0.133	0.105	0.221	0.475	0.262	+	0.076
700	0.234	0.349	0.144	0.090	0.205	0.439	0.268	V	0.075
800	0.226	0.342	0.139	0.087	0.203	0.429	0.274		0.072
900	0.217	0.332	0.120	0.097	0.212	0.457	0.278		0.078
1000	0.206	0.318	0.103	0.103	0.215	0.479	0.282		0.082
1100	0.194	0.300	0.104	0.090	0.196	0.459	0.286 .		0.080
1200	0.180	0.283	0.082	0.098	0.201	0.488	0.288		0.086
1300	0.167	0.268	0.054	0.113	0.214	0.538	0.291		0.094
1400	0.155	0.254	0.026	0.129	0.228	0.566	0.293		0.101
1500	0.141	0.241	0.001	0.140	0.240	0.583	0.295		0.105
1580	0.210	0.231	-0.016	0.226	0.247	0.915	0.297		0.166
1610	0.316	0.227	-0.023	0.339	0.250	1.36	0.298		0.246
1660	0.216	0.221	-0.032	0.248	0.253	0.982	0.299		0.179
1700	0.140	0.216	-0.040	0.144	0.220	0.655	0.299		0.119
1800	0.100	0.204	-0.054	0.154	0.258	0.597	0.301		0.110

TABLE XIX

DETERMINATION OF SPECIFIC HEATS OF "COBALT FREE" NICKEL

	A	В	C.	D	E	F	G	Н	I
Sample Temperature F	<u>∆t</u> ∆T Nickel	Δt ΔT Al ₂ 0 ₃	$\frac{\Delta t}{\Delta T}$ Theoretical Empty	A – C	B – C	D E	Cp Al ₂ O ₃	<u>Ws</u> Wu <u>43.26</u> 96.88	C _p Nickel
100	0.380	0.346	0.104	0.276	0.242	1.140	0.191	0.4465	0.097
200	0.372	0.345	0.099	0.273	0.246	1.110	0.213	1	0.106
300	0.367	0.345	0.100	0.267	0.245	1.090	0.231		0.113
400	0.368	0.348	0.105	0.263	0.243	1.080	0.244		0.118
500	0.376	0.353	0.116	0.260	0.237	1.098	0.254		0.125
600	0.388	0.354	0.133	0.255	0.221	1.154	0.262	17	0.135
650	0.395	0.351	0.139	0.256	0.212	1.207	0.265	Y	0.143
680	0.398	0.349	0.142	0.256	0.207	1.237	0.267		0.148
700	0.354	0.349	0.144	0.210	0.205	1.025	0.268		0.123
800	0.342	0.342	0.139	0.203	0.203	1.000	0.274		0.122
900	0.333	0.332	0.120	0.213	0.212	1.005	0.278		0.125
1000	0.320	0.318	0.102	0.217	0.215	1.008	0.282		0.127
1100	0.303	0.300	0.104	0.199	0.196	1.015	0.286		0.130
1200	0.286	0.283	0.082	0.204	0.201	1.014	0.288		0.131
1300	0.274	0.268	0.054	0.220	0.214	1.028	0.291		0.134
1400	0.261	0.254	0.026	0.235	0.228	1.030	0.293		0.135
1500	0.246	0.241	0.001	0.245	0.240	1.021	0.295		0.135
1600	0.230	0.228	-0.020	0.250	0.248	1.008	0.297		0.134
1700	0.214	0.216	-0.040	0.254	0.256	0.993	0.299		0.133
1800	0.198	0.204	-0.054	0.252	0.258	0.977	0.301		0.131

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VITA

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Candidate for the Degree of

Doctor of Philosophy

Thesis: A COMPARATIVE METHOD OF SPECIFIC HEAT DETERMINATION

Major Field: Engineering

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- Born: The writer was born on a ranch near Marble Falls, Texas, February 3, 1922, the son of George C. and Tessilea P. Beakley.
- Undergraduate Study: He graduated from the Levelland High School, Levelland, Texas in May, 1940, as the class valedictorian. Initial college studies were at Baylor University in 1940, where he was awarded a scholarship as a concert violinist. World War II interrupted the writer's college studies, although while in the army he was an A.S.T.P. student in mechanical engineering at Stanford University. After World War II, he returned to college at Texas Technological College and subsequently obtained his B.S. degree in Mechanical Engineering in August, 1947.
- Graduate Study: In June, 1948, the writer entered the Graduate School of the University of Texas in which he completed the requirements for the Master of Science degree, with a major in Mechanical Engineering, in August, 1952. In September, 1954, he entered the Graduate School of Oklahoma Agricultural and Mechanical College, where he completed requirements for the Doctor of Philosophy degree in May, 1956.
- Experience: The writer entered the United States Armed Forces in June, 1943. He served as a Sergeant of Infantry until January, 1945, when he received a commission in the Infantry. He served in the United States, in the Philippines, and in Japan prior to his honorable separation from the service as 1st Lt. of Infantry in September, 1946. After graduation from

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- Publications: The writer is co-author of the following college textbooks: <u>Elementary Problems in Engineering</u>, Macmillan, 1951; <u>The Slide Rule</u>, Macmillan, 1953; and <u>Workbook of</u> <u>Slide Rule and Elementary Problems in Engineering</u>, 1952.
- Professional Organizations: The writer is a member of the following professional organizations: National and Texas Society of Professional Engineers, American Society of Mechanical Engineers, American Institute of Industrial Engineers, American Society for Engineering Education, Sigma Tau, Alpha Pi Mu and Pi Tau Sigma.