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EXPERIMENTAL APPARATUS
FOR THE MEASUREMENT OF THE
ENTHALPY OF COAL DERIVED LIQUIDS

By

John R. McConnell

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A Thesis submitted to the Faculty and the Board of Trustees of the Colorado School of Mines in partial fulfillment of the requirements for the degree of Master of Science.

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ABSTRACT

An experimental apparatus capable of making accurate measurements of enthalpy differences was designed, constructed, and tested. The basic unit is a Freon 11, CFCl_3 , boil off calorimeter using three chambers; a vacuum chamber, a freon heat shield, and an inner measurement chamber.

Temperatures were measured with platinum resistance thermometers, calibrated against a standard Leeds and Northrup platinum thermometer. Resistances were measured with a Fluke multimeter.

Step by step operating procedures for all the equipment and instruments are presented for use by future operators. After further testing, future operators will use samples of coal derived liquids supplied by Bartlesville Energy Research Center, Bartlesville Oklahoma.

Enthalpy differences for water were measured for several points over the operating range of the equipment. Conditions were selected to best indicate the type of errors present in the measurements.

Steps were taken to decrease the error to within the allowed one Btu/lb_m limit. Although the accuracy was achieved on a few runs, modifications of the equipment and procedures will be required.

ACKNOWLEDGEMENTS

The author wishes to express his sincere thanks for support from the Energy Research and Development Administration, and the Colorado School of Mines Office of Research Services.

Grateful appreciation is extended to Professors Arthur J. Kidnay and Victor F. Yesavage, thesis co-advisors, who gave valuable guidance during this study. Sincere thanks is extended to the Master of Science Committee members: Professors A. J. Kidnay, V. F. Yesavage, E. D. Sloan, and A. L. Hines.

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INTRODUCTION

Coal derived liquids are a new and vital class of industrial compounds, but have thermodynamic properties that are largely unknown and, presently, unpredictable. This information of thermodynamic properties is needed to make more efficient design calculations of equipment using these new liquids. Enthalpy measurements offer a greater amount of information to the design engineer than other thermodynamic properties since they can be used directly in the determination of process heat loads.

The purpose of this project was to design and construct an experimental apparatus capable of making enthalpy measurements accurate to within one Btu/lb_m. The apparatus was tested by measuring the enthalpy of water over the pressure range of 500-1500 psi and a temperature range of 150-550° F and comparing the data obtained with that in the literature (1). Water was chosen for comparison purposes because it is readily available and reliable enthalpy data is available.

In the future this apparatus will undergo further testing with water before undergoing tests with n-pentane. Upon completion of the n-pentane tests, the apparatus will then be used on coal derived liquid samples supplied by the Energy Research and Development Administration,

Bartlesville Energy Research Center, Bartlesville,
Oklahoma.

This thesis presents a complete description of how the apparatus was constructed along with design information. Included are test run results describing operating characteristics, and accuracy of the measurements made. The thesis also contains complete calibration data for the platinum resistance thermometers, pressure gauge, and pressure transducers, and complete operating instructions for the apparatus and instruments.

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PROCESS SELECTION

A review of the calorimetric methods and interpretation of experimental results for the enthalpy of fluid mixtures under pressure has been presented recently (2). Flow calorimetry is best suited for measurement of enthalpy since, unlike batch calorimetry, enthalpy is obtained directly without the need for extensive volumetric data. The first law of thermodynamics applied to a flow calorimeter with negligible potential and kinetic energy effects is:

$$(H_{T_2, P_2} - H_{T_1, P_1})_x = \frac{\dot{Q} - \dot{W}}{F} \quad (1)$$

where H_{T_2, P_2} and H_{T_1, P_1} represent the enthalpies per unit mass of the fluid at outlet and inlet conditions respectively; \dot{Q} is the net rate of heat transfer to the fluid; \dot{W} is the net rate of work input; F is the mass flow rate; and x represents a constant overall composition. Flow calorimeters can be used to measure the variation of enthalpy with temperature and pressure depending upon the manner in which temperature and pressure are controlled.

The two major kinds of methods used to determine enthalpies of fluids by flow calorimetry are the direct measurement where a known quantity of energy is added to the fluid under study, and comparative measurement where

a knowledge of the specific heat or latent heat of a reference fluid is used to determine the energy input (2). The comparative method or more specifically a reference fluid boil off calorimeter similar to that originally developed by Nelson and Holcomb (3) was selected to be used in this study. In such a calorimeter energy is added to the fluid under study by heat transfer to a boiling fluid. The amount of energy added is determined from a knowledge of the latent heat of vaporization and the quantity of reference material boiled away.

The reasons for selecting a boil off calorimeter for this study are the following:

- 1) Boil off calorimeters are more suited for the measurement of enthalpy differences over large temperature ranges than direct measurement methods which are more suitable for determining heat capacities. For this apparatus large temperature differences are required since the processing of liquid fuels generally involves temperature changes of process streams of between room temperature to at least 700° F.

- 2) Boil off calorimeter systems are generally simpler, less costly, and less subject to equipment failures.

- 3) Although less accurate than direct calorimeters, boil off calorimeters can be developed with an accuracy of

about 1 Btu/lbm (3) which is within the accuracy of engineering process design calculations.

In all calorimetry programs the major concerns in obtaining accurate experimental data generally are the minimization of heat losses from the calorimeter and the accurate measurement of flow rate. The basic relation for a flow calorimeter, equation 1, when applied to a boil off calorimeter becomes:

$$(H_{T_2, P_2} - H_{T_1, P_1})_x = \frac{F_r \lambda_r}{F} + \frac{q}{F} \quad (2)$$

where F_r is the mass rate of reference fluid boiling off, λ_r is the latent heat of reference fluid, and q is the heat loss from the calorimeter-reference fluid system which in general must be estimated and represents a limitation in the accuracy of a measurement. The above relation is obtained since for boil off calorimeters:

$$\begin{aligned} \dot{Q} &= F_r \lambda_r + q & \text{and} \\ \dot{W} &= 0. \end{aligned} \quad (2a)$$

From equation 2, one can see that the heat loss error term is q/F and is thus inversely proportional to flow rate. Furthermore, as flow rate decreases, the accurate measurement of flow rate becomes more difficult. Thus, most calorimetry facilities, as with this one, have been developed to operate at flow rates in the order of

one gallon per hour to minimize heat loss effects and to obtain accurate flow rate measurements (3,4).

The enthalpy differences are obtained at constant total composition between a given temperature and the reference fluid boiling point temperature. Freon 11, CFCl_3 , is used as the reference fluid in this apparatus since it boils around room temperature (3). The calorimeter was constructed to operate at essentially constant pressure. Thus, the final results will consist of enthalpy differences at constant pressure levels between different temperatures and room temperature. To obtain the effect of pressure on enthalpy at any temperature it is necessary to tie in the data to results for enthalpy as a function of pressure for at least one isotherm. This can be done at room temperature for the liquid sample by integrating the relation:

$$\left(\frac{\partial H}{\partial P}\right)_T = V - T \left(\frac{\partial V}{\partial T}\right)_P \quad (3)$$

using measured values of the sample volume at room temperature. Since the effect of pressure on enthalpy is small for liquid hydrocarbons, generally of the order of 5 Btu/lb_m over a pressure range of 2000 psi (2), accurate values of volume are not required to determine the effect of pressure on enthalpy to within one Btu/lb_m.

In the two phase region the determined enthalpy values

represent the total enthalpy of a vapor-liquid mixture with a combined composition equal to that of the original sample. Such results are required to determine heat loads for heat transfer equipment; however, for phase separation equipment a knowledge of vapor-liquid equilibria is required.

EQUIPMENT DESIGN

Since the development of the original boil off calorimeter by Nelson and Holcomb (3), boil off calorimeters of numerous designs have been used under a variety of conditions, and their capabilities are well documented. Table 1 presents a listing of the boil off calorimeters that have been used, and the conditions under which they were operated. The design incorporated in this apparatus was developed after careful study of the ones listed in Table 1.

Calorimeter

The actual design work for the calorimeter was done by Dr. A. J. Kidnay and Dr. V. F. Yesavage in consultation with Cryogenic Engineering Incorporated, the fabricator. Figure 1 shows a scale drawing of the calorimeter design used in the apparatus. All calculations were based on a maximum change in enthalpy of 800 Btu/lb_m.

The calorimeter is constructed of 304 stainless steel except where it is in contact with the sample oil, where 316 stainless steel is used. The inner chamber constitutes the boiling bath. The 35 foot long coil of 1/8 inch outside diameter stainless steel tubing provides more than adequate heat transfer area to cool the sample

Table 1
Boil-Off Calorimeter Facilities

<u>Authors</u>	<u>Reference Fluid</u>	<u>System Fluid</u>	<u>Type of Data</u>
Nelson & Holcomb (3)	Freon 11	Polar Liquids	Constant P
Jenkins & Berwaldt (5)	Liq. Nitrogen	Fixed Gases	Constant P
Sahyal et al (6)	Liq. Nitrogen	Fixed Gases	Constant P
Wiener (7)	Freon 11	Gases & Liquids	Pressure Drop
Lenoir & Hipkin (4)	Freon 11	Liquids	Constant P
Laverman & Selcukoglee (8)	Liq. Nitrogen	Fixed Gases	Constant P
Dolan et al (9)	Liq. Nitrogen	Fixed Gases	Constant P & Pressure Drop
Banks & Haselden (10)	Liq. Nitrogen	Fixed Gases	Constant P
Eakin et al (11)	Freon 11	Gases & Liquids	Constant P & Pressure Drop
Thin et al (12)	Freon 11	Liquids	Constant P
Brewer (13)	Freon 11	Gases	Constant P

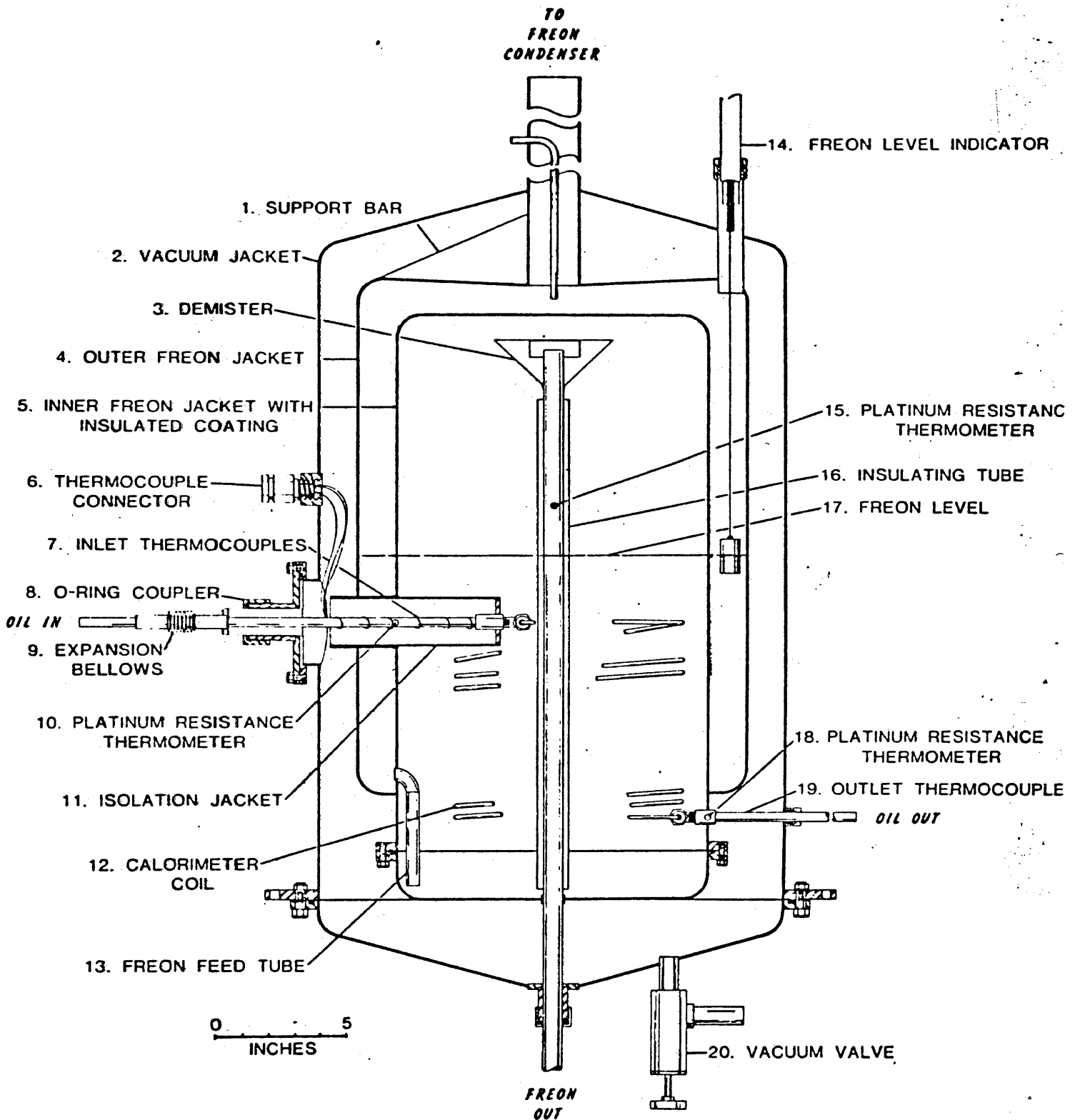


FIGURE 1 FLOW CALORIMETER

oil down to within 1° F of the freon bath.

The freon vapors that boil off from the inner chamber travel through the demister before leaving the calorimeter. The demister removes all forms of liquid from the exit stream allowing only the vapor to leave.

The middle chamber contains boiling freon. It acts as an insulating barrier by eliminating any temperature difference between the inner chamber and its surroundings. It also increases the capacity of the calorimeter for holding freon since the two chambers are connected by the freon feed tube.

The outer chamber is evacuated by the vacuum system to provide another insulating barrier. To be effective, the vacuum has to be less than 7×10^{-4} millimeters to increase the mean free path (14) until the distance is that equal to the widest distance of the chamber.

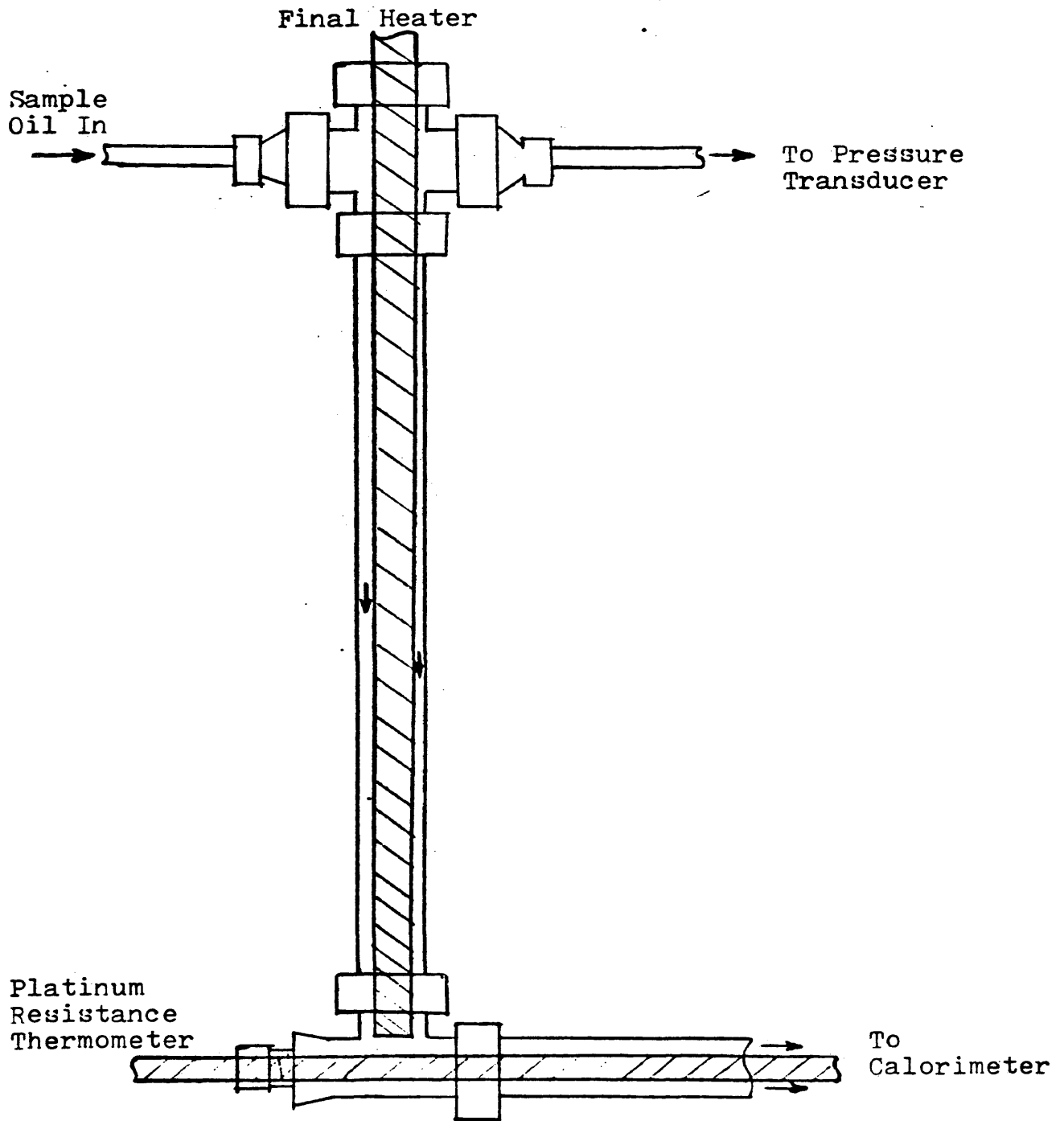
Heating System

To avoid hot spots in the sample oil lines, a gradual heating of the sample is desired. This is accomplished through the use of a fluidized bath containing a 25 foot coil to gradually heat the sample. An electric final heater is used to achieve the final temperature desired before entering the calorimeter.

The location of the final heater is shown in figure 2. The final heater is a 350 watt, two conductor element,

FIGURE 2

Inlet Tube Assembly



cartridge heater manufactured by Control Products Corporation. It is capable of heating the sample oil approximately 100° F. It is to be used to adjust the temperature for several points while the preheater bath temperature remains constant.

Pumping System

A Milton Roy dual diaphragm pump is used to deliver the sample oil through the system at the desired pressure. A Grove back-pressure regulator, dome loaded type, is located at the outlet of the calorimeter to set and maintain the pressure of the system.

To alleviate the problem of surges caused by the pump, a Greer bladder accumulator was installed in the system prior to the preheater bath. The bladder accumulator introduces a gas, nitrogen, into the system to absorb the energy of the pump strokes. The nitrogen, which is separated from the system by a barrier, releases the absorbed energy between strokes of the pump to stabilize the flow. The static pressure of the nitrogen in the bladder is set equal to the pressure of the system at the location at which it is connected.

Condensers

Based on a one cubic centimeter per second flow of

sample oil, the condensers were sized using heat transfer correlations for condensing freon (15). The temperature of the coolant for the condensers was selected to be 30° F for the calculations.

The main condenser has an area of 9.1 square feet while the small one has an area of 2.1 square feet. Both are manufactured by American Standard. The small one is mounted as a reflux condenser with the top vented to the atmosphere. The larger one is mounted as a counter flow condenser to allow for sub-cooling of the freon. All vents are connected to a dryer to prevent water from collecting in the freon system through the condensers.

Collection System

The collecting tubes for the sample oil and freon are 3 inches in diameter and 15 inches long. Samples collected in the tubes are kept below 2000 grams total to allow them to be weighed on the single pan balance, manufactured by Mettler. This eliminates the use of volumetric information and determines the mass directly.

Refrigeration System

The refrigeration unit was selected, based on the maximum heat load possible. It is a 13,000 Btu/hour water chiller manufactured by Dunham-Bush and greatly

exceeds the value of 6000 Btu/hour calculated to be needed. Flowmeters controlling the flow to the condensers are located to the left of the unit.

Vacuum System

To obtain the vacuum necessary, a diffusion pump is needed. A portable unit with gauge was assembled by Remanufactured Vacuum Products to fit the needs of the apparatus.

PROCESS FLOW

A schematic diagram of the experimental apparatus is presented in figure 3. It illustrates the positions of the different streams and the support equipment involved to operate the apparatus.

The layout of the equipment was dictated by the requirements made necessary by the samples to be handled. The initial layout was made using the calorimeter as a reference with an effort to minimize size and maximize ease of access.

The possibility of two-phase flow developing in the sample line after heating determined the layout of the piping. This consisted of a constant downward movement of the stream once the heating process began to prevent trapping of the liquid in a low spot and vapor in a high spot. Eliminating the availability of traps allows for a more uniform flow without separation of the phases.

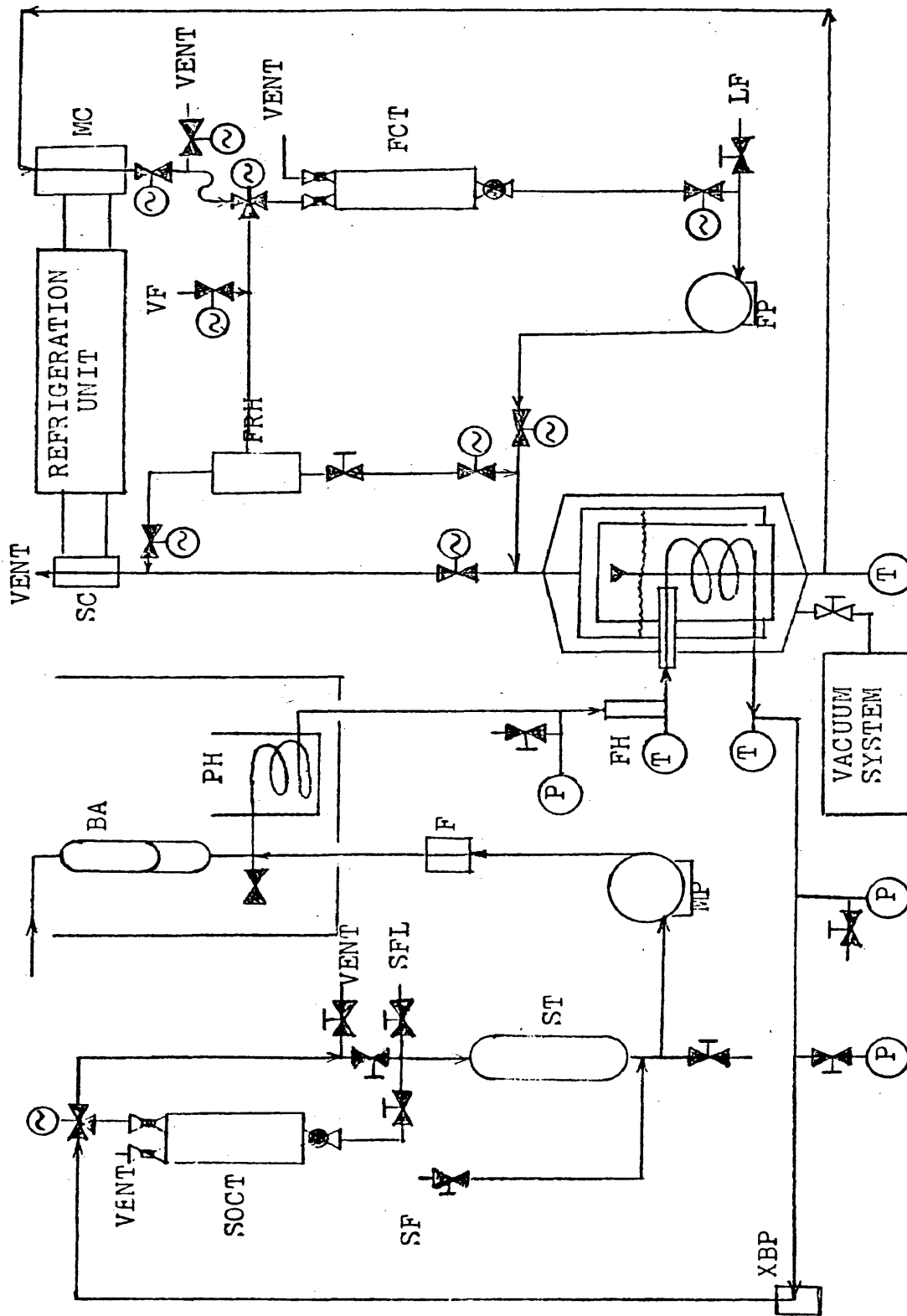
Another factor considered in the sample line layout was the expected small amount of some of the samples. This called for the shortest possible piping between equipment located in the sample stream.

Factors influencing the layout of Freon 11, CFCl_3 , lines included the gravity forced flow from the large condenser; minimizing length of lines; the low boiling point, approxi-

SYMBOL INDEX FOR FIGURE 3

BA	Bladder Accumulator
F	In-line Filter
FCT	Freon Collection Tube
FH	Final Heater
FP	Freon Pump
FRH	Freon Return Heater
LF	Liquid Fill
MC	Main Condenser
MP	Main Pump
P	Pressuring Indicating Device
PH	Preheater
SC	Small Condenser
SF	Sample Fill
SFL	Sample Flush
SOCT	Sample Oil Collection Tube
ST	Surge Tank
T	Temperature Indicating Device
VF	Vapor Fill
XBP	Back Pressure Regulator

Figure 2
 Experimental Apparatus for the Measurement of the
 Enthalpy of Coal Derived Liquids Schematic Diagram



mately 65° F, at this altitude; and the size allowed by room dimensions.

Gravity forced flow from the large condenser dictated its position above the collection tubes and at a height determined by the size of equipment required between the two. The compactness of the equipment was again necessary to maintain the length of lines as short as possible and also due to limited ceiling height in the laboratory. The low boiling point of the freon called for special measures to be taken in the handling to prevent excessive loss during filling and draining as well as during operation.

The actual equipment is mounted on a frame of slotted angle steel 8 feet wide, 4 feet deep, and 10 feet high, with a 2 1/2 foot extension on the front for the control panels. Aluminum panels were used to mount the controls and to construct the safety shielding around the preheater bath.

Sample System

The sample system is mainly made of 316 stainless steel to handle the corrosiveness of liquids derived from coal. The main lines are 1/16 inch inside diameter and 1/8 inch outside diameter and the connecting lines around the surge tank are 3/8 inch outside diameter. The surge tank is made of 304 stainless steel, has a capacity of 2.25 liters, and is mounted vertically. Manufactured by Hoke,

it is a high pressure sample cylinder with 1/2 inch NPT openings at both ends.

The fill port connects with the lower opening of the surge tank to allow air to escape out the vent, which is connected to the upper opening, without being trapped by the sample as it flows in. A drain valve is connected to the lower opening where the feed line to the main pump originates.

The main pump is a Milton Roy dual diaphragm metering pump, capable of capacities of up to two gallons per hour and of pressures up to 1800 psi. The dual diaphragm pump was selected for its ability to level out the pulsating in the flow with its double action.

An in-line filter was included after the pump to remove particles from the stream. If allowed to stay in the stream, small particles could collect in a stagnant corner created by change in tubing diameter, and dislodge and travel down stream to plug up the small diameter tubing. The filter elements are compressed stainless steel with 60 micron sized openings.

Before the preheater, a tee is located in the line to allow for the installation of a bladder accumulator to stabilize the pressure. The bladder accumulator, manufactured by Greer, incorporates a nitrogen filled bag to absorb the force of the pump stroke.

of the pump stroke. This acts as a cushion for the liquid sample system which is unable to react to the pump's stroke without pressure surges.

The preheater, manufactured by Techne, is a fluidized bath. Air is injected into the bath at the bottom to agitate the fused alumina sand to produce a safe heat transferring medium. A coil made of a 25 foot length of tubing was installed into the bath to provide an adequate heat transferring area.

The final equipment assembly before the sample stream enters into the calorimeter contains a water-cooled pressure transducer by Precise Sensors, the final heater by Control Products Corporation, and a platinum resistance thermometer, in that order. The pressure transducer is water-cooled to hold the temperature of the sensor within the limits of the transducer. The final heater is a rod-type, controlled by a Honeywell three mode controller.

After leaving the calorimeter, the sample line contains a platinum resistance thermometer to accurately measure temperature. This is followed by a pressure transducer and a Heise bourdon tube gauge to measure pressure. A back pressure regulator, made by Grove, is next in the line to maintain the systems pressure. The regulator is dome loaded with the pressure being set by

pressurized nitrogen. The sample is then sent back to the surge tank or to the collecting tube by a three way solenoid valve.

Freon System

The calorimeter's two inner chambers hold approximately twelve gallons of Freon 11, CFCl_3 . The measurement is made with the boil off freon from the inner chamber. The outer chamber of Freon 11 acts as insulation and a surge tank to increase the capacity to better maintain the level during long running periods.

The boil off from the inner chamber which is used for the actual measurement, passes through a mist trap to allow only the vapor to exit. The vapor then passes a platinum resistance thermometer which measures the temperature before entering a heated tube leading to the large condenser. The tube is heated to prevent condensing of the vapor before it reaches the condenser. Once the vapor condenses in the large condenser, it flows past a vent, through a vapor trap, to three-way valve. The three-way valve directs flow to either the recycle or the collection tube.

The recycle line goes to a return heater. The return heater is a heated glass flask that is vented to a location below the small condenser and drains through a

metering valve into the outer Freon 11, CFCl_3 , chamber of the calorimeter. The return heater is used to heat the subcooled liquid freon to the saturation point before allowing it to enter the calorimeter. This is done by allowing the freon to boil before opening the metering valve.

The outer chamber of Freon 11 is used as insulation and a surge tank. Its boil off is caused by heat transferred from the outside of the calorimeter. The vapor then travels to the small condenser, which is vented to the atmosphere, to condense and fall back to the outer layer at the saturation temperature.

INSTRUMENTATION AND MEASUREMENTTemperature

Enthalpy measurement is highly sensitive with respect to temperature. Small errors in the temperature measurement cannot be overlooked. Temperature measurements that are not directly involved in the enthalpy calculations are measured with iron-constant thermocouples connected to an Omega digital readout. The temperatures at the locations of the thermocouples are excellent indicators as to how the equipment is operating.

At locations requiring more accurate measurement, 100 ohm platinum resistance thermometers are used. They are sheathed in 316 stainless steel and are located in the sample inlet and outlet of the calorimeter and in the Freon 11 outlet of the calorimeter.

A digital volt/ohm meter, manufactured by John Fluke Company, provides accurate measurement of the resistance thermometers output. The meter incorporates a four lead connection to duplicate the type of readings available from a mueller bridge, but with greater speed.

To check for temperature gradients in the sample stream at the calorimeter inlet, several thermocouples are located on the sample inlet tube. They are spaced one inch apart on the outside of the tube. To magnify

even the slightest gradient, the digital volt/ohm meter is used along with a cold junction compensator.

Pressure

Sample system pressure is measured at both the inlet and outlet of the calorimeter to obtain operating pressure as well as the pressure drop across the calorimeter coil. The pressure drop is measured with two pressure transducers, one at the inlet and one at the outlet to the calorimeter. A Hewlett-Packard D.C. power supply is used to input the 10 volt signal necessary for the transducers. The Fluke meter is used to measure the output signal which is then converted to pressure.

The bourdon tube gauge is located at the outlet and measures the pressure directly. It is also used as a standard when calibrating the pressure transducers.

CONTROLS

The most used controls are located on the front panels with lesser used controls located throughout the equipment. The right front panel, Figure 4, is the main control panel. Several remote valves are electrically operated and are controlled from this panel as well as most of the measurement readouts. Also located on this panel are the power switches for most of the equipment, final heater controller, run switch, timer, Fluke meter, temperature indicator, and D.C. power supply. Switches to change the input to the Fluke meter and temperature are located on the lower portion of the right panel.

The middle panel houses the collection tubes. Drains for both tubes are located on their respective sides. The return flow metering valve for the freon return heater is mounted on the upper left corner of the middle panel. Coolant control valve on the upstream side of the collection tube for the freon, is located to the right of the collection tube.

The left panel, figure 5, contains the Heise bourdon tube gauge, cooling water and air flow meters, preheater controller unit, and variacs. The air flow meter controls the flow to the preheater bath to fluidize it. The water flow meter adjusts the flow of cooling water to the inlet

SYMBOL INDEX FOR FIGURE 4

1. SCR indicating light
2. Vacuum system power switch
3. Main pump power switch
4. Main electric control panel (Switches)
 - a) Blank
 - b) Blank
 - c) Blank
 - d) Blank
 - e) Solenoid Group A
 - f) Solenoid Group B
 - g) Solenoid Group C
 - h) Solenoid Group D
 - i) Freon pump
 - j) Spare
 - k) Spare
 - l) Blank
 - m) Fluke multimeter
 - n) Temperature indicator
 - o) D.C. power supply
 - p) Final heater temperature controller
5. Final heater temperature controller
6. Run Switch
7. Timer
8. Fluke multimeter
9. Temperature Indicator
10. D.C. power supply
11. Pressure transducer input voltage control switch
12. Fluke multimeter input switch
13. Thermocouple switch (Fluke multimeter)
14. Thermocouple switch (Temperature indicator)

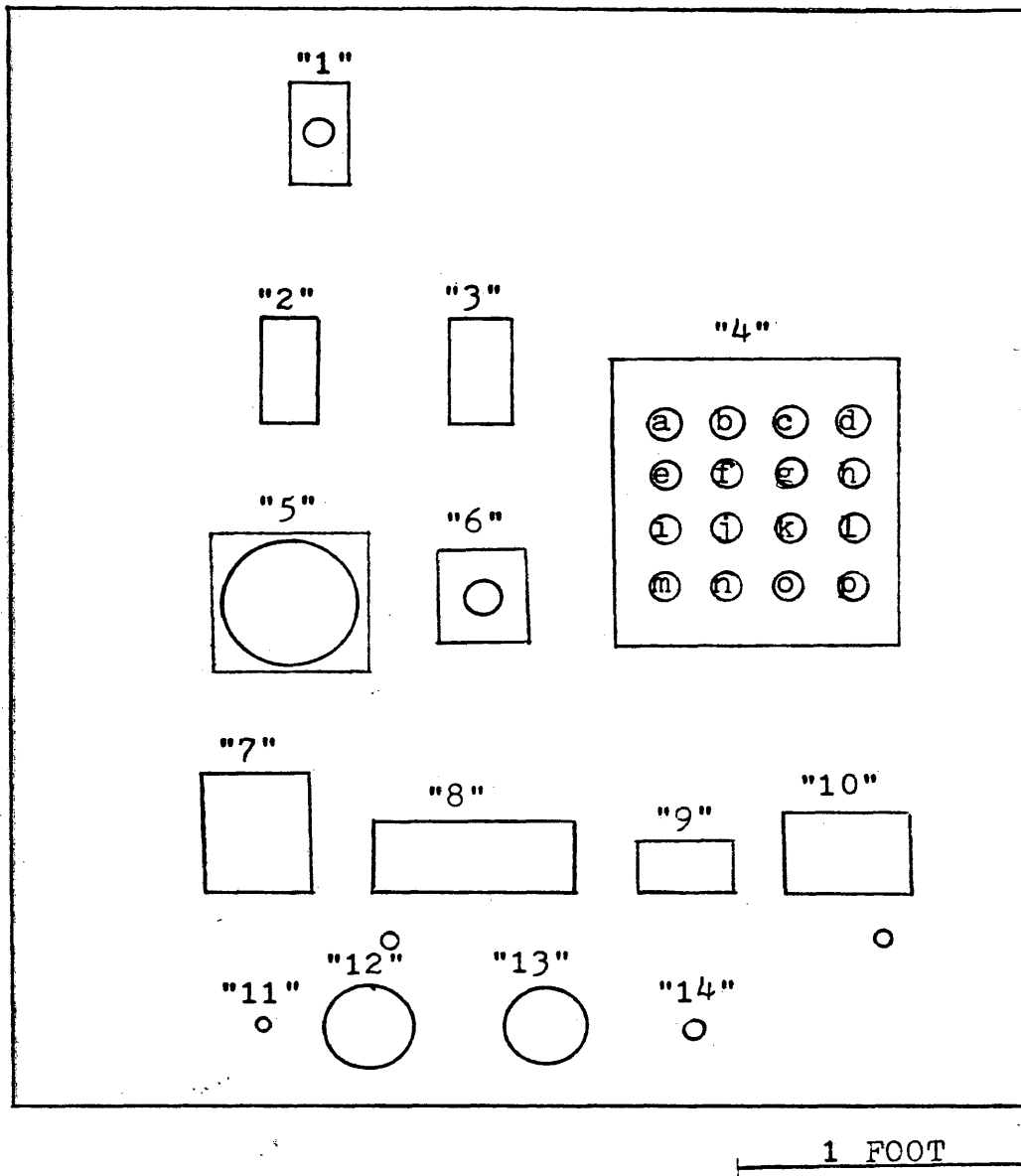


Figure 4
RIGHT MAIN CONTROL PANEL

SYMBOL INDEX FOR FIGURE 5

1. Heise bourdon tube pressure gauge
2. Air flow meter for preheater
3. Preheater temperature controller
4. Heise gauge disconnect valve.
5. Water coolant flow meter for inlet pressure transducer
6. Variac for freon return heater
7. Variac for freon outlet line heater

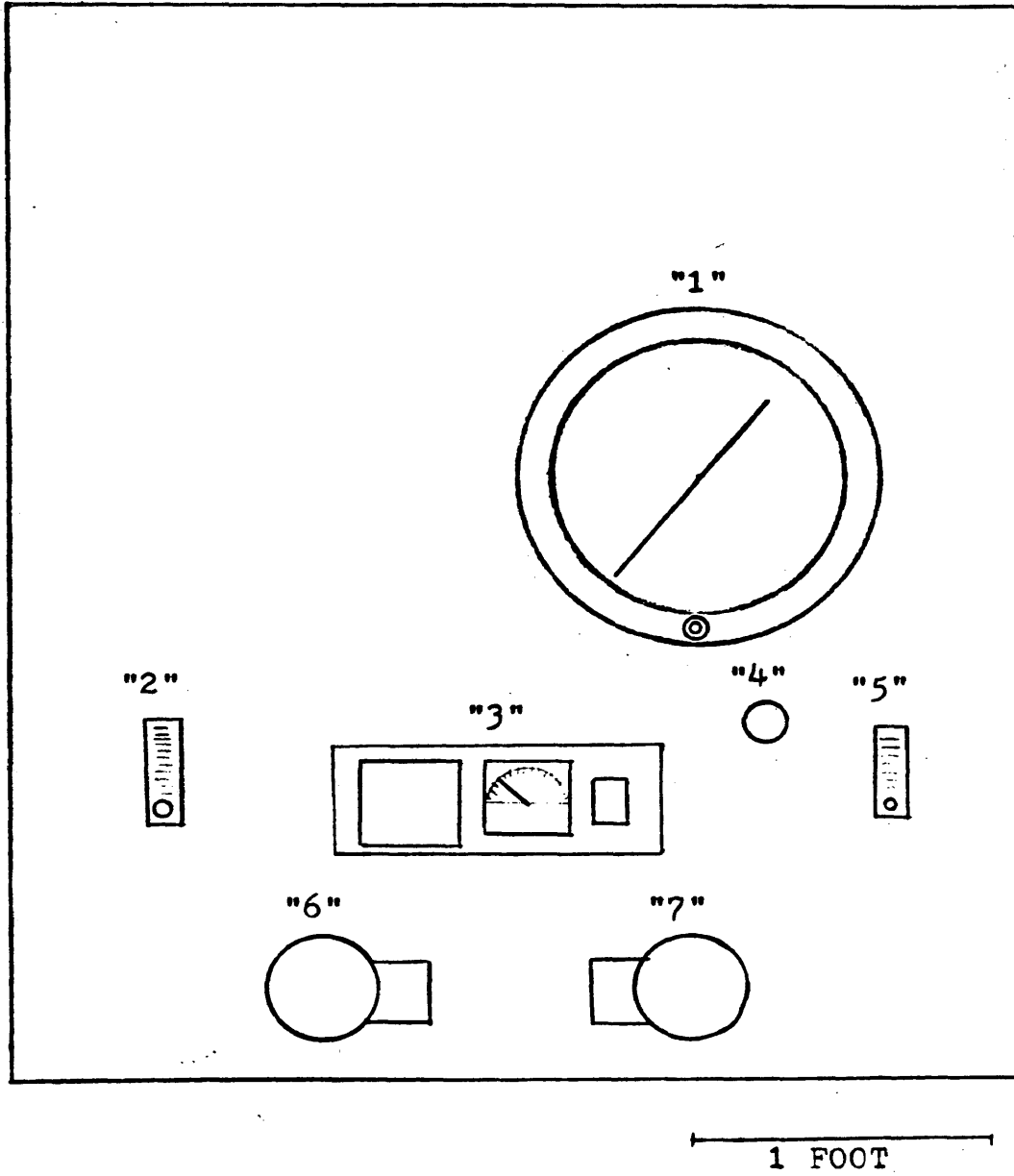


Figure 5

LEFT MAIN CONTROL PANEL

pressure transducer. One variac controls the freon return heater and the other controls the freon line to the large condenser. The valve below the Heise gauge separates the bourdon tube from the system to protect it when the system fluctuates. It should be closed during start up and shut down of the pump as well as when the system's pressure surges, to prevent damage to the bourdon tube.

The nitrogen regulators located on the cylinders to the right of the control panels are used to control the system's pressure. The left cylinder supplies the bladder accumulator which stabilizes pressure. The right regulator supplies the back pressure regulator and is used to set the operating pressure.

Between the refrigeration unit and the nitrogen cylinder rack is a small panel located on the main frame. This panel contains control valves for nitrogen flow and for coolant flow for the freon collection tube. The large valve is located downstream from the collection tube in the coolant line. It is used in conjunction with the upstream valve to isolate the tygon tubing used. The small valve on the left is located between the nitrogen regulator and the bladder accumulator to close off the system from regulator fluctuations. The three remaining valves are connected to the nitrogen regulator for the

back-pressure regulator. The left valve of the three, connects the cylinder to the back pressure regulator. The top one connects the cylinder to the sample surge tank for flushing the system. The right valve is a vent to release pressure of either the flush line or back-pressure regulator line.

Other controls are located throughtout the apparatus on the equipment they control. The equipment which have controls on them are the main pump, preheater bath, vacuum system and refrigeration system. These controls, along with valves not mentioned in the discussion of the controls, are mentioned in the operation of the particular item.

EXPERIMENTAL PROCEDURE

The following section describes the experimental procedure used in operating the equipment, given for a totally shut down apparatus. The purpose of this section is to describe what was done and to give a step by step procedure to be used by future operators.

Preparation

- 1) Fill the calorimeter with Freon-11 until the float level is approximately level with the slotted angle steel through which the float travels.
- 2) Connect two nitrogen gas cylinders to the regulators to the right of the control panel. Cylinders should have a pressure of at least 100 psi greater than the system pressure to be maintained during operation.
- 3) Fill the refrigeration unit with a 50-50 mixture by volume, of distilled water and ethylene glycol. The fill is located on the front panel above the level sight glass, which has a vent at the top to be opened only during filling. Liquid will flow out of the vent when the holding tank is full.
- 4) Fill the preheater with fused alumina sand to

within three inches from the top.

- 5) Fill surge tank with approximately 1.5 to 2.0 liters of sample to be tested.

Daily Start-up

- 1) Turn on refrigeration unit to cool the condensers.
- 2) Turn on vacuum system to evacuate the outer chamber of the calorimeter.
- 3) Turn on air flow to preheater bath and adjust the flow to 120 cubic feet per hour. Turn on preheater controller and adjust to desired temperature.
- 4) Turn on instruments, including the temperature indicator, DC power supply, and Fluke multimeter.
- 5) Activate solenoid group A when refrigeration outlet temperature drops below 30° F to open the freon system.
- 6) Adjust pressure transducer water coolant flow rate to 3.5 gallons per hour. Open valve to allow Heise gauge to read pressure. At zero gauge pressure, measure the output of the inlet pressure transducer with a 10 volt input. Record this value for later calculations.
- 7) Close the valve to the Heise gauge. Turn on the main pump and set its stroke metering to obtain

a one cubic centimeter per second flow rate. Set the back pressure regulator at approximately 100 psi greater than the desired system pressure to pressurize the system.

- 8) After allowing the pump to run for 20 to 30 seconds, pressurize the bladder accumulator to a pressure approximately 20 to 40 psi greater than the system pressure. Gradually open the valve to the Heise gauge. If fluctuations in pressure occur, adjust the pressure of the bladder accumulator to minimize pump surges.
- 9) Turn on the variac that controls the heat to the freon outlet line. Set dial reading to 30.
- 10) Close off metering valve for freon return heater until the liquid level is visible from a position in front of the control panel. Turn on the variac that controls the heat to the return heater and adjust metering valve to keep the level constant.
- 11) Adjust the final heater controller to the desired temperature and turn on.

Collecting Data

- 1) Allow time to reach steady state and record temperatures and pressures to monitor the system's conditions.

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- 3) The vacuum in the jacket should be below 0.7 microns to be effective. Check to make sure it has had time to reach this condition.
- 4) Once the temperature and pressure have stabilized, the equipment is ready to operate at the conditions set.
- 5) To collect the fluids (the sample flowing through the calorimeter and the freon that has boiled off), push the run button on the right panel. This activates the timer and three-way solenoid valves. (A manual valve was used in the freon line. A solenoid valve is being sought to replace the manual valve at a future date.) Once the system is collecting, temperatures and pressures are recorded to make required calculations and to check stability during the run. The level of the float must remain constant during the run to maintain a steady state.
- 6) To end the run, the run button is pushed again to de-energize the three-way solenoids and stop the timer. The collection tubes are weighed to determine the amounts collected.
- 7) The tube located on the left side of the center front panel is then connected to drain the sample

into the surge tank.

- 8) The tube located on the right side of the center front panel is then connected to drain the freon back to the calorimeter. Solenoid groups C and D and the freon pump are turned on to drain the collection tube.
- 9) After draining, replace the collection tubes with clean ones that have been weighed. Then repeat the steps of collecting data for the conditions of the next run.

Daily Shut-down

- 1) Turn off freon return heater variac and open the freon return metering valve completely.
- 2) Turn off the final heater controller and preheater controller, leaving the air supply to the preheater on.
- 3) Turn off the vacuum system, allowing time for the diffusion pump to cool.
- 4) Allow time for the preheater to cool to below 180° F before turning off its air supply.
- 5) After cooling off the preheater, turn off the main pump and remove the pressure from the back-pressure regulator and then from the bladder accumulator.

- 6) Turn off the freon outlet variac, Fluke meter, temperature indicator, and D.C. power supply.
- 7) When flow stops through the return heater, turn off solenoid group A. At this time the refrigeration unit can be shut off. If a run is to be made the next day, it is advisable to leave it on.

Freon Filling and Draining

- 1) Connect the Freon 11 barrel to filling lines located directly behind the calorimeter. The refrigeration unit must be on and cooled to operation temperature before filling or draining.
- 2) Open the valves on the barrel and on the apparatus.
- 3) Turn on the solenoid group D and the freon pump to fill the calorimeter. Solenoid group A must be open to allow the two freon chambers to be at the same pressure. The calorimeter is full when the float is approximately six inches high in its guide tube.
- 4) Once the calorimeter is filled, the freon pump and solenoid group D are turned off, and the valves are closed that connect the barrel to the apparatus.

- 5) Solenoid group B controls the vapor fill. This is to be open to transfer vapor from the barrel to the calorimeter through the condenser. This is used to decrease pressure in the barrel and to empty it as far as possible.
- 6) To empty the calorimeter, the barrel must be connected and valves open to permit evacuation.
- 7) The apparatus needs to be set up to run hot fluid through the calorimeter coil and set in the run position.
- 8) Open solenoid group C and stop cock at the bottom of the collection tube to allow flow through to the barrel. Pressure in the barrel is released by turning on solenoid group B, allowing vapor flow into the calorimeter.
- 9) This is continued until the outlet sample stream from the calorimeter starts increasing in temperature to a value of 80° F. The main pump and the heat controllers are then turned off.
- 10) When the flow of freon from the calorimeter stops, solenoid group C is closed along with the connecting valves to the barrel. The calorimeter is then dismantled to remove the remaining freon.

TEST RUN RESULTS

A total of twenty test runs were made with distilled water to determine the accuracy of the equipment. The equipment was modified when the data indicated an equipment or design failure. Values of enthalpy differences measured were compared to values published (1). Table 2 list the results of the last ten runs. They were made after the last corrective measure was made during the twenty test runs.

Corrective Measures

Test runs one and two were invalid due to a faulty valve. The three-way valve for the Freon 11 system was not capable of handling the freon and deteriorated to the point of not changing the flow completely.

Test runs three through seven indicated a restriction of the flow to the freon collection tube. The quick-disconnect that was being used in that line was replaced by tubing of a larger inside diameter.

During test run ten a leak developed inside the calorimeter and made necessary its dismantling. Data collected on runs eight through ten were disallowed due to the corrections made on the system after run ten.

TABLE 2

Run	Flow Rate cc/sec	Pressure psia	Temp. °F	Enthalpy (Determined) Btu/lb	Enthalpy (Literature) Btu/lb
11	1.16	564	256.2	182.6	190.7
12	1.24	565	254.2	182.7	188.9
13		Leakage Through Collection Tube			
14	.73	1418	174.5	102.8	107.5
15	.72	1430	175.5	105.1	109.2
16	.70	1526	547.6	485.2	507.3
17	.67	1517	515.0	447.7	461.3
18	.43	546	275.1	202.9	210.2
19	.95	565	277.3	209.1	212.5
20	1.93	600	274.5	208.3	209.3

Indications

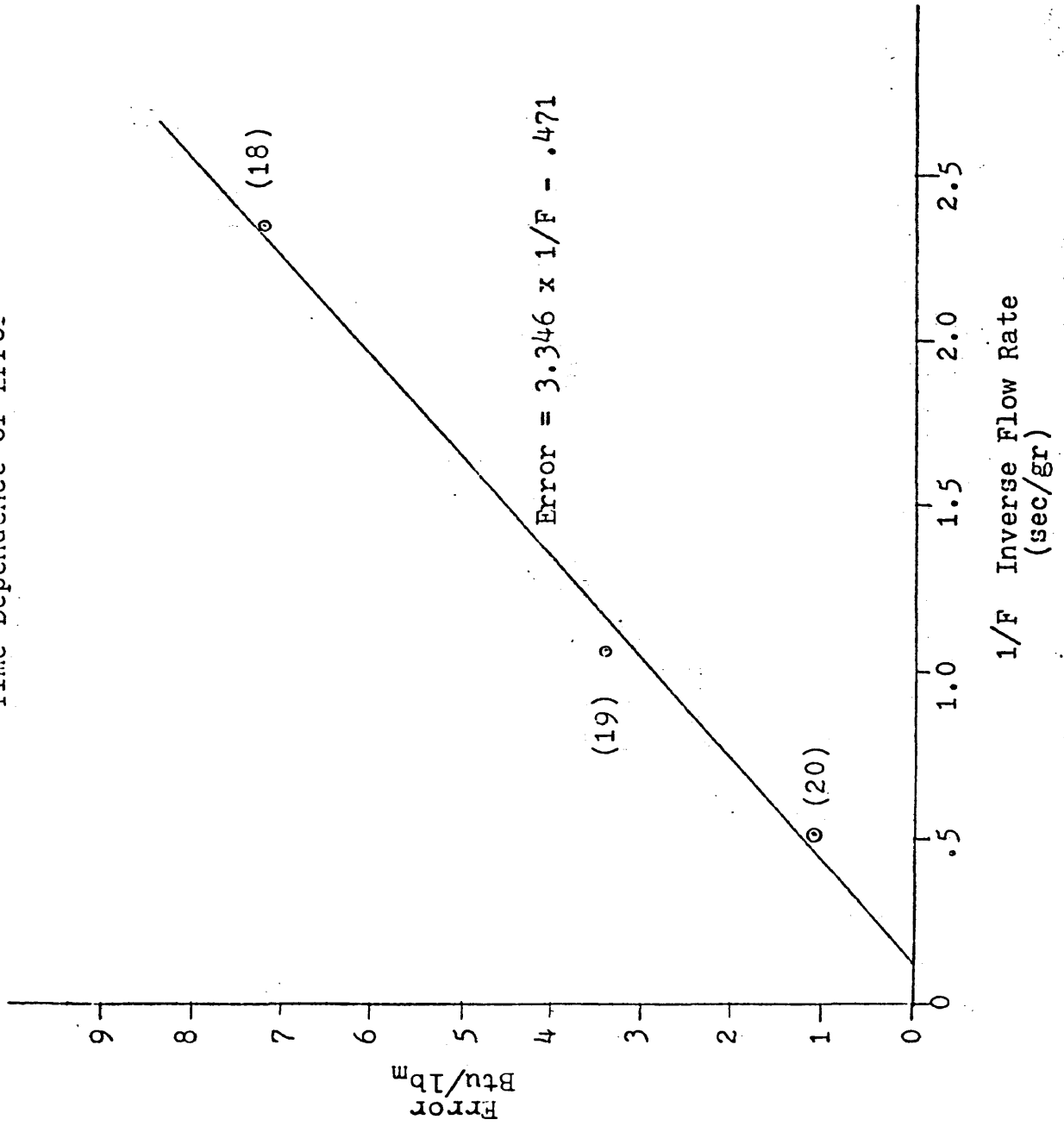
The results of the following runs were then used to determine the type of errors still remaining in the equipment. Graphs one and two illustrate the trends in the measurements taken.

Graph one indicates a heat leak that is dependent on time. The error values are shown to be inversely proportional to the flow rate. As the length of the run is increased, the error in measurement is increased by the constant heat flux from the boil off chamber of freon in the calorimeter to its surroundings. The middle chamber of freon was found to be at a lower temperature than the inner chamber. This would cause the type of error realized. To compensate for this, the freon from the small condenser needs to be heated to its saturation temperature before entering the outer chamber.

Graph two indicates a conduction heat leak, since the error is proportional to the inlet temperature of the sample. This would point to a path for conduction from the inlet tube assembly to the outer chamber of freon. the inlet thermocouples were removed to eliminate any possible path for conduction.

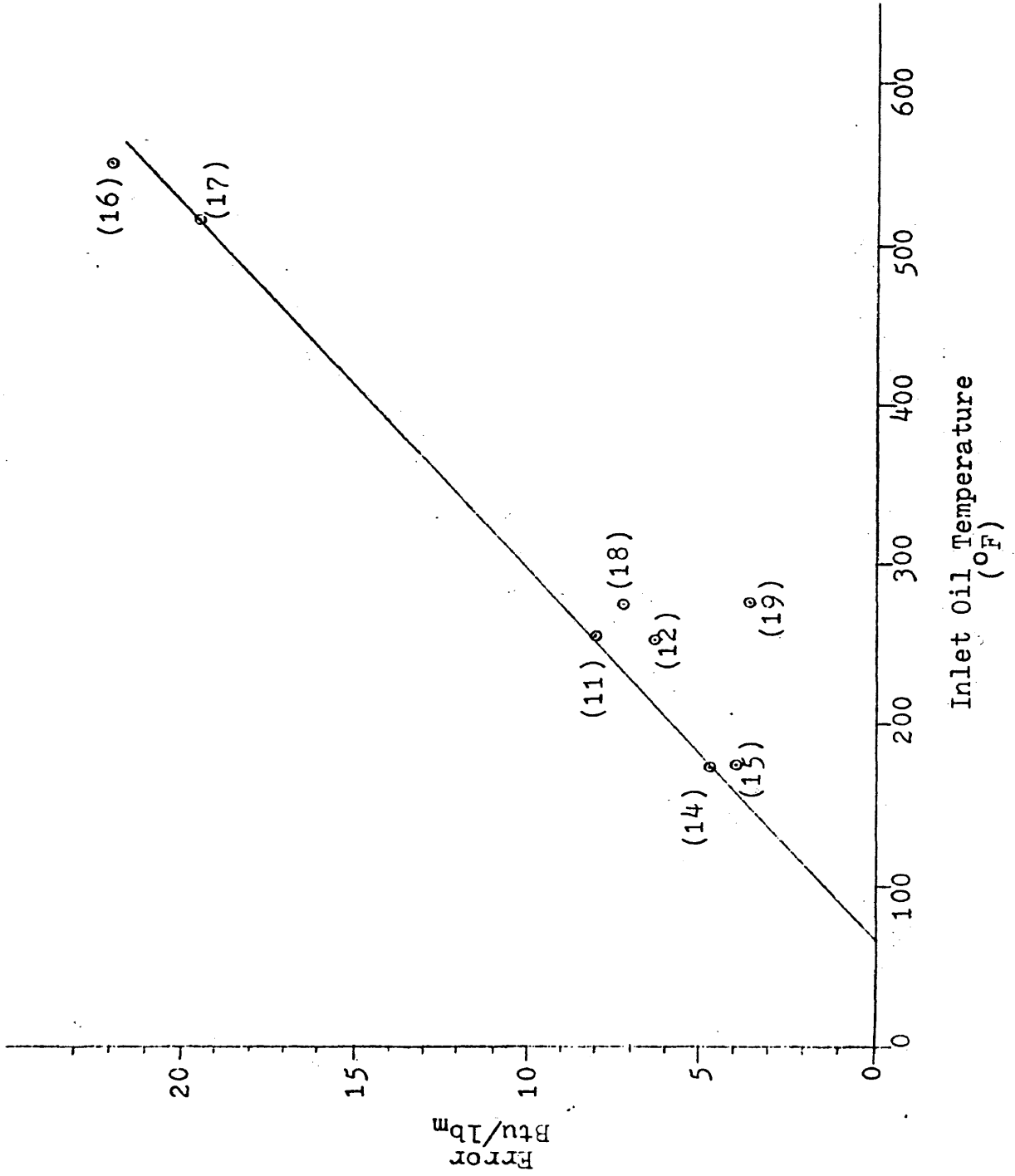
GRAPH 1

Time Dependence of Error



GRAPH 2

Temperature Dependence of Error



CONCLUSIONS

Although several faults in the equipment were corrected during the first twenty test runs, it is necessary to make more test runs to see if any more errors exist. The runs should be made using distilled water and then n-pentane with data points covering the full range of the calorimeter capabilities

RECOMMENDATIONS

1. Replace inlet thermocouples with a better set-up to eliminate any form of heat loss.
2. Reroute the return flow from the small condenser through an apparatus similiar to the return heater before allowing it to enter the calorimeter.
3. Replace the drain lines for the freon collection tube with 3/8 inch copper tubing that is insulated.
4. Modify the freon collection lines to establish 1/4 inch as a minimum inside diameter.

APPENDIX A

Platinum Resistance Thermometer Calibration

The Report of Calibration from Leeds and Northrup Company for the calibrated thermometer is presented in this appendix. The report is followed by information on the calibration of the 100-ohm platinum resistance thermometers used in the system, including calibration procedures, results, and the method to determine the results from the raw data.



LEEDS & NORTHRUP COMPANY Sumneytown Pike • North Wales, Pennsylvania 19454

REPORT OF CALIBRATION

FOR

8167-25-B PLATINUM RESISTANCE THERMOMETER

SERIAL NO. 1835855

-oOo-

The above designated platinum resistance thermometer was calibrated in October, 1975 for use with continuous current of 1.0 milliamp through the thermometer.

The following values were found for constants in the formulas given in the International Practical Temperature Scale of 1968 (IPTS-68):

$$\text{Alpha } (\alpha) = 0.003926573$$

$$A_4 = 6.063 \times 10^{-7}$$

$$\text{Delta } (\delta) = 1.496340$$

$$C_4 = 2.122 \times 10^{-14}$$

The pertinent International Practical Temperature Scale formulas are given in the discussion on the following pages.

The resistance at 0° C was found to be 25.5104 ohms.

The values given were determined from measurements at the triple point of water, tin point, zinc point and the boiling point of oxygen.

All observations were made by comparison with platinum resistance thermometer reference standards calibrated at regular intervals by the National Bureau of Standards. The most recent reference standard calibration is dated October, 1974; N. B. S. Test No. 211432.

For the Vice President-Instruments & Products Group

R. H. Verity
R. H. Verity
Manager, Standards Laboratory

October 21, 1975

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TEMPERATURE RANGE: 0° TO 630.74° C

Temperatures between 0° C and 630.74° C on the new International Practical Temperature Scale of 1968 (IPTS-68) are defined by the indications (resistance values) of standard platinum resistance thermometers and the following expressions:

$$t = t' + M(t') \quad (1)$$

$$t' = \frac{1}{\alpha} \left(\frac{R_t}{R_0} - 1 \right) + \delta \left(\frac{t'}{100} - 1 \right) \frac{t'}{100} \quad (2)$$

$$M(t') = .045 \left(\frac{t'}{100} \right) \left(\frac{t'}{100} - 1 \right) \left(\frac{t'}{419.58} - 1 \right) \left(\frac{t'}{630.74} - 1 \right) \quad (3)$$

where t is the temperature, at the outside of the tube protecting the platinum resistor, in ° C on the International Practical Temperature Scale of 1968, and R_t and R_0 are the resistances of the platinum resistor at t° and 0° C respectively, measured with a continuous current through the platinum resistor. The value of this current and the values of the constants α and δ found for this thermometer are given on the previous page. The value of $M(t')$, given by expression (3), is the same for all thermometers and is a function only of the quantity t' . The addition of the small value represented by (3) serves to make the IPTS-68 conform more closely to the thermodynamic scale than can be done with only the simple quadratic of expression (2).

An alternate form which is completely equivalent to expression (2) is

$$R_t = R_0 (1 + At' + Bt'^2) \quad (4)$$

In some instances expression (4) is less difficult to calculate than (2). The constants A and B used in (4) are related directly to α and δ .

$$A = \alpha (1 + \delta/100) \quad (5)$$

$$B = -\alpha\delta/10^4 \quad (6)$$

CAUTION: THE VALUES OF A , B , AND δ ON THE NEW 1968 SCALE ARE DISTINCTLY DIFFERENT FROM THE CORRESPONDING VALUES ON THE OLD 1948 OR 1927 SCALE. THE VALUES OF α AND R_0 ARE ALSO DIFFERENT BUT ONLY TRIVIAALLY SO.

TEMPERATURE RANGE: -182.962° C TO 0° C (90.188K TO 273.15K)

Temperatures below 0° C on the new 1968 scale are calculated using a standard reference table which gives values of R_t/R_0 for a fictitious "mean" standard thermometer. This reference table and a specified deviation equation are combined to give the values for a particular thermometer. The standard reference table used for IPTS-68 is referred to as the "CCT-68" table. It is convenient to use the symbol W_t in place of R_t/R_0 . For the special reference values of R_t/R_0 tabulated in CCT-68 the designation $W_t(\text{CCT-68})$ is used. The table giving values of W_t for a particular thermometer from 0° C down to -182.962° C (90.188K) may be calculated from the following expressions,

$$W_t = W_t(\text{CCT-68}) + \Delta W_t \quad (7)$$

$$\Delta W_t = A_4 t + C_4 t^3 (t - 100) \quad (8)$$

Expression (8) is the specified deviation equation in the range 0° C to -182.962° C. The constants A_4 and C_4 to be used in expression (8) for this particular thermometer are given on the first page.

CALIBRATION TABLE

A table calculated from the constants for this thermometer is on the following pages. The first column of the table gives values of temperature. Unless a different function is requested, the second column gives R_t/R_0 (i.e. the ratio of the resistance at the stated temperature to the resistance at the ice point). The third column gives the inverse (reciprocal) of the difference between successive values in the second column. These reciprocal first differences are included to facilitate interpolation. The error introduced by using the linear interpolation will be less than 0.0001° C.

The resistance at the ice point should be determined with the bridge which is used with the thermometer.

This table was computed and printed by machine. Although it has been made with care, a complete check of every value actually printed has not been made.

TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
			-150.0	0.385453	235.20
			-149.0	0.389702	235.37
			-148.0	0.393947	235.54
			-147.0	0.398190	235.71
			-146.0	0.402429	235.88
			-145.0	0.406666	236.04
			-144.0	0.410899	236.21
			-143.0	0.415130	236.37
			-142.0	0.419358	236.53
			-141.0	0.423582	236.69
			-140.0	0.427804	236.85
			-139.0	0.432024	237.01
			-138.0	0.436240	237.17
			-137.0	0.440454	237.32
			-136.0	0.444665	237.48
			-135.0	0.448873	237.63
			-134.0	0.453079	237.78
			-133.0	0.457281	237.93
-183.0	0.243560		-132.0	0.461482	238.08
-182.0	0.247399	230.45	-131.0	0.465680	238.22
-181.0	0.252238	230.51	-130.0	0.469875	238.37
-180.0	0.256574	230.59	-129.0	0.474067	238.51
-179.0	0.260909	230.68	-128.0	0.478258	238.65
-178.0	0.265242	230.78	-127.0	0.482445	238.79
-177.0	0.269573	230.89	-126.0	0.486631	238.93
-176.0	0.273903	231.00	-125.0	0.490813	239.07
-175.0	0.278229	231.12	-124.0	0.494994	239.21
-174.0	0.282553	231.25	-123.0	0.499172	239.34
-173.0	0.286875	231.39	-122.0	0.503348	239.47
-172.0	0.291194	231.53	-121.0	0.507521	239.61
-171.0	0.295511	231.68	-120.0	0.511692	239.74
-170.0	0.299824	231.82	-119.0	0.515861	239.87
-169.0	0.304135	231.98	-118.0	0.520028	239.99
-168.0	0.308343	232.14	-117.0	0.524193	240.13
-167.0	0.312748	232.30	-116.0	0.528355	240.25
-166.0	0.317050	232.46	-115.0	0.532515	240.38
-165.0	0.321348	232.63	-114.0	0.536673	240.50
-164.0	0.325644	232.79	-113.0	0.540829	240.62
-163.0	0.329937	232.96	-112.0	0.544983	240.74
-162.0	0.334226	233.13	-111.0	0.549135	240.86
-161.0	0.338512	233.30	-110.0	0.553284	240.98
-160.0	0.342795	233.47	-109.0	0.557432	241.10
-159.0	0.347075	233.65	-108.0	0.561576	241.21
-158.0	0.351352	233.82	-107.0	0.565721	241.33
-157.0	0.355626	233.99	-106.0	0.569863	241.45
-156.0	0.359896	234.17	-105.0	0.574003	241.56
-155.0	0.364164	234.34	-104.0	0.578141	241.67
-154.0	0.368428	234.51	-103.0	0.582277	241.78
-153.0	0.372689	234.69	-102.0	0.586411	241.90
-152.0	0.376946	234.86	-101.0	0.590543	242.01
-151.0	0.381201	235.03			

TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
-100.0	0.554673	242.11	-50.0	0.799083	246.82
-99.0	0.598801	242.22	-49.0	0.803133	246.90
-98.0	0.602928	242.33	-48.0	0.807182	246.99
-97.0	0.607053	242.44	-47.0	0.811229	247.07
-96.0	0.611176	242.54	-46.0	0.815275	247.15
-95.0	0.615297	242.65	-45.0	0.819320	247.23
-94.0	0.619415	242.75	-44.0	0.823364	247.32
-93.0	0.623534	242.86	-43.0	0.827406	247.40
-92.0	0.627650	242.96	-42.0	0.831446	247.48
-91.0	0.631764	243.06	-41.0	0.835486	247.56
-90.0	0.635877	243.17	-40.0	0.839524	247.65
-89.0	0.639987	243.26	-39.0	0.843560	247.73
-88.0	0.644096	243.36	-38.0	0.847596	247.80
-87.0	0.648204	243.46	-37.0	0.851630	247.89
-86.0	0.652310	243.56	-36.0	0.855663	247.96
-85.0	0.656414	243.66	-35.0	0.859694	248.05
-84.0	0.660516	243.76	-34.0	0.863724	248.13
-83.0	0.664617	243.86	-33.0	0.867753	248.21
-82.0	0.668716	243.95	-32.0	0.871781	248.29
-81.0	0.672813	244.05	-31.0	0.875807	248.37
-80.0	0.676909	244.15	-30.0	0.879832	248.45
-79.0	0.681004	244.24	-29.0	0.883856	248.53
-78.0	0.685096	244.34	-28.0	0.887878	248.60
-77.0	0.689188	244.43	-27.0	0.891899	248.68
-76.0	0.693277	244.52	-26.0	0.895919	248.76
-75.0	0.697365	244.62	-25.0	0.899938	248.84
-74.0	0.701452	244.71	-24.0	0.903955	248.92
-73.0	0.705537	244.80	-23.0	0.907971	249.00
-72.0	0.709620	244.89	-22.0	0.911986	249.07
-71.0	0.713702	244.98	-21.0	0.916000	249.15
-70.0	0.717782	245.08	-20.0	0.920012	249.23
-69.0	0.721861	245.16	-19.0	0.924023	249.30
-68.0	0.725939	245.26	-18.0	0.928033	249.38
-67.0	0.730015	245.34	-17.0	0.932042	249.49
-66.0	0.734089	245.44	-16.0	0.936049	249.54
-65.0	0.738162	245.52	-15.0	0.940055	249.62
-64.0	0.742233	245.61	-14.0	0.944060	249.69
-63.0	0.746303	245.70	-13.0	0.948064	249.78
-62.0	0.750372	245.79	-12.0	0.952066	249.84
-61.0	0.754439	245.87	-11.0	0.956067	249.93
-60.0	0.758504	245.97	-10.0	0.960067	250.00
-59.0	0.762569	246.05	-9.0	0.964066	250.08
-58.0	0.766631	246.14	-8.0	0.968064	250.15
-57.0	0.770693	246.22	-7.0	0.972060	250.23
-56.0	0.774753	246.31	-6.0	0.976055	250.31
-55.0	0.778811	246.40	-5.0	0.980049	250.38
-54.0	0.782868	246.48	-4.0	0.984041	250.46
-53.0	0.786924	246.56	-3.0	0.988033	250.54
-52.0	0.790978	246.65	-2.0	0.992023	250.62
-51.0	0.795031	246.73	-1.0	0.996012	250.69

8167-25-0 PLATINUM RESISTANCE THERMOMETER 1835855
(IPTS - 68) OCTOBER 1975

T.C. 1 M.A.

TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
0.0	1.000000	250.75	50.0	1.197833	254.65
1.0	1.003987	250.85	51.0	1.201759	254.73
2.0	1.007972	250.92	52.0	1.205684	254.80
3.0	1.011956	251.00	53.0	1.209607	254.88
4.0	1.015939	251.08	54.0	1.213529	254.96
5.0	1.019920	251.15	55.0	1.217450	255.04
6.0	1.023901	251.23	56.0	1.221370	255.12
7.0	1.027880	251.31	57.0	1.225289	255.19
8.0	1.031858	251.39	58.0	1.229206	255.27
9.0	1.035834	251.46	59.0	1.233122	255.35
10.0	1.039810	251.54	60.0	1.237037	255.43
11.0	1.043784	251.62	61.0	1.240951	255.51
12.0	1.047757	251.70	62.0	1.244864	255.59
13.0	1.051729	251.77	63.0	1.248775	255.60
14.0	1.055700	251.85	64.0	1.252685	255.74
15.0	1.059669	251.93	65.0	1.256594	255.82
16.0	1.063637	252.01	66.0	1.260502	255.90
17.0	1.067604	252.08	67.0	1.264409	255.98
18.0	1.071570	252.16	68.0	1.268314	256.06
19.0	1.075535	252.24	69.0	1.272218	256.13
20.0	1.079498	252.31	70.0	1.276121	256.21
21.0	1.083460	252.39	71.0	1.280023	256.29
22.0	1.087421	252.47	72.0	1.283924	256.37
23.0	1.091380	252.55	73.0	1.287823	256.45
24.0	1.095339	252.62	74.0	1.291721	256.53
25.0	1.099296	252.70	75.0	1.295618	256.61
26.0	1.103252	252.78	76.0	1.299514	256.68
27.0	1.107207	252.86	77.0	1.303409	256.76
28.0	1.111161	252.93	78.0	1.307302	256.84
29.0	1.115113	253.01	79.0	1.311195	256.92
30.0	1.119064	253.09	80.0	1.315086	257.00
31.0	1.123014	253.17	81.0	1.318976	257.08
32.0	1.126963	253.25	82.0	1.322864	257.16
33.0	1.130910	253.32	83.0	1.326752	257.23
34.0	1.134857	253.40	84.0	1.330638	257.31
35.0	1.138802	253.48	85.0	1.334523	257.39
36.0	1.142746	253.56	86.0	1.338407	257.47
37.0	1.146688	253.63	87.0	1.342290	257.55
38.0	1.150630	253.71	88.0	1.346171	257.63
39.0	1.154570	253.79	89.0	1.350052	257.71
40.0	1.158509	253.87	90.0	1.353931	257.79
41.0	1.162447	253.95	91.0	1.357809	257.87
42.0	1.166384	254.02	92.0	1.361686	257.95
43.0	1.170319	254.10	93.0	1.365561	258.02
44.0	1.174253	254.18	94.0	1.369436	258.10
45.0	1.178186	254.26	95.0	1.373309	258.18
46.0	1.182118	254.33	96.0	1.377181	258.26
47.0	1.186049	254.41	97.0	1.381052	258.34
48.0	1.189978	254.49	98.0	1.384922	258.42
49.0	1.193906	254.57	99.0	1.388790	258.50

8167-25-R PLATINUM RESISTANCE THERMOMETER 1835855
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T.C. 1 M.A.

TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
100.0	1.392657	258.59	150.0	1.584516	262.58
101.0	1.396523	258.66	151.0	1.588324	262.66
102.0	1.400388	258.74	152.0	1.592130	262.74
103.0	1.404252	258.82	153.0	1.595934	262.82
104.0	1.408115	258.90	154.0	1.599738	262.90
105.0	1.411976	258.98	155.0	1.603541	262.99
106.0	1.415836	259.05	156.0	1.607342	263.07
107.0	1.419695	259.13	157.0	1.611142	263.15
108.0	1.423553	259.21	158.0	1.614941	263.23
109.0	1.427410	259.29	159.0	1.618739	263.31
110.0	1.431265	259.37	160.0	1.622535	263.39
111.0	1.435119	259.45	161.0	1.626331	263.47
112.0	1.438972	259.53	162.0	1.630125	263.55
113.0	1.442824	259.61	163.0	1.633918	263.63
114.0	1.446675	259.69	164.0	1.637710	263.72
115.0	1.450525	259.77	165.0	1.641501	263.80
116.0	1.454373	259.85	166.0	1.645291	263.88
117.0	1.458220	259.93	167.0	1.649079	263.96
118.0	1.462066	260.01	168.0	1.652866	264.04
119.0	1.465911	260.09	169.0	1.656653	264.12
120.0	1.469755	260.17	170.0	1.660438	264.20
121.0	1.473597	260.25	171.0	1.664221	264.29
122.0	1.477438	260.33	172.0	1.668004	264.37
123.0	1.481278	260.41	173.0	1.671785	264.45
124.0	1.485117	260.49	174.0	1.675566	264.53
125.0	1.488955	260.57	175.0	1.679345	264.61
126.0	1.492792	260.65	176.0	1.683123	264.70
127.0	1.496627	260.73	177.0	1.686899	264.78
128.0	1.500461	260.81	178.0	1.690675	264.86
129.0	1.504294	260.89	179.0	1.694449	264.94
130.0	1.508126	260.97	180.0	1.698223	265.02
131.0	1.511957	261.05	181.0	1.701995	265.10
132.0	1.515786	261.13	182.0	1.705766	265.19
133.0	1.519615	261.21	183.0	1.709535	265.27
134.0	1.523442	261.29	184.0	1.713304	265.35
135.0	1.527268	261.37	185.0	1.717071	265.43
136.0	1.531093	261.45	186.0	1.720838	265.52
137.0	1.534916	261.53	187.0	1.724603	265.60
138.0	1.538739	261.61	188.0	1.728367	265.68
139.0	1.542560	261.69	189.0	1.732130	265.76
140.0	1.546380	261.77	190.0	1.735891	265.84
141.0	1.550199	261.85	191.0	1.739652	265.93
142.0	1.554017	261.93	192.0	1.743411	266.01
143.0	1.557833	262.02	193.0	1.747169	266.09
144.0	1.561649	262.10	194.0	1.750926	266.17
145.0	1.565463	262.18	195.0	1.754682	266.26
146.0	1.569276	262.26	196.0	1.758436	266.34
147.0	1.573088	262.34	197.0	1.762190	266.42
148.0	1.576899	262.42	198.0	1.765942	266.50
149.0	1.580708	262.50	199.0	1.769693	266.59

8167-25-B PLATINUM RESISTANCE THERMOMETER 1835855
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T.C. 1 M.A.

TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
200.0	1.773443	266.67	250.0	1.959458	270.87
201.0	1.777192	266.75	251.0	1.963149	270.95
202.0	1.780939	266.84	252.0	1.966838	271.04
203.0	1.784686	266.92	253.0	1.970527	271.12
204.0	1.788431	267.00	254.0	1.974214	271.21
205.0	1.792175	267.08	255.0	1.977900	271.29
206.0	1.795918	267.17	256.0	1.981585	271.38
207.0	1.799660	267.25	257.0	1.985269	271.46
208.0	1.803401	267.33	258.0	1.988951	271.55
209.0	1.807140	267.42	259.0	1.992633	271.63
210.0	1.810878	267.50	260.0	1.996313	271.72
211.0	1.814616	267.58	261.0	1.999992	271.81
212.0	1.818352	267.67	262.0	2.003670	271.89
213.0	1.822086	267.75	263.0	2.007347	271.98
214.0	1.825820	267.83	264.0	2.011022	272.06
215.0	1.829553	267.92	265.0	2.014697	272.15
216.0	1.833284	268.00	266.0	2.018370	272.23
217.0	1.837014	268.08	267.0	2.022042	272.32
218.0	1.840743	268.17	268.0	2.025713	272.41
219.0	1.844471	268.25	269.0	2.029383	272.49
220.0	1.848198	268.33	270.0	2.033052	272.58
221.0	1.851923	268.42	271.0	2.036719	272.67
222.0	1.855648	268.50	272.0	2.040386	272.75
223.0	1.859371	268.59	273.0	2.044051	272.84
224.0	1.863093	268.67	274.0	2.047715	272.92
225.0	1.866814	268.75	275.0	2.051378	273.01
226.0	1.870533	268.84	276.0	2.055039	273.10
227.0	1.874252	268.92	277.0	2.058700	273.18
228.0	1.877969	269.01	278.0	2.062359	273.27
229.0	1.881686	269.09	279.0	2.066018	273.36
230.0	1.885401	269.17	280.0	2.069675	273.44
231.0	1.889115	269.26	281.0	2.073331	273.53
232.0	1.892827	269.34	282.0	2.076985	273.62
233.0	1.896539	269.43	283.0	2.080639	273.70
234.0	1.900249	269.51	284.0	2.084291	273.79
235.0	1.903959	269.60	285.0	2.087943	273.88
236.0	1.907667	269.68	286.0	2.091593	273.96
237.0	1.911374	269.76	287.0	2.095242	274.05
238.0	1.915079	269.85	288.0	2.098889	274.14
239.0	1.918784	269.93	289.0	2.102536	274.23
240.0	1.922487	270.02	290.0	2.106181	274.31
241.0	1.926190	270.10	291.0	2.109826	274.40
242.0	1.929891	270.19	292.0	2.113469	274.49
243.0	1.933591	270.27	293.0	2.117111	274.58
244.0	1.937290	270.36	294.0	2.120752	274.66
245.0	1.940987	270.44	295.0	2.124391	274.75
246.0	1.944684	270.53	296.0	2.128030	274.84
247.0	1.948379	270.61	297.0	2.131667	274.93
248.0	1.952073	270.70	298.0	2.135303	275.01
249.0	1.955766	270.78	299.0	2.138939	275.10

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TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
300.0	2.142572	275.19	350.0	2.322786	279.66
301.0	2.146205	275.28	351.0	2.326361	279.75
302.0	2.149837	275.36	352.0	2.329934	279.84
303.0	2.153467	275.45	353.0	2.333507	279.93
304.0	2.157096	275.54	354.0	2.337078	280.02
305.0	2.160724	275.63	355.0	2.340648	280.11
306.0	2.164351	275.72	356.0	2.344217	280.20
307.0	2.167977	275.80	357.0	2.347784	280.30
308.0	2.171602	275.89	358.0	2.351351	280.39
309.0	2.175225	275.98	359.0	2.354916	280.48
310.0	2.178847	276.07	360.0	2.358480	280.57
311.0	2.182468	276.16	361.0	2.362043	280.66
312.0	2.186088	276.25	362.0	2.365605	280.75
313.0	2.189707	276.34	363.0	2.369166	280.85
314.0	2.193325	276.42	364.0	2.372725	280.94
315.0	2.196941	276.51	365.0	2.376284	281.03
316.0	2.200557	276.60	366.0	2.379841	281.12
317.0	2.204171	276.69	367.0	2.383397	281.21
318.0	2.207784	276.78	368.0	2.386952	281.31
319.0	2.211396	276.87	369.0	2.390505	281.40
320.0	2.215006	276.96	370.0	2.394058	281.49
321.0	2.218616	277.05	371.0	2.397609	281.58
322.0	2.222224	277.14	372.0	2.401159	281.67
323.0	2.225831	277.22	373.0	2.404709	281.77
324.0	2.229437	277.31	374.0	2.408256	281.86
325.0	2.233042	277.40	375.0	2.411803	281.95
326.0	2.236646	277.49	376.0	2.415349	282.05
327.0	2.240248	277.58	377.0	2.418893	282.14
328.0	2.243850	277.67	378.0	2.422436	282.23
329.0	2.247450	277.76	379.0	2.425978	282.32
330.0	2.251049	277.85	380.0	2.429519	282.42
331.0	2.254647	277.94	381.0	2.433059	282.51
332.0	2.258244	278.03	382.0	2.436597	282.60
333.0	2.261839	278.12	383.0	2.440135	282.70
334.0	2.265434	278.21	384.0	2.443671	282.79
335.0	2.269027	278.30	385.0	2.447206	282.88
336.0	2.272619	278.39	386.0	2.450740	282.98
337.0	2.276210	278.48	387.0	2.454272	283.07
338.0	2.279800	278.57	388.0	2.457804	283.16
339.0	2.283388	278.66	389.0	2.461334	283.26
340.0	2.286976	278.75	390.0	2.464863	283.35
341.0	2.290562	278.84	391.0	2.468391	283.45
342.0	2.294147	278.93	392.0	2.471918	283.54
343.0	2.297731	279.02	393.0	2.475444	283.63
344.0	2.301314	279.11	394.0	2.478968	283.73
345.0	2.304895	279.20	395.0	2.482492	283.82
346.0	2.308476	279.29	396.0	2.486014	283.92
347.0	2.312055	279.38	397.0	2.489535	284.01
348.0	2.315633	279.43	398.0	2.493055	284.10
349.0	2.319210	279.57	399.0	2.496573	284.20

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T.C. 1 M.A.

TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
400.0	2.500091	284.29	450.0	2.674470	289.12
401.0	2.503607	284.39	451.0	2.677927	289.22
402.0	2.507122	284.48	452.0	2.681384	289.32
403.0	2.510636	284.58	453.0	2.684839	289.42
404.0	2.514149	284.67	454.0	2.688293	289.52
405.0	2.517661	284.77	455.0	2.691746	289.62
406.0	2.521171	284.86	456.0	2.695198	289.71
407.0	2.524681	284.96	457.0	2.698648	289.81
408.0	2.528189	285.05	458.0	2.702097	289.91
409.0	2.531696	285.15	459.0	2.705546	290.01
410.0	2.535201	285.24	460.0	2.708992	290.11
411.0	2.538706	285.34	461.0	2.712438	290.21
412.0	2.542210	285.43	462.0	2.715883	290.31
413.0	2.545712	285.53	463.0	2.719326	290.41
414.0	2.549213	285.63	464.0	2.722768	290.51
415.0	2.552713	285.72	465.0	2.726209	290.61
416.0	2.556212	285.82	466.0	2.729649	290.71
417.0	2.559709	285.91	467.0	2.733088	290.81
418.0	2.563206	286.01	468.0	2.736525	290.91
419.0	2.566701	286.10	469.0	2.739962	291.01
420.0	2.570195	286.20	470.0	2.743397	291.11
421.0	2.573688	286.30	471.0	2.746831	291.21
422.0	2.577179	286.39	472.0	2.750264	291.31
423.0	2.580670	286.49	473.0	2.753695	291.41
424.0	2.584159	286.59	474.0	2.757125	291.51
425.0	2.587647	286.68	475.0	2.760555	291.61
426.0	2.591135	286.78	476.0	2.763983	291.72
427.0	2.594620	286.88	477.0	2.767409	291.82
428.0	2.598105	286.97	478.0	2.770835	291.92
429.0	2.601588	287.07	479.0	2.774260	292.02
430.0	2.605071	287.17	480.0	2.777683	292.12
431.0	2.608552	287.26	481.0	2.781105	292.22
432.0	2.612032	287.36	482.0	2.784526	292.32
433.0	2.615511	287.46	483.0	2.787945	292.42
434.0	2.618988	287.55	484.0	2.791364	292.53
435.0	2.622465	287.65	485.0	2.794781	292.63
436.0	2.625940	287.75	486.0	2.798197	292.73
437.0	2.629414	287.85	487.0	2.801612	292.83
438.0	2.632887	287.94	488.0	2.805026	292.93
439.0	2.636359	288.04	489.0	2.808439	293.04
440.0	2.639829	288.14	490.0	2.811850	293.14
441.0	2.643299	288.24	491.0	2.815260	293.24
442.0	2.646767	288.33	492.0	2.818669	293.34
443.0	2.650234	288.43	493.0	2.822077	293.44
444.0	2.653700	288.53	494.0	2.825484	293.55
445.0	2.657164	288.63	495.0	2.828889	293.65
446.0	2.660628	288.73	496.0	2.832293	293.75
447.0	2.664090	288.83	497.0	2.835696	293.86
448.0	2.667551	288.92	498.0	2.839098	293.96
449.0	2.671011	289.02	499.0	2.842499	294.06

8167-25-P PLATINUM RESISTANCE THERMOMETER 1835855 T.C. 1 M.A.
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TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
500.0	2.845898	294.16	550.0	3.014345	299.45
501.0	2.849297	294.27	551.0	3.017683	299.56
502.0	2.852694	294.37	552.0	3.021020	299.66
503.0	2.856089	294.47	553.0	3.024356	299.77
504.0	2.859484	294.58	554.0	3.027691	299.88
505.0	2.862878	294.68	555.0	3.031024	299.99
506.0	2.866270	294.79	556.0	3.034356	300.10
507.0	2.869661	294.89	557.0	3.037687	300.21
508.0	2.873051	294.99	558.0	3.041017	300.32
509.0	2.876440	295.10	559.0	3.044346	300.43
510.0	2.879827	295.20	560.0	3.047673	300.54
511.0	2.883214	295.31	561.0	3.050999	300.65
512.0	2.886599	295.41	562.0	3.054324	300.76
513.0	2.889983	295.51	563.0	3.057648	300.86
514.0	2.893365	295.62	564.0	3.060971	300.97
515.0	2.896747	295.72	565.0	3.064292	301.08
516.0	2.900127	295.83	566.0	3.067612	301.19
517.0	2.903506	295.93	567.0	3.070931	301.30
518.0	2.906884	296.04	568.0	3.074249	301.41
519.0	2.910261	296.14	569.0	3.077565	301.52
520.0	2.913637	296.25	570.0	3.080880	301.64
521.0	2.917011	296.35	571.0	3.084194	301.75
522.0	2.920384	296.46	572.0	3.087507	301.86
523.0	2.923756	296.56	573.0	3.090819	301.97
524.0	2.927127	296.67	574.0	3.094129	302.08
525.0	2.930496	296.77	575.0	3.097438	302.19
526.0	2.933865	296.88	576.0	3.100746	302.30
527.0	2.937232	296.99	577.0	3.104053	302.41
528.0	2.940598	297.09	578.0	3.107359	302.52
529.0	2.943963	297.20	579.0	3.110663	302.63
530.0	2.947326	297.30	580.0	3.113966	302.75
531.0	2.950689	297.41	581.0	3.117268	302.86
532.0	2.954050	297.52	582.0	3.120569	302.97
533.0	2.957410	297.62	583.0	3.123868	303.08
534.0	2.960769	297.73	584.0	3.127166	303.19
535.0	2.964126	297.84	585.0	3.130463	303.30
536.0	2.967482	297.94	586.0	3.133759	303.42
537.0	2.970838	298.05	587.0	3.137054	303.53
538.0	2.974191	298.16	588.0	3.140347	303.64
539.0	2.977544	298.26	589.0	3.143639	303.75
540.0	2.980896	298.37	590.0	3.146930	303.87
541.0	2.984246	298.48	591.0	3.150220	303.98
542.0	2.987595	298.59	592.0	3.153508	304.09
543.0	2.990943	298.69	593.0	3.156795	304.21
544.0	2.994290	298.80	594.0	3.160082	304.32
545.0	2.997635	298.91	595.0	3.163366	304.43
546.0	3.000980	299.02	596.0	3.166650	304.55
547.0	3.004323	299.12	597.0	3.169932	304.66
548.0	3.007665	299.23	598.0	3.173213	304.77
549.0	3.011005	299.34	599.0	3.176493	304.89

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RESISTANCE THERMOMETER 1835855

T.C. 1 M.A. 56

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TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.	TEMP. DEG. C	RESISTANCE RATIO	INVERSE DIFF.
600.0	3.173772	305.00	650.0	3.342137	310.85
601.0	3.183049	305.11			
602.0	3.186326	305.23			
603.0	3.189601	305.34			
604.0	3.192874	305.46			
605.0	3.196147	305.57			
606.0	3.199418	305.69			
607.0	3.202688	305.80			
608.0	3.205957	305.92			
609.0	3.209225	306.03			
610.0	3.212491	306.15			
611.0	3.215757	306.26			
612.0	3.219021	306.38			
613.0	3.222283	306.49			
614.0	3.225545	306.61			
615.0	3.228805	306.72			
616.0	3.232064	306.84			
617.0	3.235322	306.95			
618.0	3.238579	307.07			
619.0	3.241834	307.19			
620.0	3.245088	307.30			
621.0	3.248341	307.42			
622.0	3.251593	307.54			
623.0	3.254843	307.65			
624.0	3.258092	307.77			
625.0	3.261340	307.89			
626.0	3.264587	308.00			
627.0	3.267832	308.12			
628.0	3.271077	308.24			
629.0	3.274320	308.35			
630.0	3.277561	308.47			
631.0	3.280802	308.59			
632.0	3.284041	308.71			
633.0	3.287279	308.83			
634.0	3.290516	308.94			
635.0	3.293752	309.06			
636.0	3.296986	309.18			
637.0	3.300219	309.30			
638.0	3.303451	309.42			
639.0	3.306682	309.54			
640.0	3.309911	309.65			
641.0	3.313139	309.77			
642.0	3.316366	309.89			
643.0	3.319592	310.01			
644.0	3.322816	310.13			
645.0	3.326039	310.25			
646.0	3.329261	310.37			
647.0	3.332482	310.49			
648.0	3.335702	310.61			
649.0	3.338920	310.73			

CALIBRATION PROCEDURES

The procedure used to calibrate the platinum resistance thermometer to be used in the system, is outlined below. The preheater bath is incorporated as a multi-temperature bath used to set the value of the data points. Suggested temperatures for the calibration points are room temperature, 380° F, and 700° F.

- 1) Insert the calibrated thermometer, used as a standard, into the middle hole in the shield above the preheater bath.
- 2) Insert the thermometers to be calibrated in the surrounding holes to the same depth, and angle them in so that they are in contact with the calibrated thermometer.
- 3) Connect the leads of the calibrated thermometer to one Fluke meter and the leads of the thermometer to be calibrated to the other Fluke meter.
- 4) Set the bath temperature and read values for both thermometers once they stabilize. (During the calibration of the thermometers a cycling effect was noticed in the temperatures. The median value was used for the calibration purposes.)
- 5) Repeat step four for each thermometer at each temperature.

Table A1Results of Calibration

Thermometer, Sample Oil Inlet

Temperature	62.3687	208.912	385.572	561.622	711.607
Resistance	106.702	137.953	174.602	210.210	239.240

$$\text{ALPHA} = .0038448$$

$$\text{DELTA} = 1.541015$$

$$\text{RESISTANCE AT } 32^{\circ} \text{ F} = 100.121838$$

Thermometer, Sample Oil Outlet

Temperature	62.3153	208.703	385.488	561.632	711.936
Resistance	106.559	137.674	174.287	210.675	239.015

$$\text{ALPHA} = .0038366$$

$$\text{DELTA} = 1.475683$$

$$\text{RESISTANCE AT } 32^{\circ} \text{ F} = 100.014847$$

Thermometer, Freon Outlet

Temperature	62.3865	208.556	385.817	561.690	711.7068
Resistance	106.673	137.755	174.612	209.945	239.220

$$\text{ALPHA} = .0038455$$

$$\text{DELTA} = 1.537737$$

$$\text{RESISTANCE AT } 32^{\circ} \text{ F} = 100.090153$$

CALCULATION PROCEDURES

The values of alpha, delta, and resistance at zero degrees centigrade are calculated using the equations in the Leeds and Northrup Report of Calibration. A computer program using successive substitution proved to be adequate in solving the equations.

Once alpha, delta, and resistance at zero degrees centigrade was determined, the values were then used to determine the resistances at temperatures between 32 and 700° F at increments of 1° F.

APPENDIX B

Pressure Measurement Calibration

The certification report of the Heise gauge is included in this appendix as well as the calibration of the pressure transducers based on the Heise gauge readings.

A least-squares fit of the data was incorporated. The use of a linear equation for the transducers is assured because of the calibration certification received for both transducers. They show the pressure and the output to be directly proportional.



INDUSTRIAL VALVE & INSTRUMENT DIVISION NEWTOWN, CONNECTICUT 06470
 HEISE PLANT TEL. 203/428-4408 TWX: 710-467-0817

CERTIFICATION REPORT

of
HEISE GAUGE No. 69132

This gauge has been calibrated with a piston gauge which has been compared with master piston gauges whose effective areas were determined with an estimated accuracy of 3 parts in 100,000 by the National Bureau of Standards Identification No. P6744 and No. P6745. The weights for those dead weight piston gauges have also been certified by the Bureau of Standards to have an average accuracy of within one part in 35,000.

READING NUMBER	DEAD WEIGHT OR Hg COL. READING	GAUGE READING (Deviation from Dead Wt. or Hg Col.)
1	100 <i>psig</i>	-
2	200	-
3	300	-
4	400	-
5	500	-
6	600	-
7	700	-
8	800	-
9	900	-
10	1000	-
11	1100	-
12	1200	-
13	1300	-
14	1400	-
15	1500	-
16	1600	-
17	1700	-
18	1800	-
19	1900	-
20	2000 <i>psig</i>	-

Room Temperature at Test *70° F*

Maximum Hysteresis *2.0 psig*

Remarks: *Corrections are indicated where error is 2.0 psig or more.*

Date Tested *May 27, 1976*

Signed *A. K. Arndt*

CALIBRATION PROCEDURE

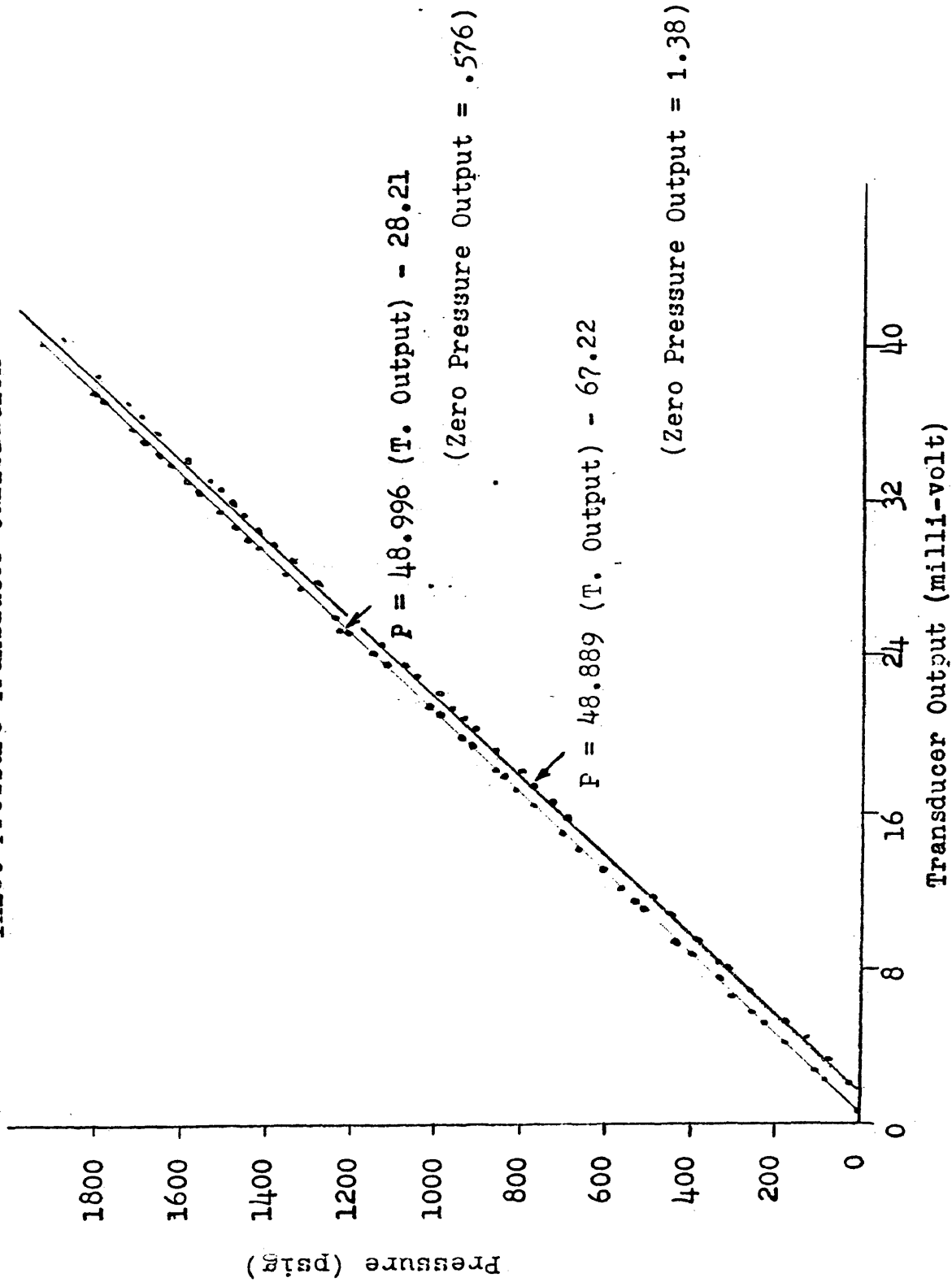
- 1) Adjust the back pressure regulator to a pressure of 2000 psig.
- 2) Start the main pump, keeping track of the pressure on the Heise gauge. When the pressure reaches 50 psig stop the main pump.
- 3) Pressurize the system using the bladder accumulator.
- 4) Adjust the pressure with the nitrogen regulator for the bladder accumulator to obtain the desired pressure for a data point.
- 5) Record the input and output to the transducer and the Heise gauge reading. (The input should be as close to 10.0 volts D.C. as possible.)
- 6) Repeat steps 4 and 5.

CALIBRATION RESULTS

The results of the calibration are displayed in graphs B1 and B2. A point of interest is the dependence of the inlet transducer's reading on the water flow rate. A change in flow rate changes the zero pressure output from the transducer. This is shown in graph B1. The slope of the equation remains almost constant and the intercept should be adjusted to fit the zero value reading for the day.

GRAPH B1

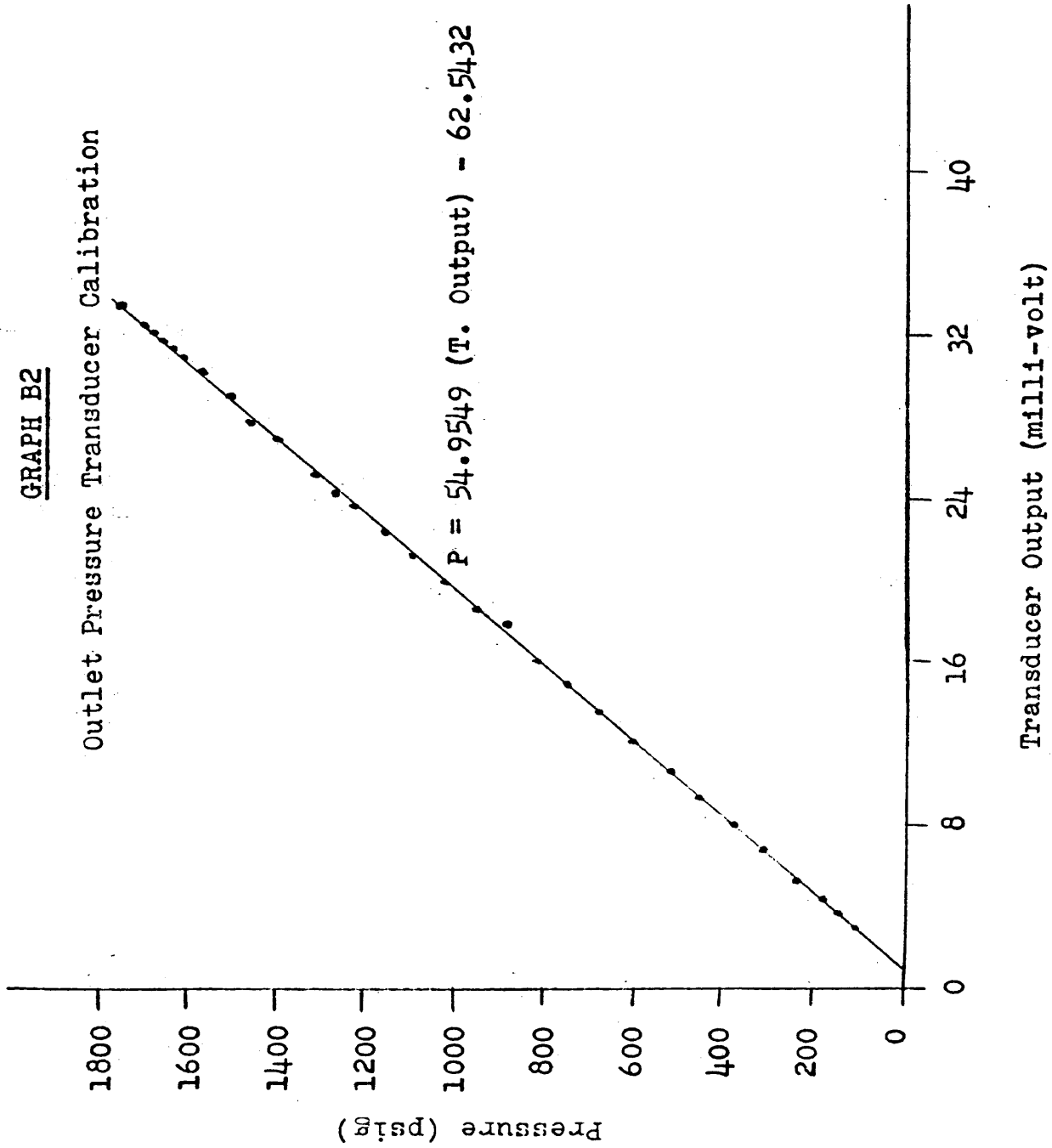
Inlet Pressure Transducer Calibration



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GRAPH B2

Outlet Pressure Transducer Calibration



APPENDIX CSuppliers of Major Equipment

Back-Pressure Regualtor (Grove)	Brock Easley Inc. 3700 Havana St., Suite 212 Denver, Colorado 80239
Balance (Mettler)	VWR Scientific 3700 Havana Denver, Colorado 80207
Bladder Accumulator (Greer)	Warren Engineering 2301 S. Inca Denver, Colorado 80223
Calorimeter	Cryenco Div. of Cryogenic Technology 4955 Bannock Denver, Colorado
Condenser (American Standard)	Air Mac Manufacturing Co. 1158 South Jason Street Denver, Colorado 80223
Final Heater	Control Products Corporation 4431 W. Division Street Chicago, Illinois 60651
Final Heater Controller (Honeywell)	Honeywell Inc. 7825 East Prentice Englewood, Colorado 80110
Fittings (Swagelock)	Denver Valve and Fitting P. O. Box 15636 970 Simms Denver, Colorado 80215
(Autoclave Engineers)	D. D. Frederick Company 115 W. 3rd Street Tulsa, Oklahoma
Fluke Meter	Burnhill Three 1980 S. Quebec, Unit 4 Denver, Colorado 80231

Freon Pump	Sargent Welch Scientific Co. 4040 Dahlia Street Denver, Colorado 80207
Glassware	R. H. Allen Company Box 27 Boulder, Colorado 80302
Heise Gauge	Heise Bourdon Tube Co., Inc. Newtown, Ct. 06470
Main Pump	Fluid Systems Inc. 1777 S. Bellaire Denver, Colorado 80222
Platinum Resistance Thermometer (Calibrated)	Leeds and Northrup 2715 S. Locust Street Denver, Colorado 80222
(Uncalibrated)	Omega Engineering Box 4047 Springdale Station Stamford, Ct. 06907
Power Supply	Hewlett Packard 7965 East Prentice Englewood, Colorado 80110
Preheater	Techne Inc. 3700 Brunswick Pike Princeton, N.J. 08540
Pressure Transducers	Precise Sensors Inc. 235 W. Chestnut Ave. Monrovia., Ca. 91016
Refrigeration Unit	Cecil Boling Co. 1050 Yuma Street Denver, Colorado 80204
Surge Tank	Ross Equipment Co. 2149 S. Clermont Denver, Colorado 80222
Timer	Sargent Welch Scientific Co. 4040 Dahlia Street Denver, Colorado 80207

Vacuum System

Remanufactured Vacuum Products
7250 W. 38th Ave.
Wheatridge, Colorado 80033

Variacs

VWR Scientific
3700 Havana
Denver, Colorado 80207

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