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DESIGN AND DEVELOPMENT OF A COMPUTERIZED HIGH TEMPERATURE PULSE CALORIMETER

A Thesis

Presented for the

Master of Science

Degree

The University of Tennessee, Knoxville.

Debasis Basak

December 1992

DEDICATION

This thesis is dedicated to
the memory of my paternal grandparents
Late Mr. Nalini K. Basak and Late Ms. Giribala Basak,
to the memory of my maternal grandfather
Late Mr. Aswini K. Dalal,
to my maternal grandmother
Ms. Karunamayee Dalal
and to my parents
Dr. Ajit K. Basak and Ms. Anita Basak

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ABSTRACT

A computerized pulse heating calorimeter was designed to measure the specific heat of electrical conductors. A computer assisted data acquisition and control system was built and computer programs for acquisition and specific heat calculation were written. The calorimeter was tested from room temperature to 480 °C with nickel as the specimen.

The next few paragraphs contain brief description of the vacuum pumping system, instrument rack, power supply, data acquisition and control programs, overall manager program and preliminary data analysis.

The vacuum pumping system consisted of a mechanical pump and a diffusion pump whose outlet was connected to the vacuum chamber where the specimen was held in a vertical position by the specimen holder. The electrical connections (current leads, voltage taps and thermocouple wires) coming from the specimen holder were connected to terminals on the top platform. These terminals were connected to the instruments situated in the rack and then to a personal computer.

The instrument rack mainly consisted of (1) three separate Preston Amplifiers for amplification of the acquired signals, (2) a programmable power supply, (3) a standard resistor and (4) a digital voltmeter. The amplifiers were set to accept thermoelectric voltage from the thermocouple welded to the specimen, voltage across the

effective specimen length and the current signal from across a standard resistor. The amplified analog signals were fed to the analog-to-digital converter, and converted to digital signals which were then fed to the computer.

The input of the power supply was set to accept a digital to analog input signal from the computer, process it, then send the specified current to the specimen for the specified length of time.

Two main computer programs were written to conduct the specific heat experiment from the keyboard of the computer. The first program is a control and acquisition program and directs the power supply to send a one step current pulse to the specimen. The operator can specify the current for the pulse and its duration by typing their values using the keyboard or by selecting some standard runs. The acquisition part of the program loads the current, voltage and temperature signals from the specimen and arranges them in three data arrays. Calculations are performed on these data to compute the instantaneous power and the electrical resistivity of the specimen which are displayed on the screen for each data point. The data array for time, power and temperature can be stored on the hard disk to be used in another program.

The second program is the overall manager program and reads the raw data created by the acquisition program from the hard disk and carries out conversions, differentiations and other calculations to get equations for the rate of change of temperature during pulsing and cooling and the equation for the power input to the specimen

with time (t) and temperature (T). The calculated power and slope values are then substituted into the specific heat (C_p) equation and a Cp-T curve in the desired range is obtained.

A section on preliminary data analysis has been included which shows the effect of current reversal, changing interval limits for data fitting and degree of fit on the specific heat curve. However, the main goal of the project was the design of the calorimeter rather than data analysis.

The main problems in the design of the calorimeter were due to vacuum leaks in the pumping system and disturbance in the thermocouple emf due to external electromagnetic fields.

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PART I
INTRODUCTION

PART I: CHAPTER 1
GENERAL INTRODUCTION

HISTORICAL BACKGROUND OF CALORIMETRY RESEARCH AT THE UNIVERSITY OF TENNESSEE

The University of Tennessee has had an active project on adiabatic calorimetry as early as 1950. Picklesimer [1] worked on Calorimetric Measurements of High Purity Iron and Eutectoid Steels in 1954, Pawel [2] conducted research on The Application of Dynamic Adiabatic Calorimetry to the Copper-Nickel System from 50 to 620 °C in 1956 and McElroy [3] worked on The Application of Dynamic Adiabatic Calorimetry to the Iron-Iron Carbide System from 50 to 950 °C, just to name a few. The equipment was housed in Estabrook Hall first and then in Dougherty Hall. These projects were sponsored by the United States Atomic Energy Commission. The first effort in designing a pulse calorimetry technique by a University of Tennessee student was in 1965 by Kollie [4] who, in association with the Oak Ridge National Laboratory, designed and used a pulse calorimeter for measuring the specific heat of pure iron from 100 to 1400 °C. In 1985 calorimetry research at University of Tennessee came to a halt due to the lack of sponsorship and the unavailability of interested graduate students.

The early months of 1990 saw a renewed endeavor to initiate a pulse calorimetry technique and hence a new calorimetry laboratory

was set up in Estabrook Hall under the direction of Dr. C. R. Brooks and Dr. D. L. McElroy, with the involvement of the author, and with the support of the Department of Materials Science and Engineering and the Center for Materials Processing of the University of Tennessee.

A dismantled calorimeter from earlier research at the University of Tennessee was transported from Dougherty Engineering Building to Estabrook Hall. The calorimeter was cleaned, old O-ring seals were replaced, a mechanical pump was installed, water lines were fixed and it was brought back to working condition. An instrumentation rack was assembled. A new Dell 310 System personal computer and the necessary peripherals were purchased. The calorimeter was electrically connected to the instrument panel and to the computer. All this was done as an effort to build the new calorimetry laboratory and to satisfy the current thesis goal which was to design and develop a high temperature pulse calorimeter for the measurement of the specific heat of electrical conductors from room temperature to the melting point of metals with full computer control. The laboratory now has, among other facilities, a computerized pulse calorimeter.

PULSE HEATING TECHNIQUE IN GENERAL

Thermodynamic properties of metals are important for their study and use in engineering calculations. Specific heat is one such property. Specific heat of a material is the amount of heat required

to raise or lower the temperature of a unit mass of a material by an unit amount. The calorimeter is an important tool for determining the specific heat of a material. The pulse calorimeter, in particular, has certain advantages over other calorimeters for determining the specific heat of electrical conductors (e.g., metals) at high temperatures.

The author expects that such a high temperature process coupled with a high speed acquisition system can broaden the domain of interaction between theory and experiment. The physical and magnetic changes, for example, and the energy associated with such changes, can be determined and compared with theoretical explanations. The discontinuities in the specific heat curve, such as those due to order-disorder transformations or ferromagnetic to paramagnetic change, can be detected and the temperature at which they occur can be determined accurately from the high resolution data set. A discontinuity in the specific heat versus temperature curve for nickel, for example, occurs at 633 K [5]. Determining the temperature at which this discontinuity occurs and comparing with the data in literature serves as a method of checking the thermometry of the calorimeter.

The literature provides accounts of several other calorimetry methods which are in common use for measuring specific heat. They may be grouped under drop, levitation, differential, modulation and adiabatic calorimetry. Above 1300 K the accuracy of measurements by adiabatic calorimetry degrades to a level at which specific heat

values of better accuracy can be obtained by pulse calorimeters or drop calorimeters. Drop calorimeters fail to give accurate values of specific heat if the specimen is in a non-equilibrium state. Also, extended exposure to high temperature in other calorimetric methods causes a host of problems to the specimen and the calorimeter (e.g., heat loss, specimen contamination, oxidation, loss of mechanical strength, loss of electrical insulation). The pulse method attempts to minimize the severity of some of these problems by enabling the high temperature exposure to be over in a very short time (e.g., few seconds). This is the principal reason for adopting the pulse method for high temperature specific heat measurement.

Pulse calorimetry essentially consists of passing a high current for a short period of time, through the specimen, which is in the form of a wire, such that the current is in the form of a square wave or a pulse. The temperature rise during the pulse due to resistive self-heating of the specimen is noted and so are the instantaneous current to the specimen and voltage across the effective specimen length. After the power to the specimen is shut off the temperature during cooling is also noted. This temperature data are needed for cooling correction. The temperature is usually recorded with a thermocouple. By acquiring these electrical quantities in arrays in a time function it is possible to process and fit these data. Then using the generated fit statistics and the specific heat equation (equation 5, Part III, Chapter 5) the specific heat is calculated. This is discussed in further detail in Part III, Chapter 5. At temperatures at which a

pulse calorimeter is usually used, heat loss due to radiation from the specimen to its surroundings is a major factor. Conduction losses through the ends of the specimen become a dominant factor at lower temperatures. To correct for the finite heat loss from the specimen, the slope of the cooling data is calculated and subtracted from the slope of the heating data, at the same temperature. The design of the calorimeter and the program structure takes into account strict environmental control, proper recording of electrical signals, proper statistical treatment of data and usage of the correct power balance equation to extract an accurate value for specific heat. The designed calorimeter can then be made available to the interested materials scientist for measuring the specific heat of metals and alloys in a wide range of temperatures. Almost any material which conducts electricity can be made the specimen. But, depending on the electrical resistivity of the material, appropriate specimen dimensions need to be selected to generate enough Joule heat to raise the specimen temperature somewhat above the maximum temperature at which the specific heat of the substance is to be determined.

External heating can be used to raise the temperature of the specimen initially, before the application of the pulse. This will allow going to a higher temperature or achieving the temperature in a shorter time, thus minimizing heat loss. Alternatively, the initial rise in temperature can be attained by applying a steady-state current to the specimen. Both methods will allow going to higher temperatures.

The calorimeter, the acquisition system components and the program modules have been installed in a way to allow the operator to apply different experimental methods to measure the same thermodynamic quantity. For example, instead of using a short duration pulse method, which will last for a few seconds, the operator can apply power in steps of increasing current allowing the temperature to come to a steady state, and then calculating the specific heat from cooling data. This technique was proposed by Zemansky [6]. With this method the experiment can run for even a few days. With the pulse calorimetry technique, physical property data, other than specific heat, can also be measured with a high accuracy (e.g., 1% error) with no alterations in the calorimeter and acquisition set-up and with only little alterations in the computer software. For example, electrical resistance, electrical resistivity and total hemispherical emittance can be measured or determined. Equations for the functional relationship between each of these quantities and the temperature can also be generated by fitting data over the desired temperature range. Quantities like enthalpy and entropy can then be determined by integrating the Cp=f(T) relationship and Cp/T=f(T) relationship, respectively.

PART I: CHAPTER 2
DEFINITION OF SPECIFIC HEAT AND ITS RELATIONSHIP WITH OTHER THERMODYNAMIC QUANTITIES

DEFINITION OF SPECIFIC HEAT

If we think of a system having arbitrary mass then its heat capacity, c, can be defined with the following limit

$$c = Lt\,(\Delta T \rightarrow 0)\,\frac{\Delta Q}{\Delta T} = \frac{dq}{dT} \qquad \ldots.(1)$$

where ΔQ is the quantity of heat added to the system in order to raise its temperature by an amount ΔT. Dividing the above equation by the system mass, m, we arrive at the expression of specific heat, C

$$C = \frac{c}{m} = \frac{dq}{dT} \qquad \ldots. (2)$$

where dq is the quantity of heat required to raise the unit mass of the system by an amount dT. 'd' in 'dq' refers to an infinitesimal change in 'q'. The Systeme International (SI) unit for specific heat is J gm-mol^{-1} K^{-1} or J kg^{-1} K^{-1} (1000 J gm^{-1} °C^{-1}) [8].

The specific heat is path dependent so the path must be specified in order to determine its value. For any substance the equation of state will be of the form f(P,V,T) = 0, where P is hydrostatic pressure, V is volume and T is temperature. Since any two variables among the three are independent variables, only two

variables can have arbitrary values at the same time. For example if T is increased only one of the two remaining variables, P or V, can be kept constant. So two major specific heats have been defined, one at constant volume, the other at constant pressure.

$$C_p = \left[\frac{dq}{dT}\right]_p \quad ....(3) \quad \text{and} \quad C_v = \left[\frac{dq}{dT}\right]_v \quad ....(4)$$

The specific heat at constant volume, C_v, is also an intensive property of the substance. The determination of this fundamental quantity for various metals and alloys is of great interest to physicists. For solids it is difficult to measure C_v directly. Usually the specific heat at constant pressure, C_p, is determined and the corresponding C_v is calculated from a suitable thermodynamic relationship. This general relationship between the specific heat capacity at constant pressure and that at constant volume enables us to calculate one from the knowledge of the other. The relationship is derived as follows.

$$C_p = (dq/dT)_p \quad ....(5)$$

$$C_p = [d(U+PV)/dT]_p \quad ....(6)$$

$$C_p = (dU/dT)_p + P(dV/dT)_p \quad ....(7)$$

Internal energy U is a function of state and can be expressed as U=U(T,V)

$$dU = (\delta U/\delta T)_v \, dT + (\delta U/\delta V)_T \, dV \quad ....(8)$$

$$(\delta U/\delta T)_p = (\delta U/\delta T)_v + (\delta U/\delta V)_T (\delta V/\delta T)_p \quad ....(9)$$

$$C_p = C_v + (\delta U/\delta V)_T \, (\delta V/\delta T)_p \quad ....(10)$$

From equations (7) & (9),

$$C_p = C_v + [P + (\delta U/\delta V)_T] (\delta V/\delta T)_p \quad \text{....(11)}$$

$$C_p = C_v + C_d \quad \text{....(12)}$$

In equation 12, C_d is called the dilation correction. This contribution from the dilation of the lattice can be expressed in terms of isothermal compressibility, κ, and thermal expansion coefficient, α.

From the first law and the second law of thermodynamics

$$dU = TdS - PdV \quad \text{....(13)}$$

$$(\delta U/\delta V)_T = T (\delta S/\delta V)_T - P \quad \text{....(14)}$$

From (11) and (14)

$$C_p = C_v + T (\delta S/\delta V)_T (\delta V/\delta T)_p \quad \text{....(15)}$$

$$C_p = C_v + T (\delta P/\delta T)_v (\delta V/\delta T)_p \quad \text{....(16)}$$

But

$$(\delta P/\delta T)_v (\delta T/\delta V)_p (\delta V/\delta P)_T = -1 \quad \text{....(17)}$$

$$(\delta P/\delta T)_v = - (\delta V/\delta T)_p (\delta V/\delta P)_T = \alpha / \kappa \quad \text{....(18)}$$

where $\alpha = (1/V) (\delta V/\delta T)_p$ and $\kappa = -(1/V) (\delta V/\delta P)_T$.

From (16) and (18)

$$C_p = C_v - T (\delta V/\delta T)_p^2 / (\delta V/\delta P)_T = \alpha^2 VT / \kappa$$

Or $$C_p - C_v = -T (\delta V/\delta T)_p^2 / (\delta V/\delta P)_T = \alpha^2 VT / \kappa \quad \text{....(19)}$$

Thus $C_d = \dfrac{\alpha^2 VT}{\kappa}$ where,

α = volumetric coefficient of thermal expansion

κ = coefficient of isothermal compressibility

$$C_d = C_p - C_v \quad \text{....(20)}$$

where C_d is the dilation correction.

The main contributions to the specific heat capacity at constant volume, C_v, can be stated theoretically by considering the micro-mechanisms which lead to the changes in the internal energies of a substance [5]. The contributions come from the following:

C_{vt} = Contribution of transitional motion of atoms

C_{ve} = Contribution of electronic structure changes

C_{vv} = Contribution of vibrational motion of atoms

C_{vw} = Contribution of rearrangement of atoms

C_{vr} = Contribution of rotational motion of atoms

C_{vn} = Contribution of nuclear structure changes

C_{vi} = Contribution from interaction of the different mechanisms

Thus, $C_v = C_{vt} + C_{ve} + C_{vv} + C_{vw} + C_{vr} + C_{vn} + C_{vi}$ ....(21)

In this project our interest will be restricted to simple solid materials. For such solids, the translational and rotational motion contribution can be neglected. Above 10 K the nuclear contribution C_{vn} also becomes negligible.

The term C_{vw} takes into account configurational ordering and vacancy and divacancy formation. C_{ve} also includes contributions from excitations of valence electrons and alignment of spin in magnetic effects. The contribution of C_{vi} is very difficult to assess. It is assumed to be negligible. But sometimes it is necessary to make adjustments in other contributions to validate this assumption. With the aforesaid assumptions, equation (21) can be written as

$$C_v = C_{vv} + C_{ve} + C_{vw} \quad \text{....(22)}$$

The specific heat capacity at constant pressure, C_p, has contributions from all the factors involved in equation (21) plus that due to dilation of the lattice, C_d. Thus the expression for C_p will be :

$$C_p = C_d + C_{vh} + C_{va} + C_{ve} + C_{vw} \quad \text{....(23)}$$

In this equation the contribution from vibrational motion has been broken up into two components - the harmonic contribution, C_{vh}, and the anharmonic contribution, C_{va}.

The value of C_v for a substance can be determined from the measured value of C_p and the value of C_d at the required temperature. However, little data are available for isothermal compressibility coefficient, κ, for high temperatures for most metals and alloys. For this reason approximation methods are usually employed to obtain C_d.

RELATIONSHIP OF SPECIFIC HEAT TO OTHER THERMODYNAMIC QUANTITIES

It follows from the first law of thermodynamics that whenever a quantity of heat, dQ, is added to a system, part of the heat is retained in the system as internal energy dU of the system and part of it is utilized in performing the external work, dW.

$$dQ = dU + dW \quad \text{....(24)}$$

If the work is done only in the form of changing the volume of the system, against the external pressure, then $dW = P\ dV$.

From equation (4) and (24) it follows that

$$C_V = \left[\frac{dU}{dT}\right]_v \qquad \text{....(25)}$$

As mentioned before, the quantity C_V is of definite theoretical interest since it gives an idea about the change in the internal energy of the system with change in temperature, which in turn can be related to changes in the translational, rotational, vibrational, electronic and magnetic energy of its atoms.

Some of the properties of specific heat can be explained with the second law of thermodynamics. For a reversible process the change in entropy is

$$dQ = T\,dS \qquad \text{....(26)}$$

From equations (3), (4) and (26) C_p and C_V can be expressed as

$$C_p = T\left[\frac{\delta S}{\delta T}\right]_p \quad \text{....(27)} \quad \text{and} \quad C_V = T\left[\frac{\delta S}{\delta T}\right]_v \quad \text{....(28)}$$

In terms of P, V, S and T, the change in the internal energy of the system may be written as $dU = T\,dS - P\,dV$.

Other principal thermodynamic functions, in differential form, may be expressed as

$$dA = d(U - TS) = -SdT - PdV \qquad \text{....(29)}$$

$$dH = d(U + PV) = TdS + VdP \qquad \text{....(30)}$$

$$dG = d(U - TS + PV) = SdT + VdP \qquad \text{....(31)}$$

where A is Helmholtz free energy, H is enthalpy and G is Gibb's free energy. These functions are essentially measures of the energy content of the system and can be related to C_p and C_v. For example at constant pressure,

$$dH = T\,dS = dQ \qquad \text{....(32)}$$

So C_p can be expressed as both

$$C_p = \left[\frac{dQ}{dT}\right]_p = \left[\frac{dH}{dT}\right]_p \qquad \text{....(33)}$$

PART I: CHAPTER 3
PULSE CALORIMETRY

The first successful attempt to use pulse calorimetry for the measurement of specific heat of electrically conducting materials was by Worthing [7]. This technique, reported in 1918, employed passage of short time (e.g., 1 sec.) electric current through a specimen and the measurement of absorbed power by the specimen (in this case tungsten and carbon) while going from one steady state to another. Electrical measurements were made with a modified potentiometer-galvanometer technique, in which the galvanometer was pre-adjusted to give zero deflection under pulse conditions. The temperature of the specimen was determined from electrical resistivity measurements on the specimen during the pulse experiment and from the knowledge of the resistance-temperature relation, which was obtained separately under steady-state conditions [8].

Pasternak [9], Parker [10] and Kollie [4] also used this general concept, but after considerable refinements and modifications. Pasternak [9] made an exploratory study of platinum by the method in which the total hemispherical emittance could be obtained in addition to the specific heat. For a specimen of uniform cross-section, the energy balance during electrical heating (neglecting conduction losses) can be described by

$$I E = m C_p (dT/dt) + A \sigma \varepsilon (T^4 - T_o^4) \qquad ....(1)$$

where,

I = Current through specimen

E = Voltage across effective specimen

m = Specimen mass

ε = Total emissivity

T = Specimen temperature

T_o = Ambient temperature

t = Time

σ = Stefan-Boltzmann constant

A = Specimen emittance area

C_p = Specific heat

The term on the left of the equation is for the power input to the specimen. The first term on the right of the equation is for the heat absorbed by the effective specimen and the second term on the right is for the radiation loss from the effective specimen to the surrounding.

In Pasternak's experiment a long filament was flash heated in high vacuum (10^{-8} Torr or less). For establishing a functional relationship between C_p and ε, the parameters of interest, only I and E need to be measured. They define the power input as well as the specimen temperature if the temperature-resistance correlation is known. At least two experimental runs differing in the rate of heating are required. Typical lower and higher heating rates used were 35 K/sec and 2000 K/sec, respectively. The potential and current were recorded with a fast two-channel recorder. From the two C_p-ε equations determined from the two runs the two unknown

parameters C_p and ε were determined. Pasternak's determination of specific heat of platinum between 400 and 1300 K were 5% lower than the values reported in literature.

Parker [10] used a high speed capacity discharge system. He used this system to carry out experiments on heat of transformation and the rates involved in solid-solid transformations of titanium.

Kollie [4,5] designed a pulse calorimeter capable of operating up to 1200 K and worked on nickel, iron and the alloy Ni_3Fe. He utilized a digital voltmeter and a computer to record his readings at a speed of 400 readings per second with a full scale resolution of 0.1%. This system was an attractive one for the measurement of specific heats around transition points at moderately high temperatures. Later he added a furnace to the system and modified his data acquisition system. This extension allowed him to measure specific heat up to 1500 K. A full-scale resolution of 0.01% was obtained with a speed of 2000 readings per second.

The calorimeter designed in this study employs a pulse of about thirty seconds. The current through the specimen, the voltage across the effective specimen length and the thermal emf of the thermocouple attached to the specimen are all recorded with a certain operator specified speed for the entire pulse period and the subsequent cooling period. Power is calculated from the acquired voltage and current data. Thermal emf is converted to temperature with the help of conversion equations. The slope of the temperature-time relationship, for heating and cooling, is calculated by

differentiating the corresponding functions for temperature-time. The calculated power and slope data are then substituted in the general equation (equation 2) from which the specific heat at any temperature can be calculated.

$$Cp = \frac{E \quad I}{m \left[\left(\frac{dT}{dt}\right)_h - \left(\frac{dT}{dt}\right)_c \right]} \qquad \ldots.(2)$$

where,

E = Voltage drop across the effective length of the specimen

I = Current to the specimen

m = Effective mass of the specimen

$(dT/dt)_h$ = Slope of temperature-time during heating

$(dT/dt)_c$ = Slope of temperature-time during cooling

The pulse method, basically, is a resistive self-heating of the specimen by the passage of electric current through it. The measuring system consists of an electrical power-pulsing circuit along with high speed measuring devices interfaced with amplifiers.

CLASSIFICATION

Cezairlian [8] classifies pulse calorimetry into two types, depending on the nature of initial heating and the way the current pulse is applied.

Category I

The specimen is initially at a temperature above room-temperature under steady-state conditions. Then the specimen is pulsed with electric current for a very short time. This category can be further divided into two types.

a. Where the specimen is initially heated by resistive self-heating, before the application of the pulse.

b. Where the specimen is initially heated in a furnace before the application of the pulse.

In Category I the initial temperature is achieved by steady-state power dissipation. In this method there is only a small temperature rise (e.g. 10 K) from the initial high temperature, because the magnitude of the pulse current is small, usually less than 10 A.

In Category Ia the steady-state temperature is reached by resistive self-heating and the radiative heat loss becomes appreciable. So a correction for the lost power needs to be made based on either an estimated emittance of the specimen or on the data for the cooling period following the pulse. In Category Ib, since the specimen is initially in thermal equilibrium with its surroundings the difference in temperature of the specimen and its surroundings, during the pulse, is small. Correspondingly, the heat loss is small. This allows for a heating rate slower than that in Category I, which is advantageous from the data recording point of view.

Category II

In this method the specimen is initially at room temperature. A current pulse of very short duration is applied to raise the temperature to the desired level.

The experiments of this category are faster than those in Category I. The specimen and its surroundings are initially at room-temperature and only the specimen is heated to a high temperature with a short pulse. Since the current pulse is of a shorter duration in Category II it does not have the limitations of Category I, which are due to extended high temperature exposure of the specimen. But a limitation of this category is that since the surrounding is at room temperature during and after the pulse the vast temperature difference between the specimen and the surrounding leads to high radiation heat loss. In order to reduce this loss high heating rates are required. This requires the pulse to be as short as possible. So the method in Category II is faster than that in Category I. But a shorter pulse requires a higher pulse current which can create strong electromagnetic fields which disturb the sensitive measuring instruments.

Techniques in both Category I and Category II can be implemented using the designed calorimeter, but so far only that in Category II has been tried.

RELEVANT EQUATIONS FOR DETERMINATION OF SPECIFIC HEAT

This section deals with the thermodynamic equations pertaining to pulse calorimetry. The electrical power imparted to the specimen is mostly stored in the specimen in the form of the heat content of the system distributed among its components; the rest is lost to the surroundings. If the applied power is equated to the stored power and power lost then,

$$E\,I = C_p\,m\,(dT/dt)_h + Q_L \quad ....(3)$$

where,

E = Potential drop across the effective length of the specimen

I = Current to the specimen

C_p= Specific heat of the specimen material

m = Effective mass of the specimen

Q_L= Total power loss from the specimen

$(dT/dt)_h$= Rate of heating the specimen

The term specimen, here, indicates the effective specimen. The effective length of the specimen is different from its physical length. The effective length of the specimen is the length between the voltage probes. The effective length is made small enough to minimize the temperature gradient across this length of the

specimen due to end effects, since the ends are at a lower temperature.

Figure 1.1 shows the temperature-time relationship of the specimen as it is taken through the heat pulse and is subsequently allowed to cool.

Here,

T_r = room temperature

T_m = maximum temperature

t_o = start of pulse time

t_m = end of pulse time

Since no power is supplied during the cooling period, the term EI is zero. So, for the cooling period, we can write the heat balance equation as

$$- C_p \; m \; (dT/dt)_c = Q_L \qquad \text{....(4)}$$

where $(dT/dt)_c$ is the cooling rate of the specimen.

Combining equations (3) & (4) , C_p can be written as

$$C_p = \frac{E \; I}{m \; (dT/dt)_h \; (1+1/M)} \qquad \text{....(5)}$$

Here we have introduced a new term, M, which is the negative ratio of the rate of change of temperature during heating and cooling stages.

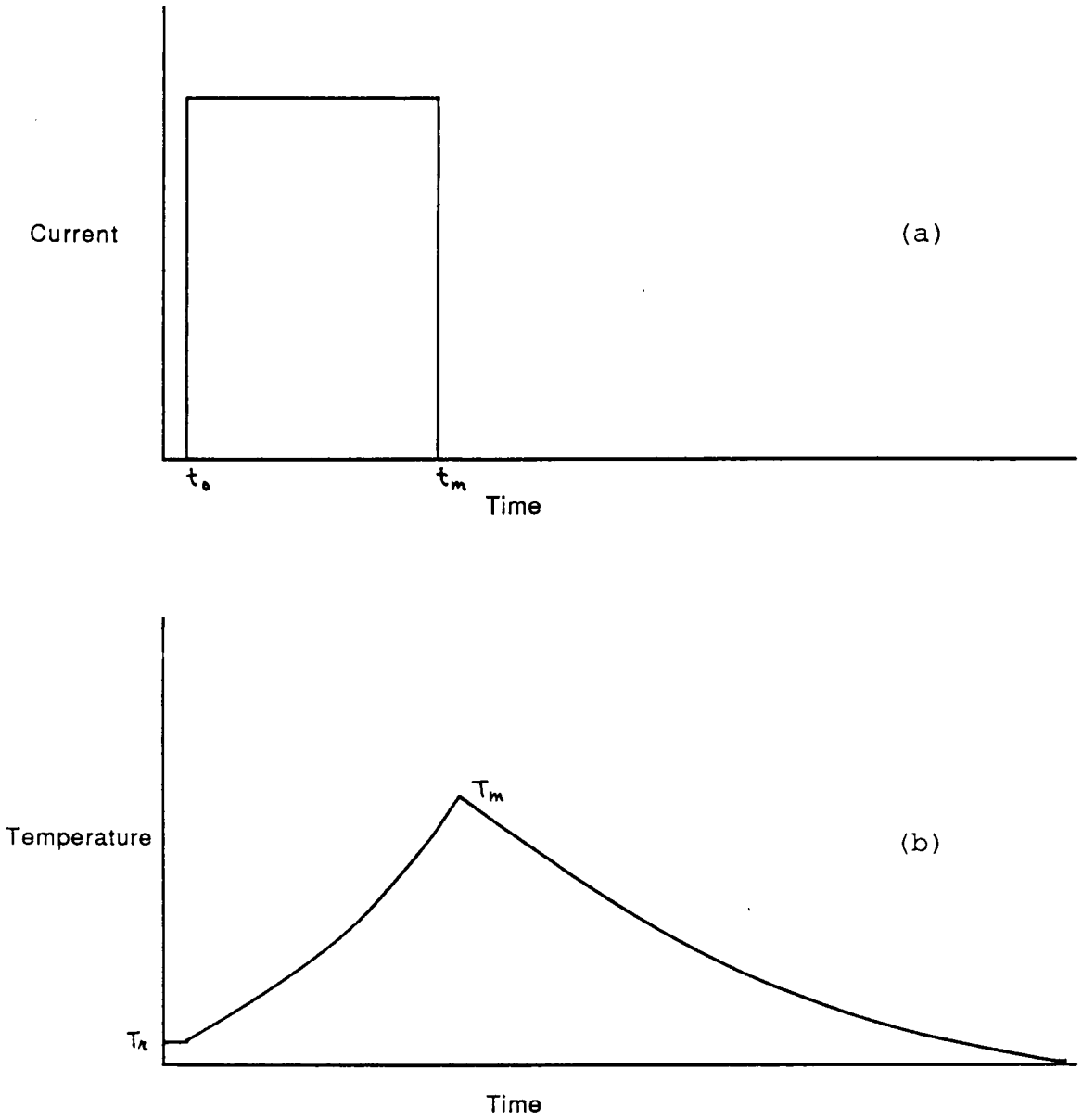


Fig. 1.1 (a) Current profile during pulsing. (b) Temperature profile of the specimen during pulsing and during subsequent cooling.

$$M = - \frac{(dT/dt)_h}{(dT/dt)_c} \qquad \text{....(6)}$$

The factor, M, has an important significance to the experimenter. Ideally an experiment would be designed to make the value of M sufficiently large so as to render the factor 1/M negligible. This would minimize the correction due to heat loss. Physically, this would require a high rate of change of temperature during the heating stage as compared to the cooling stage, as can be inferred from equation (6). Pulse calorimetry is basically devised to minimize this factor, M, by raising the temperature of the specimen over a very short time (e.g., 1 to 3 sec. or 10 to 100 °C/sec.).

The heat loss correction factor is not just typical of pulse calorimetry but is also a part of the expression for C_p for adiabatic calorimetry. In adiabatic calorimetry the drift rate is taken as the correct value of the heat loss (or gain) during dynamic operation. In adiabatic calorimetry the shield is controlled at the temperature of the specimen at the location of the thermocouple. But the specimen will lose heat with time which results in the temperature of the specimen decreasing. Hence, the temperature of the shield goes down too. To estimate the drift rate, the power to the specimen is shut off. Then the rate at which the specimen temperature changes is noted. The change in temperature of the specimen with time, with no power going into the specimen, is the drift rate. Pawel [2] obtained a relationship between the apparent specific heat, true specific heat and the drift rate and presented it in the form

$$C_p = \frac{C_{pa}}{1 - \frac{C_{pa}\, R(T)}{K}} \qquad \text{....(7)}$$

where,

C_p = True specific heat

C_{pa} = Apparent specific heat

R(T) = Static drift rate (due to lack of perfect adiabatic conditions)

K = A quantity directly proportional to the heating rate during dynamic operation and inversely proportional to the specimen mass.

Equation (5) can be written as

$$C_p = \frac{C_{pa}}{1 + \frac{1}{M}} \qquad \text{....(8)}$$

since,

$$C_{pa} = \frac{E\ I}{m\ (dT/dt)_h} \qquad \text{....(9)}$$

From equation (7) and (8)

$$1 + \frac{1}{M} = 1 - \frac{C_{pa}\, R(T)}{K} \qquad \text{....(10)}$$

$$M = \frac{-\ K}{C_{pa}\, R(T)} \qquad \text{....(11)}$$

Thus, in equation (11) a correlation between factor M, used in pulse calorimetry calculations, and the drift rate, R(T), used in adiabatic calculations, has been established.

Coming back to pulse calorimetry, from equation (5), the

specific heat of the specimen can be calculated if the voltage, current and temperature during the heating period are available as a function of time, and if temperature as a function of time is available during cooling.

The power loss, by radiation, during the high temperature pulse period is usually calculated from the cooling data. But if the surface emittance data on the specimen material are available, power loss by radiation can be calculated using the relation for thermal radiation [11].

$$Q_r = \varepsilon\sigma A_S (T^4 - T_O^4) \qquad ....(12)$$

where,

ε = Total hemispherical emittance

σ = Stefan-Boltzmann constant

A_s = Effective surface area of the specimen

T = Specimen absolute temperature.

T_0 = Ambient absolute temperature.

ACCURACY OF THE PULSE EXPERIMENTS

The equation for specific heat in terms of the energy supplied to the specimen, the energy absorbed by the specimen and the energy lost to the surroundings involves measurement of the voltage and current passed through the specimen as a function of time. Also temperature data, as a function of time, both during the pulse and during the subsequent cooling period, are needed. The mass of the

specimen needs to be determined too. The measurement of current, voltage and temperature requires, in turn, the interception of electrical signals pertaining to the physical quantities, storing them in computer's memory and fitting them to a polynomial. There is a factor of uncertainty associated with each step of the measurement process. Also correct estimation of the specimen mass necessitates sufficiently accurate values for the density of the specimen material and the dimensions of the specimen. The uncertainty in the measurement of the length and diameter of the specimen, which is in the form of a thin wire, increases the uncertainty in C_p.

An evaluation of the accuracy of the calorimetry system, as a whole, becomes necessary. There are two different approaches to arriving at the overall accuracy - adding up the errors generated due to all the uncertainties involved in the experiment and comparing the measured data with some standard data established earlier. An assessment by Kollie [4] on the errors of measurement of all the quantities that go into the calculation of the specific heat showed a total error of 1.05% when the power loss was less than 10% of the power. The errors of measurement of absolute temperature, T, during the pulse yielded an uncertainty of 3.0 degrees in T. Estimation of errors by comparing with a standard poses a problem in high temperature calorimetry, because no electrically conducting material is widely accepted as a standard for specific heat

measurements above 300 K.

Error analysis and accuracy have been discussed further in Part IV, Chapter 1.

PART II
ELEMENTS OF CALORIMETER SYSTEM DESIGN

PART II: CHAPTER 1
VACUUM SYSTEM

Operations involving the heating and melting of metals are most conveniently carried out in vacuum to prevent reaction with atmospheric gases, conduction and convection heat loss and disturbance due to air currents. The pressure of the enclosed gas can be read continuously and monitored as required. Vacuum systems become almost indispensable for critical high temperature measurements, especially those involving temperatures in excess of 1000 °C. High temperature calorimetry is one example of such an application. Inside the vacuum chamber the only appreciable heat loss is by radiation; conduction and convection losses are minimized. This increases the thermal efficiency and higher temperatures can be attained with the same expenditure of power.

PUMPING SYSTEMS

To attain pressures below 10^{-2} Torr requires both a mechanical pump and a diffusion pump. The diffusion pump operates at much lower air pressures than that of the mechanical pump. The mechanical pump removes the air in the vacuum system to the region where the diffusion pump operates.

Mechanical Pumps

Beside evacuating the system to the pressure level where the diffusion pump operates, the mechanical pump also maintains the operation of the diffusion pump by keeping the exit end of the diffusion pump below the critical pressure required for operation. The size of the mechanical pump determines its capacity to suck air out of the system. The following two considerations are important while selecting the capacity of the mechanical pump - (1) It must be large enough to carry off the gas removed from the entire system and fast enough to maintain the required forepressure at the inlet of the diffusion pump. (2) It should have sufficient capacity to evacuate the chamber from atmospheric pressure to the required low level in the required length of time. For our case a Welch Duo-Seal Vacuum Pump, Model No. 1402 was found suitable and was used.

Mechanical pumps do not vary much in their basic design. They have an eccentric cam which squeezes out a crescent shaped volume of air at each revolution. Figure 2.1 shows a typical example of a two-stage pump. Gas is drawn through the intake I into the chamber A, which assumes the shape of a crescent for all positions of the cam. On the other side of the cam, gas is squeezed through the valve D into the chamber C. Some of it exhausts through the valve to the atmosphere. At some point of time the pressure in the exhaust chamber C falls below the atmospheric pressure. Then the gas is sucked into the second stage which operates in the same way. The

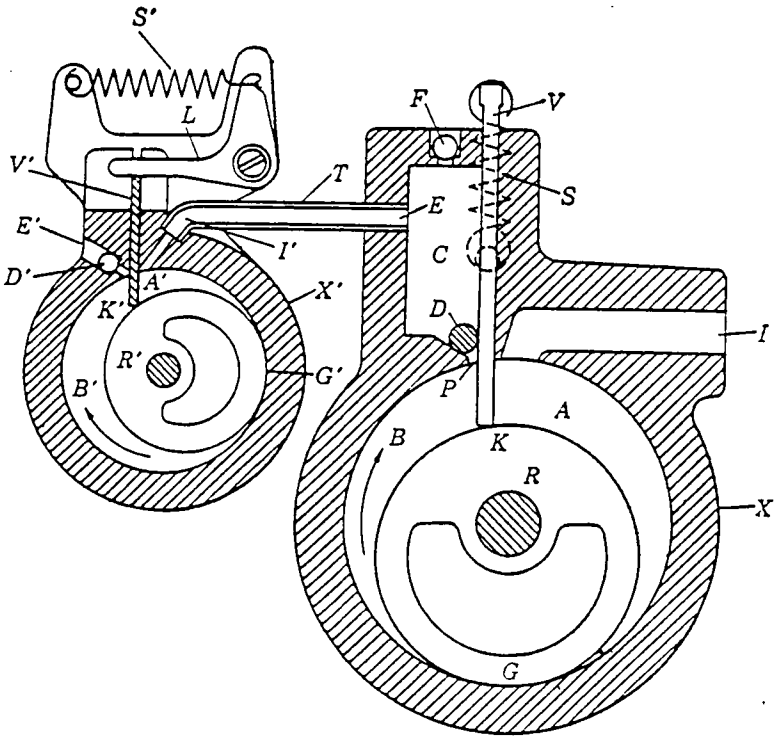


Fig. 2.1 Schematic diagram of a two-stage mechanical pump. I, I', intake; E, E', exhaust; D, D', exhaust valve; C, interstage; F, interstage exhaust valve; V, V', vane; S, S', vane spring; L, vane lever; R, R', rotor; X, X', stator; A, A', intake chamber; B, B', exhaust chamber; P, exhaust port; K, K', contact line of vane with rotor; G, G' contact line of rotor with stator; T, connecting tube. Source: Dushman[12].

second stage pump evacuates chamber C and acts as a fore-pump to the main stage since the pressure differential between B and A is reduced by the action of the second stage.

The efficiency of such systems depends a lot on the fit of the various parts, especially the joints, which are most leak prone. The rotating cam is required to press very tightly on the cam housing. The oil should always be maintained at a certain level. This oil is a sealing medium and also acts as a lubricant, so its presence is always necessary. The oil needs to be changed from time to time to ensure that it is free from dissolved air, water and impurities.

Diffusion Pump

The diffusion pump is a vapor jet pump which transports gas by momentum transfer on collision with the vapor stream. A hydrocarbon oil is heated and vaporized in the boiler which then flows up the chimney and out through a series of nozzles. Figure 2.2 shows a sectional view of a vacuum pumping system in which the main diffusion pump parts are shown. Once the vapor is released from the chimney it is directed downwards and comes into contact with the cooler outer wall where it condenses and reaches the boiler again. The metal wall of the pump is constantly cooled by circulating water through the pipes fitted to the outer walls of the pump.

The flow of vapor in the diffusion pump is supersonic. Gases that diffuse into the supersonic vapor stream are given a downward

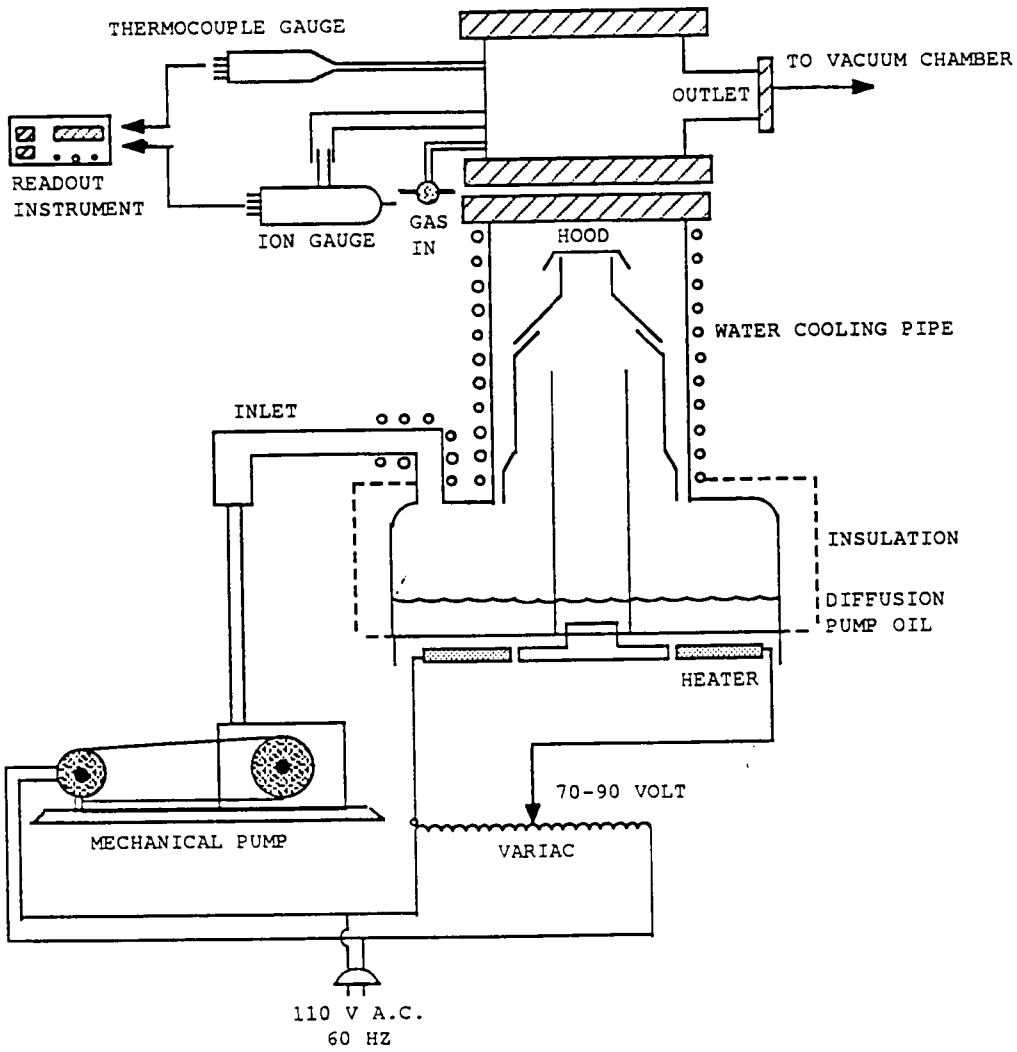


Fig. 2.2 Sectional view of the vacuum pumping system.

momentum and ejected into a region of high pressure. The pump has two compression stages. Each stage compresses the gas to a successively higher pressure than the preceding stage as it transports it toward the outlet. The diffusion pump must be backed by another pump to keep the pressure below the critical forepressure. If the pressure exceeds the critical value, all pumping action will cease because the supersonic jet stream will end closer to the jet and never reach the wall at all.

The four operating regions of the diffusion pump are the constant speed, constant throughput, mechanical pump and the compression ratio region. They are shown graphically in Figure 2.3. In its normal operating range the diffusion pump is a constant speed device.

The gas throughput in the constant speed range is a product of the inlet pressure and the speed of the pump at the inlet flange. It rises linearly with pressure until a critical inlet pressure is reached. Above this pressure the pump throughput remains constant until the critical forepressure of the pump is exceeded and the jets cease to function.

It has been observed that the temperature of the boiler containing the diffusion pump oil is an important factor determining the vacuum obtained (refer to figure 2.4). The voltage supplied by the heater is controlled by a variac which determines the power input to the heater element. As the voltage to the heater element is increased the pressure in the vacuum chamber is initially lowered,

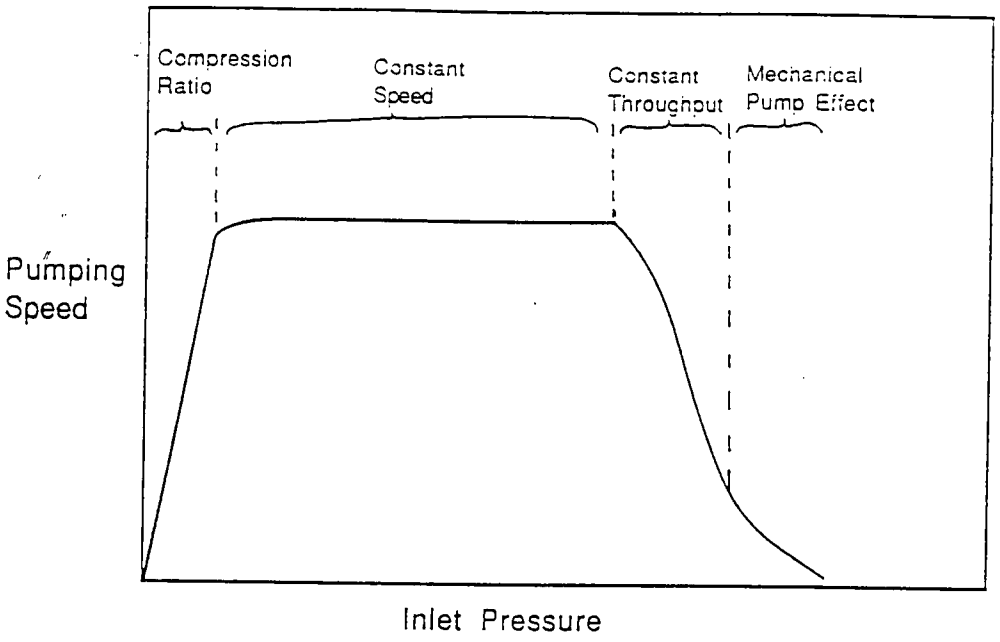


Fig. 2.3 Operating regions of the diffusion pump.

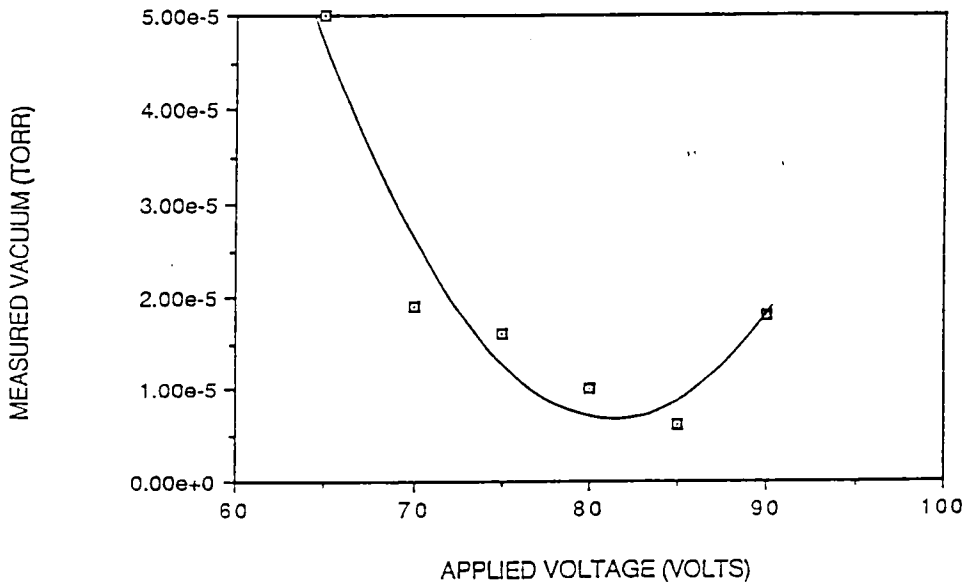


Fig. 2.4 Plot of pressure in the vacuum chamber vs. applied voltage to the heater.

whereafter the pressure rises. A test on the change in the measured vacuum with the change in the applied voltage to the heater was conducted in the laboratory, the results of which are displayed in Figure 2.4. The type of diffusion pump, used in our laboratory, is unknown.

Vacuum Measuring Devices

Thermocouple Gauge

The thermocouple gauge, shown in Figure 2.5, makes use of the changing temperature in a fine tungsten wire due to impingement of gas molecules on it. The wire is heated by a constant current. A thermocouple is spot welded to the wire and generates a thermal emf. Both the wire and the thermocouple are made very small to increase the sensitivity of the gauge. A Varian Type 0531 Thermocouple Gauge was used in our case.

Care must be taken to keep the thermocouple gauge away from the contaminants in the heating chamber. If either the tungsten wire or the thermocouple wires are contaminated there will be an error in the reading. The thermocouple gauge consists of a metal tube with a reduced neck fitted with threaded pipe which screws into the metal system.

Ion Gauge

Figure 2.6 shows the essentials of an ion gauge and Figure 2.7a shows the cross-section of an O-ring seal that fits into the inlet of the

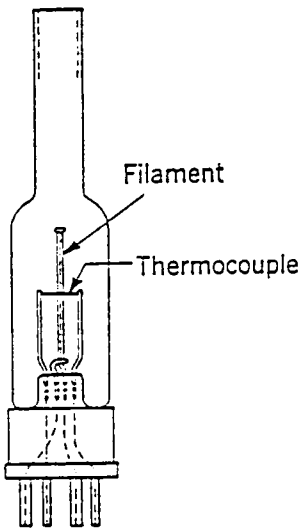


Fig. 2.5 Thermocouple gauge. Source: Guthrie and Walkerling[13].

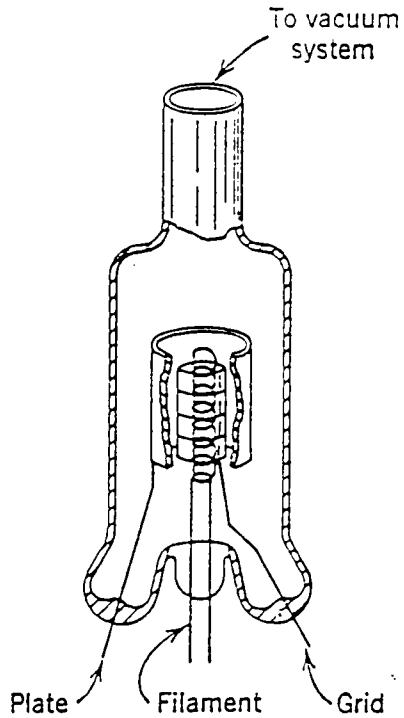


Fig. 2.6 Ion gauge. (Source: Guthrie and Walkerling[13]).

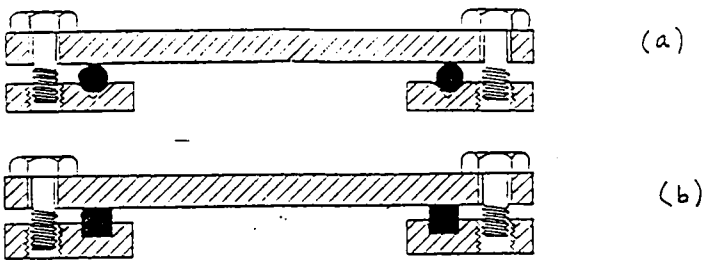


Fig. 2.7 O-ring seals.(a)Circular cross-section (b)Rectangular cross-section.

gauge. For the measurement of high vacuum (e.g., $< 10^{-3}$ Torr) the sensitivity of the thermocouple gauge is not sufficient. The ion gauge reads the vacuum accurately down to about 10^{-8} Torr. A Veeco Instruments RG75P Ion Gauge was used in our case. There are different types of ion gauges but the most common is the hot cathode type. This type of ion gauge, in essence, is a triode. It consists of a cathode, anode and a grid enclosed in a glass tube. There is an inlet which goes to the vacuum system. The filament itself acts as the cathode, which is heated high enough to emit thermoelectrons. The plate or the anode is at a higher potential so that electrons generated in the cathode goes to the anode. Since the grid is placed in between, the electrons are deflected to the grid. But while passing to the grid the electrons collide with the atoms of the gas and ionize them. The ions created between the grid and the plate pass to the plate creating an ion current which can be measured by a suitable microammeter. Hence, the greater the amount of gas the greater is the ionization and a higher ion current is detected.

The location of the vacuum measuring device is also of some significance. The pressure gradient in a vacuum system is always from the mouth of the diffusion pump to the farthest recess of the evacuated chamber. Any bend or crookedness in the system increases this gradient. Obviously, it is desirable to locate the vacuum gauge as close as possible to the area of the vacuum chamber whose pressure is to be known. In general, location of the gauge at a site where the vacuum is poorer is more proper so that the observer

gets a more conservative reading than that which he would have gotten had the gauge been very close to the inlet of the diffusion pump.

VACUUM CHAMBER

The outside shell to the vacuum chamber is a cylindrical structure made out of steel. Water lines on the outer wall of the shell are used to cool the outside, if a furnace is used for external heating of the specimen. The heaters are fitted to the cylindrical vacuum chamber. There are six heaters in all, four of which are fitted to the side and two at the bottom. Figure 2.8 is a photograph of the vacuum chamber with the heater connections. The vacuum chamber is inserted into the outside shell.

The cylindrical space inside houses the specimen holder. This space is 99.1 cm deep and has a diameter of 9.8 cm. Since the height of the specimen holder is 97.8 cm, there is a clearance of about 1.3 cm between the bottom screws of the holder and the bottom of the vacuum chamber. A thermocouple is welded to the inside wall of the vacuum chamber, and is insulated with ceramic and then passed through a metal tube. The hot junction is welded to a small platinum strip which is then welded to the inner wall. The distance from the top of chamber to the hot-junction is 82.6 cm.

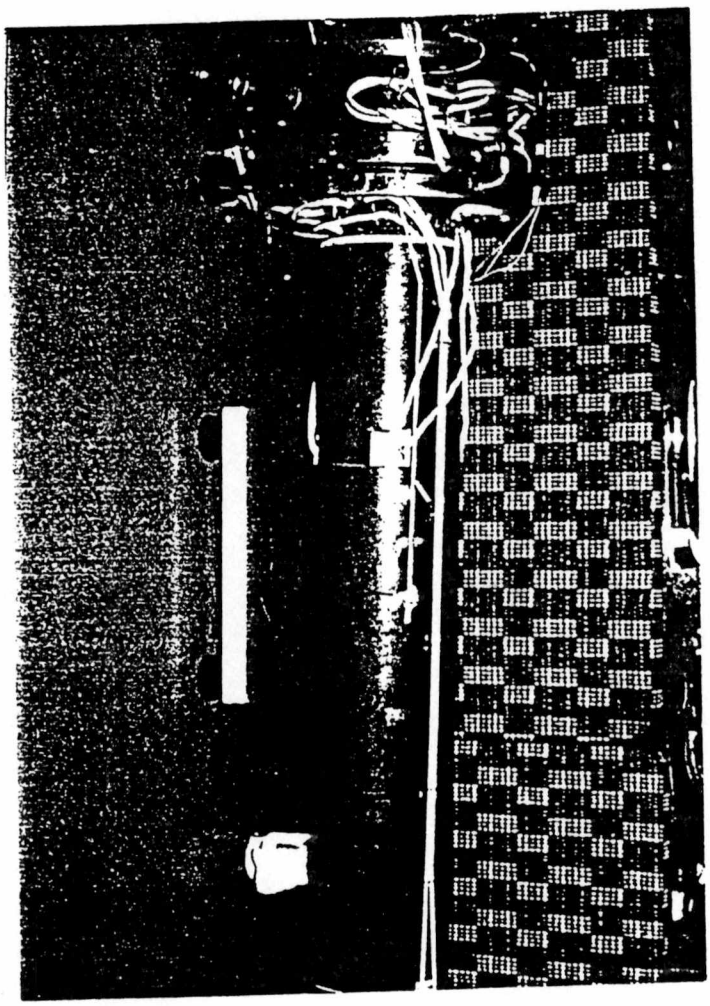


Fig. 2.8 Photograph of internal cylinder which is the vacuum chamber.

VACUUM SEALS

Making effective vacuum seals is technology by itself which has been brought to high degree of perfection in recent years. Any metal vacuum system requires effective sealing where it is connected to other system parts or where devices need to be taken out of the vacuum chamber. The most common places where seals are required are listed below :

(1) Flanges

(2) Tubes

(3) Electrical leads outlet

(4) Windows

(5) Motion introducer

Several O-ring seals, in the form of rubber gaskets, were used in our calorimetry system. Seals of other cross sections were also used. Seals with different inside and outside diameters were used depending upon their location. Both inside and outside diameter was properly selected so that the ring fits into the groove. Seals were used in the flanges between the mechanical pump, and the diffusion pump, between the diffusion pump and the vacuum chamber, in the ion gauge attachment and in resting the top lid. Except the seal used to rest the metal top all other seals have a circular cross section. Figure 2.7(a) shows the cross section of an O-ring seal with an attachment. The ring is recessed into a groove to the depth of its radius. There should not be any deep scratches in the metal-rubber

surface of contact. The flange, lid or cover is screwed in place by screws and nuts which need to be well tightened to attain a snug fit.

O-rings are complete rings of circular cross section and are available in a wide range of sizes from commercial suppliers. O-ring suppliers have manuals which give the correct groove size and design for rings of all sizes. Generally, O-rings are used as seals up to 1 inch in diameter. The use of rings of rectangular cross-section are also common. The metal lid on top of the vacuum chamber rests on the platform, on a rubber seal, which is rectangular in cross section. A rectangular cross-section rubber seal is shown in Figure 2.7(b). When a vacuum is pulled the metal lid pushes against the flat ring to give a good vacuum seal. Thus, flat section rings are preferable to O-rings at certain locations.

LEAK HUNTING

Even though a lot of care is taken in putting together a vacuum system and even though new defect-free O-rings are used as vacuum seals, a newly completed vacuum system will almost always have leaks. Much of the time of a calorimeter designer may be spent initially in detecting the location of the leaks and then setting them right. In this project, several months were spent without the location of the leaks being detected. One reason for getting a low vacuum was the usage of a long vacuum hose. Also, the hose used initially collapsed under vacuum and prevented sufficient suction. It was

replaced by a more sturdy and a shorter hose. A better vacuum was obtained after these changes were made. Also, some of junctions did not have a good seal initially. The seal at the mechanical pump outlet showed a leak which took several days to fix. A soft metal ring, made of indium, was inserted instead of the regular rubber ring to get over the problem. Several detecting techniques are available. Some of these techniques require a vacuum gauge such as a calibrated readout instrument. Crude techniques, such as spraying acetone or painting soap solution, or more sophisticated ones using hydrogen ionization gauge or helium mass spectrograph, are commonly used.

If there is more than one gauge available, then the pressure readings from different parts of the system can be compared. This may help in deciding whether the major trouble lies in the pumping system or in the vacuum chamber. However, if just one gauge is available then one has to proceed by the process of elimination. This may consist of isolating most of the system and starting from the vacuum source, then proceeding in a direction so as to include more parts of the vacuum system and checking for any considerable rise in pressure. The rise in pressure would indicate that the component of the system attached last has the leak. Since just one ion gauge was used in our project, this is the way we had to proceed in order to locate the leak. We started by ascertaining that the mechanical pump was pumping down to the pressure that it should ($\sim 10^{-2}$ Torr). This involved attachment of the thermocouple gauge to the outlet of the mechanical pump and reading the pressure directly with the readout

instrument. This needed a special adapter to be made, one side of which tightly fitted into the hose coming out of the mechanical pump outlet and the other side threaded to the thermocouple gauge. The oil level in the mechanical pump was checked and enough time (e.g., 48 hours) was allowed to drive out the air and moisture from the oil. The mechanical pump was then fitted to the diffusion pump and the vacuum chamber. The diffusion pump and the vacuum chamber did not show any major leaks.

PART II: CHAPTER 2
THERMOMETRY

SPECIFICATIONS

Type R thermocouples were chosen to measure the temperature of the specimen. Type R is a Platinum-13% Rhodium alloy versus Platinum thermocouple. The composition of the positive thermoelement is Platinum-13 ± 0.05 wt% Rhodium. The negative thermoelement is commercially pure platinum. Type R thermocouples have a higher Seebeck coefficient than that of the type S (Pt+10% Rh - Pt) thermocouples.

The restrictions that apply on the usage of these are that they should not be used in reducing atmospheres or in atmospheres containing metallic vapors (e.g., Pb, Zn) or non-metallic vapors (e.g., P, S, As). They should never be inserted directly into the metallic primary tubes [14].

ASTM Standard E230-72 specifies the standard limit and error for type R commercial thermocouples to be ± 1.4 °C between 0 and 538 °C and ± 0.25% between 538 and 1482 °C.

DISCUSSION OF PUBLISHED TEMPERATURE VERSUS EMF DATA

Since the temperature, in °C, was calculated from the fitting functions for type R thermocouples, care had to be taken in selecting the ranges of temperatures for curve-fit, ascertaining the degree of fit in that range and determining how good the fit is. If the data are fitted over wide ranges of temperatures in which there is a change in slope, then the error range and the standard deviation for the fit will increase. Bedford [15] has published values for thermocouple voltages based on IPTS-68. He carried out experiments on twelve meters of wire for each thermoelement and calibrated the thermocouples from - 50 °C to the gold freezing point (1064.43 °C). All the data below 630.74 °C were fitted with a seventh degree function. The fit had a standard deviation of 0.26 μV for seventy experimental points. Two different cubic functions were used to fit data for the ranges 1064.43 to 1665 °C and 1665 to 1767 °C. The standard deviation for the first range was 1.0 μV for eight points and for the second range was 1.5 μV for eight points. Figure 2.9 compiles the degree of fit and curve fit coefficients for the different temperature ranges for power series expansion for the thermoelectric voltage of type R thermocouples.

Work on curve-fitting of mV - °C data has been carried out by other institutions as well. Figure 2.10 shows the comparison of the difference in thermoelectric voltages for type R thermocouples

Temperature range	Degree	Coefficients	Term
−50 to 630.74 °C	7	$5.2891395059 \times 10^{0}$ $1.3911109947 \times 10^{-2}$ $-2.4005238430 \times 10^{-5}$ $3.6201410595 \times 10^{-8}$ $-4.4645019036 \times 10^{-11}$ $3.8497691865 \times 10^{-14}$ $-1.5372641559 \times 10^{-17}$	T T^2 T^3 T^4 T^5 T^6 T^7
630.74 to 1064.43 °C	3	$-2.6418007025 \times 10^{2}$ $8.0468680747 \times 10^{0}$ $2.9892293723 \times 10^{-3}$ $-2.6876058617 \times 10^{-7}$	T^0 T^1 T^2 T^3
1064.43 to 1665 °C	3	$1.4901702702 \times 10^{3}$ $2.8639867552 \times 10^{0}$ $8.0823631189 \times 10^{-3}$ $-1.9338477638 \times 10^{-6}$	T^0 T^1 T^2 T^3
1665 to 1767.6 °C	3	$9.5445559910 \times 10^{4}$ $-1.6642500359 \times 10^{2}$ $1.0975743239 \times 10^{-1}$ $-2.2289216980 \times 10^{-6}$	T^0 T^1 T^2 T^3
1064.43 to 1665 °C $T^* = \frac{T-1365}{300}$	3	$1.5540414086 \times 10^{4}$ $4.2357772712 \times 10^{3}$ $1.4693087343 \times 10^{1}$ $-5.2213889624 \times 10^{1}$	T^{*0} T^{*1} T^{*2} T^{*3}
1665 to 1767.6 °C $T^* = \frac{T-1715}{50}$	3	$2.0416695016 \times 10^{4}$ $6.6850914082 \times 10^{2}$ $-1.2301472524 \times 10^{1}$ $-2.7861521235 \times 10^{0}$	T^{*0} T^{*1} T^{*2} T^{*3}

Fig. 2.9 Power series expansion for the thermoelectric voltage of Type R thermocouples. Source: NBS Monograph 125.

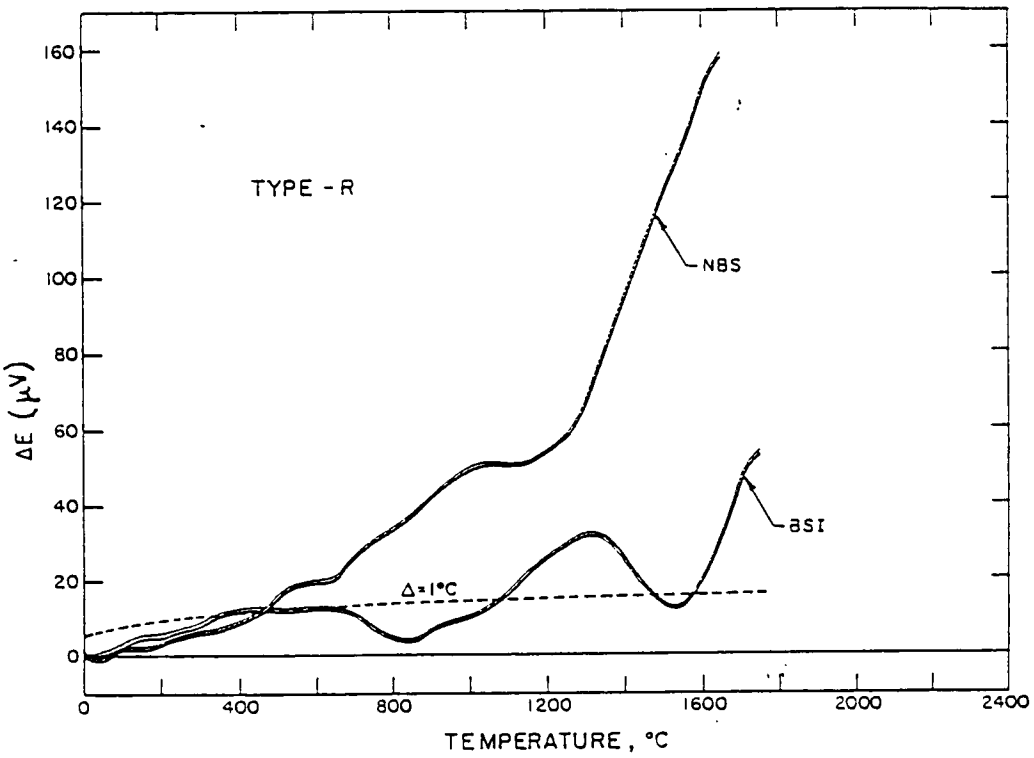


Fig.2.10 Comparison of difference in values for thermoelectric voltages given in NBS Monograph 125 and those given in BSI (BS.1826:1952), based on the values given in NBS Monograph 125.

published in National Bureau of Standards Circular 561 and British Standards Institute (B.S. 1826 : 1952) based on the values published in NBS Monograph 125. The width of the curve represents an uncertainty of 1 μV in the tabular values. Figure 2.11 shows a graph of thermoelectric voltage as a function of temperature for type R thermocouples. The circles indicate the various thermoelectric fixed points on the IPTS-68 definitions. Figure 2.12 shows the first derivative of the thermocouple voltage plotted against temperature, which is also the Seebeck coefficient for the thermocouple, and Figure 2.13 is a plot between the second derivative and temperature for type R thermocouples. From Figure 2.9 and Figure 2.13 it can be inferred that the temperature intervals for which the coefficients have been determined are directly related to the changes in slopes in $\frac{d^2V}{dT^2}$ versus T curve. It is necessary to divide the empirical function into the required number of intervals if there are drastic changes in slope in the function. If thermocouple emf versus temperature curve or any of its derivatives shows a drastic change in slope, such a change needs to be appropriately located and separate functions are needed on both sides of the location to describe the relationship. Furukawa [16] commented that for Pt - 13%Rh thermocouples, even though the thermoelectric voltage vs. temperature curve shows discontinuities, when plotted against resistivity of platinum the thermoelectric voltage does not show any discontinuity. This fact can be taken advantage of by using two different relationships, $dV/d\rho$ and $d\rho/dT$, so that a single function can be possibly used over the

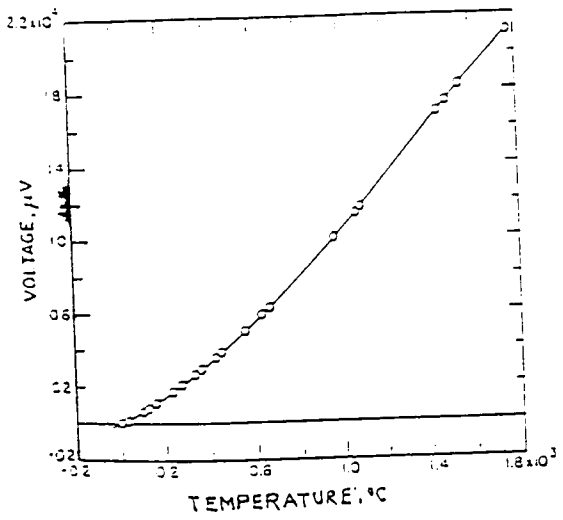


Fig.2.11 Thermoelectric voltages for Type R thermocouples. Source: NBS Monograph 125.

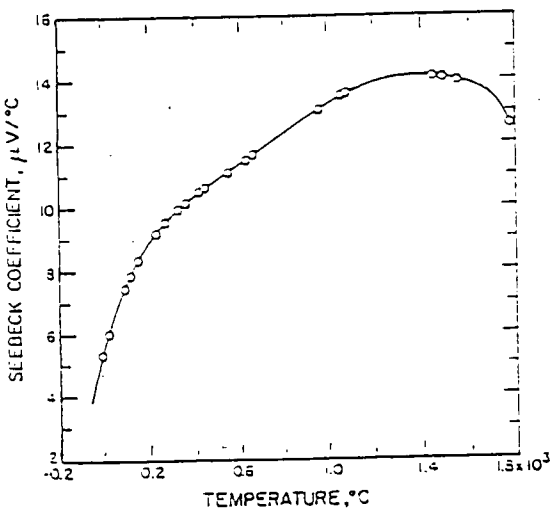


Fig.2.12 Seebeck coefficients for Type R thermocouples. Source: NBS Monograph 125.

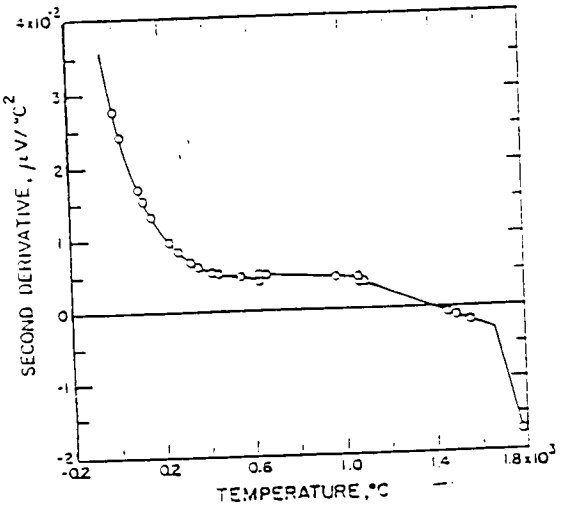


Fig.2.13 Second derivative of thermoelectric voltage for Type R thermocouples. Source: NBS Monograph 125.

whole temperature range if both V versus ρ and ρ versus T are free from discontinuities in that range.

Another important consideration while generating and selecting coefficients for thermocouples is the error range associated with the coefficients. Figure 2.14 shows the quadratic, cubic and quartic approximations of E = f(T) data. By comparing the error range for quadratic (second degree), cubic (third degree) and quartic (fourth degree) approximations it is concluded that the more the number of coefficients used the better is the fit and the narrower is the error range. Figure 2.14 shows how the error range is significantly reduced by increasing the number of coefficients from 3 to 4. Also, due to the nature of the curve, at higher temperatures the error range is narrower.

Using the quartic equations shown in Figure 2.14 for E = f(T), the generated data fit very closely (within < 0.1%) with those given in NBS Monograph 125 table. But for the inverse relationship, T = f(E), the coefficients provided in the Monograph 125 do not generate data which fit closely with those given in their table. In the worst case, temperature (°C) data generated in the range 0 - 1768 °C, by using the coefficients provided for the cubic equation, may differ as much as 43 °C from the tabulated data. This obviously cannot be accepted for accurate thermometry. Such big differences between the computed and tabulated data are due to the fact that large temperature intervals were used to fit the data. It is unlikely that the fit will be good over such large ranges (e.g., 0 to 1768°C or 400 to

Temperature Range (°C)	a_0		a_1		a_2		a_3		a_4		Error Range (μV)
	Argument	Exp.	Argument	Exp.	Argument	Exp.	Argument	Exp.	Argument	Exp.	Exact-Approx.
I. Quartic Equation											
- 50 to 900	. . . . .	. .	5.4295008	+0	1.1446885	-2	-1.1295306	-5	5.0020496	-9	-7 to 15
0 to 1100	. . . . .	. .	5.7622358	+0	9.2715271	-3	-7.1346883	-6	2.5877458	-9	-16 to 12
0 to 1400	. . . . .	. .	6.1429722	+0	7.1515857	-3	-5.7539447	-6	9.6963832	-10	-35 to 25
0 to 1650	. . . . .	. .	6.4615269	+0	5.7010917	-3	-1.3683292	-6	2.3636365	-10	-55 to 35
0 to 1768	. . . . .	. .	6.5962120	+0	5.1559203	-3	-1.2385309	-6	1.8827643	-11	-65 to 35
400 to 1100	-4.0674108	+2	8.7490294	+0	1.7115155	-3	7.5039035	-7	-3.0096240	-10	-.4 to .3
400 to 1400	-5.6047484	+2	9.6731111	+0	-2.6994046	-4	2.5536988	-6	-8.9155491	-10	-1.7 to 1.6
400 to 1650	-5.4505828	+2	9.5942872	+0	-1.2813352	-4	2.4468512	-6	-8.6286756	-10	-2.1 to 1.8
1050 to 1400	1.6618159	+3	2.3048526	+0	8.7635426	-3	-2.3016819	-6	7.4284923	-11	-.05 to .05
1050 to 1650	1.5132838	+3	2.7958847	+0	8.1571403	-3	-1.9701159	-6	6.5568964	-12	-.05 to .05
1400 to 1550	2.4008703	+3	4.1606579	-1	1.0549178	-2	-3.0383621	-6	1.8540516	-10	-.05 to .05
1400 to 1650	1.5787334	+3	2.6321144	+0	8.3100314	-3	-2.0332036	-6	1.6260416	-11	-.05 to .05
1400 to 1768	-7.1904948	+4	1.9442383	+2	-1.7913090	-1	7.9264764	-5	-1.3187245	-8	-1.0 to 1.3
1666 to 1768	8.8532076	+4	-1.5014129	+2	9.5376167	-2	-1.6644901	-5	-8.3062870	-10	-.05 to .05
II. Cubic Equation											
- 50 to 900	. . . . .	. .	6.0632060	+0	7.0729964	-3	-2.7461531	-6	. . . . .	. .	-30 to 60
0 to 1100	. . . . .	. .	6.4312595	+0	5.7063515	-3	-1.6171323	-6	. . . . .	. .	-51 to 31
0 to 1400	. . . . .	. .	6.6602336	+0	4.9866412	-3	-1.1222144	-6	. . . . .	. .	-70 to 35
0 to 1650	. . . . .	. .	6.6681844	+0	4.9675968	-3	-1.1120895	-6	. . . . .	. .	-70 to 40
0 to 1768	. . . . .	. .	6.6164788	+0	5.0888113	-3	-1.1739757	-6	. . . . .	. .	-65 to 40
400 to 1100	-3.3173012	+2	8.2968261	+0	2.6896906	-3	-1.5226719	-7	. . . . .	. .	-.6 to .8
400 to 1400	-1.4900615	+2	7.4755900	+0	3.8372620	-3	-6.5522906	-7	. . . . .	. .	-7 to 10
400 to 1650	7.0511331	+1	6.5679703	+0	4.9717004	-3	-1.0905912	-6	. . . . .	. .	-17 to 19
1050 to 1400	1.4980000	+3	2.8453533	+0	8.0970910	-3	-1.9377143	-6	. . . . .	. .	-.05 to .05
1050 to 1650	1.4925741	+3	2.8588183	+0	8.0860331	-3	-1.9347086	-6	. . . . .	. .	-.05 to .05
1400 to 1550	1.5265340	+3	2.7908769	+0	8.1313270	-3	-1.9447717	-6	. . . . .	. .	-.05 to .05
1400 to1650	1.4915384	+3	2.8617020	+0	8.0835951	-3	-1.9340590	-6	. . . . .	. .	-.05 to .05
1400 to 1768	9.8946473	+3	-1.3614385	+1	1.8829682	-2	-4.2655016	-6	. . . . .	. .	-2 to 4
1666 to 1768	9.5736985	+4	-1.6693822	+2	1.1005861	-1	-2.2348111	-5	. . . . .	. .	-.05 to .05
Variable reference junction correction											
0 to 50	. . . . .	. .	5.2891411	+0	1.3844426	-2	-2.0889531	-5	. . . . .	. .	-0.01 to +0.01
III. Quadratic Equation											
- 50 to 900	. . . . .	. .	7.0091249	+0	3.6680622	-3	. . . . .	. .	. . . . .	. .	-100 to 120
0 to 1100	. . . . .	. .	7.2706944	+0	3.2499597	-3	. . . . .	. .	. . . . .	. .	-130 to 60
0 to 1400	. . . . .	. .	7.6038268	+0	2.8172081	-3	. . . . .	. .	. . . . .	. .	-160 to 90
0 to 1650	. . . . .	. .	7.9670282	+0	2.4339570	-3	. . . . .	. .	. . . . .	. .	-240 to 170
0 to 1768	. . . . .	. .	8.1907195	+0	2.2229871	-3	. . . . .	. .	. . . . .	. .	-330 to 230
400 to 1100	-3.8627420	+2	8.5408889	+0	2.3470378	-3	. . . . .	. .	. . . . .	. .	-3 to 2
400 to 1400	-5.2441977	+2	8.9543660	+0	2.0679233	-3	. . . . .	. .	. . . . .	. .	-31 to 25
400 to 1650	-8.0082944	+2	9.7098356	+0	1.6179512	-3	. . . . .	. .	. . . . .	. .	-95 to 75
1050 to 1400	-2.0139987	+3	1.1528256	+1	9.7566289	-4	. . . . .	. .	. . . . .	. .	-3.2 to 3.2
1050 to 1650	-3.1042936	+3	1.3315918	+1	2.5046320	-4	. . . . .	. .	. . . . .	. .	-16 to 16
1400 to 1550	-4.7053323	+3	1.5479630	+1	-4.7535391	-4	. . . . .	. .	. . . . .	. .	-.3 to .3
1400 to 1650	-5.3395443	+3	1.6339581	+1	-7.6649197	-4	. . . . .	. .	. . . . .	. .	-1.1 to 1.2
1400 to 1768	-6.9060256	+3	1.9401469	+1	-1.4430259	-3	. . . . .	. .	. . . . .	. .	-10 to 9
1666 to 1768	-1.7351473	+4	3.0715022	+1	-5.0686458	-3	. . . . .	. .	. . . . .	. .	-.9 to 1.0
Variable reference junction correction											
0 to 50	. . . . .	. .	5.3139863	+0	1.2244376	-2	. . . . .	. .	. . . . .	. .	-0.1 to +0.2

Fig. 2.14 Quadratic, cubic and quartic approximations to the E=f(T) data, where E is in microvolts and T is in degrees Celsius. Source: NBS Monograph 125.

1650°C). Also, using only five coefficients in the equation partially broadened the error range. Breaking up of the temperature range into several small intervals and using more coefficients would have resulted in much better fits over the temperature range of interest.

Since the coefficients given in NBS Monograph 125, Table A3.2.3 yielded temperature data which were inaccurate to the extent that they could not be used for our purpose, it was decided that we would generate our own data in the temperature range 100-400 °C. T(°C) versus E(mV) data from Table A3.2.1 of NBS Monograph 125 were entered into a fitting program LEASTSQR.BAS with intervals of 10 °C in the interval 0 to 200 °C, 200 to 400 °C and 100 to 400 °C, and three sets of coefficients were recorded and the temperature computed with the help of these coefficients compared with NBS Monograph 125 T=f(E) data. It was found that the temperature data computed with the coefficients in the range 100 to 400 °C matched closely (within 0.01%) with the NBS Monograph 125 table data. Thus, the coefficients generated by us were used from 100 to 400 °C. The temperature data generated between 100 and 400 °C when plotted along with the temperature data generated in the range 400 to 1100 °C, against time, result in a smooth curve all the way from 100 to 1100 °C. Figure 2.15 shows a typical plot of temperature (°C) versus time (second). Note that there is no discontinuity in the T=f(t) curve at 400 °C even though different sets of T=f(E) coefficients were used above and below 400 °C. The T=f(E) coefficients used by us in the range 100 - 400 °C and 400 - 1100 °C are tabulated in Table 2.2.

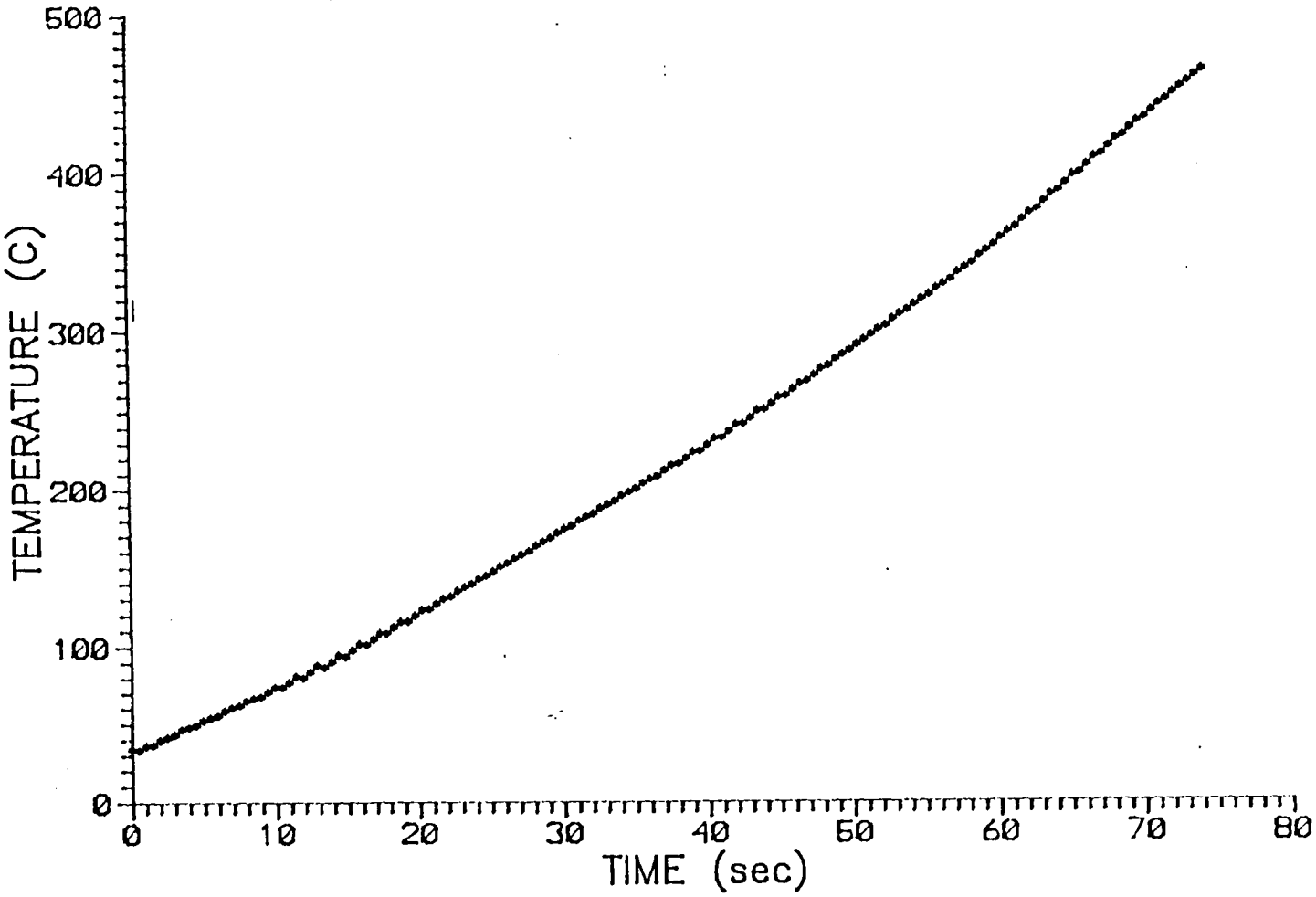


Fig.2.15 Temperature versus time data. Temperature data has been generated from thermocouple emf data by using different sets of T=f(E) coefficients above and below 400 °C.

Table 2.1 Comparison of temperature (°C) data generated using the NBS Monograph 125 coefficients and the coefficients generated in the UTK laboratory.

EMF (μV)	T(UTK) (°C)	T(NBS) (°C)	T(UTK) - T(NBS) (°C)
1000	144.99	139.86	5.13
1500	203.64	201.67	1.97
2000	258.22	259.15	-0.93
2500	310.19	312.98	-2.79
3000	360.35	363.85	-3.50

Table 2.2 T=f(E) coefficients used to convert thermocouple emf (mV) to temperature (°C). The conversion equation is of the form $T = a_0+a_1E+a_2E^2+a_3E^3+a_4E^4$.

Temperature range 100 - 400°C	
a_0	0.6414181×10^{1}
a_1	1.586447×10^{-1}
a_2	-2.464001×10^{-5}
a_3	5.013749×10^{-9}
a_4	$-4.396420 \times 10^{-13}$
Temperature range 400 - 1100°C	
a_0	4.5509556×10^{1}
a_1	1.1284875×10^{-1}
a_2	$-2.8603978 \times 10^{-6}$
a_3	$8.5173702 \times 10^{-11}$
a_4	$-1.1440038 \times 10^{-15}$

PART II: CHAPTER 3
SPECIMEN, SPECIMEN HOLDER AND PARTS

SPECIMEN

A machined nickel rod, 28 cm in length, has been used as the specimen. The purity of the nickel is unknown. Nickel wire manufactured by Alfa Products [17], with a diameter of 0.25 mm, was used as the voltage taps. The voltage taps were welded to the specimen with a separation of 4.00 cm, which marks the effective length of the specimen. The specimen has an average diameter of 0.2395 cm (0.0943 inch). The diameter range is discussed in Part IV, Chapter I. The individual measurements of the diameter are tabulated in Table 2.3.

Table 2.3 Individual measurements of specimen diameter.

Measurement Number	Measured Diameter (inch)
1.	0.0972
2.	0.0963
3.	0.0952
4.	0.0943
5.	0.0924
6.	0.0922
7.	0.0922
8.	0.0930
9.	0.0939
10.	0.0964

A thermocouple was spot welded near the center of the specimen,

between the voltage taps. The platinum thermoelement was first spot welded to the specimen using the spot welder. Platinum-13% rhodium electrode was then carefully welded on top of the already welded platinum electrode. This was done to minimize the axial misalignment between the two electrodes and to minimize an unwanted voltage pick-up due to the applied current. A low power optical microscope was used for viewing while welding the thermoelements to the specimen. Figure 2.16c and d shows photographs of the specimen being held vertically in the specimen holder.

SPECIMEN HOLDER DESCRIPTION

The specimen holder for the pulse calorimeter is designed to fit inside the cylindrical vacuum furnace chamber of the pulse calorimeter, which is a 10.2 cm inside diameter and 101.6 cm long nickel tube. The holder consists of several components, some of which are described in the following paragraphs.

(1) The Teflon™ top flange is 12.1 cm in diameter and 2.9 cm thick with a recessed shoulder that is 9.5 cm diameter and 1.6 cm thick. The shoulder positions the holder axially in the pulse calorimeter chamber. Four cylindrical cut-outs have been removed from the top flange to provide openings for evacuation of the pulse calorimeter chamber and for chamber instrumentation leads. The top flange contains four holes for the current leads to the specimen lower

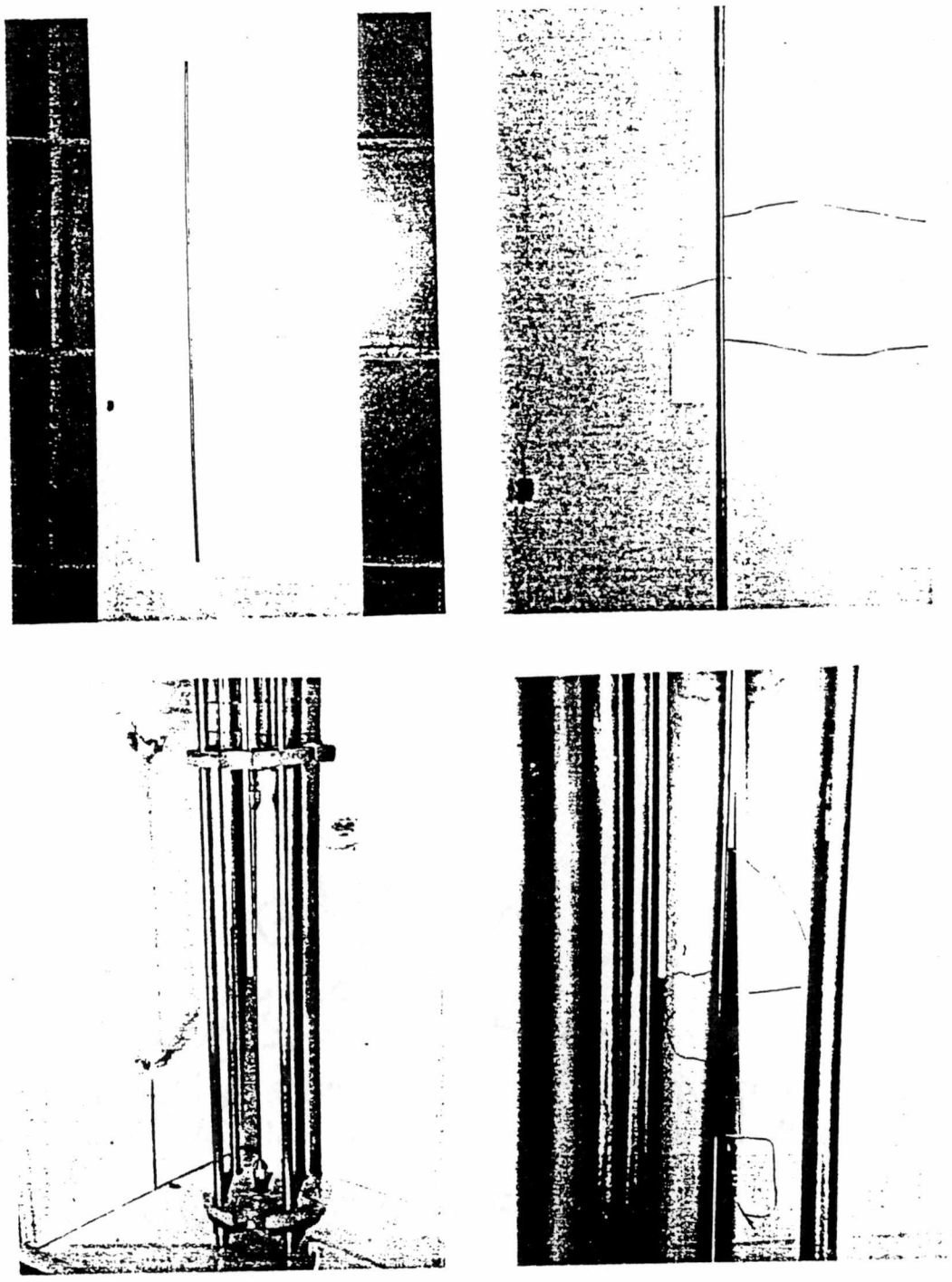


Fig. 2.16 (a) Thermocouple wires and voltage taps welded to the specimen. (b) Close-up of (a). (c) Specimen being held vertically in the specimen holder. (d) Close-up of (c).

electrode, a central hole for the current lead to the specimen upper electrode and eight small holes for specimen instrumentation leads. The planar design of the Teflon™ top flange is repeated on the nickel radiator shields, the three lavite mechanical spacers and the lower specimen electrode.

(2) Four current leads for the specimen lower electrode are made from 1 cm outside diameter nickel tube with end extensions to produce a total length of 101.6 cm.

DESCRIPTION OF CURRENT LEADS AND TERMINALS

There are two brass screw terminals in the instrument rack to facilitate the connections between the power supply and the calorimeter terminals. The power supply is connected to the screw terminals with 1.2 cm outside diameter Laribee AWG EP Welding Cable. One of the two connecting wires connects directly from the power supply to the screw terminal while the other is connected through the standard resistor.

The instrumentation rack screw terminals are connected to the current lead terminals of the calorimeter via 1.6 cm diameter insulated copper wires. These wires are not connected directly to the instrumentation rack screw terminals but are first connected to 2.2 cm wide flexible strip which are then connected to the screw terminals. This has been done to facilitate connections.

The two current lead terminals are copper rods 7.9 cm long and

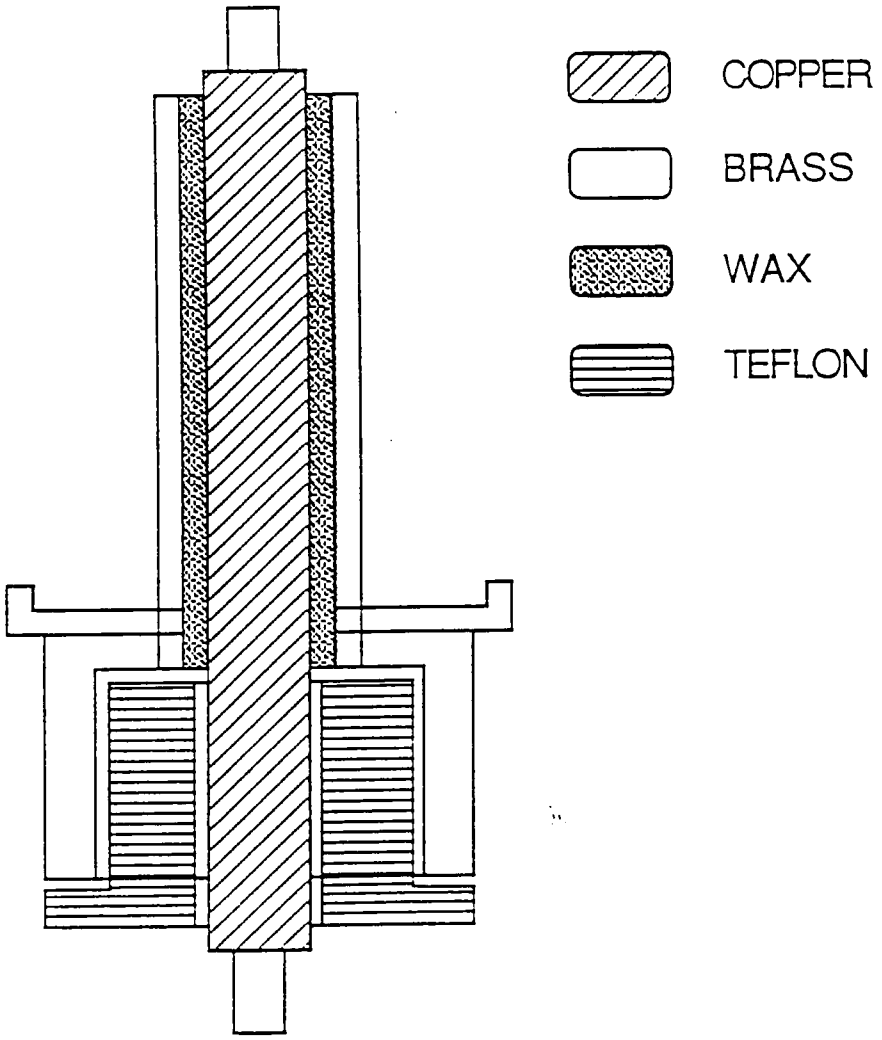


Fig. 2.17 Schematic diagram of the cross-section of a current lead terminal showing the different materials with which the parts were made.

1.0 cm diameter, and each is mounted in a brass cylinder to create a vacuum seal. Figure 2.17 shows a schematic diagram of such a terminal. It is a vacuum feed-through terminal which connects the current leads from inside the vacuum chamber to those outside. The brass cylinder is 5.3 cm long and has two cylindrical sections. The lower section is 2.5 cm outside diameter with a 1.8 cm inside diameter and has a 2.5 cm deep cavity to accommodate a Teflon™ alignment sleeve for the copper electrode and the brass section. The upper section is a sleeve 1.8 cm in outside diameter and 1.5 cm in inside diameter. This contains the wax sealant to the copper electrode. The upper and lower sections have an O-ring space where they join and thus seal the brass ring to the flange on the main plate.

Part III, Chapter 5 contains further discussion of the electrical circuit for the acquisition system.

PART III
ELEMENTS OF ACQUISITION SYSTEM DESIGN

PART III: CHAPTER 1
PROGRAMMABLE POWER SUPPLY

POWER REQUIREMENT OF THE SPECIMEN

The heat capacity of the specimen determines how much power is needed by the specimen to heat it to a certain temperature. If it is required to raise the temperature of a specimen to a certain degree within a given time, the current that would be required can be estimated from the energy balance equation if the specific heat of the specimen substance in that range is known. If it is required to go to a higher temperature in the same time span, then a higher value of the current and/or voltage would be required. If the rate of heating of the specimen is not critical, then the same temperature can be achieved over a longer length of time, i.e., by increasing the pulse duration. In both cases the energy supplied to the specimen (area under the power vs. time curve) is the same, assuming no heat losses. Thus a higher temperature can be obtained either by increasing the pulse current or the pulse time. But in certain cases the choice may be limited. Certain experiments require the specimen to heat at a high rate to suppress the kinetics of certain transformations taking place in the specimen. Then a higher current becomes a must and the power supply should have the current and voltage capacity to supply the required power. One method of bypassing this problem is to change the specimen dimensions so that

the resistance of the specimen is increased (e.g., use thinner specimen). But then this has a physical limitation to it. Reducing the dimensions of the specimen may increase the error in measurement of the dimensions.

To form an idea about the ideal system resistance to obtain maximum power from the power supply, the electrical resistance of the specimen should be matched with that of the power supply. Power supply can produce 90 Amps at 6 Volts.

From Ohm's law, R=E/I

Circuit resistance = R(circuit) = 6/90 ohms

R(circuit) = 0.067 ohms.

The ideal matching resistance to obtain maximum power from the power supply is 0.067 ohms.

Since Power(P) = E I

P = E I = 6 x 90 = 540 watts.

This is the maximum power that can be obtained from the power supply.

THE POWER SUPPLY

The specimen power source was a high current range, high gain voltage regulated power supply, with a maximum output rating of 90 Amperes and 6 Volts. It is an operational power supply (OPS) of Group VIII type manufactured by Kepco Incorporated [18]. Group VIII type OPS models are designed for applications requiring a wide

band, programmable source. They are unipolar devices which may be connected to the circuit as d.c. amplifiers to operate either as a constant voltage source or a constant current source, or to operate as a controlling device by using feedback.

In this project, the programmable feature of the power supply was used. The output of the power supply can be remotely controlled by a d.c. voltage signal. The power supply can also be controlled locally by using the knobs on the chassis. This option was not used much except in the initial stages of the project when the digital-to-analog connections between the acquisition board (inside the computer) and the signal input terminal of the power supply were not available.

Pulse calorimetry often requires using such small pulsing time that manual opening or closing of a switch or changing the current on the potentiometer dial may not be feasible. So a programmable power supply is a requirement. Since everything in the acquisition process is controlled from the computer by a program called CALPUL.FOR, the output of the power supply was controlled by the same acquisition program. There was no external switching device to apply or shut off the current to the specimen. Instead, a set of carefully written communication codes, in the CALPUL.FOR program, could trigger the power supply in a way such that the desired current pulse characteristics could be obtained. The program CALPUL.FOR set the current to zero before the experiment started and also switched it back to zero before exiting the program. Using

the remote mode enabled high switching speed and eliminated operator error in generating the current-time pattern. Also a greater stability in the current could be obtained in the remote control mode than in the manual control mode. This is also stated in the specification of the power supply which says that approximately 2% current stabilization can be obtained in the manual (front panel) control mode while a 0.1% stabilization can be obtained in the remote controlled mode. Thus the remote mode provides much better control over the current.

OPERATIONAL FEATURES OF THE POWER SUPPLY

For fast calorimetric experiments such as this, which require transient signal measurement and adjustment, a standard power supply is not well suited because of its slow response time. The standard power supply is generally optimized as a voltage source and its bandwidth is deliberately restricted mainly by means of a large output capacitor. It approaches the characteristics of an ideal voltage source - low output ripple, low source resistance and a large storage capacity.

An Operational Power Supply, such as the one used in this project, by contrast, can exhibit the characteristics deliberately suppressed in a standard power supply, most important of which are wide bandwidth and fast output response. The wide bandwidth characteristics can be generated by removing the large capacitor at

the output and shaping the open-loop response by means of lag networks. The drawbacks are higher output ripple, higher output impedance and restrictions on reactive loading.

In this project a Kepco Power Supply was used as a constant current source. It can be used as a programmable d.c. source which can provide high speed voltage or current level changes.

Operational Power Supply Operation

There are no established parameters to describe the standards of Operational Power Supplies. However, the following parameters are the generally accepted ones:

1) D.C. Offset Variations
2) Open-Loop Gain
3) Maximum Slewing Rate
4) Maximum Sine Wave Frequency
5) Programming Time Constant

Some of these factors are discussed subsequently in this chapter.

The heart of an operational amplifier consists of a Comparison Amplifier, a Reference Source and a Power Amplifier, as shown in Figure 3.1. Small variations in the d.c. output are caused by certain external factors (e.g., temperature, load, a.c. source variations). They cause small changes in the input of the comparison amplifier. These variations, called offset variations, are transferred to the output as an amplified signal, the amplification depending upon the gain of the

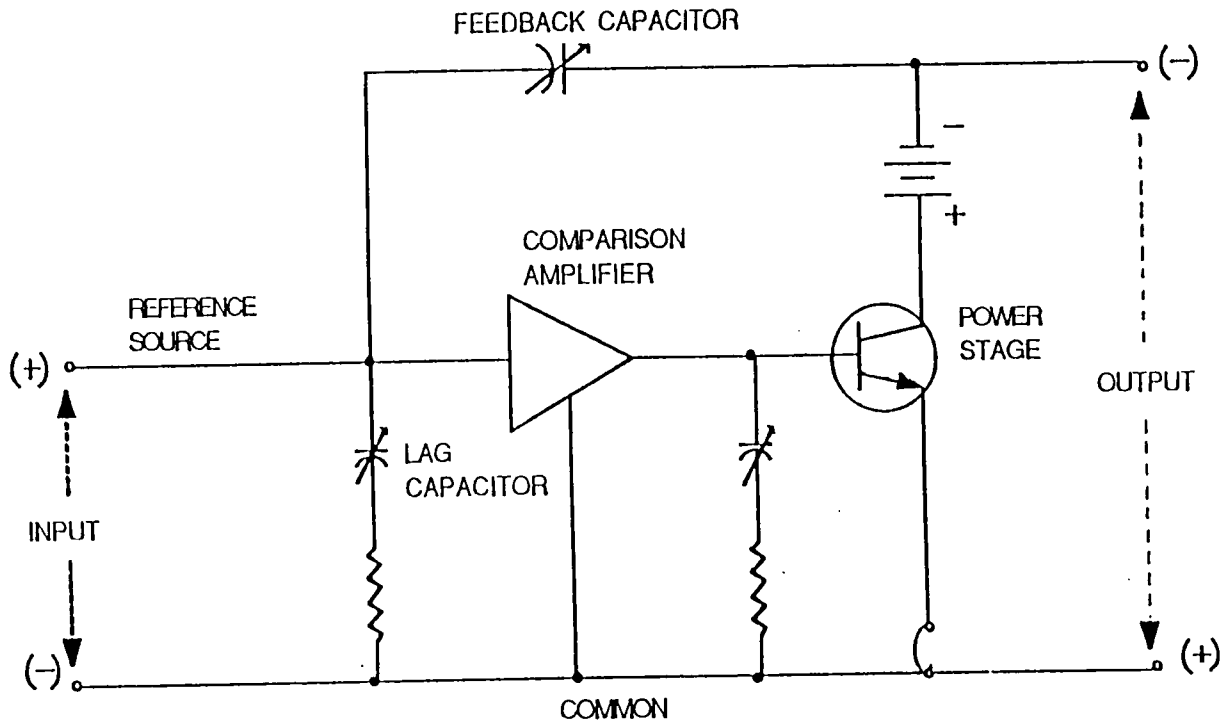


Fig. 3.1 Simplified circuit diagram of an Operational Power Supply showing the reference source, comparison amplifier and power stage.

power transistor in the output stage (refer to Figure 3.1).

Figure 3.2 shows a further simplified schematic of the Operational Power Supply, where the power stage and the comparator amplifier have been represented by the triangle. This figure represents an ideal operational power supply for which the following equation is valid

$$\frac{E_i}{R_i} = \frac{E_o}{R_f}$$

where,

E_i = Input voltage

R_i = Input resistance

E_o = Output voltage

R_f = Feedback resistance

The equation assumes that the null junction voltage and current are both zero. In reality, however, non-idealities do exist at the null junction or the input of the amplifier. These errors are in the form of an offset voltage (E_{io}) and offset current (I_{io}). Figure 3.3 shows the modified version of the amplifier model with the two errors taken into account.

Dynamic Specifications

The dynamic characteristics of the operational power supply can be specified by two parameters - The Programming Time Constant and the Maximum Slewing Rate. The first describes the output response in the linear region of the Emf versus Time curve

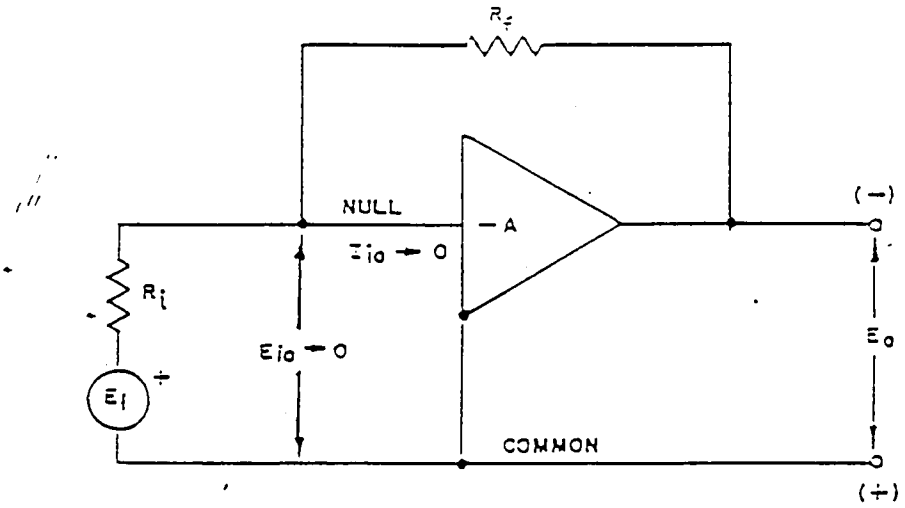


Fig 3.2 Simplified diagram of operational amplifier without input offset sources.

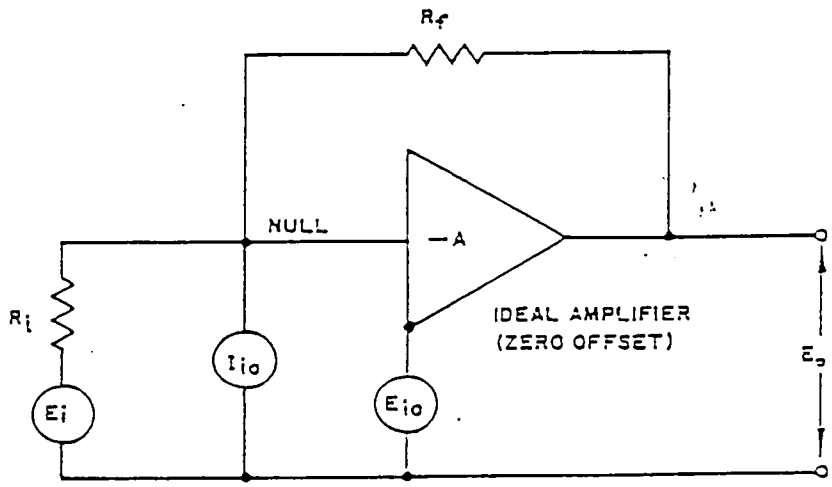


Fig. 3.3 Simplified diagram of operational amplifier with input offset sources.

and the other represents the output response to overridden or forced input signals in the non-linear region of the Emf versus Time curve.

a) Programming time constant

Operational Power Supplies have a linear operating region. For Programmable Operational Power Supplies, such as the one we are using, the speed at which they can be programmed determines the speed at which their output voltage can be varied. If it is desired to apply a square wave type current to the calorimeter specimen, a high programming speed is desired. The programming speed depends upon the value of the feedback components -- the feedback capacitor (C_f), and the feedback resistance (R_f) (refer to Figures 3.1 and 3.2). The programming time constant (τ) is defined by the relation $\tau = C_f \times R_f$. It is the time required to raise the output from 0 to 63% of the exponential output voltage in response to an input step function (refer to Figure 3.4).

b) Maximum slewing rate

Maximum slewing rate is defined as the fastest time rate of change of which the operational power supply is capable. It is a measure of the output response to an input step function with the output fully loaded. If an increase in the driving signal amplitude does not produce a change in the slope of the output response (E_o/τ) (refer to Figure 3.5), then the operational power supply is responding with the maximum slewing rate. The ratio E_o/τ can be measured

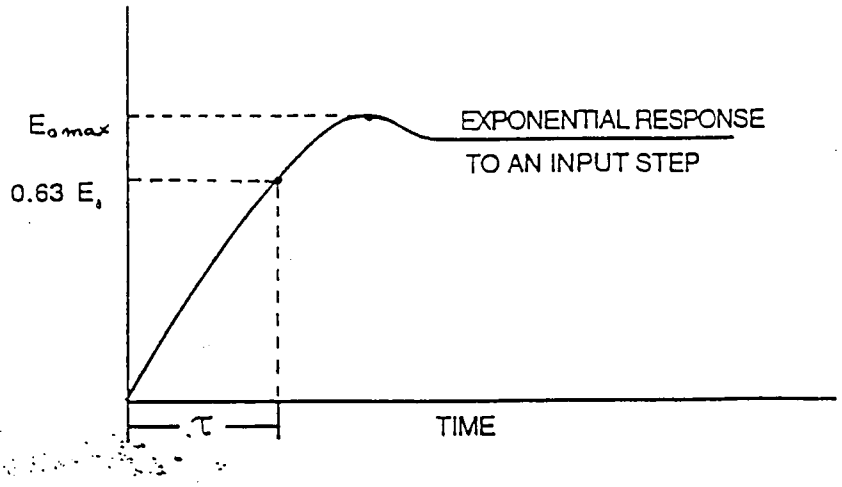


Fig. 3.4 Definition of Programming Time Constant.

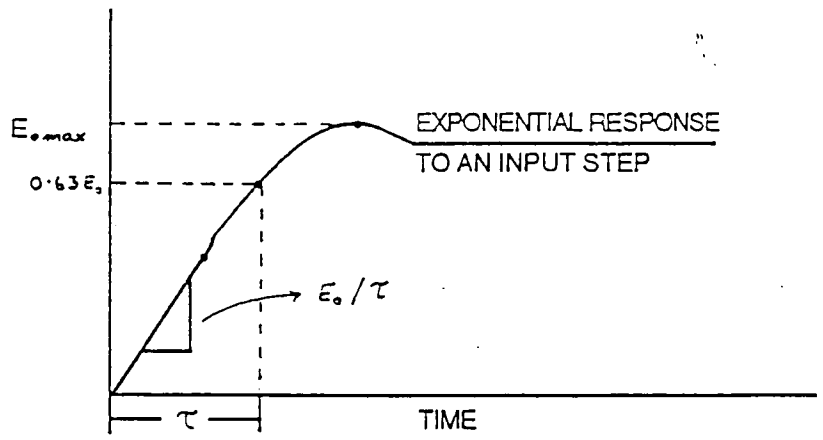


Fig. 3.5 Definition of Maximum Slewing Rate

microseconds.

Though a low programming time constant and a high maximum slewing rate are desirable, their non-availability is not a serious limitation for this kind of work. A low programming time constant would mean that it takes more time for the power supply to attain the specified stepped-up output signal. The instantaneous output signal usually fluctuates about the specified output signal for some time before attaining a steady state. The time required to attain steady-state output is slewing rate dependent. This time is not very critical as long as we have the instantaneous voltage and current data to compute the power to the specimen. Still, for the reference of the reader, the time response of the output current of the Kepco Group VIII Power Supply in presented in Figure 3.6 as actual recorded data. The figure shows the current dependence on time for the input signal stepped up from 0 to 40 amperes and from 0 to 80 amperes. Data were recorded at the speed of a data point per 55 millisecond. The time unit, Time Tag# is equal to 55 milliseconds for this data set. Curves for 40 amps and 80 amps both show fluctuations about a mean which is the set current, before attaining stability. Magnification of the fluctuating region reveals that the fluctuation continued for at least 550 milliseconds for both cases. The magnitude of fluctuation, however, was more for the higher current.

At 80 amps the maximum slewing rate measured for the used Kepco power supply is 140 milliseconds and the corresponding maximum slewing rate is 0.36 amps/millisecond.

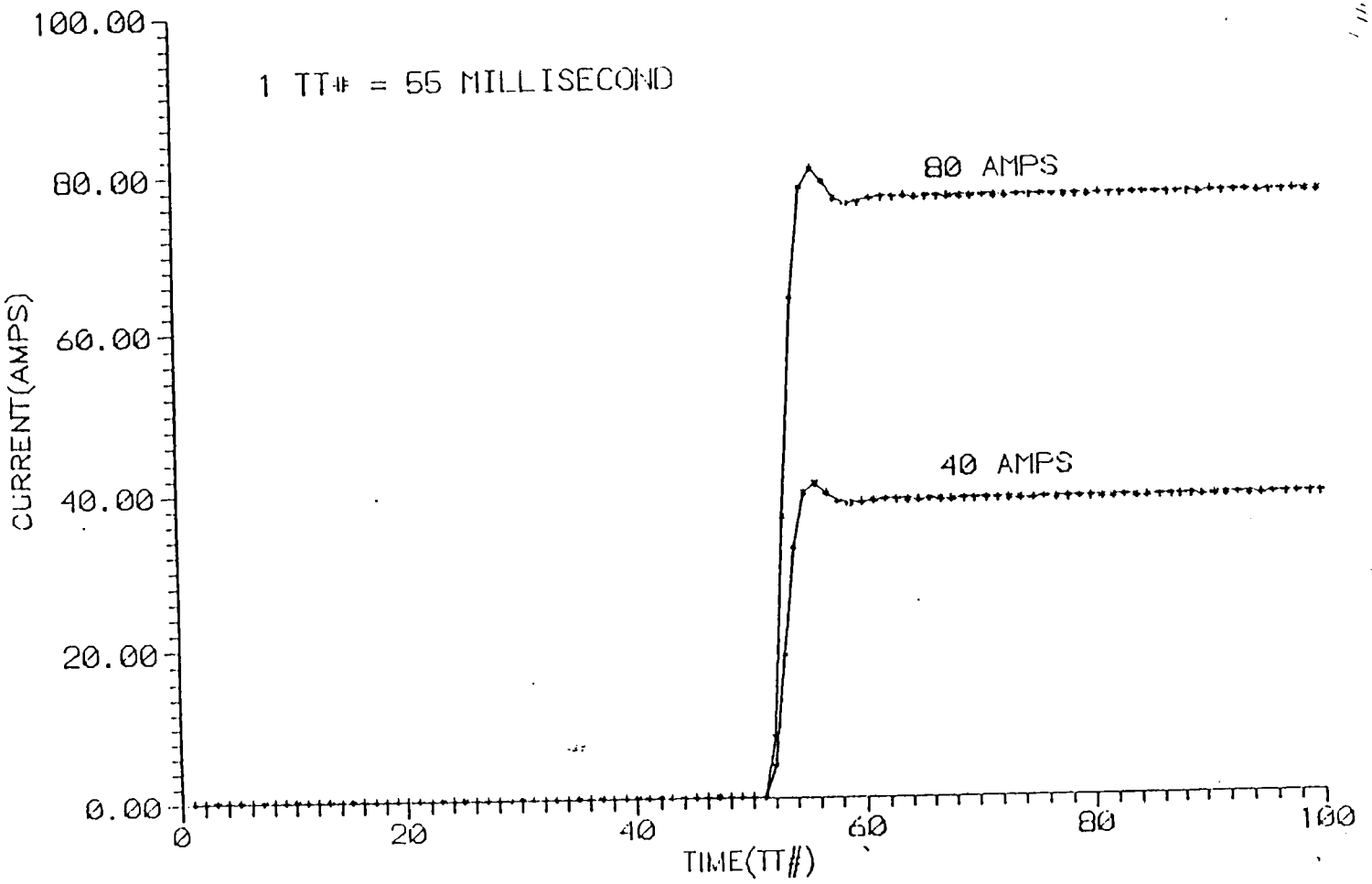


Fig. 3.6 Figure showing the front end of the current pulse to the specimen for 40 amperes and 80 amperes. The initial fluctuation about the mean set current is evident.

Simplified Circuit Diagram

Figure 3.7 shows the simplified circuit diagram of the Kepco Group VIII Power Supply. The two transformers in the input section of the power supply, the power transformer and the auxiliary transformer, convert the a.c. source voltage to the levels required to produce the d.c. operating potentials. The main d.c. supply is connected in series with the power output stage, consisting mainly of the power transistor and the current sensing resistor which finally leads to the output terminals. The output is controlled by varying the impedance of the power output stage. The driver stage controls the impedance of the output. The driver stage, in turn, receives its drive from either the voltage mode or the current mode amplifiers.

The Current Limit Circuit

During operation, the voltage drop across the current sensing resistor (R_S) is thus continuously compared to the reference voltage, set by the current limit control. As long as the voltage developed across the current sensing resistor is less than the preset voltage reference level, the current limit amplifier will be biased to its inactive state and will not affect the output. But if the output current increases the voltage drop across R_S it will overcome the reference

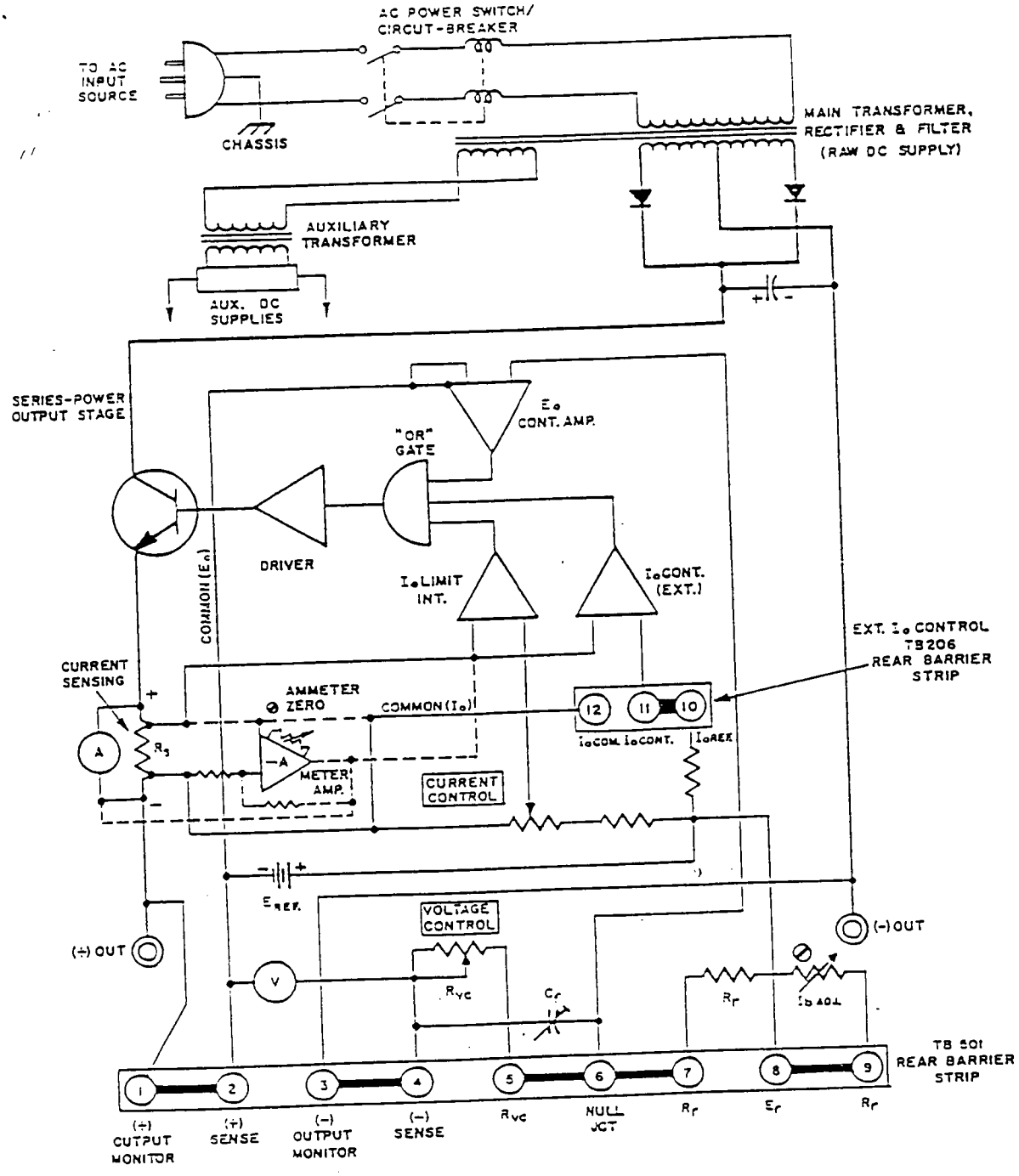


Fig. 3.7 Simplified circuit diagram of Kepco Group VIII Power Supply.

level set up by the current limit control and the power supply will be transferred into the current limit mode thus maintaining a constant current.

PART III: CHAPTER 2
SIGNAL AMPLIFICATION AND PRECISION RECORDING

DESIGN CONSIDERATIONS OF RECORDING SYSTEM

For accurate calorimetry work the experimenter should have a way of judging the reliability of the specific measuring instruments he deals with and the precision with which they measure or record incoming data. So the design considerations of the calorimeter and its acquisition system should include :

1) Methods of eliminating errors in incoming signals.
2) The sensitivity of the instruments.
3) Eliminating random noise.
4) Eliminating long term noise.
5) Improving linearity of instruments and calorimeter.
6) Improving repeatability and random uncertainty.
7) Improving accuracy by decreasing total error.

Some of these factors necessitate the amplification of the incoming low voltage electrical signals to generate an accurate output signal with a high signal-to-noise ratio. Since the characteristic of the amplified signal is related to these factors, they will be briefly discussed here.

i) **Errors in incoming signals** - There can be multiple

sources of such errors. The calorimeter designer should be able to correctly detect the source of such errors and adopt measures to eliminate them. One frequent cause of instability in the input signal in our apparatus was inadequate grounding of the power supply. After a series of observations it was found that grounding the thermocouple at the hot junction improved the stability of the signal. An unregulated power supply is another example of instability of signals. In the course of our work it was found that much of the fluctuation in the recorded temperature signal was due to the interference of external electromagnetic fields with the signal-carrying thermocouple wires. Effective shielding of the thermocouple wires minimized this disturbance to an acceptable limit (e.g. <1% fluctuation).

ii) **Sensitivity** - Sensitivity can be defined in several ways depending upon the sense in which it is used. A general definition of sensitivity is the ratio of the change in effect to the change in cause of an event. For example, if a change in temperature, ΔT, of a thermocouple generates a thermal emf change, ΔE, then $\Delta E/\Delta T$ is a measure of the sensitivity of the thermocouple. The most common way of increasing the sensitivity of the signals is to amplify them. Amplification causes the signal to increase and thus a higher signal-to-noise ratio is obtained. In the current project three separate Preston amplifiers were used and the current, voltage and thermocouple emf signals were amplified through them. The amplification factor (gain) used for each channel is given in Table 3.1.

Table 3.1 Gain used for the three A/D channels.

Channel Number	Signal	Gain
0	Current	10
1	Voltage	10
2	Thermocouple emf	1000

iii) **Random noise** - The random fluctuations of the output signal are termed as noise. Unless otherwise mentioned, random noise refers to short-time noise. It is the random changes in the recorded signal. Noise is a source of significant inaccuracy for the incoming signal. Amplification of the input signal can sufficiently decrease the effect of noise in the output signal. Figure 3.8a shows the waveform of a typical random noise, such as may be generated in an amplifier. A typical graph of noise would show several maxima and minima in a span of a few seconds. Waveform 2, in Figure 3.8b, is the kind which may be applied to the input of an amplifier. This will give an output signal similar to Waveform 3 in Figure 3.8b. When random noise as shown in Figure 3.8a is superimposed over Waveform 2 and Waveform 3 the resulting curves are like what is shown in Figure 3.8c and Figure 3.8d, respectively. Comparison of Figures 3.8c and 3.8d shows that the amplification of the incoming signals greatly increases the signal-to-noise ratio and significantly increases the accuracy of the signals. It is most convenient to express noise in the units of the output signal (e.g., ± 1°C for the temperature

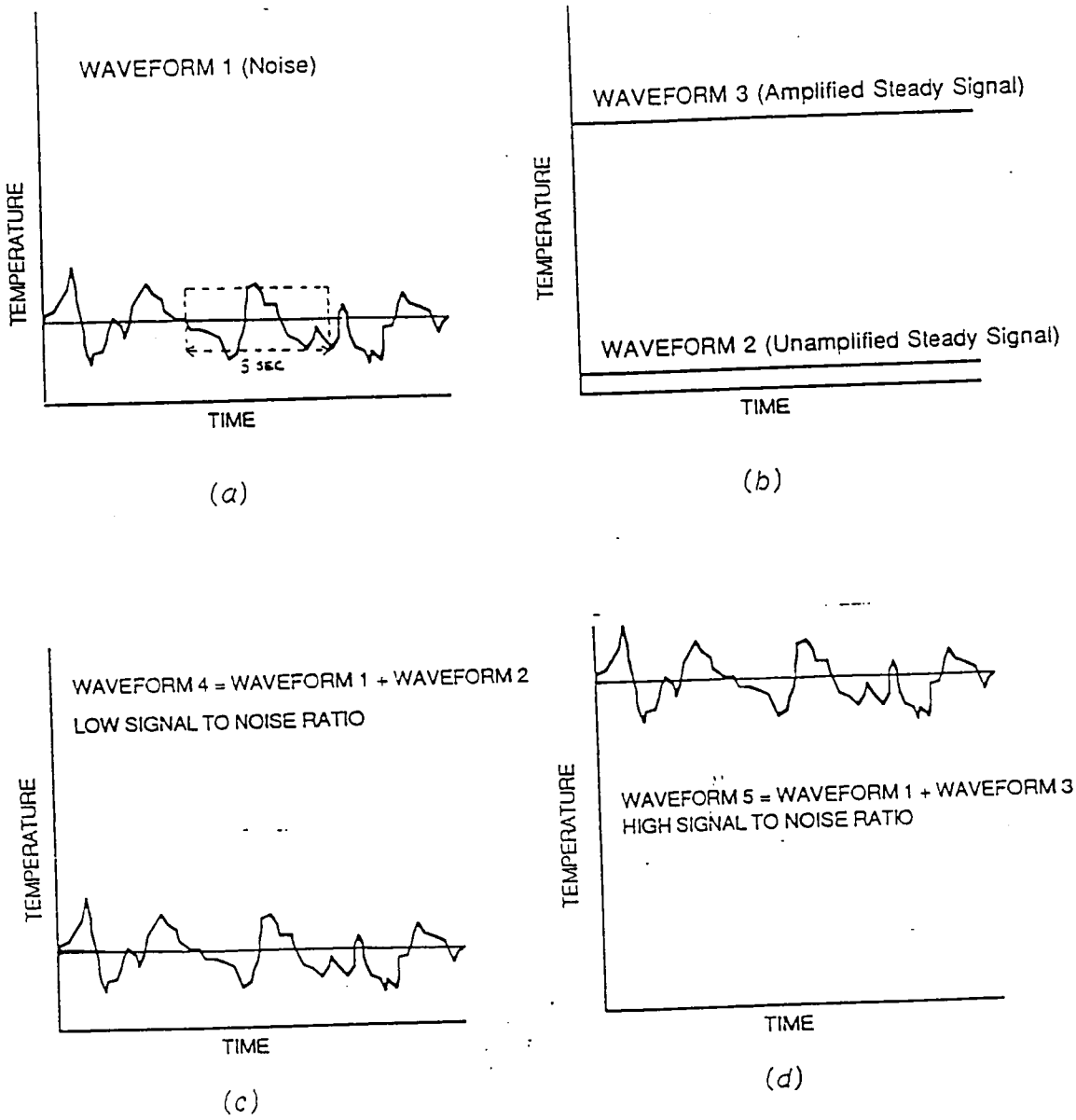


Fig. 3.8 Waveform for (a) typical random noise, (b) unamplified and amplified steady signal, (c) noise superimposed over unamplified steady signal, and (d) noise superimposed over amplified steady signal.

channel). Noise seriously limits the resolution of the data set obtained. Noise can originate from the measured quantity itself (e.g., temperature) or from the various devices that the signal has to pass through (e.g., thermocouple, amplifiers, power supply, electromagnetic interference). An acquisition system with minimum noise level greatly improves the quality of the experiment.

iv) **Long-term noise** - This refers to the overall fluctuations of the baseline over a longer time interval as opposed to the short-time fluctuations about the baseline, in the case of random noise. It affects the repeatability of the baseline over a number of repeated measurements of similar nature. The baseline drift during measurement also limits the resolution of the calorimeter. Long-term noise can be more troublesome than random noise, as the latter can be easily eliminated by statistically fitting data and deriving a running average [19]. Long term noise is not a problem in pulse calorimetry since the experiment is over in a very short time.

v) **Linearity** - In general, linearity is the proportionality between two quantities. When two quantities are directly proportional to each other they can be said to be linear. In pulse calorimetry, linearity is desired between the three input quantities of interest (current, voltage and temperature) and their respective output signals. In practice it is almost impossible to achieve linearity over a full scale of an instrument. A function called linearity error measures the maximum deviation of the actual functional relationship from the ideal (straight) line. A good linearity also

indicates that the amplification and recording device are not sensitive to the specimen parameters (e.g. size, mass, thermal conductivity, etc.). These parameters indirectly affect the gain of the amplifier by deciding the region in the scale where the amplifier will be operated. All instruments should be checked for their linearity and calibrated accordingly before they are used in the experiments [19].

The three Preston amplifiers used in the acquisition system have been calibrated and the range in which they are linear determined. Voltage from 0.0 to 0.01 V was applied to the input of the Preston amplifiers, the gain of the amplifiers set at 1000. The voltage was applied at a step of 0.001 V. The voltage input as shown on the potentiometer was plotted against the voltage output as recorded by the computer. A program called SETUP was then used to calculate the gain of the amplifiers from zero to full scale. Calibration using SETUP program is described in Part III, Chapter 4.

Figure 3.9a shows a schematic between the input voltage and the amplified output voltage and Figure 3.9b shows the amplifier gain as a function of the output voltage. These are not actual data. It can be inferred from these figures that the amplifiers are linear approximately over the middle 80% of the scale. The upper and lower end of the scale cannot yield the specified gain. Hence, corrections in the input signals need to be made before using the signals in the calculations if the voltage happens to be in these ranges. Figure 3.9c plots the linearity error (maximum deviation

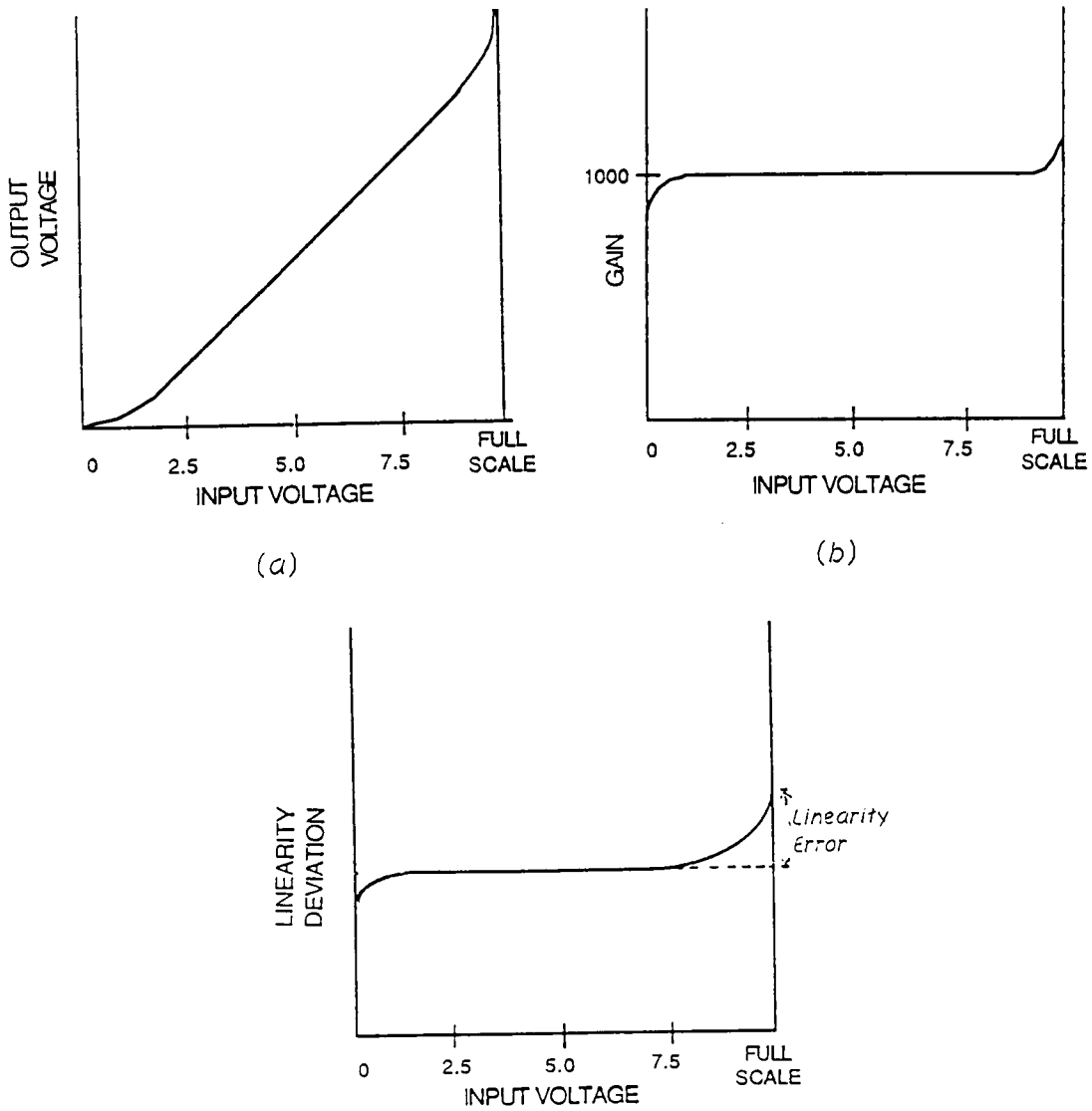


Fig. 3.9 Schematic diagram of (a) amplified output voltage, (b) amplifier gain, and (c) linearity deviation shown as functions of the input voltage. These are not actual data.

from ideal) of the voltage signal over the whole scale.

vi) Repeatability and random uncertainty - The term repeatability is more applicable to a calorimeter than the term reproducibility. Reproducibility refers to the agreement of results between the same experiment conducted in different places by different individuals under different environments [19]. In addition to the random errors, reproducibility also involves systematic errors (those arising out of differences in the instruments and the devices used). Going by this definition reproducibility does not have much significance for a particular calorimeter. However if the calorimeter is totally dismantled and then rebuilt and after reusing the experimental results are noted down and compared with those in the previous case, then their agreement is also a measure of reproducibility.

Repeatability is only affected by the random fluctuations accruing from the measurements of the same quantity, most often conducted by the same person or group of persons under the same conditions. It is usually expressed as a plus/minus semi-range about a mean value in absolute units or percentages. The overall repeatability of a calorimeter depends on the repeatability of the individual instruments involved in acquiring data.

vii) Accuracy and total error - Accuracy refers to the agreement between the measured value (e.g., specific heat) at a certain value of an experimental parameter (e.g., temperature) and the corresponding "true" value. The true value, in the absolute sense,

is somewhat nebulous. It generally refers to a value established by round-robin experiments and/or accepted by a Standards Institution. Overall accuracy is related to the proper calibrations of all the instruments involved in the process. If an instrument is properly calibrated the accuracy will be affected by random errors only. If not then systematic errors will contribute too.

Total error refers to the deviation from the true measured quantity and is expressed as

$$Total\ Error = \frac{Q_{measured} \cdot Q_{true}}{Q_{true}}$$

An error analysis of this technique was performed, and the results are discussed in Part IV, Chapter 1. In order to assess the total error of this technique the error involved with each measured quantity needs to be determined accurately.

PART III: CHAPTER 3
DIGITAL TO ANALOG AND ANALOG TO DIGITAL CONVERTERS

Communications through Digital-to-Analog (D/A) and Analog-to-Digital (A/D) channels and D/A and A/D conversions are accomplished by DT2821 series boards [20], which are high speed analog and digital Input/Output boards designed to be used with the IBM Personal Computer AT [21] or compatible computer systems. The connection of the board to the computer is via fully bussed system expansion slots in the computer backplane. The board can be programmed through the computer to perform A/D and D/A conversions, and the Digital Input and Output (DIO) transfers, on a specified channel, at a specified speed. The boards are currently being used in the Direct Memory Access (DMA) mode with interrupt capability. The interrupt capability allows interruption of acquisition at a specified rate to scan the keyboard or perform specific tasks specified in the interrupt subroutines. The acquisition boards for the A/D and D/A both have a resolution of 12 bits, an A/D throughput of 50 kHz and a D/A throughput of 130 kHz. The maximum A/D gain is 8. No gain is available in D/A channel.

The DT2821 series board also contains an on-board programmable pacer clock, which can be used to provide clock pulses to control the rate of conversions from the boards A/D and D/A subsystems. The pacer clock operates under program control and

provides a usable range of 1.25 microseconds (800 kHz) to 2 seconds (0.5 Hz). The A/D and D/A convertors were operated simultaneously at the same clock speed.

The Direct Memory Access (DMA) channels have been used to transfer data from the board to the computer. Channels 5, 6, and 7 are available. Data transfer in the 16-bit mode is possible using channels 5 and 6 simultaneously. A unique configuration using two DMA channels is available for acquisition in the special mode - the burst mode and the continuous mode. With the power level (90 amps max.) and pulse widths used in our project, a speed of 1 to 18 data points per second, per channel, was used in obtaining the power versus temperature and temperature versus time results. The interrupt mode can give a maximum of 18 data points per second. Getting more proximate data points, e.g., 300 data points per second, with Burst Mode operation may not contribute much additionally, in determining the equation for the best fit curve . But with high power pulses involving higher temperature rise, Burst Mode may be required.

Burst mode was not tried in our project but may be an option for our future experiments. To operate the D/A and A/D boards in the Burst Mode requires declaring buffer memory before acquisition. This is because at such high speeds the boards do not have time to write into the computer's regular memory. DMA buffers may be located anywhere in the 16 MB memory space of the computer and may be up to 128 KB long. It may sometimes be required to declare

more than one buffer depending upon the speed of acquisition and the duration of the direct memory access. Burst Mode and Continuous Mode are methods which provide gap-free transfers of large volumes of data.

A/D CONVERSION

An analog to digital converter (A/D) changes an analog voltage (e.g., output of a thermocouple) into a digital code of 0's and 1's which are intelligible to the computer. Figure 3.10 shows the block diagram of the A/D Convertor along with the 18-Channel Multiplexer, Gain Amplifier and Sample and Hold Circuit.

The output signals from the thermocouple, the voltage taps and the standard resistor are connected to the multiplexer. The multiplexer chooses one input channel at a time from the three channels connected to the board. The Programmable Gain Amplifier intercepts the analog input and may increase its voltage level depending upon the gain specified by the user. Since the analog input signal to the multiplexer can be only +1.25 Volt maximum, the internal amplifier is used to boost the signal's voltage to the +10 Volt level, which is the full scale operating level of the board's A/D convertor.

The Sample and Hold Circuit samples the selected analog signal from the multiplexer after it has passed through the amplifier. An internal trigger now marks the beginning of the scan. On the first

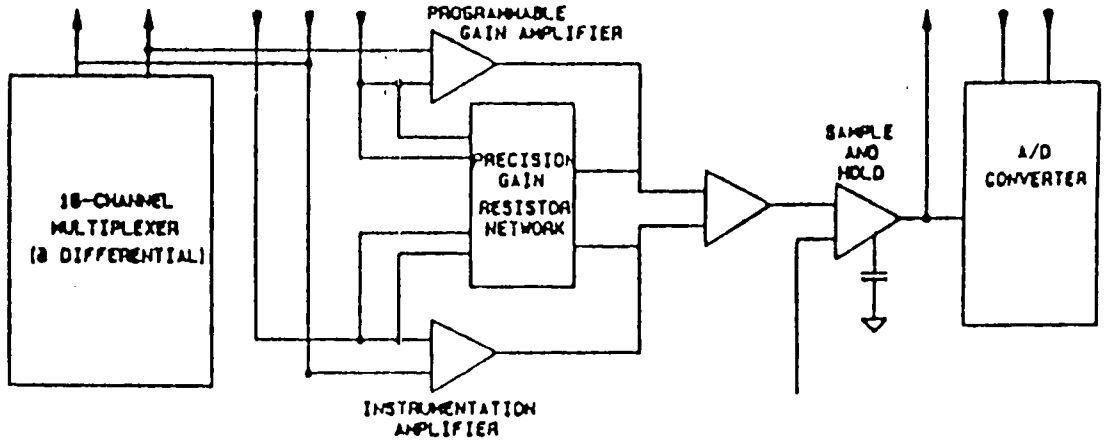


Fig. 3.10 Block diagram of A/D Converter showing multiplexer, gain amplifier and sample and hold circuit.

clock pulse after the trigger, the Sample and Hold Circuit switches to 'hold' and the A/D Convertor translates the analog signal held by the specimen into a digital code. At the next trigger the multiplexer loads the next channel and sends it through to the Amplifier and Converter Circuit. When all the three channels are accessed, the multiplexer scans the first channel again and the sequence continues. The multiplexer is ready to scan a channel only when the signal from the previous channel has been converted and the Sample and Hold Circuit is switched back to 'Sample' [22].

D/A CONVERSION

Two 12-Bit D/A Convertors (DAC 0 and DAC 1) are available, but only one has been used in the current pulse heating calorimeter. DAC 0 has been used to send control signals to the Programmable Power Supply.

The value of current, in amperes, for the pre-pulse, pulse and post-pulse period is first converted to the corresponding digital value using a conversion formula in the program. The digital conversion is for the computer's own processing. This digital signal is then converted back to analog using DAC 0 and then sent to the power supply. This is further discussed in Part III, Chapter 4.

The DACs provide constant output after the first data point is written to them. Subsequent 'writes' to the DACs simply change the output voltage. For example, data are written to DAC 0 which results

in a +1 Volt output. This +1 Volt signal remains on the output of DAC 0 until another data point is written to it. Whenever the system passes from the pre-pulse to pulse period or from the pulse to the post-pulse period a new data point is written to the DAC and the output voltage is correspondingly adjusted.

ACCESSORIES

A new terminal panel has been used to house the connections between the converters (inside the computer) and the external instrumentation. The terminal is screwed to the back of the work-station table. A flat ribbon cable, which plugs directly into the jumper connector of the board, serves as the connection between the A/D and D/A board and the screw terminal. Screw connections are labelled for analog input connections, analog output connections, Digital Input and Output (DIO) lines, external clock input and external trigger input. The analog input lines (e.g., those coming from the thermocouple, voltage tap and standard resistor) are connected from the screw terminals to the output of the three amplifiers individually. The external screw terminal at the rack makes it easy to make or change the connections.

BOARD CONTROL SOFTWARE

The real-time software package for Data Translation's IBM

Personal Computer AT compatible DT2821 analog and digital I/O Boards is called ATLAB [20]. The ATLAB package contains routines which can be called from Microsoft FORTRAN operated under MS-DOS. The ATLAB package allows the programmer to control all the analog and digital I/O capabilities of the interface boards through operation specific routines which greatly simplify the programming of the hardware interfaces. With ATLAB, direct access to the control registers is not required. An error processing subroutine checks for argument errors and generates an error report, if any are detected. Codes for A/D and D/A communications can be written along with the original FORTRAN codes and can be placed anywhere in the program. They are mostly single line statements with one or more arguments. The arguments, among other parameters, specify the channel number, gain, trigger (e.g., external or internal), number of buffers, buffer size, etc.

The codes which have been most frequently used are AL_SET_DAC(), AL_SET_A(), ALAV(), ALDV(). AL_SET_DAC() and AL_SET_A() are used to set up the D/A channel and the A/D channels, respectively. The specifications and arguments are put inside the parenthesis. The code ALAV() is used to remotely control the current level of the Kepco Power Supply through the DAC channel 0. Depending upon the pulse characteristics entered by the operator, the 'control routine' of the acquisition program, using ALDV() codes, sends signals through the DAC channel at proper times with the proper current signals to the power supply. Acting on this signal the

power supply sends the required current to the specimen. This will be further discussed in the next chapter.

Thus, the ATLAB library codes play a great role in the communication between the converter board and the external devices.

PART III: CHAPTER 4
COMPUTER AND PROGRAMS

THE COMPUTER

The computer is the heart of the process control, data acquisition and calculation system. It controls the current going through the specimen, records and converts incoming signals, processes stored data with a calculation program and displays and plots the desired thermodynamic data on the screen or with the printer. The main functions of the computer, thus, are current control, data acquisition, data processing, data printing and plotting. It is also involved in amplifier calibration. These processes require a high speed of operation and vast system board memory space. For example, after recording the first set of data points, the computer has to go through conversion steps and logic control steps before it acquires the second set of data points at a set time. The computer should be fast enough to complete these operations before the set time for calling the communications port. Thus slower computers are not suitable for this purpose. The computer should also have sufficient storage space to accommodate the controlling program and the data arrays, which take up a lot of space. It must accommodate the GW-BASIC [23] Interpreter to call and use several programs. These are some of the important factors which are to be considered while selecting the computer to be used.

The computer used in this calorimetry project is a DELL 310 System [24] based on an Intel 386 microprocessor running at a clock speed of 20 MHz. It is an IBM compatible with MS-DOS operating system. It has eight expansion slots, 1 MB of system board memory and supports a 20 MHz math coprocessor. It has dual disk drives ($3\frac{1}{2}$ inch and $5\frac{1}{4}$ inch). It has two serial ports for communications and a parallel port to connect an external peripheral such as a modem, printer or a mouse. Inside the computer is also installed Data Translation's DT 2821 series A/D input-output boards [20] having 7 A/D channels and 2 D/A channels. The other components of the computer workstation are the VGA Color Plus Monitor, Enhanced Keyboard, Mouse with pad and Epson FX-850 [25] Line Printer.

The computer is connected to the calorimeter via the instrument rack. Using proper programs, the computer, amplifiers, power supply and the calorimeter can be made to exchange information to perform a specific experiment. The next section discusses the programs used in this project.

COMPUTER PROGRAMS

Since calorimetry experiments were directed entirely from the computer's keyboard, specific programs were written to send control signals, acquire relevant data, process data and generate required thermodynamic quantities. Since the process, as a whole, involves diverse operations and is somewhat sophisticated, several programs

were written which were capable of performing specific tasks. These programs were then run in a particular sequence to yield the desired output. Each of these programs was further divided into several subroutines or modules to perform a more specific job. This simplified the task of writing the computer software for the programmer and the job of explaining the program codes to the reader or the next user was made easier. Also, all programs were extensively documented to furnish instant explanation of the program lines to the reader. All the programs used in the calorimetry experiments are explained with some detail in this chapter and flowcharts of these programs have been provided in Appendix I. Copies of these programs, in their final form, have been provided in Appendix II. Data disks containing the source codes and the executable files of these programs are available upon request. Appendix II provides more information.

These discussions and explanations were designed to make the programs easier for the user to operate and easier for the interested programmer to improve. Attempts were also made to make the programs user friendly and to minimize operator involvement as far as possible. Provisions were included to issue self-executable system commands from within the programs so that the operator did not need to go to the DOS prompt to issue these commands. Since several programs are involved in the calorimetry experiment, codes were written to make it possible to call one program from the other and pass the necessary variables from one program to the other. This,

again, reduced operator involvement since the operator was not required to call individual programs from the DOS prompt by typing their names.

When it was not possible to transfer variables directly from one program to another, a data file with the required data was generated so that the next program in the line could read the data from the file into the memory and process it. Multi-file-management was required to transfer data across programs. Program modules were written in a way so that no information is ever asked of the operator which can be read from another file. Files were also used extensively to store experimental data both in the raw and processed form to be retrieved later for usage. From time to time these files were transferred from the WORK directory to the DATAFILE directory where they resided permanently unless deleted. Usage of files were also extensively made while presenting data in a particular format to GRAPHER [26], a commercial software, which could display the best-fit-curve on the screen. By observing these curves along with the data points and comparing the fit statistics, the operator can decide the closeness of the fit.

The following sections explain the individual programs with the help of flowcharts and actual program lines.

Explanation of programs used in calorimetry experiments

Table 3.2 is a list of the programs written for the calorimetry

experiments in this project:

Table 3.2 List of programs written for calorimetry experiments.

No.	Program Filename	Function	See Flowchart
1.	CALPUL.FOR	Process control and acquisition program for pulse.	I
2.	MASTER.BAS	Overall control and calculation program.	II
3.	CONVERT.BAS	Converts thermocouple emf (μV) to temperature (°C).	II
4.	SETUP.FOR	Program for calibration of amplifiers.	III
5.	CALZEM.FOR	Process control and acquisition program for step heating.	-
6.	CALRES.FOR	Program to determine electrical resistivity.	-

Of these programs, only the first four have been used in this project, so only these will be described subsequently in this chapter.

Appendix I has the flowchart for the four programs. CONVERT.BAS has been treated as a part of MASTER.BAS to maintain continuity. So, a separate flowchart has not been provided. Appendix II has the source code of the four programs separately.

The Acquisition Program [CALPUL.FOR]

Name

CALPUL.FOR stands for Calorimeter Pulse and is the main acquisition program used in our calorimetry experiments. The ".FOR" extension indicates that the source code of the program is in FORTRAN. A Microsoft Compiler 3.0 [27] was used to compile the program. The program has been linked and an executable file has been created. Program I in Appendix II lists the source code of this program and Flowchart I in Appendix I is the flowchart for this program.

Features

The main features of the acquisition program are listed below.

1) Allows operator to select a standard run or enter parameters for a run.

2) Sends a control signal to the programmable power supply for sending controlled current to the specimen for a set time.

3) Changes and shuts off the current at the set time.

4) Acquires specimen current, specimen voltage, thermoelectric voltage and elapsed time as digital signals.

5) Converts these digital signals into their corresponding analog values and arranges them in data arrays.

6) Stores these data arrays in hard disk in a format MASTER.BAS program can read.

Comment lines

The first code in the program is the name of the program --

Program Calorimeter Pulse. This is different from the filename under which it has been stored -- CALPUL.FOR (short for Calorimeter Pulse). The first set of comment lines broadly describes what this program does. The program supplies a control signal via the D/A channel to the input of the power supply, which corresponds to a certain current. The Kepco power supply then supplies this current to the specimen. The signals for the current supplied to the specimen, the voltage across the effective length of the specimen and the thermocouple emf are then sent back to the computer through the three A/D channels. It is this acquisition which has the provision of recording the signals as digital values at a certain speed defined by the user, and subsequently converting them into analog values before displaying on the screen.

Define Variables

After the comment line is the section for variable definition. Forty seven variables have been defined. Depending on its needs to be shared by other modules, a variable can be defined as either local or common. Local variables are defined first. Four types of variables have been used - integer, real, character and external type. Variables like number of intervals, interval length, load and exit flags have been defined as integer variables. The current, voltage and thermocouple emf are stored as analog values which have been defined as double-precision real data, and so are the calculated power and resistivity. Depending upon the current range of operation the acquired data can have significant figures beyond the fifth or

sixth place of decimal. So it is always safer to work with double-precision variables. The only variable used as character variable is the filename. An example of filename would be DATAFILE. The operator is required to enter it through the keyboard to assign a name to a set of data.

After local variable declaration the common locations are established. This allows two or more program units to directly share these variables, without having to pass them as arguments. The common declaration begins with the name of the common block and is followed by the variable contents of the block.

Pre-main loop

Before the main loop starts, there is a small module under the name *Pre Main Loop* which performs certain tasks. The first thing it does is set the current through the specimen to zero, if it is not already so. This is an important step because the experiment should not start with power already in the specimen. Next it displays information to the operator which is relevant to him at that stage. It mainly shows the channel numbers corresponding to the signals coming in. It then waits for the operator to hit the spacebar to start the test. Figure 3.11 shows a printout of the screen at this stage.

Main loop

Once the spacebar is hit the control enters the *Main Loop* . The Main Loop immediately calls the subroutine *Pre-Info* and *Pre-Info* subsequently calls *Pulse* subroutine. This is the data entry module. This module first displays information on the D/A and A/D channels

```
** PROGRAM - CALORIMETER VERSION 2.1 **

    CAPS LOCK MUST BE ON

TEMPERATURE INPUT 1    ON A/D CH0
SAMPLE VOLTAGE INPUT 2    ON A/D CH1
SHUNT CURRENT INPUT 3    ON A/D CH2
KEPCO CURRENT OUTPUT 1 ON D/A CH0

**** SPACE TO PROCEED ****
```

Fig. 3.11 Screen printout in the Pre-Main loop.

on the screen (refer to block 4, Flowchart 1). The pulse characteristics defined by the user are entered to the computer through this module. The operator is prompted to enter the current for interval 1, 2 & 3 (i.e., for the pre-pulse, pulse and post-pulse period). Next, the interval between subsequent readings, during heating, is prompted. The value is to be entered in tics, where 1 tic = 55 milliseconds. Next the total number of readings in each of the three intervals is entered. This is all that is required to define the pulse. For specific heat measurement, the rate of decrease of temperature during cooling is also required to obtain the heat loss during the pulse. This requires a cooling period after the pulse and the acquisition to be active during this period. The acquiring speed during cooling can be different from that during heating. Since the cooling period lasts much longer than the pulse period, acquisition speed as high as that during heating may not be required during cooling. The other parameter required to define the cooling period is the number of data points in it.

Acquisition style

At this point it is pertinent to tell the reader that data points, as recorded by this program, are done against a Time-Tag-Number (TT#). This is a method of artificial time-keeping. This was adopted because there was no provision for acquiring data against time in seconds. The total number of data points in an interval is numerically equal to the TT# of the last data in that interval. Consider the example data given in Table 3.3 and the corresponding illustration in

Figure 3.12(a), which shows a pulse having 75 amps and the pulse current lasting for TT#=25-5=20. Both pre-pulse and post-pulse intervals last for TT#=5. The current in the cooling period is essentially zero and lasts for TT#=70-30=40, in this example. Since the time between two data points is known, the time in terms of TT# can be re-calculated in terms of time in seconds. Figure 3.12(b) shows the temperature profile during pulsing and cooling for the reader to correlate it with figure 3.12(a).

Table 3.3 Example data for Figure 3.12, representing pulse characteristics.

Intervals	Duration (TT#)
Interval 1 (pre-pulse)	5
Interval 2 (pulse)	20
Interval 3 (post-pulse)	5
Cooling Period	40

Screen information

After the program assigns the value of the entered quantities into the respective variables, it computes the TT#s which mark the end of the three intervals and the cooling period (block 10, Flowchart 1, Appendix I). The computed TT#s are used later in the program as flags for activating and deactivating the current and for stopping data acquisition automatically after the specified period. After this, the control is transferred back to *Pulse* and from *Pulse* to *Pre-info*.

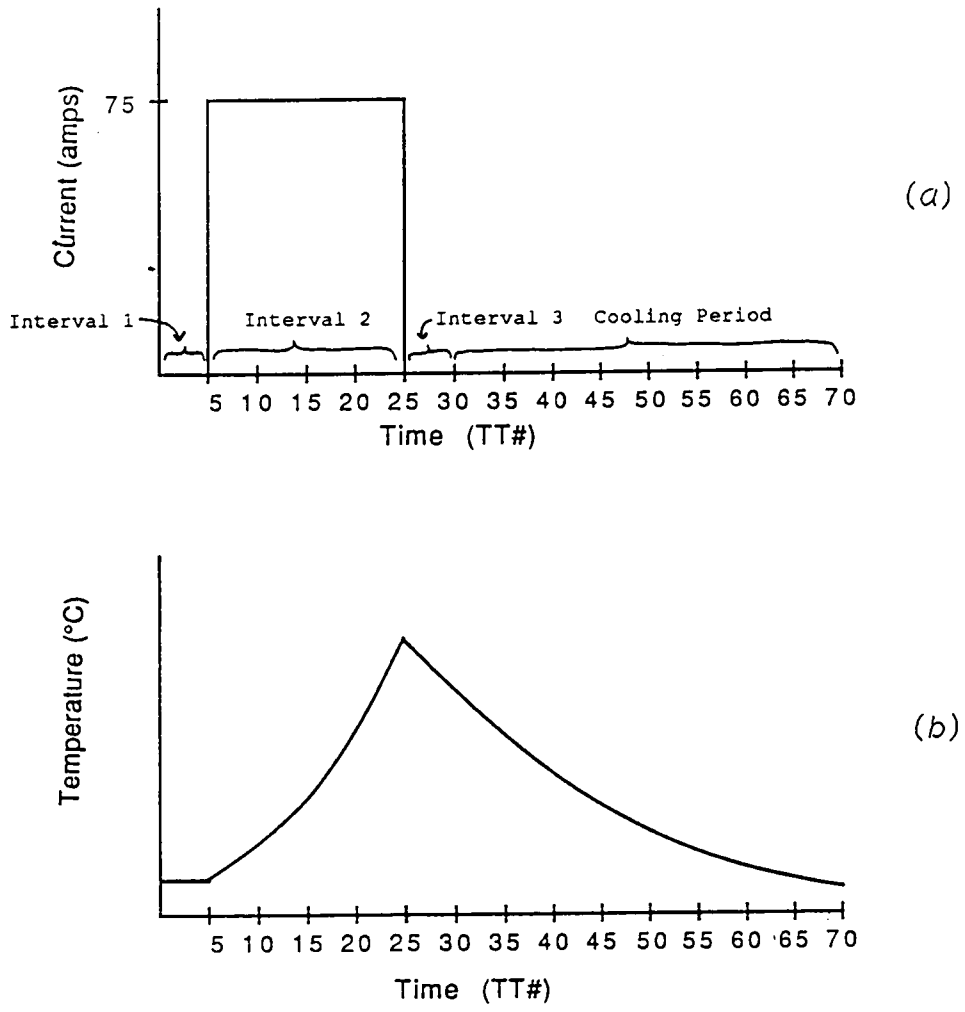


Fig. 3.12 (a) Current and (b) temperature data during a typical pulse and its cooling period, where time has been plotted in TT#.

The *Pre-info* subroutine then displays the last information set before the start of the experiment. The information mainly consists of how to start the acquisition from the keyboard, how to stop acquisition in case of an emergency, the TT# at which the program automatically deactivates output and the TT# at which acquisition is terminated. Figure 3.13 shows the pre-experiment information screen. Another function of the subroutine is to display the headers of the various quantities which appear in columns during acquisition (block 14). Next it returns to *Main loop* (block 15).

Initialization and interruption

In the Main-loop a series of variable initialization follows. The loop-number is initialized to zero. Variables responsible to control the acquisition speed, ITIC and STIM, are initialized too. ITIC and STIM are loop controlling variables of the interrupt subroutine. The initialization of the interrupt subroutines follows. Now the functions of the interrupt subroutines will be briefly discussed. The three IRSET statements in the main-loop initializes interrupt servicing. The routine establishes a specified FORTRAN subroutine to be an interrupt service routine. Both hardware and software interrupts are intercepted. The first argument is the interrupt number. The second argument is the name of the FORTRAN subroutine to intercept interrupt. The third argument is the workspace to contain saved information and the interrupt servicing mechanism. Whenever an interrupt subroutine is called, the control is transferred to the specific interrupt subroutine. Interrupt subroutine#1, ISR1, performs

**** PRESS SPACEBAR TO ACTIVATE OUTPUT ****

AND START DATA ACQUISITION

THIS PROGRAM AUTOMATICALLY DEACTIVATES OUTPUT
AT THE END OF INTERVAL3 (TT# 85 FOR THIS RUN)
AND STOPS DATA ACQUISITION AT THE END OF THE
COOLING PERIOD (TT#1045 FOR THIS RUN)

Fig. 3.13 Pre-experiment information screen.

the task of achieving the desired wait time by decrementing the loop number from the user specified tic value and checking for its equivalence to zero. Once this equivalence is true the load flag is set to 1 so that the control is ready to load the next data point. Before exiting this subroutine, ITIC is reset to the operator specified tic value so that it can be used in the next loop again (refer to Interrupt Service Routine, Program I, Appendix II). The other two interrupt subroutines, ISR2 and ISR3, perform a hardware interrupt which enables them to leave the program and scan the keyboard to check whether control-C (^C) or control-break (^Break) have been pressed at any instant. If these keys are pressed then the exit flag is set to 1 and the acquisition is terminated. However, one seldom needs to abort the program in the middle of a run. So at the end of ISR1, control is returned to the main loop IRSET call.

A/D conversion

The next set of instructions performs the analog to digital conversion of the input signal to be sent to the input of the Kepco Power Supply to trigger its output to the desired current level (block 19). The variables ICURRENT1, ICURRENT2 and ICURRENT3 carry the analog values, in amperes, for the three intervals, as entered by the operator through the keyboard. The variables ICO1, ICO2 and ICO3 contain the converted digital values which are compatible with the D/A channel through which the signal will be sent. The conversion factor takes into account the fact that at the full scale 4096 counts corresponds to 10 volts and 90 amps DC for this power supply

corresponds to 9 Volts.

Data loading

After this conversion the load flag, ILOAD, is checked (block 20). If ILOAD=0 control is transferred to the Pre-exit line (block 65). However, ILOAD≠0 until the required number of loops for a particular experiment is over. So, when the program sees that ILOAD=1 it calls up the *Load Module* (block 22). It is in this module that the three channels are accessed through the communication ports into the ATLAB board and then to the program. The first executable code in the module deals with the incrementing of the TT#. This is done so that each data point can be stored under an unique TT# which can give information about the time in seconds at which it was acquired. (However, conversion from time, in TT#, to time, in seconds, has not been done anywhere in the acquisition program. It is converted later in the calculation program). Channel#0 of the A/D series is now called with an internal gain of 1 and the digital signal is stored under IN (block 24). The interrupt subroutine#1 interacts with the main-loop program in such a way that the signals coming through the three A/D channels are read at the moment the system clock updates itself, i.e., between 1 and 18 tics, as entered by the operator through the *Pulse* subroutine. How this is exactly done has not been discussed here because this will require a thorough knowledge of interrupt handling. After the digital signal is acquired it needs to be converted to analog so that when it is displayed on the screen the operator can view it in terms of

familiar units (amperes, volts or millivolts) and so that the analog quantities can be used directly in calculations. For this conversion to be made the digital value stored in IN is 'floated' to convert it from Integer type to Real type. This real quantity, then, is multiplied by the necessary multiplication factor (block 25). The converted value of current, in amperes, is now stored in a real type data array with array number starting from IVAL=0. This analog value will be N times more than that actually flowing through the specimen, where N is the gain of the amplifier for that particular channel. To remove this gain from the recorded signal, the array variable is divided by N to yield the correct value (block 26). The above series of procedures is performed for CH#0, CH#1 and CH#2, which correspond to the specimen current, specimen voltage and the thermocouple emf, respectively. Thus, the current through the specimen, the voltage across the effective specimen and the thermocouple emf are all in their conventional analog units so that they can be used in subsequent calculations with ease. An array variable for time, TIME(IVAL), is created and the value of IVAL at each loop is transferred to it so that it shows the TT# for each data point. This is because TT# for a particular set of data is numerically equal to the value of IVAL during the loop in which that data was acquired. Next the LOAD flag was reset to zero and control transferred to the main-loop (blocks 34 & 35).

Calculation module

Next, the control enters the *Calculation Module* for the

acquisition program. This module performs some elementary computations. The product of current and voltage is computed and stored in another data array called POWER(IVAL). The contents of this array are displayed under 'Power' on the screen and stored for further processing. The specimen electrical resistivity is calculated using values of specimen dimensions and the value of current and voltage in that loop. The resistivity calculated in each loop is shown on the screen each time and also stored in a data-array called RES(IVAL). In each loop there is a check on whether the value of IVAL for that particular loop is greater than, less than or equal to the flag for interval 3 (block 38). If IVAL ≤ INTERVAL3 then the tic value assigned to STIM will be that during pulsing (block 39). If, however, IVAL > INTERVAL3 then, since the cooling period has begun, the tic value assigned to STIM is that for cooling (block 40). This value of STIM is passed to the interrupt subroutine #1 which controls the time delay between two successive data recordings.

Control Module

Upon exiting the *Calculation Module* the program enters the *Control Module*, which is the module for controlling the D/A parameters. Some of the features of the pulse which are controlled by this module are :

1) Setting the current for pre-pulse, pulse and post-pulse to the value entered by the operator in the pulse module.

2) Controlling the time for pre-pulse, pulse and post-pulse as entered by the operator.

3) Setting the current to zero during the cooling period.

4) Controlling the length of the cooling period.

5) Terminating acquisition when the 'end flag' is reached.

6) Sounding beeps at the beginning of the cooling period and the end of run.

Blocks 41 to 50 illustrate the operation of the *Control Module*. Before communicating to the programmable power supply, the D/A channel needs to be set up for proper communications. The first code of the *Control Module* does exactly this. The AL_SETUP_DAC (abbreviated as ALSETD) procedure establishes the transfer parameters for subsequent D/A series and D/A Direct Memory Access (DMA) operations (block 41). The first argument of the procedure assigns the timing source. The number '0' indicates that the internal clock in the Data Translation's Acquisition Board is the trigger source for conversion timing. The second argument specifies the D/A channel used to transfer the data value. The number '0' in this argument indicates that D/A channel #0 has been specified for communications.

Next follows a series of IF statements (blocks 42 to 50) which, by comparing the value of IVAL with the flags set for intervals 1, 2, 3 and 'End', transfer the control sequentially from interval 1 to interval 2 to interval 3, then onto the cooling period and through to the end of acquisition. When the control is in a particular interval the value of the pulse current, in counts, for that interval is communicated to the power supply. Also the analog current value is

loaded to a variable called ICURR for displaying it on the screen. This may be useful at times, for comparison, if the set current is different from the acquired current. In the block diagram, blocks 42, 44 and 46 correspond to interval 1, 2 and 3 respectively. Block 48 is for cooling period and block 50 marks the end of acquisition if the comparison yields a YES. The conditional branchings are a set of two statements each, which checks the current value of IVAL to ascertain the interval in which the data acquisition is occurring. Depending upon which interval is active the instructions specific to that interval are performed. The common point of return merges between blocks 48 and 50, as partially indicated by curcuits 5 and 6. Instructions have also been written to sound a beep the moment the end flag is reached. If the comparison in block 50 yields a YES (i.e., IVAL > 'end flag') blocks 54 to 56 are first executed. The 'End of Cooling Period' sign is displayed and the operator has the choice of saving or discarding the acquired data. The operator can call the *Save Module* by hitting spacebar (block 57).

At this point let us go back to block 50 to find out the path travelled by the program if the comparison yields a 'NO'. A 'NO' will result for all values of IVAL except the last value which terminates acquisition. That is, when data acquisition is active, block 50 yields a 'NO'. A conditional WRITE statement follows which prints the message 'COOLING PERIOD BEGINS' on the screen to notify the operator. This and the sounding of the beep are simultaneous. Block 53 is the only real time display of the acquired and calculated

parameters on the computer screen. It helps the operator to monitor the current, voltage and temperature associated with the specimen, instantaneously. This WRITE statement prints the values of TIME, CURRENT, VOLTAGE, TEMPERATURE and POWER in a single line for each loop. Refer to Appendix III to look at how the data are displayed and/or printed. The three interrupt subroutines are then terminated and control is transferred to the beginning of the loop again (block 20), so that the next set of data points can be acquired under an incremented value of IVAL.

Save module

Let us return to block 57 now. If the operator hits the spacebar, blocks 59 through 64 are executed. The *Save* subroutine is called where the following options are displayed on the screen:

S : Store Data on Disc

P : Print Data on Printer

B : Both Store and Print Data

E : Exit (without storing or printing)

The operator enters a selection by pressing the letter of his/her choice. A series of IF statements and a GOTO statement has been written to perform the function of a switch (block 61). If the operator wants to store data, the program prompts for the filename under which the data are to be stored. A sequential file under that name is opened and the acquired data stored in the hard disc for further processing. The operator also has the choice of printing the data on the printer by pressing the 'P' key. Pressing 'B' allows both

storing data in the hard disc and printing it on the line printer. If he/she is not interested in saving the data he/she can exit by pressing 'E'. After the control returns to the *Main Module* from the *Save Module*, the next statement transfers the control to the *Pre-exit* location (block 65). The flags that are checked are the exit flag and the flag for the maximum array size. The next statement sets the current through the A/D channel to zero if it is accidentally high (block 66). This is a precautionary measure. The interrupt subroutines are terminated and the *Exit* subroutine is called.

Exit module

The *Exit* subroutine displays an option to the operator - 'Run' or 'Quit'. The operator has to type in his/her choice. If the operator runs the program again, control is transferred from block 70 to block 4 which immediately displays the introductory pre-experiment information on the screen. This is the longest loop in the program and all the other loops are nested inside this one. However this loop is optional and is executed only if the operator wants to run the program more than one time. If the operator's choice in block 70 is 'Quit', control is transferred to the STOP statement (Label 90, Exit subroutine, Program I, Appendix II). This terminates the program and returns control to the DOS level.

There is another subroutine at the end of the program called *Block Data*. This subroutine does not contain any executable statements. The variables and array elements declared under 'common' are initialized in this section.

This above discussion, which has frequent references to the flowchart, is expected to help the reader to form a basic idea about the program structure.

The Overall Control Program [MASTER.BAS]

General Information

This is the control program which calls other programs to interact with its own program codes to carry out a specific heat experiment and process the data after the experiment to yield the specific heat of the specimen. Tasks including data acquisition, conversion, curve fitting, file handling, calculation and display of results can be done from this program. This program can call other programs directly, whenever required, and transfer information between each other to carry out a task. At other times this program can generate files with appropriate data in a specific format for other programs to read and use the data. The MASTER program is basically a calculation program but performs other specific tasks too.

Name

The name of this program is MASTER CONTROL and has been stored under the filename MASTER.BAS. The '.BAS' extension indicates that it has been written in BASIC. The GWBASIC interpreter has been used to write this program. The program can be executed without linking and compiling. The actual program listing of MASTER is provided in Program II of Appendix II. The flowchart of this program is illustrated in Flowchart II of Appendix I. The flowchart

and program listing are referred to frequently during the following program explanation.

Functions

The main functions of the MASTER program are listed as follows.

1) Call acquisition program.

2) Read pulse characteristics from the file generated by acquisition program.

3) Read acquired data from the file generated by acquisition program.

4) Convert TT# to seconds.

5) Call CONVERT.BAS to convert thermocouple emf, in microvolts, to temperature, in °C.

6) Create new file having heating data.

7) Create new file having cooling data.

8) Call GRAPHER to fit data whenever required.

9) Formulate equations for T= f(t) both for heating and cooling.

10) Differentiate equations to get dT/dt = f(t).

11) Append dT/dt data to heat and cool file.

12) Enter coefficients for dT/dt = f(T) both during heating and cooling.

13) Use these coefficients and other data in the final specific heat equation to calculate specific heat at a given temperature.

The logical sequence of events in the program can be described

as route selection, file selection, data conversion, writing 'heat' and 'cool' files, curve fitting, power and slope calculation and specific heat calculation. These events are described in more detail in the next few paragraphs.

Route Selection

After displaying the program title and setting the screen parameters, the program prompts the operator to enter choice of calling or bypassing the acquisition program (refer to block 2, Flowchart II, Appendix I). If the operator wants to perform a new experiment then the acquisition program (CALPUL) will be called by hitting the spacebar. If, however, the operator only wants to process data from a previous experiment, any other key is pressed to bypass acquisition. This is shown on the flowchart by the conditional branching from block 2 to block 6.

File Selection

As discussed in the last section, the *Save Module* of the acquisition program writes a file containing the voltage, current and thermoelectric voltage data acquired. In the saved file is also written some information on the pulse characteristics. Specifically the duration, in TT#, of the three intervals, the duration of the cooling period, and the acquisition speed during heating and during cooling are written (refer to Figure 3.12). The MASTER program requires this information to create the 'heat' and 'cool' file and to convert the time data. Block 7 asks the operator to input the name of the raw data file to be processed. This raw file may be the one just created by the

acquisition program or any other previously saved file. Once the MASTER gets the name of the file to be processed, it goes to the current directory, the C:/PROJECT/WORK directory, and opens that file to read the data in it (block 10).

Data Conversion

At first the data on pulse characteristics are read. The value of the variables INTERVAL1, INTERVAL2, INTERVAL3, INTCOOL, ITIC, ITICOOL are thus transferred from CALPUL to MASTER via the data file. The variables introduced in this paragraph are defined as follows:

INTERVAL1 - Length of the Pre-Pulse period

INTERVAL2 - Length of the Pulse period

INTERVAL3 - Length of the Post-Pulse period

INTCOOL - Length of the cooling period

ITIC - Time interval between two subsequent data points during heating

ITICOOL - Time interval between two subsequent data points during cooling

Refer to lines 170 to 220 in Program II, Appendix II, for these variables.

Next the flags marking the ends of the intervals and the cooling period are calculated and set. Several array variables need to be declared and dimensioned before they can be used during processing. Since the time data and the temperature data need to be converted in this program, data arrays holding the old values and the

converted new values should be declared. The dimensions of these arrays should be more than or equal to the maximum number of data points acquired by the acquisition program. Otherwise an 'Out of Memory Error' will occur. The dimensions of array variables TIMEOLD, TIMENEW, TEMPOLD, TEMPNEW, CURR, VOLT and POWER have been declared in lines 260 and 270 of this program listing, which can be found in Program II, Appendix II. Then the time, current, voltage, thermocouple emf and power data are read and stored in arrays TIMEOLD(), CURR(), VOLT(), TEMPOLD() and POWER(), respectively. The thermocouple emf stored under the array variable TEMPOLD() now needs to be converted to temperature (°C). The conversion requires one or more equations for T=f(E), where T is the temperature (°C) and E is the thermocouple emf (millivolts), depending upon the temperature range of operation. A separate program called CONVERT.BAS has been used for this purpose. The variables required to make the conversion are chained to the CONVERT from the MASTER (lines 350 & 360). CONVERT then processes the thermal emf data using equations for Platinum-13% Rhodium thermocouples. A program code listing of CONVERT is given in Program IV, Appendix II. After the thermocouple voltage is converted to temperature (°C), the TEMPNEW data is chained to the MASTER which is stored for later use.

The next task of the MASTER program is to convert the artificial time (in TT#) to real time (in seconds). Separate formulae are used for heating and cooling data because the acquisition speed

during heating may be different from that during cooling. First the time conversion for heating data is done (lines 440 to 460). This involves knowing the acquisition speed during heating, given by ITIC, the TT# for each data, given by J, and the number of tics per second, which is a constant equal to 18.20651 tics/second. A simple loop of J is carried out from J=1 to the difference in the numerical value of INTERVAL2 and INTERVAL1. This difference is also numerically equal to the total number of data points in Interval 2. Thus within this loop all the time data acquired during the pulse are converted from TT# to seconds. The pre-pulse and the post-pulse data are discarded because they are not required for the calculation of the specific heat.

Writing 'Heat' and 'Cool' Files

Next, a filename with a postscript 'H.DAT' is created and the acquired data during pulse, i.e., Interval 2, is written to it (lines 470 to 540). This means that if a datafile called DATA, for example, is created by the acquisition program, the 'heat file' created by MASTER will have the name DATAH.DAT. The contents of this heat file will be time, temperature and power data. The file is automatically created and named without any operator involvement. The extension .DAT is essential because the commercial package GRAPHER only recognizes files with .DAT extension as data files.

Next, the 'cool' file is identically named and created. It contains the time and temperature data for the cooling period. For the above example a datafile called DATAC.DAT will be created, where 'C'

indicates cooling data. Refer to lines 600 to 670. An array element called 'K' has been defined and used in the PRINT# statement with the time array instead of the usual 'J' (line 640). This was done to reset time = 0 at the beginning of the cooling period. Also, for converting cooling data from TT# to seconds, the tic value in ITICOOL is used instead of ITIC, since ITIC may or may not be equal to ITICOOL for a particular run.

Curve Fitting

Next a series of interactions with the fitting program, GRAPHER, starts. After the 'heat' and 'cool' files are created in the format GRAPHER can read, MASTER fetches GRAPHER into the computer memory (block 25, Flowchart II, Appendix I). With appropriate menu selections the 'heat' file can be imported into GRAPHER. The contents of the file can be viewed. This is the first time the operator gets to view the processed file where the time is in seconds and the temperature is in °C. Power vs. Temperature data are also plotted on the screen. The curve that best fits these data points is then plotted. The degree of fit needs to be specified. With a few more selections the data points along with the best-fit curve are viewed on the screen. The goodness of the fit can either be determined from looking at the graph or by noting the value of the 'sum of the residuals' of the fit. The smaller the number the better the fit. For the best fitting curve the sum of the residuals should be a minimum. The meaning of 'sum of residuals' and its significance has been explained in Chapter 5 of Part III. After the operator decides the degree to which the

curve will be fitted, the coefficients for the fit can be looked at. The equation will be of the form $P = a+bT+cT^2+....$, where P is the power input to the specimen, T is the specimen temperature, and a,b,c, ... are the fitting coefficients. A printout of these coefficients can be obtained on the line printer. The printed coefficients are retained for later entry into the *Calculation Module* of the MASTER program. The next graph plotted is between temperature(°C) and time(seconds). The degree of fit is decided and the generated coefficients of the polynomial fit are printed again. Blocks 26 and 27 pertain to the discussions in this paragraph.

Power and Slope Calculation

Once the control returns to the MASTER the calculation routine starts. The message 'Enter Coefficients for POWER vs. TEMPERATURE fit during heating' is displayed on the screen and the value of the coefficient 'a' is prompted (lines 720 and 730). After the value of 'a' is entered by the operator from the printout, the values of the other coefficients are also prompted. Once this is done the operator is requested to enter the coefficients for Temperature vs. Time fit during heating, i.e., the coefficients 'a' through 'f', where $T = a+bt+ct^2+....$ (block 29). This curve gives the temperature at any time 't' during heating. The slope of this curve is required to know the rate of increase in temperature during the pulse. Line 900 formulates an equation between 'T' and 't' with the help of the coefficients a,b,c, ... This equation is differentiated giving

$$dT/dt = b + 2\,c\,t + 3\,d\,t^2 + 4\,e\,t^3 + \ldots\ldots \text{ (line 910)}$$

The above two equations are put inside a loop with the loop increment variable J operating between INTERVAL1 and INTERVAL2 (lines 870 to 920). The loop increment variable K, for time, is in effect operating between 0 and INTERVAL2 - INTERVAL1. This may not be evident from the program codes but substituting the value of J (line 870) into the equation (line 880) will yield the limits of operation, which is the loop termination value for J. As mentioned earlier, different loop increment variables J and K have been used to reset the time to zero at the beginning of pulsing and at the beginning of the cooling period. The two equations are operated within a loop so that for each data point (time) a corresponding value of dT/dt is obtained. The 'heat' file is then opened in the append mode (block 31) and the slope (dT/dt) is appended to the file as a separate column (block 32). The 'heat' file is then closed and GRAPHER is called. The new 'heat' file, though having the same filename, is imported and its contents viewed. In addition to time, temperature and power column, the column for slope data is also there now. A curve for Slope = f(Temp) (not Slope = f(time)) is plotted. This is because all parameters need to be calculated as a function of temperature if C_p as a function of temperature is ultimately desired. A print out of the coefficients is obtained. GRAPHER is exited. The coefficients for $dT/dt = f(T)_H$, for heating, are prompted by the program and stored into appropriate variables (block 34). The processing of the pulse data is now complete.

GRAPHER is called again and the processing of the cooling data

begins. The 'cool' file with the suffix 'C.DAT' is now imported and its contents viewed. As expected it contains the temperature vs. time data only during cooling, with time starting from zero at the beginning of the cooling period. The temperature versus time data for cooling has the curvature similar to that shown in Figure 3.14. The coefficients of this curve, as generated by GRAPHER, are entered into the appropriate variables in MASTER (block 36). An equation for temperature as a function of time during cooling, $T = f(t)_C$, is formulated (line 1220) and in the very next step this equation is differentiated (line 1230). These equations are put inside a loop (lines 1190 to 1240) which calculates the slope of the $T = f(t)_C$ curve for each time increment. This slope data are then appended to the cool file as a separate column (lines 1250 to 1300, block 39). The updated 'cool' file now has the data for time, acquired temperature, smoothed temperature and slope (line 1280). The smoothed temperature values are those calculated by the equation $T = a + b\ t + c\ t^2 + ...$ (line 1220). GRAPHER is called again and the updated 'cool' file is imported. The operator has the choice of plotting the slope against either the acquired temperature or the smoothed temperature. Plotting against smoothed temperature is perhaps better because it will give smoother $dT/dt = f(T)_C$ curve. The degree of fit is determined and the coefficients of the fit obtained. A printout of the coefficients is made. MASTER then transfers control from GRAPHER to itself and prompts for the coefficients of the $dT/dt = f(T)_C$ fit. The operator enters the coefficients which are stored in

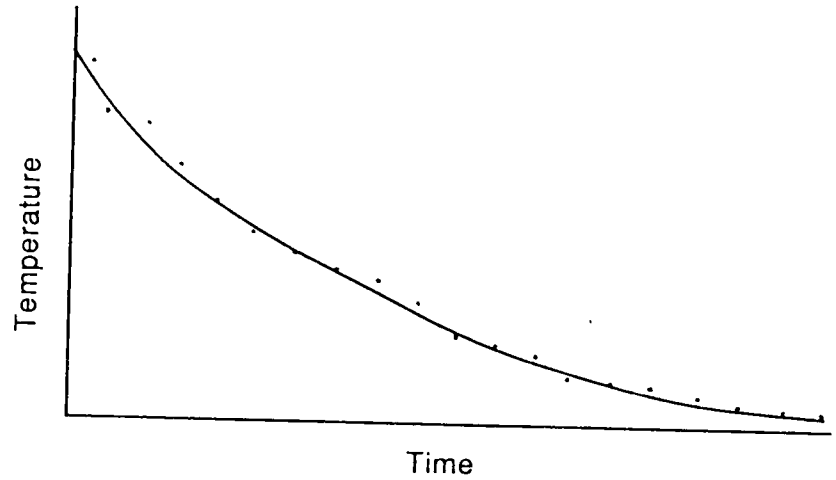


Fig. 3.14 Schematic typical profile of temperature-time data during cooling shown along with the best fit line.

appropriate variables (block 41).

Specific Heat Calculation

The specimen dimensions are specified in the program in line 1430. The data required to calculate the specific heat are all in the MASTER program now. The program displays the specimen dimensions on the screen and asks the operator to confirm whether the same dimensions are being used. If not, the operator has the choice of changing the values of the length and diameter of the specimen (blocks 44 & 45). Equations of power as a function of temperature $P = f(T)_H$, and slope as a function of temperature during cooling, $dT/dt = f(T)_C$, are formulated using the coefficients already stored.

First the volume and then the mass of the specimen is calculated using the specimen dimensions and the density of nickel (line 1470). The program then requests the operator to input the temperature range in which the specific heat of nickel is to be calculated. Once the temperature range is entered, the program selects the lower temperature limit and calculates the power (P), slope during pulsing (dT/dt_H), and slope during cooling (dT/dt_C), at this temperature using equations in lines 1440 to 1460 respectively. These calculated parameters and the specimen mass are then substituted for the variables in the final specific heat equation, $C_p = EI / (m (dT/dt_H - dT/dt_C))$ (line 1480). The specific heat at the lower temperature limit is thus calculated and displayed on the screen. The set of formulae are put inside a loop so that specific heat

in the specified temperature range is calculated with the specified temperature increment. The calculated set of specific heat values are then displayed on the screen or optionally stored as a file or printed on the printer. The operator also has the choice of getting a plot between the calculated Cp vs. T data. The MASTER program is then terminated.

Program for Curve-Fitting and Plotting [GRAPHER]

GRAPHER [26] is a commercial package which has been used extensively in the processing of raw data for calculation of the specific heat. It is a powerful and flexible tool for creating high resolution X-Y graphs for viewing on screen or as a printer or plotter output. In addition, it can do certain statistical regression analysis to generate best least-squares fit curves for a given set of data and calculate the corresponding error of fit (e.g., sum of residuals). The best fit curve can be viewed superimposed over the data points, with good resolution. The fit statistics (e.g. the fitting coefficients) can be viewed and printed for later use.

GRAPHER has been called several times from the MASTER program to plot data graphically and generate the coefficients of the best fit plot for a given degree of fit. Most of the graphs presented in this thesis have also been plotted by GRAPHER.

Program for Amplifier Calibration [SETUP.FOR]

This program written in FORTRAN is involved in the calibration

of the three amplifiers used in this project.

A steady output signal from the output of the potentiometer is supplied to the input of the amplifier whose gain is to be determined. The gain of the amplifier is set by the knobs in the front panel. The SETUP program is then run. A brief explanation of the program codes is provided here. Flowchart III in Appendix I and Program III in Appendix II pertain to this program.

As in other FORTRAN programs, the local and common variables are declared in the beginning. The interrupt variables are defined. The *Parameter Module* is then called (block 4, Flowchart III, Appendix I). Two values are entered in this subroutine (blocks 5 & 6), the voltage supplied by the potentiometer and the A/D channel# to scan. The three A/D channels are connected to the three amplifiers. The channel number specifies which amplifier output to tap.

Four flags are then initialized to zero - exit flag, loop flag, load flag, count flag (block 7). The array element is initialized to 1. The message 'Press Spacebar to Begin Acquisition' is displayed on the screen. When the operator presses the spacebar, acquisition starts and the message 'Acquisition in Progress' is displayed on the screen. The acquisition continues till 100 data points are acquired, unless terminated in the middle by pressing spacebar, ^C or ^Break. The speed of acquisition can be varied but is not critical here. If acquisition is terminated before acquiring 100 data points then the average is calculated on the basis of the number of data points taken,

which is numerically equal to the value of the variable ILOOP. The interrupt subroutines for delay, ^C and ^Break are initialized (block 11). While the acquisition is in progress the keyboard is scanned periodically to see whether the spacebar has been pressed again (line 30, Program III, Appendix II). If spacebar is pressed, control is transferred to line 50, which is the pre-exit location, via path#3 (refer to Flowchart III). Next, if the load flag is high the *Load Module* is called.

The four tasks performed in the *Load Module* are acquisition of data through A/D channel# as specified in the *Parameter Module*, storage of acquired value in the variable IARRAY(), incrementing the array# and incrementing the loop#. After these tasks are accomplished control returns to the line from where the *Load* routine was called. The next statement in the program initializes the load flag to zero. The control is then transferred back to block 12 via path# 2. Next the IF statements check the load flags again and the program enters the *Load Module* to acquire the next data point. This loop continues till 100 data points are acquired (except when terminated by spacebar, ^C or ^Break). When any one of the conditions for exiting the program is satisfied, the control enters the Pre-exit location and terminates interrupt subroutines # 1, 2 and 3.

Some simple calculations and the display of results follow (block 20). The contents of the variable ICOUNT, which are the individual counts of data, are transferred to COUNT. This is done to change integer value to real value. The value stored under ILOOP is

either 100 or its current value while being terminated by spacebar, ^C or ^Break. The cumulative count value is divided by the number of loops and stored under the variable COUNT. The statement COUNT = COUNT / ILOOP performs this (between lines 50 and 60, Program III, Appendix II) and the COUNT value is called average count. The averaged count is then displayed on the screen. This average count value is then converted from digital to analog by the conversion equation COUNT = COUNT * (10./4096.). The conversion factor is based on the fact that a full scale reading of 10 DC Volts correspond to 4096 counts for a 12 bit A/D converter. The converted value of average counts is stored in the same variable COUNT.

The gain of the amplifier is then calculated by dividing the analog cumulative count value by the potentiometer output voltage, stored in DIAL. The contents of COUNT and DIAL are then printed on the screen. Finally the calculated gain is printed. The operator then has a choice of viewing the individual counts by pressing backslash(\) (block 27). If the answer is 'NO', control goes to block 29. If the operator responds with a 'YES', the COUNT() array is displayed on the screen. These are the individual data points. The *Save Module* is subsequently called and the operator is requested to enter a filename for data storage (line 10, Save and Print Module). Next, he/she is required to input his choice of whether he would like the data to be stored. If the response is affirmative, a file with the given filename is opened for output and the data array written into it along with the channel number (blocks 31 to 34). A negative

response bypasses the 'Store' routine. If the operator wants a printout, a 'YES' to the query in block 35 allows him to obtain a printout of the file on the line printer (blocks 36 to 40). A 'NO' bypasses the 'Print' routine.

The program then prompts the operator - 'Do You Want to Run Again?'. A 'YES' transfers the control back to block 4 and repeats the whole process. A 'NO' terminates the program.

The SETUP program is an important tool for measuring the amplifier gain at various input voltages. It is desirable to run this program before every experiment to check the gain. In our project, the company specified gain was used in the calculation program and the specific heat experiments were conducted with uncalibrated amplifiers.

PART III: CHAPTER 5
MEASUREMENT METHOD AND CALCULATIONS

DESCRIPTION OF MEASUREMENT CIRCUITRY

The calorimetry circuitry is of the closed loop type. Figure 3.15 shows a block diagram of the calorimeter, its acquisition system and circuit connections. The operator has the choice of controlling the current locally by a local/remote switch situated in the control panel of the instrumentation rack. In the local control mode the computer has no control over the power supply. The current knob in the power supply can be used for increasing or decreasing the current and the on/off switch can be used to send or discontinue current to the specimen. If the remote mode is selected the experiment is initiated from the computer keyboard and is terminated with displaying the output on the screen and/or saving the output on the hard-disk. The process is triggered by the computer sending an input signal to the power supply. The operational power supply then sends the direct current, indicated to it by the input signal, to the specimen. The current stays at this level till another input signal is supplied to the input of the power supply. A single DAC channel is required to send signals from the D/A converter to the calorimetry system.

The specimen rests in the specimen holder which is put inside the vacuum chamber (see Figure 2.17). The current leads supply the specimen with the required power to raise its temperature to the

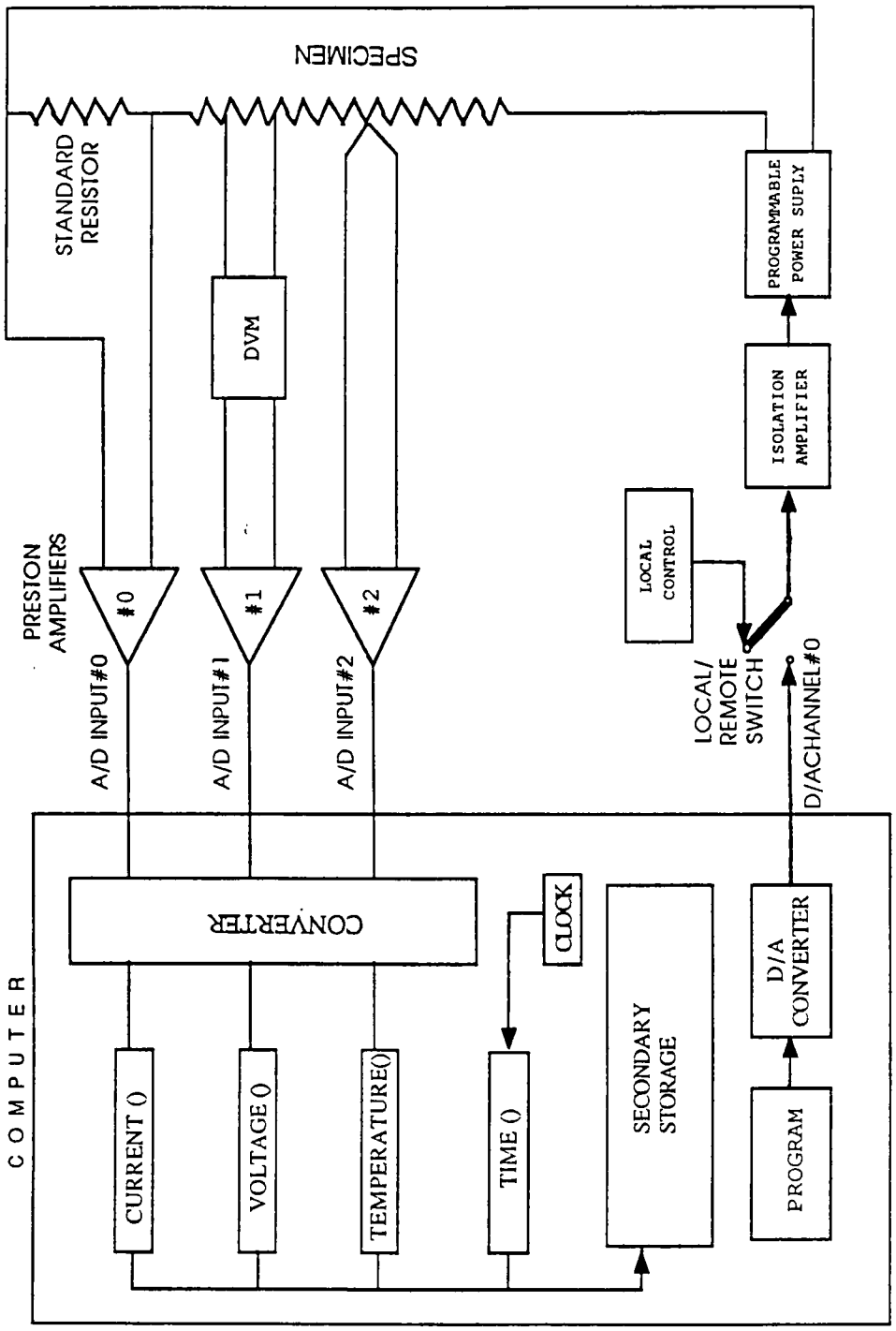


Fig. 3.15 Block diagram of the calorimeter and its acquisition system showing the computer, power supply, amplifiers, specimen and standard resistor.

desired degree. The time and current during the pre-pulse, pulse and post-pulse (see Figure 3.16) period are controlled by the computer program by sending appropriate analog signals at the appropriate times through the DAC channel#0. The power input to the specimen causes a rise in temperature. This rise in temperature causes the resistance of the specimen material to rise. Since E=IR (where E is the voltage, I is the current and R is the electrical resistance of the specimen) the rise in temperature causes the voltage drop across the effective length of the specimen to rise. The current during the pulse remains almost constant. The current, voltage and temperature signals are fed to three separate amplifiers with variable gains. The amplified outputs are then sent to the Analog-to-Digital (A/D) Converter board over the three A/D channels. The A/D converter sends these signals through DMA (Direct Memory Access) channel#s 5 and 6 which are 16-bit channels. The data are thus logged into the array variables already declared and created in the acquisition program. The speed of this acquisition is controlled by an interrupt service routine which is a part of the main acquisition. The fastest speed available with the current configuration is 1 tic, which is equal to 55 milliseconds. The following paragraphs describe what is involved in the measurement of the three input signals.

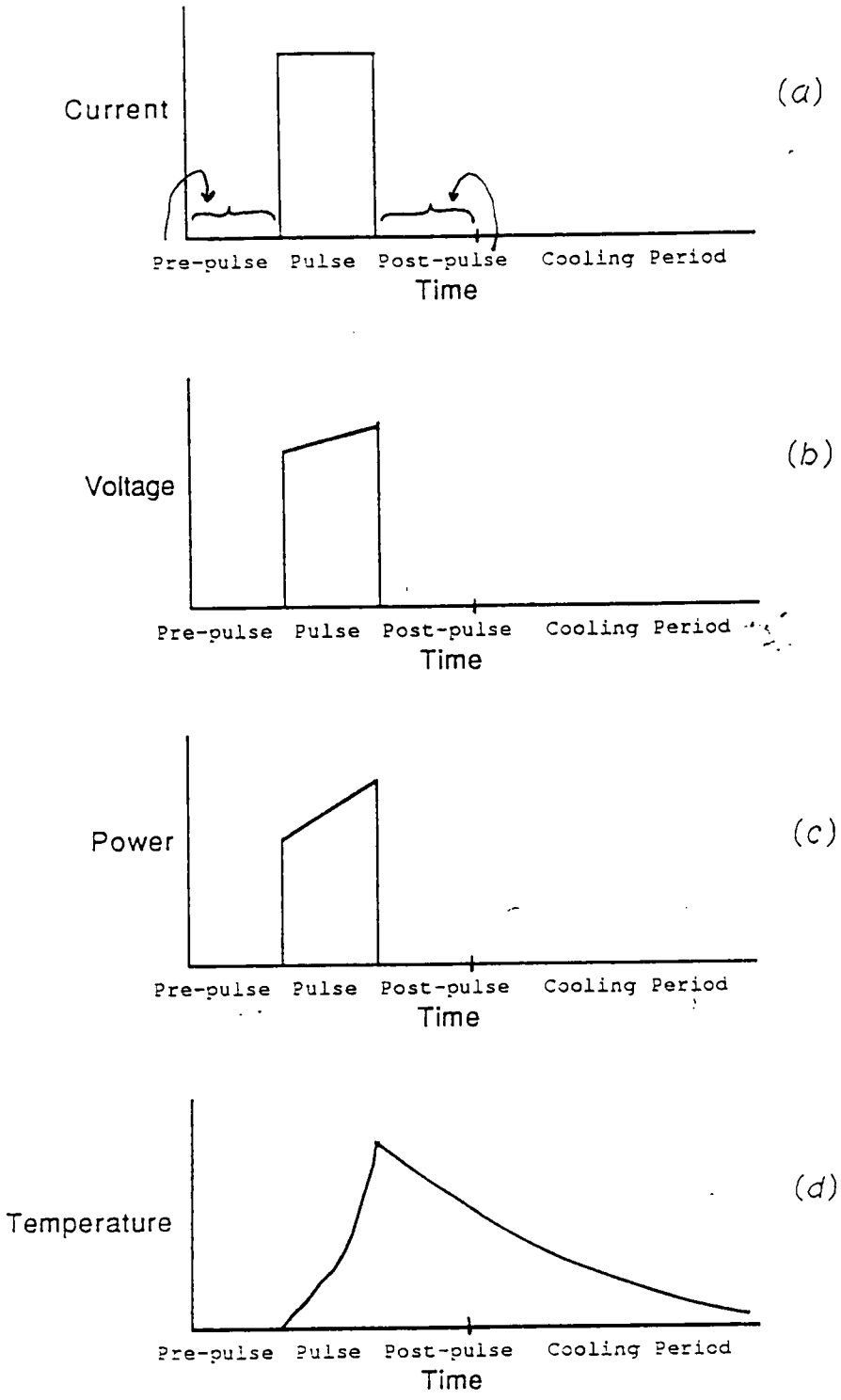


Fig. 3.16 Schematic representation of (a) Current, (b) voltage, (c) power and (d) temperature profile during a specific heat experiment.

Measurement of Current

To measure the current through the specimen the potential drop across the standard resistor, which is in series with the power supply and specimen (see Figure 3.15), is detected and sent to the D/A converter, via amplifier#0 (gain=10). The D/A converter converts the voltage signal (for current) into its corresponding digital value for the computer's internal processing. This digital value is loaded into the computer program and then converted to analog again so that it is intelligible to the viewer and so that its value, in ampere, is available to the 'Calculation Module' for computing the power, in watts. The resistance of the standard resistor, manufactured by Leeds and Northrup Company [28], is known to be [0.01 $\pm$ 0.001%] ohm and is present in the 'Calculation Module' of the acquisition program. The current through the specimen, in amperes, is obtained from the relationship I= E/R.

Measurement of Voltage

Voltage taps are welded to the specimen as shown in Figure 2.17. The length of the specimen between voltage taps marks its effective length. The voltage taps are 0.25 mm diameter nickel wires which are further attached to 0.50 mm diameter nickel wires. This is done to reduce conduction loss from the specimen through the wires.

The effective length is made much smaller than its physical length to minimize temperature gradients between the voltage taps. The voltage taps were 4.00 cm apart and the physical length of the specimen was 28 cm. The signal from the voltage taps was supplied to the input of the amplifier#1. The gain of this amplifier was set at 20. The voltage signal from the taps does not usually exceed 0.5 Volts d.c., thus a gain of 20 allows the maximum possible signal amplification without overflowing the A/D registers of the converter, which operates at a full scale of 10 Volts.

As in the case of current signals, the voltage signals are converted to digital values by the board and back to analog by the computer program. After acquisition of each data point, the product of current and voltage is computed to get the power for that data set.

Measurement of Temperature

Temperature measurement involves recording the thermoelectric voltage from the thermocouple and converting them later, using the T = f(E) relationships. Platinum vs. Platinum + 13% Rhodium thermocouples have been used for their resistance to oxidation and stability. The thermocouple has been spot welded to the specimen midway between the voltage taps. If more than one thermocouple is used then the temperature at different points along the length of the specimen can be known. This will help determine the temperature gradient, if any, in the linear direction of the

specimen. But since we were limited to three amplifiers, all of which were already being used, we could not use more than one thermocouple at a time. However, using only one thermocouple allowed us to reduce the conduction loss through the thermocouple wires. The two wires of the thermocouple were welded one on top of the other to minimize voltage pick-up due to their misalignment. The thermocouple wires pass through a two-hole ceramic tubing and are taken out from the top of a holder. They are connected to the terminals on top of the vacuum chamber. From these terminals the thermocouple wires are taken to and immersed in an ice-bath maintained at 0° C. The thermoelectric voltage is then fed through copper wires to Preston Amplifier #2, which has a fixed gain of 1000. Since the thermoelectric voltage is very small, a gain of 1000 is required to amplify the signal to the 10 Volts range and thus improve the signal to noise ratio. For example, at 960 °C a thermocouple emf of 9.978 millivolts will be boosted to 9.978 Volts. This will give a high signal-to-noise ratio and at the same time will not overflow the converter. Thus a maximum temperature of about 960 °C can be measured with the gain set at 1000. The thermoelectric voltage passes through the converter and is stored in the computer's memory and then in the hard disk in units of voltage (mV). It is used later by the MASTER program under a data array and passed to the CONVERT program where the codes for conversion to degrees Celsius are present. The correct equation for that temperature range is selected and the temperature in °C calculated.

A new data array, with temperature in °C, is formed which is passed back to the MASTER for processing.

Calculation of the Specimen Mass

It is the mass of the specimen between the voltage taps which is needed in the specific heat calculation. This mass is called the effective mass of the specimen. It is an important parameter which needs to be determined with high accuracy. The specimen mass is calculated from the specimen geometry. The specimen is in the form of a cylinder and the cylindrical effective volume is calculated from the effective length and the average diameter. Thus for accurate calculation of the specimen mass the length between voltage taps and the specimen diameter needs to be measured with high accuracy. An error analysis performed on the mass reveals that an error in the calculation of the specimen diameter of 0.01 cm causes an error of 8% in the specific heat value. The ten individual measurements of the specimen diameter are given in Table 2.2, Part II, Chapter 3.

Ten readings of the specimen diameter were taken and the average used. The specimen volume was calculated from the relationship

$$\mathcal{V}=\frac{\pi}{4}\, d^2\, \ell$$

and the specimen mass from

$$m = \frac{\pi}{4} d^2 \rho \ell$$

where,

$\mathcal{V}$ = effective volume of specimen

m = effective mass of specimen

d = diameter of specimen

ρ = density of specimen material

ℓ = effective length of specimen

The density of nickel specimen used was 8.902 gm/cm^3. This value was taken from Metals Handbook Volume 2, Properties and Selection: Non-ferrous Alloys and Pure Metals, American Society for Metals, Metals Park, Ohio. These formulae and the value of the specimen density are in the MASTER program, which calculates the specimen mass.

SPECIFIC HEAT MEASUREMENT TECHNIQUE

The single stage pulse technique was used to determine the specific heat of nickel. As described earlier (see Part I, Chapter 3), the pulse method consists of applying a single-stage, steady-state current pulse through the specimen. There is a pre-pulse and a post-pulse period which usually carries zero current through them and have been involved so that any instability in the current falls in these regions, keeping the pulse as steady as possible. Figure 3.16(a) shows a typical current pulse. The voltage and power during pulsing rise steadily as shown in Figure 3.16(b) and 3.16(c), respectively. The

current and voltage are recorded at regular intervals of time. The product of voltage and current, the power, is the rate of energy input to the specimen. Power is calculated by the computer instantaneously after each current and voltage acquisition.

$$\text{Energy Input Rate} = \text{Power (P)} = EI \qquad \ldots. (1)$$

The term 'm C_p ΔT' gives the energy absorbed by the specimen. So, the rate of energy absorption will be given by

$$\text{Rate of Energy Absorption} = m\,Cp \left(\frac{dT}{dt}\right)_{\text{Heating}} \qquad \ldots. (2)$$

The above quantity would have been equal to the energy input to the specimen if all the supplied heat were spent in raising the temperature of the specimen. Since there is a finite heat loss from the specimen, the rate of heat loss at a particular temperature needs to be determined. The rate of energy loss will be given by

$$\text{Rate of Energy Loss} = -\,m\,Cp \left(\frac{dT}{dt}\right)_{\text{Cooling}} \qquad \ldots. (3)$$

if it is assumed that the conditions in and around the specimen during cooling is the same as that during heating.

From power balance equation

$$\text{Power Input} = \text{Rate of Energy Absorption} + \text{Rate of Energy Loss}$$

$$\mathcal{E}I = m\,Cp \left[\left(\frac{dT}{dt}\right)_H - \left(\frac{dT}{dt}\right)_C\right] \qquad \ldots. (4)$$

$$Cp = \frac{\mathcal{E}I}{m\left[\left(\frac{dT}{dt}\right)_H - \left(\frac{dT}{dt}\right)_C\right]} \qquad \ldots. (5)$$

ELECTRICAL RESISTIVITY MEASUREMENT

The electrical resistivity of the specimen can be calculated from the same experimental setup. E/I gives the resistance of the specimen. From the following formula the electrical resistivity of the specimen can be calculated from the knowledge of the specimen dimensions and the electrical resistance.

$$\rho = \frac{E}{I} \frac{\pi d^2}{4 l}$$

where ρ is the electrical resistivity of specimen. If the electrical resistivity of the specimen is calculated in the same range the specific heat is calculated and if the $T=f(\rho)$ function for the specimen material is known then the calculated resistivity can be used to check the temperature. So, resistivity measurements are highly recommended. A program for measuring resistivity (CALRES.FOR) has been given in Appendix II.

CALCULATIONS

Power (watt) and thermoelectric voltage (millivolts) are recorded against time during pulsing of the specimen. These data are recovered in the Calculation program. Time units are first converted from TT# to seconds. Thermoelectric voltage in millivolts is then converted to temperature in °C, using the T=f(E) coefficients for R type thermocouples. Separate graphs are plotted between Temperature vs. Time and Power vs. Temperature. The best fit

curves for the two data sets are then computed. The best fit curve is that which gives the closest fit to a given set of data. Since the graphs of the data points superimposed with the best fit curve can be viewed on the screen, an eye estimation of the closeness of the fit can be made. But this may be erroneous. So the error function called the Sum of Residuals for several degrees of fit are compared. The degree of fit which gives the least value for the Sum of Residuals is chosen. A brief explanation of the term Sum of Residuals and the Least Square Fitting Method follows.

Sum of Residuals and the Best Fitting Line

More than one curve of a given type can be fitted to a set of data. To avoid individual judgement and eye-estimation for the determination of the best curve, it is necessary to agree to a definition of a best fit line.

To formulate a possible definition consider Figure 3.17 in which the data points are (t_1,T_1), (t_2,T_2), ... (t_n,T_n). For a given value of t, say t_1, there will be a difference between the value T_1 and the corresponding value as determined from the least squares fit curve, C. We denote this difference by d_1, which is sometimes referred to as a deviation, error or a residual, and may be positive, negative or zero. Similarly, corresponding to the values t_2, ..., t_n we obtain the deviations d_2, ..., d_n.

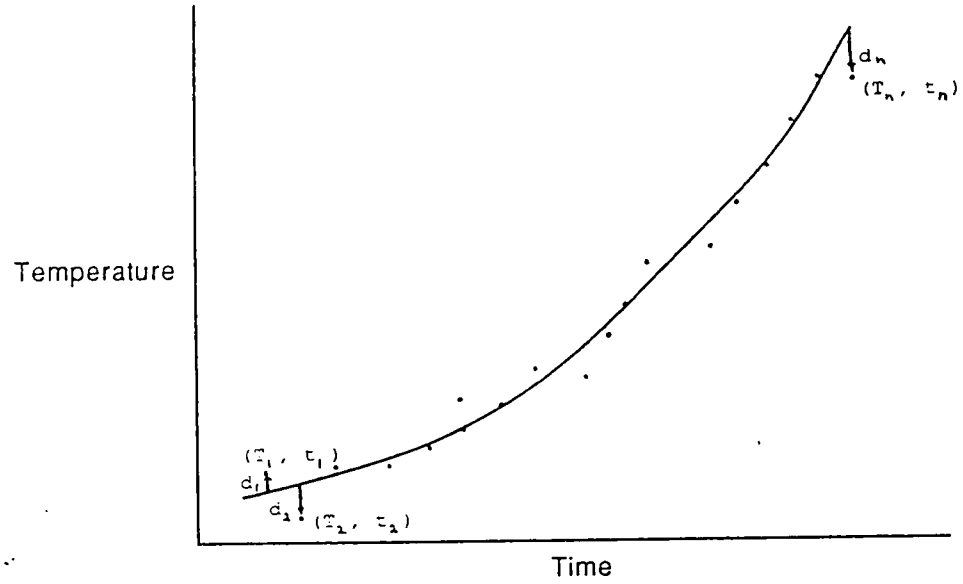


Fig. 3.17 Data points for temperature vs. time during heating shown with the best fit curve.

A measure of the 'goodness of fit' of the curve C to the given set of data is provided by the quantity $d_1^2+d_2^2+ \ldots\ldots\ldots\ldots +d_n^2$, which is called the Sum of Residuals for a given data set and the least-squares curve. The smaller the Sum of Residuals the better the fit. So, it can be summed up that of all curves approximating a given data set, the curve having a property that

Sum of Residuals = $d_1^2+d_2^2+ \ldots\ldots\ldots\ldots +d_n^2$ = minimum

is the best fitting curve [29].

Mathematical Procedure

Once the degree of fit has been determined, the coefficients are calculated. The Power-Temperature and the Temperature-Time equations are of the form

$$P = a_1+b_1T+c_1T^2+ \ldots\ldots\ldots\ldots \quad \ldots\ldots (6)$$

$$T_H = a_2+b_2t+c_2t^2+ \ldots\ldots\ldots\ldots \quad \ldots\ldots (7)$$

where,

P = Power input through the specimen

T_H = Temperature during heating

t = Time

a1, b1, ..., a2, b2, ... are coefficients of fit.

Equation (7) is differentiated with respect to time to get

$$\frac{dT_H}{dt} = b_2+2c_2t+ \ldots\ldots\ldots\ldots \quad \ldots\ldots (8)$$

Next, $\frac{dT_H}{dt}$ data are plotted against T_H and curve fitted. The resulting equation is of the form

$$\frac{dT_H}{dt} = a_3+b_3T+c_3T^2+ \ldots\ldots\ldots \quad \ldots.(9)$$

Values of Power (P) given by equation (6), and the slope $\left(\frac{dT_H}{dt}\right)$ given by equation (9) for all acquired temperature and time, are the necessary data for the specific heat equation, equation (5).

Next the cooling data are processed. Temperature during cooling (T_C) can be plotted against time.

$$T_C = a_4+b_4t+c_4t^2+ \ldots\ldots\ldots \quad \ldots. (10)$$

Equation (10) is differentiated once to get

$$\frac{dT_C}{dt} = b_4+2c_4t+ \ldots\ldots\ldots \quad \ldots. (11)$$

The calculated $\frac{dT_C}{dt}$ for each time (t) can be tabulated and plotted against T_C to get the curve for

$$\frac{dT_C}{dt} = a_5+b_5T+c_5T^2+ \ldots\ldots\ldots \quad \ldots. (12)$$

The mass is calculated using the equation

$$m = \frac{\pi}{4} d^2 \rho l \quad \ldots. (13)$$

The instantaneous power during heating (P_H), the slope of the temperature-time curve during heating (dT_H/dt), and the slope of the temperature-time curve during cooling (dT_C/dt), are now all determined as functions of temperature, given by the equations 6, 9 and 12, respectively. When the operator enters the temperature of interest, the quantities P_H, dT_H/dt and dT_C/dt are calculated for that temperature and substituted into the specific heat equation :

$$Cp = \frac{\mathcal{E}I}{m\left[\left(\frac{dT}{dt}\right)_H - \left(\frac{dT}{dt}\right)_C\right]} \qquad \text{.... (14)}$$

The value of the calculated specific heat at the entered temperature is displayed on the screen.

PART IV
RESULTS AND DISCUSSIONS

PART IV: CHAPTER 1
RESULTS AND DISCUSSION

ANALYSIS OF DATA FOR NICKEL

This chapter deals with the results of the specific heat experiments conducted on the pulse heating calorimeter. The discussion includes effect of fitting intervals near the Curie point, effect of current reversal and effect of degree of fitting. Data presentation and error analysis are also included.

Effect of fitting intervals near the Curie point

The specific heat versus temperature data is discontinuous at the Curie point of nickel (360 °C) and so the data from which C_p is calculated are fitted separately on either side of the Curie point, and so are the calculated specific heat data. Also, since the general curvature and slope of the specific heat curve is different on both sides of the Curie point, the nature of curve fitting on the two sides will be different. Also improper determination of the Curie temperature will have different effects on the calculated specific heat curves on the two sides. A discussion of the data analysis for the specific heat of nickel on both sides of the Curie point is presented here.

Specific heat is a measure of the change in temperature of a

specimen with a corresponding addition of heat. Whatever method is applied to measure this amount of heat, the measurement has to be done while the temperature is transient. In pulse calorimetry, the change in temperature is attained by constant application of electrical power. This requires the temperature of the specimen to be determined as a function of time. The estimation of the function $T=f(t)$, where T is temperature and t is time, may become extremely difficult at the critical temperature of a second order transformation since T is a very sensitive function of time in this region.

Also, the fact that the specimen has a temperature gradient in the axial direction affects the measured specific heat near the Curie point. Since the thermocouple is fixed on the surface near the center of the specimen, it only gives the temperature of the center. But, when the center has attained the Curie temperature the rest of the specimen may be at a lower temperature. Also, the temperature at the center of cross-section of the specimen will be higher than the temperature at the surface. These factors affect the value of the calculated specific heat. If we knew the temperature gradient profile from the center to each side of the specimen, we would know the temperature range in which the specific heat is actually affected. However, the temperature gradient was not determined in our experiment because this would require more than one thermocouple to be attached to the specimen and a corresponding number of amplifiers, which we did not have available to us.

To quantitatively determine the effect of interval limits near

the Curie point, on the specific heat of nickel, the C_p vs. T data was fitted with three different upper limits below T_C and with two different lower limits above T_C.

Below the Curie point

Figure 4.1 shows the plots of specific heat versus temperature data for temperatures below the Curie point. The three plots are for three different fitting intervals as shown in the legend. These curves are called Curve I, Curve II and Curve III. Each fitting interval not only indicates the interval in which the specific heat data have been fitted but also the interval in which the P=(T), T=(t), dT/dt=f(T) data during heating and T=f(t), dT/dt=f(T) data during cooling have been fitted. The Sum of Residuals and number of data points for each interval and the degree of fit for each function is given in Table 4.1. Taking the interval 100-360 °C as a reference, when the upper limit of fit is decreased, the specific heat curves go up at higher temperatures and come down at lower temperatures. At about 180 °C the curves cross each other. The specific heat near this temperature is not sensitive to the fitting interval. Another observation is that at higher temperatures (e.g., 275 °C) the difference in specific heat, on changing limits, is more than that at lower temperatures (e.g., 100 °C).

A comparison shows that at 275 °C, reducing the upper limit of fitting from 360 °C (Curie point) to 320 °C increases the specific heat from 0.540 to 0.544 J/gm-°C. This is a difference of 0.8%. When the upper limit of fitting is further reduced to 280 °C, the specific heat

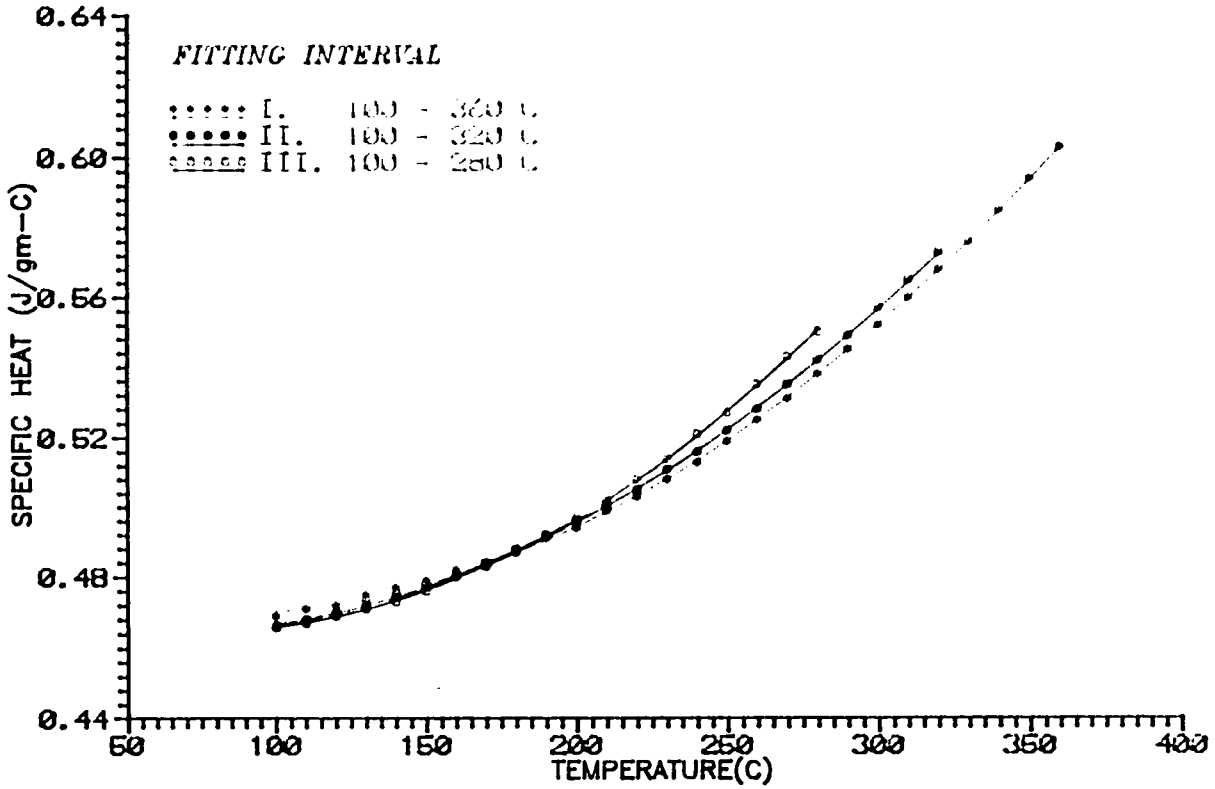


Fig. 4.1 Effect of changing the upper limit of fit on the specific heat versus temperature curve, below the Curie point.

Table 4.1 Sum of Residuals and number of data points for the five functions for different temperature intervals. The degree of fit for each function is given in brackets in column 1

Functions (Degree)	**Curve I 100 - 360°C**	**Curve II 100 - 320 °C**	**Curve III 100 - 280 °C**
	Sum of Residuals		
$P=f(T)_H$ (2)	1.0197	0.661859	0.461393
$T=f(t)_H$ (2)	54.4135	38.5426	28.6179
$dT/dt=f(T)_H$ (2)	0.0289116	0.0194469	0.0144322
$T=f(t)_C$ (3)	193.135	82.5821	23.0171
$dT/dt=f(T)_C$ (4)	0.000599067	0.000229393	0.000135694
	No. of data points		
$P=f(T)_H$ (2)	194	170	144
$T=f(t)_H$ (2)	194	170	144
$dT/dt=f(T)_H$ (2)	194	170	144
$T=f(t)_C$ (3)	422	385	337
$dT/dt=f(T)_C$ (4)	422	385	337

rises to 0.550 J/gm-°C, a difference of 1.9% from Curve I. As mentioned earlier, the specific heat at lower temperatures is also affected. At 100 °C the difference between curves I and III is 1.3%, but in this case the change is negative.

There is no way by which one can conclude from the graphs which one of these curves is the most realistic. For that, one has to refer to the Sum of Residuals of fit (explained in Part III, Chapter 5), and compare its value for the different intervals. Table 4.1a shows the Sum of Residuals for the three curves fitted with different intervals for the five functions. In Part III, Chapter 5, we had concluded that the curve having the least value of the Sum of Residuals is the best fit curve. But here we cannot directly compare the Sum of Residuals because different intervals contain different number of data points in them. Other things being equal, the larger the number of data points the greater will be the Sum of Residuals, which is the sum of squares of the differences between the individual data points and the best fit curve. For a fair comparison of the 'goodness of fit' the Sum of Residuals in each interval should be divided by the total number of data points in that interval, and the quotient compared. To this, the term Mean Sum has been defined as the Sum of Residuals divided by the number of data points in the interval. So,

$$\text{Mean Sum} = \frac{\text{Sum of Residuals}}{\text{Number of Data Points}}$$

Table 4.2 shows the Mean Sum for the different fitting intervals for the different functions. Comparing the Mean Sum for the five

different functions shows that below the Curie point, the smaller the fitting interval the smaller the Mean Sum. For the slope data, which are most sensitive to fitting intervals, Mean Sum varies significantly

Table 4.2 Mean Sum of fit for the five functions for different temperature intervals, where Mean Sum has been defined as Sum of Residuals / Number of data points in the interval. The degree of fit for each function is given in brackets in column 1.

Functions (Degree)	**Curve I 100 - 360°C**	**Curve II 100 - 320 °C**	**Curve III 100 - 280 °C**
$P=f(T)_H$ (2)	5.256×10^{-3}	3.893×10^{-3}	3.204×10^{-3}
$T=f(t)_H$ (2)	0.2784	0.2267	0.1987
$dT/dt=f(T)_H$ (2)	1.490×10^{-4}	1.144×10^{-4}	1.002×10^{-4}
$T=f(t)_C$ (3)	0.4577	0.2145	0.0683
$dT/dt=f(T)_C$ (4)	14.196×10^{-7}	5.955×10^{-7}	4.023×10^{-7}

with fitting intervals. For $dT/dt=f(T)_H$, going from 360 to 280 °C as the upper limit, the Mean Sum changed from 1.490×10^{-4} to 1.002×10^{-4}, a percentage change of 32%. For dT/dt=f(T) for cooling, Mean Sum for curves I and III shows a difference from 14.196×10^{-7} to 4.023×10^{-7}, a 71% difference. This decrease in the Mean Sum with decrease in the fitting interval size is also evident for the other functions. From these observations it can be concluded that smaller intervals of fit will give better 'goodness of fit' and the 'best fit' curve will be a better representation of the data set, provided there are enough data points in the interval to fit the data well. So, for

example, it would be better to fit the data over an interval from 100 to 280 °C and then from 280 to 360 °C separately than to fit the whole data set with a single curve. Fitting the data with more number of smaller intervals had a better effect on the 'goodness of fit'. But, there will be a limiting case beyond which smaller interval will not give a better fit. This has not been tried in our analysis.

Above Curie point

Figure 4.2 shows two specific heat versus temperature curves fitted in two different intervals, above T_C. The reader is reminded here that the indicated interval of fit is not only for fitting specific heat data but is also valid for the five functions which are involved in the determination of specific heat. The same degrees of fit were used. The upper limit has been fixed at 480 °C. The two curves are for two different lower limits, 360 and 380 °C. Making curve I (360-480 °C) the reference, it is observed that at 380 °C the specific heat changes from 0.519 to 0.517 J/gm-°C when the lower limit of fit is moved 20 °C away from T_C (curve II). This is a percentage difference of 0.4%. At temperatures near the Curie point the specific heat curve is lower, the smaller the fitting range. But at higher temperatures (e.g., 480 °C) the effect is reversed and specific heat curve is higher, the smaller the fitting range. At 480 °C, the the specific heat goes up by 0.4% from curve I to curve II. This reverse effect was also observed for specific heat data fitted below T_C.

Contrary to what was expected for the $P=f(T)_H$ and $T=f(t)_C$, the

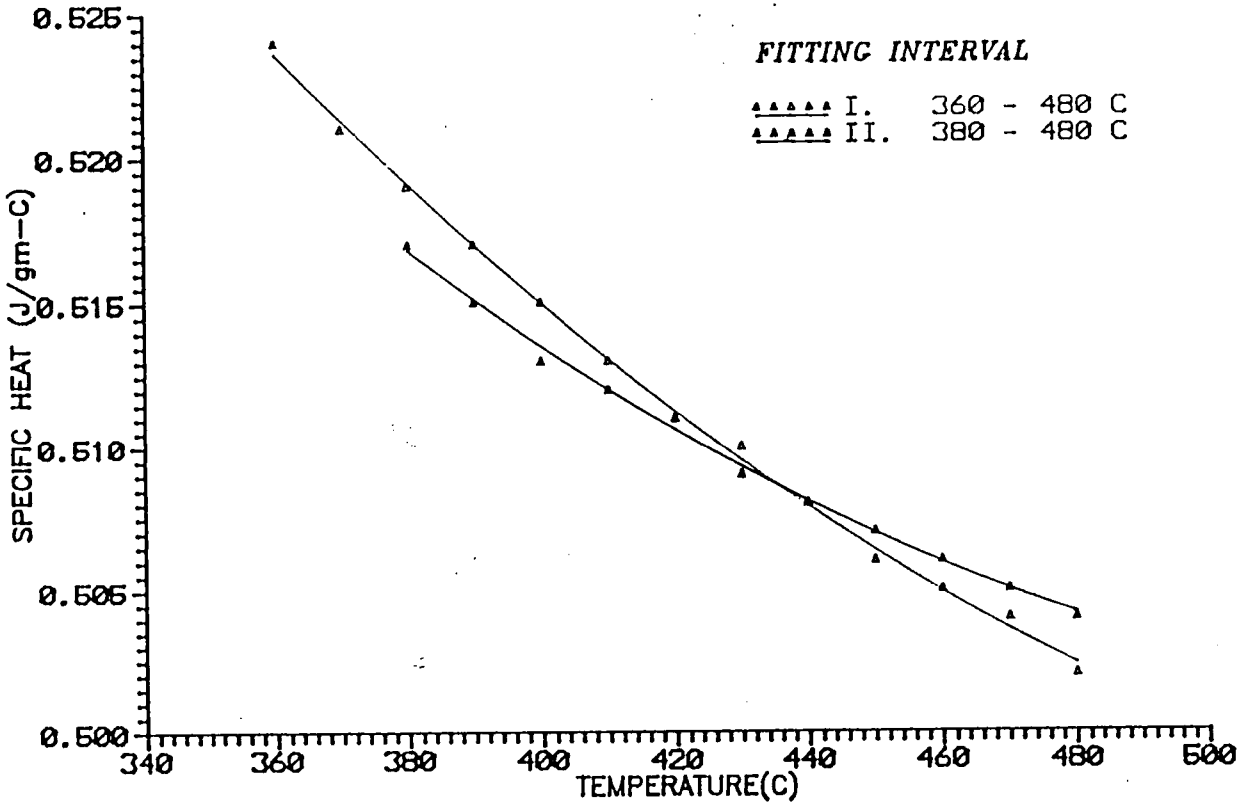


Fig. 4.2 Effect of changing the lower limit of fit on the specific heat versus temperature curve, above the Curie point.

Mean Sum increased when the interval of fit was decreased from 360-480 °C to 380-480 °C. But the functions $T=f(t)_H$, $dT/dt=f(T)_H$ and $dT/dt=f(T)_C$ showed a decrease in the Mean Sum. Table 4.3 summarizes the Mean Sum of fit for the five functions, in this range. It is difficult to come out with a definite conclusion regarding the determination of the intervals of fit in this range since the changes in Mean Sum do not follow the same trend for all the functions with changes in the interval limits.

Table 4.3 Mean Sum of fit for the five functions for different temperature intervals. The degree of fit for each function is given in brackets in column 1.

Functions (Degree)	**Curve I 360 - 480 °C**	**Curve II 380 - 480 °C**
$P=f(T)_H$ (2)	6.918×10^{-3}	7.068×10^{-3}
$T=f(t)_H$ (2)	0.1167	0.1128
$dT/dt=f(T)_H$ (2)	2.583×10^{-5}	1.692×10^{-5}
$T=f(t)_C$ (3)	0.1275	0.1304
$dT/dt=f(T)_C$ (4)	7.8425×10^{-5}	3.918×10^{-5}

Effect of current reversal

If there is no voltage pickup by the thermocouple welded to the specimen, then reversing the direction of current going into the specimen should not affect the thermoelectric voltage from the thermocouple and the specific heat determination. But even with

utmost care during welding of the thermocouple, it is almost impossible to eliminate the axial misalignment of the two thermocouple wires with respect to each other. When the direction of the current is reversed this voltage pick-up has a reverse effect on the thermocouple voltage. The effect, off course, shows up in the calculated specific heat. The pick-up in our case was of the order of 7 μV, at room temperature.

Figure 4.3 shows the specific heat vs. temperature curves plotted between 100 and 280 °C and between 400 and 480 °C. Curve I is for current flowing in one direction and curve II is for current flowing in the other direction. The Curie point occurs at 360 °C, so the curves have been fitted and presented in the regions sufficiently removed from 360 °C so that the effect of the Curie point on these curves is insignificant. Changing the current from one direction to the other will raise or lower the specific heat at all temperatures. Below the Curie point this change is more the lower the temperature. Above the Curie point, this change in specific heat is more too, the lower the temperature. This difference is more at lower temperatures because at lower temperatures the ratio of the voltage pick-up from the thermocouple to the thermoelectric voltage is higher. At higher temperatures the voltage pick-up is more because the resistance of the specimen is more, and the thermoelectric voltage is also significantly higher. But their ratio is less, so the net effect on the pick-up is less. At 100 °C the difference in the specific heat between curves I and II is 7.1%. At 480 °C this difference decreases to 5.1%.

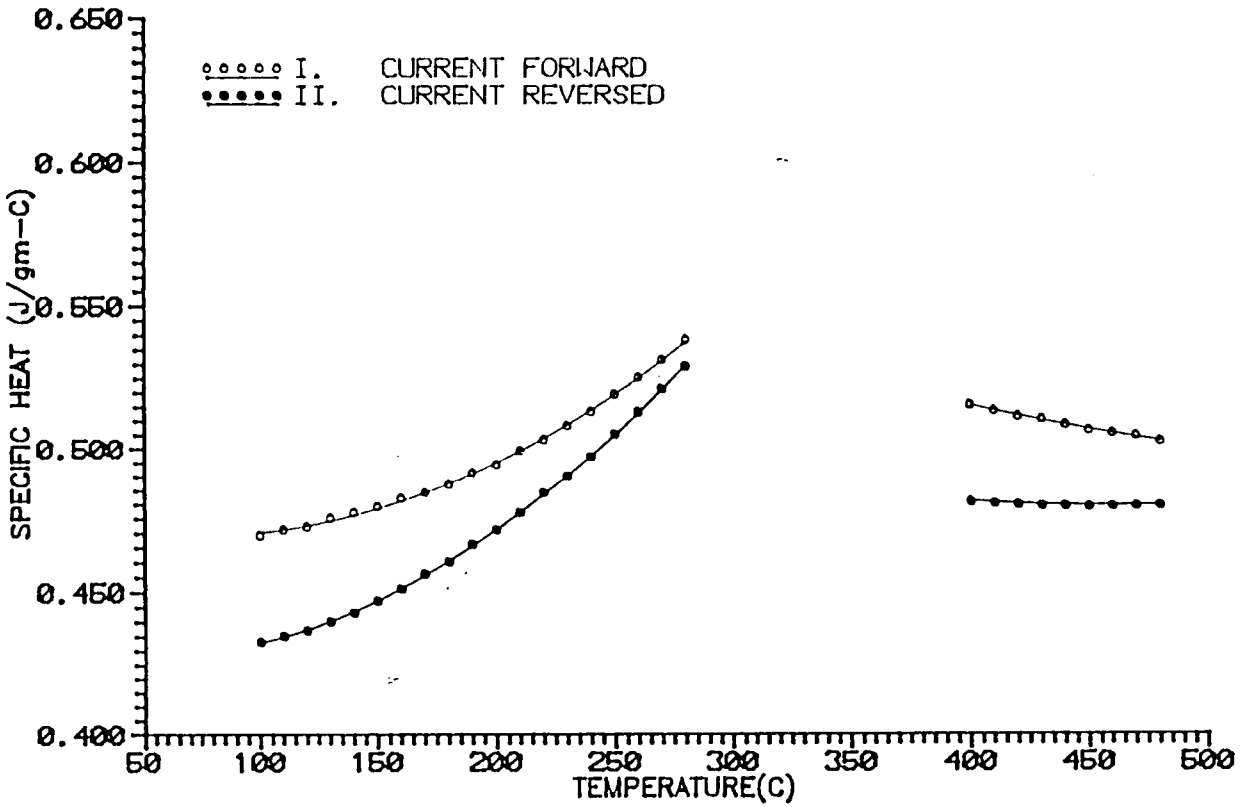


Fig. 4.3 Effect of current reversal on the specific heat versus temperature curve.

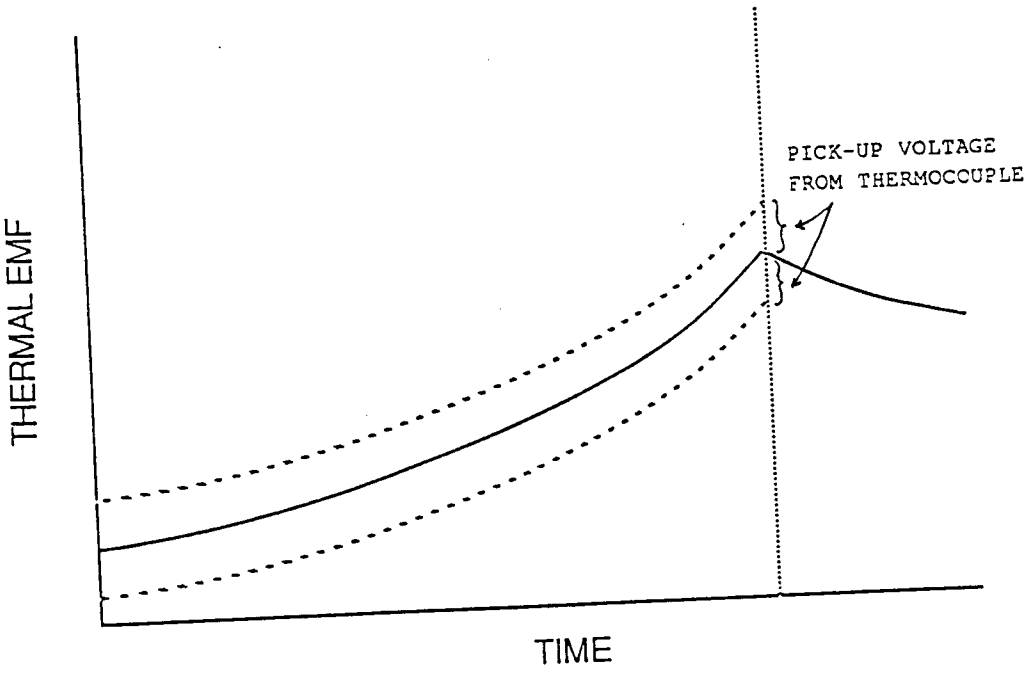


Fig. 4.4 Curves with solid lines are for thermal emf versus time during heating and cooling unaffected by voltage pick-up. The two curves with broken lines are for thermal emf versus time, during heating, with positive and negative voltage pick-up.

Figure 4.4 shows a schematic of thermal emf versus time curve for current in either direction, along with the thermal emf versus time curve for cooling. The curve shown with solid line for heating is the one with no voltage pick-up. The curve higher than this one, shown with broken line, is the one with the influence of positive pick-up and the curve below, shown with broken line, is the one with the influence of negative pick-up. Since there is no current flowing through the specimen during cooling there will be no voltage pick-up. From the difference in the thermal emf between heating and cooling curve at the current shut off time the voltage pick-up can be estimated. However, this pick-up is not constant throughout and decreases with temperature due to the decrease in the resistance of the specimen. Since, the specimen resistance decreases with temperature, so a constant current yields a lower IR drop at lower temperature.

Figure 4.5 is an exaggerated schematic of thermal emf versus time curves for heating, which shows how thermal emf versus time curve affected by voltage pick-up can cause an error in the calculated slope. The curve with the solid line shows the true thermal emf while the curves with broken lines show the thermal emf for the current flowing in either direction. At an emf corresponding to temperature T_1, notice how the positive and negative pick-up decreases and increases the slope of the temperature vs. time curve. This error in slope causes an error in the calculated specific heat.

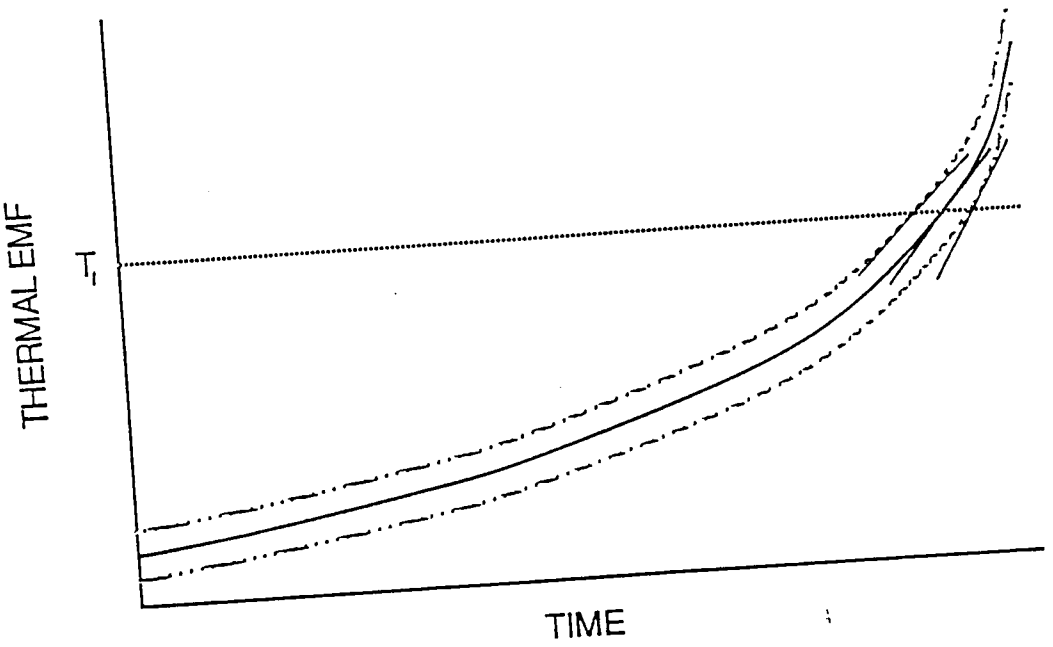


Fig. 4.5 Exaggerated schematic of thermal emf versus time curve, showing the effect of voltage pick-up on the determined slope of the curve.

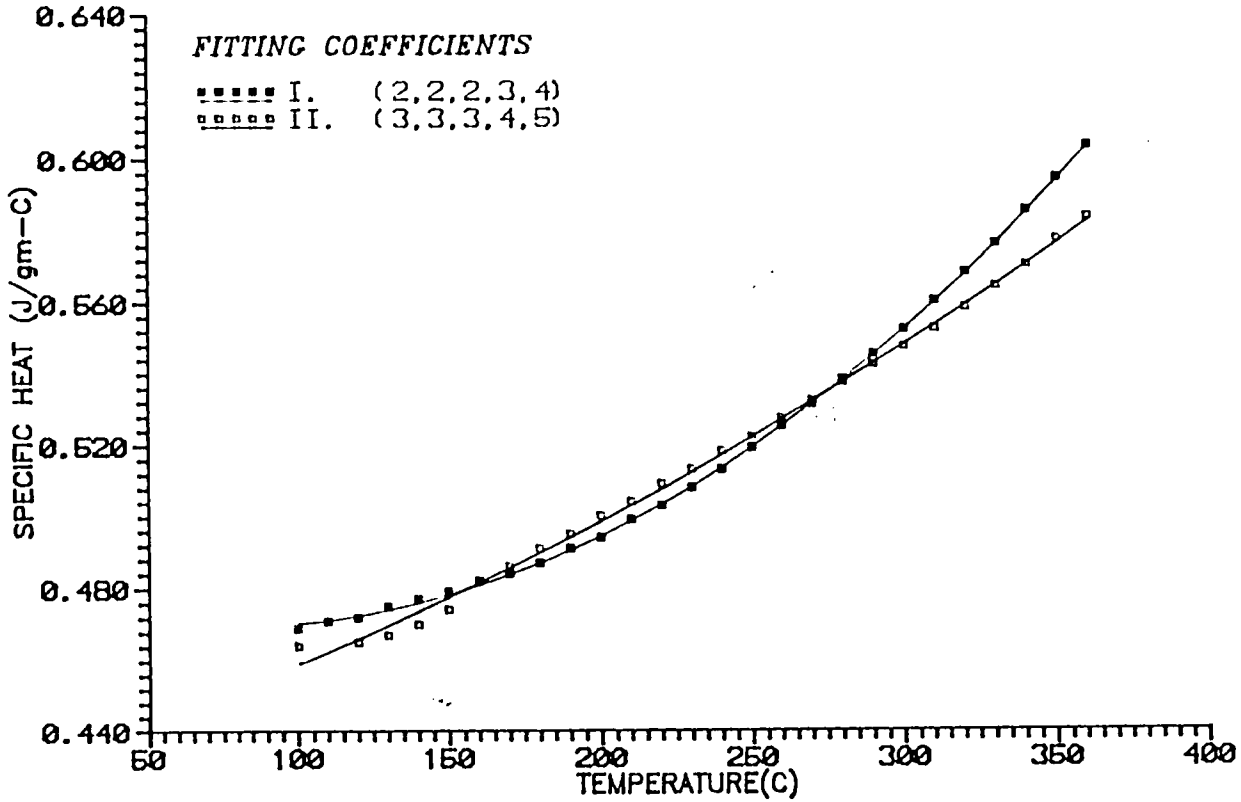


Fig. 4.6 Effect of increasing the degree of fit for all five functions used for the determination of specific heat, on the resultant specific heat curve.

Effect of the degree of fitting

Figure 4.6 shows the two specific heat curves fitted in the same temperature interval, 100-360 °C, with a different set of coefficients. For curve I, all the three functions for heating have been fitted to a second degree polynomial of the form $a+bx+cx^2$. The T=f(t) curve, for cooling, has been fitted to third degree and the dT/dt=f(T) curve, for cooling, has been fitted to fourth degree. The specific heat versus temperature curve obtained from these five curves is fitted to a second degree polynomial. Curve II has been plotted using the five functions $P=f(T)_H$, $T=f(t)_H$, $dT/dt=f(T)_H$, $T=f(t)_C$ and $dT/dt=f(T)_C$, which were fitted with a polynomial one degree higher than that of the corresponding functions for curve I. The specific heat versus temperature data in Curve I and Curve II have both been fitted to a second degree polynomial.

Since we have kept the limits of fit for the two curves the same and used the same degree of fit for the polynomial for fitting the specific heat versus temperature data, we can compare the effect of fitting the five functions with an increased degree of fit. The degree of fit is given in brackets in the legend of Figure 4.6 in the order $P=f(T)_H$, $T=f(t)_H$, $dT/dt=f(T)_H$, $T=f(t)_C$ and $dT/dt=f(T)_C$. It is evident from the curves that fitting all the above mentioned functions to a higher degree lowers the curvature of the specific heat versus temperature curve. The maximum difference in specific heat due to this higher degree of fitting occurs at the Curie point where the

difference is 3.8%. A comparison of the Sum of Residuals of fit for the functions from Table 4.4 clearly shows that for a higher degree of fit the Sum of Residuals is smaller for curve II, except for the function dT/dt_c. This may lead the experimenter to believe that curve II is more accurate.

Table 4.4 Sum of Residuals of fit for the same data fitted with different degrees of fit, in the same interval.

Functions	**Curve I** **100 - 360 °C**	**Curve II** **100 - 360 °C**
$P=f(T)_H$	1.0197	0.97601
DEGREE:	2	3
$T=f(t)_H$	54.4135	34.2347
DEGREE:	2	3
$dT/dt=f(T)_H$	0.0289166	0.0187214
DEGREE:	2	3
$T=f(t)_C$	193.135	34.3316
DEGREE:	3	4
$dT/dt=f(T)_C$	0.000599067	0.000864656
DEGREE:	4	5

It might not be so, because fitting a set of data with too high a degree than that which the data actually represents, may cause unrealistic curvature in the best fit curve. Sometimes, just by viewing the graph such unrealistic curvatures are revealed. It was

found out by visual inspection that a second degree polynomial was sufficient to fit the functions $P=f(T)_H$, $T=f(t)_H$, $dT/dt=f(T)_H$. This was further verified by observing that for $dT/dt=f(T)$, for example, increasing the degree of fit from first degree to second degree decreased the Sum of Residuals by 30%, whereas, increasing the degree of fit from second degree to third degree decreased the Sum of Residuals by an amount which is less than 1% of the Sum of Residuals for the first degree. This is summarized in Table 4.5.

Table 4.5 Percentage change in the Sum of Residuals with change in the degree of fit for the function $dT/dt=f(T)_H$

Change in degree of fit	% Change in Sum of Residuals based on degree 1
1 to 2	-30 %
2 to 3	-1 %

Making certain that the degree of fit for the three functions for heating should be 2, we next studied the effect of increasing the degree of fit for the cooling data only. The degree of fit for the three 'heating' functions were kept at 2, while the degree of fit for the $T=f(t)_C$ data was increased from 3 to 4 and for $dT/dt=f(T)_C$ data from 4 to 5. The specific heat data calculated from these functions in the range 100-280 °C are shown in Figure 4.7. Between 180-280 °C the data seem to agree well (<0.5% difference). Below 180 °C the difference starts increasing, as the slope of curve II (calculated from cooling functions fitted with a higher degree) keeps decreasing till it

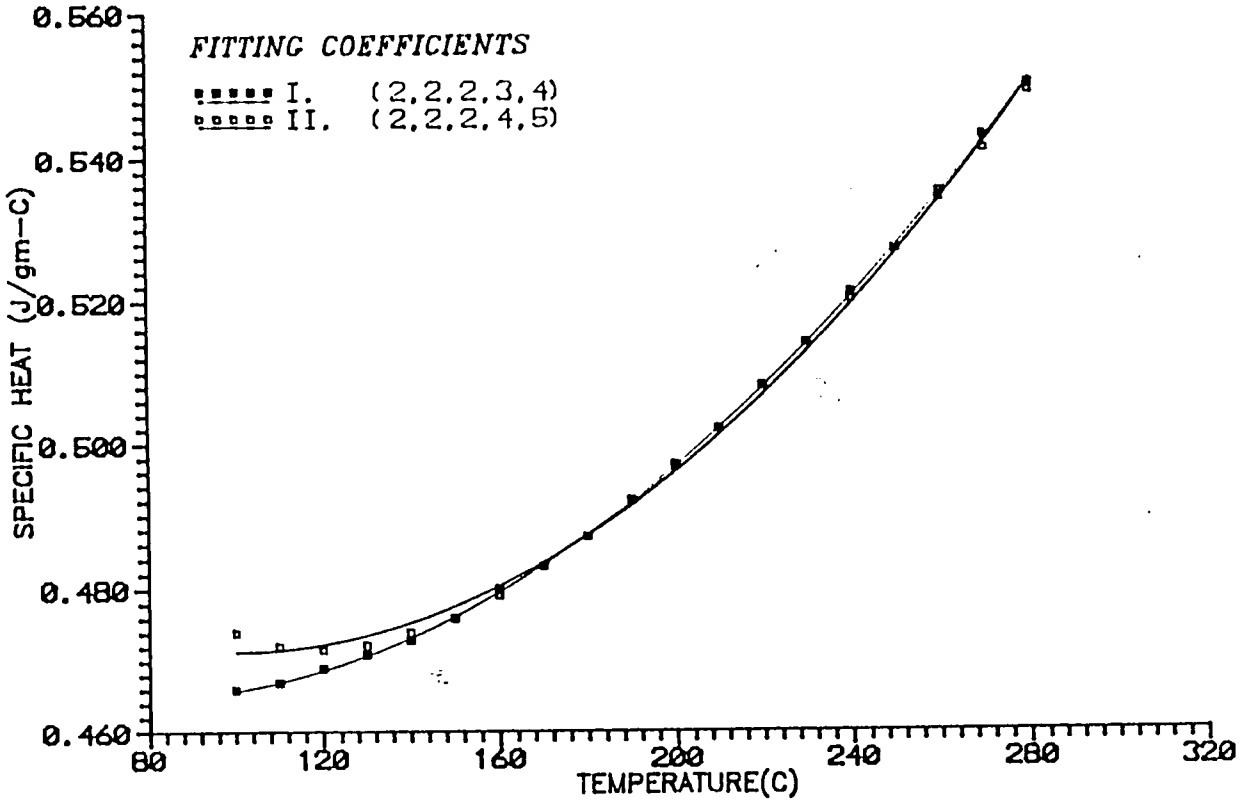


Fig. 4.7 Effect of increasing the degree of fit for the two functions for cooling ($T=f(t)_C$ and $dT/dt=f(T)_C$) only, on the resultant specific heat curve.

reaches about 120 °C, whereafter the slope is negative. This negative slope does not show up in the best fit curve because only a second degree polynomial has been used to fit the data, but the negative slope is evident from looking at the data points. We know from the specific heat data for nickel established earlier that such a negative slope in the lower temperature region (e.g., 100 °C) is just incorrect. Also, by comparing the Sum of Residuals for 4^{th} and 5^{th} degree fit for the function $dT/dt=f(T)_C$, from Table 4.4, it is observed that the Sum of Residuals for 4^{th} order is less than that for 5^{th} order. This proves that 4^{th} order is a better fit. If the data in Figure 4.7 is extrapolated below 100 °C, the negative slope of the curve would keep on increasing which is contrary to the data for nickel established earlier [5,30].

Thus, for the determination of accurate specific heat data the experimenter should make the correct choices about the degree of fit, intervals of fit, number of intervals to fit, and about other statistical parameters. Data analysis, thus, forms an important part of specific heat determination.

DATA PRESENTATION

Data for specific heat at constant pressure for nickel determined by the author from 100 to 480 °C, using the designed pulse calorimeter, is presented in Figure 4.9, along with the data in the same range determined by Meschter, Wright, Brooks and Kollie

Temperature (°C)	Specific Heat (J/gm-°C)
100	0.469
110	0.471
120	0.472
130	0.475
140	0.477
150	0.479
160	0.482
170	0.484
180	0.487
190	0.491
200	0.494
210	0.499
220	0.503
230	0.508
240	0.513
250	0.519
260	0.525
270	0.531
280	0.538
290	0.545
300	0.552
310	0.560
320	0.568
330	0.576
340	0.585
350	0.594
360	0.603
360	0.524
370	0.521
380	0.519
390	0.517
400	0.515
410	0.513
420	0.511
430	0.510
440	0.508
450	0.506
460	0.505
470	0.504
480	0.502

Fig. 4.8 Tabulation of author's data on the specific heat of nickel in the range 100 - 480 °C.

[30]. Both pulse and adiabatic calorimeters have been used by Meschter et al. to measure the specific heat of nickel. The specific heat data presented by the author have been fitted in the intervals 100-360 °C and 360-480 °C from the functions $P=f(T)_H$, $T=f(t)_H$, $dT/dt=f(T)_H$, $T=f(t)_C$, $dT/dt=f(T)_C$, which were also fitted in the same intervals with degrees 2, 2, 2, 3 and 4 respectively. The author's data for specific heat are tabulated in Figure 4.8 and are plotted in Figure 4.9. They were calculated from functions fitted in the interval 100-480 °C, then the calculated specific heat data were curve fitted with a second degree polynomial. In the 100-320 °C range the average difference between the author's data and Meschter et al.'s data is 5%. The author's curve is the one which has been influenced by positive voltage pick-up. If the curve with negative voltage pick-up was compared then the difference between the the two data sets would have further increased (se Figure 4.3. Both investigators have determined the Curie point to be 360 °C. There is a considerable difference in the specific heat of both ferromagnetic and paramagnetic nickel at 360 °C. Kollie had a better resolution of data near the Curie point because he recorded data at the speed of 2000 readings/second, whereas, the author's data were recorded at a speed of 9 readings/second. Kollie, with his higher resolution, could determine the slope of the specific heat curve with better accuracy, approaching the Curie point from both sides. For temperatures considerably higher than the Curie point, e.g., 375 °C, the average difference between the data sets is 4%. The difference in

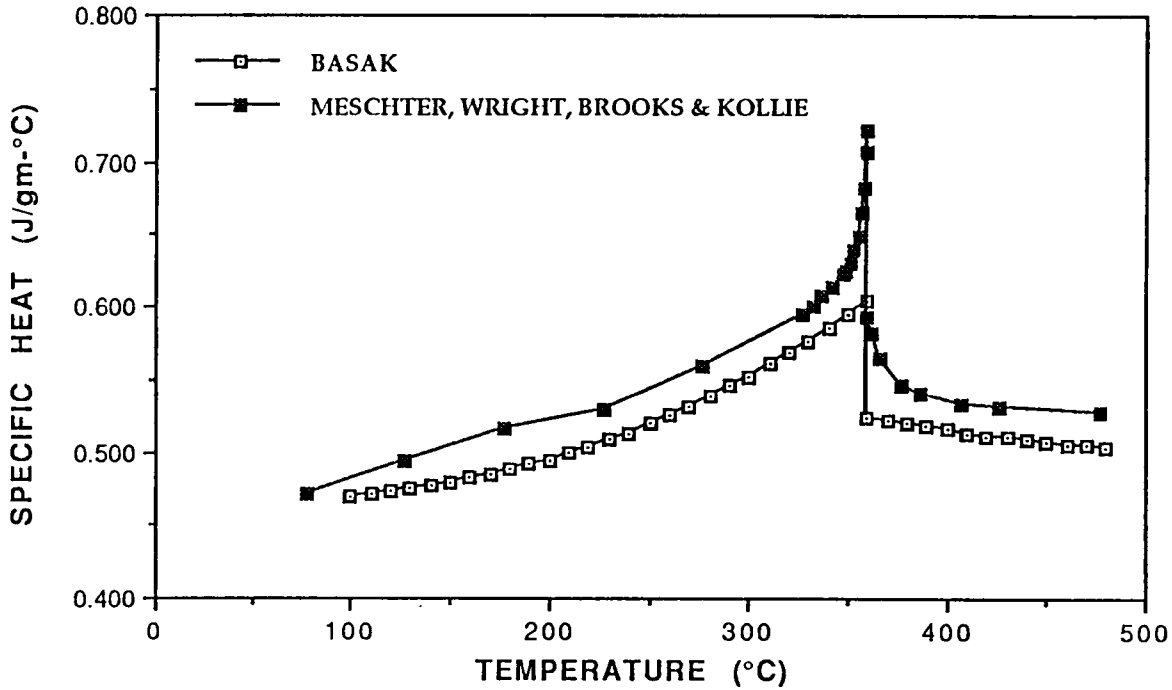


Fig. 4.9 Comparison of author's data on the specific heat of nickel with Meschter et al.'s data, in the range 100 - 480 °C.

discontinuity between the two data at the Curie point is 0.120 J/gm-°C, for ferromagnetic nickel. This is a difference of 16%.

ERROR ANALYSIS

The total error in the calculated specific heat at any temperature is due to the combined effects of errors accruing from experimental and physical factors. Some of the important factors which can significantly affect the accuracy of the calculated specific heat will be discussed in the next few paragraphs.

Before the specimen is inserted into the specimen holder, which goes into the vacuum chamber, the effective length and diameter of the specimen needs to be measured. The error in mass is the summation of the errors in length, diameter squared and density. The specific heat at constant pressure can be expressed as

$$C_p = \frac{4\,\mathcal{E}\,I}{\rho\ \pi\ d^2\ l\left[\frac{dT}{dt_H}\ .\ \frac{dT}{dt_C}\right]} \qquad \ldots. (1)$$

In order to come up with a quantitative total percentage error in the calculated specific heat one needs to evaluate the errors due to all the terms given in the equation above.

Error in mass

Mass of the specimen, m, can be expressed as $m=\rho\ \pi\ d^2\ l/4$. To find out the total error in mass, the errors in l, d^2, and ρ need to be

estimated and summed up.

The maximum error in the measured length is 1.25 %. By using knife edge method the effective length of the specimen can be measured with better accuracy. The area of contact between the knife edge and the specimen is extremely small and can accurately span the effective length. The knife edge can measure the resistance of the effective specimen. Then, from the knowledge of the resistivity of the specimen material and the specimen diameter the effective length can be precisely determined.

To calculate the errors in d^2 we must first calculate d. The ten individual measurements of diameter are tabulated in Table 2.2. Figure 4.10 is a plot of these measured diameters. The non-uniformity of the specimen is, at once, evident. Also, the plot indicates that the specimen has a tapered middle portion and bulges out toward the end. Since, the specimen has been assumed to be cylindrical this non-uniformity is the source of considerable error in diameter. The arithmetic average of these ten readings were computed and is shown in Figure 4.10. This computer average value of diameter has been used in the computer program to calculate the specimen mass. The values of the measured diameter were entered in the LEASTSQR program and fitted with degree 0. This gave the arithmetic average of the diameter to be 0.0943 inch. The % difference and difference were also computed. These values are tabulated in Figure 4.11. The variance, as computed by LEASTSQR, is 3.5454×10^{-6}. This calculated value of variance is based on the

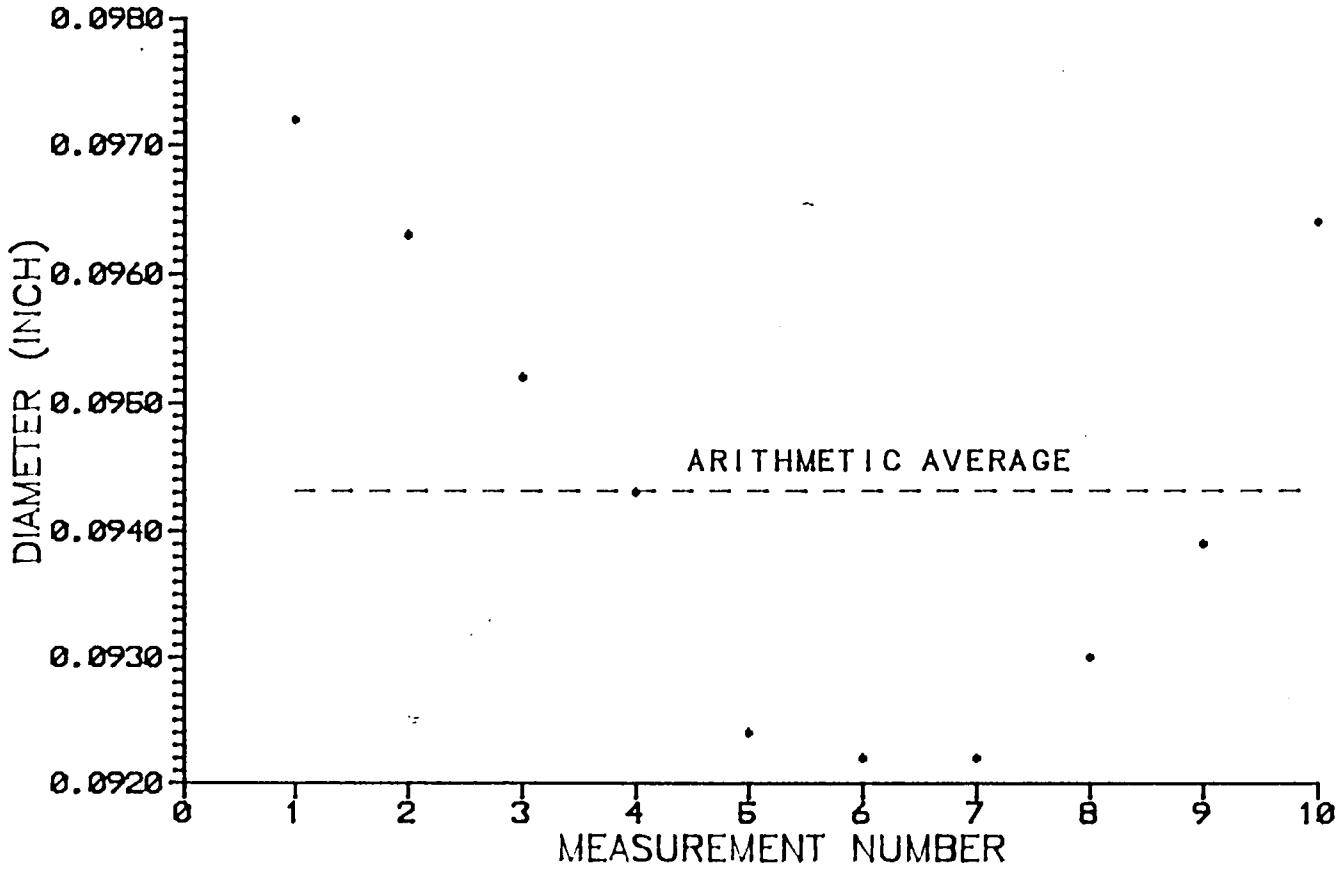


Fig. 4.10 Plot of the ten measured specimen diameters, showing the arithmetic average.

	X	Y	YCALC	% DIFF	DIFF
1)	1.0000	0.0972	0.0943	2.9733	0.0029
2)	2.0000	0.0963	0.0943	2.0665	0.0020
3)	3.0000	0.0952	0.0943	0.9349	0.0009
4)	4.0000	0.0943	0.0943	0.0106	-0.0000
5)	5.0000	0.0924	0.0943	2.0671	-0.0019
6)	6.0000	0.0922	0.0943	2.2885	-0.0021
7)	7.0000	0.0922	0.0943	2.2885	-0.0021
8)	8.0000	0.0930	0.0943	1.4086	-0.0013
9)	9.0000	0.0939	0.0943	0.4366	-0.0004
10)	10.0000	0.0964	0.0943	2.1680	0.0021

VARIANCE : 3.545444D-06

Fig. 4.11 Printout from the program LEASTSQR showing the ten measured diameters (inch) under column Y, the calculated value of the arithmetic average under column YCALC, the % difference, the difference and the variance.

formula $v = \frac{\Sigma (y - \bar{y})^2}{n-1}$, where v is variance, y is dependant variable, $\bar{y}$ is arithmetic mean and n is the number of data points.

The values of the measured diameter were also entered into GRAPHER. The computed average was 0.2395 cm (0.0943 inch), which agrees with the value computed by LEASTSQR. The calculated Sum of Residuals is 3.1909×10^{-5}. This value has been calculated based on the formula $s = \Sigma (y - y_{est})^2$, where s is Sum of Residuals, y is the dependent variable and y_{est} is the estimated value of y for a given value of x. The degree of fit is 0 since we are computing the arithmetic average. For the case where the degree of fit is equal to 0, $y = y_{est}$. So, by examining the equations for variance and Sum of Residuals it can be seen that it is possible to arrive at the value of variance, for the given set of data, from the value of the sum of residuals, simply by dividing the Sum of Residuals, by n-1. If the error in the measured diameter is random, then 95.45 % of the data will fall between 2σ, where σ is standard deviation. This can be inferred from the standard normal curve [29]. The standard deviation can be calculated from the relation

$$\sigma = \frac{\sqrt{v}}{y} = \frac{\sqrt{3.5454 \times 10^{-6}}}{0.0943} = 0.01984$$

Therefore, $\sigma \times 100\ \% = 1.98\ \%$. So, $2\sigma \times 100\ \% = 3.96\ \%$. Since, the d term arrears as a square in the mass equation, the error in d^2 will be $4\sigma \times 100\ \% = 7.92\ \%$. This is the error in mass due to an error in diameter. The error in the measured density is insignificant in comparison to the errors in the measurement of dimensions. If the

error in length (1.25 %) and the error in diameter (7.92 %) add up in the same direction the calculated mass can be upset by about 9.2 %.

Error in I and E

The maximum variation in current from the power supply, in the local control mode, is 2%. The same for remote control mode is 0.1%. The pulse experiments reported in this thesis have all been controlled using the remote mode. Thus the maximum variation in the current channel is 0.1%. The current flowing through the specimen was determined by measuring the voltage across the standard resistor and from the knowledge of the standard resistance. Thus, the uncertainty in the measured resistance of the standard resistor also comes into consideration. This uncertainty is 0.001% [28].

The maximum offset variation in voltage channel is 0.0001 % and the maximum drift over a 8 hour period is 0.005% [18]. The voltage across the effective specimen length was measured independently by the computer and a DVM and was seen to agree very closely (<0.01% error).

Error in T

The accuracy of the determined temperature is affected by the following factors.

1) Error in the measured emf.

2) Statistical error in emf to temperature conversion.

3) Voltage pick-up by the thermocouples.

4) Error due to the usage of uncalibrated thermocouple.

The accuracy of the measured emf is limited by the accuracy of the amplifier gain which, in turn, is dependent upon the resolution of the potentiometer which is used to calibrate the amplifier. The potentiometer used by us had a resolution of 0.00001 V. At an average temperature of 290 °C, which corresponds to an emf of 2.303 mV, the error in the measured emf will be about 0.4 %.

The other factor that affects the accuracy of the measured emf is the resolution of the analog to digital convertor. A 12 bit converter was used which has a full scale of 2^{12} = 4096 counts. Full scale corresponds to 10 Volts, in analog units. Thus 1 count will correspond to 2.4 mV. Now, an average temperature of 290 °C corresponds to an emf of 2.303 mV. At a gain of 1000 the voltage going into the converter is 2.303 Volts. So, the error due to the resolution limitation is 0.1 %.

The measured thermocouple emf are converted to temperature using T=f(E) coefficients generated by fitting the tabulated temperature versus emf data given in Monograph 125. The T=f(E) function has a statistical error associated with it. In the temperature range we are operating, using a quartic conversion equation gives an error in temperature of about 0.04 °C. At 290 °C the percentage error due to this error in temperature is 0.01 %.

Voltage pick-up by the thermocouple can cause significant error in the measured emf and hence an error in the determined temperature. It was found out that at a thermocouple emf of 100 μV, the pick-up voltage, which was 7 μV, accounted for about 7 % error. This value corresponds to room temperature. At a temperature of 290 °C, which corresponds to 230 μV, the percentage error is 3 %. This value will actually be somewhat higher than 3 % because the pick-up voltage will also increase at higher temperatures.

Since calibrated thermocouples were not used in this project an error of about 2 °C is expected [32], due to this. This would result in an error of about 0.7 % at an average temperature of about 290 °C.

Comparing the errors due to the four factors discussed, it is observed that the error due to the voltage pick-up is of a higher order than the others, so this is the factor which determines the total error in temperature.

Error in dT/dt

The accuracy of dT/dt is most elusive [4]. The factors which can affect the determined slope can be grouped into the following categories.

1) Any factor which cause an error in temperature.
2) Error in time measurement.
3) Statistical error associated with fitting dT/dt=f(T) data.

The last section discussed the factors which affect the error in

determined temperature. The error in determined temperature causes the T=f(t) curve to shift higher or lower. Due to this shift the slope at a slightly different temperature (e.g., 1 °C) will be calculated. From a practical standpoint, however, this error in slope will be negligible because the error in temperature will not change the curvature of the T=f(t) curve to any appreciable extent.

The computer clock is very accurate and the data points for time are not affected to the extent that an error in time can affect the accuracy in dT/dt.

There is a statistical error associated with fitting dT/dt data. The reader is reminded here that the T data have first been fitted versus time to obtain the T=f(t) relation. Then, the dT/dt=f(t) function has been determined by differentiating the T=f(t) function. However, finally the dT/dt=f(T) function is required. So, the dT/dt data have been fitted against smoothed T data. Since both T and dT/dt data have been smoothed by polynomials first, the data points for dT/dt=f(T) should lie on a smooth curve. But, depending on the nature of fitting dT/dt=f(T) data there may be small statistical error. Based on a mean temperature of 290 °C, the percentage error due to this is 0.01 %. This value has been arrived at by dividing the square root of Mean Sum of fit for the function by the slope at 290 °C.

All the three factors mentioned above have very little effect on the calculated dT/dt. So, it is concluded that the error in dT/dt is negligible.

Summary of Errors

The three electrical signals, current, voltage and thermal emf, are all affected by the error in the amplification factor of the amplifiers, which were not calibrated before use. But a check on the linearity was performed and it was seen to deviate a maximum of 0.6%, only at very low input voltages (e.g., 0.001 V). In the middle 80 % of the input voltage range the deviation was much smaller.

Table 4.6 Estimated errors in the quantities that are used to determine the specific heat.

Quantities	Percentage Error
Mass (m)	9.2 %
Current (I)	0.1 %
Voltage (E)	0.005 %
Temperature (T)	4.2 %
Slope (dT/dt)	NEGLIGIBLE

From Table 4.6 it can be inferred that the largest error in the specific heat is due to the mass.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions have been drawn from the pulse heating calorimetry experiments conducted in the UTK Calorimetry Laboratory.

1. A pulse heating calorimeter for measuring the specific heat of electrical conductors was designed. It mainly consists of a vacuum pumping system, a vacuum chamber with furnace, a programmable power supply, three d.c. amplifiers and a computer.

2. Four computer programs were written for the pulse experiment. They were the current control and data acquisition program (CALPUL.FOR), the overall control and calculation program (MASTER.BAS), the program for determining the amplifier gain (SETUP.FOR) and the program for converting the thermocouple voltage (mV) to temperature (°C) (CONVERT.BAS). The programs MASTER, CALPUL and CONVERT could call each other and transfer data between them to perform a complete specific heat experiment from the computer keyboard, with minimum operator involvement.

3. The specific heat of nickel was measured from 100 - 480 °C using the designed calorimeter. The average difference between the author's data and Meschter et al.'s data is -5 % in the range 100 - 320 °C and -4 % in the range 400 - 480 °C. This difference at the Curie temperature is 38 %. The Curie temperature determined by the author is 360 °C which is same as that determined by Kollie [5].

4. The determined specific heat data are affected by statistical

methods adopted while fitting the five source functions $P=f(T)_H$, $T=f(t)_H$, $dT/dt=f(T)_H$, $T=f(t)_C$, $dT/dt=f(T)_C$. The effects of intervals of fit and the degrees of fit were studied.

5. Without proper shielding of the thermocouple and proper grounding of the power supply the incoming thermocouple emf was disturbed considerably. Shielding and grounding restored the disturbance to a minimum.

6. An optimum value of variac a.c. voltage to the diffusion pump heater gave the best vacuum in the chamber. For the present set-up an optimum voltage of 82 Volts generated a vacuum of $6x10^{-6}$ Torr.

The following recommendations are given for future experiments using the pulse calorimeter.

1. A specimen with more uniform diameter should be used in order to estimate the mass of the specimen more accurately. Measured diameter of the specimen shows a standard deviation of 1.98 %. Since, diameter appears as a square term in the mass equation the error in calculated mass is twice this. Also, a specimen of known purity should be used, in order to make a more legitimate comparison of the calculated specific heat with earlier established values.

2. The temperature profile across the length of the effective specimen should be estimated using either several thermocouples or a scanning optical pyrometer. If it is observed by this analysis that the temperature between the voltage probes is not uniform, then the ratio of the effective length of the specimen to its physical length

should be decreased.

3. Separate and simultaneous electrical resistivity tests are recommended in the same temperature range the specific heat is being measured. This would serve as a secondary thermometry since from the $T=f(\rho)$ function the temperature of the specimen can be checked.

4. The three amplifiers used in the current, voltage and temperature channels should be calibrated before each experiment. The measured correct gain should be entered in the calculated program. An even better option would be to enter the calibration curve into the calculation program so that the correct gain at each input voltage is used.

5. Tests on repeatability should be performed. To do this the experiment can be repeated several times keeping all the test parameters the same. A test on reproducibility is also recommended. To do this, the calorimeter can be partially dismantled and assembled again. Then the test should be repeated with identical parameters.

6. A detailed study should be made on how the variation of statistical parameters on raw data affects the calculated specific heat. As examples, the effect of variation of interval length, interval limits and degrees of fit, are suggested.

7. Two different experiments should be performed: 1) Several pulses covering different temperature intervals can be applied and specific heat data can be calculated in each interval separately from raw data

in that interval only, and 2) one complete pulse covering the entire temperature interval can be applied and specific heat can be calculated from the single data set. The specific heat data determined from these two methods can then be compared.

8. Provision should be made in the program structure to transfer the coefficients of fit computed by the fitting program to the calculation program directly, without the operator having to type them through the computer keyboard. This would eliminate operator error and reduce the time for data analysis.

9. In order to use a higher heating rate on the specimen a power supply with higher current and voltage rating should be used. In the present project a maximum heating rate of about 25 °C/second was obtained using a power supply of rating 6 Volts - 90 amperes.

10. To minimize radiation heat loss from the specimen and conduction heat loss through the thermocouples, the specimen can be enclosed inside a furnace. Also, a furnace can be used to raise the temperature of the specimen to a steady state above room temperature, before the pulse.

11. The thermocouple wires coming from the specimen should be shielded so that external electromagnetic fields do not disturb the measured thermocouple voltage.

12. The ionization gauge should be checked for proper vacuum and the ice-bath should be checked for the presence of ice before every experiment.

13. Tests with forward and reverse current should be performed to

evaluate unwanted specimen thermocouple voltages. Simple switches to achieve forward and reverse current should be designed.

14. Auxilliary uses of this test facility should be explored including (a) measuring total hemispherical emittance, (b) conducting tests to measure heats of transformation, such as that of iron, (c) stored energy due to cold working, etc.

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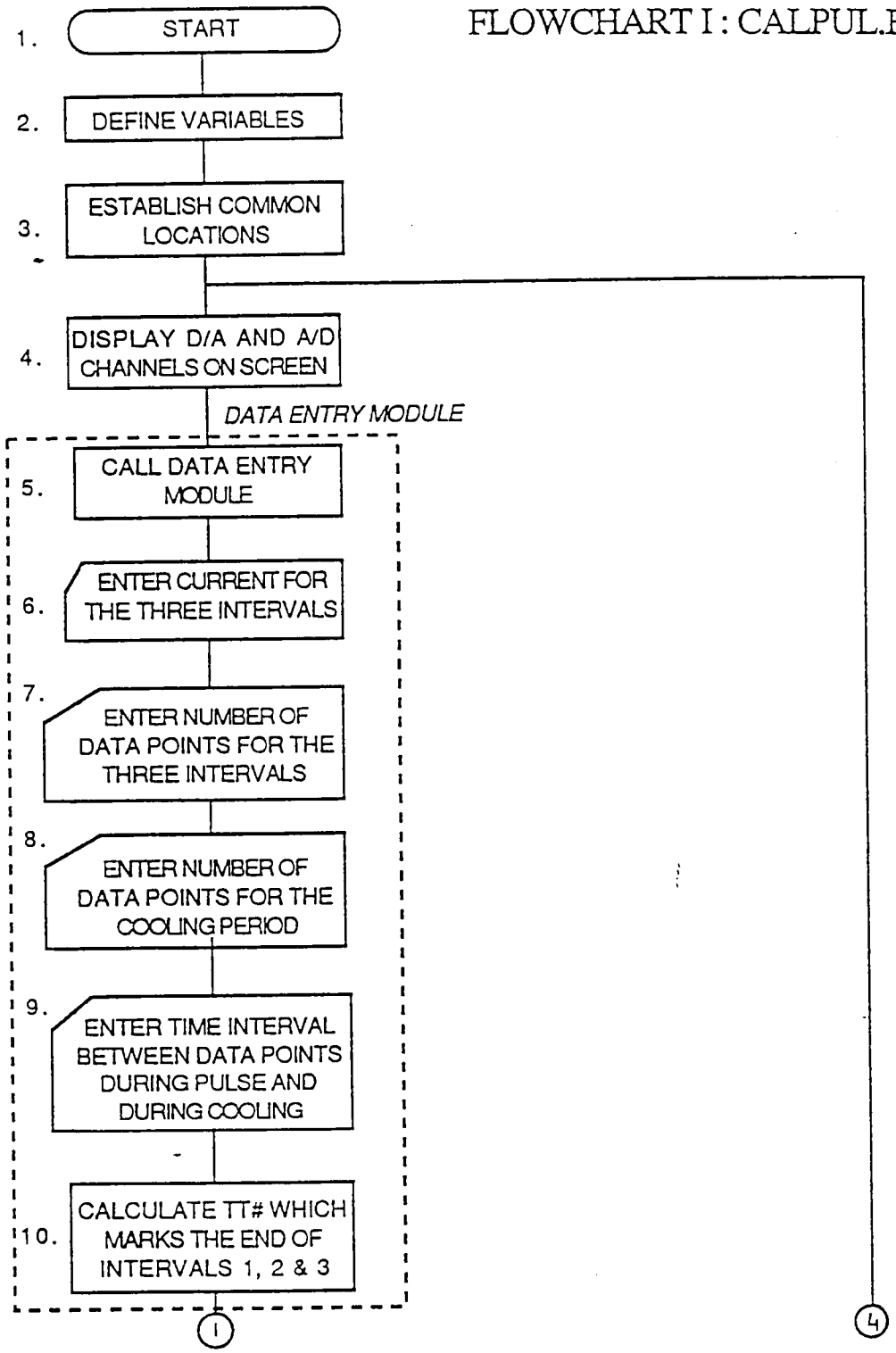
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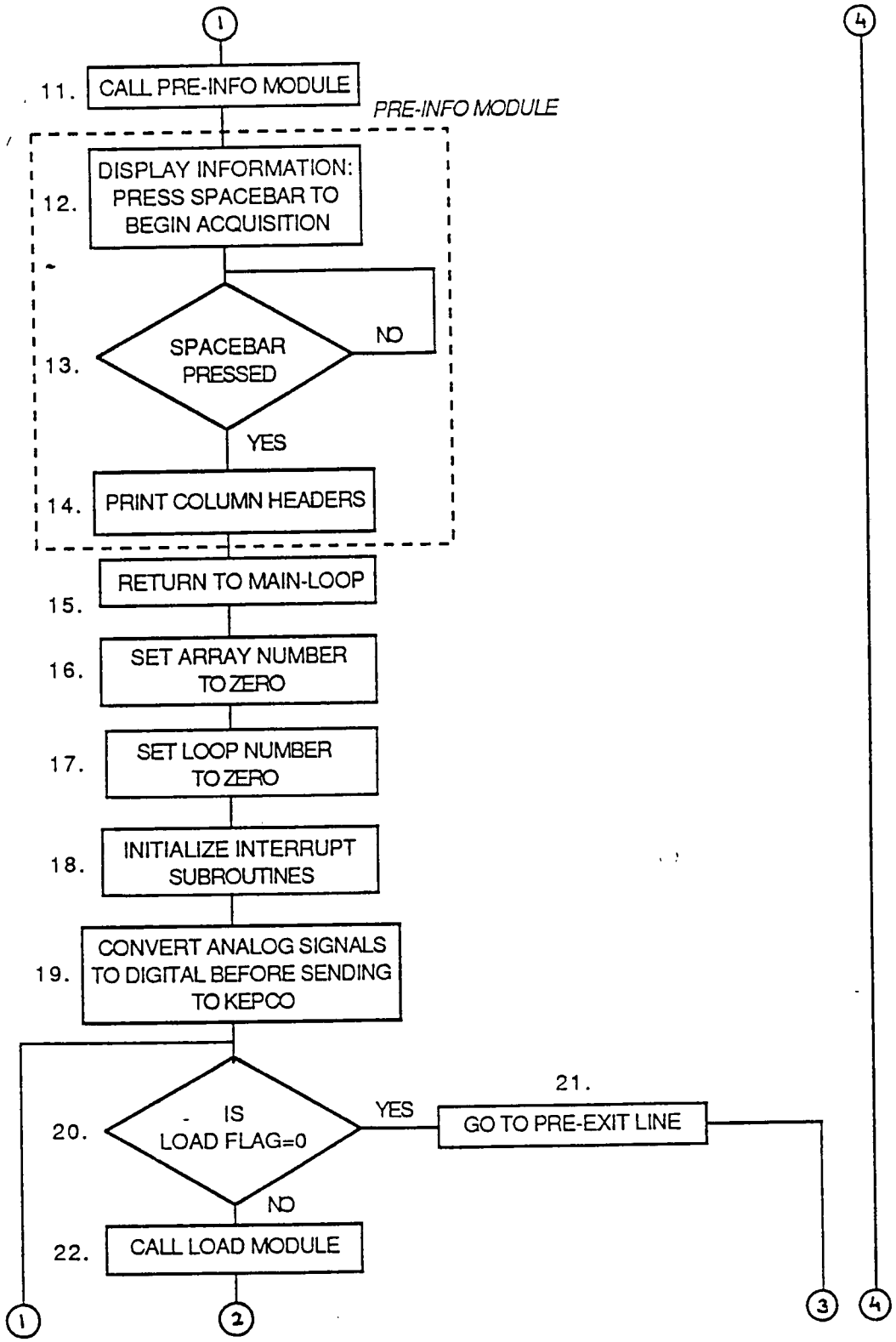
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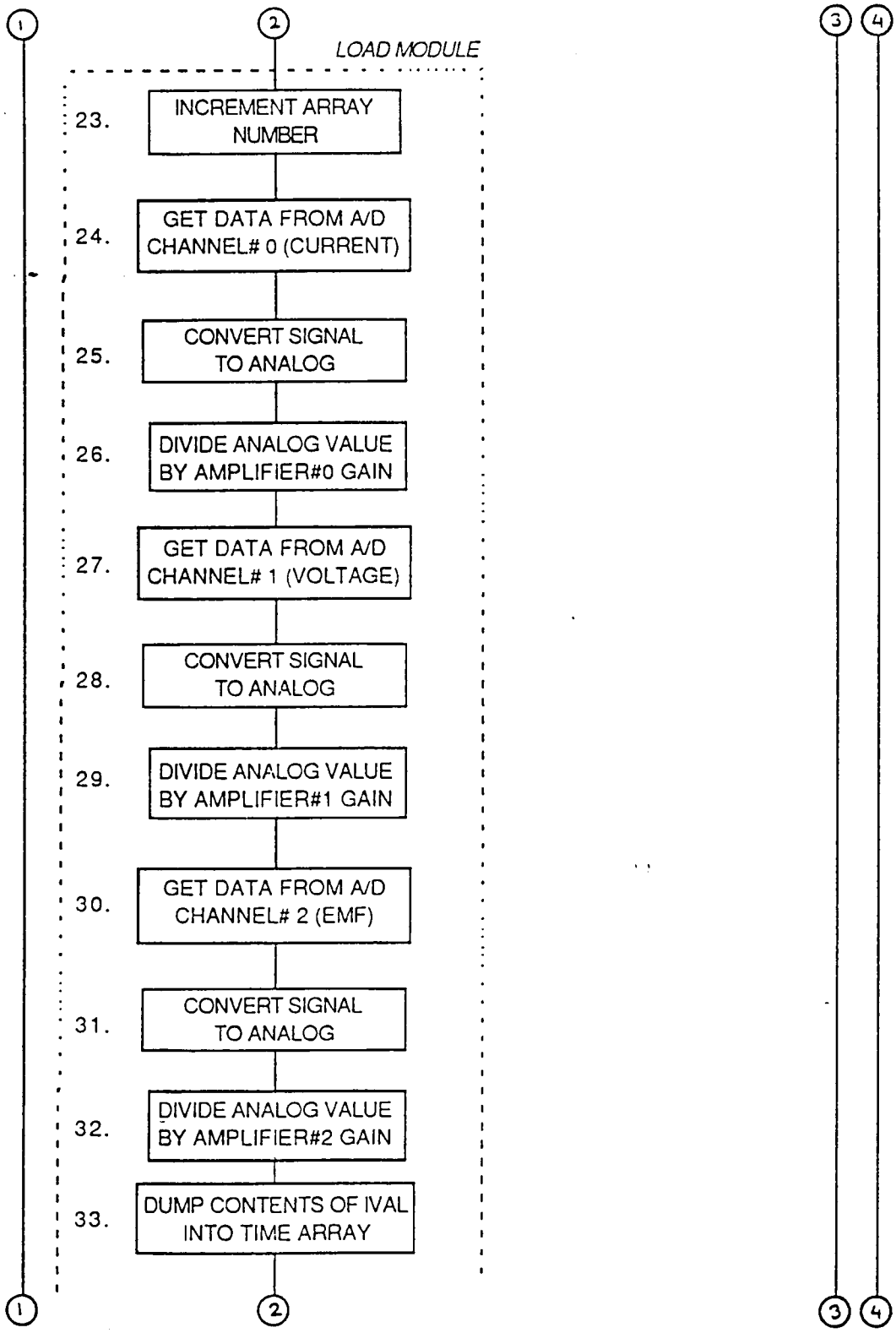
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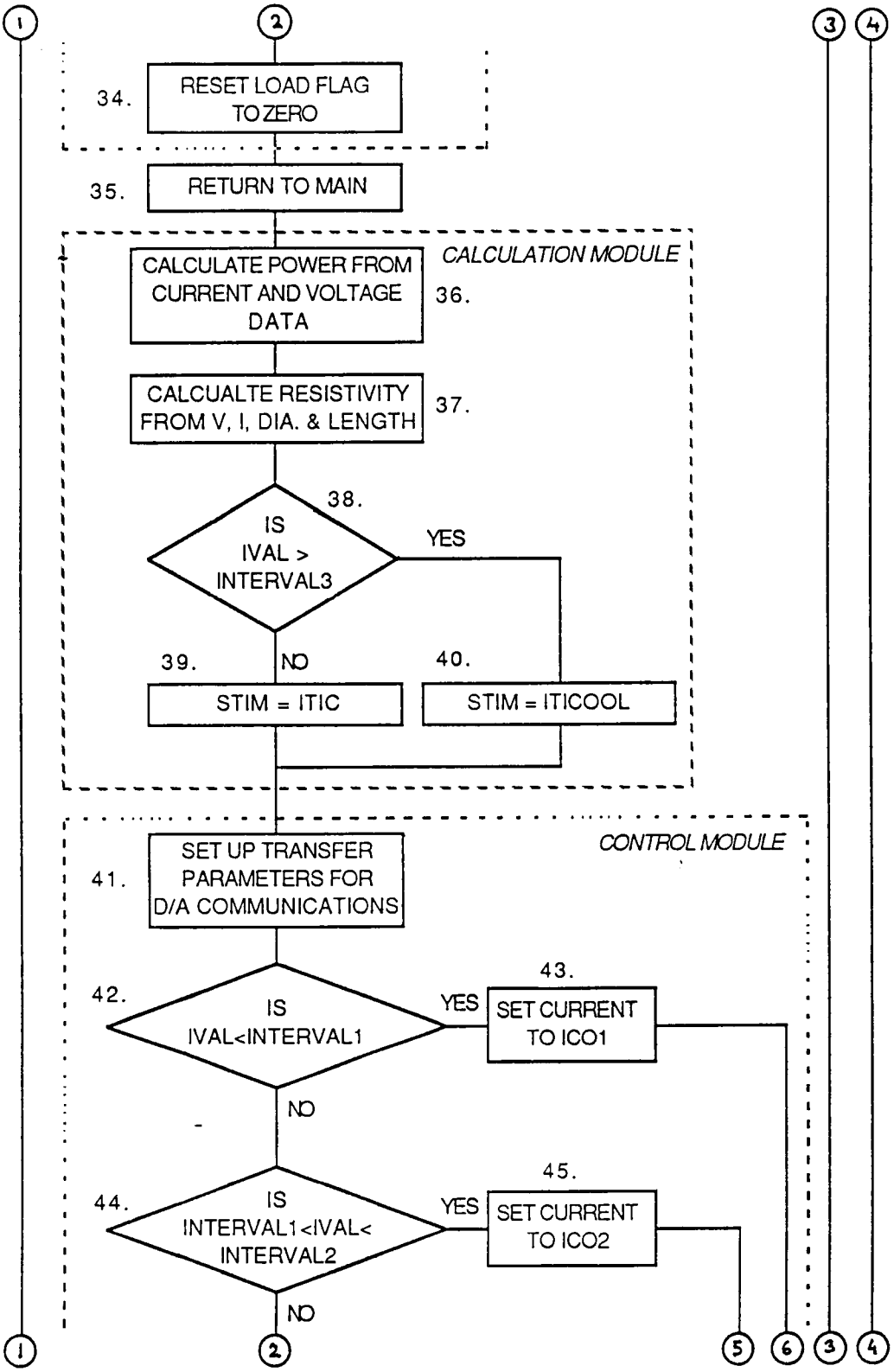
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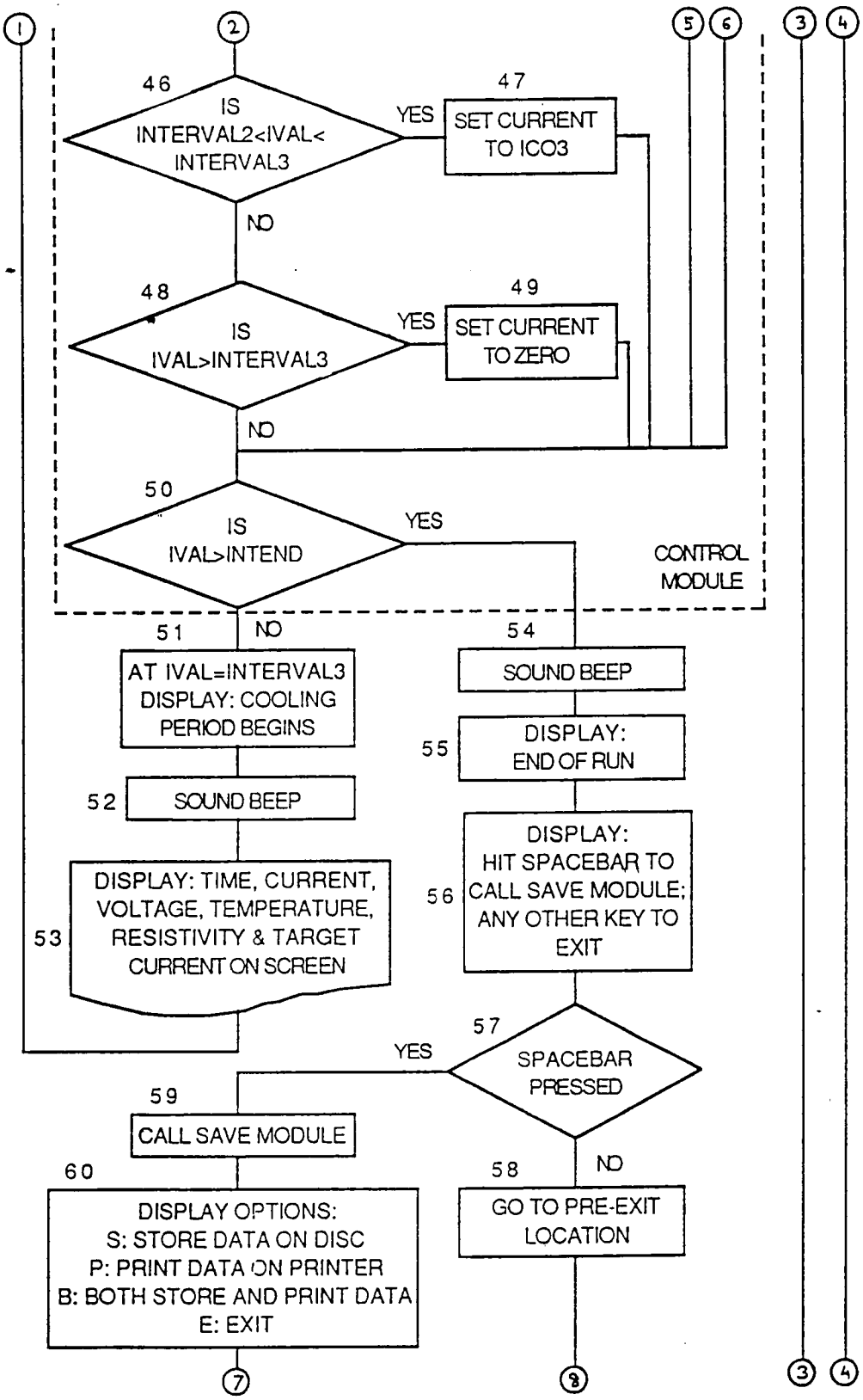
FLOWCHART I : CALPUL.FOR

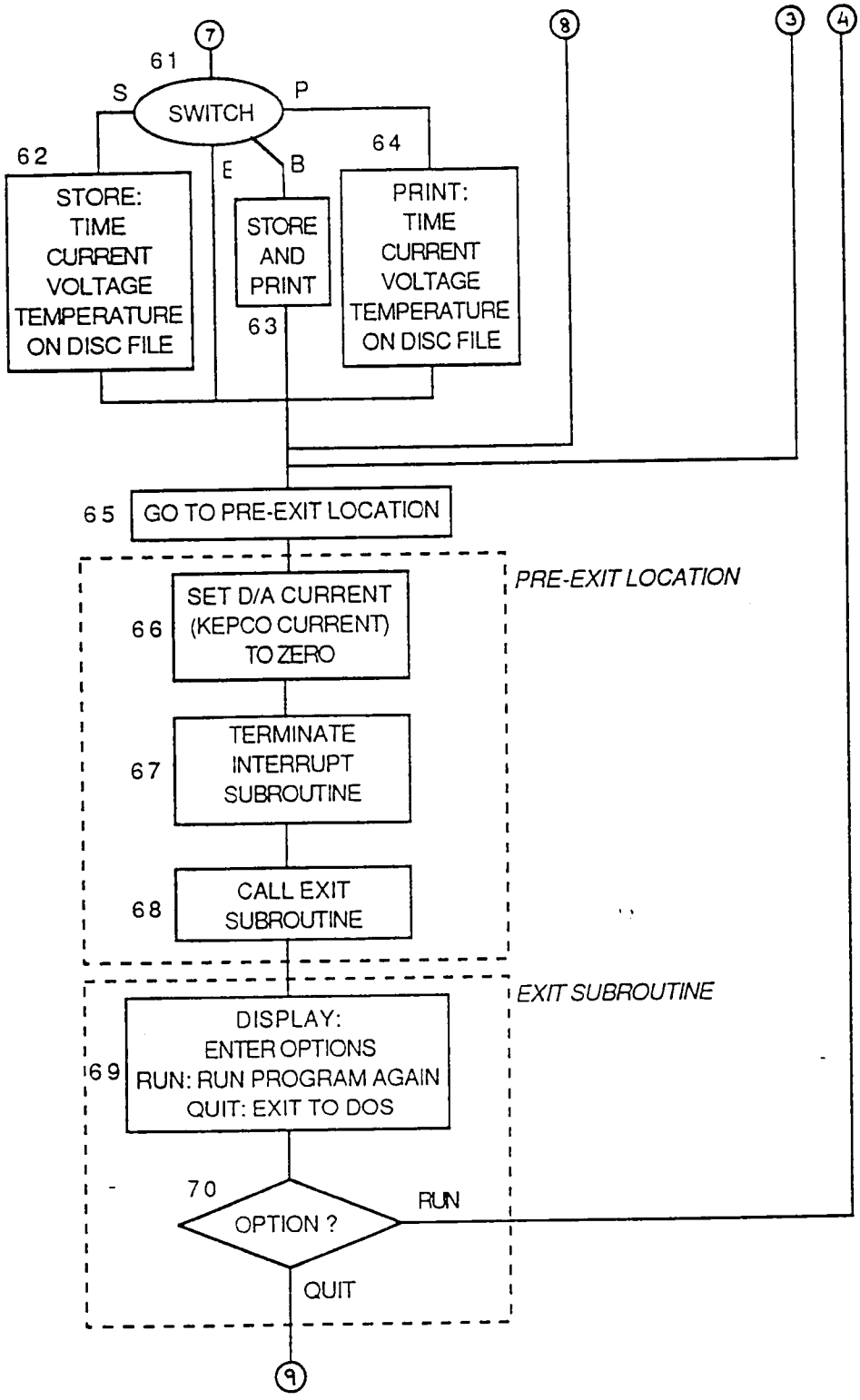


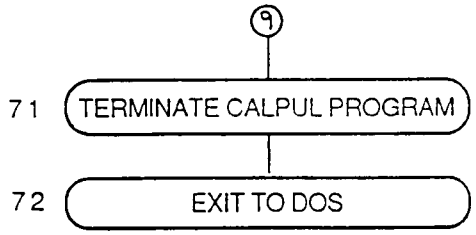

1
11.
CALL PRE-INFO MODULE
PRE-INFO MODULE
12.
DISPLAY INFORMATION: PRESS SPACEBAR TO BEGIN ACQUISITION
13.
SPACEBAR PRESSED
NO
YES
14.
PRINT COLUMN HEADERS
15.
RETURN TO MAIN-LOOP
16.
SET ARRAY NUMBER TO ZERO
17.
SET LOOP NUMBER TO ZERO
18.
INITIALIZE INTERRUPT SUBROUTINES
19.
CONVERT ANALOG SIGNALS TO DIGITAL BEFORE SENDING TO KEPCO
20.
IS LOAD FLAG=0
YES
21.
GO TO PRE-EXIT LINE
NO
22.
CALL LOAD MODULE
1
2
3
4
4


1
2
LOAD MODULE
23.
INCREMENT ARRAY NUMBER
24.
GET DATA FROM A/D CHANNEL# 0 (CURRENT)
25.
CONVERT SIGNAL TO ANALOG
26.
DIVIDE ANALOG VALUE BY AMPLIFIER#0 GAIN
27.
GET DATA FROM A/D CHANNEL# 1 (VOLTAGE)
28.
CONVERT SIGNAL TO ANALOG
29.
DIVIDE ANALOG VALUE BY AMPLIFIER#1 GAIN
30.
GET DATA FROM A/D CHANNEL# 2 (EMF)
31.
CONVERT SIGNAL TO ANALOG
32.
DIVIDE ANALOG VALUE BY AMPLIFIER#2 GAIN
33.
DUMP CONTENTS OF IVAL INTO TIME ARRAY
1
2
3
4
3
4

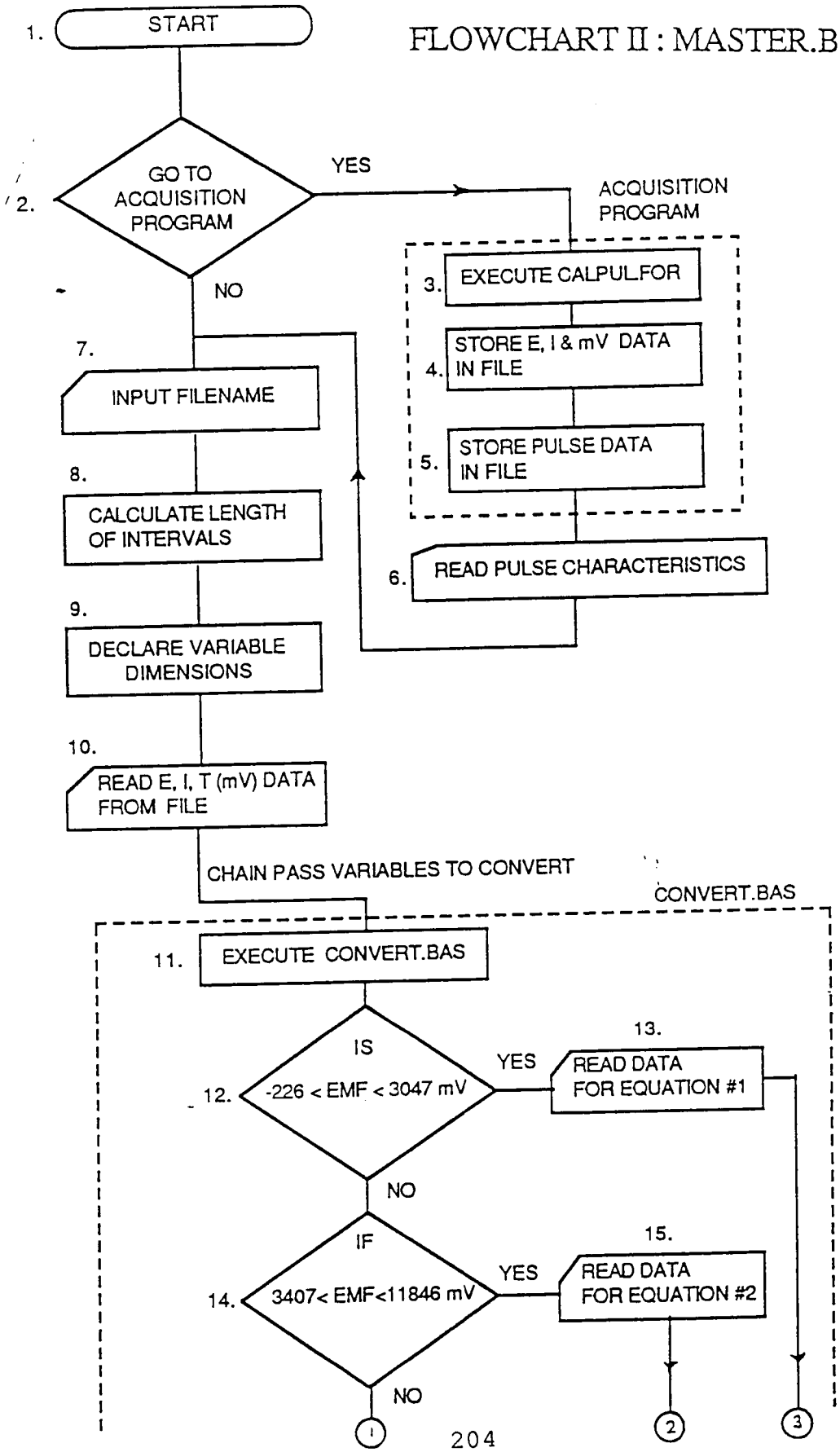

1
2
3
4
34.
RESET LOAD FLAG TO ZERO
35.
RETURN TO MAIN
CALCULATION MODULE
CALCULATE POWER FROM CURRENT AND VOLTAGE DATA
36.
CALCUALTE RESISTIVITY FROM V, I, DIA. & LENGTH
37.
38.
IS IVAL > INTERVAL3
YES
NO
39.
STIM = ITIC
40.
STIM = ITICOOL
CONTROL MODULE
41.
SET UP TRANSFER PARAMETERS FOR D/A COMMUNICATIONS
42.
IS IVAL<INTERVAL1
YES
43.
SET CURRENT TO ICO1
NO
44.
IS INTERVAL1<IVAL< INTERVAL2
YES
45.
SET CURRENT TO ICO2
NO
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2
5
6
3
4

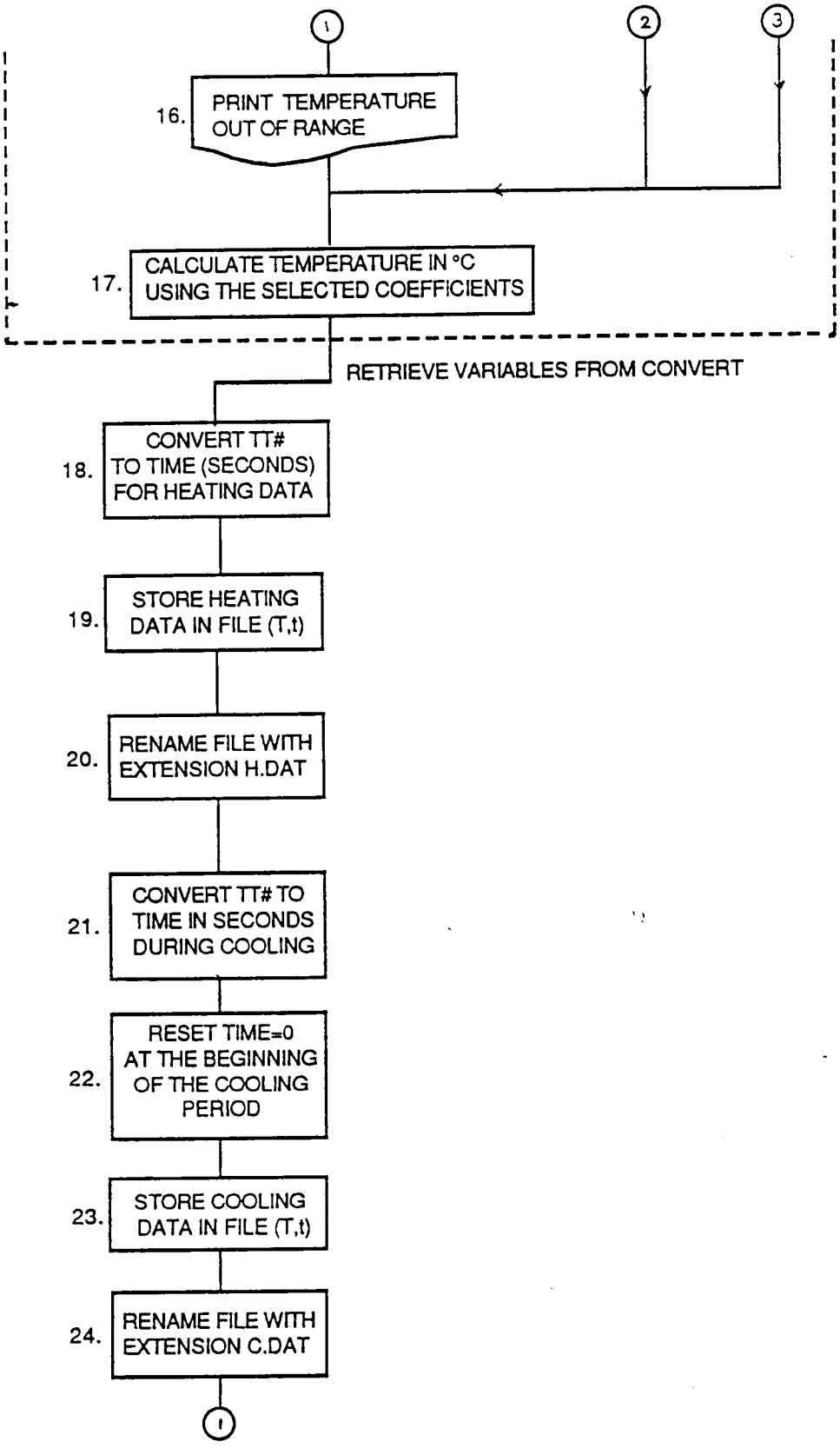

1
2
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3
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46
IS
INTERVAL2<IVAL<
INTERVAL3
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SET CURRENT
TO ICO3
NO
48
IS
IVAL>INTERVAL3
YES
49
SET CURRENT
TO ZERO
NO
50
IS
IVAL>INTEND
YES
CONTROL
MODULE
NO
51
AT IVAL=INTERVAL3
DISPLAY: COOLING
PERIOD BEGINS
52
SOUND BEEP
53
DISPLAY: TIME, CURRENT,
VOLTAGE, TEMPERATURE,
RESISTIVITY & TARGET
CURRENT ON SCREEN
54
SOUND BEEP
55
DISPLAY:
END OF RUN
56
DISPLAY:
HIT SPACEBAR TO
CALL SAVE MODULE;
ANY OTHER KEY TO
EXIT
57
SPACEBAR
PRESSED
YES
59
CALL SAVE MODULE
60
DISPLAY OPTIONS:
S: STORE DATA ON DISC
P: PRINT DATA ON PRINTER
B: BOTH STORE AND PRINT DATA
E: EXIT
NO
58
GO TO PRE-EXIT
LOCATION
7
8
3
4

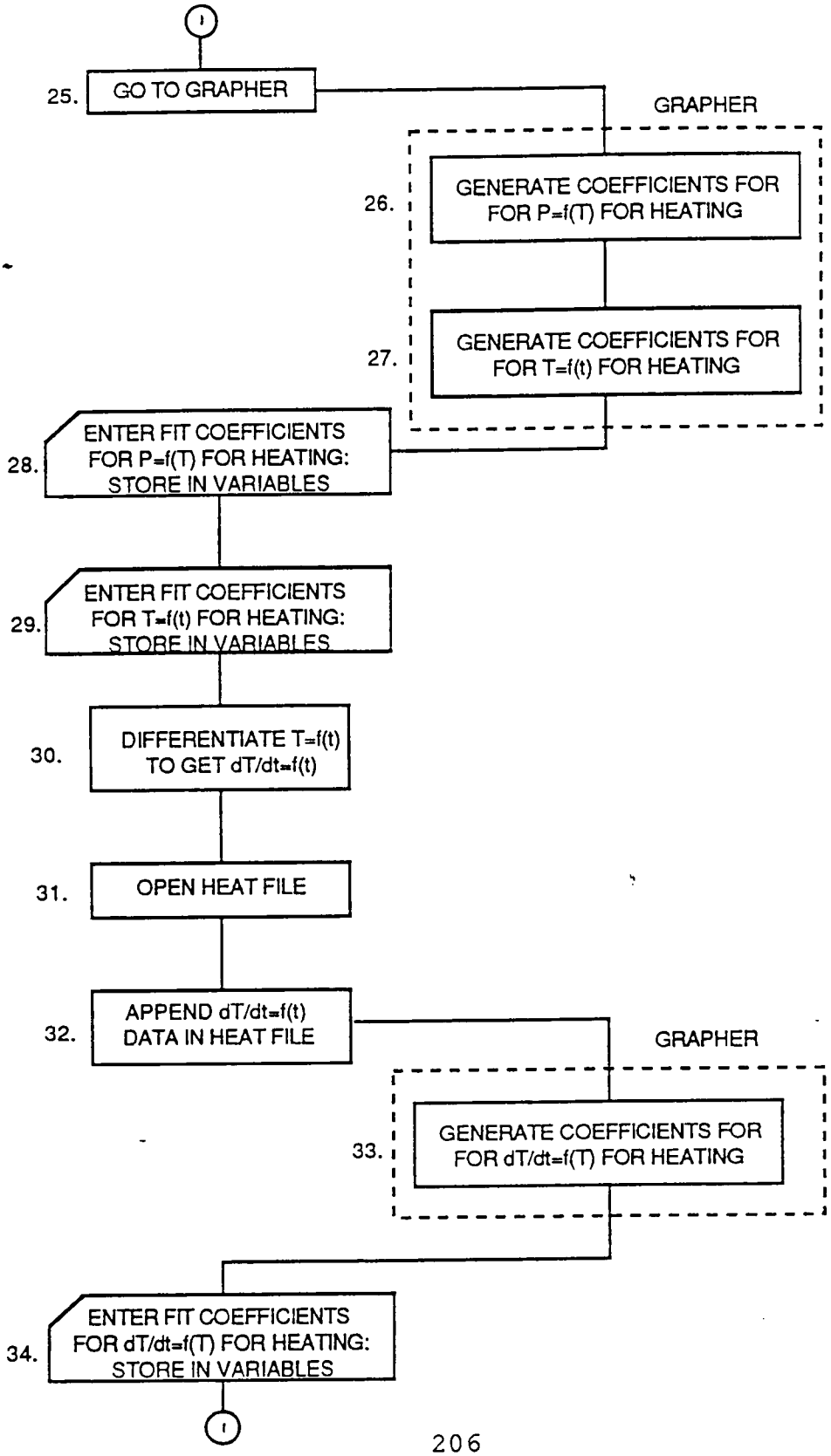

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61
S
SWITCH
P
62
E
B
64
STORE:
TIME
CURRENT
VOLTAGE
TEMPERATURE
ON DISC FILE
STORE
AND
PRINT
63
PRINT:
TIME
CURRENT
VOLTAGE
TEMPERATURE
ON DISC FILE
8
3
4
65
GO TO PRE-EXIT LOCATION
PRE-EXIT LOCATION
66
SET D/A CURRENT
(KEPCO CURRENT)
TO ZERO
67
TERMINATE
INTERRUPT
SUBROUTINE
68
CALL EXIT
SUBROUTINE
EXIT SUBROUTINE
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DISPLAY:
ENTER OPTIONS
RUN: RUN PROGRAM AGAIN
QUIT: EXIT TO DOS
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OPTION ?
RUN
QUIT
9

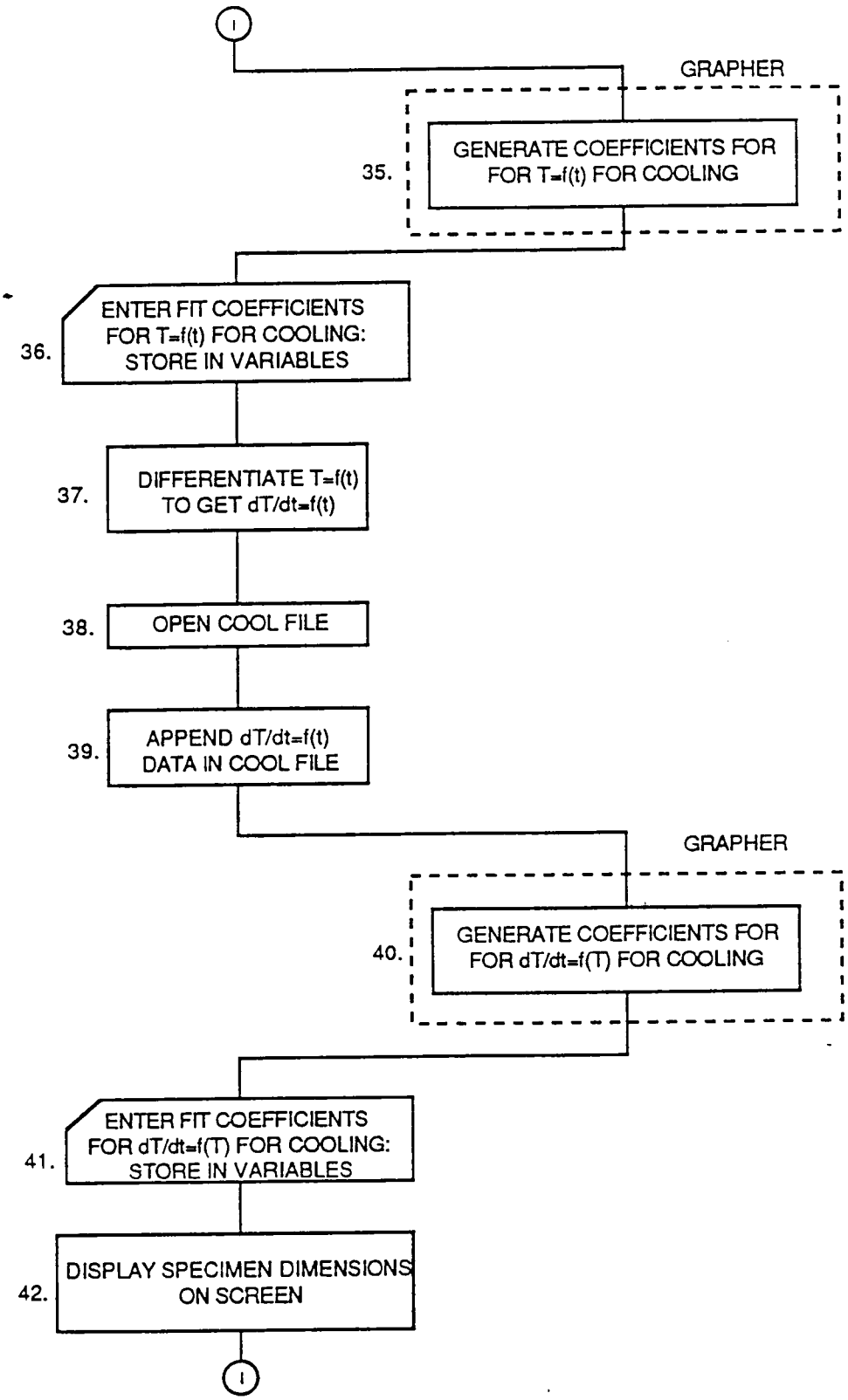

9
71
TERMINATE CALPUL PROGRAM
72
EXIT TO DOS

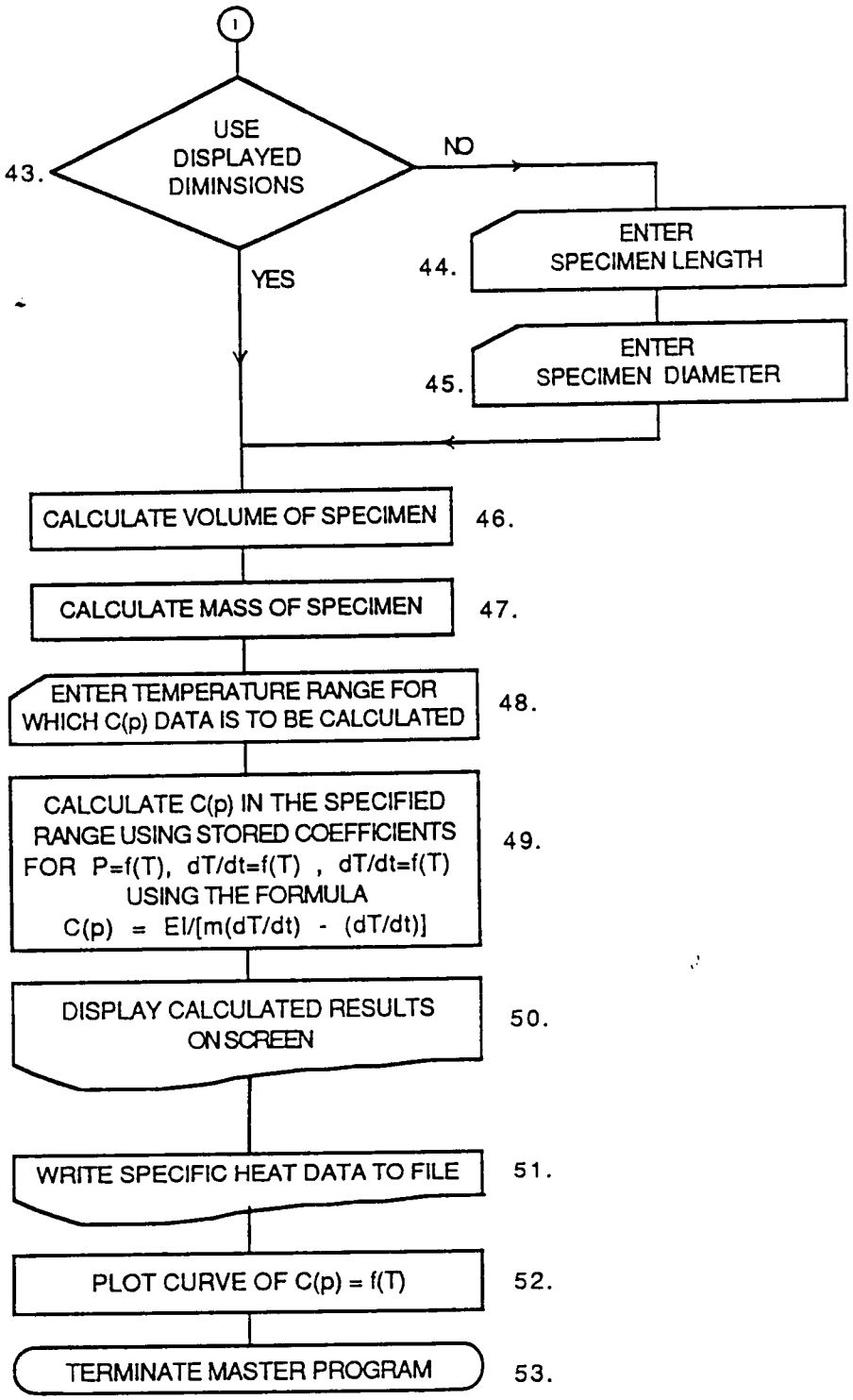
FLOWCHART II : MASTER.BAS



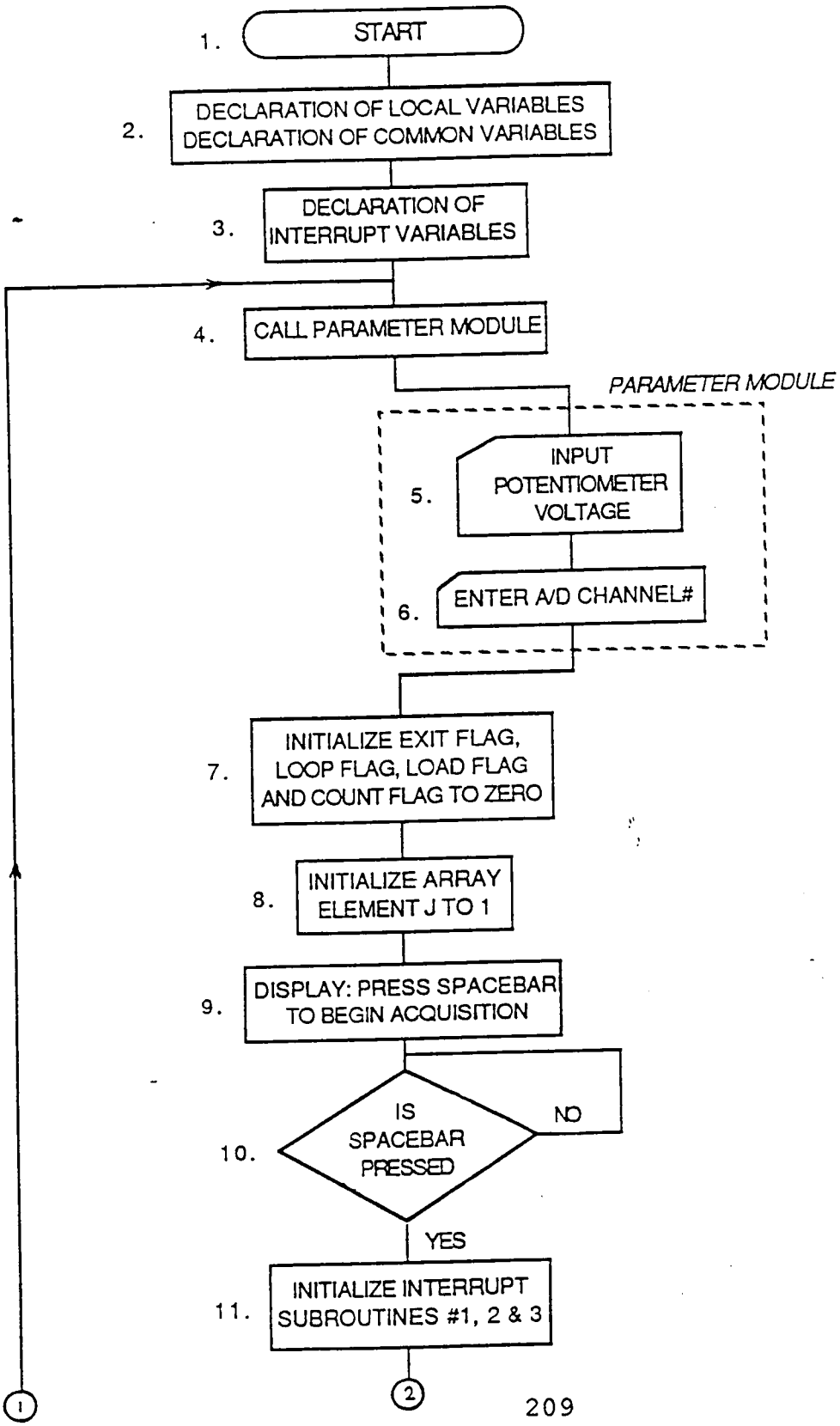

1
2
3
16.
PRINT TEMPERATURE OUT OF RANGE
17.
CALCULATE TEMPERATURE IN °C USING THE SELECTED COEFFICIENTS
RETRIEVE VARIABLES FROM CONVERT
18.
CONVERT TT# TO TIME (SECONDS) FOR HEATING DATA
19.
STORE HEATING DATA IN FILE (T,t)
20.
RENAME FILE WITH EXTENSION H.DAT
21.
CONVERT TT# TO TIME IN SECONDS DURING COOLING
22.
RESET TIME=0 AT THE BEGINNING OF THE COOLING PERIOD
23.
STORE COOLING DATA IN FILE (T,t)
24.
RENAME FILE WITH EXTENSION C.DAT
1

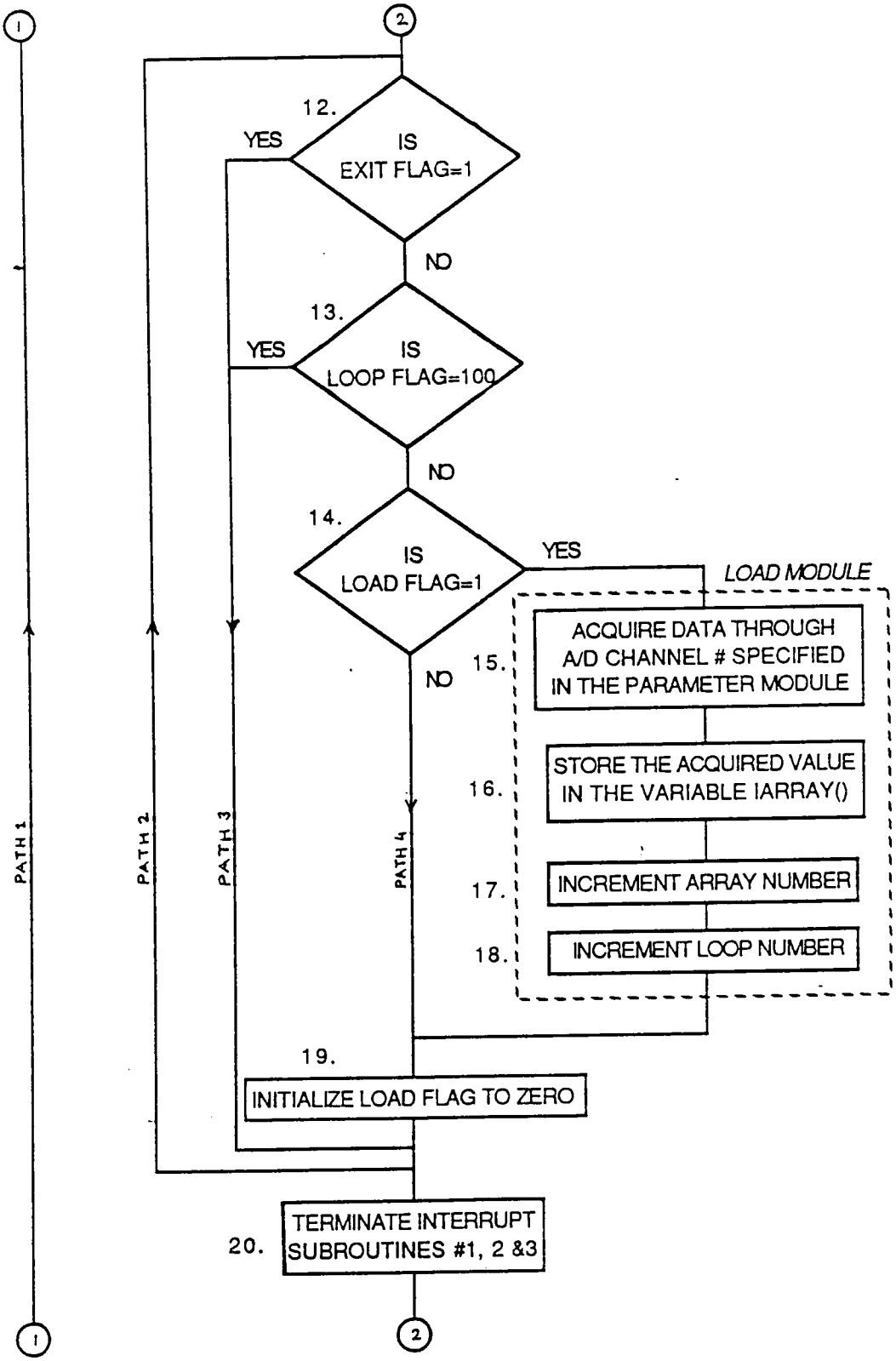

1
25.
GO TO GRAPHER
GRAPHER
26.
GENERATE COEFFICIENTS FOR
FOR P=f(T) FOR HEATING
27.
GENERATE COEFFICIENTS FOR
FOR T=f(t) FOR HEATING
28.
ENTER FIT COEFFICIENTS
FOR P=f(T) FOR HEATING:
STORE IN VARIABLES
29.
ENTER FIT COEFFICIENTS
FOR T=f(t) FOR HEATING:
STORE IN VARIABLES
30.
DIFFERENTIATE T=f(t)
TO GET dT/dt=f(t)
31.
OPEN HEAT FILE
32.
APPEND dT/dt=f(t)
DATA IN HEAT FILE
GRAPHER
33.
GENERATE COEFFICIENTS FOR
FOR dT/dt=f(T) FOR HEATING
34.
ENTER FIT COEFFICIENTS
FOR dT/dt=f(T) FOR HEATING:
STORE IN VARIABLES
1

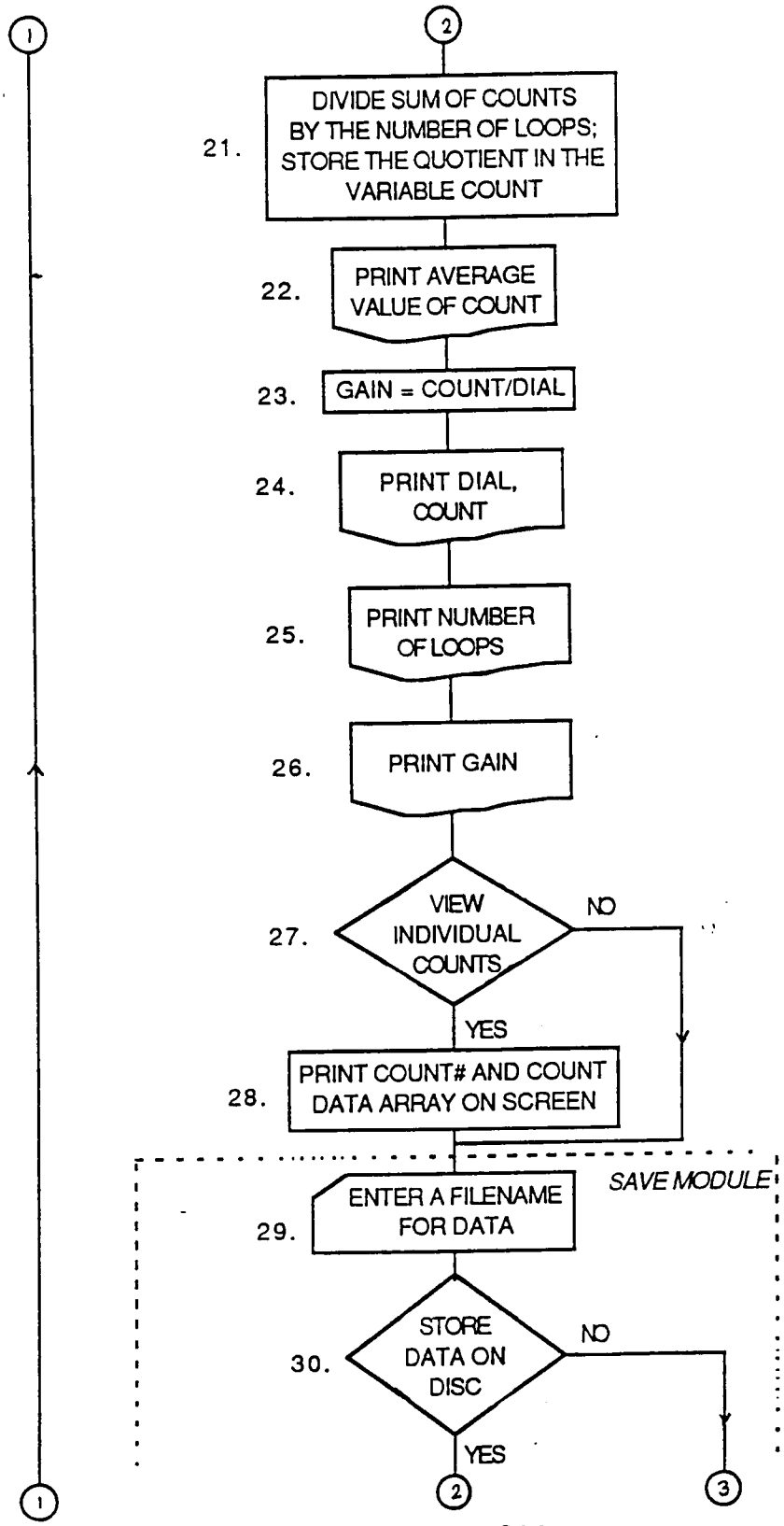

1
GRAPHER
35.
GENERATE COEFFICIENTS FOR
FOR T=f(t) FOR COOLING
36.
ENTER FIT COEFFICIENTS
FOR T=f(t) FOR COOLING:
STORE IN VARIABLES
37.
DIFFERENTIATE T=f(t)
TO GET dT/dt=f(t)
38.
OPEN COOL FILE
39.
APPEND dT/dt=f(t)
DATA IN COOL FILE
GRAPHER
40.
GENERATE COEFFICIENTS FOR
FOR dT/dt=f(T) FOR COOLING
41.
ENTER FIT COEFFICIENTS
FOR dT/dt=f(T) FOR COOLING:
STORE IN VARIABLES
42.
DISPLAY SPECIMEN DIMENSIONS
ON SCREEN
1

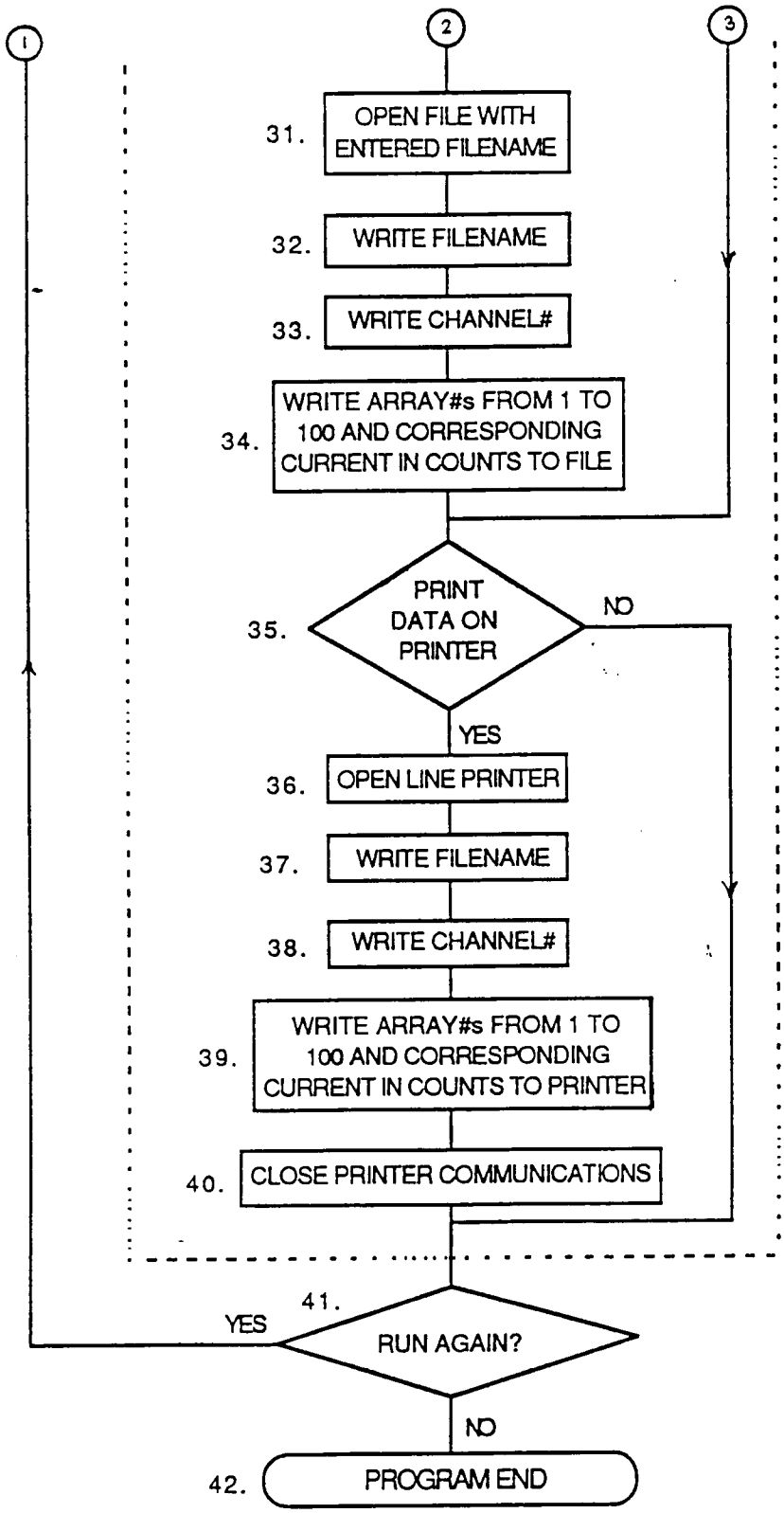

1
43.
USE DISPLAYED DIMINSIONS
NO
YES
44.
ENTER SPECIMEN LENGTH
45.
ENTER SPECIMEN DIAMETER
CALCULATE VOLUME OF SPECIMEN
46.
CALCULATE MASS OF SPECIMEN
47.
ENTER TEMPERATURE RANGE FOR WHICH C(p) DATA IS TO BE CALCULATED
48.
CALCULATE C(p) IN THE SPECIFIED RANGE USING STORED COEFFICIENTS FOR P=f(T), dT/dt=f(T) , dT/dt=f(T) USING THE FORMULA
C(p) = EI/[m(dT/dt) - (dT/dt)]
49.
DISPLAY CALCULATED RESULTS ON SCREEN
50.
WRITE SPECIFIC HEAT DATA TO FILE
51.
PLOT CURVE OF C(p) = f(T)
52.
TERMINATE MASTER PROGRAM
53.

FLOWCHART III : SETUP.FOR




1
2
12.
IS
EXIT FLAG=1
YES
NO
13.
IS
LOOP FLAG=100
YES
NO
14.
IS
LOAD FLAG=1
YES
NO
LOAD MODULE
15.
ACQUIRE DATA THROUGH
A/D CHANNEL # SPECIFIED
IN THE PARAMETER MODULE
16.
STORE THE ACQUIRED VALUE
IN THE VARIABLE IARRAY()
17.
INCREMENT ARRAY NUMBER
18.
INCREMENT LOOP NUMBER
19.
INITIALIZE LOAD FLAG TO ZERO
20.
TERMINATE INTERRUPT
SUBROUTINES #1, 2 &3
PATH 1
PATH 2
PATH 3
PATH 4
1
2


1
2
21.
DIVIDE SUM OF COUNTS BY THE NUMBER OF LOOPS; STORE THE QUOTIENT IN THE VARIABLE COUNT
22.
PRINT AVERAGE VALUE OF COUNT
23.
GAIN = COUNT/DIAL
24.
PRINT DIAL, COUNT
25.
PRINT NUMBER OF LOOPS
26.
PRINT GAIN
27.
VIEW INDIVIDUAL COUNTS
NO
YES
28.
PRINT COUNT# AND COUNT DATA ARRAY ON SCREEN
SAVE MODULE
29.
ENTER A FILENAME FOR DATA
30.
STORE DATA ON DISC
NO
YES
2
3
1


1
2
3
31.
OPEN FILE WITH ENTERED FILENAME
32.
WRITE FILENAME
33.
WRITE CHANNEL#
34.
WRITE ARRAY#s FROM 1 TO 100 AND CORRESPONDING CURRENT IN COUNTS TO FILE
35.
PRINT DATA ON PRINTER
NO
YES
36.
OPEN LINE PRINTER
37.
WRITE FILENAME
38.
WRITE CHANNEL#
39.
WRITE ARRAY#s FROM 1 TO 100 AND CORRESPONDING CURRENT IN COUNTS TO PRINTER
40.
CLOSE PRINTER COMMUNICATIONS
41.
YES
RUN AGAIN?
NO
42.
PROGRAM END

APPENDIX II

PROGRAM I: CALPUL.FOR

```
C****     11-14-91 **************************DEBASIS*

          PROGRAM CALORIMETER PULSE

C****     CALORIMETER PULSE WAS WRITTEN TO TAKE THREE A/D
C****     INPUTS ON CHANNELS 0-2 AND TO OUTPUT A CONTROL
C****     SIGNAL WITH THE D/A ON CHANNEL 0 FOR THE
C****     APPLICATION OF THE PULSE.


C****     DEFINE VARIABLES ***************************

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*4 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 IWS1(22),IWS2(22),IWS3(22),IREG(8),IVAL
      INTEGER*2 TIME(5000),IN
      INTEGER*2 IEXIT,ILOAD
      INTEGER*2 KEY,INKEY,WAITKY
      INTEGER*2 INTERVAL1,INTERVAL2,INTERVAL3
      INTEGER*2 ICURRENT1,ICURRENT2,ICURRENT3,ICURR
      INTEGER*2 INTCOOL,ITICOOL,INTEND

      REAL*8
     DATAR0(5000),DATAR1(5000),DATAR2(5000),POWER(5000)
      REAL*8 RHO(5000)
      REAL*8
     STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
      CHARACTER*12 FNAME

      EXTERNAL ISR1,ISR2,ISR3

C****     ESTABLISH COMMON LOCATIONS ****************

      COMMON/TIME/STIM,JTIM,HTIM,ITIC

     COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
     QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN
      COMMON/FILE/FNAME
```

```
      COMMON/CURRENT/ICURRENT1,ICURRENT2,ICURRENT3
      COMMON/INTERVAL/INTERVAL1,INTERVAL2,INTERVAL3
      COMMON/DATAR/DATAR0,DATAR1,DATAR2,POWER,RHO
      COMMON/COOL/INTCOOL,ITICOOL,INTEND

C****     PRE MAIN LOOP ********************************

      CALL ALDV(0,0)              !  SET D/A OUTPUT TO 0
5     WRITE(*,10)
10    FORMAT(1X,/,/,/,/,/,/,/,/,/,/,/,/,/)
      WRITE(*,20)
20    FORMAT(9X,'** PROGRAM - CALORIMETER VERSION 2.1 **
',/,/)
      WRITE(*,30)
30    FORMAT(13X,'CAPS LOCK MUST BE ON',/,/,/)
      WRITE(*,70)
70    FORMAT(9X,'TEMPERATURE INPUT 1   ON A/D CH0')
      WRITE(*,75)
75    FORMAT(9X,'SAMPLE VOLTAGE INPUT 2   ON A/D CH1')
      WRITE(*,80)
80    FORMAT(9X,'SHUNT CURRENT INPUT 3   ON A/D CH2')
      WRITE(*,82)
82    FORMAT(9X,'KEPCO CURRENT OUTPUT 1 ON D/A CH0',/,/)
      WRITE(*,85)
85    FORMAT(9X,'**** SPACE TO PROCEED ****')
      KEY=WAITKY()
      IF (KEY.NE.32)GO TO 5

C****     MAIN LOOP ************************************

115   CALL PREINFO
      IVAL=0
      ITIC=STIM                          !  SET LOOP FOR SAMPLE
RATE
C     CALL WTIME(0,0,0,0)                !  SET CLOCK TO ZERO
      CALL ISRSET(28,ISR1,IWS1)          !  INITIALIZE ISR1
      CALL ISRSET(27,ISR2,IWS2)          !  SET UP ^BREAK ISR
      CALL ISRSET(35,ISR3,IWS3)          !  SET UP ^C ISR

      ICO1=ICURRENT1*(4096/100)          ! CONVERT ANALOG TO
DIGITAL
      ICO2=ICURRENT2*(4096/100)          ! BEFORE SENDING TO KEPCO
      ICO3=ICURRENT3*(4096/100)

C120       KEY=INKEY()
120   IF(ILOAD.EQ.0)GO TO 125            !  DEB CHANGE(120 ON)
      CALL LOAD

C****   CALCULATION MODULE **********************
```

```
      POWER(IVAL)=DATAR0(IVAL)*DATAR1(IVAL)
      IF (DATAR0(IVAL).NE.0) THEN
      RHO(IVAL)=((3.14159265*0.2395**2)/(4.0*4.00))*
     1    (DATAR1(IVAL)/DATAR0(IVAL))*1D6
      ENDIF
      IF (IVAL.GT.INTERVAL3)ITIC=ITICOOL

C****      END CALCULATION ****

C****      CONTROL MODULE ************************

      CALL ALSETD(0,0)                ! SET DAC OUTPUT
      IF (IVAL.LE.INTERVAL1) THEN     ! TIME LOGIC FOR INTERVAL1
      CALL ALDV(0,ICO1)               ! SEND SIGNAL TO KEPCO
      ICURR=ICURRENT1                 ! VARIABLE FOR SET CURRENT1
      ENDIF
      IF (IVAL.GT.INTERVAL1) THEN     ! TIME LOGIC FOR INTERVAL2
      IF (IVAL.LE.INTERVAL2) THEN
      CALL ALDV(0,ICO2)               ! SEND SIGNAL TO KEPCO
      ICURR=ICURRENT2                 ! VARIABLE FOR SET CURRENT2
      END IF
      END IF
      IF (IVAL.GT.INTERVAL2) THEN     ! TIME LOGIC FOR INTERVAL3
      IF (IVAL.LE.INTERVAL3) THEN
      CALL ALDV(0,ICO3)               ! SEND SIGNAL TO KEPCO
      ICURR=ICURRENT3                 ! VARIABLE FOR SET CURRENT3
      END IF
      END IF
      IF(IVAL.GT.INTERVAL3) THEN      ! TIME LOGIC FOR COOLING
PERIOD
      CALL ALDV(0,0)                  ! SET CURRENT TO ZERO
      ICURR=0
      ENDIF
      IF(IVAL.GT.INTEND) THEN         ! TIME LOGIC FOR END
      DO 410, I=1,5
      CALL BEEP(700,25)
      DO 420, J=1,100
420   CONTINUE
      CALL BEEP(900,50)
410   CONTINUE
      WRITE(*,450)
450   FORMAT(24X,'END OF COOLING PERIOD')
      WRITE(*,500)INTEND
500   FORMAT(/,/,12X,'PAST  TIME  TAG#',I5,8X,' END  OF
RUN')
      WRITE(*,600)
600   FORMAT(9X,'HIT SPACEBAR TO CALL THE SAVE MODULE',/)
      WRITE(*,700)
700   FORMAT(9X,'ANY OTHER KEY TO EXIT')
```

```
      KEY=WAITKY()
      IF (KEY.EQ.32) CALL DISK    ! FETCH SAVE MODULE
      IF (KEY.NE.0) GO TO 140     ! GO TO COMMON PRE-EXIT
POINT
      ENDIF


C****      END CONTROL ****

      IF (IVAL.EQ.INTERVAL3+1) THEN
      WRITE(*,100)
100   FORMAT(24X,'COOLING PERIOD BEGINS')
      CALL BEEP(500,10)
      ENDIF

WRITE(*,121)TIME(IVAL),DATAR0(IVAL),DATAR1(IVAL),DATAR2(IVAL
)
     1   ,POWER(IVAL),RHO(IVAL),ICURR !  SCREEN OUTPUT
121
FORMAT(3X,I5,6X,F9.6,7X,F8.6,2X,F8.3,1X,F8.3,2X,F6.3,2X,I5)
C125 IF (KEY.EQ.32)GO TO 140        !  SCREEN OUTPUT FORMAT

125   IF (IEXIT.NE.0)GO TO 140      !  IF ON ^C OR ^BREAK:DEB
130   IF (IVAL.NE.10000)GO TO 120   !  IF ON MAX ARRAY SIZE
140   CALL  ALDV(0,0)               !   SET D/A TO 0
      CALL ISRCLR(IWS1)             !  TERMINATE ISR1
      CALL ISRCLR(IWS2)             !  TERMINATE ISR2
      CALL ISRCLR(IWS3)             !  TERMINATE ISR3
      CALL EXIT                     !  EXIT
      GO TO 5       ! FLOW RETURNS TO THIS POINT ON, RUN AGAIN


      STOP
      END

C****      PRE-EXPERIMENT INFORMATION SCREEN***************

      SUBROUTINE PREINFO

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(5000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD
      INTEGER*2 KEY,WAITKY
      INTEGER*2 ICURRENT1,ICURRENT2,ICURRENT3
      INTEGER*2 INTERVAL1,INTERVAL2,INTERVAL3
      INTEGER*2 INTCOOL,ITICOOL,INTEND
```

```
      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
      CHARACTER*12 FNAME

      COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
/QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN
      COMMON/CURRENT/ICURRENT1,ICURRENT2,ICURRENT3
      COMMON/INTERVAL/INTERVAL1,INTERVAL2,INTERVAL3
      COMMON/COOL/INTCOOL,ITICOOL,INTEND
      COMMON/FILE/FNAME


60    CALL PULSE    !  FETCH PULSE MODULE
62    WRITE(*,70)
      WRITE(*,63)
63    FORMAT(9X,/,/,/,/,/,/,/)
      WRITE(*,65)
65    FORMAT(15X,'**** PRESS SPACEBAR TO ACTIVATE OUTPUT
****',/)
      WRITE(*,66)
66    FORMAT(24X,'AND START DATA ACQUISITION',/,/,/,/,/,/,/)
      WRITE(*,67)
67    FORMAT(15X,'THIS PROGRAM AUTOMATICALLY DEACTIVATES
OUTPUT',/)
      WRITE(*,68)INTERVAL3
68       FORMAT(15X,'AT THE END OF INTERVAL3 (TT#',I4
      1   ,' FOR THIS RUN)',/)
      WRITE(*,69)
69    FORMAT(15X,'AND STOPS DATA ACQUISITION AT THE END OF
THE',/)
      WRITE(*,691)INTEND
691   FORMAT(18X,'COOLING PERIOD (TT#',I4,' FOR THIS RUN)',/)
      WRITE(*,70)
      KEY=WAITKY()
      IF (KEY.NE.32)GO TO 62
      WRITE(*,70)
70    FORMAT(/,/,/,/,/,/)
      WRITE(*,80)
80    FORMAT(9X,'*** OUTPUT ACTIVE AND ACQUISITION IN
PROGRESS ***',/,/)
      WRITE(*,90)
90    FORMAT(10X,'--- CTRL BREAK WILL ABORT DATA ACQUISITION
---'
```

```
     1    ,/,/,/)
     WRITE(*,100)
100  FORMAT(1X,'TIME TAG#',7X,'CURRENT',5X,'VOLTAGE',5X,
     2  'TEMP',3X,'POWER',8X,'RHO',2X,'KEPCO I',/)
     RETURN
     END


C**** OPERATOR QUERY ********************************

     SUBROUTINE PULSE

     INTEGER*2 STIM,JTIM,HTIM,ITIC
     INTEGER*2 TEMP1
     INTEGER*2 VOLT1,VOLT2
     INTEGER*2 TIME(5000),IN
     INTEGER*2 IEXIT,IVAL,ILOAD
     INTEGER*2 KEY,WAITKY,CHOICE
     INTEGER*2 ICURRENT1,ICURRENT2,ICURRENT3
     INTEGER*2 INTERVAL1,INTERVAL2,INTERVAL3
     INTEGER*2 INTCOOL,ITICOOL,INTEND

     REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
     CHARACTER*12 FNAME

     COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT
     COMMON/TEMP/TEMP1
     COMMON/VOLT/VOLT1,VOLT2
     COMMON/FLAG/IEXIT,IVAL,ILOAD
     COMMON/INPUTS/TIME,IN
     COMMON/FILE/FNAME
     COMMON/CURRENT/ICURRENT1,ICURRENT2,ICURRENT3
     COMMON/INTERVAL/INTERVAL1,INTERVAL2,INTERVAL3
     COMMON/COOL/INTCOOL,ITICOOL,INTEND

5    WRITE(*,10)
10   FORMAT(/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/)
     WRITE(*,15)
15   FORMAT(12X,'**ENTER PULSE CHARACTERISTICS**')
     WRITE(*,16)
16   FORMAT(12X,'==========================================',/,/)
     WRITE(*,20)
20   FORMAT(9X,'ENTER CURRENT FOR INTERVAL 1 IN "AMPS"',/)
```

```
      WRITE(*,30)
      READ(*,30)ICURRENT1
30    FORMAT(I5)
      WRITE(*,40)
40    FORMAT(9X,'ENTER CURRENT FOR INTERVAL 2 IN "AMPS"',/)
      READ(*,50)ICURRENT2
50    FORMAT(I5)
      WRITE(*,60)
60    FORMAT(9X,'ENTER CURRENT FOR INTERVAL 3 IN "AMPS"',/)
      READ(*,70)ICURRENT3
70    FORMAT(I5)

      WRITE(*,140)
140   FORMAT(9X,'ENTER INTERVAL BETWEEN READINGS DURING
HEATING
      1  IN "TICS" ')
      WRITE(*,145)
145   FORMAT(9X,'(1 TIC IS 55 MILLISECONDS,18 TICS IS 1
SECOND)')
      READ(*,150)STIM
150   FORMAT(I5)


      WRITE(*,80)
80    FORMAT(9X,'ENTER TOTAL NUMBER OF READINGS IN INTERVAL 1

      1  AS "TT#"',/)
      READ(*,90)INTERVAL1
90    FORMAT(I5)
      WRITE(*,100)
100   FORMAT(9X,'ENTER TOTAL NUMBER OF READINGS IN INTERVAL 2

      1  AS "TT#"',/)
      READ(*,110)INTERVAL2
110   FORMAT(I5)
      WRITE(*,120)
120   FORMAT(9X,'ENTER TOTAL NUMBER OF READINGS IN INTERVAL 3

      1  AS "TT#"',/)
      READ(*,130)INTERVAL3
130   FORMAT(I5)
      WRITE(*,165)
165   FORMAT(9X,'ENTER INTERVAL BETWEEN READINGS DURING
COOLING
      1  IN "TICS"')
      WRITE(*,167)
167   FORMAT(9X,'(1 TIC IS 55 MILLISECONDS, 18 TICS IS 1
SECOND)')
      READ(*,170)ITICOOL
```

```
170   FORMAT(I5)

      WRITE(*,155)
155   FORMAT(9X,'ENTER TOTAL NUMBER OF READINGS DURING
COOLING
     1  AS "TT#"',/)
      READ(*,160)INTCOOL
160   FORMAT(I5)

175   INTERVAL3=INTERVAL1+INTERVAL2+INTERVAL3 ! COMPUTE FLAGS
      INTERVAL2=INTERVAL1+INTERVAL2           ! FOR INTERVALS
     - INTEND= INTERVAL3+INTCOOL
      WRITE(*,180)
180   FORMAT(/,/)
      RETURN
      END


C****      LOAD ARRAYS    ******************************

      SUBROUTINE LOAD

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(5000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD
      INTEGER*2 ICURRENT1,ICURRENT2,ICURRENT3
      INTEGER*2 INTERVAL1,INTERVAL2,INTERVAL3

      REAL*8
DATAR0(5000),DATAR1(5000),DATAR2(5000),POWER(5000)
      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT


      COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN
      COMMON/CURRENT/ICURRENT1,ICURRENT2,ICURRENT3
      COMMON/INTERVAL/INTERVAL1,INTERVAL2,INTERVAL3
      COMMON/DATAR/DATAR0,DATAR1,DATAR2
      IVAL=IVAL+1                    !  INCREMENT SAMPLE NUMBER
```

```
5     FORMAT(/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/)
      WRITE(*,6)
6     FORMAT(9X,'PRESS "S" TO STORE DATA ON DISK')
      WRITE(*,7)
7     FORMAT(9X,'PRESS "P" TO PRINT DATA ON PRINTER')
      WRITE(*,8)
8     FORMAT(9X,'PRESS "B" TO BOTH STORE AND PRINT DATA')
      WRITE(*,9)
9     FORMAT(9X,'PRESS "E" TO EXIT WITHOUT STORING OR
PRINTING')

      KEY=WAITKY()
      IF (KEY.EQ.80) GOTO 45
      IF (KEY.EQ.69) GOTO 80
      IF (KEY.NE.83) THEN
      IF (KEY.NE.80) THEN
      IF (KEY.NE.66) THEN
      IF (KEY.NE.69) THEN
      CALL BEEP(200,500)
      GO TO 4
      ENDIF
      ENDIF
      ENDIF
      ENDIF


      WRITE(*,10)
10    FORMAT(9X,'ENTER FILENAME FOR DATA',/)
      WRITE(*,15)
15    FORMAT(9X,'FORMAT - XXXXXXXX.XXX',/)
      READ(*,20)FNAME
20    FORMAT(A12)

      OPEN(3,FILE=FNAME,STATUS='NEW')
      DO 1 J=1,IVAL

WRITE(3,40)TIME(J),DATAR0(J),DATAR1(J),DATAR2(J),POWER(J)
     1   ,RHO(J)
40    FORMAT(2X,I5,2X,F8.3,2X,F8.5,2X,F8.3,2X,F8.3,2X,F8.3)
1     CONTINUE
      CLOSE(3,STATUS='KEEP')

      IF (KEY.EQ.83)GOTO 80

45    WRITE(*,50)
50    FORMAT(/,/,/,/,/)
      OPEN(2,FILE='PRN')
      WRITE(2,60)
60    FORMAT(5X,'TIME
```

```
TAG#',3X,'CURRENT',5X,'VOLTAGE',5X,'TEMPERATURE'
     2   ,2X,'POWER',/)
     DO 2 J=1,IVAL

WRITE(2,70)TIME(J),DATAR0(J),DATAR1(J),DATAR2(J),POWER(J)
70   FORMAT(5X,I5,3X,F8.3,5X,F8.3,5X,F8.3,2X,F8.3)
2    CONTINUE
     CLOSE(2)
80   RETURN
     END


C****      DEB END **************

C****      INTERUPT SERVICE ROUTINES ********************

     SUBROUTINE ISR1(IREG)

     INTEGER*2 STIM,JTIM,HTIM,ITIC
     INTEGER*2 IEXIT,IVAL,ILOAD

     COMMON/TIME/STIM,JTIM,HTIM,ITIC
     COMMON/FLAG/IEXIT,IVAL,ILOAD

     ITIC=ITIC-1                    !  DECREMENT LOOP VALUE
     IF (ITIC.NE.0)GO TO 10         !  LOOP FOR SAMPLE RATE
     ILOAD=1                        !  SET FLAG TO ACQUIRE DATA
     ITIC=STIM                      !  RESET LOOP VALUE
10   RETURN
     END



     SUBROUTINE ISR2(IREG)

     INTEGER*2 IREG(8)
     INTEGER*2 IEXIT,IVAL,ILOAD

     COMMON/FLAG/IEXIT,IVAL,ILOAD

     IEXIT=1                        !  SET FLAG ON ^BREAK
     RETURN
     END

     SUBROUTINE ISR3(IREG)

     INTEGER*2 IREG(8)
     INTEGER*2 IEXIT,IVAL,ILOAD
```

```
      COMMON/FLAG/IEXIT,IVAL,ILOAD

      IEXIT=1                          !  SET FLAG ON ^C
      RETURN
      END

C****      EXIT ************************************

      SUBROUTINE EXIT

      REAL*8 ANS
     *REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT

5     WRITE(*,10)
10    FORMAT(/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/)
      WRITE(*,15)
15    FORMAT(9X,'ENTER OPTION',/,/)
      WRITE(*,30)
30    FORMAT(9X,'RUN        ---    RUN PROGRAM AGAIN')
      WRITE(*,40)
40    FORMAT(9X,'QUIT       ---    EXIT TO DOS',/)
      READ(*,50)ANS
50    FORMAT(A8)
      IF (ANS.EQ.XRUN)GO TO 80
      IF (ANS.EQ.XQUIT)GO TO 90
      GO TO 5
80    RETURN
90    STOP
      END

C****      BLOCK DATA ***********************************

      BLOCKDATA

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(5000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD
      INTEGER*2 ICURRENT1,ICURRENT2,ICURRENT3
      INTEGER*2 INTERVAL1,INTERVAL2,INTERVAL3

      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
      CHARACTER*12 FNAME
```

```
      COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN
      COMMON/CURRENT/ICURRENT1,ICURRENT2,ICURRENT3
      COMMON/INTERVAL/INTERVAL1,INTERVAL2,INTERVAL3
      COMMON/FILE/FNAME
      COMMON/DATAR/DATARO,DATAR1,DATAR2

      DATA STIME/'STIM'/,JTIME/'JTIM'/,HTIME/'HTIM'/
      DATA JTIM/18/,HTIM/5/
      DATA IEXIT/0/,IVAL/0/
      DATA XSAVE/'SAVE'/,XPRINT/'PRINT'/,XEND/'END'/
      DATA XRETURN/'RETURN'/,XRUN/'RUN'/,XQUIT/'QUIT'/
      END
```

PROGRAM II : MASTER.BAS

```
2  REM ***********************************DEBASIS*
4  REM #########MASTER CONTROL FOR PULSE ###########
6  REM #############SAVED AS "MASTER"################
8  REM ***************************************
10 CLS:KEY OFF:SCREEN 7:COLOR 4,6
20 LOCATE 12,12:PRINT"╔═══════════════╗"
30 LOCATE 13,12:PRINT"║MASTER CONTROL ║"
40 LOCATE 14,12:PRINT"╚═══════════════╝"
50 FOR I=1 TO 25000!:NEXT I
60 CLS:SCREEN 7:SCREEN 8:COLOR 4,15
70 LOCATE 13,17:PRINT"PRESS SPACEBAR TO GO TO THE ACQUISITION
   PROGRAM"
80 LOCATE 14,17:PRINT"ANY OTHER KEY TO BYPASS ACQUISITION
   PROGRAM"
90 Z$=INKEY$:IF Z$="" THEN 90    'INTERCEPT OPTION KEYPRESS
100 SCREEN 9:SCREEN 0
110 IF Z$=" " THEN SHELL "CALPUL.EXE"  'FETCH ACQUISITION
    PROGRAM
120 CLS:SCREEN 7:SCREEN 8:COLOR 4,15
130 LOCATE 13,17
140 IF Z$=" " THEN PRINT"RETURNED FROM ACQUISITION PROGRAM"
150 IF Z$<>" " THEN PRINT"BYPASSED ACQUISITION PROGRAM"
160 LOCATE 14,17:INPUT"ENTER NAME OF THE FILE TO BE
    PROCESSED";F$
    'ENTER PULSE CHARACTERISTICS
170 CLS:LOCATE 14,17:INPUT"ENTER LENGTH OF INTERVAL
    1";INTERVAL1
180 LOCATE 15,17:INPUT"ENTER LENGTH OF INTERVAL 2";INTERVAL2
190 LOCATE 16,17:INPUT"ENTER LENGTH OF INTERVAL 3";INTERVAL3
200 LOCATE 17,17:INPUT"ENTER LENGTH OF COOLING PERIOD";INTCOOL
210 LOCATE 18,17:INPUT"ENTER HEATING TIC VALUE";ITIC
220 LOCATE 19,17:INPUT"ENTER COOLING TIC VALUE";ITICOOL
     'CALCULATE FLAG VALUES
230 INTERVAL3=INTERVAL1+INTERVAL2+INTERVAL3
240 INTERVAL2=INTERVAL1+INTERVAL2
250 INTEND=INTERVAL3+INTCOOL
260 DIM TIMEOLD(1200):DIM TEMPOLD(1200):DIM TIMENEW(1200):DIM
    TEMPNEW(1200)                  'DECLARE DIMENSIONS
270 DIM CURR(1200):DIM VOLT(1200):DIM POWER(1200):DIM
    RHO(1200)
280 OPEN "I",#1,F$       'OPEN INDICATED FILE TO READ RAW DATA
290 J=1
300 INPUT#1,TIMEOLD(J),CURR(J),VOLT(J),TEMPOLD(J),POWER(J),
    RHO(J)
```

```
310 IF EOF(1) THEN GOTO 340          'EXIT LOOP AT END OF FILE
320 J=J+1                            'INCREMENT LOOP NUMBER
330 GOTO 300
340 CLOSE #1
    'DECLARE COMMON VARIABLES TO CHAIN TO CONVERT PROGRAM
     WHICH CONVERTS EMF TO °C FOR PT-13%RH THERMOCOUPLES
350 COMMON TEMPOLD(),TIMEOLD(),POWER(),INTERVAL1,INTERVAL2,
    INTERVAL3,INTCOOL,INTEND,ITIC,ITICOOL,LAST,F$
360 CHAIN "CONVERT"                  'FETCH CONVERT INTO MEMORY
370 CLS:SCREEN 7:SCREEN 8:COLOR 4,15
380 CLS:LOCATE 13,17:PRINT"COPYING FILE....."
390 LOCATE 14,17:PRINT"ADDING EXTENSIONS....."
400 LOCATE 15,17:PRINT"SEARCHING  FOR  GRAPHER....."
410 DIM TIMENEWH(1200),TEMPH(1200),FIRSTH(1200)
420 DIM TIMENEWC(1200),TEMPC(1200),FIRSTC(1200)
430 REM HEAT FILE
440 FOR J=1 TO INTERVAL2-INTERVAL1     'CONVERT TT# TO SECOND
450 TIMENEWH(J)=J*ITIC/18.20651
460 NEXT J
470 G$=F$+"H.DAT"                     'ADD H EXTENSION
480 S$="COPY "+F$+" "+G$:SHELL S$
490 OPEN "O",#1,G$                   'OPEN HEAT FILE FOR OUTPUT
500 FOR J=INTERVAL1 TO INTERVAL2      'EXTRACT HEATING DATA
510 K=J-INTERVAL1                     'RESET CLOCK TO ZERO
520 PRINT#1,TIMENEWH(K),TEMPNEW(J),POWER(J)
530 NEXT J
540 CLOSE#1                           'CLOSE HEAT FILE
550 REM END HEAT
560 REM COOL FILE
570 FOR J=1 TO INTEND-INTERVAL3       'CONVERT TT# TO SECOND
580 TIMENEWC(J)=J*ITICOOL/18.20651
590 NEXT J
600 H$=F$+"C.DAT"                     'ADD C EXTENSION
610 S$="COPY "+F$+" "+H$:SHELL S$
620 OPEN "O",#2,H$                   'OPEN COOL FILE FOR OUTPUT
630 FOR J=INTERVAL3 TO INTEND         'EXTRACT COOLING DATA
640 K=J-INTERVAL3                     'RESET CLOCK TO ZERO
650 PRINT#2,TIMENEWC(K),TEMPNEW(J)
660 NEXT J
670 CLOSE#2                           'CLOSE COOL FILE
680 REM END COOL
690 SHELL "GRAPHER.EXE"               'FETCH GRAPHER
700 CLS:SCREEN 7:SCREEN 8:COLOR 4,15
710 LOCATE 14,17:PRINT"RETURNED FROM GRAPHER"
720 LOCATE 15,17:PRINT "ENTER COEFFICIENTS FOR Power vs. Temp
    FIT DURING HEATING"
    'STORE P=f(T) COEFFICIENTS IN VARIABLES
730 LOCATE 16,17:INPUT "A";AP
740 LOCATE 17,17:INPUT "B";BP
```

```
750 LOCATE 18,17:INPUT "C";CP
760 LOCATE 19,17:INPUT "D";DP
770 LOCATE 20,17:INPUT "E";EP
780 LOCATE 21,17:INPUT "F";FP
790 CLS
800 LOCATE 14,17:PRINT"ENTER COEFFICIENTS FOR Temp vs. Time
    FIT DURING HEATING"
    'STORE T=f(t) COEFFICIENTS FOR HEATING IN VARIABLES
810 LOCATE 15,17:INPUT"A";AH
820 LOCATE 16,17:INPUT"B";BH
830 LOCATE 17,17:INPUT"C";CH
840 LOCATE 18,17:INPUT"D";DH
850 LOCATE 19,17:INPUT"E";EH
860 LOCATE 20,17:INPUT"F";FH
870 FOR J= INTERVAL1 TO INTERVAL2
880 K=J-INTERVAL1
890 TIME=TIMENEWH(K)
900 TEMPH(J)=AH+BH*TIME+CH*TIME^2+DH*TIME^3+EH*TIME^4
    +FH*TIME^5
910 FIRSTH(J)=BH+2*CH*TIME+3*DH*TIME^2+4*EH*TIME^3
    +5*FH*TIME^4
920 NEXT J
930 OPEN "O",#3,G$                  'APPEND dT/dt DATA TO FILE
940 FOR J= INTERVAL1 TO INTERVAL2
950 K=J-INTERVAL1
960 PRINT#3,TIMENEWH(K),TEMPNEW(J),POWER(J),FIRSTH(J)
970 NEXT J
980 CLOSE#3                         'CLOSE APPENDED FILE
990 SHELL "GRAPHER.EXE"
1000 CLS:SCREEN 7:SCREEN 8:COLOR 4,15
1010 CLS:LOCATE 13,17:PRINT"RETURNED FROM GRAPHER"
1020 LOCATE 14,17:PRINT"ENTER COEFFICIENTS FOR dT/dt vs. Temp
     FIT DURING HEATING"
     'STORE dT/dt=f(T) COEFFICIENTS FOR HEATING IN VARIABLES
1030 LOCATE 15,17:INPUT"A";AH
1040 LOCATE 16,17:INPUT"B";BH
1050 LOCATE 17,17:INPUT"C";CH
1060 LOCATE 18,17:INPUT"D";DH
1070 LOCATE 19,17:INPUT"E";EH
1080 LOCATE 20,17:INPUT"F";FH
1090 SHELL "GRAPHER.EXE"
1100 CLS:SCREEN 7:SCREEN 8:COLOR 4,15
1110 CLS:LOCATE 13,17:PRINT"RETURNED FROM GRAPHER"
1120 LOCATE 15,17:PRINT "ENTER COEFFICIENTS FOR Temp vs. Time
     FIT DURING COOLING"
     'STORE T=f(t) COEFFICIENTS FOR COOLING IN VARIABLES
1130 LOCATE 16,17:INPUT "A";AC
1140 LOCATE 17,17:INPUT "B";BC
1150 LOCATE 18,17:INPUT "C";CC
```

```
1160 LOCATE 19,17:INPUT "D";DC
1170 LOCATE 20,17:INPUT "E";EC
1180 LOCATE 21,17:INPUT "F";FC
1190 FOR J=INTERVAL3 TO INTEND
'1200 K=J-INTERVAL3
1210 TIME=TIMENEWC(K)
1220 TEMPC(J)=AC+BC*TIME+CC*TIME^2+DC*TIME^3+EC*TIME^4
     +FC*TIME^5
1230 FIRSTC(J)=BC+2*CC*TIME+3*DC*TIME^2+4*EC*TIME^3
     +5*FC*TIME^4
1240 NEXT J
1250 OPEN "O",#3,H$              'APPEND SLOPE DATA TO FILE
1260 FOR J=INTERVAL3 TO INTEND
1270 K=J-INTERVAL3
1280 PRINT#3,TIMENEWC(K),TEMPNEW(J),TEMPC(J),FIRSTC(J)
1290 NEXT J
1300 CLOSE#3                     'CLOSE APPENDED FILE
1310 SHELL "GRAPHER.EXE"
1320 CLS:SCREEN 7:SCREEN 8:COLOR 4,15
1330 CLS:LOCATE 13,17:PRINT"RETURNED FROM GRAPHER"
1340 LOCATE 14,17:PRINT"ENTER COEFFICIENTS FOR dT/dt vs. Temp
     FIT DURING COOLING"
     'STORE dT/dt=f(T) COEFFICIENTS FOR COOLING IN VARIABLES
1350 LOCATE 15,17:INPUT"A";AC
1360 LOCATE 16,17:INPUT"B";BC
1370 LOCATE 17,17:INPUT"C";CC
1380 LOCATE 18,17:INPUT"D";DC
1390 LOCATE 19,17:INPUT"E";EC
1400 LOCATE 20,17:INPUT"F";FC
1410 CLS:LOCATE 13,17
1420  INPUT "ENTER TEMPERATURE AT WHICH C(p) IS TO BE
DETERMINED";TEMPI
1430 LENGTH=4!:DIA=.2395:RHO=8.902001 'SPECIMEN PHYSICAL DATA
     'CALCULATE POWER FOR HEATING
1440 POWER=AP+BP*TEMPI+CP*TEMPI^2+DP*TEMPI^3+EP*TEMPI^4
     +FP*TEMPI^5
     'CALCULATE dT/dt FOR HEATING
1450 FIRSTIH=AH+BH*TEMPI+CH*TEMPI^2+DH*TEMPI^3+EH*TEMPI^4
     +FH*TEMPI^5
     'CALCULATE dT/dt FOR COOLING
1460 FIRSTIC=AC+BC*TEMPI+CC*TEMPI^2+DC*TEMPI^3+EC*TEMPI^4
     +FC*TEMPI^5
     'CALCULATE MASS OF SPECIMEN
1470 VOL=(3.14159*DIA^2*LENGTH)/4:MASS=VOL*RHO
     'CALCULATE SPECIFIC HEAT
1480 SPHT=POWER/(MASS*(FIRSTIH-FIRSTIC))
1490 PRINT "THE SPECIFIC HEAT AT ";TEMPI;" °C IS ";SPHT
1500 GOTO 1420
```

PROGRAM III : SETUP.FOR

```
C****      03-11-91  **********************DEBASIS*

      PROGRAM SETUP VERSION 4.0

C****      SETUP WAS WRITTEN TO TAKE 100 INPUTS AND
C****      DISPLAY AVERAGE, THEN FROM THIS AVERAGE
C****    AND THE POTENTIOMETER OUTPUT CALCULATE
C****    THE GAIN.


C****      MAIN LOOP  ************************

      INTEGER*2
IWS1(22),IWS2(22),IWS3(22),IREG(8),IARRAY(101)
      INTEGER*2 ICHAN
      INTEGER*2 KEY,WAITKY,INKEY,IEXIT,ILOOP,ILOAD
      INTEGER*4 ICOUNT,J
      REAL*4 COUNT,DIAL,GAIN
      CHARACTER ANS

      EXTERNAL ISR1,ISR2,ISR3

      COMMON/INPUTS/ICHAN,ICOUNT,DIAL,GAIN
      COMMON/FLAG/IEXIT,ILOOP,ILOAD
      COMMON/ARRAY/J,IARRAY
      COMMON/KEYS/KEY

1     CALL PARAM

      IEXIT=0                  ! INITIALIZE LOOP VARIABLES
      ILOOP=0
      ILOAD=0
      ICOUNT=0
      J=1

10    WRITE(*,20)
20    FORMAT(/,/,1X,'** PRESS SPACEBAR TO BEGIN ACQUISITION
** ',/,/)
      KEY=WAITKY()
      IF (KEY.NE.32)GO TO 10

      WRITE(*,25)
25    FORMAT(/,/,8X,'-ACQUISITION IN PROGRESS-',/,/)
      CALL ISRSET(28,ISR1,IWS1)      ! INITIALIZE INTERRUPT
```

```
ROUTINE1
      CALL ISRSET(27,ISR2,IWS2)      ! SET UP ^BREAK
      CALL ISRSET(35,ISR3,IWS3)      ! SET UP ^C

30    KEY=INKEY()
      IF (KEY.EQ.32)GO TO 50           ! PRE-EXIT LOCATION
      IF (IEXIT.EQ.1)GO TO 50
      IF (ILOOP.EQ.100)GO TO 50
      IF (ILOAD.EQ.1)CALL LOAD         ! GO TO LOAD SUBROUTINE
      ILOAD=0
      GO TO 30

50    CALL ISRCLR(IWS1)          ! TERMINATE INTERRUPT ROUTINES
      CALL ISRCLR(IWS2)
      CALL ISRCLR(IWS3)

C****   CALCULATION AND DISPLAY MODULE

      COUNT=ICOUNT
      COUNT=COUNT/ILOOP                ! COMPUTE AVERAGE

      WRITE(*,60)COUNT
60    FORMAT(/,/,1X,'DEBS COUNT IS  ',F9.4)
      COUNT=COUNT*(10./4096.)
      GAIN=COUNT/DIAL                  ! COMPUTE GAIN
      WRITE(*,67)DIAL,COUNT
67    FORMAT(/,/,1X,'DIAL',F9.7,4X,'COMPUTER',F9.4)
      WRITE(*,65)ILOOP
65    FORMAT(/,/,1X,'NO OF LOOPS IS  ',I9)
      WRITE(*,68)GAIN
68    FORMAT(1X,'GAIN IS  ',F10.2)

      WRITE(*,70)
70    FORMAT(1X,'PRESS BACK-SLASH (\) TO VIEW INDIVIDUAL
COUNTS')

      KEY=WAITKY()
      IF (KEY.NE.92)GO TO 78
      DO 75, J=2,101                   ! PRINT ARRAY
      WRITE(*,72)J-1,IARRAY(J-1)
72    FORMAT(1X,'COUNT (',I3,') = ',I5)
75    CONTINUE

         CALL DISK                     ! CALL SAVE MODULE

78    WRITE(*,80)
80    FORMAT(/,/,1X,'DO YOU WANT TO RUN AGAIN ?   (Y/N)')
      KEY=WAITKY()
      IF (KEY.EQ.121)GO TO 1
```

```
      IF (KEY.EQ.89)GO TO 1
      STOP

      END

C****     PARAMETER ENTRY  ********************

      SUBROUTINE PARAM

      INTEGER*2 ICHAN
      INTEGER*4 ICOUNT
      REAL*4 DIAL

      COMMON/INPUTS/ICHAN,ICOUNT,DIAL,GAIN
      COMMON/ARRAY/J,IARRAY

C****     READ DIAL-A-SOURCE VOLTAGE
      WRITE(*,2)
2     FORMAT(/,/,1X,'INPUT DIAL-A-SOURCE VOLTAGE',/,/)
      READ(*,5)DIAL
5     FORMAT(F9.7)
      WRITE(*,10)
10    FORMAT(/,/,/,/,/,/,/,/,/,/,/,/,/,/)
      WRITE(*,20)
20    FORMAT(/,1X,'ENTER INPUT CHNNEL# (0-7)')
      READ(*,30)ICHAN
30    FORMAT(I5)
      RETURN
      END


C****     LOAD SUBROUTINE  ********************

      SUBROUTINE LOAD

      INTEGER*2 ICHAN
      INTEGER*2 IEXIT,ILOOP,ILOAD
      INTEGER*4 ICOUNT,IN
      INTEGER*2 IARRAY(100)
      INTEGER*2 J

      COMMON/INPUTS/ICHAN,ICOUNT,DIAL,GAIN
      COMMON/FLAG/IEXIT,ILOOP,ILOAD
      COMMON/ARRAY/J,IARRAY

      CALL ALAV(ICHAN,1,IN)  ! ACQUIRE SIGNAL ON CHANNEL#
SPECIFIED BY ICHAN
      J=J+1                          ! INCREMENT ARRAY NUMBER
      IARRAY(J)=IN                   ! STORE ACQUIRED SIGNAL
```

```
IN ARRAY

      ICOUNT=ICOUNT+IN              ! CALCULATE CUMULATIVE
COUNTS
      ILOOP=ILOOP+1
      WRITE (*,10)ICOUNT
'10   FORMAT(/,/,1X,'VALUE OF ICOUNT IS  'I9,/,/)
      WRITE (*,20)IN
20    FORMAT(/,/,1X,'VALUE OF IN IS  'I8,/,/)

      RETURN
      END

C**** SAVE AND PRINT MODULE*******************************

      SUBROUTINE DISK

      REAL*4 COUNT,DIAL,GAIN
      INTEGER*2 IARRAY(101)
      INTEGER*2 ICHAN
      INTEGER*4 ICOUNT,J
      CHARACTER*12 FNAME
      INTEGER*2 KEY,WAITKY

      COMMON /INPUTS/ICHAN,ICOUNT,DIAL,GAIN
      COMMON /ARRAY/J,IARRAY
      COMMON /KEYS/KEY

      WRITE(*,5)
5     FORMAT(2X,'╔═══════════════════════════════╗')
      WRITE(*,10)
10    FORMAT(2X,'‖ENTER A FILENAME FOR DATA‖')
      WRITE(*,15)
15    FORMAT(2X,'╚═══════════════════════════════╝')
      READ(*,20)FNAME
20    FORMAT(A12)
      WRITE(*,30)
30    FORMAT(/,1X,'DO YOU WANT TO STORE DATA IN DISK ?')
      KEY=WAITKY()

      IF(KEY.EQ.89) GO TO 40
      IF(KEY.EQ.121) GO TO 40
      IF(KEY.NE.89)GO TO 78
      IF(KEY.NE.121)GO TO 78

40    OPEN (3,FILE=FNAME,STATUS='NEW')
      WRITE(3,50)FNAME
50    FORMAT(1X,'THIS DATA HAS BEEN STORED UNDER FILENAME -
',A12)
```

```
      WRITE(3,60)ICHAN
60    FORMAT(/,1X,'THE CHANNEL# IS 'I5)
      WRITE(3,70)GAIN
70    FORMAT(/,1X,'GAIN IS ',F8.2)
      DO 77, J=1,101
      WRITE(3,75)J-1,IARRAY(J-1)
'75   FORMAT(1X,'COUNT (',I3,')= 'I5)
77    CONTINUE
      CLOSE(3,STATUS='KEEP')

78 -  WRITE(*,79)
79    FORMAT(/,1X,'DO YOU WANT A PRINTOUT ?')
      KEY=WAITKY()

      IF (KEY.EQ.89)GO TO 80
      IF (KEY.EQ.121)GO TO 80
      IF (KEY.NE.89) GO TO 140
      IF (KEY.NE.121) GO TO 140

80    WRITE(*,82)
82    FORMAT(/,/,/,/,/)
      OPEN(2,FILE='PRN')
      WRITE(2,90)FNAME
90    FORMAT(/,1X,'THIS DATA  HAS BEEN STORED UNDER FILENAME
- ',A12)
      WRITE(2,100)ICHAN
100   FORMAT(/,1X,'THE CHANNEL# IS',I5)
      WRITE(2,110)GAIN
110   FORMAT(/,1X,'GAIN IS ',F8.2)
      DO 130, J=1,101
      WRITE(2,120)J-1,IARRAY(J-1)
120   FORMAT(1X,'COUNT (',I3,')= 'I5)
130   CONTINUE
      CLOSE(2)
140   RETURN
      END



C****      INTERUPT SUBROUTINE  ***************

      SUBROUTINE ISR1(IREG)

      INTEGER*2 IREG(8)
      INTEGER*2 IEXIT,ILOOP,ILOAD
      COMMON/FLAG/IEXIT,ILOOP,ILOAD
```

```
      ILOAD=1

      RETURN
      END

      SUBROUTINE ISR2(IREG)

      INTEGER*2 IREG(8)
      INTEGER*2 IEXIT,ILOOP,ILOAD
      COMMON/FLAG/IEXIT,ILOOP,ILOAD

      IEXIT=1

      RETURN
      END

      SUBROUTINE ISR3(IREG)

      INTEGER*2 IREG(8)
      INTEGER*2 IEXIT,ILOOP,ILOAD
      COMMON/FLAG/IEXIT,ILOOP,ILOAD

      IEXIT=1

      RETURN
      END
```

PROGRAM IV : CONVERT.BAS

```
10 REM ********************************DEBASIS*
20 REM ############CONVERT FOR PULSE############
30 REM ############NBS MONOGRAPH 125######R TYPE#
40 REM ***********SAVED AS "CONVERT"**********
50 REM TEMPERATURE FROM THERMOELECTRIC VOLTAGE FOR
   PLATINUM-13%RHODIUM
60 DIM TEMPNEW(1200)
70 CLS:KEY OFF:SCREEN 7:COLOR 11,13
80 LOCATE 10,10:PRINT"CONVERTING mV TO °C.."
90 FOR I=1 TO 2              'READ BOTH SET OF COEFFICIENTS
100 READ A(I),B(I),C(I),D(I),E(I),F(I)
110 NEXT I
120 RESTORE
130 FOR J=1 TO INTEND        'CONVERT MICROVOLTS TO MILLIVOLTS
140 TEMPOLD(J)=TEMPOLD(J)*1000
150 EMF=TEMPOLD(J)
160 IF EMF>0# AND EMF<3407# THEN I=1   'SELECT EQUATION
170 IF EMF> 3407# AND EMF<11846# THEN I=2
180 IF EMF<0# OR EMF>11846# THEN LOCATE 10,22:PRINT
    "TEMPERATURE OUT OF RANGE":PLAY"MBDF":GOTO 80
190TEMPNEW(J)=A(I)+B(I)*EMF+C(I)*EMF^2+D(I)*EMF^3+E(I)*EMF^4
    +F(I)*EMF^5              'COMPUTE TEMPERATURE FROM EMF
200 NEXT J
210 REM SUBROUTINE DATA
220 DATA 6.414181,1.586447D-1,-2.464001D-5,5.013749D-9,
    -4.396420D-13,0
230DATA4.5509556D1,1.1284875D-1,-2.8603978D-6,8.5173702D-11,
    -1.1440038D-15,0
240 COMMON TIMENEW(),TEMPNEW(),TIMEOLD(),POWER(),INTERVAL1,
    INTERVAL2,INTERVAL3,INTCOOL,INTEND,ITIC,ITICOOL,F$
250 CHAIN "MASTER",370       'GO BACK TO MASTER
```

PROGRAM V : CALRES.FOR

```
C****      09-30-91**********************************DEBASIS*

      -       PROGRAM RESISTIVITY

C****      SAVED AS "CALRES"

C****      THIS PROGRAM APPLIES A USER SPECIFIED CURRENT TO
THE SAMPLE
C****      FOR A SPECIFIED TIME AND CALCULATES THE E/I RATIO
AND
C****      RESISTIVITY FOR EACH READING. AT THE END OF THE
TEST IT
C****      DISPLAYS THE CALCULATED AVERAGE RESISTIVITY.

C****      DEFINE VARIABLES ***************************

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 IWS1(22),IWS2(22),IWS3(22),IREG(8),IVAL,IADD
      INTEGER*2 TIME(2000),IN
      INTEGER*2 IEXIT,ILOAD
      INTEGER*2 KEY,INKEY,WAITKY
      INTEGER*2 INTERVAL
      INTEGER*2 ICURRENT

      REAL*4 DATAR0(2000),DATAR1(2000),DATAR2(2000)
      REAL*8
RATIO(2000),RES(2000),RHOSUM,RHOAVG,DR2SUM,DR2AVG
      REAL*8 DIA,ELENGTH
      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
      CHARACTER*12 FNAME

      EXTERNAL ISR1,ISR2,ISR3

C****      ESTABLISH COMMON LOCATIONS ****************

      COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT
      COMMON/TEMP/TEMP1
```

```
      COMMON/VOLT/VOLT1.VOLT2
      COMMON/FLAG/IEXIT.IVAL.ILOAD
      COMMON/INPUTS/TIME.IN
      COMMON/FILE/FNAME
      COMMON/CURRENT/ICURRENT
      COMMON/INTERVAL/INTERVAL
      COMMON/SAMPLE/DIA.ELENGTH
      COMMON/DATAR/DATAR0,DATAR1.DATAR2,RES

C****     PRE MAIN LOOP ********************************

      CALL ALDV(0,2048)              !  SET D/A TO 0
5     WRITE(*,10)
10    FORMAT(1X,/,/,/,/,/,/,/,/,/,/)
      WRITE(*,20)
20    FORMAT(9X,'** PROGRAM - RESISTIVITY VERSION 1.0 **
',/,/)
      WRITE(*,30)
30    FORMAT(13X,'CAPS LOCK MUST BE ON',/,/,/)
      WRITE(*,70)
70    FORMAT(9X,'SHUNT CURRENT INPUT 1    ON A/D CH0')
      WRITE(*,75)
75    FORMAT(9X,'SAMPLE VOLTAGE INPUT 2    ON A/D CH1')
      WRITE(*,80)
80    FORMAT(9X,'TEMPERATURE INPUT 3       ON A/D CH2')
      WRITE(*,82)
82    FORMAT(9X,'KEPCO CURRENT OUTPUT 1 ON D/A CH0',/,/)
      WRITE(*,85)
85    FORMAT(9X,'**** SPACE TO PROCEED ****')
      KEY=WAITKY()
      IF (KEY.NE.32)GO TO 5

C****     MAIN LOOP ************************************

115   CALL PARAM
117   IVAL=0
      ITIC=STIM                      !  SET LOOP FOR SAMPLE
RATE
C     CALL WTIME(0,0,0,0)            !  SET CLOCK
      CALL ISRSET(28,ISR1,IWS1)      !  INITIALIZE ISR1
      CALL ISRSET(27,ISR2,IWS2)      !  SET UP ^BREAK ISR
      CALL ISRSET(35,ISR3,IWS3)      !  SET UP ^C ISR

      ICO1=(ICURRENT*2.29)+2048      !  CALCULATE OUTPUT TO
MATCH RANGE
C     ICO2=(ICURRENT2*2.29)+2048
C     ICO3=(ICURRENT3*2.29)+2048
```

```
C120        KEY=INKEY()
120   IF(ILOAD.EQ.0)GO TO 125          !  DEB CHANGE(120 ON)
      CALL LOAD                        !  ACQUIRE DATA
C     WRITE(*,100)INTERVAL1,INTERVAL2,INTERVAL3,IVAL
C100  FORMAT(5X,I5,5X,I5,5X,I5,5X,I5)

C****   RESISTIVITY CONTROL
ROUTINE**************************

C     DATAR1(IVAL)=REAL(DATA1(IVAL))
C     DATAR2(IVAL)=REAL(DATA2(IVAL))
      RATIO(IVAL)=DATAR1(IVAL)/DATAR0(IVAL)
      RES(IVAL)=((3.1415*DIA**2)/(400.*ELENGTH))*
     1   (DATAR1(IVAL)/DATAR0(IVAL))*1D4
      IF(IVAL.GT.INTERVAL) THEN
      WRITE(*,500)INTERVAL
500   FORMAT(/,/,12X,'PAST  TIME  TAG#',I5,8X,
     1   ' END OF RESISTIVITY TEST.')       ! DEB CHANGE(AUTO
STOP)
      IVAL=IVAL-1
      RHOSUM=0
      DR2SUM=0
      DO 600 IADD=1,IVAL
      RHOSUM=RHOSUM+RES(IADD)
      DR2SUM=DR2SUM+DATAR2(IADD)
600   CONTINUE
      RHOAVG=RHOSUM/IVAL
      DR2AVG=DR2SUM/IVAL
      WRITE(*,700)
700   FORMAT(12X,'THE CALCULATED AVERAGE OF RESISTIVITY
FOR',1X)
      WRITE(*,710)IVAL,RHOAVG
710   FORMAT(9X,I5,' READINGS = ',F10.4,' µOHM-CM')
      WRITE(*,720)DR2AVG
720   FORMAT(12X,'AVERAGE TEMPERATURE CORRESPONDS TO '
     1   ,F6.3,' MILLIVOLTS')

      KEY=WAITKY()                    ! DEB CHANGE(KEY 2:VIEW
ENABLE)
      IF (KEY.EQ.80) CALL DISK     ! DEB REM(LINE# 140 EXIT
COMMON POINT)
      GO TO 140
      ENDIF

C****      END OF CONTROL************

WRITE(*,121)TIME(IVAL),DATAR0(IVAL),DATAR1(IVAL),DATAR2(IVAL
```

```
),
     1  RATIO(IVAL),RES(IVAL)          !  SCREEN OUTPUT
121
FORMAT('1',2X,I5,3X,F8.3,3X,F8.3,3X,F8.3,5X,F8.4,3X,F10.4)
C125  IF (KEY.EQ.32)GO TO 140          !  DEB CHANGE (STOP FLAG
OFF)
125   IF (IEXIT.NE.0)GO TO 140        !  IF ON 'C OR  BREAK:DEB
CH(125 ON)
130   IF (IVAL.NE.20000)GO TO 120     !  IF ON MAX ARRAY SIZE
140   CALL  ALDV(0,2048)              !  SET D/A TO 0
      CALL ISRCLR(IWS1)               !  TERMINATES ISR1
      CALL ISRCLR(IWS2)               !  TERMINATES ISR2
      CALL ISRCLR(IWS3)               !  TERMINATES ISR3
      CALL ALDV(0,2048)               !  SET A/D TO 0
      CALL EXIT                       !  EXIT
      GO TO 5      !  FLOW RETURNS TO THIS POINT ON. RUN AGAIN

      STOP
      END

C*****      KEYBOARD ENTRY ***************************************

      SUBROUTINE PARAM

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(2000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD
      INTEGER*2 KEY,WAITKY
      INTEGER*2 ICURRENT
      INTEGER*2 INTERVAL

      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
      CHARACTER*12 FNAME

      COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN
      COMMON/CURRENT/ICURRENT
      COMMON/INTERVAL/INTERVAL
      COMMON/FILE/FNAME
```

```
60    CALL PULSE    !  D/A SETUP
62    WRITE(*,70)
      WRITE(*,63)
63    FORMAT(8X,/,/,/,/,/,/,/)
      WRITE(*,65)
65    FORMAT(15X,'*** PRESS SPACEBAR TO BEGIN TEST ***',/)

C -   WRITE(*,66)
C66   FORMAT(24X,'AND START DATA ACQUISITION',/,/,/,/,/,/)
      WRITE(*,67)
67    FORMAT(14X,'THIS PROGRAM AUTOMATICALLY STOPS THE
TEST',/)
      WRITE(*,69)
69    FORMAT(28X,'AT THE SPECIFIED TIME',/)
      WRITE(*,691)INTERVAL
691   FORMAT(19X,'(TIME TAG#',I5,' FOR THIS RUN).',/)
      WRITE(*,68)
68       FORMAT(15X,'PERFORMS THE AVERAGE AND RETURNS
CONTROL.',/)
      KEY=WAITXY()
      IF (KEY.NE.32)GO TO 62
      WRITE(*,70)
70    FORMAT(/,/,/,/,/,/,/,/)
      WRITE(*,80)
80    FORMAT(15X,'*** RESISTIVITY TEST IN PROGRESS ***',/,/)
      WRITE(*,90)
90    FORMAT(10X,'---  CTRL BREAK WILL ABORT TEST  ---'
      1     ,/,/,/)
      WRITE(*,100)
100   FORMAT(5X,'TIME
TAG#',3X,'CURRENT',9X,'VOLT',5X,'TEMPERATURE',6X,
      2  'E/I',11X,'RHO')
      WRITE(*,200)
200   FORMAT(4X,'(READING#)',3X,'(I:AMPS)',7X,'(E:MV)',
      3  3X,'(T:MV)',5X,'(R:MOHMS)',2X,'(μOHM-CM)',/)

      RETURN
      END


C****      PULSE SPECIFICATIONS
**********************************

      SUBROUTINE PULSE

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
```

```
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(2000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD
      INTEGER*2 KEY,WAITKY
      INTEGER*2 ICURRENT
      INTEGER*2 INTERVAL

     *  REAL*8 DIA,ELENGTH
      REAL*8
     STIME,JTIME,HTIME,KSAVE,KPRINT,KEND,KRETURN,KRUN,KQUIT
      CHARACTER*12 FNAME

      COMMON/TIME/STIM,JTIM,HTIM,ITI?

     COMMON/ID/STIME,JTIME,HTIME,KSAVE,KPRINT,KEND,KRETURN,KRUN,K
     QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN
      COMMON/FILE/FNAME
      COMMON/CURRENT/ICURRENT
      COMMON/INTERVAL/INTERVAL
      COMMON/SAMPLE/DIA,ELENGTH



5     WRITE(*,10)
10    FORMAT(/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/)
      WRITE(*,15)
15    FORMAT(12X,'**ENTER TEST PARAMETERS**')
      WRITE(*,16)
16    FORMAT(12X,'==========================',/,/)
      WRITE(*,20)
20    FORMAT(9X,'ENTER THE VALUE OF CURRENT IN "AMPS"',/)
      WRITE(*,30)
      READ(*,30)ICURRENT
30    FORMAT(I5)
      WRITE(*,31)
31    FORMAT(9X,'ENTER DIAMETER OF SAMPLE IN "mm"',/)
      READ(*,32)DIA
32    FORMAT(F9.4)
      WRITE(*,33)
33    FORMAT(9X,'ENTER EFFECTIVE LENGTH OF SAMPLE IN "cm"',/)
      READ(*,34)ELENGTH
34    FORMAT(F9.4)
```

```
C     WRITE(*,40)
C40   FORMAT(9X,'ENTER CURRENT FOR INTERVAL 2  IN "AMPS"',/)
C     READ(*,50)ICURRENT2
C50   FORMAT(I5)
C     WRITE(*,60)
C60   FORMAT(9X,'ENTER CURRENT FOR INTERVAL 3 IN "AMPS"',/)
C     READ(*,70)ICURRENT3
C70   FORMAT(I5)



      WRITE(*,80)
80    FORMAT(9X,'ENTER TIME FOR OUTPUT TO BE ACTIVE AS
"TT#"',/)
      READ(*,90)INTERVAL
90    FORMAT(I5)
C     WRITE(*,100)
C100  FORMAT(9X,'ENTER TIME OF INTERVAL 2 AS "TT#"',/)
C     READ(*,110)INTERVAL2
C110  FORMAT(I5)
C     WRITE(*,120)
C120  FORMAT(9X,'ENTER TIME OF INTERVAL 3 AS "TT#"',/)
C     READ(*,130)INTERVAL3
C130  FORMAT(I5)

      WRITE(*,140)
140   FORMAT(9X,'TIME BETWEEN SUBSEQUENT DATA RECORDINGS
"TICS" ?',/)
      READ(*,150)STIM
150   FORMAT(I5)
C     INTERVAL3=INTERVAL1+INTERVAL2+INTERVAL3
C     INTERVAL2=INTERVAL1+INTERVAL2
C     WRITE(*,180)
C180  FORMAT(/,/)
C     READ(*,190)CURRENT
C190  FORMAT(I5)
C     IF (CURRENT.GT.90)GO TO 5
C     WRITE(*,100)CURRENT
C200  FORMAT(9X,'NEW VALUE OF CURRENT IS  ',I5,' AMPS',/)
C     WRITE(*,110)
C210  FORMAT(9X,'IS THIS VALUE OK  "Y/N"',/,/)
C     KEY=WAITKY()
C     IF (KEY.NE.89)GO TO 5
      RETURN
      END


C****      LOAD ARRAYS    ********************************
```

```
      SUBROUTINE LOAD

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(2000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD
      INTEGER*2 ICURRENT
      INTEGER*2 INTERVAL

      REAL*4 DATAR0(2000),DATAR1(2000),DATAR2(2000)
      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT


      COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN
      COMMON/CURRENT/ICURRENT
      COMMON/INTERVAL/INTERVAL
      COMMON/DATAR/DATAR0,DATAR1,DATAR2

      IVAL=IVAL+1                    !  INCREMENT SAMPLE NUMBER
      CALL ALAV(0,1,IN)          !  ADC CH0 TEMP1
      DATAR0=(100./4096.)*FLOAT(IN)    !  SCALE INPUT TO MATCH
RANGE
C     DATA0(IVAL)=IN             !  LOAD ARRAY 0
      CALL ALAV(1,1,IN)          !  ADC CH1 VOLT1
      DATAR1(IVAL)=(10./4096.)*FLOAT(IN)
      DATAR1(IVAL)=DATAR1(IVAL)*10
C     DATAR1(IVAL)=REAL(IN)
      CALL ALAV(2,1,IN)          !  ADC CH2 CURR2
      DATAR2(IVAL)=(10./4096.)*FLOAT(IN)
C     DATAR2(IVAL)=REAL(IN)
      TIME(IVAL)=IVAL       !  ARRAY FOR SAMPLE NUMBER
      ILOAD=0               !  RESET LOAD FLAG
      RETURN
      END


C****      SAVE MODULE **********************************

      SUBROUTINE DISK
```

```
      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(2000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD
      REAL*4 DATAR0(2000),DATAR1(2000),DATAR2(2000)
      REAL*8 RES(2000)
      REAL*8
     *STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
      CHARACTER*12 FNAME

      COMMON/TIME/STIM,JTIM,HTIM,ITIC

     *COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
     *QUIT
      COMMON/FLAG/IEXIT,IVAL,ILOAD
      COMMON/INPUTS/TIME,IN,CURRENT
      COMMON/DATAR/DATAR0,DATAR1,DATAR2,RES
      COMMON/FILE/FNAME

      WRITE(*,5)
5     FORMAT(/,/,/,/,/)
      WRITE(*,10)
10    FORMAT(9X,'ENTER FILENAME FOR DATA',/)
      WRITE(*,15)
15    FORMAT(9X,'EXAMPLE - XXXXXXXX.XXX',/)
      READ(*,20)FNAME
20    FORMAT(A12)

      OPEN (3,FILE=FNAME,STATUS='NEW')
      WRITE(3,30)
30    FORMAT(2X,'TEMP',2X,'VOLT',2X,'CURRENT')
      DO 1 I=1 ,IVAL
      WRITE(3,40)TIME(I),DATAR0(I),DATAR1(I),DATAR2(I),RES(I)
40    FORMAT(2X,I5,2X,F8.3,2X,F8.3,2X,F8.3,2X,F10.4)
1     CONTINUE
      CLOSE(3,STATUS='KEEP')

      WRITE(*,50)
50    FORMAT(/,/,/,/,/,/)
      OPEN(2,FILE='PRN')
      WRITE(2,60)
60    FORMAT(2X,'TIME',2X,'TEMP',2X,'VOLT',2X,'CURRENT',/)
      DO 2 I=1,IVAL
      WRITE(2,70)TIME(I),DATAR0(I),DATAR1(I),DATAR2(I)
70    FORMAT(2X,I5,2X,F8.3,2X,F8.3,2X,F8.3)
2     CONTINUE
      CLOSE(2)
      RETURN
```

```
      END
C****   SELF REMINDER :THIS IS WHERE I LEFT

C****      INTERUPT SERVICE ROUTINES ********************

      SUBROUTINE ISR1(IREG)

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 IEXIT,IVAL,ILOAD

      COMMON/TIME/STIM,JTIM,HTIM,ITIC
      COMMON/FLAG/IEXIT,IVAL,ILOAD

      ITIC=ITIC-1                    ! DECREMENT LOOP VALUE
      IF (ITIC.NE.0)GO TO 10         ! LOOP FOR SAMPLE RATE
      ILOAD=1                        ! SET FLAG TO ACQUIRE DATA
      ITIC=STIM                      ! RESET LOOP VALUE
10    RETURN
      END


      SUBROUTINE ISR2(IREG)

      INTEGER*2 IREG(8)
      INTEGER*2 IEXIT,IVAL,ILOAD

      COMMON/FLAG/IEXIT,IVAL,ILOAD

      IEXIT=1                        ! SET FLAG ON ^BREAK
      RETURN
      END

      SUBROUTINE ISR3(IREG)

      INTEGER*2 IREG(8)
      INTEGER*2 IEXIT,IVAL,ILOAD

      COMMON/FLAG/IEXIT,IVAL,ILOAD

      IEXIT=1                        ! SET FLAG ON ^C
      RETURN
      END

C****      EXIT ***************************************

      SUBROUTINE EXIT
```

```
      REAL*8 ANS
      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT

5     WRITE(*,10)
10    FORMAT(/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/,/)
      WRITE(*,15)
15    FORMAT(9X,'ENTER OPTION',/,/)
      WRITE(*,30)
30    FORMAT(9X,'R  ---    RUN PROGRAM AGAIN')
      WRITE(*,40)
40    FORMAT(9X,'Q  ---    EXIT TO DOS',/)
      READ(*,50)ANS
50    FORMAT(A8)
      IF (ANS.EQ.XRUN)GO TO 80
      IF (ANS.EQ.XQUIT)GO TO 90
      GO TO 5
80    RETURN
90    STOP
      END

C*****      BLOCK DATA **********************************

      BLOCKDATA

      INTEGER*2 STIM,JTIM,HTIM,ITIC
      INTEGER*2 TEMP1
      INTEGER*2 VOLT1,VOLT2
      INTEGER*2 TIME(2000),IN
      INTEGER*2 IEXIT,IVAL,ILOAD,IADD
      INTEGER*2 ICURRENT
      INTEGER*2 INTERVAL

      REAL*8 RATIO,RES,RHOSUM,RHOAVG
      REAL*8 DIA,ELENGTH
      REAL*8
STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,XQUIT
      CHARACTER*12 FNAME

      COMMON/TIME/STIM,JTIM,HTIM,ITIC

COMMON/ID/STIME,JTIME,HTIME,XSAVE,XPRINT,XEND,XRETURN,XRUN,X
QUIT
      COMMON/TEMP/TEMP1
      COMMON/VOLT/VOLT1,VOLT2
      COMMON/FLAG/IEXIT,IVAL,ILOAD
```

```
      [illegible]
      [illegible]
      [illegible]
      [illegible]
      [illegible]
      [illegible]

      DATA [illegible]
      DATA [illegible]
      DATA [illegible]
      DATA [illegible]
      DATA [illegible]
      END
```

Note - The source codes and the executable files of the four programs listed in this appendix are available upon request from

Debasis Basak
Room 422, Dougherty Engineering Building
University of Tennessee
Phone 615-974-5551

or, in his absence, from

Dr. J. S. Brooks
Professor, Dept. of Materials Science and Engineering
University of Tennessee
Phone 615-974-5814

APPENDIX III

RAW DATA FROM A TYPICAL RUN

TIME TAG#	CURRENT (AMPERES)	VOLTAGE (VOLTS)	TEMPERATURE (MILLIVOLTS)	POWER (WATT)
1	.220	.00000	.100	.000
2	.244	.00000	.100	.000
3	.269	.00366	.103	.001
4	.220	.00171	.100	.000
5	.244	.00293	.100	.001
6	.269	.00000	.103	.000
7	37.866	.03540	.103	1.340
8	38.159	.03613	.110	1.379
9	38.232	.03613	.125	1.381
10	38.159	.03638	.122	1.388
11	38.184	.03613	.129	1.380
12	38.232	.03662	.142	1.400
13	38.159	.03638	.139	1.388
14	38.184	.03687	.146	1.408
15	38.232	.03687	.161	1.409
16	38.159	.03711	.156	1.416
17	38.184	.03711	.164	1.417
18	38.232	.03735	.176	1.428
19	38.159	.03760	.176	1.435
20	38.184	.03760	.183	1.436
21	38.232	.03784	.195	1.447
22	38.159	.03784	.193	1.444
23	38.184	.03809	.198	1.454
24	38.232	.03809	.215	1.456
25	38.159	.03833	.212	1.463
26	38.184	.03857	.220	1.473
27	38.232	.03857	.234	1.475
28	38.159	.03882	.232	1.481
29	38.184	.03906	.237	1.492
30	38.232	.03906	.251	1.493
31	38.159	.03906	.249	1.491
32	38.184	.03955	.259	1.510
33	38.232	.03979	.271	1.521
34	38.159	.03955	.269	1.509
35	38.184	.03979	.276	1.520
36	38.257	.04004	.291	1.532
37	38.159	.04028	.288	1.537
38	38.184	.04053	.298	1.547
39	38.232	.04053	.310	1.549
40	38.159	.04053	.308	1.546
41	38.184	.04077	.317	1.557

42	38.257	.04102	.330	1.569
43	38.159	.04102	.330	1.565
44	38.184	.04126	.337	1.575
45	38.232	.04126	.352	1.577
46	38.159	.04126	.347	1.574
47	38.184	.04175	.359	1.594
48	38.232	.04175	.371	1.596
49	38.159	.04224	.369	1.612
50	38.208	.04224	.381	1.614
51	38.232	.04248	.393	1.624
52	38.159	.04248	.391	1.621
53	38.184	.04297	.400	1.641
54	38.232	.04272	.415	1.633
55	38.159	.04321	.413	1.649
56	38.184	.04346	.420	1.659
57	38.232	.04321	.435	1.652
58	38.159	.04346	.435	1.658
59	38.184	.04370	.442	1.669
60	38.232	.04370	.457	1.671
61	38.159	.04419	.454	1.686
62	38.184	.04395	.464	1.678
63	38.208	.04443	.479	1.698
64	38.159	.04443	.476	1.696
65	38.184	.04468	.486	1.706
66	38.208	.04492	.500	1.716
67	38.184	.04492	.498	1.715
68	38.184	.04517	.510	1.725
69	38.208	.04541	.522	1.735
70	38.184	.04565	.520	1.743
71	38.184	.04565	.532	1.743
72	38.184	.04590	.544	1.753
73	38.184	.04614	.544	1.762
74	38.184	.04639	.554	1.771
75	38.184	.04639	.569	1.771
76	38.184	.04639	.569	1.771
77	38.184	.04688	.579	1.790
78	38.159	.04688	.586	1.789
79	38.184	.04712	.591	1.799
80	38.184	.04712	.601	1.799
81	38.159	.04761	.608	1.817
82	.513	.00073	.608	.000
83	.244	.00049	.605	.000
84	.269	.00024	.605	.000
85	.244	.00049	.605	.000
86	.244	.00049	.603	.000
87	.269	.00049	.601	.000
88	.244	.00049	.601	.000
89	.244	.00049	.601	.000
90	.244	.00049	.598	.000

91	.244	.00024	.598	.000
92	.244	.00049	.596	.000
93	.269	.00049	.596	.000
94	.244	.00024	.593	.000
95	.244	.00000	.591	.000
96	.244	.00049	.593	.000
97	.244	.00049	.591	.000
98	.244	.00024	.588	.000
99	.269	.00073	.588	.000
100	.244	.00024	.586	.000
101	.244	.00024	.583	.000
102	.269	.00049	.583	.000
103	.244	.00049	.581	.000
104	.244	.00049	.581	.000
105	.244	.00049	.581	.000
106	.244	.00049	.579	.000
107	.244	.00024	.579	.000
108	.269	.00024	.576	.000
109	.244	.00049	.574	.000
110	.244	.00049	.574	.000
111	.269	.00024	.574	.000
112	.244	.00024	.569	.000
113	.244	.00049	.571	.000
114	.244	.00049	.569	.000
115	.244	.00049	.569	.000
116	.244	.00049	.566	.000
117	.244	.00024	.564	.000
118	.220	.00024	.566	.000
119	.244	.00024	.564	.000
120	.244	.00049	.562	.000
121	.244	.00049	.562	.000
122	.244	.00049	.562	.000
123	.269	.00049	.559	.000
124	.220	.00024	.559	.000
125	.244	.00024	.557	.000
126	.244	.00024	.557	.000
127	.244	.00024	.554	.000
128	.220	.00049	.554	.000
129	.269	.00049	.554	.000
130	.244	.00049	.552	.000
131	.244	.00049	.552	.000
132	.244	.00024	.549	.000
133	.220	.00049	.547	.000
134	.220	.00024	.547	.000
135	.269	.00024	.547	.000
136	.220	.00049	.542	.000
137	.220	.00049	.544	.000
138	.269	.00024	.542	.000
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148	.220	.00049	.532	.000
149	.220	.00049	.530	.000
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152	.220	.00024	.530	.000
153	.244	.00024	.527	.000
154	.220	.00024	.525	.000
155	.220	.00049	.525	.000
156	.269	.00024	.525	.000
157	.220	.00049	.522	.000
158	.244	.00024	.522	.000
159	.269	.00049	.522	.000
160	.220	.00049	.520	.000
161	.244	.00073	.520	.000
162	.244	.00049	.522	.000
163	.220	.00049	.518	.000
164	.244	.00049	.518	.000
165	.244	.00024	.518	.000
166	.220	.00024	.515	.000
167	.244	.00049	.515	.000
168	.244	.00024	.515	.000
169	.220	.00049	.510	.000
170	.244	.00049	.510	.000
171	.244	.00049	.513	.000
172	.244	.00024	.508	.000
173	.244	.00024	.510	.000
174	.244	.00049	.508	.000
175	.244	.00049	.505	.000
176	.244	.00049	.508	.000
177	.244	.00024	.505	.000
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179	.244	.00049	.505	.000
180	.244	.00049	.503	.000
181	.220	.00024	.503	.000
182	.244	.00049	.503	.000
183	.269	.00049	.500	.000
184	.244	.00024	.500	.000
185	.269	.00049	.498	.000
186	.244	.00049	.498	.000
187	.244	.00024	.498	.000
188	.244	.00049	.498	.000

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194	.269	.00024	.496	.000
195	.269	.00049	.493	.000
196	.244	.00024	.491	.000
197	.220	.00024	.491	.000
198	.269	.00073	.491	.000
199	.244	.00024	.491	.000
200	.244	.00049	.491	.000
201	.269	.00049	.491	.000
202	.244	.00024	.488	.000
203	.269	.00073	.493	.000
204	.269	.00049	.488	.000
205	.269	.00049	.486	.000
206	.317	.00024	.486	.000
207	.317	.00024	.491	.000
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209	.244	.00049	.486	.000
210	.244	.00049	.486	.000
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212	.269	.00024	.486	.000
213	.293	.00073	.481	.000
214	.220	.00024	.481	.000
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225	.244	.00049	.474	.000
226	.269	.00024	.474	.000
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228	.244	.00049	.471	.000
229	.293	.00049	.469	.000
230	.269	.00024	.469	.000
231	.220	.00024	.469	.000
232	.244	.00049	.466	.000
233	.244	.00073	.469	.000
234	.220	.00049	.464	.000
235	.317	.00049	.464	.000
236	.269	.00024	.464	.000
237	.220	.00049	.464	.000

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261	.244	.00049	.447	.000
262	.244	.00049	.447	.000
263	.244	.00024	.447	.000
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265	.269	.00049	.447	.000
266	.439	.00049	.447	.000
267	.220	.00000	.447	.000
268	.269	.00049	.444	.000
269	.220	.00830	.439	.002
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271	.269	.00024	.444	.000
272	.269	.00049	.444	.000
273	.244	.00000	.442	.000
274	.269	.00049	.444	.000
275	.269	.00049	.442	.000
276	.244	.00049	.442	.000
277	.244	.00049	.444	.000
278	.244	.00049	.442	.000
279	.244	.00024	.439	.000
280	.244	.00024	.444	.000
281	.244	.00073	.439	.000
282	.244	.00049	.437	.000
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284	.244	.00024	.437	.000
285	.244	.00049	.437	.000
286	.269	.00024	.437	.000

287	.244	.00073	.435	.000
288	.244	.00024	.437	.000
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293	.244	.00049	.432	.000
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297	.220	.00073	.430	.000
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300	.244	.00024	.427	.000
301	.269	.00024	.427	.000
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321	.244	.00024	.417	.000
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323	.220	.00049	.413	.000
324	.269	.00049	.415	.000
325	.269	.00024	.415	.000
326	.244	.00024	.415	.000

VITA

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