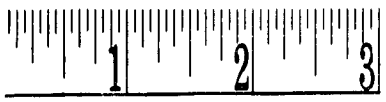
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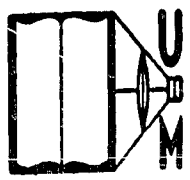
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AUTHOR Gopal Tripathi

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TO

MY MOST VALUED FRIEND N.V.S.

THERMAL PROPERTIES OF ORTHODICHLOROBENZENE

by

Gopal Tripathi

Committee in Charge:

Professor G. G. Brown, Chairman
Professor D. L. Katz
Professor R. R. White
Assistant Professor E. F. Westrum, Jr.
Mr. J. T. Banchero

A Dissertation Submitted in Partial Fulfillment of the Requirements for the Degree of Doctor of Science in Chemical Engineering at the University of Michigan.

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ABSTRACT

The purpose of this investigation was to determine the thermal properties of orthodichlorobenzene. The experiments consisted of the separate determination of the enthalpies of the liquid and the vapor, in addition to the measurements of the Joule-Thompson expansions. The following data were obtained:

1) The enthalpy of liquid orthodichlorobenzene was determined as a function of temperature at pressures of 15, 215, and 662 pounds per square inch absolute. The temperature range covered was from 60°F to 760°F. The enthalpy of the liquid referred to pure liquid at 32°F. and one atmosphere pressure can be represented - up to 800°F. - as follows:

$$H_L = -9.29 + 0.288t + 0.49 \times 10^{-4}t^2 + 0.106 \times 10^{-6}t^3$$

where H_L = Btu/pound and t = °F.

The isothermal effect of pressure on the enthalpy of the liquid was found to be negligible.

2) The enthalpy of orthodichlorobenzene vapor was determined from 400-800°F. at a constant pressure of 35 pounds per square inch absolute. The enthalpy of the vapor at one atmosphere pressure referred to pure liquid at 32°F. and one atmosphere, and the specific heat of the vapor at atmospheric pressure can be represented as:

$$H_v = 139.5 + 0.2092t + 8.891 \times 10^{-5}t^2$$

$$(C_p)_v = 0.2092 + 1.778 \times 10^{-4}t$$

where H_v = Btu/pound, C_p = Btu/pound/°F, and t = °F.

3) The vapor pressure of orthodichlorobenzene was determined experimentally up to 800°F. from the Joule-Thompson expansion data. The vapor pressure can be expressed as

$$\log_{10} P = 5.3987 - \frac{2972}{t + 350}$$

where P = psia, and t = °F.

The Joule-Thompson expansion data for the liquid, the liquid-vapor region, and the superheated vapor of orthodichlorobenzene was obtained. The temperature range covered was 400-874°F. and the pressure range was from atmospheric to 815 pounds per square inch absolute.

Charts of enthalpy versus temperature at lines of constant pressure, and pressure versus enthalpy at lines of constant temperature, were constructed from the Joule-Thompson expansion and enthalpy data. The saturated liquid, the saturated vapor, the two-phase region, and the single-phase regions are shown in these diagrams.

The critical temperature was estimated by extrapolation of the latent heat data. The estimated critical temperature of orthodichlorobenzene is 852°F. The estimated critical pressure is 845 pounds per square inch absolute.

The effect of pressure on the enthalpy of orthodichlorobenzene at constant reduced temperatures was determined and was found to be greater than the corresponding values obtained for water.

A valve-type (variable) throttling orifice was tested and found to reproduce the corresponding data obtained by a progressive-throttling type orifice. It was also found to possess many experimental advantages over the latter.

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THERMAL PROPERTIES OF ORTHODICHLOROBENZENE

INTRODUCTION

Orthodichlorobenzene is widely used in the chemical industry as a heat transfer medium in the range of 300-500°F. In view of the capital investment in the high-temperature heat transfer installations utilizing this medium, an accurate investigation of the thermal properties of orthodichlorobenzene would be of value in the engineering design of such installations. This investigation was therefore undertaken to determine the vapor pressure, liquid enthalpies, and vapor enthalpies up to the maximum temperature permissible before decomposition occurs.

Orthodichlorobenzene is commonly used under the trade name of "Dowtherm E" which consists predominately of orthodichlorobenzene with minor amounts of the paradichlorobenzene isomer. Dowtherm E, a special purified grade of orthodichlorobenzene, was the material studied in this investigation.

Due to the high temperature and pressures incurred in the measurements of the thermal properties of orthodichlorobenzene, it was decided to use the flow method for making these measurements. According to Callendar[4] the advantages of the flow method over the batch method are: (a) There is no time lag in measuring the temperature. (b) The size of

the equipment can be greatly reduced for a given heat exchange, thus reducing the external heat loss and improving response to any change of conditions. (c) There is no need for a correction for the water equivalent, which is so great a source of uncertainty in the batch methods at higher temperatures. (d) The flow of fluid itself, when properly directed, supplies sufficient stirring and makes it possible to dispense with mechanical devices for stirring. (e) Each part of the apparatus can be jacketed with its own flow, thereby reducing the heat loss. (f) If the material is decomposible at high temperatures and pressures, the properties can be evaluated without any appreciable decomposition because of the short time of contact of the fluid with the heated surface.

By the flow method the enthalpies of both liquid and vapor orthodichlorobenzene were measured at low and high pressures, isoenthalpic lines were obtained from regions of low pressures to regions of high pressures, and the vapor pressure curve was located by the lines of constant enthalpy expansions in the two-phase region.

METHODS FOR MEASURING THERMAL PROPERTIES

For the determination of the thermal properties it is essential to determine the enthalpy of the liquid and vapor as a function of temperature at a constant pressure. For

determination of liquid enthalpies the calorimetric method is used. However, for the determination of the vapor enthalpies, either a spectroscopic or a calorimetric method may be used. The spectroscopic method is reported to be very accurate and precise and gives values only at zero pressure or at pressures where the vapor behaves as an ideal gas. The molecular structure, the moment of inertia, and all the various translational, rotational, vibrational, and the combinations of rotational and vibrational energy levels should be known and well established. The calorimetric method is used extensively for the determination of vapor enthalpies, and good agreement has been obtained between the calorimetric and spectroscopic methods. Having once established the enthalpies of the liquid and vapor as a function of temperature at a definite pressure, the question arises as to how to extend the data to other pressures and temperatures. A direct course would be to determine calorimetrically the enthalpy of the substance as a function of temperature at all different pressures. This would be tedious and time consuming. It is more practical, experimentally, to extend the enthalpy data from one pressure to other pressures by any of several available methods. These extensions can be made directly by the use of isoenthalpic expansion data, directly by isothermal expansion data, or indirectly from

thermodynamic relations based on pressure, volume, and temperature (P-V-T) data. These methods will be discussed briefly in the following sections.

Isothermal Free Expansion.

In this method the fluid is throttled from a high pressure to a low pressure isothermally. During the expansion the material is maintained at a constant temperature by the addition of a measured quantity of electrical energy. Eucken and Berger[6] used the isothermal expansion method in studying the thermal properties of methane. They combined their isothermal data and available specific heat data in the construction of an enthalpy-temperature diagram. Gilliland and co-workers[8,9] have used this method for the study of the isothermal expansion of methane, n-pentane, n-heptane, and benzene vapors. This method is experimentally more difficult than the isoenthalpic expansion. It is a trial-and-error process, experimentally, to adjust the heat input to the flowing material in order to obtain the desired steady state final temperature. In view of the comparatively small heat quantities involved, the proportional experimental error is magnified by the experimental uncertainties in the isothermal expansion method. An additional disadvantage is that the properties of the two-phase region and the vapor pressure can not be determined by this method.

Extension of Enthalpy by P-V-T Data.

Low-pressure enthalpies of liquids and vapors can be extended to high pressures by the following thermodynamic relation:

$$\left(\frac{dH}{dP}\right)_T = T\left(\frac{dV}{dT}\right)_P - V$$

Lindsay and Brown[15] used Young's equation of state to calculate the isothermal changes in enthalpy with pressure for n-pentane. Using the P-V-T data of Beattie and co-workers, York[26] has calculated the isothermal changes in enthalpies with pressures for ethane, propane, butane, and heptane. Sage and Lacey and co-workers[21 and 22] have made an extensive use of this method in calculating the isothermal changes in enthalpy with pressure for paraffinic hydrocarbons.

In order to calculate the thermal properties from P-V-T properties, it is essential to have a precise measurement of their values because the first derivative of these values is used in the computation. Differentiation of P-V-T data inherently leads to a reduction in accuracy because of the uncertainties encountered in the differentiation. Further, this method is not reliable for thermal properties in the neighborhood of the critical as it is very difficult to get accurate P-V-T values in this region.

Recently the generalized compressibility factors and pseudo critical concepts have been used[25,27] to evaluate the generalized isothermal changes in enthalpy with pressures. If there is no available data, these generalized charts are satisfactory for estimates. However, the values predicted by the generalized charts are not always in agreement with actual experimental values, and the disagreement may be serious, especially for polar compounds.

Joule-Thompson Method.

In this method the fluid is expanded adiabatically from a high pressure and temperature to a lower pressure and temperature.

Patee and Brown[8] used the Joule-Thompson method in studying the thermal properties of n-pentane. In their investigation they determined isoenthalpic expansion data and also enthalpies of vapor at atmospheric pressure. These data were used in preparing a chart of pressure versus temperature for lines of constant enthalpy. Lindsay and Brown[15], Konz and Brown[14], Fekula[7], and Kemp[13] determined isoenthalpic expansion data for benzene, pentane, mixtures of paraffinic hydrocarbons, and mixtures of pentane and benzene, respectively. They have also determined the enthalpy data of the vapor at atmospheric pressure. Sage, Lacey and co-workers[2,19,20,23] have used this method for

determining the thermal properties of ethane, propane, n-butane, i-butane, n-pentane, methane, and mixtures of methane and n-butane.

By means of the Joule-Thompson method, it is possible: (1) To obtain data on the two-phase region, (2) To determine experimentally the vapor-pressure, (3) To determine accurately lines of constant enthalpy at high and low pressures and temperatures, and (4) To check the independent measurements of liquid and vapor enthalpies.

From this brief survey of the various methods of extending the enthalpy data from low pressures to high pressures it is evident that the Joule-Thompson method is the most desirable from the experimental point of view. This method provides data over a wider range than can be obtained by isothermal and P-V-T methods.

The Joule-Thompson expansion method, in conjunction with separate determinations of enthalpy as a function of temperature at a definite pressure, was used in this work for the determination of the thermal properties of ortho-dichlorobenzene.

EQUIPMENT

A flow diagram of the equipment is shown in Figure 1. It is similar to that used by Brown and co-workers[7,13,14,15,18] with modifications.

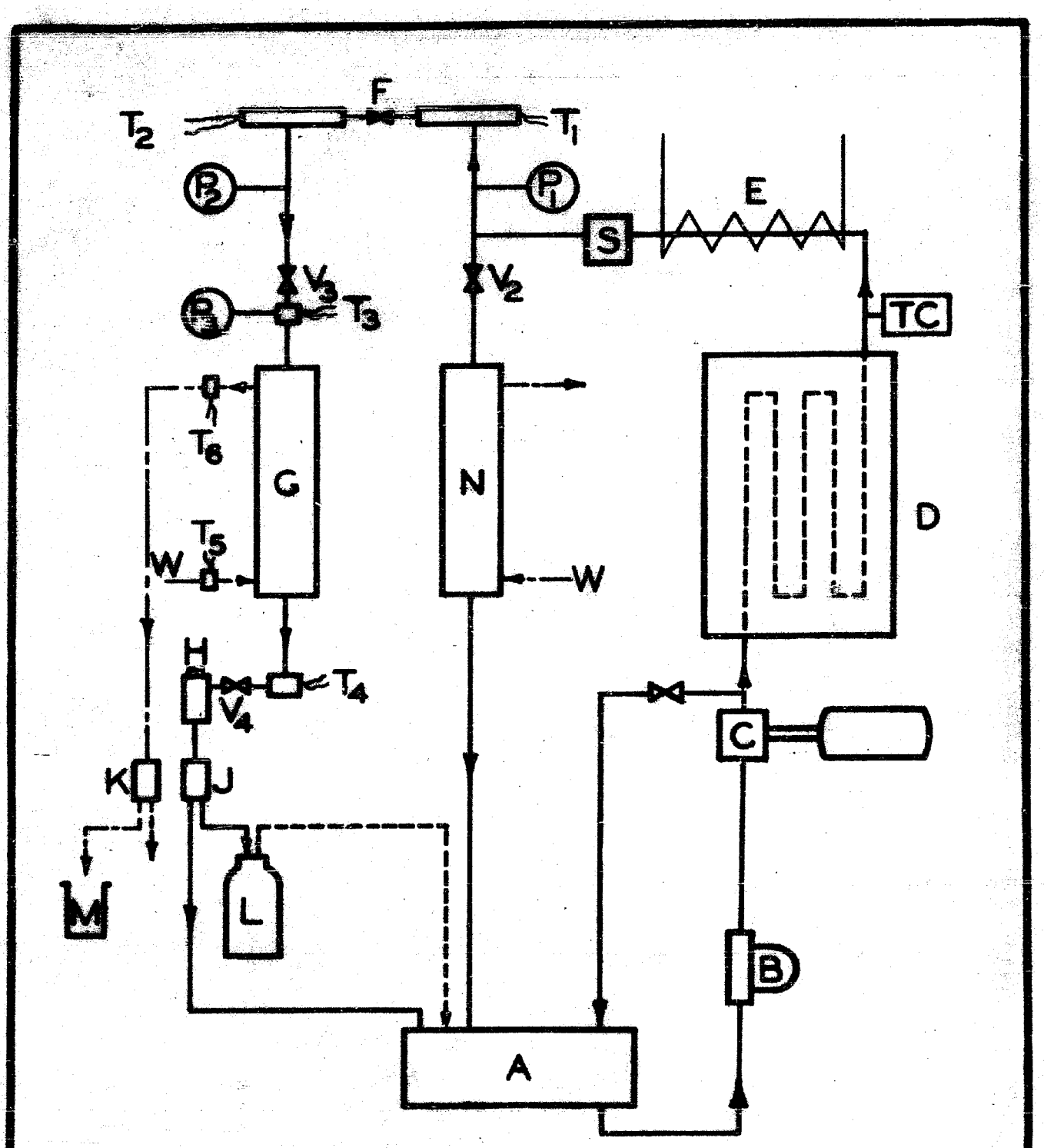


FIGURE 1. FLOW DIAGRAM OF APPARATUS

The material is continuously recirculated, flowing from supply tank A through a skinner filter B, to a Vickers hydraulic pump C, radiant electric furnace D, temperature controller TC, transfer line heater E, high pressure filter and settler S, adiabatic throttle F, high pressure flow calorimeter G, pressure regulator metric valve V_4, two-way valve j, and back to supply tank A. M is a cooler for the by-pass stream. K and L are containers for water and orthodichlorobenzene, respectively. These containers are used only during enthalpy measurements. Pressures are measured at P_1 and P_2 for Joule-Thompson measurements and at P_3 for enthalpy measurements. Temperatures are measured with chromel-alumel thermocouples T_1, T_2, T_3, T_4, T_5, and T_6. For enthalpy measurements, in addition to measuring the temperatures T_3, T_4, T_5, and T_6, the differences T_3-T_4 and T_5-T_6 are also measured by providing switches which connect these thermocouples in series.

Pump.

The Vickers hydraulic transmission unit, model AA16801A, which was developed during World War II, was used as a pump in this investigation. Although this type of unit was designed primarily for use as a speed reducer, by making certain modifications, it can be adapted as a pump which has a

capacity of 0-1 gallon per minute and a maximum discharge pressure of 1250 pounds per square inch. This pump is driven by a 1 Horsepower, 1750 R.P.M. motor, which is connected to the pump through another Vickers speed reducer. The speed can be regulated from 0-450 R.P.M. As this unit was designed to be used with lubricating fluids for short time intervals, the maximum pressure obtainable with ortho-dichlorobenzene (which is a non-lubricant) decreased appreciably after running for a period of time. As a result these pumps had to be discarded and replaced from time to time.

Electrical Heating Units.

The electric radiant heat furnace was built by Konz[14]. The furnace contains twelve lengths of National Shelby seamless steel tube (3 feet, 8 inches long; 3/8 inch inside diameter; and 3/4 inch outside diameter) electrically welded into return headers. These headers were machined from 2-inch square, cold-rolled, steel bar stock and were tapped for 3/4 inch pipe plugs. The 3/4 inch standard pipe plugs were made of Hills-McCanna bronze alloy No. 45. The pipe plug threads were lubricated with colloidal graphite in oil (Oil-dag).

The tube bank is surrounded on its sides and top by three equal lengths (107 ft x 1/2 inch x .036 inch thick

heating element) of Hoskins Chromel A resistance ribbon which are placed in the grooves of the unit brick. The electric wires are connected in Y to the three-phase circuit at one end of the furnace. Power is supplied to the furnace from a three-phase, 220 volt, sixty cycle line through an Allen Bradley relay. The total input power of the furnace is 15 kilowatts.

The temperature of the fluid leaving the furnace was controlled by a Leeds & Northrup platinum resistance thermometer (thermohm) and Leeds & Northrup Electromax Serial No. 705776, which operated the relay in the power line to the furnace. The temperature regulator has a range of 0-1000°F. and is reported to control the temperature within ±0.3%.

A transfer-line heater, E (Figure 1) was installed between the furnace outlet and the throttle to act as a superheater. This heater consists of 20 feet of 18 gage asbestos-insulated chromel wire wrapped around the line between the furnace and the throttle. An 110-volt Variac permits current adjustments between 0 and 5 amperes.

Filters.

To prevent the plugging of the orifices, two high pressure filter settlers were designed and connected in series. These high pressure filters were made from 2½-inch cold-

rolled hexagonal steel bar stock, 12 inches in length. A one-inch hole was drilled through each block. The top and bottom of each block were tapped for one-inch standard pipe plugs. The upstream filter contains a 60-mesh Monel screen supported on a ring frame which screws into the body of the filter. The downstream filter is similarly constructed but contains a 200-mesh screen. The coarse filter (60 mesh size) has a 3-inch open space below the inlet pipe to the filter to permit the settling of the larger particles. It was found that, due to very fine corrosion products, a pressure drop of 600 pounds per square inch across the filters would be built up in a few hours of running at 700°F. Under these conditions it was impossible to get any data, and most of the time was spent in cleaning the filters. After many repeated attempts to eliminate this difficulty it was decided to replace the screen filters by loosely-packed pyrex glass wool. The same effect of high pressure drop across the filters was noted. Many other types of filtering materials were tried but without any success. Finally, the high pressure filter bodies were packed with ½-inch Raschig rings, and the fixed orifices in the adiabatic throttle were replaced by valve orifices which do not require a clear filtrate. (See page 24.)

Adiabatic Throttle.

A progressive series of orifices, separated by spacers and increasing gradually in size, known as progressive throttling, were first used for adiabatic throttling. The size of holes drilled in 3/8" O.D. x 1/8" thick, circular stainless steel orifices were 1/64", 1/32", 1/16" and 1/8". A typical example of the arrangement of the progressive throttles was: An orifice with a 1/16" hole, a 1/8" spacer, an orifice with two 1/16" holes, spacer, one 1/8" hole, spacer, two 1/8" holes, spacer, etc. The selection of the orifices depended upon the desired pressure drops and flow rates.

As the material under investigation was corrosive, the orifices became plugged easily; therefore, they were replaced by a variable orifice which simply consisted of a 1/4" Metric needle valve.

High Pressure Calorimeter.

The same calorimeter as described by Fekula[7] was used for the first few runs. After some time a leak developed in the calorimeter due to corrosion. A new calorimeter was then designed and constructed.

The flow calorimeter was designed to operate up to 5000 pounds per square inch and 1000°F. It consists of 21 ft. of

3/16-inch I.D. x 5/16-inch O.D. stainless steel tubing coiled on a 1½-inch pipe which is 24 inches long and which is closed on both ends. The tubing and pipe were placed inside the shell of the calorimeter. The main purpose of the pipe on which the tubing is coiled is to decrease the free space for the flow of water and thereby increase the velocity of the water which is being circulated in the shell. The net result is to produce a very uniform outlet water temperature.

The shell of the calorimeter consists of a 2-inch, Schedule 40, pipe, 27 inches in length. The inlet and outlet connections for the cooling water that circulates through the shell are ¼-inch, Schedule 40, pipe and are welded into the bottom and top of the shell. Openings for a thermocouple-well and pressure gage connections to the tubing were made by means of a high pressure fitting which was welded to the top of the calorimeter. A similar fitting, having only a thermocouple-well, was welded to the bottom of the calorimeter for measuring the outlet temperature of the fluid in the tubing. The calorimeter and fittings were insulated with 2 inches of 85 per cent magnesia insulation. The pressure in the calorimeter was controlled by the valve V_4, Figure 1.

The fluid condensate outlet and water outlet from the calorimeter are connected to the two-way valves J and J'. During the enthalpy runs, there are no orifices in the adiabatic throttle, and the two-way valves are opened to permit water and orthodichlorobenzene to flow into the receivers K and L, respectively, for flow measurements. During adiabatic expansion experiments the condensate is returned to supply tank A, and the water into the drain.

The pipe line from the outlet of the furnace to the calorimeter, the adiabatic throttle, the high pressure filter-settler, part of the calorimeter, and part of the by-pass line for cooler M were insulated by placing them in a rockwool packing of 14 inches in height. The packing was loose in order to increase the efficiency of the insulation.

CALIBRATION OF INSTRUMENTS

The chromel-alumel thermocouples were calibrated up to 175°C. in a constant temperature oil bath against a thermometer which was standardized by the National Bureau of Standards. For temperatures above 175°C. the thermocouples were calibrated against freezing points of lead, tin and zinc which were standardized by the National Bureau of Standards. The procedure followed was as recommended in the Bureau of Standards Research Paper RP768.

The thermocouples were also calibrated against a National Bureau of Standards certified platinum to platinum-rhodium thermocouple.

Check calibrations made at different times during experimental work agreed within 0.2°F.

The thermocouple potentials were plotted against temperatures and the following equation was derived:

$$t = 43.78E + 32$$

t is the temperature in °F, E is the electromotive potential in millivolts. Corrections had to be applied to the above equation. The magnitude of these corrections are shown in Figures12A, 12B, 12C, 12D of the Appendix.

A Leeds & Northrup portable precision potentiometer, Serial No. 325550, was used for all electromotive force measurements and was calibrated periodically with a standard cell.

Pressure gages were calibrated with an "American" gage tester, Type No. 1315, to ±1 lb./sq.in. for pressures up to 1000 lbs./sq.in. The gages were calibrated at frequent intervals to check previous calibrations. Calibration curves for the gages used are presented in Figure 13A, 13B, 13C of the Appendix.

MATERIALS USED

Dowtherm E, a special grade of orthodichlorobenzene, was the material used in this investigation. The material was supplied by the Dow Chemical Company. True boiling point distillation analyses are presented in Table I of the Appendix and Figure 2. A special high-temperature fractional distillation analysis column was used. Density and refractive index for the pure material together with densities and refractive indices of the cuts from the true boiling point distillation are presented in Table II of the Appendix. The densities were obtained by the pycnometer method and the refractive indices were determined by an Abbe refractometer.

The physical properties of this material as reported by Dow Chemical Company are:

Viscosity	1.05	centipoise	at 100°F.
	0.62	"	at 200°F.
	0.41	"	at 300°F.

Molecular wt	147
Freezing point	-6.7°F.
Heat of fusion	38 Btu per pound
Flash point	154°F.
Fire point	284°F.
Normal boiling point	352°F.
Density at 60°F.	81.7 lbs./cu.ft.

Control distillations before and after many of the experimental adiabatic expansions and enthalpy experiments were made to determine if any change in compositions of the

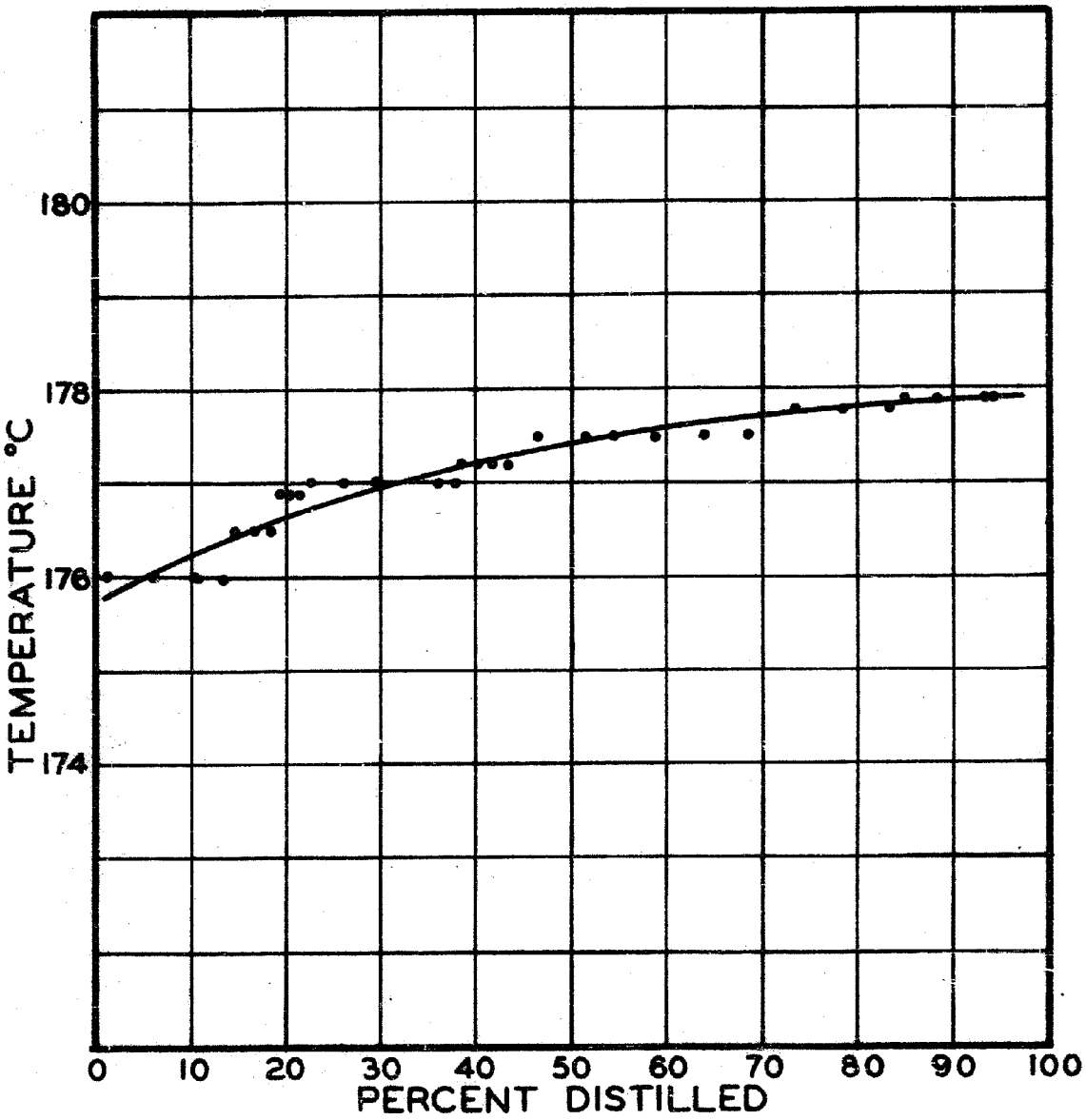


FIGURE 2

TRUE-BOILING-POINT DISTILLATION

material occurred from evaporation loss or thermal cracking. Density and refractive index measurements were also made. All control data checked within the accuracy of the methods used, indicating that no appreciable change in composition occurred in the course of experiments.

OPERATING PROCEDURES

Enthalpy Measurements.

The flow calorimetric method was used to measure the enthalpy of the fluid as a function of temperature at any pressure. In the calorimetric process, the hot fluid exchanges heat with water and the amount of the fluid and water and their inlet and outlet temperatures are noted. The enthalpy of the fluid is obtained by a heat balance.

The high pressure flow calorimeter was used for measuring the enthalpies of the fluid. To measure the enthalpy at any definite pressure, the Joule-Thompson throttle orifices were removed from the adiabatic throttle. The fluid was circulated through the furnace and the calorimeter during the warm-up period (3 to 4 hours) until the desired temperature, T_3, (Figure 1) was attained. The pressure at which the measurement was to be made was regulated manually by the metric needle valve, V_4. Fixed orifices for controlling

the pressure in the calorimeter could not be used because of the plugging by corrosion products. When the inlet and outlet temperatures of the fluid and cooling water became constant, valves J and J' (Figure 1) were turned simultaneously so that the fluid and cooling water from the calorimeter flowed into their respective receivers, at which time readings were begun. The fluid and water were collected for 3-6 minutes depending upon the flow rates. During this time interval various readings of thermocouples T_3, T_4, T_5, and T_6 and differences (T_3-T_4) and (T_6-T_5) were read on the potentiometer. Several readings of the pressures in the calorimeter were also recorded. Arithmetic average values of each temperature were taken for the heat balance. In order to calculate a value for enthalpy, it was necessary to know the differences of (T_3-T_4) and (T_6-T_5) thermocouples. In calculating these temperature differences from individual thermocouple readings a difference of two large numbers must be taken, which lowers the accuracy in the region where the temperature differences were small. By providing switches which connected these thermocouples in series, these differences could be read directly on the potentiometer, thus considerably increasing the accuracy of the measurements.

Duplicate and some triplicate check runs with different flow rates of the fluid were made. When the enthalpy value obtained from all the runs checked with each other, the

temperature of thermocouple T_3 was changed by resetting the temperature controller. The different flow rates were used to determine the heat losses, if any. If the enthalpy values had not checked with each other, heat losses could have been calculated easily from these runs. During this investigation it was found that the heat losses from the calorimeter were negligible.

A constant head device was used for delivering water to the calorimeter. In some runs, the fluctuations in the outlet temperature of the water were quite appreciable. These runs were discarded.

In the measurement of enthalpy, the flow rate of the fluid was kept between 1-2 lbs./minute.

The enthalpy values are referred to liquid orthodichlorobenzene at 1 atmosphere and 32°F. as the standard state. The enthalpy of the water corresponding to the inlet and outlet temperatures of the water were read from the Keenan and Keyes Steam Tables.(12)

A sample calculation for calculating the enthalpy is shown below:

Run No. 170

Data

Orthodichlorobenzene E.M.F. Inlet E_3	Outlet E_4	E_3-E_4	Cooling Water E.M.F. Inlet E_5	Outlet E_6	E_6-E_5	Pressure Lbs./Sq.In. Gage
8.938	1.913	7.025	.66	1.96	1.30	200
		7.025			1.30	200
		7.020			1.30	200
		7.025			1.30	200
		7.020			1.303	200
8.930	1.913	7.020	.66	1.95	1.303	200
	Average =	7.022		Average =	1.301	

$E_3 = 8.934$ $E_4 = 8.934-7.022 = 1.912$ $E_5 = .66$ $E_6 = 1.301+.66 = 1.961$

$t_3 = 423.13°F$ $t_4 = 115.79$ $t_5 = 60.89°F$ $t_6 = 120.83$

Pressure 215 P.S.I.A.

Weight of orthodichlorobenzene = 3.256 kilogram
Weight of cooling water = 5.945 kilogram
Time of experimental run = 3½ minutes

Heat balance around the calorimeter:

$$H_3-H_4 = \frac{(\text{wt. of water collected})}{(\text{wt. of orthodichlorobenzene})}(H_6-H_5)$$

where

H_3 and H_4 are enthalpies of the orthodichlorobenzene at 423.13°F. and 115.79°F. and at a pressure of 215 pounds per square inch absolute.

H_6 and H_5 are the enthalpies of the water at 120.83°F. and 60.89°F.

$$H_3-H_4 = \frac{5.945}{3.256}(88.75-28.95)$$

$$= 109.44$$

In order to refer the enthalpies of H_3 with reference to liquid orthodichlorobenzene at 32°F. and 1 atmosphere, it is necessary to know the change in temperature of liquid orthodichlorobenzene when it is expanded from 215 pounds per square inch absolute and 115.79°F. to 1 atmosphere. From the Joule-Thompson expansion of liquid orthodichlorobenzene, it was found that the temperature change equals zero. H_4 is then calculated from the equation:

$$H_L = -9.15 + 0.283t + 0.000094t^2 \quad \text{(Equat. 1, page 31)}$$

$$H_4 = 24.82 \text{ B.t.u./lb.}$$

Final enthalpy of orthodichlorobenzene at 423.14°F. and 215 pounds per square inch absolute referred to liquid orthodichlorobenzene at 32°F. and 1 atmosphere is 109.44 + 24.82 = 134.26 B.t.u./lb.

Joule-Thompson or Iso-Enthalpic Expansion

The function of Joule-Thompson expansion is to reproduce the enthalpy condition of a fluid at a lower pressure while keeping the kinetic effects at a negligible value.

The experimental aims of Joule-Thompson expansion consist essentially of two factors: (1) To reduce the pressure of the fluid and (2) To thoroughly mix the stream on the low pressure side so that it is of uniform temperature and pressure throughout. The porous plug performs both these

operations simultaneously. A single orifice succeeds in reducing the pressure but provides for very little mixing. The progressive throttle, a series of orifices increasing gradually in size and separated from each other by baffles or open spaces, performs mixing action along with pressure drop. The needle valve type orifice provides a throttling action which creates a pressure drop and also produces thorough mixing as a result of the turbulence created by the impingement of the expanded fluid against the valve body and by the abrupt change in direction.

With the proper mixing conditions in the Joule-Thompson expansion, the final velocity of the fluid is independent of the type of the orifice used. With the usual flow rates employed in Joule-Thompson expansions, the thermal effects of the resulting velocity changes are of such a small magnitude that they can be neglected.

Progressive throttling was used initially. However, due to frequent plugging of the orifices at about 600°F., it was decided to change over to the valve-type orifice. It was found that the valve-type orifice offered several practical advantages in obtaining isoenthalpic data. The valve orifice was readily cleared of obstructions which clog

the passage opening by moving the valve spindle. In the case of progressive throttling orifices, such clogging action necessitated the complete shut down of the equipment and dismantling of the pipe to clean the unit. A very useful feature of the valve or variable orifice was that the orifice size could be varied as desired to maintain substantially a constant rate of flow through a series of Joule-Thompson expansion measurements. This adjustment was not possible in the case of progressive throttling without shutting down the equipment to change the orifices.

Adiabatic conditions can be approached only by maintaining the flow rate above that point where the rate of heat loss experienced is negligible in comparison with the quantity of the fluid flowing. For this reason the adiabatic conditions cannot be approached at the very small flow rates. Maintenance of adiabatic conditions in Joule-Thompson expansion is essential; otherwise for the same initial pressure and temperature conditions, "isoenthalpic" lines will have different slopes. In the case of progressive throttling, non-adiabatic conditions may result when the downstream pressure is approaching the upstream pressure because of the decreased flow rate. This behavior has been observed in the case of steam[3] when the data of various workers is compared with each other for the same enthalpy.

Isoenthalpic expansion was obtained by expanding adiabatically from a given inlet temperature and pressure through the adiabatic throttle to the minimum outlet pressure available. The constant enthalpy line was followed by increasing the outlet pressure in small increments and by taking temperature and pressure readings when equilibrium had been attained after each readjustment of valve V_3. By this method all results were obtained from one thermocouple and one pressure gauge, thus eliminating errors arising from slight changes in the relative characteristics of the separate gauges and thermocouples. In some cases, instead of expanding the fluid to the lowest pressure obtainable, the run was started with the highest pressure obtainable on the downstream side for a given initial pressure and temperature and then progressively decreased to the lowest available pressure. The check between both procedures was within experimental accuracy. The temperature of the fluid entering the throttle was maintained within $\pm 0.5^{\circ}$F. by adjusting the temperature controller and the current to the line heater. The pressure of the fluid entering the throttle was maintained within ± 1 lb. in the range of 500 lbs./sq.in. and ± 2 lbs. in the range of 500-1000 lbs./sq.in. The inlet pressure was maintained either by changing the orifice size

or flow rate. Final regulation was done by the by-pass valve V_2 (Figure 1).

Before starting to record the data for constant enthalpy runs, the heat losses between the two thermocouples in the throttle were checked. This was done by changing the flow rate of the fluid through the throttle and measuring the downstream temperature while keeping the inlet pressure and temperature constant. The outlet temperature of the throttle T_2 was plotted against $\frac{1}{\text{flow rate}}$. The line joining the temperature readings at high flow rates was extended to $\frac{1}{\text{flow rate}} = 0$. The difference between the intercept reading of T_2 and T_2 obtained at a given flow rate was the correction for non-adiabatic conditions due to a change in flow through the throttle. It was found that the minimum flow rate for which no appreciable correction for heat loss was needed was 200 cc/minute.

Preliminary experiments for Joule-Thompson expansions were made at inlet pressures of 800 lbs./sq.in. and temperatures ranging from 450-700°F. It was observed that the entire vapor range could not be covered. This necessitated the study of Joule-Thompson expansions for superheated vapor alone. The inlet pressure was kept at least 50 lbs./sq.in. less than the vapor pressure at the inlet temperature.

If the inlet pressure to the throttle was kept close to the saturation pressure any slight increases in pressure brought about by the accumulation of fine particles in the throttle valve would tend to produce saturation conditions at this point. As a result, the first part of the Joule-Thompson expansion would follow the vapor-pressure curve instead of remaining entirely in the superheat region. At temperatures above 700°F. and pressures above 500 psi, fine scale plugged the expansion valve so rapidly that only instantaneous readings for complete isoenthalpic expansions could be obtained.

Vapor-Pressure Determination.

According to the phase rule for a single component, where the two phases are in equilibrium, there is only one degree of freedom. This principle was utilized in determining the vapor pressure from Joule-Thompson expansion data. The actual experimental procedure consisted of determining various isoenthalpic expansions from the liquid phase for different inlet temperatures and pressures. When equilibrium existed between the liquid and vapor, the temperatures were identical for the same outlet pressure regardless of the different initial pressures and temperatures. The establishment of two phases and thermal equilibrium was very rapid.

The temperature during this investigation was read within ±0.5°F. The pressure readings were as follows:

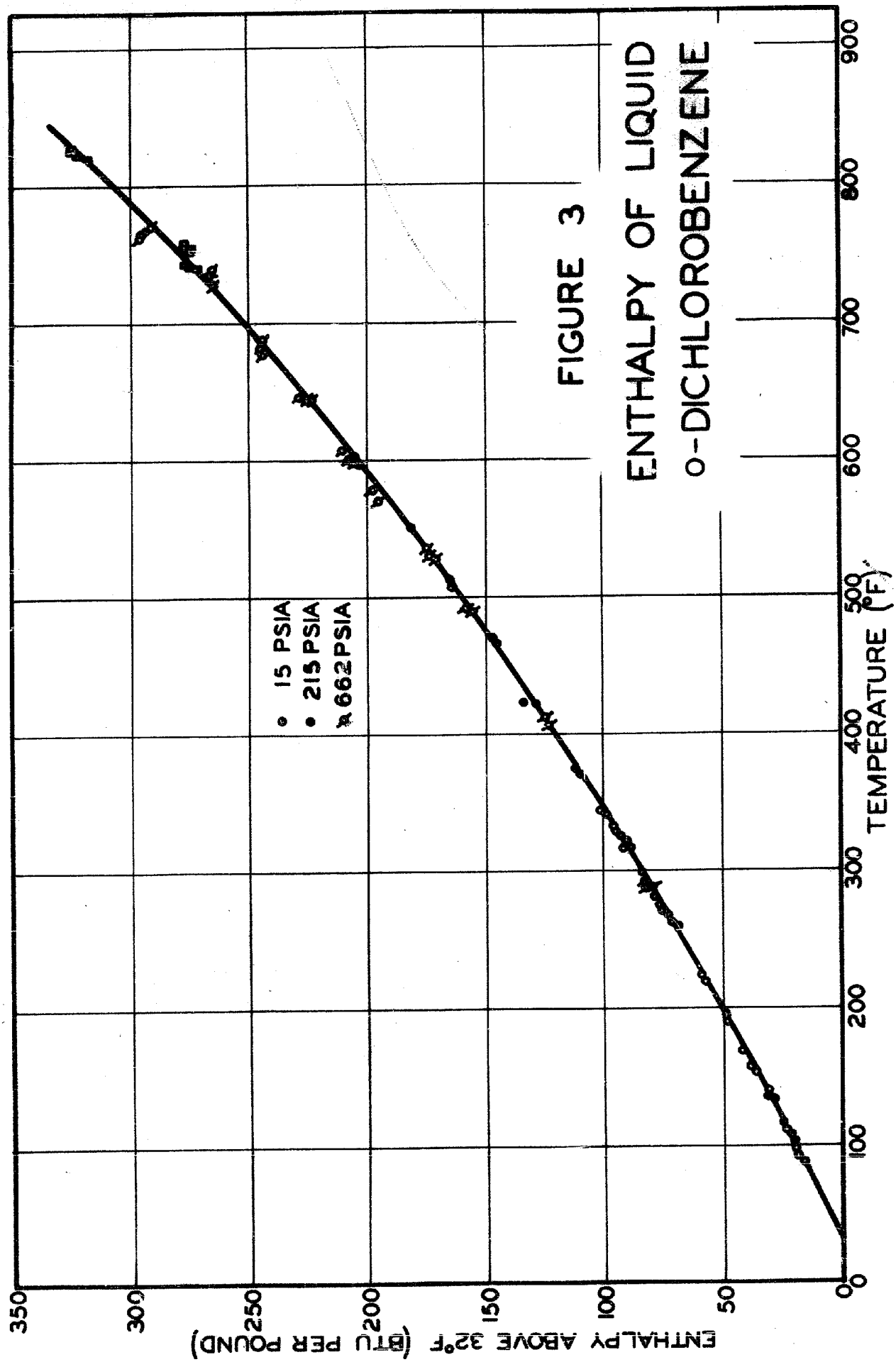
In the range of	0-50	pounds/sq.in.	±.2 lbs./sq.in.
" " "	50-200	" " "	±1.0 lbs./sq.in.
" " "	200-1000	" " "	±2 lbs./sq.in.

EXPERIMENTAL RESULTS AND DISCUSSION

Liquid Enthalpy.

The enthalpy of liquid orthodichlorobenzene was determined at 15, 215 and 662 pounds per square inch absolute. The results are presented in Tables III, IV, and V of the Appendix and in Figure No. 3. The liquid enthalpies are referred to a standard state of liquid orthodichlorobenzene at 32°F. and 1 atmosphere pressure.

Since the experimental results obtained in measurements on liquid enthalpies are in the form of enthalpy differences and temperature differences, an empirical expression of the enthalpy as a function of temperature was utilized to obtain relative enthalpy values from the experimental data. From the experimental data the necessary constants in this empirical expression were evaluated.


FIGURE 3
ENTHALPY OF LIQUID
O-DICHLOROBENZENE
15 PSIA
215 PSIA
662 PSIA
ENTHALPY ABOVE 32°F (BTU PER POUND)
TEMPERATURE (°F)
0
50
100
150
200
250
300
350
100
200
300
400
500
600
700
800
900

In the determination of enthalpy by a flow calorimeter, $H_3-H_4 = \frac{w_w}{w_o}(H_6-H_5)$, where H_4 and H_3 are the enthalpy values corresponding to the inlet and outlet temperatures of the orthodichlorobenzene liquid; H_6 and H_5 are the enthalpy values corresponding to the outlet and inlet temperatures of the water as read from the Keenan and Keyes Steam Tables[12]; w_w and w_o are the weights of water and orthodichlorobenzene, respectively. In order to find H_3 it was necessary to know the value of H_4. As there were no reliable specific heat data of the liquid orthodichlorobenzene at any temperature, it was assumed that the enthalpy of the liquid can be expressed in the form of

$$H_L = a + bt + ct^2, \text{ where } t = {}^\circ F.$$

Since the values for the quantity (H_3-H_4) and the corresponding temperatures were obtained as experimental data, the values for the constants a, b and c in the above equation were evaluated by plotting $\frac{H_3-H_4}{t_3-t_4}$ versus (t_3+t_4). The equation thus developed fits the data up to the maximum temperature of 350°F., obtainable on the runs performed at 15 p.s.i.a., and may be expressed as

$$H_L = -9.15 + 0.283\ t + 0.000094\ t^2. \qquad (1)$$

In calculating the enthalpy of the liquid at 215 and 615 p.s.i.a. with reference to pure liquid at 32°F. and one atmosphere, it was necessary to evaluate the differential ratio

$\left(\frac{dT}{dP}\right)_H$ for the liquid. In the Joule-Thompson experiments for liquid it was observed that the change in temperature with pressure for constant enthalpy in the liquid phase is negligible. This was confirmed by the liquid enthalpy data at 215 and 615 p.s.i.a., as the values of H obtained experimentally for a certain temperature differences at these pressures checked very well with the calculated values of H obtained from the Equation (1) for 15 p.s.i.a. However, the enthalpy of the liquid at different pressures was measured in order to extend the data of liquid emthalpies to the higher temperatures.

The enthalpy and specific heat of the liquid up to 800°F. can be represented as follows:

$$H_L = -9.29 + 0.288\ t + 0.49 \times 10^{-4} t^2 + 0.106 \times 10^{-6} t^3 \qquad (2)$$

and $$\left(\frac{dH_L}{dT}\right)_P = (C_p)_L = 0.288 + 0.98 \times 10^{-4}\ t + 0.324 \times 10^{-6} t^2 \qquad (3)$$

These constants were obtained by using the smoothed curve shown on Figure No. 3. Since the effect of pressure on the enthalpy of the liquid is negligible, Equations (2) and (3) apply to the saturated liquids as well.

Vapor Enthalpy.

The enthalpy of the superheated vapor at 35 p.s.i.a. was obtained and is presented in Table No. VI of the Appendix and Figure No. 4. This enthalpy was determined in order to

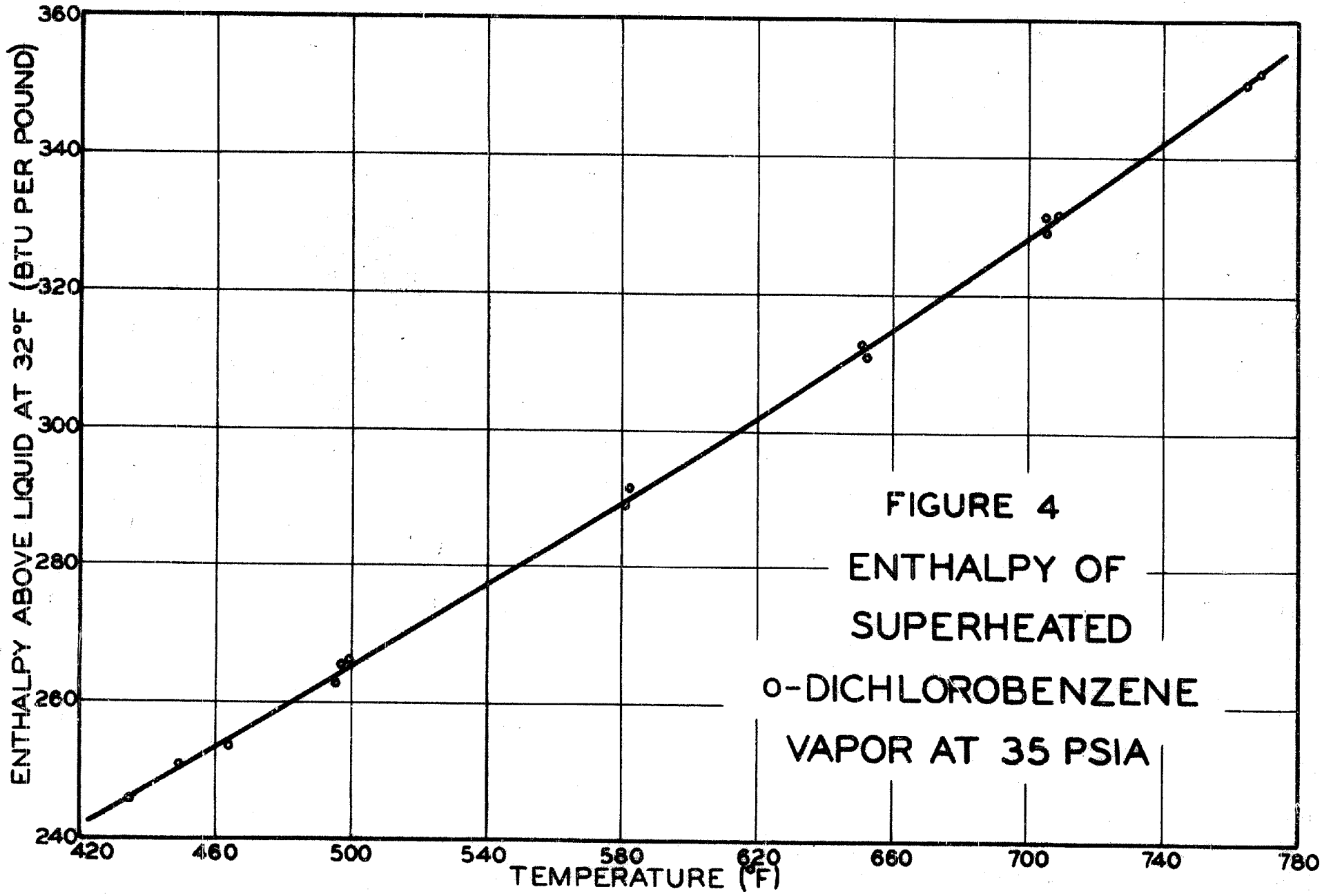


FIGURE 4

ENTHALPY OF SUPERHEATED o-DICHLOROBENZENE VAPOR AT 35 PSIA

identify the Joule-Thompson isoenthalps for superheated vapor.

The enthalpy and specific heat of the vapor at atmospheric pressure is represented by the equations:

$$H_V = 139.5 + 0.2092\ t + 8.891 \times 10^{-5} t^2 \qquad (4)$$

and $$\left(\frac{dH_V}{dT}\right)_P = (C_p)_V = 0.2092 + 1.778 \times 10^{-4} t \qquad (5)$$

The maximum deviation of the experimental points from the smoothed data is 2 Btu per pound and the average deviation is less than 1 Btu per pound.

The separate determinations of the liquid and vapor enthalpies were checked for mutual consistency by means of the complete Joule-Thompson expansion from liquid to superheated vapor based on the assumption that the enthalpies of the superheated vapor at 35 p.s.i.a. were correct. The enthalpies of the liquid obtained by the Joule-Thompson expansion data and the vapor enthapies at 35 p.s.i.a. are shown as rectangular points in Figure No. 3 and are presented in Table VII of the Appendix. That these points fall on the smooth curve of the liquid enthalpies is a good indication of the consistency of the data.

Vapor Pressure Determination.

The data for the vapor pressure are presented in Table No. VIII of the Appendix. Vapor pressure data are shown together with Joule-Thompson data. Antoine's[1] type of equation for vapor pressure has been plotted in Figure No. 5. The vapor pressure can be expressed as

$$\text{Log}_{10} P = 5.3987 - \frac{2972}{t + 350} \qquad (6)$$

where P = pressure, p.s.i.a.

t = degrees °F.

The Dow Chemical Company[5] obtained the vapor pressure of this material up to the atmospheric boiling point and, on the basis of limited data, extrapolated to 500°F. by plotting the logarithm of the pressure versus $\frac{1}{T°R}$. The extrapolation is not in agreement with the data obtained in this investigation, being 10% lower at 500°F. However, the agreement of the Equation (6) with vapor pressure data obtained in a study of the stability of Dowtherm E[5] is excellent.

The accuracy of the vapor pressure data is estimated as follows: Temperatures are accurate to plus or minus 0.5°F. Accuracy of the pressure reading in the range of 0 to 50 p.s.i. is plus or minus 0.2 p.s.i., in the range of 50 to 200 p.s.i. is plus or minus 1.0 p.s.i. and in the range of 200 to 1000 p.s.i. is plus or minus 2 p.s.i.

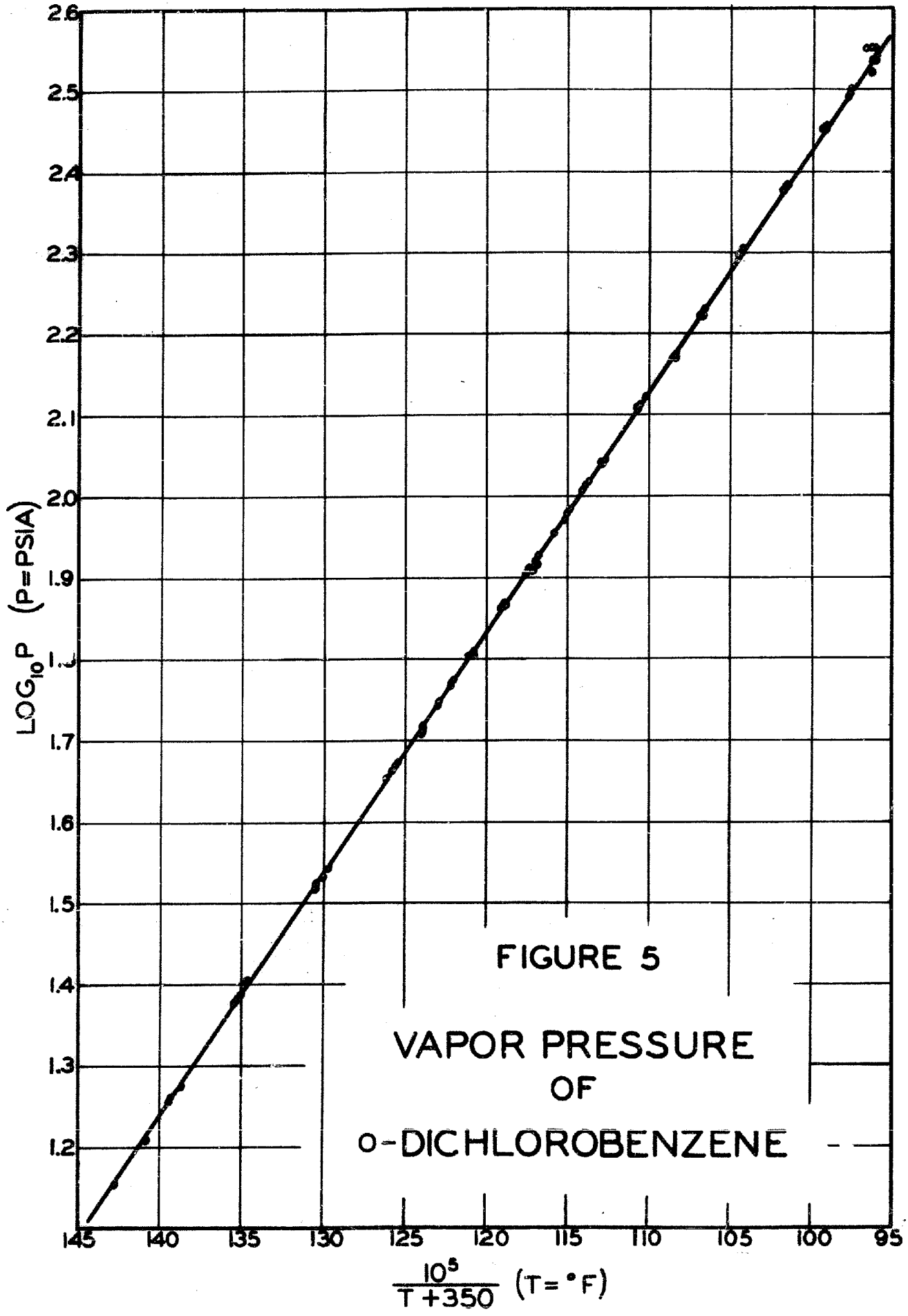


FIGURE 5

VAPOR PRESSURE
OF
o-DICHLOROBENZENE

Joule-Thompson Expansion Data.

The Joule-Thompson expansion data are presented in Tables IX and X of the Appendix and Figure No. 6. The open circles indicate data obtained by a throttle valve orifice, and the filled circles indicate data obtained by a progressive throttle. The vapor-pressure curve of Figure 6 is taken from the smoothed data of Figure 5 which was obtained by the valve-type orifice. As can be seen from Figure 6, this curve fits the progressive-orifice data extremely well. Also, it can be seen that the agreement of the isoenthalpic data for the liquid and vapor phases, which were obtained by both the valve-orifice and the progressive-throttling orifice is good.

Within the accuracy of the data, the isoenthalpic lines are straight when plotted on pressure and temperature coordinates. The data were smoothed and all extrapolations and interpolations were made from a plot of these Joule-Thompson coefficients against the temperature at which the isoenthalps crossed the 35 p.s.i.a. ordinate. Agreement between individual points and the smoothed curves is shown by the isoenthalpic lines in Figure 6 which were calculated from the smoothed slopes. Enthalpy versus temperature has been plotted in Figure No. 7. This figure shows the lines of saturated liquid, saturated vapor, the two-phase regions,

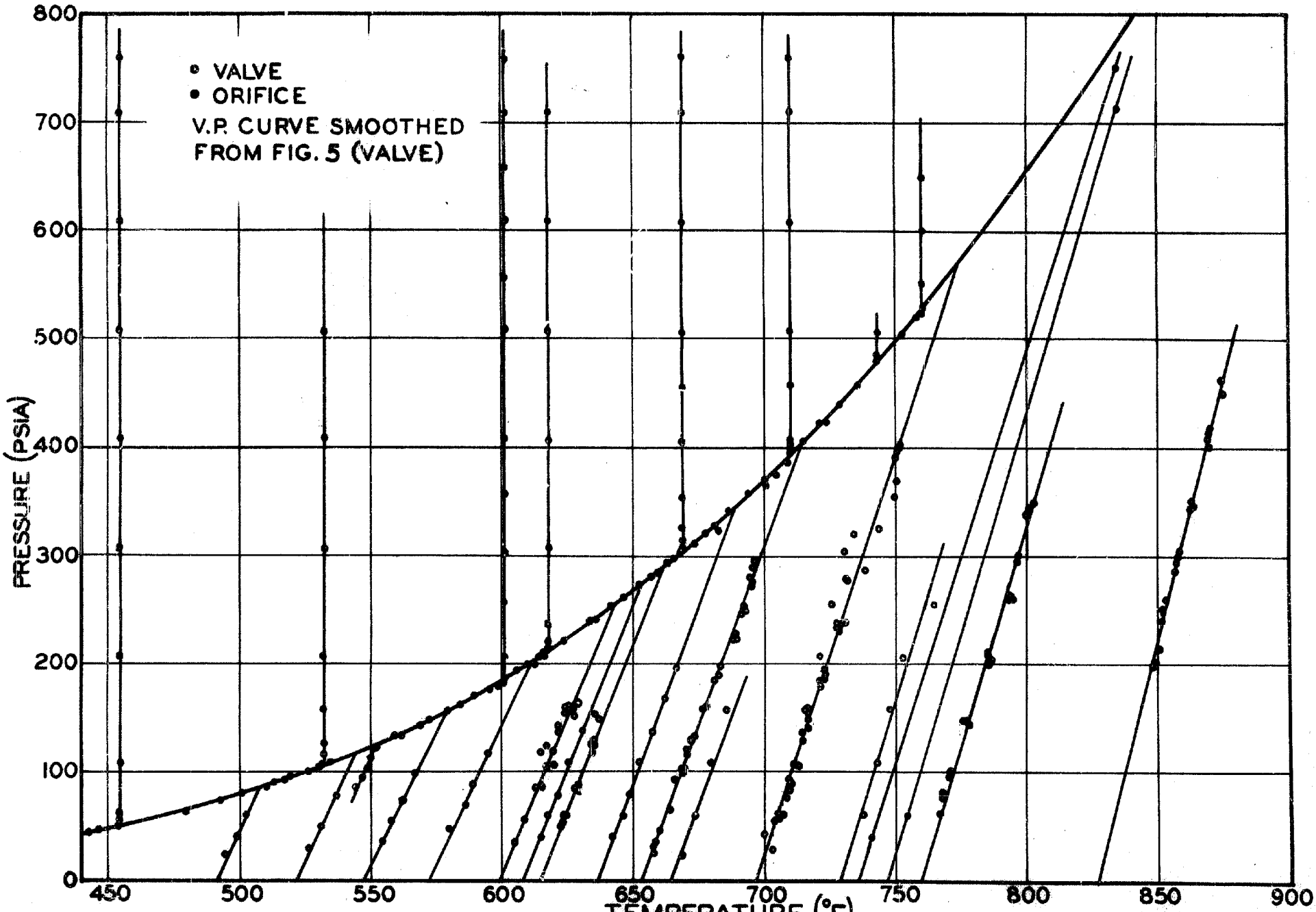


FIGURE 6 JOULE-THOMPSON EXPANSIONS OF O-DICHLOROBENZENE

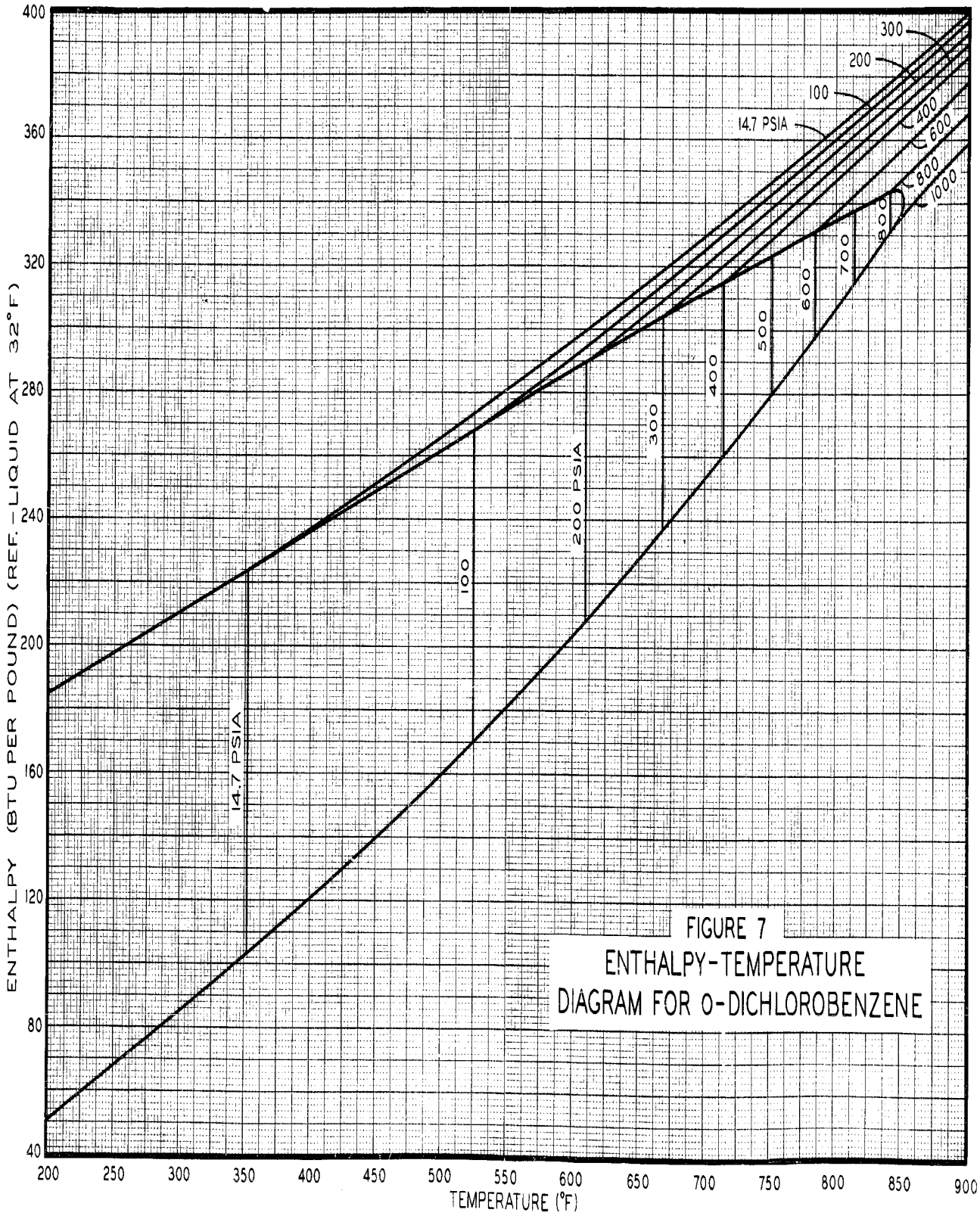


FIGURE 7
ENTHALPY-TEMPERATURE
DIAGRAM FOR O-DICHLOROBENZENE

and the single-phase regions. The enthalpies of the saturated liquid in this figure were obtained from Figure No. 3. In order to close the loop between the saturated liquid and saturated vapor, values for the critical temperature and pressure are required. The methods of obtaining these values will be discussed later. The pressure versus temperature for constant enthalpy lines has been plotted in Figure No. 8. From this plot, a plot of log pressure versus enthalpy for constant temperatures was made as shown in Figure No. 9. This plot is useful in extending the data to the pressures higher than those covered in the investigations. The saturated liquid, the saturated vapor, the two-phase regions, and the single-phase regions are shown.

The maximum deviation of the Joule-Thompson data in the experimental range is 3 Btu. The maximum possible deviation for saturated vapor is 5 Btu per pound, while the average deviation is 2 Btu per pound.

Determination of the Critical Temperature and Pressure.

The corrosion of the steel tubes in the radiant-tube furnace beyond 750°F. was very great, and it was difficult to run isoenthalps at high pressures and temperatures. This necessitated estimation of the critical temperature by a

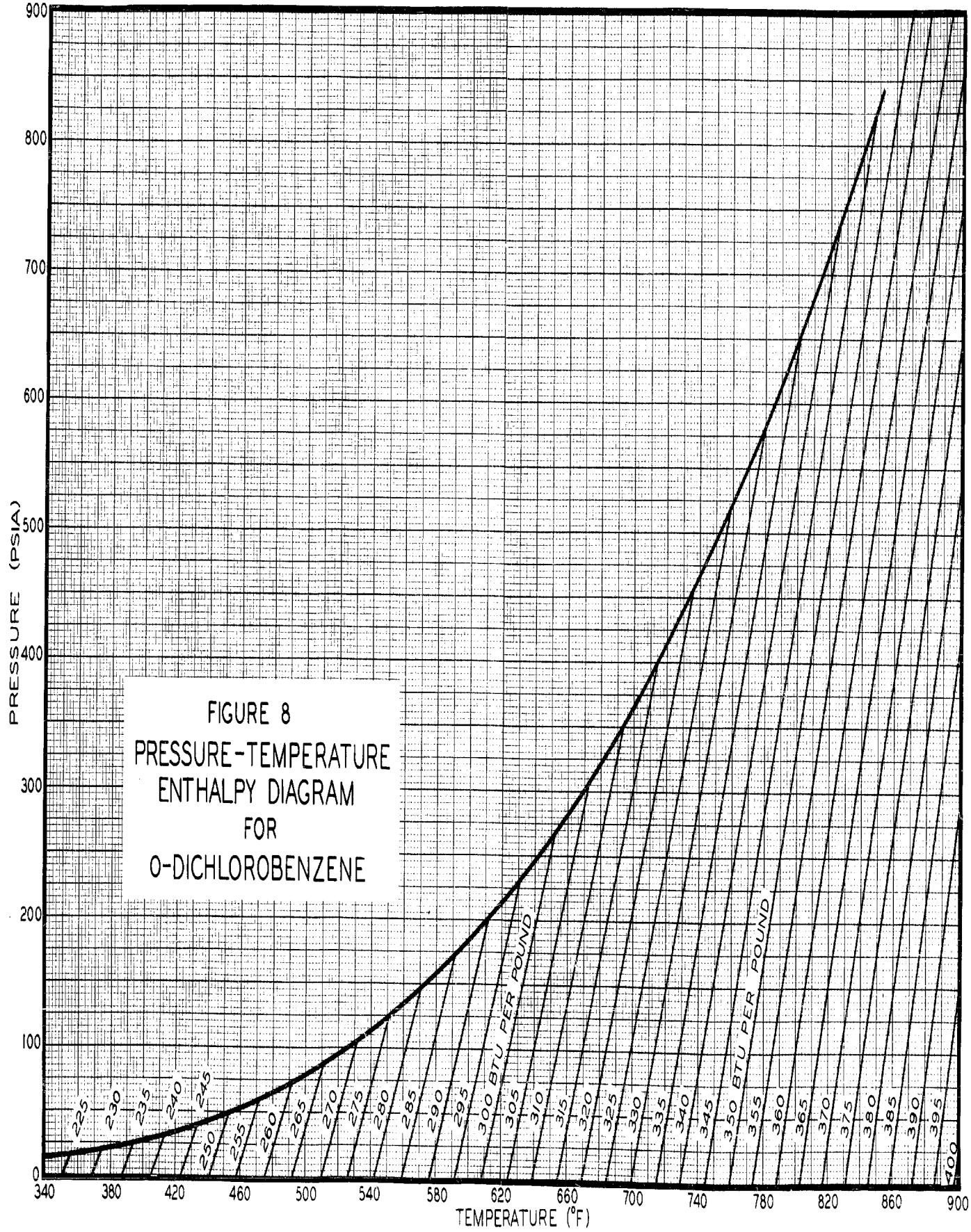


FIGURE 8
PRESSURE-TEMPERATURE
ENTHALPY DIAGRAM
FOR
O-DICHLOROBENZENE

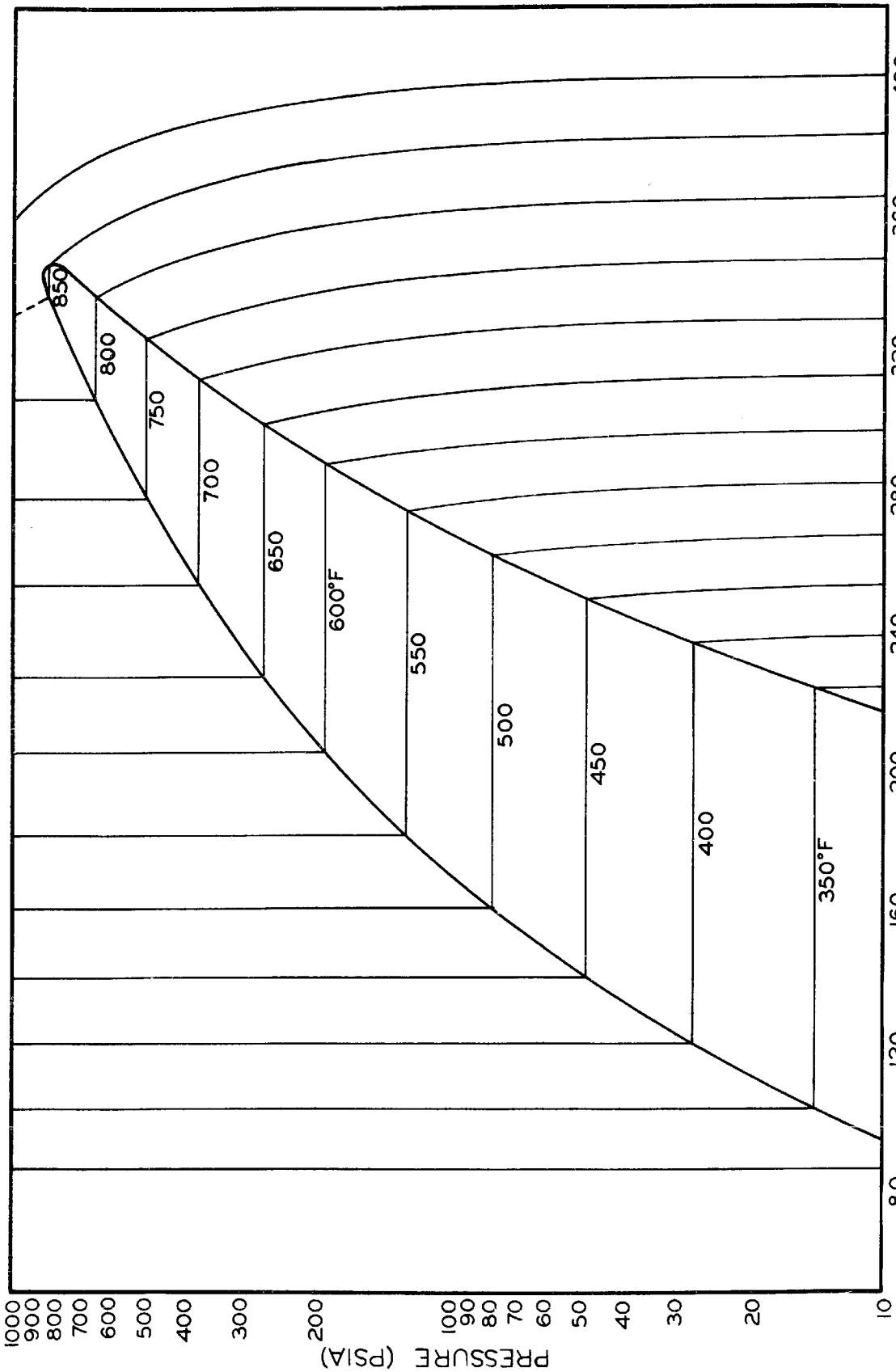


FIGURE 9 PRESSURE-ENTHALPY DIAGRAM FOR O-DICHLOROBENZENE

different method. Even if it were possible to obtain the data at high temperatures and pressures, it would be difficult to run an isoenthalp which would pass exactly through the critical. In the Joule-Thompson method for determining the critical, the critical conditions either have to be known or they have to be determined indirectly. The critical temperature was estimated by plotting the latent heat of vaporization versus temperature as in Figure No. 10. At the critical temperature and pressure the latent heat of vaporization is zero, and the slope of the latent heat of vaporization versus temperature should be infinite. The plot of the latent heat of vaporization versus temperature was extended to latent heat equal to zero. The temperature at which the curve intersects the latent heat of vaporization equal to zero is the estimated critical temperature. The data obtained experimentally for latent heat is extended to within 15°F. of the estimated critical and the maximum error for the critical temperature obtained by extrapolation is probably less than 10°F. In order to make the extrapolation easier and more accurate, the latent heat of vaporization was also plotted against $\frac{1}{T^{\circ} R}$. From this extrapolation, the value of the critical temperature obtained was 852°F. The critical pressure obtained from the extrapolation of the vapor pressure versus temperature curve up to the critical temperature was 845 p.s.i.a.

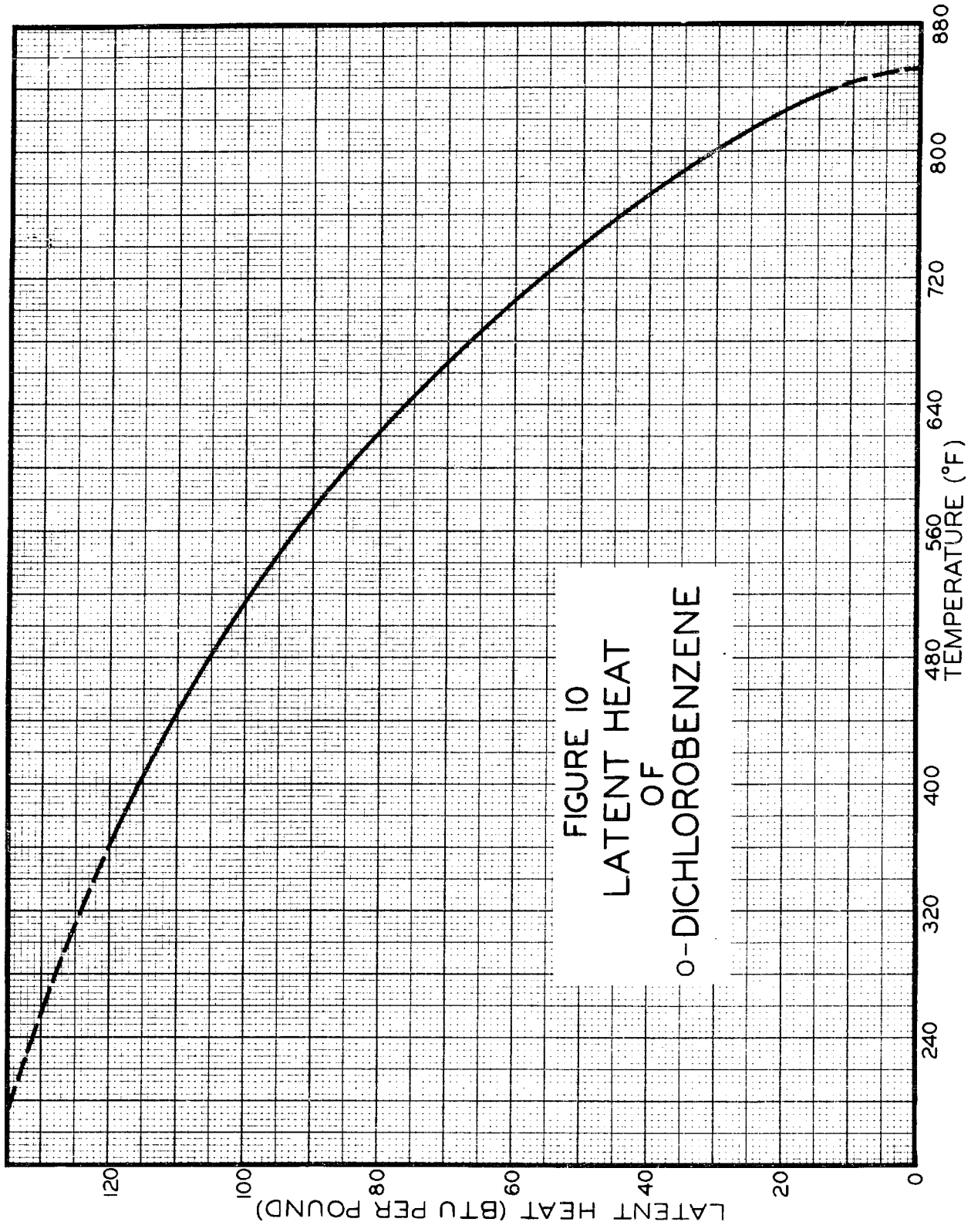


FIGURE 10
LATENT HEAT
OF
O-DICHLOROBENZENE

The value for the critical temperature as estimated in this work is compared with the various values as predicted by the equations of Meissner and Redding[17], Watson[24], and Herzog[10] and Meissner[16] in the following list:

	Critical Temp., °F.	Percent Deviation in Abs. Temp.
Estimation from experimental data	852	0
Watson	808	3.7
Meissner and Redding	804	4.0
Herzog	757	7.9
Meissner	760	7.6

These deviations are within the range of accuracy claimed for the empirical equations indicated.

The effect of pressure on the enthalpy of orthodichlorobenzene at constant reduced temperatures is shown in Figure No. 11. The solid lines are those obtained in this investigation, and the dotted lines are drawn from steam-table data[11]. The agreement between the two sets of data at T_r equal to 0.7 is fair. However, at other reduced temperatures, the values obtained in this investigation are higher than those based on steam-table data.

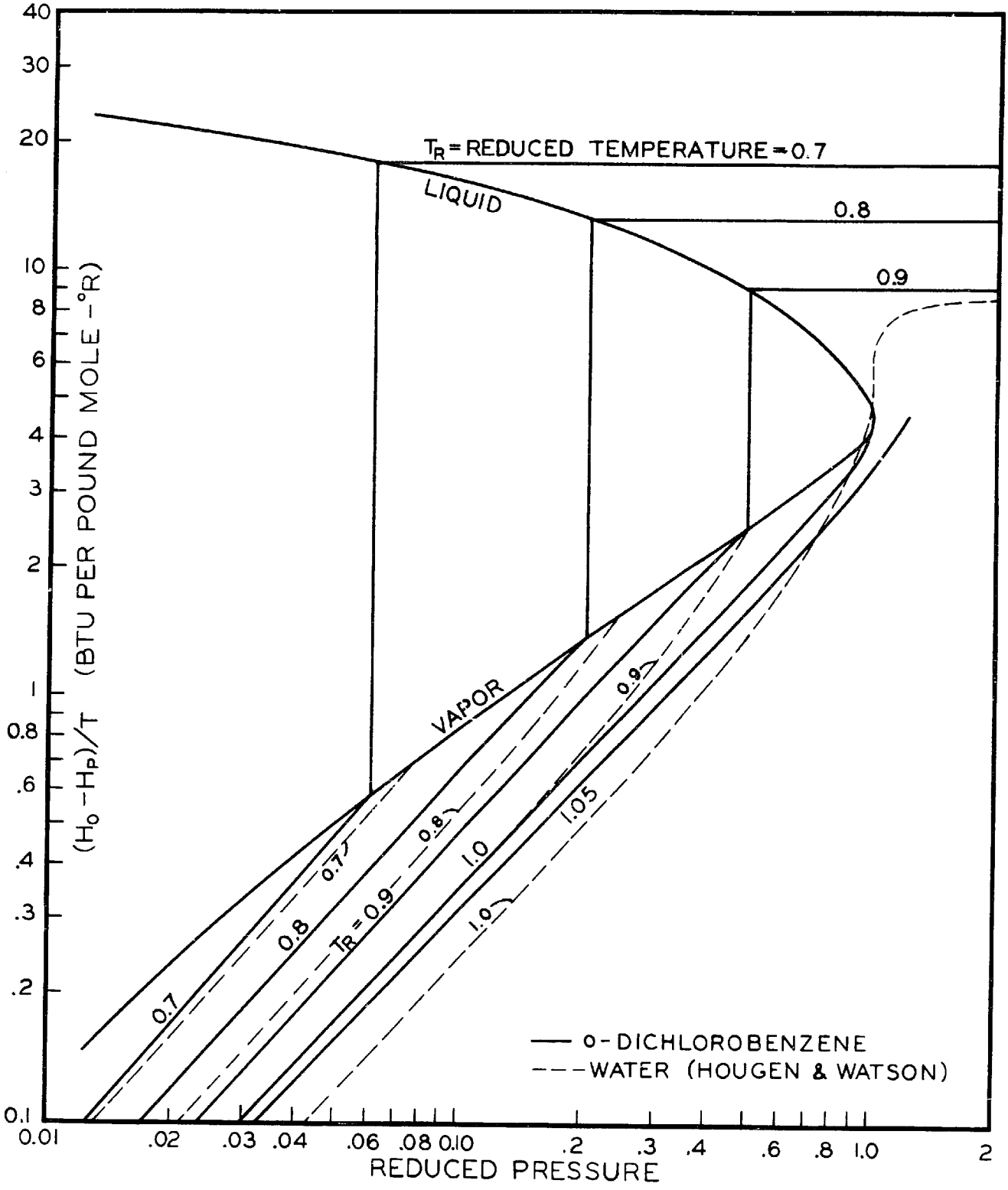


FIGURE 11

REDUCED ENTHALPY DIFFERENCE CHART FOR o-DICHLOROBENZENE

CONCLUSIONS

1. The enthalpy referred to pure liquid at 32°F. and 1 atmosphere and specific heat of liquid orthodichlorobenzene up to 800°F. is represented by the equations.

$$H_L = -9.29 + 0.288\ t + 0.49 \times 10^{-4} t^2 + 0.106 \times 10^{-6} t^3$$

$$(C_p)_L = 0.288 + 0.98 \times 10^{-4} t + 0.324 \times 10^{-6} t^2$$

2. The effect of pressure upon the enthalpy of liquid orthodichlorobenzene is negligible.

3. The enthalpy referred to pure liquid at 32°F. and 1 atmosphere and specific heat of orthodichlorobenzene vapor at one atmospheric pressure are represented by the equations

$$H_v = 139.5 + 0.2092\ t + 8.891 \times 10^{-5}\ t^2$$

$$(C_p)_v = 0.2092 + 1.778 \times 10^{-4}\ t$$

4. The vapor pressure in psia has been determined and may be represented by the equation

$$\text{Log}_{10} P = 5.3987 - \frac{2972}{t + 350°F.}$$

5. Charts of pressure versus enthalpy at constant temperatures, and enthalpy versus temperature at constant pressures have been prepared. These charts show the lines of saturated liquid, saturated vapor, the two-phase region, and the single-phase regions.

6. The critical temperature of orthodichlorobenzene was estimated to be 852°F., and the critical pressure is estimated 845 p.s.i.a.

7. The effect of pressure on the enthalpy of orthodichlorobenzene at constant reduced temperature was determined and compared with the corresponding values for water. The values at reduced conditions obtained in this investigation are slightly higher than those derived from steam table data.

8. A valve-type throttling orifice has been tested and was found to reproduce the corresponding data obtained by the progressive-throttling type orifice. It also has been found to possess many experimental advantages over the latter.

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A P P E N D I X

TABLE NO. I

TRUE-BOILING-POINT DISTILLATION ANALYSIS
OF ORTHODICHLOROBENZENE

Volume Percent Recovered C.C.	Temperature °C.	Volume Percent Recovered C.C.	Temperature °C.
0.9	175.0	46.6	176.2
2.3	175.0	47.6	176.5
3.4	175.0	49.6	176.5
4.6	175.0	51.6	176.5
5.8	175.0	53.6	176.5
6.5	175.0	55.6	176.5
8.5	175.0	57.6	176.5
10.5	175.0	59.6	176.5
12.2	175.0	61.6	176.5
13.4	175.2	63.6	176.5
14.4	175.5	65.6	176.5
15.4	175.5	66.6	176.5
18.4	175.5	67.6	176.5
19.4	175.8	68.6	176.5
21.5	175.8	69.6	176.8
22.3	176.0	71.6	176.8
24.3	176.0	73.6	176.8
26.3	176.0	75.6	176.8
28.3	176.0	77.6	176.8
30.3	176.0	79.6	176.8
32.3	176.0	81.6	176.8
34.3	176.0	83.6	176.8
36.3	176.0	85.6	176.8
38.1	176.0	87.6	176.8
39.0	176.2	88.6	176.9
40.6	176.2	90.6	176.9
41.8	176.2	92.6	176.9
43.8	176.2	94.6	176.9
45.8	176.2	96.1	176.9

Recovery	96.1%
Residue	2.8%
Loss	1.1%

TABLE NO. II

SPECIFIC GRAVITY AND REFRACTIVE INDEX OF TRUE-BOILING FRACTIONS OF ORTHODICHLOROBENZENE

Fraction Volume Percent	Volume Average Boiling Point °C.	Specific Gravity at 69.5°F.	Refractive Index at 71.6°F.
0-100		1.3057	1.5503
0-19.3	175.5	1.3035	1.5498
19.3-80.4	176.5	1.3057	1.5505
80.4-96.1	176.9	1.3065	1.5504
96.1-100		1.3065	1.5504

TABLE NO. III

ENTHALPY OF ORTHODICHLOROBENZENE LIQUID AT 15 PSIA WITH REFERENCE TO LIQUID ORTHODICHLOROBENZENE AT 32°F. AND 1 ATMOSPHERE

Run No.	Orthodichlorobenzene Temperatures Inlet °F. T_3	Orthodichlorobenzene Temperatures Outlet °F. T_4	Cooling Water Temperatures Inlet °F. T_5	Cooling Water Temperatures Outlet °F. T_6	Wt. of orthodichlorobenzene kg	Wt. of Water kg	H* B.t.u/lb.	Enthalpy of Liquid B.t.u./lb. H_L
130	111.73	73.55	62.72	72.25	1.42	1.746	11.70	24.48
131	113.91	73.64	62.41	72.50	1.409	1.693	12.11	24.88
133	155.68	77.98	62.41	80.88	3.244	4.171	23.70	37.75
134	155.16	74.43	62.86	79.78	3.017	4.308	24.20	37.15
135	168.28	68.98	63.30	75.00	1.962	5.256	31.35	42.60
142	91.00	75.87	64.56	77.31	3.389	1.156	4.34	17.69
143	91.00	74.45	64.06	77.30	3.527	1.537	5.76	18.71
144	191.09	92.21	64.48	92.14	3.169	3.500	30.55	48.95
146	191.54	91.32	63.84	92.21	3.136	3.402	30.80	48.85
147	263.86	107.84	64.65	122.55	3.295	2.764	48.60	71.65
148	263.38	107.84	64.65	125.00	3.280	2.628	48.30	71.35
149	324.68	119.90	63.86	148.64	3.502	2.690	65.00	91.85
151	325.50	108.98	64.06	134.55	2.704	2.672	69.60	93.10
152	100.00	72.36	62.18	72.82	2.912	2.212	8.09	20.43
154	220.54	102.50	62.86	97.54	3.682	3.894	36.65	58.15
155	221.37	102.73	62.41	97.98	3.674	3.803	36.80	58.45
156	267.79	107.50	62.41	96.36	3.718	5.572	50.80	73.80
157	268.86	107.46	61.96	96.14	3.692	5.557	51.50	75.80
158	299.77	110.95	61.64	101.59	3.156	4.783	60.51	84.66
159	299.70	110.91	61.96	102.04	3.156	4.780	60.60	84.75
160	331.23	115.63	63.00	110.27	3.195	4.765	70.60	96.15
161	332.27	115.45	63.21	110.59	3.251	4.860	70.80	96.35
163	137.08	84.43	63.30	82.32	3.915	3.339	16.20	32.20
185	272.64	69.86	65.13	89.67	2.186	5.817	65.40	76.90
186	274.42	69.94	65.13	89.55	2.351	6.423	66.80	78.30
189	281.91	80.64	65.86	116.00	2.713	3.500	64.61	79.56
190	317.73	80.77	65.13	125.33	2.663	3.435	77.50	92.50
191	318.64	79.73	65.13	122.51	2.619	3.411	74.80	89.35
192	343.98	79.50	65.13	132.50	2.502	3.243	87.40	101.90
193	343.07	76.00	65.13	123.50	2.258	3.393	86.10	99.55

* $H = \frac{(\text{wt. of water})(H_6 - H_5)}{(\text{wt. of orthodichlorobenzene})}$

where H_6 and H_5 are the enthalpies of the water corresponding to outlet and inlet temperatures.

TABLE NO. IV

ENTHALPY OF LIQUID ORTHODICHLOROBENZENE AT 215 PSIA WITH REFERENCE TO LIQUID ORTHODICHLOROBENZENE AT 32°F. AND 1 ATMOSPHERE

Run No.	Orthodichlorobenzene Temperatures Inlet °F. T_3	Orthodichlorobenzene Temperatures Outlet °F. T_4	Cooling Water Temperatures Inlet °F. T_5	Cooling Water Temperatures Outlet °F. T_6	Wt. of orthodichlorobenzene kg	Wt. of Water kg	H* B.t.u/lb	Enthalpy of Liquid B.t.u./lb. H_L
164	139.00	79.11	61.77	75.52	3.300	4.175	17.24	31.03
165	136.72	78.41	62.16	75.65	3.347	4.140	16.69	30.18
166	104.24	72.71	62.16	69.17	3.323	4.113	8.68	20.58
167	106.07	72.71	61.77	69.52	3.293	4.135	9.73	21.63
168	372.78	102.53	61.03	104.81	3.277	6.744	90.10	110.92
169	374.62	104.41	61.20	106.25	3.370	6.744	90.15	111.53
170	423.13	115.79	60.89	120.83	3.256	5.945	109.44	134.25
171	422.65	114.70	60.67	116.36	3.230	6.056	104.4	128.90
172	468.49	121.75	60.46	123.46	3.583	6.809	119.72	146.87
173	467.83	119.56	60.54	121.92	3.482	6.815	120.10	146.08
174	511.83	120.00	60.68	121.27	3.024	6.856	138.4	164.38
175	511.97	118.82	60.76	120.35	2.941	6.801	138.8	164.78
176	551.14	121.75	60.89	126.7	2.914	6.836	154.40	181.70
177	553.81	122.19	60.89	126.13	2.891	6.825	154.00	181.80
178	598.10	117.81	60.89	139.70	3.069	6.823	175.20	203.64
179	603.33	123.5	61.11	139.98	3.028	6.831	177.80	204.98
180	676.79	113.87	61.46	161.02	2.436	6.926	283.1	307.35**
181	677.54	104.89	61.73	152.26	2.130	6.826	290.1	311.62**

* $H = \frac{(\text{wt. of water})(H_6 - H_5)}{(\text{wt. of orthodichlorobenzene})}$

where

H_6 and H_5 are the enthalpies of the water corresponding to the outlet and inlet temperatures.

**Enthalpy of vapor.

TABLE NO. V

ENTHALPY OF LIQUID ORTHODICHLOROBENZENE AT 662 PSIA WITH REFERENCE TO LIQUID ORTHODICHLOROBENZENE AT 32°F. AND 1 ATMOSPHERE

Run No.	Orthodichlorobenzene Temperatures Inlet °F. T_3	Orthodichlorobenzene Temperatures Outlet °F. T_4	Cooling Water Temperatures Inlet °F. T_5	Cooling Water Temperatures Outlet °F. T_6	Wt. of orthodichlorobenzene kg	Wt. of Water kg	H* B.t.u/lb	Enthalpy of Liquid B.t.u/lb. H_T
217	740.14	72.72	59.36	129.85	1.758	6.309	252.97	264.82
218	736.86	77.40	59.14	129.63	1.737	6.257	253.92	266.21
220	729.63	69.56	60.02	114.03	1.247	5.855	253.5	264.46
222	764.44	76.65	60.02	141.01	1.887	5.878	282.19	295.25
223	764.00	77.09	59.58	132.88	1.532	5.910	282.96	296.15
224	687.82	70.55	60.46	114.74	1.227	5.275	233.35	244.60
225	681.04	68.12	60.02	108.18	1.230	6.095	238.65	248.19
226	679.29	76.00	60.02	126.08	1.535	5.455	234.76	247.63
227	680.74	69.13	60.02	109.49	1.235	5.913	237.00	247.94
228	644.92	86.72	60.02	146.05	2.406	5.840	208.82	224.88
229	647.54	76.48	60.02	126.08	1.743	5.691	215.69	228.70
231	603.77	85.41	60.89	144.21	2.229	5.150	192.25	207.92
232	607.27	78.41	61.77	128.54	1.746	5.164	197.48	211.07
233	579.06	75.02	61.68	108.26	1.308	5.200	185.18	197.76
234	571.37	75.78	61.55	114.00	1.489	5.206	183.38	196.19
235	536.35	80.11	61.33	119.00	1.865	5.193	160.58	174.67
237	529.34	78.84	61.55	123.28	2.391	6.153	158.87	172.58
238	529.34	81.82	61.55	128.00	2.563	6.144	159.24	173.84
239	490.38	82.65	61.55	126.21	2.783	6.146	142.79	156.64
240	489.72	77.77	61.55	124.37	2.678	6.136	143.94	157.34
242	412.45	77.53	61.77	111.33	2.699	6.082	111.67	124.99
244	406.76	77.10	60.89	111.55	2.749	5.984	110.23	123.43
245	292.05	77.53	61.33	104.28	2.997	4.897	70.18	83.50
246	289.43	81.91	61.55	112.16	3.769	4.892	65.69	80.32
247	289.00	80.60	61.33	112.07	3.512	4.753	68.67	83.81

* $$H = \frac{(\text{wt. of water})(H_6 - H_5)}{(\text{wt. of orthodichlorobenzene})}$$

H_6 and H_5 are the enthalpies of the water corresponding to the outlet and inlet temperatures.

TABLE NO. VI

ENTHALPY OF VAPOR ORTHODICHLOROBENZENE AT 35 PSIA WITH REFERENCE TO LIQUID AT 32°F. AND 1 ATMOSPHERE

Run No.	Orthodichlorobenzene Temperatures Inlet °F. T_3	Orthodichlorobenzene Temperatures Outlet °F. T_4	Cooling Water Temperatures Inlet °F. T_5	Cooling Water Temperatures Outlet °F. T_6	Wt. of orthodichlorobenzene kg	Wt. of Water kg	H* B.t.u/lb	Enthalpy of Vapor B.t.u/lb. H_v
249	769.03	64.62	57.392	134.48	1.210	5.391	343.0	352.50
250	764.88	62.21	57.392	127.27	1.094	5.357	341.99	350.80
251	709.28	66.15	58.71	144.90	1.699	6.373	322.50	332.00
252	706.00	63.96	58.71	127.7	1.446	6.830	319.82	329.13
253	705.30	65.71	58.71	132.69	1.593	6.934	321.90	331.73
254	652.14	65.71	58.27	128.32	1.606	6.908	301.16	310.98
255	650.17	64.83	58.71	125.69	1.543	6.993	303.44	312.98
256	582.10	65.10	58.71	127.10	1.676	6.919	282.18	291.82
259	580.56	67.02	58.71	123.46	1.627	7.013	278.97	289.23
260	495.61	68.55	58.71	126.08	1.643	6.150	252.09	262.72
261	497.38	66.02	58.71	135.45	1.867	6.223	255.69	265.61
262	499.57	68.77	58.71	124.81	1.619	6.257	255.37	266.19
264	464.00	71.05	59.14	131.99	1.897	6.313	242.33	253.75
265	434.34	71.18	59.14	135.34	2.045	6.271	234.0	245.50
266	449.66	67.68	59.14	131.39	1.902	6.298	239.50	250.90

* $H = \dfrac{(\text{wt. of water})(H_6 - H_5)}{(\text{wt. of orthodichlorobenzene})}$

H_6 and H_5 are the enthalpies of water corresponding to the outlet and inlet temperatures.

TABLE NO. VII

ENTHALPY OF LIQUID ORTHODICHLOROBENZENE FROM COMPLETE JOULE-THOMPSON EXPANSIONS OF LIQUID TO SUPERHEATED VAPOR BASED ON ENTHALPY DATA OF SUPERHEATED VAPOR AT 35 POUNDS PER SQUARE INCH ABSOLUTE

Inlet to Throttle Liquid		Outlet from Throttle Vapor		Temperature T_2' at 35 P.S.I.A.	Enthalpy of Vapor
T_1 °F	P_1 P.S.I.A.	T_2 °F	P.S.I.A.	°F.	B.t.u/lb.
		Data from Run No. 108			
740	797	524.1	36.5	524	272.7
740	797	527.2	44.0	525	273.3
743	797	542.0	77.2	534	276.0
743.5	797	545.5	86.8	533.5	276.4
743.5	797	547.7	95.8	536.	276.7
743.5	797	549.5	105.4	536	276.7
743.0	797	551.2	115.0	535	276.2
		Data from Run No. 292			
755	602	535.0	47	532	275.3
755	606	534.8	48	532	275.3
755	599	533.9	45	531.5	275.0
754	600	534.2	59	529.5	274.7
754	610	534.7	60	530.0	274.8
755.5	605	552.0	114	536.5	276.7
754.5	600	551.5	115	536.0	276.4
		Data from Run No. 293			
823	836	677.2	24	678.5	321.8
822	831	667.8	24	669.5	318.5
828	787	688.7	25	690.0	325.6
824.5	812	687.8	21	689.0	325.3

Note: Liquid at T_1 and P_1 are expanded adiabatically to vapor at T_2 and P_2. T_2 values are then extrapolated to P_2 = 35 pounds per square inch absolute using the experimental Joule-Thompson superheated vapor slopes to permit reading of vapor enthalpy from Figure No. 4. The liquid enthalpy at P_1 and T_1 is then equal to the vapor enthalpy at T_2' and 35 pounds per square inch absolute.

TABLE NO. VIII

VAPOR PRESSURE OF ORTHODICHLOROBENZENE BY JOULE-THOMPSON EXPANSIONS INTO THE LIQUID-VAPOR REGION

T_1 and P_1 are inlet temperature in °F. and pressure in PSIA to the throttle.
T_2 and P_2 are outlet temperature in °F. and pressure in PSIA from the throttle.

T_1	P_1	T_2	P_2
Run No. 288			
500	200	360.6	16.2
		360.8	16.2
		350.8	14.3
		416.9	33.1
		419.9	34.1
		418.8	34.0
		421.1	35.0
		416.2	33.4
Run No. 289			
600	450	371.3	19.0
		368.1	18.3
		368.0	18.3
		389.1	23.8
		388.8	23.8
		389.9	24.2
		390.3	24.5
		393.1	25.3
		443.0	45.1
		446.2	46.4
		446.8	46.6
		447.5	47.0
		447.9	47.1
		457.0	52.1
		457.0	51.8
		457.0	52.0
		457.2	52.1
		462.5	55.0
		462.8	55.1
		462.8	55.1
		469.5	59.0
		469.7	59.1
		477.7	64.3

T_1	P_1	T_2	P_2
Run No. 289 (continued)			
600	450	477.8	64.1
		478.1	64.3
		476.6	63.1
		476.3	63.2
		490.3	73.2
		490.9	73.2
		491.5	73.7
		491.4	73.7
		501.8	81.6
		504.0	83.1
		504.8	82.6
		505.6	83.6
		506.1	83.9
		513.2	90.1
		514.2	90.4
		518.5	94.6
		519.3	95.1
		519.8	95.6
		528.3	103.1
Run No. 290			
675	560	447.5	47.0
		462.5	55.0
		491.4	73.7
		502.2	80.2
		502.7	81.2
		503.8	82.8
		528.8	103.6
		529.3	104.6
		536.6	111.1
		537.0	111.6
		537.4	112.1
		554.4	129.3

TABLE NO. VIII (continued)

Run No. 290 (continued)

T_1	P_1	T_2	P_2
675	500	554.7	129.0
		558.2	133.8
		554.9	129.3
		554.7	129.8
		571.8	149.3
		572.9	150.3
		572.6	149.3
		571.6	148.3
		587.4	168.0
		588.3	171.0
		586.7	167.5
		587.4	169.0
		609.1	200.0
		609.5	200.1
		609.2	200.0

Run No. 291

T_1	P_1	T_2	P_2
750	650	588.3	171.0
		609.1	200.0
		634.9	240.0
		635.0	240.0
		634.3	240.0
		634.5	240.0
		657.5	283.0
		658.7	282.5
		659.7	284.0
		674.5	317.5
		675.2	317.0
		673.4	313.0
		672.8	313.0
		687.5	332.0
		689.8	347.0
		691.1	352.0
		691.4	349.0
		690.3	357.0
		683.3	329.0
		689.9	356.0
		688.9	347.0

TABLE NO. IX

JOULE-THOMPSON EXPANSION DATA OF ORTHO-DICHLOROBENZENE FOR LIQUID AND LIQUID-VAPOR REGION

T_1 and P_1 are inlet temperature in °F. and pressure in PSIA to the throttle.
T_2 and P_2 are outlet temperature in °F. and pressure in PSIA from the throttle.

T_1	P_1	T_2	P_2
Run No. 101			
426.36	815	373.77	21.0
		386.91	26.4
		401.4	29.6
		405.95	31.2
		410.44	34.2
		420.4	39.0
		426.36	44.0
		426.36	65.0
		426.36	107.0
		426.36	207.0
		426.36	307.0
		426.36	407.0
		426.36	508.0
		426.36	608.0
		426.36	709.0
		426.36	760.0
Run No. 102			
455.2	815	377.86	23.2
		402.81	28.4
		421.81	36.3
		440.0	44.0
		443.0	45.0
		447.0	46.0
		455.0	52.0
		455.0	56.7
		455.0	63.55
		455.0	108.0
		455.0	207.0
		455.0	307.0
		455.0	407.0
		455.0	508.0
		455.0	608.0
		455.0	709.0
		455.0	760.0
Run No. 103			
532.0	815	510.79	87.2
		514.10	90.0
		518.00	94.0
		521.63	96.8
		527.60	100.0
		531.10	105.0
		532.0	109.0
		532.0	118.0
		532.0	127.0
		532.0	160.0
		532.0	207.0
		532.0	307.0
		532.0	408.0
		532.0	507.0
Run No. 104			
601.0	815	480.1	65.0
		508.2	85.0
		527.1	100.0
		552.0	125.0
		573.0	150.0
		596.1	178.0
		599.2	180.0
		601.0	187.0
		601.0	207.0
		601.0	256.0
		601.0	307.0
		601.0	357.0
		601.0	409.0
		601.0	508.0
		601.0	610.0
		601.0	659.0
		601.0	708.0
		601.0	758.0

TABLE NO. IX (continued)

T_1	P_1	T_2	P_2
Run No. 105			
618.08	815	494.20	75.0
		510.10	87.2
		521.02	96.8
		535.0	110.0
		562.3	135.0
		574.2	150.0
		592.46	175.0
		603.0	190.0
		606.0	194.9
		610.2	200.0
		615.0	206.0
		616.0	210.0
		618.08	215.0
		618.08	220.0
		618.08	307.0
		618.08	407.0
		618.08	507.0
		618.08	609.0
		618.08	710.0
Run No. 106			
672.3	815	502.0	82.0
		532.0	110.2
		560.0	135.0
		574.0	150.0
		589.8	170.0
		618.2	210.0
		642.1	253.8
		653.5	274.0
		658.3	283.8
		663.0	293.6
		667.2	300.0
		669.1	303.8
		669.1	315.0
		669.1	325.0
		669.1	355.0
		669.1	405.0
		669.1	455.0
		669.1	505.0
		669.1	605.0
		669.1	709.0
		669.1	760.0

T_1	P_1	T_2	P_2
Run No. 107			
710.45	815	521.61	97.8
		551.80	125.0
		569.41	145.0
		585.67	165.0
		600.27	185.0
		613.90	204.7
		623.95	224.2
		635.25	243.8
		646.50	263.8
		659.41	283.8
		674.83	313.8
		683.58	325.0
		687.99	344.0
		695.67	359.0
		702.0	365.0
		705.0	375.0
		708.7	385.0
		710.45	395.0
		710.45	402.0
		710.45	413.0
		710.45	458.0
		710.45	507.0
		710.45	608.0
		710.45	710.0
		710.45	760.0
Run No. 108			
740	797	524.1*	36.5*
740	797	527.2*	44*
743	797	542.0*	77.2*
743.5	797	545.5*	86.8*
743.5	797	547.7*	95.8*
743.5	797	549.5*	105.4*
743.5	797	551.2*	115.0*
743.5	797	653.45	274.0
743.5	797	678.9	324
743.5	797	700.16	374
		715.75	408
		721.80	424
		729.50	444
		736.2	459
		743.5	487
		743.5	508

*Superheated vapor region.

TABLE NO. IX (continued)

T_1	P_1	T_2	P_2
Run No. 109			
761.0	815	616.0	210.0
		634.2	240.0
		654.0	275.0
		682.1	330.0
		700.0	370.0
		722.2	425.0
		744.0	480.0
		761.0	526.0
		761.0	552.0
		761.0	601.0
		761.0	650.0

TABLE NO. X

JOULE-THOMPSON EXPANSION DATA OF SUPERHEATED VAPOR OF ORTHODICHLOROBENZENE

T_1 and P_1 are the inlet temperature in °F. and pressure in PSIA to the throttle.
T_2 and P_2 are outlet temperature in °F. and pressure in PSIA from the throttle.

T_1	P_1	T_2	P_2
Run No. 271			
636.1	158	615.5	41
		617.7	61
		621.6	80
		624.7	109
		631.3	139
		635.9	154
Run No. 272			
668.0	206	642.4	41
		646.4	61
		649.0	80
		652.3	109
		657.7	139
		662.1	168
		666.5	197
Run No. 273			
597.3	130	580.2	48
		586.2	70
		589.2	89
		594.5	119
Run No. 274			
570.2	111	554.6	37
		558.1	55
		562.5	75
		567.6	99
Run No. 275			
538.1	83	526.6	31
		531.9	51
		537.3	79
Run No. 276			
502.1	61	494.2	26
		498.8	41
		502.1	61
Run No. 277			
697.5	225	668.9	23
		673.7	61
		679.8	109
		684.9	158
Run No. 278			
769.5	321	738.5	61
		743.3	109
		746.8	158
		752.5	207
		764.7	256
Run No. 279			
735.0	331	703.7	29
		705.9	61
		710.9	109
		715.7	158
		720.8	207
		724.9	256
		731.0	305
Run No. 280			
637.8	152	623.6	-51.9
		622.9	51.6
		621.5	50.4
		624.6	60.8
		623.1	61.4
		624.1	62.4
		623.0	54.4
		628.4	84.4

TABLE NO. X (continued)

Run No. 280 (continued)

T_1	P_1	T_2	P_2
		628.3	85.9
		626.7	85.4
		627.4	87.4
		634.4	119.9
		634.3	119.9
		634.0	119.4
		634.0	125.0
		634.9	124.0
		634.8	131.0
		637.3	150.0

Run No. 281

T_1	P_1	T_2	P_2
632.0	210	604.8	35
		606.1	35
		610.9	58
		608.6	56
		613.0	84
		615.5	87
		620.0	108
		619.6	108
		617.6	105
		619.7	120
		616.9	126
		615.4	119
		621.5	144
		621.7	138
		620.8	144
		623.9	160
		628.1	152
		625.4	162
		627.3	160
		629.2	165
		624.3	156

Run No. 282

T_1	P_1	T_2	P_2
696.0	305	657.8	26
		657.8	26
		657.3	27
		657.6	29
		658.1	35
		659.7	44

Run No. 282 (continued)

T_1	P_1	T_2	P_2
		659.7	43
		663.3	66
		664.2	66
		665.7	94
		670.1	120
		669.5	104
		667.5	103
		668.3	101
		671.8	132
		672.9	131
		676.2	158
		677.6	162
		677.7	161
		681.0	186
		683.0	192
		682.6	194
		687.9	223
		689.2	228
		689.3	222
		691.4	249
		691.3	249
		692.1	250
		695.0	277
		695.0	276
		695.2	291
		695.3	292
		696.3	295
		695.7	296

Run No. 283

T_1	P_1	T_2	P_2
747.0	405	699.9	43
		699.9	43
		708.8	90
		709.4	94
		714.5	137
		714.9	130
		722.0	186
		721.6	179
		730.3	234
		731.7	238
		738.5	287
		738.7	287

TABLE NO. X (continued)

Run No. 283 (continued)

T_1	P_1	T_2	P_2
		743.6	326
		744.2	327
		750.4	357
		750.7	369
		751.0	397
		750.9	396

Run No. 284

T_1	P_1	T_2	P_2
748.0	410	705.0	56
		706.4	58
		707.4	60
		713.5	107
		710.5	88
		708.7	77
		710.1	92
		716.5	185
		716.7	149
		716.8	143
		722.4	194
		723.0	193
		723.3	185
		726.9	239
		727.8	235
		727.9	232
		731.2	279
		731.7	279
		732.0	280
		734.1	322

Run No. 285

T_1	P_1	T_2	P_2
798.0	400	769.9	98
		770.4	99
		771.0	100
		775.9	148
		776.7	148
		777.6	146
		778.0	145
		784.7	211
		785.9	202
		787.7	204
		793.8	262

Run No. 285 (continued)

T_1	P_1	T_2	P_2
		794.2	260
		793.9	259
		796.8	301
		796.7	303
		796.3	298
		800.0	341
		801.6	347
		802.3	351
		766.6	63
		767.4	76
		768.1	81

Run No. 286

T_1	P_1	T_2	P_2
863.0	500	848.2	199
		848.9	200
		850.7	214
		853.2	260
		851.7	241
		852.1	251
		857.3	295
		857.0	287
		858.5	306
		863.0	350
		862.6	343
		863.4	345
		869.7	410
		869.9	403
		870.0	419
		870.0	404
		874.5	463
		874.5	453
		875.1	453

Run No. 287

T_1	P_1	T_2	P_2
835.7	714	755.5	59
834.9	752	741.5	39

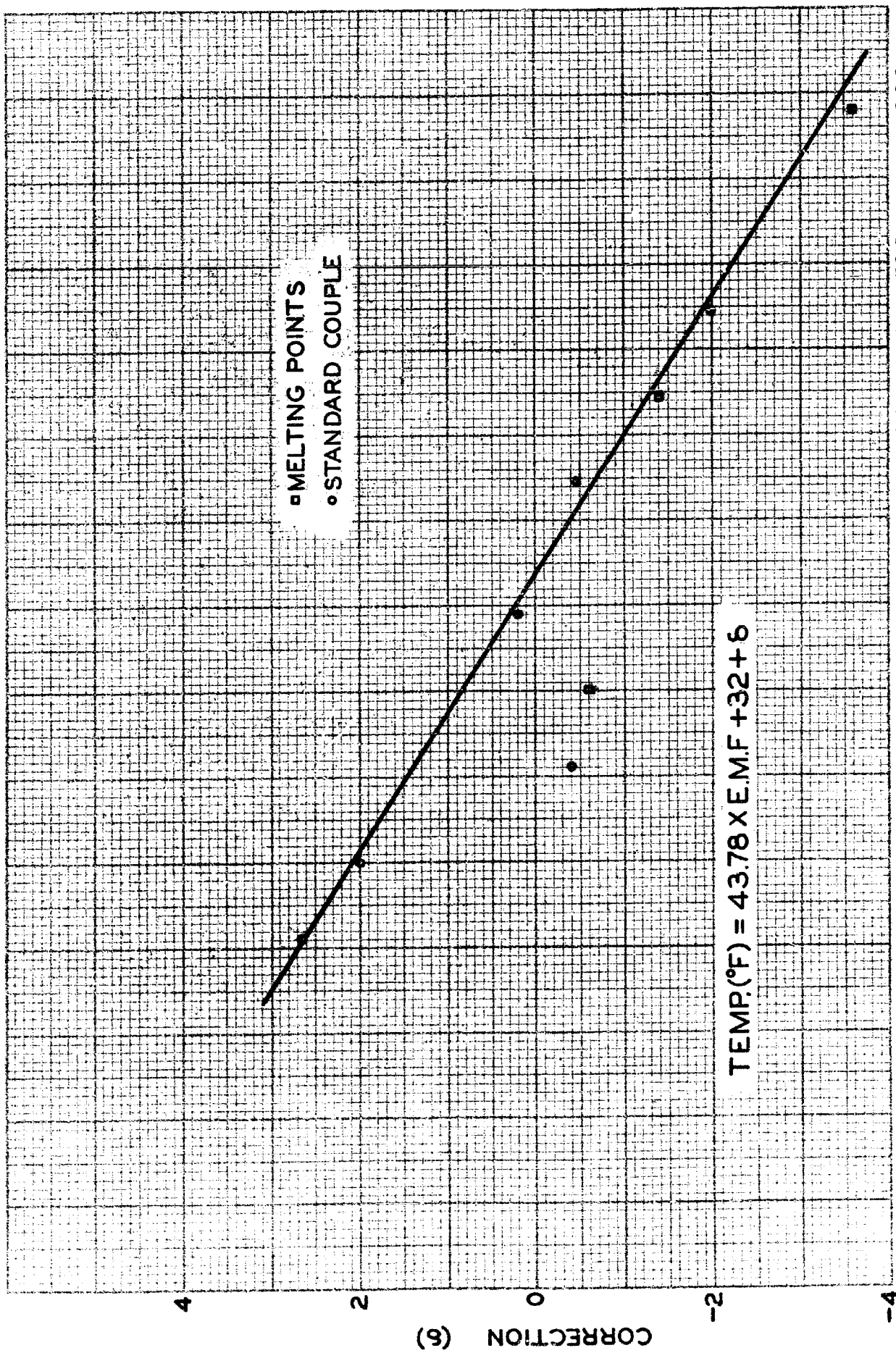


FIGURE 12A CALIBRATION OF THERMOCOUPLE NO. 1

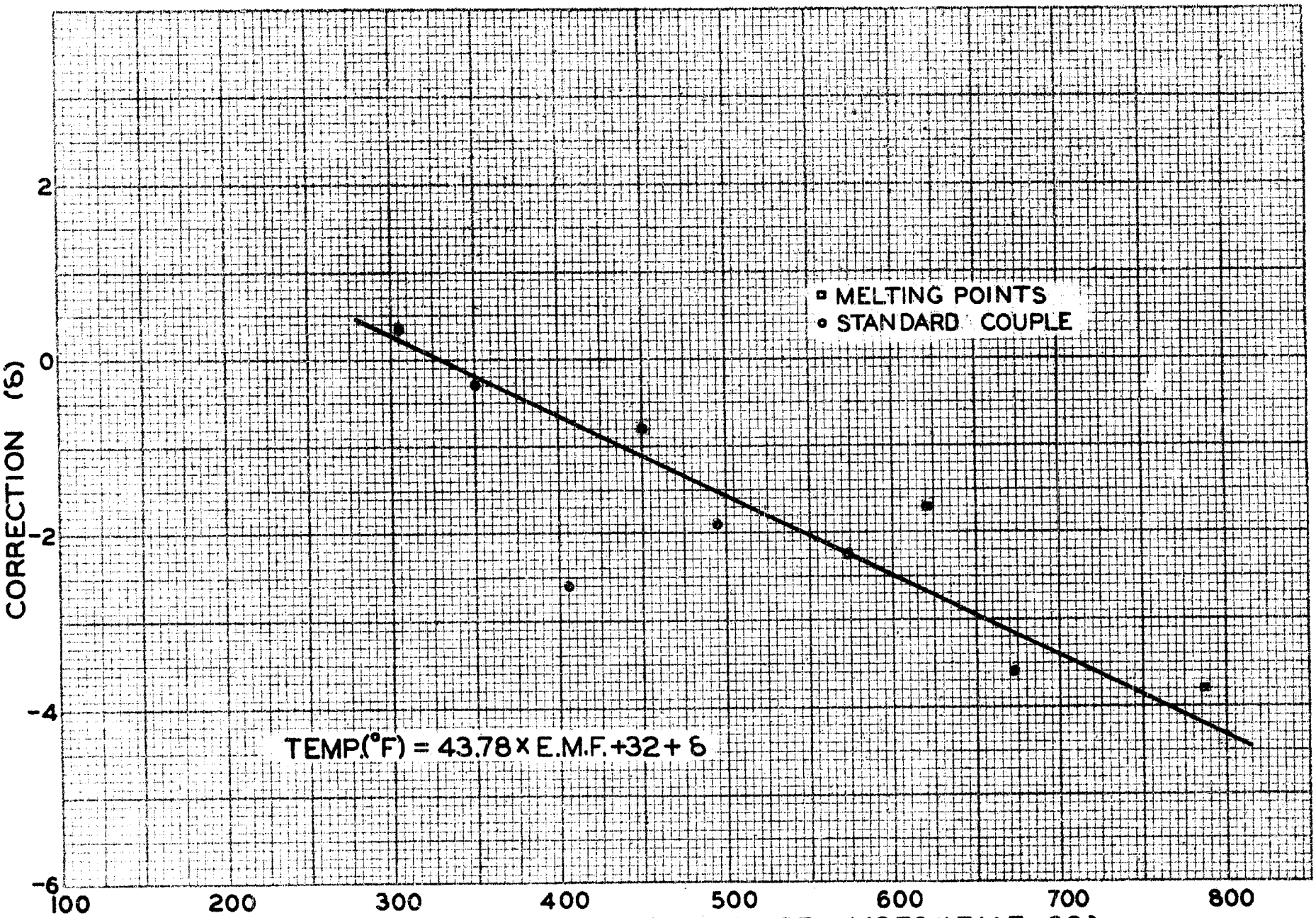


FIGURE 12B CALIBRATION OF THERMOCOUPLE NO. 2

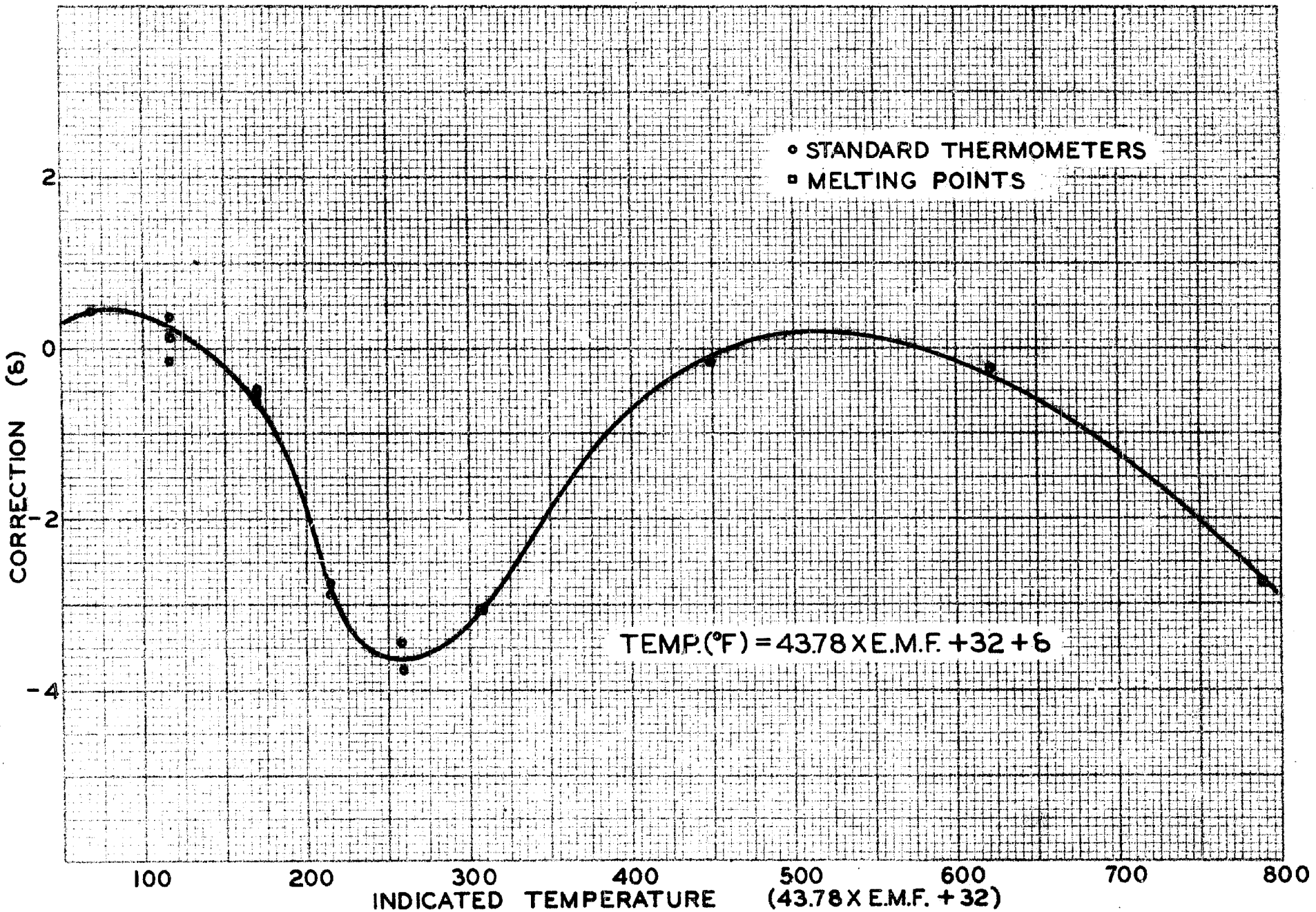


FIGURE 12C CALIBRATION OF THERMOCOUPLE NO.3

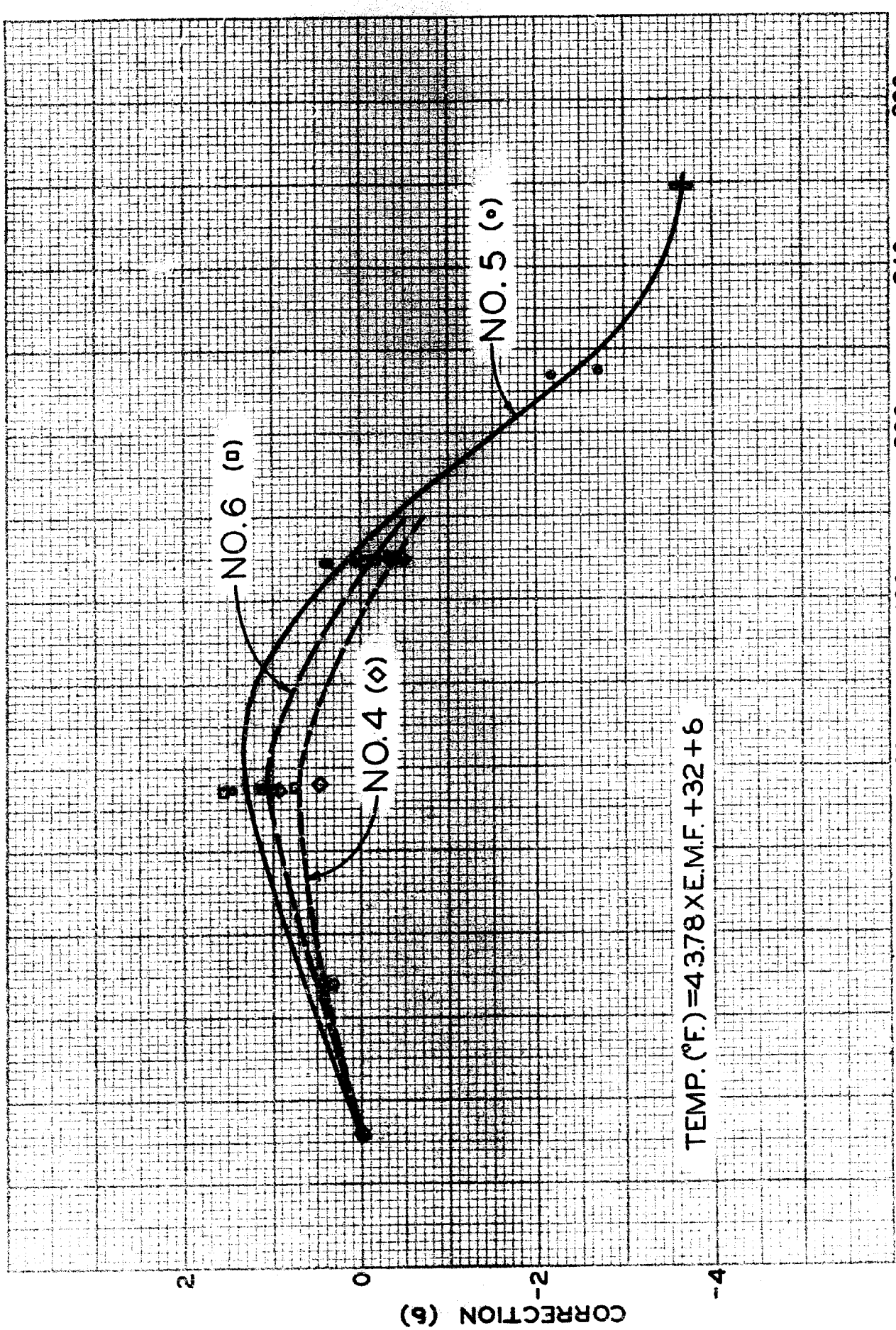


FIGURE 12D CALIBRATION OF THERMOCOUPLES NO. 4, 5, & 6

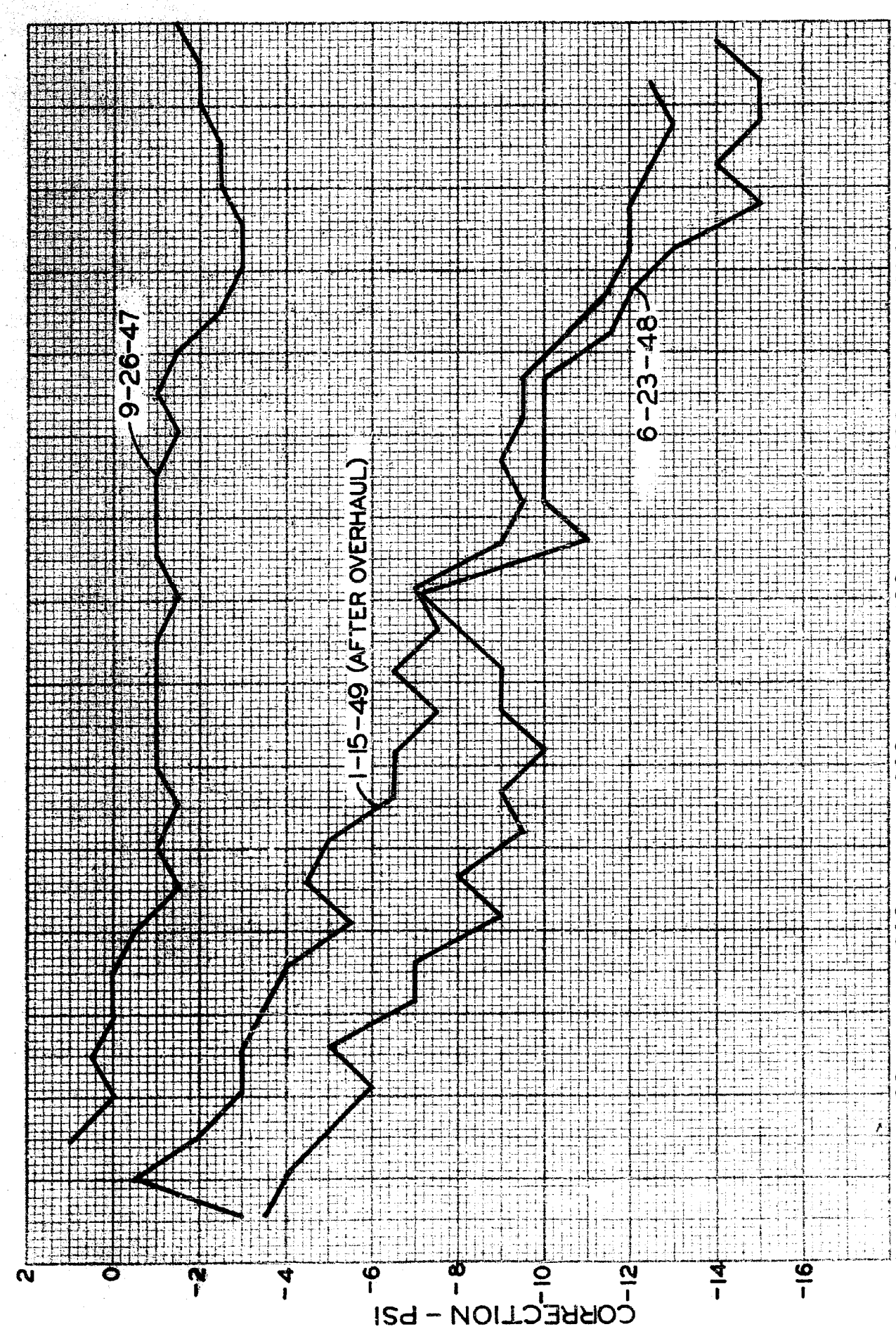


FIGURE 13-A CALIBRATION OF GAUGE NO. C2-131

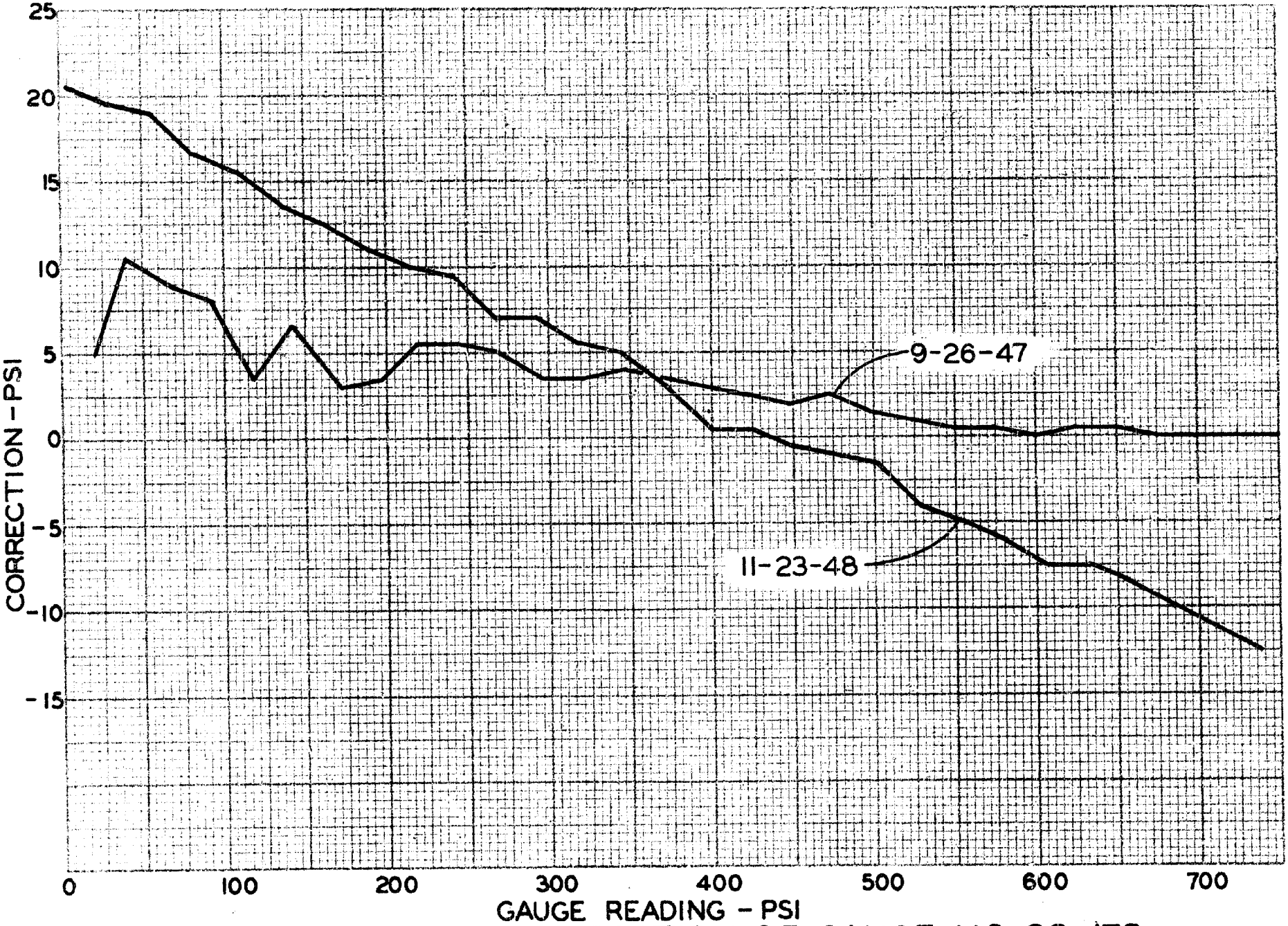
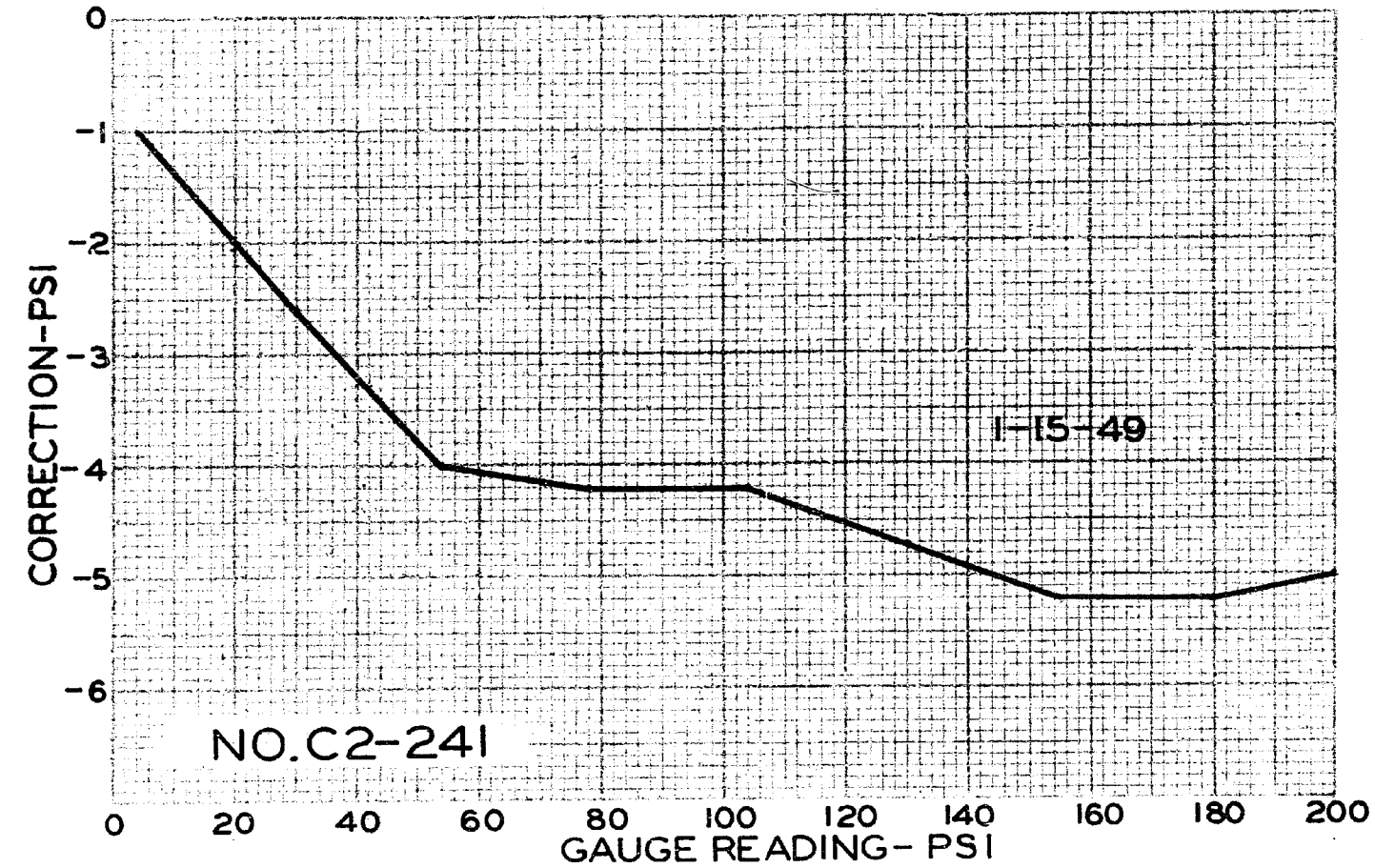


FIGURE 13-B CALIBRATION OF GAUGE NO. C2-172



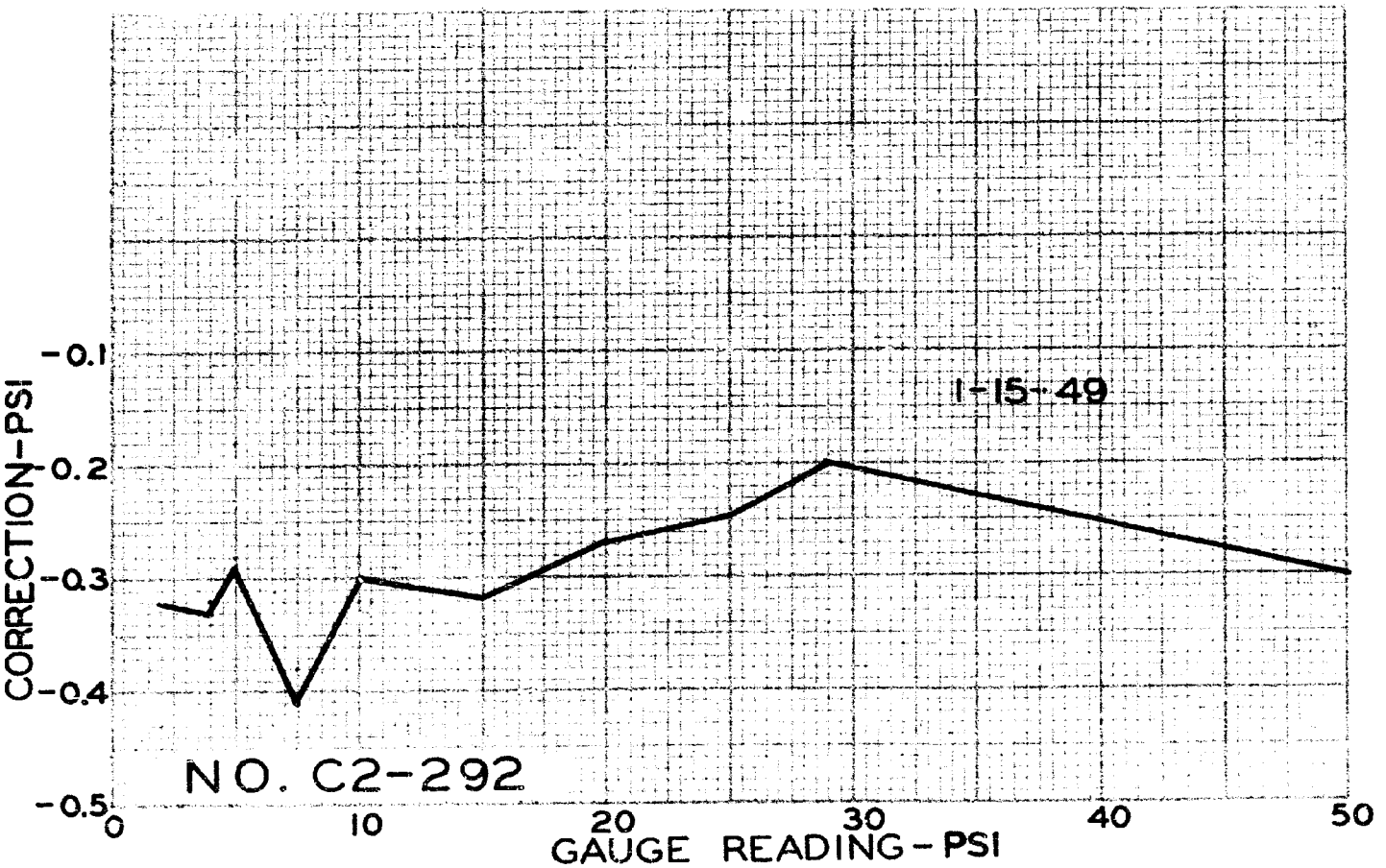


FIGURE 13-C CALIBRATION OF GAUGES