OPERATIONAL CHARACTERISTICS OF A TEN PLATE BUBBLE-CAP DISTILLATION COLUMN USING AS A SYSTEM A PETROLEUM MIXTURE

by

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I. INTRODUCTION

The process of distillation originated as a means by which spirits and "medical waters" could be purified and separated from essential oils and herbs. From this humble beginning, the art of distillation took on a new perspective when brewers in France developed the modern fractionating tower for the production of brandy. Fractional distillation is the separation of the constituents of a liquid mixture by partial vaporization of the mixture and separate recovery of the vapor and residue. This type of distillation is carried out in such a manner that the rising vapor comes into contact with a condensed portion of the vapor previously evolved.

From the standpoint of internal construction, most fractionating columns may be classified as bubble-cap, tray, sieve plate, or packed towers. The bubble-cap, tray, and sieve plate towers have many applications for industrial operations because of their large capacities. The packed tower, in addition to being used in industry, is used extensively in research and development both on pilot plant studies and bench scale work because of its low initial cost and ease of construction. The important variables for the successful operation of a distillation unit include feed temperature, column design, reflux temperature, pressure, and reflux ratio.

The bubble-cap column is used extensively in the petroleum industry to separate crude petroleum into various fractions and for further separation of those boiling range fractions. The important variables necessary for the successful operation of a fractionation unit include boiling range of the feed, column design, reflux ratio, reflux temperature, and pressure.

The purpose of this investigation was to determine the individual plate and overall column efficiencies for a ten-plate bubble-cap distillation column using as a system a petroleum fraction. The variables that were studied included product valve opening, calandria section temperature, and feed flow rate.

II. LITERATURE REVIEW

The following section of the thesis contains a review of the literature on distillation in general and bubble-cap fractional distillation in particular. The material presented will have the following headings: basic concepts of distillation, definitions, theory of distillation, and calculations for rectification of binary systems.

Basic Concepts of Distillation

Carey(10) defines distillation as "the separation of the constituents of a liquid mixture by partial vaporization of the mixture and separate recovery of the vapor and residue". The more volatile components of the original mixture are obtained in increased composition in the vapor, whereas the less volatile components appear in greater concentrations in the residue. Distillation is feasible for the separation of miscible liquid components only if the composition of the liquid phases to be separated into components is different from the composition of the vapor phase in equilibrium with it(41).

Equilibrium Distillation. There are two important kinds of distillation that do not involve rectification, namely: (1) equilibrium of flash distillation, and (2) simple or differential distillation. Equilibrium distillation is concerned with vaporizing a definite batch of liquid, keeping all of the liquid and all of the vapor in contact so that at the end of the operation the vapor will be in equilibrium with the liquid(2). This process is not of great importance in two component distillation but is used primarily in the petroleum industry(4).

Differential Distillation. In differential distillation the vapor formed by boiling the liquid is removed and condensed as rapidly as it is formed(4). The first portion of the distillate formed will be richer in the more volatile component, and as the distillation proceeds, the product becomes leaner(60); this type of distillation is commonly referred to as batch distillation.

Rectification. Rectification or fractional distillation is "the partial removal of the less volatile component from the vapor by allowing the vapor to interact with the partial condensate from a previous fraction or vapor from the same still"(65). This type of distillation is equivalent to a series of redistillations without the consumption of additional heat(66). A transfer of material and an interchange of heat results from this contact, thereby securing a greater enrichment of the more volatile component in the vapor than would be possible with a single distillation operation utilizing the same amount of heat.

Continuous Rectification. Continuous rectification is essentially fractional distillation in which feed is continuously introduced into the column, and a product and a bottoms are continuously withdrawn from the top and the bottom of the column, respectively(68). Either the product or the bottoms is a relatively pure component, depending on the specifications desired.

Definitions

The following section contains definitions of some of the basic terms of distillation theory.

Reflux Ratio. Reflux ratio is defined as the ratio of the number of mols of liquid passing down the column per unit of time to the number of mols of product per unit time(52).

Holdup. During any time period, a certain amount of the liquid entering the column is actually in the still as reflux and rising vapor(53); this material is known as holdup. The proportion of holdup to the total mixture being distilled is of great importance in batch distillation since it limits the percentage of the charge that can be distilled, and also it affects the sharpness of separation.

Vapor Pressure. The vapor pressure of a liquid is the pressure of the vapor of that liquid at any given temperature at which the vapor and liquid are in equilibrium(11). The value of the vapor pressure is constant at any given temperature and increases with an increase in temperature. Since vapor pressure is an equilibrium value, the tendency to approach equilibrium is dependent upon whether condensation of the vapor or vaporization of the liquid is necessary. The distance from equilibrium in any practical commercial operation represents the driving force or activating potential which is directly related to the rate of mass transfer and consequently to the size of equipment required for carrying out the operation(63).

Vapor pressures can be obtained from the Cox chart(39) by any of the following methods where the latent heats of vaporization are essentially constant over small temperature ranges:

Duhring's Rule. Duhring's rule is useful for estimating approximately the vapor pressures at various temperatures for any liquid if the vapor pressure-temperature data are available for a reference liquid$^{(24)}$:

$$T_B = \frac{T'_B}{T'_A} T_A$$

where:

T_B = temperature of liquid B at which the vapor pressures of A and B are equal, °K

T_A = temperature of reference liquid A corresponding to the vapor pressure of A and B at T_B, °K

T'_B = temperature different from T_B at which the vapor pressures of A and B are equal, °K

T'_A = temperature of reference liquid B corresponding to the vapor pressure of A and B at T'_B, °K.

Clausius-Clapeyron Equation. The Clausius-Clapeyron equation may be used to determine the vapor pressures of a liquid provided the assumption of ideal gas behavior is made. The equation is as follows(22):

$$\frac{dp}{dT} = \frac{p\,\Delta H}{R\,T^2}$$

where:

$\frac{dp}{dT}$ = rate of change of the vapor pressure of the liquid with temperature, atm/°K

p = vapor pressure of liquid at temperature T, atm

ΔH = molal latent heat of vaporization of the liquid, cal/mol

T = temperature of liquid, °K

R = gas constant, cal/mol-°K.

Integrated Clausius Equation. Upon integrating the Clausius-Clapeyron equation between limits p_2 at T_2 and p_1 at T_1, the following relationship is obtained(23):

$$\log \frac{p_2}{p_1} = \frac{\Delta H \, (T_2 - T_1)}{(2.303)\ (R)\ (T_1 T_2)}$$

where:

p_2 = vapor pressure of liquid at temperature T_2, atm

p_1 = vapor pressure of liquid at temperature T_1, atm

T_2 = temperature corresponding to vapor pressure p_1, °K

T_1 = temperature corresponding to vapor pressure p_1, °K

ΔH = molal latent heat of vaporization of liquid, cal/mol - °K

R = gas constant, cal/mol - °K.

Since the magnitude of the vapor pressure at any given temperature is directly related to the intermolecular attractive forces of the molecules, and since these forces tend to increase with increase in molecular size and complexity it follows that the vapor pressure decreases with increasing molecular weight for similar liquids(64).

Volatility. In general, the term volatility is used to compare the vapor pressures of two substances, the substance having the higher vapor pressure being the more volatile. For use in distillation, volatility can be defined as the ratio of the partial pressure of any component in a liquid mixture to the mol fraction of that component in the liquid(12).

Mathematically,

$$V_A = \frac{P\, y_A}{x_A}$$

where:

V_A = volatility of component A, mm Hg

P = total vapor pressure of the mixture, mm Hg

y_A = concentration of A in vapor phase, mol fraction

x_A = concentration of A in liquid phase, mol fraction.

Relative volatility is the ratio of volatilities of the two components and is a direct measure of the ease of separation of components by distillation operations. It can be seen from the following equation(59) that the larger the value of the relative volatility, the easier will be the separation:

$$\alpha = \frac{y/(1-y)}{x/(1-x)}$$

where:

α = relative volatility, dimensionless

y = concentration of the more volatile component in the vapor phase, mol fraction

x = concentration of the more volatile component in the liquid phase, mol fraction.

Another method for determining relative volatility is given by Rose and Biles(8).

Theory of Distillation

The following sections contain the basic considerations of theory as applied to the fractional distillation of binary systems. A more complete discussion is given by Robinson(50) and Carey(10).

Boiling Point Diagram. The basic requirement for a distillation separation is that the composition of the vapor phase at equilibrium be different from the composition of the liquid from which it was formed. Thus, by holding the total pressure constant and plotting the boiling points against the composition of the more volatile component in both the liquid and vapor phases, a curve is obtained which is similar to Figure 1, page 14. Vapor compositions in equilibrium with the liquid are plotted horizontally.

Equilibrium Diagram. For the purpose of solving distillation problems, a simplified form of the boiling point diagram is used. This form consists of plotting the concentration of the more volatile component in the liquid phase against the concentration of the more volatile component in the vapor phase at equilibrium. This is presented by Figure 2, page 15. An equilibrium curve is easily constructed when the boiling point

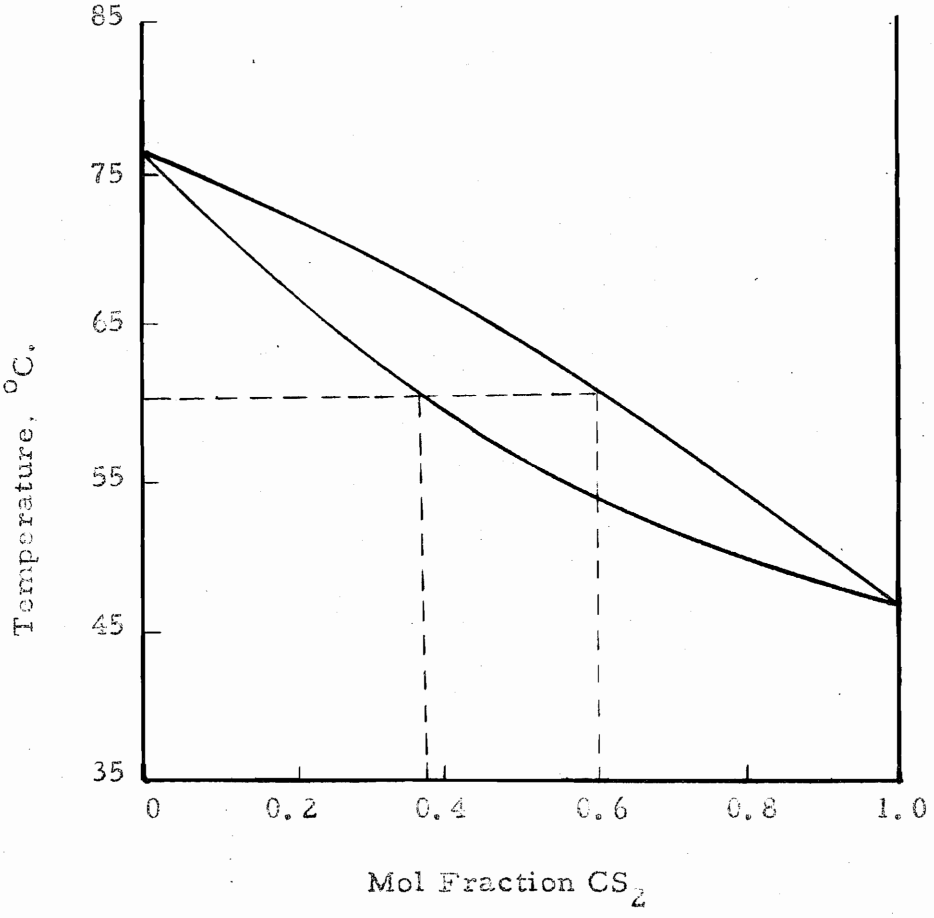


Figure 1. Boiling Point Diagram for

CCl_4- CS_2 Mixtures

Robinson, C. S. and E. R. Gilliland: "The Elements of Fractional Distillation", p. 12. McGraw-Hill Book Co., Inc., New York, N. Y., 1939. 3ed.

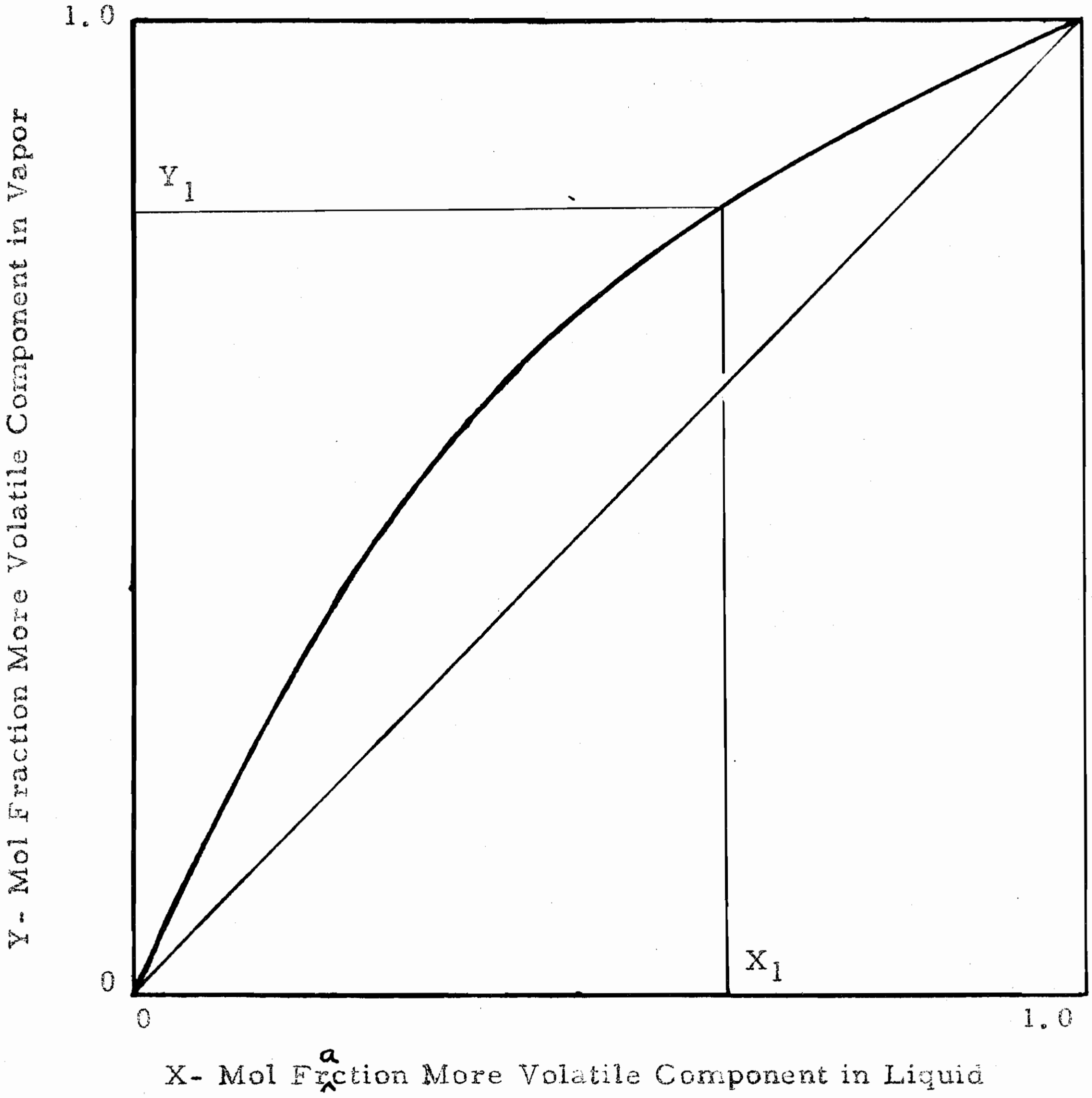


Figure 2. Equilibrium Diagram

diagram is known. Tables and curves giving equilibria data are found in handbooks(17,44) and in other literature.

Even though a great deal of equilibria data have been published, there is considerable discrepancy among data of various investigators using different stills. Since very few systems form ideal solutions, equilibria data have to be determined for every practical design. There is doubt about the validity of these data, both from a theoretical thermodynamic aspect and for commercial design(62).

Raoult's Law. When one liquid is dissolved in another the partial pressure of each is decreased(67). Hence, the partial pressure of each component is equal to the product of the vapor pressure of that component in its pure state and its mol fraction in the mixture(51). This is known as Raoult's law and is stated mathematically as follows(25):

$$p = p_o x_o$$

where:

p = partial pressure of one component, mm Hg

p_o = vapor pressure of that component in the pure state, mm Hg

x_o = concentration of that component in the solution, mol fraction.

The assumption of ideal behavior is made when using Raoult's law; although a slight error is encountered due to non-ideality of solutions, it is usually neglected in most engineering problems.

Henry's Law. At high temperatures and pressures or at low concentrations, Raoult's law is inaccurate and must be corrected. The correction is incorporated in Henry's constant that appears in the following equation(25):

$$p = Kx_o$$

where:

p = partial pressure of a component, mm Hg

K = Henry's constant, mm Hg

x_o = concentration of that component in the liquid mixture, mol fraction.

Henry's constant is not the vapor pressure of a pure component, as in the case of Raoult's law, but is a constant that must be evaluated experimentally for each solution. It may be said that Raoult's law applies to the solvent, and that Henry's law applies to the solute of a solution(3).

Theoretical Plate. The simplest measure of the ease of a distillation rectification process is the theoretical plate. A perfect plate is defined as one in which the liquid leaving is in equilibrium with the vapor leaving(18). The number of theoretical or ideal plates required for the separation depends upon the equilibria involved, the ratio of liquid to gas flow, purity, and the recovery required(18). The number of actual plates is usually somewhat greater than the number of ideal plates. A measure of column efficiency is to divide the theoretical number of plates required for the separation by the actual number of plates used for the same separation.

Bubble-cap Column. There are many different types of distillation columns, the most important of which are sieve tray, bubble-cap tray, and packed columns. The distinguishing feature between these columns is the liquid-vapor contacting medium used. The sieve plate tower employs perforated plates spaced at specified intervals normal to the flow of vapor. The packed tower consists of a vertical shell filled with suitable packing material.

The bubble-cap column differs from the sieve plate column only in the construction of the plate. A bubble-cap plate is pierced with a number of holes into which are fitted "chimneys", or risers, through which the vapors from the plate below may pass. Each of the risers is covered with a bell-shaped cap which is fastened to the riser by means of a "spider" or other suitable mounting. The lower edge of the cap may be serrated, or the lower periphery of the cap may be pierced with a number of holes in order to permit passage of the vapors into the liquid on the plate(9).

Bubble-cap trays should be considered in four parts(69): (1) a contacting zone, (2) an entrainment eliminating zone, (3) a liquid collecting zone, and (4) a liquid inflow zone. This approach to bubble-cap tray design and evaluation of pressure drop through each plate has been reported by Atkins(1) and Cauley and coworkers(16).

Factors that affect the performance of a bubble-cap column include vapor velocity, liquid gradient on a plate, bubble-cap size and spacing, as well as overall column design. Equations and quantitative data on the magnitude of the different effects have been worked out and reported in the literature(7,26,40). In addition to those variables already mentioned, Gautreaux and O'Connell(37) found that plate efficiency was a function of the length of liquid path on a plate.

For many years it was thought that entrainment, or the mechanical carrying over of small liquid droplets from plate to plate(15) defined the limiting vapor velocity for any given conditions. The results of many quantitative studies led to the following conclusions(38,43,55): (1) entrainment may reach a high ratio in terms of weight of entrained liquid per unit weight of vapor before fractionating efficiency is altered appreciably, and (2) other factors such as (a) froth or foam buildup on the plate, (b) jetting or spouting action between caps, and (c) weir and downflow performance usually become controlling before vapor rates are reached at which entrainment interferes with fractionating efficiency(15).

Calculations Involved in Binary Distillation

Sorel(56) was the first to study theoretical considerations of binary distillation by applying heat and material balances from plate to plate throughout the column and calculating the enrichment of the vapor. Savarit(54) and Thiele(58) greatly simplified Sorel's method by solving graphically for the number of equilibrium plates required to perfect a given separation.

Material and Enthalpy Balances. The basic requirements for the analysis of any type distillation operation are complete heat and material balances for the piece of equipment used. No matter how complex the operation may be, it can always be represented by a simple flow sheet that indicates the material introduced in the form of feed is equal to the material leaving in the form of distillate and bottoms. The following equation can be applied to all rectification operations:

$$F = D + B$$

where:

F = rate of flow of feed, lb mol/hr

D = rate of flow of distillate, lb mol/hr

B = rate of flow of bottoms, lb mol/hr

By using the same analogy a heat balance for the column or for each plate may be determined. The following equation may be used to obtain an overall heat balance for a distillation operation:

$$FH_F + Q = DH_D + BH_B$$

where:

F = feed flow rate, lb mol/hr

H_F = enthalpy of feed, Btu/lb mol

Q = heat lost by heating medium, Btu/hr

D = distillate flow rate, lb mol/hr

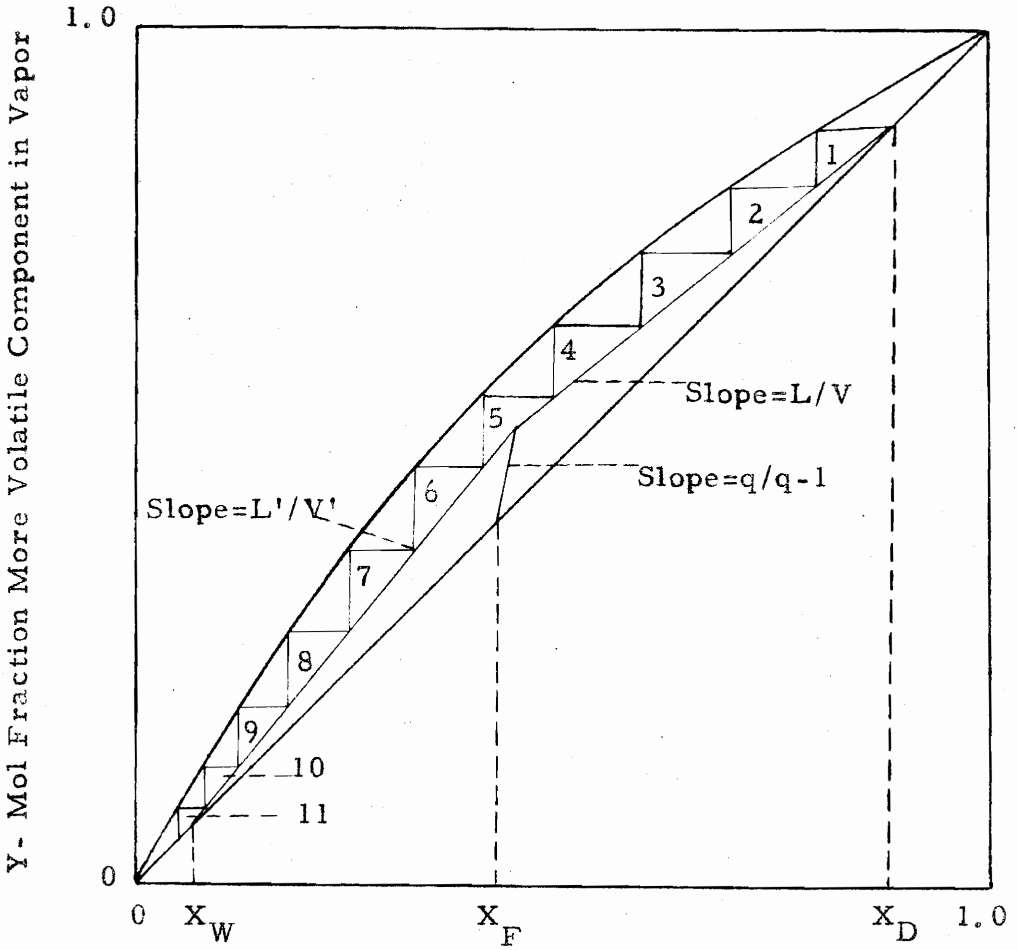
H_D = enthalpy of distillate, Btu/lb mol

B = bottoms flow rate, lb mol/hr

H_B = enthalpy of bottoms, Btu/lb mol.

McCabe-Thiele Method of Calculation. Assuming constant molal overflow, Raoult's law, negligible tower heat losses, and negligible heats of mixing(13), the number of equilibrium plates needed to perform a given separation may be graphically determined by using the equilibrium diagram and two operating lines. In addition to the two operating lines a third line is constructed for the purpose of simplification. An example of the McCabe-Thiele method of calculation is shown in Figure 3, page 25.

Enrichment Line. The operating line for the rectification section of the column is determined from a material balance for the section of the column above the feed plate. The enrichment line represents the composition of the vapor leaving and the liquid entering(19). The



X- Mol Fraction More Volatile Component in Liquid

Figure 3. Graphical Analysis of Continuous Fractionation by McCabe-Thiele Method

Teller, A. J.: Binary Distillation, Chem. Eng., 61, 176 (1954) following equation expresses the operating line mathematically(5):

$$y_{n+1} = \frac{L}{L+D} x_n + \frac{D}{L+D} x_D$$

where:

y_{n+1} = composition of more volatile component in vapor phase rising from plate <u>n+1</u>, mol fraction

x_n = composition of more volatile component in liquid phase leaving plate <u>n</u>, mol fraction

x_D = composition of more volatile component in the product, mol fraction

L = reflux flow rate, lb mol/hr

D = product flow rate, lb mol/hr.

Stripping Line. The operating line for the section of the column below the feed plate is determined by the same analogy that is used for obtaining the enriching line. The equation for the stripping line is as follows(61):

$$y_{m+1} = \frac{\overline{L}}{\overline{L}-W} x_m - \frac{W}{\overline{L}-W} x_W$$

where:

y_{m+1} = composition of more volatile component in vapor leaving plate m+1, mol fraction

x_m = composition of more volatile component in liquid leaving plate m, mol fraction

x_W = composition of more volatile component in bottoms, mol fraction

$\overline{L}$ = flow rate of liquid leaving plate m, lb mol/hr

W = flow rate of bottoms, lb mol/hr.

Feed Enthalpy Line. The third line that is constructed on the equilibrium diagram is the q-line. This line represents the heat condition of the feed and has a slope of $\frac{q}{q-1}$. The definition of q is the total amount of heat needed to convert one mol of feed into saturated vapor divided by the molal latent heat of vaporization of the feed. The q-line originates at the feed composition on the 45-degree diagonal of the equilibrium curve and is drawn through the intersection of the operating lines. The equation is as follows:

$$y = \frac{q}{q-1}x - \frac{x_f}{q-1}$$

where:

y = composition of more volatile component in vapor phase, mol fraction

x = composition of more volatile component in liquid phase, mol fraction

x_f = composition of more volatile component in feed, mol fraction

q = total amount of heat necessary to vaporize one mol of feed divided by the molal latent heat of vaporization of the feed, dimensionless.

In approaching a distillation calculation using the McCabe-Thiele method, the usual procedure is to construct the rectifying operating line and the q-line, and then to draw the operating line for the stripping section from the bottoms composition through the intersection of the previous two lines.

<u>Minimum Reflux Ratio</u>. The theoretical minimum reflux ratio is that ratio at which an infinite number of theoretical plates is required to perfect a given separation(14). For most binary distillation operations the minimum reflux is calculated by using the coordinates of the point at which the operating lines and the *q*-line intersect on the equilibrium curve. The equation(6) for minimum reflux is as follows:

$$R' = \frac{x_D - y'}{y' - x'}$$

where:

R' = minimum reflux ratio, dimensionless

x_D = composition of more volatile component in product, mol fraction

y' = composition of more volatile component in vapor phase at intersection of equilibrium curve, *q*-line, and operating lines, mol fraction

x' = composition of more volatile component in liquid phase at intersection of equilibrium curve, *q*-line, and operating lines, mol fraction.

If the equilibrium curve is concave upward in the upper regions of compositions, then the $\underline{x'}$ and $\underline{y'}$ values will be taken from the point of intersection of the rectifying operating line and the equilibrium curve.

The number of theoretical plates is found by stepping off, horizontally and vertically, equilibrium stages using the operating lines, as shown in Figure 3, page 25.

Instrumentation and Control of Fractionating Columns

The primary purpose of instrumentation is the measurement of process variables(20). The measured values of the variables must also be presented to the process operators in a useful manner as an indication of the state of the process and/or be used in a manner to control the process. The primary consideration in designing a process control system is the process itself and it is usually the process that determines the control system and thus the accuracy of measurement and degree of automation that can be attained(20).

According to Brown(9), the general procedure involved in the instrumentation and control of fractionating columns is to establish controls so that each independent variable is controlled by one and only one primary control and that sufficient flexibility is allowed for variations in feed and product specifications. Stream flow rates, stream and column temperatures, and liquid levels in the column and off-site equipment are all available impetuses for control elements. The Minneapolis-Honeywell Regulator Company(36) recommends three primary control elements for the efficient automatic operation of a continuous distillation column. The use of flow rate regulators on the feed and steam lines and a liquid level controller to regulate the rate of bottoms discharge are the minimum control elements necessary to control a simple distillation installation using a feed with constant composition. A more complex installation would require the use of a temperature controller to adjust the reflux rate to provide for constant overhead product, a liquid level controller to maintain a constant level in the reflux accumulator, and temperature controllers on the feed preheater and product condenser(20). The three principal elements associated with the more common types of control instruments are: (1) the sensing element, (2) the converting element, and (3) the functioning element.

The Sensing Element. The measurement of process variables is performed by a device generally termed a sensing element. The sensing element is that part of the instrument which first utilizes energy from the measured medium to produce a condition representing the value of the measured variable(27). In measuring temperature, a thermocouple, a resistance thermometer, a pressure spring thermometer, or any of several other specialized temperature sensing devices may be the sensing element. In measuring fluid flow, head meters such as orifice meters are among the more common sensing elements. Liquid level measurement is usually effected with pressure elements or floats.

The Converting Element. The nature of the converting element is dependent upon the type of signal received from the sensing element and the form of the final or functioning element of the instrument. The signal generated from the sensing element may be electrical, hydraulic, or pneumatic, which means that the converting element must transform this condition into a condition that is useful to the functioning of the instrument(28). In the case of the chemical process industries, most of the instruments used are of a mechanical nature; therefore, the differential signal from the sensing element must be converted into a mechanical motion. The bourdon tube, helix, or another form of pressure spring is commonly used to convert hydraulic pressure into mechanical motion(30). The conversion of a differential pressure, as encountered in flow measurement or liquid level determinations, to mechanical motion is enacted by an iron leg manometer incorporating a steel float with suitable mechanical linkage(31). For remote transmission purposes, it is sometimes desirable to convert the differential pressure to an air signal with a force balance type differential converter(34) or flow transmitter(35).

The Functioning Element. The functioning or final element denotes that part of an instrument used for transmitting, signaling, registering, indicating, recording, or controlling(29). The difference between the functioning and converting elements at times exists in definition only. Their function may overlap, or one of the two may not even be present in some types of instruments(29).

Of the various forms of functioning elements, the most common types are the transmitting, the recording, and the controlling types. The transmitting element may or may not be incorporated into a recording and/or controlling instrument. It may also be considered a form of converting element or actually a complete functioning unit. The recording functioning element is some form of bellows or pressure spring activated by a pneumatic signal from the transmitting element, thus causing a pen to move across a chart, recording the value of a variable. The recorder may be included in the controller instrument or housed in a separate case.

The fundamental purpose of the control functioning element is to transmit to the final control device or diaphragm motor value a pneumatic signal whose strength is determined by the value of a measured variable and to constantly modulate that signal to maintain that variable at a predetermined level(45).

The more common control devices consist of three elements that allow for a continuous adjustment of the control valve position. Proportional band is defined as that per cent of full scale through which the pen must move in order to move the control valve to the full open or full closed position(46). Reset is shifting the proportional band or throttling range around the control point so the pen can be maintained at the control point regardless of the process changes(46). Rate action provides for a continuous linear relation between rate of change of the controlled variable and position of the final control element(47).

It must be emphasized that the methods of employing the previously discussed principles of instrumentation are peculiar to each individual manufacturer.

Method of Analysis

There are many different methods by which mixtures of hydrocarbons can be analyzed and separated into individual components. Among these are (1) refractive index determination, (2) chromatography, (x) x-ray pattern, and (4) mass spectrophotometry. The following paragraphs contain a discussion of the first two methods mentioned above.

Refractive Index. According to Daniels(21), the extent to which a beam of light is bent or refracted when it passes from one substance into another depends on the relative number of atoms in the two substances which are in the path of the light, on the kinds of atoms, and on the arrangement of the atoms within the molecules. The refractive index, which is a quantitative measure of this refraction of light, may be used to determine the concentration of materials, to establish the identity and purity of a chemical compound, or to ascertain the arrangement of atoms within the molecule(21).

Various relationships may exist between refractive indices and concentration of two components in a mixture. For mixtures of hydrocarbons a linear relationship is obtained.

Chromatography. One of the more useful methods for the resolution of mixtures and for the isolation and identification of the components is known as chromatography or chromatographic adsorption analysis(33). According to Podbielniak and Preston(48), the principle utilized in separating the components of a hydrocarbon mixture by vapor-phase partition chromatography is that of selective sorption in columns having up to 2,000 theoretical plates, packed with solid adsorbent or with liquid solvent supported on inert packing. The hydrocarbon sample is carried through the analyzer by means of a carrier gas, each component moving at a rate corresponding to its "solubility" in the stationary packing. The components are identified by the volume of carrier gas passed from the start of the analysis until each component reaches the column effluent as indicated by the detector(48).

III. EXPERIMENTAL

The experimental section of this thesis is concerned with the purpose of investigation, plan of experimentation, materials, apparatus, methods of procedure, data and results, and sample calculations used throughout the investigation.

Purpose of Investigation

The purpose of this investigation was to determine the individual plate efficiencies and overall column efficiencies of a ten-plate bubble-cap distillation column using a petroleum fraction as a feed mixture. The variables that were studied include product valve opening, feed flow rate, and calandria section temperature.

Plan of Experimentation

An outline of the plan of experimentation for this investigation includes a review of the literature, preliminary work, and column operation.

Review of the Literature. The purpose of the literature review was to acquaint the author with the general principles of distillation and, in particular, distillation as applied to the bubble-cap column. Emphasis was also applied to instrumentation as it affects distillation control systems in procuring a specified overhead product from a bubble-cap fractionating unit. This review also pertained to methods of calculation, previous work done by others on the same topic, and a method of analysis.

Preliminary Work. Minor modifications were made to the auxiliary equipment previously constructed. The control instruments were repaired and tested for satisfactory operation and correct operating range. The hydrocarbon flow recording instruments, which include feed, product, bottoms, and reflux recorders, were calibrated to function within the range of operational variability of the fractionation unit.

Column Operation. In order to gather the necessary data for determining the individual plate and overall column efficiencies of the hydrocarbon fractionating unit, eight tests were made. Throughout all the tests the feed composition was approximately constant at 55 mol per cent hexane and 45 mol per cent heavy virgin naphtha, the feed temperature was 141 °F, and the condensate temperature was 146 °F. The feed flow rates used were 9.9 and 12.7 mols per hour, the calandria section temperatures were 257 and 275 °F, and the product valve settings were 13 and 15 pounds per square inch, gage.

Materials

The following paragraphs contain a listing of the materials used in the test operations of the automatically controlled hydrocarbon fractionating unit.

Air. Supplied at 35-100 lb/sq in., gage, filtered and reduced to 20 lb/sq in., gage. Supplied by Nash Hytor air compressor in the Unit Operations Laboratory, Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used as supply air for pneumatic control instruments.

Heavy Virgin Naphtha. Boiling range 220-309 °F at 28.18 inch Hg pressure. Obtained from the Baltimore Refinery of the Esso Standard Oil Company. Used as one component of the feed mixture for the fractionating tower.

Hexane. Commercial grade, boiling range 148-152 °F at 28.18 inch Hg pressure. Obtained from Hercules

Powder Co., Inc., Wilmington, Del. Used as one component of the feed mixture for the fractionating column.

Mercury. Technical grade, triple distilled. Obtained from Mexican Metals, Inc., New York, N. Y. Used in the manometer legs of the indicating flow transmitters.

Steam. Supplied at 80 lb/sq in., gage. Obtained from the steam mains of the Unit Operations Laboratory, Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used as heat supply for feed mixture and calandria section.

Water. Tap, temperature approximately 58 °F, supplied at 80 lb/sq in., gage. Obtained from the water mains of the Unit Operations Laboratory, Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used for:

(1) condensing overhead vapor product
(2) cooling bottoms stream
(3) preliminary tests on the fractionating tower
(4) calibrating product and reflux flow rate recorders.

Apparatus

Included in the following section is a listing of all apparatus installed in the hydrocarbon fractionation unit and the instrument panel board.

Clock. Electric, Telechron, model No 1HA1608, sweep second hand, 115 v, 60 cy, 2 w. Manufactured by General Electric Co., Ashland, Mass. Used for timing operation periods.

Column, Distillation. Size 2 x 21-1/3 ft, ✓ constructed of 24-inch diameter ASTM A-106 grade A pipe, design pressure 350 lb/sq in., design temperature 150 °F, contains 10 plates, spaced 1 ft apart each having twenty-two 2-inch bubble-caps, integral calandria constructed of 1-inch extra heavy pipe having total heating area of 60 sq ft. Manufactured in and a gift of the Baltimore Refinery of the Esso Standard Oil Co. Used as the hydrocarbon fractionation column.

Controller. Serial No J-49075, D/P Cell, range ✓ 0-50 inches, working pressure 750 lb/sq in., gage. Manufactured by the Foxboro Co., Foxboro, Mass. Used as the liquid level controller on the reflux accumulator drum.

<u>Controller and Recorder</u>. Air-O-Line, proportional plus reset, 24-hour electric clock chart drive. Manufactured by the Brown Instrument Co., Division of Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Presented by the Baltimore Refinery, Esso Standard Oil Co. Used in conjunction with a differential pressure transmitter to record and control the feed stream flow rate to the distillation column. ✓

<u>Controller and Recorder</u>. Fulscope temperature controller, serial No 122RM2038, scale No OP3320, 11-1/2 inch bulb immersion, temperature range 0-250 °C, proportional plus reset, integral manual by-pass, mechanical 24-hour chart drive. Manufactured by the Taylor Instrument Co., Rochester, N. Y. Given by the Union Carbide Chemicals Co., Charleston, W. Va. Used to control steam flow into column calandria and record temperature in reboiler. ✓

<u>Controller and Recorder</u>. Stabilog temperature controller, serial No C61090, range 0-200 °C, 5-inch bulb immersion, tubing length 50 ft, class 4C, proportional plus reset. Manufactured by the Foxboro Co., Foxboro, Mass. Donated by the Union Carbide Chemicals Co., Charleston, W. Va. Used to ✓

control the flow rate of cooling water to the tops condenser by variations in the condensate temperature from the condenser.

Controller and Recorder. Stabilog temperature controller, serial No C61090, chart No 898442, range 0-250 °C, 5-inch bulb immersion, tubing length 35 ft, class 4B, proportional plus reset. Manufactured by the Foxboro Co., Foxboro, Mass. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Used to control the flow of cooling water to the bottoms cooler by variations in the cooled bottoms temperature.

Controller and Recorder. Stabilog temperature controller, serial No C32362, chart No 898445, range 0-300 °F, 10-inch bulb immersion, tubing length 50 ft, class 4D, proportional plus reset. Manufactured by the Foxboro Co., Foxboro, Mass. Given by the Union Carbide Chemicals Co., Charleston, W. Va. Used to control the flow of steam to the feed preheater by variations in the preheated feed temperature.

Controller and Recorder. Air-O-Line, proportional plus reset, mechanical flow controller, model No 220131-X99, serial No 402891, range 0-15000, mechanical 24-hour clock drive. Manufactured by the Brown

Instrument Co., Division of Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Donated by the Baltimore Refinery of the Esso Standard Oil Co. Used to regulate the discharge rate of the bottoms by the measurement of the liquid level in the reboiler.

Controller and Recorder. Air-O-Line, proportional plus reset, mechanical flow controller, model No 702P26C14-91-74, serial No 601817, range 0-15000, mechanical 24-hour clock drive. Manufactured by the Brown Instrument Co., Division of Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Donated by the Baltimore Refinery of the Esso Standard Oil Co. Used to regulate the flow of distillate from the accumulator drum by measuring the liquid level in the drum.

Control Panel, Manual. Range 0-30 lb/sq in., gage, consists of supply air pressure gage, 4 position pneumatic switch, and pressure reducing valve. Manufactured by the Taylor Instrument Co., Rochester, N. Y. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Used to operate pneumatic motor valves manually.

Control Panel, Master. Consists of pressure reducing valve and inlet and outlet pressure gages, range 0-60 lb/sq in., gage. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used to reduce air pressure from compressor to 20 lb/sq in., gage, air pressure required for operation of pneumatic instruments.

Control, Pushbutton Station. Type SD-2, class 15-020, model No 1253069, start-stop. Manufactured by the Westinghouse Electric Corp., Pittsburgh, Pa. Used for remote control of motor pumps.

Filter, Air. Mild steel, constructed from discarded inverted bucket type steam trap housing, 6-inch height, packed with treated fiber air filter media. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used to filter air supply from compressor to control panel.

Heat Exchanger. Single pass, countercurrent, shell and tube type; contact surface of nineteen 5/8-inch copper tubes 45.5 inches long enclosed in mild steel shell. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Used as the feed preheater.

Heat Exchanger. Single pass, countercurrent, shell and tube type; contact surface of eighty-nine 3/4-inch admiralty metal tubes 69 inches long enclosed in mild steel shell. Manufactured by the M. W. Kellogg Co., New York, N. Y. Donated by the Baton Rouge Refinery of the Esso Standard Oil Co. Used as the overhead vapor condenser.

Heat Exchanger. Four-pass, shell and tube type; contact surface of thirty-seven 3/4-inch copper tubes 79 inches long enclosed in mild steel shell. Manufactured by the Ingersoll-Rand Co., New York, N. Y. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Used as the bottoms cooler.

Motor. Electric, type CSP, induction, class 1, style 1261426, serial No 546, 2 hp, 3490 rpm, 60 cy, 3 ph, 220 v, 5.6 amp per terminal. Manufactured by the Westinghouse Electric Corp., Pittsburgh, Pa. Given by the Union Carbide Chemicals Co., Charleston, W. Va. Used to drive reflux and product pumps.

Motor. Electric, type CSP, induction, class 1, style 1261426, serial No 2046, 1.5 hp, 3450 rpm, 60 cy, 3 ph, 220 v, 4.6 amp per terminal. Manufactured by the Westinghouse Electric Corp., Pittsburgh, Pa.

Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Two used; one to drive feed pump and one to drive bottoms pump.

Potentiometer Pyrometer. Electronik, model ✓ No 156x63P48, serial No 540265, temperature range 0-500 °F, 110-125 v, 0.45 amp, 60 cy, 50 w, 48-point indicator. Manufactured by the Brown Instrument Co., Division of Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Given by the Baltimore Refinery of the Esso Standard Oil Co. Used in conjunction with chromel-alumel thermocouples to determine temperatures at various points in the column and off-site equipment.

Pump. Centrifugal, size 204, type ECD, serial No KE-465, capacity 20,000, head 40 lb. Manufactured by the Economy Pumps, Inc., Hamilton, Ohio. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Two used; one to pump feed stream and one to pump bottoms stream.

Pump. Centrifugal, size 254, type ECD, serial No KED-463, capacity 25,000, head 40 lb. Manufactured by the Economy Pumps, Inc., Hamilton, Ohio. Donated by the Union Carbide Chemicals Co., Charleston, W. Va. Used to pump reflux and product streams.

Receiver, Recording. Metric mechanical flow meter, model No 39005, operating pressure 100 lb/sq in., gage, range 0-100 inches of water. Manufactured for the Metric Metal Works by the Taylor Instrument Co., Rochester, N. Y. Presented by the Baltimore Refinery of the Esso Standard Oil Co. Used to record steam flow rate to calandria.

Receiver, Recording. Flow, two pen, 12-inch diameter chart, electric 24-hour chart drive, input air range 3-15 lb/sq in., gage. Manufactured by the Brown Instrument Co., Division of the Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Donated by the Baltimore Refinery of the Esso Standard Oil Co. Three used to record terminal stream flow rates of the column and off-site equipment.

Starter, Magnetic. Model No CR7006B101B, size 0, 3 ph, 220 v, 60 cy, heater No 81D549. Manufactured by the General Electric Co., Schenectady, N. Y. Used to start the product and reflux pump.

Starter, Magnetic. Size 0, form 2, type 1, series No 61AH57, 220 v, 3 ph, 60 cy. Manufactured by the Allen-Bradley Co., Milwaukee, Wis. Used to start the bottoms pump.

Starter, Magnetic. Size 0, form 2, type 1, series No 536706, 220 v, 3 ph, 60 cy. Manufactured by the Allen-Bradley Co., Milwaukee, Wis. Used to start the feed pump.

Switch Box. Single throw, fusible, 3 pole, catalog No 88352, 60 amp, 230 v, 7.5 hp max. Manufactured by the Square D Co., Detroit, Mich. Used in the pump motor control circuit.

Switch Box. Single throw, fusible, 2 pole, catalog No N33, 30 amp, 125 v, 2 hp max. Manufactured by the Wadsworth Electric Manufacturing Co., Inc., Covington, Ky. Used in the electric clock drive circuit.

Tank. Capacity 500 gal, mild steel, welded construction, 4-ft outside diameter, 6 ft long. Constructed in the Unit Operations Laboratory, Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used as the feed storage tank.

Tank. Pre-act stabilizer, capacity 500 ml, model No 33S22. Manufactured by the Taylor Instrument Co., Rochester, N. Y. Used in conjunction with the Taylor Fulscope controller as part of the pre-act time control function.

Tank. Capacity 20-gal, mild steel, welded construction, 14.5-inch outside diameter, 39-inch height less support, 55-inch overall height including angle iron tripod support, contains 15-ft heating coil of 1/4-inch copper tubing. Donated by the Union Carbide Chemicals Co., Charleston, W. Va. Used as the reflux accumulator drum.

Tank. Knock-out, capacity 1 liter, constructed of a 9-inch section of 3-inch mild steel pipe, welded ends, equipped with drain cock. Constructed in the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used in the air line from the compressor to the panel board to remove entrained particles from the supply air.

Thermocouples. Nine chromel-alumel, type K, 14 gage, equipped with 304 stainless steel protecting tube 12 inches long fitted with 1/2-inch mounting thread, catalog No 5B10S. Manufactured by the Brown Instrument Co., Division of Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Used in conjunction with the Brown precision indicator to determine the operating temperatures in the column.

Thermocouple Lead Wire. Catalog No 5W2A14, for use with type K chromel-alumel thermocouples, weatherproof over polyvinyl insulation, 16 gage. Manufactured by the Brown Instrument Co., Division of the Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Used to extend thermocouples from head assembly to precision indicator.

Transmitter. Mercury manometer type, range 0-54.57 inches of water, transmits 3-15 lb/sq in., gage, indicating type. Manufactured by the Brown Instrument Co., Division of the Minneapolis-Honeywell Regulator Co., Philadelphia, Pa. Presented by the Baltimore Refinery of the Esso Standard Oil Co. Seven used in conjunction with recorders and temperature and flow controllers to measure the flow rates of the terminal streams of the column and off-site equipment.

Trap, Steam. Anderson Super Silvertop, model No 110, capacity 400 lb/hr, operating pressure 80 lb/sq in., gage. Manufactured by the V. D. Anderson Co., Cleveland, Ohio. Used on the feed preheater to facilitate the removal of steam condensate.

<u>Trap, Steam</u>. Anderson Super Silvertop, model No 120, capacity 1320 lb/hr, operating pressure 80 lb/sq in., gage. Manufactured by the V. D. Anderson Co., Cleveland, Ohio. Used on the calandria to facilitate the removal of steam condensate.

<u>Valve, Diaphragm Motor</u>. Size 1/2-inch, 250 lb/sq in., gage, bronze body, 3/8-inch diameter cut-away type inner valve, air to open action, serial No J-1188. Manufactured by the Mason-Neilan Regulator Co., Boston, Mass. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Used to throttle product stream.

<u>Valve, Diaphragm Motor</u>. Size 1/2-inch, Lunkenheimer bronze body, stainless steel trim, single seat cut-away plug type inner valve, air to open action. Manufactured by the Foxboro Co., Foxboro, Mass. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Used to throttle the feed stream.

<u>Valve, Diaphragm Motor</u>. Size 1/2-inch, AE make, Lunkenheimer bronze body, stainless steel trim, cut-away plug, single seat type, air to open action. Manufactured by the Foxboro Co., Foxboro, Mass. Presented by the Union Carbide Chemicals Co.,

Charleston, W. Va. Used to throttle the bottoms discharge stream.

<u>Valve, Diaphragm Motor</u>. Size 1/2-inch, bronze body, stainless steel trim, cut-away type inner valve, air to open action. Manufactured by the Bristol Co., Waterbury, Conn. Used in conjunction with valve positioner to regulate flow of distillate from reflux accumulator drum.

<u>Valve, Diaphragm Motor</u>. Size 1-inch, cast steel body, stainless steel trim, cut-away type inner valve, flange fitting, air to close action. Manufactured by the Foxboro Co., Foxboro, Mass. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Used to throttle steam flow to calandria section of column.

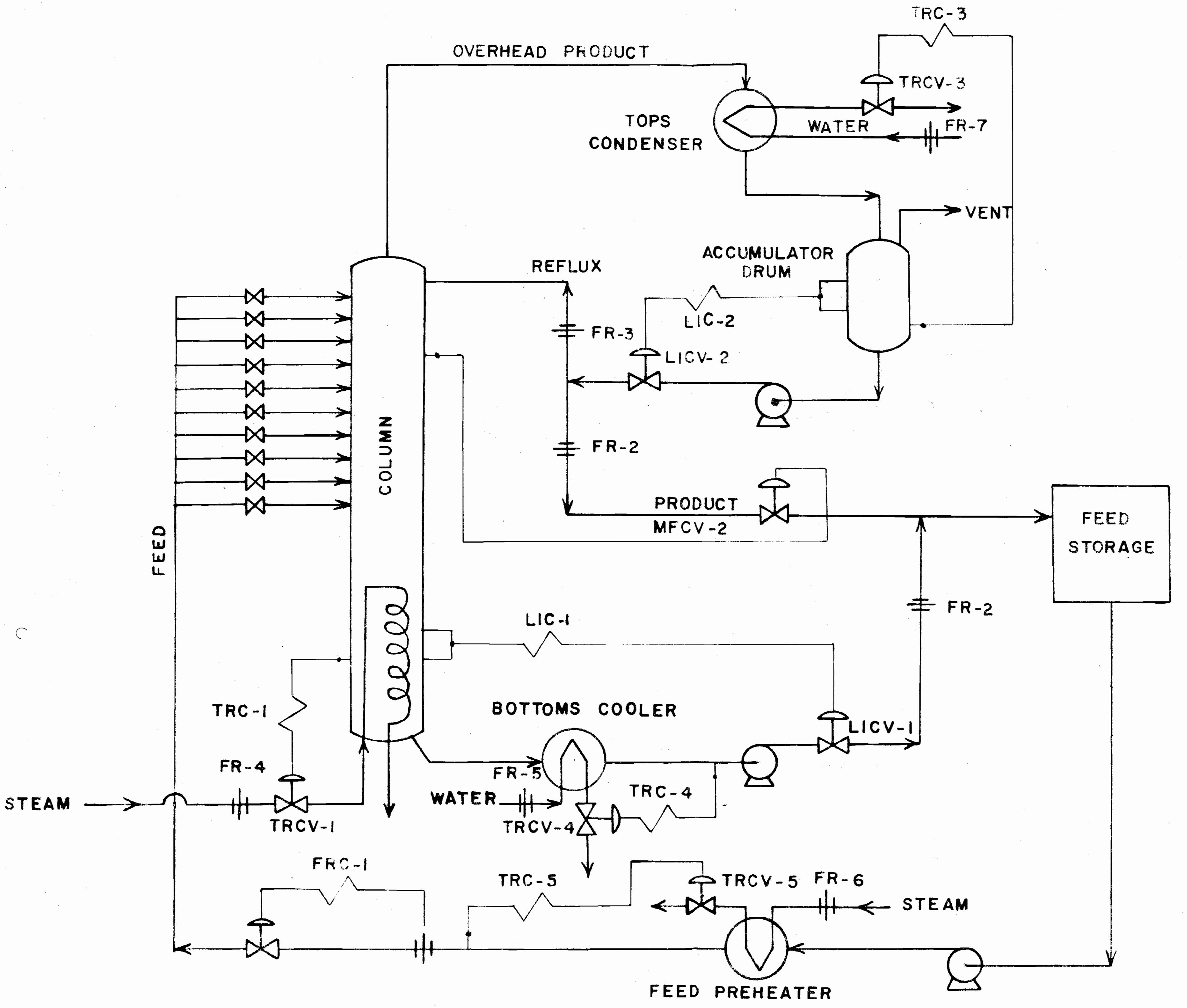
<u>Valve, Diaphragm Motor</u>. Size 1/2-inch, bronze body, stainless steel trim, cut-away plug, single seat type inner valve, air to open action. Manufactured by the Foxboro Co., Foxboro, Mass. Presented by the Union Carbide Chemicals Co., Charleston, W. Va. Three used; one to throttle water flow to bottoms cooler, one to throttle water flow to tops condenser, and one to throttle steam flow to feed preheater.

Method of Procedure

The following paragraphs contain an outline of the procedure used in the preliminary tests on the hydrocarbon fractionating unit, repair of off-site equipment and instruments, and calibration of feed, reflux, product, and bottoms flow rate recorders. A detailed procedure for the operation of the fractionating unit is also included in this section.

Preliminary Tests. As the initial step in determining the condition of the off-site equipment and the instruments, the column, as shown in Figure 4, page 56, was operated under test conditions using water as a feed. Water was used so as to conserve the inventory of petroleum feed stock. In all preliminary tests using water as the feed,total reflux was used at first, after which no reflux was set up. It was not possible to obtain partial reflux until the reflux control valve was changed to the product line.

The instruments, shown in Figure 5, page 57, were checked for response and control, and all flow controllers and liquid level controllers were adjusted so that they operated in the proper range of control. The temperature controllers on the distillate and



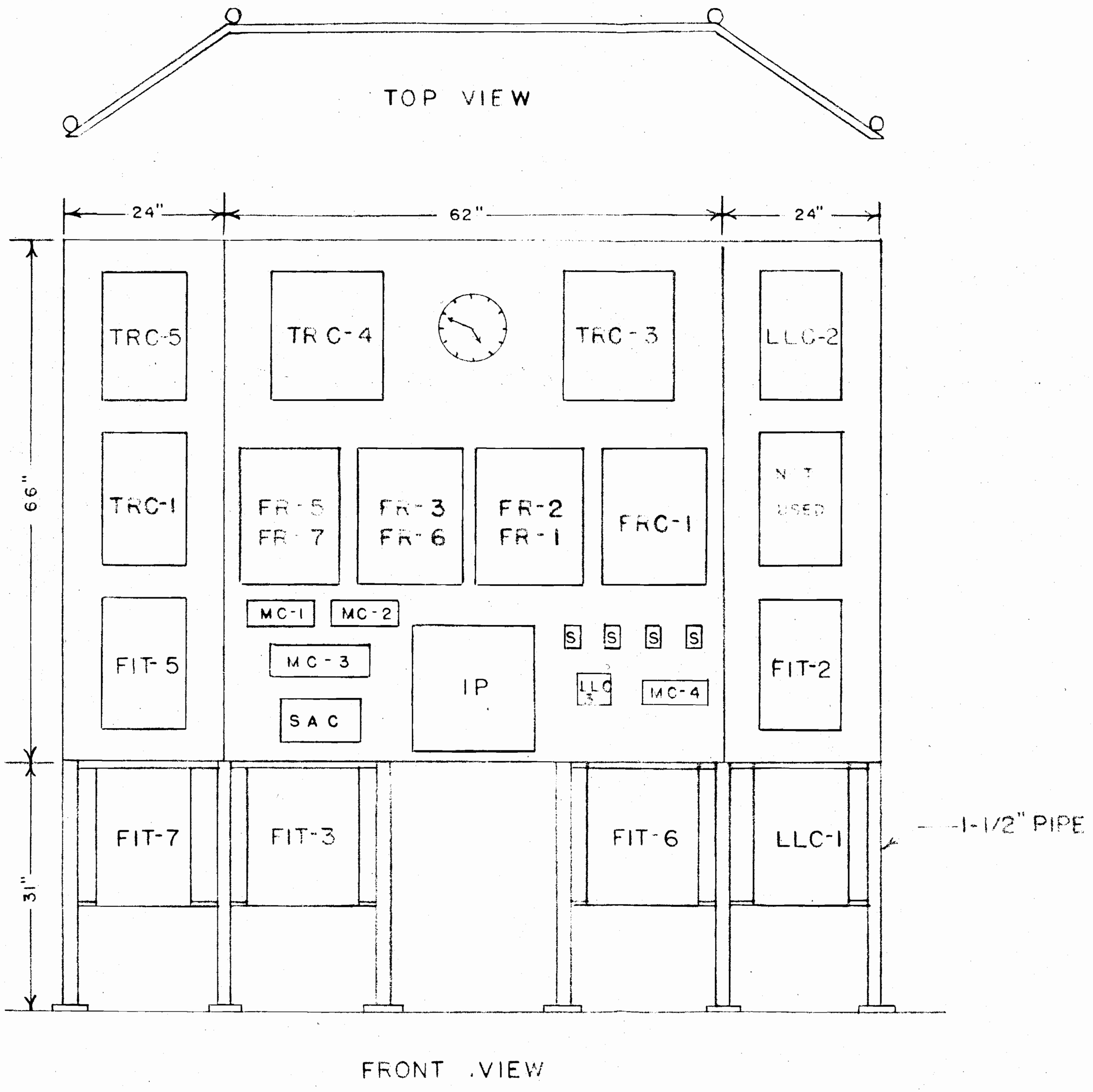
LEGEND

LIC	LEVEL INDICATOR AND CONTROLLER
LICV	LEVEL INDICATOR AND CONTROLLER VALVE
FR	FLOW RECORDER
FRC	FLOW RECORDER AND CONTROLLER
FRCV	FLOW RECORDER AND CONTROLLER VALVE
TRC	TEMPERATURE RECORDER AND CONTROLLER
TRCV	TEMPERATURE RECORDER AND CONTROLLER VALVE
MFCV	MANUAL FLOW CONTROL VALVE

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

INSTRUMENTATION DIAGRAM FOR FRACTIONATING COLUMN AND ACCESSORY EQUIPMENT

SCALE: NONE DATE 6-11 CASE NO: 599
DRAWN BY: W.C. Richard FILE NO: 57
CHECKED BY: FIGURE NO: 4
APPROVED BY: FCV 9/11/57 SHEET NO: 1



LEGEND

FR- FLOW RECORDER
FIT- FLOW TRANSMITTER
TRC- TEMPERATURE RECORDER-CONTROLLER
FRC- FLOW RECORDER-CONTROLLER
LLC- LIQUID LEVEL CONTROLLER
MC- MANUAL CONTROL PANEL
SAC- SUPPLY AIR CONTROL
IP- INDICATING POTENTIOMETER
S- REMOTE MOTOR SWITCH

NOTE

PANEL CONSTRUCTED OF 3/4" PLYWOOD

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

CONTROL PANEL FOR THE
HYDROCARBON FRACTIONATION
UNIT

SCALE: 1" = 16"
DATE 8-28-56
CASE NO: 599
DRAWN BY:
FILE NO: 56
CHECKED BY:
FIGURE NO: 5
APPROVED BY: 8/28/56
SHEET NO: 1

bottoms lines, feed preheater, and column reboiler were not adjusted until tests using the petroleum mixture were made. The reflux flow controller, which controlled the temperature on the seventh plate from the bottom of the column was found to function improperly and was deleted from the system. The reflux flow was thereafter controlled manually by setting the product control valve opening.

Repair of Off-site Equipment and Instruments. The following paragraphs contain an outline of the procedure used in the repair, movement, and replacement of equipment and instruments. These changes include movement of the reflux accumulator drum, product and reflux pump, reflux control valve; replacement of differential converter, accumulator level controller, and accumulator level control valve.

> Reflux Accumulator Drum. Upon operating the column using water as the feed, it was found that the distillate would not flow of its own accord from the condenser to the pump because the pump was located on a higher level than the condenser. This meant that the pump would have to be primed before each test. Therefore, it was deemed

feasible to move the accumulator drum and pump to a position below the condenser, as shown in Figure 6, page 60. As a result of this movement one pump was alleviated from the entire system.

Differential Converter and Accumulator Level Controller. During preliminary tests on the column using water as the feed it was discovered that the differential converter was not functioning properly in the measurement of the differential pressure in the accumulator drum. The instrument was removed and a Foxboro pneumatic D/P Cell was put in its place.

During those same tests it was noticed that instrument LLC-3, as shown in Figure 5, page 57, was not controlling the accumulator level properly. The addition of a new part to the instrument did not augment its operation, so it was replaced by instrument LLC-2.

Reflux Control Valve. During the preliminary tests using a petroleum mixture as the feed, no reflux could be obtained. At first it was thought that the valve was working improperly, but later it was found that, due to the arrangement of the

Figure 6. Piping Diagram of Fractionating Column and Accessory Equipment

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Plan View

(See pocket inside back cover)

Figure 7. Piping Diagram of Fractionating Column and Accessory Equipment

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Southwest Elevation

(See pocket inside back cover)

reflux and product lines, a greater pressure drop occurred in the product line than in the reflux line. Therefore, the reflux control valve was changed to the product line, thus creating more equal pressure distribution in the two lines. After this change the amount of product was controlled, and the remainder of the distillate returned to the column as reflux.

Accumulator Level Control Valve. The control valve that controlled the level in the accumulator drum was found to have an opening too small to permit flow of distillate out of the drum at a high enough rate to maintain the level constant; that is, material was being condensed faster than it was being withdrawn from the reflux accumulator drum. The control valve was replaced by one having a greater pressure drop and capacity.

Calibration of Instruments. The following paragraphs contain an outline of the procedure used in calibrating the feed, bottoms, product, and reflux flow recorder charts; and feed, bottoms, and distillate temperature recording charts.

Feed Flow Rate Recorder Controller. In calibrating the feed flow rate controller chart readings in pounds per minute a petroleum mixture was used. The material was pumped through the feed line to the base of the column where it was collected in a large tub during the entire time of calibration. At one minute time intervals samples of the feed were collected in a bucket, weighed, and recorded along with the chart reading corresponding to the particular flow rate. Eight different settings were automatically obtained on the chart by moving the setpoint, each reading being allowed to stabilize before collecting a sample. A plot was made of flow rate in pounds per minute against setpoint reading on the chart. The recording pen corresponded to the setpoint within ± 0.5 division. At the conclusion of the calibration the material collected in the tub was returned to the feed storage tank through the bottoms line. This was done by closing the gate valve between the bottoms outlet from the column and the bottoms pump, connecting a piece of suction hose to the suction side of the pump, and switching on the pump. The product line and bottoms sample collecting line were closed before the pump was turned on, making certain that the bottoms return line to the storage tank was open.

Bottoms Flow Rate Recorder. The bottoms flow rate recorder was calibrated using a petroleum mixture as the material. The mixture was pumped into the column to a height substantially above the bottoms outlet, pumped out through the bottoms line, and collected in a large tub. Instrument MC-4, as shown in Figure 5, page 57, was put on manual control so that valve LICV-1, the bottoms flow rate control valve, could be manually operated.

For one minute time intervals samples of the petroleum mixture were collected which corresponded to nine different bottoms flow rates. The flow rates were controlled by the valve opening, and after each setting of the valve the flow rate was allowed to become steady before collecting the sample in a bucket. A plot was made of bottoms flow rates, in pounds per minute, versus recorder chart reading. At the conclusion of the calibration the material was returned to the feed storage tank in the same manner as after the feed calibration tests.

Product Flow Rate Recorder. The product flow rate recorder was calibrated using tap water as the material. A continuous supply of water was put into the reflux accumulator drum and pumped out onto the ground through the product line. The ten different flow rates used were set by instrument MC-2. The accumulator level control valve was by-passed and the reflux line was manually shut off. Samples of water flowing through the product line were collected over one minute time intervals for each valve setting and the flow rates in pounds per minute were plotted against the product chart reading.

Reflux Flow Rate Recorder. Calibration of the reflux flow rate recorder was conducted by pumping water from the accumulator drum through the reflux line onto the ground. The accumulator level control valve was by-passed and the product line was completely closed before calibration began. The reflux sample globe valve was manually operated, eight different settings being made. Samples, measured in pounds per minute, were collected, over one minute time intervals, in a bucket, weighed, and recorded along with the recorder chart readings. A plot was prepared of flow rates versus chart readings.

Feed, Bottoms, and Distillate Temperature Recorders. The temperature recorders which record the feed, bottoms, and distillate temperature were calibrated simultaneously using a constant temperature water bath obtained from the Chemical Engineering Department, Virginia Polytechnic Institute. A standard ASTM thermometer was used in the bath with the thermocouples. Ten different temperature readings and their corresponding chart readings were recorded. Plots were made of temperatures, in °F, versus temperature recorder chart readings.

Thermocouples Located on Column Plates. The readings of the thermocouples that measure the temperatures on each plate in the column were found to correspond closely enough with thermometer readings so that calibration curves were deemed unnecessary.

Operating Procedure. The following paragraphs contain a detailed procedure of the startup, continuous operation, and shutdown of the fractional distillation column. During all eight tests the column was operated under essentially atmospheric pressure. The only difference in operation occurred when charges in the setpoints of the variables was necessary.

Startup Procedure. The first consideration in the startup procedure was to be certain that the Nash Hytor was in operation, supplying the panel board with at least 40 pounds per square inch, gage, air pressure. The next step was to set the supply air control, as shown in Figure 5, page 57, at a reading of 18 pounds per square inch, gage, supply pressure on the instruments side of the pressure reducing valve. Since there was a discrepancy in the readings of the supply air control pressure and the instrument supply pressure, as indicated by readings on the instrument pressure gages, a setting of 18 pounds per square inch, gage, supply pressure actually supplied the instruments with approximately 20 pounds per square inch, gage, pressure.

After the air supply to the instruments was fixed, the electric clocks which operate instruments FRC-1, FR-1, FR-2, FR-3, FR-5, FR-6, and FR-7, as shown in Figure 5, page 57, were turned on. The instruments TRC-1, TRC-3, TRC-4, TRC-5, and LLC-1 contain hand wind clocks, which were also started. The instrument charts were next installed according to the following arrangement: on all Minneapolis-Honeywell Brown instruments, charts with No 12501 were placed; charts with No OP3320 were placed on all Taylor instruments; and on the Foxboro instruments, charts with No 898442 were placed.

The next operation included manually turning on the water line valve which is located to the rear and to the right of the panel board, directly above the feed flow rate indicator. Immediately following this, the instruments TRC-3, TRC-4, and MC-2, shown in Figure 5, page 57, were changed to manual control, and the output air pressure was adjusted to 18, 5, and 13 pounds per square inch, gage, respectively. The indicating potentiometer was switched on at this point, and after the tubes warmed up for about one minute the instrument was standardized according to the procedure outlined on the inside face of the instrument.

Following the potentiometer standardization, the steam valve which allows steam flow to the control valve, TRCV-1, as shown in Figure 4, page 56, was moved one-quarter turn to the left, manually, making certain that all the water was drained out inside the building through the steam drain before closing the drain and turning the valve full open; the steam valve is located to the right and behind the panel board, directly above the reflux flow indicator.

After allowing the steam to flow from inside the building to the control valve on the outside, and making certain that the setpoint on instrument TRC-1 was zero, as indicated by Figure 5, page 57, a feed flow rate was set by moving the setpoint on instrument FRC-1 to the predetermined value of 90. The setpoint of 90 was used so as to reduce the time necessary for filling the column reboiler section. The feed pump motor switch was then turned on. The feed was entering the column in the vapor space above plate five.

As soon as the level in the column transmitted a reading of eight on the chart of instrument LLC-1, as shown in Figure 5, page 57, the bottoms pump was switched on and the setpoint on instrument TRC-1 was moved to 125, corresponding to a reboiler temperature of 257 °F. Steam was now flowing into the calandria section of the column. The feed flow rate setpoint on instrument FRC-1 was changed to 65, the setpoints on instruments LLC-1, LLC-2, TRC-3, and TRC-5, as shown by Figure 5, page 57, were changed to 8, 18, 91, and 130, respectively.

When the temperature on plate nine reached 160 °F, as indicated by the potentiometer, and the level in the reflux accumulator, as recorded by instrument LLC-2, rose to the control point of 18, the accumulator pump was switched on. The next operation was to put instrument TRC-3, as shown in Figure 5, page 57, on automatic control, adjusting the setpoint until a reading of 95 was recorded. The column was then operating automatically and no more changes were made until shutdown.

Continuous Operation. After the final adjustments were made on instrument settings, as indicated in the previous paragraph, the column flow rates, temperatures, and liquid levels were allowed to become steady. The column remained at steady state for a period of at least 30 minutes before any samples were taken. Samples were then taken of the feed, bottoms, and product and analyzed by means of an Abbé refractometer. At the end of another 30-minute period, samples of the same streams were taken and analyzed as before. This same procedure was repeated once again in order to test for constancy of composition. The column had thus far been operating at steady state for one and one-half hours, at which time liquid samples were taken from each plate, and from the feed, bottoms, and product lines. The test was then concluded and shutdown began.

Shutdown Procedure. At the conclusion of the test, the first objective in the shutdown procedure was to make certain that steam to the calandria section was shut off. This was accomplished by changing the setpoint of instrument TRC-1 from 125 to zero, and also by manually shutting off the steam valve inside the building behind the panel board, directly above the reflux flow indicator. The next consideration was to move the setpoint of instrument FRC-1 back to zero and shut off the feed pump. As soon as the temperature on the ninth plate from the bottom of the column reached 150 °F, the reflux and bottoms pumps were shut off. In this paragraph reference was made to Figure 5, page 57.

After the pumps were shut off, the condenser and cooling water valve inside the building behind the panel board, directly above the feed flow indicator was manually closed. The electric clocks of instruments FRC-1, FR-1, FR-2, FR-3, FR-5, FR-6, and FR-7 were stopped, all the charts were removed, and the potentiometer was cut off. The supply air control valve was shut off, thus removing all air supply to the instruments. The Nash Hytor was stopped, completing the shutdown procedure.

Sample Analysis. The samples that were taken during the eight tests were analyzed by means of an Abbé refractometer, using normal hexane, distilled by Phillips Petroleum Company, and heavy virgin naphtha, produced by Esso Standard Oil Company, as references. According to the literature(21), the refractive indices of hydrocarbon mixtures exhibit a straight line relationship with composition by volume. This principle was utilized in drawing Figure 8, page 74, since this system was considered as a two-component system.

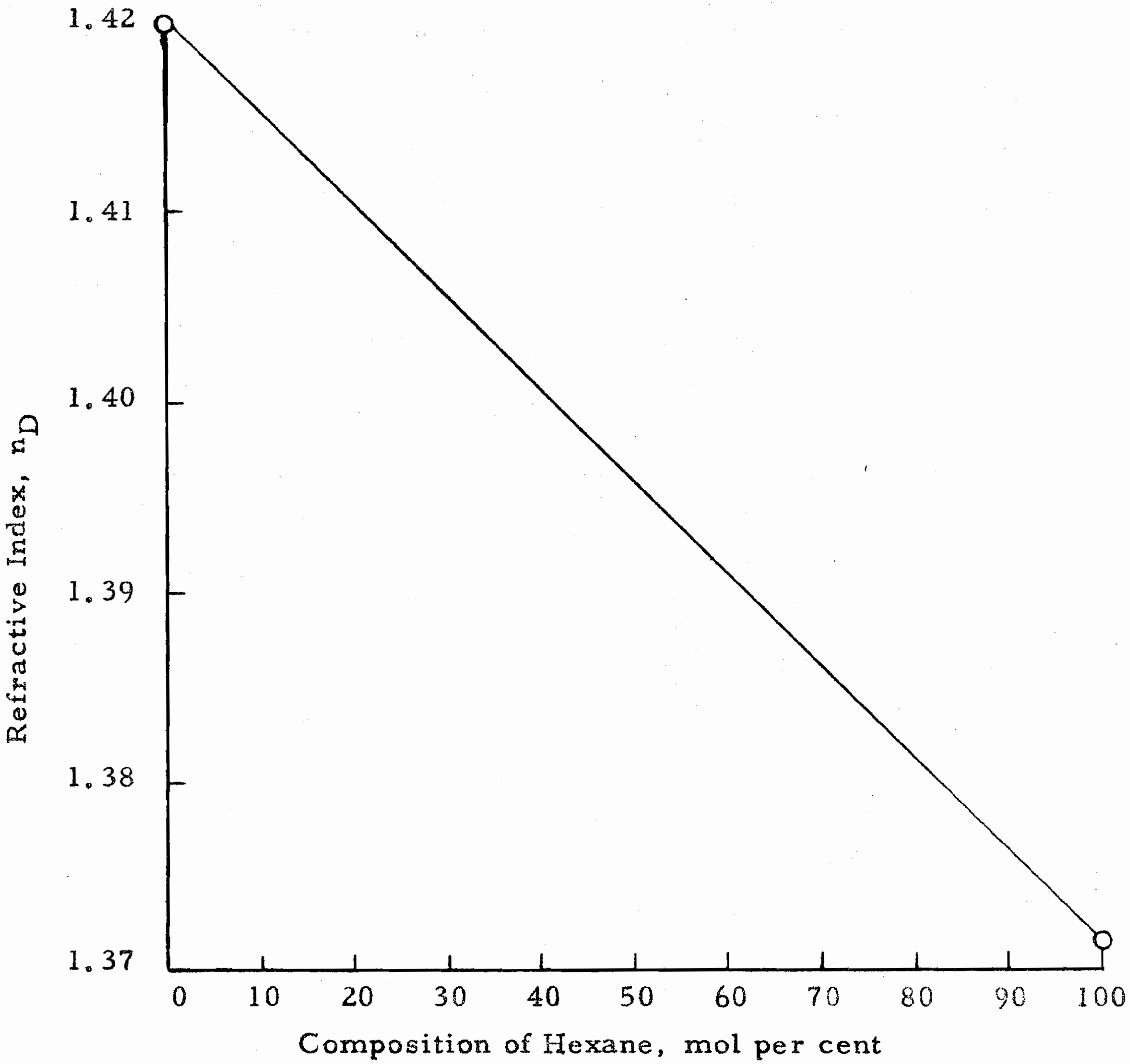

Figure 8 . Refractive Indices for Mixtures of Hexane(1) and Heavy Virgin Naphtha(2)

(1) Hexane used was distilled to 99.5 per cent purity by Phillips Petroleum Company

(2) Boiling range of Heavy Virgin Naphtha- 220-324 °F.

Sample Temperature- 25 °C.

Data and Results

The following section contains a presentation of the data and results involved in the operation of the hydrocarbon fractionating unit.

Analysis of Hexane and Heavy Virgin Naphtha. The analysis of the hexane and the heavy virgin naphtha used in the feed stock is presented in Table I, page 79. The analysis is given in weight per cent.

Physical Properties of Hexane and Heavy Virgin Naphtha. The physical properties of the components of the feed stock are given in Table II, page 70. The properties include distillation curve data.

Flow Recorder Calibration Data. The data amassed in the calibration of feed flow controller setpoint and the bottoms, product, and reflux flow recorders are presented in Table III, page 83. All flow rates are expressed in pounds per minute.

Calibration of Temperature Recorders. The data taken during the calibration of the feed, bottoms, and product temperature recorders are given in Table IV, page 88. All temperatures are expressed in °F.

Equilibrium Curve Data. The equilibrium data calculated for the system hexane and heavy virgin naphtha are tabulated in Table V, page 92.

Operating Data and Results. The operating data and results for the hydrocarbon fractionating unit are presented in Table VI, page 95.

Compositions of Liquid Samples. The compositions of liquid samples from each plate taken during the tests on the hydrocarbon fractionating unit are presented in Table VII, page 96.

Individual Plate Efficiencies. The individual plate efficiencies for the tests on the fractionating column are tabulated in Table VIII, page 97. The Murphree method of calculation was used for all tests.

Distillation Curve for Hexane. Figure 9, page 81, is a graphical representation of the distillation curve for the commercial grade hexane used in the feed mixture.

Distillation Curve for Heavy Virgin Naphtha. Figure 10, page 82, is a graphical representation of the distillation curve for the heavy virgin naphtha used in the feed mixture.

Feed Flow Controller Setpoint Calibration. Figure 11, page 84, is a graphical representation of the data taken during calibration of the feed flow rate controller setpoint.

Bottoms Flow Rate Recorder Calibration. Figure 12, page 85, is a graphical representation of the calibration data for the bottoms flow recorder.

Product Flow Rate Recorder Calibration. Figure 13, page 86, is a graphical representation of the data amassed in calibrating the product flow recorder.

Reflux Flow Rate Recorder Calibration. Figure 14, page 87, is a graphical representation of the calibration data obtained for the reflux flow recorder.

Feed Temperature Recorder Calibration. Figure 15, page 89, is a graphical representation of the data obtained for the calibration of the feed temperature recorder.

Bottoms Temperature Recorder Calibration. Figure 16, page 90, is a graphical representation of the data amassed during the calibration of the bottoms temperature recorder.

Product Temperature Recorder Calibration. Figure 17, page 91, is a graphical representation of the calibration data for the product temperature recorder.

Equilibrium Curve for Hexane and Heavy Virgin Naphtha. Figure 18, page 93, is a graphical representation of the liquid-vapor equilibrium data calculated for the system hexane and heavy virgin naphtha.

Boiling Temperature Versus Mol Per Cent Hexane Curve. Figure 19, page 94, is a graphical representation of the assumed boiling points and calculated hexane compositions for mixtures of hexane and heavy virgin naphtha.

TABLE I

Analysis of Commercial Grade Hexane and Heavy Virgin Naphtha

Heavy Virgin Naphtha(1)		Hexane(2)	
Compound	Weight Per Cent	Compound	Weight Per Cent
Benzene	0.2	nC_6	56.3
Toluene	2.0	$3MeC_5$	13.4
C_8 Aromatics	6.6	nC_4	0.5
C_9 Aromatics	3.6	$i\text{-}C_5, nC_5$	0.3
C_{10} Aromatics	0.6	2, 2 diMe C_4	trace
Naphthalenes	0.2	Cyclo C_5, $2MeC_5$, 2, 3 diMe C_4	8.9
Indan	0.1		
Naphthenes	35.1	MeCyclo C_5, 2, 2 diMe C_5, 2, 4 diMe C_5	14.4
Paraffins	47.7		
Condensed naphthenes	3.9	Cyclo C_6, 3, 3 diMe C_5, 2, 3 diMe C_5	2.3
		Benzene	4.2

(1) Analysis by mass spectrophotometry

(2) Analysis by gas-liquid partition chromatography

Knox, W. T.: Personal Communication, March 29, 1957,
Linden, N. J.

TABLE II

Physical Properties of Commercial Grade Hexane and Heavy Virgin Naphtha

Volume Distilled %	Boiling Point(1) °F	
	Hexane	Heavy Virgin Naphtha
0	148	220
10	149	244
20	150	252
30	150	258
40	151	264
50	151	271
60	151	278
70	152	285
80	152	295
90	152	309
Molecule weight	86	120
Specific gravity, 25 °C	0.684	0.753
Specific heat, Btu/lb-°F Liquid	0.565	0.607
Latent heat of vaporization, Btu/lb	138	152
Refractive index, n_D	1.3715(2)	1.4200

(1) Atmospheric pressure, 28.18 inches mercury

(2) Hexane sample was distilled by Phillips Petroleum Company to 99.5 per cent purity

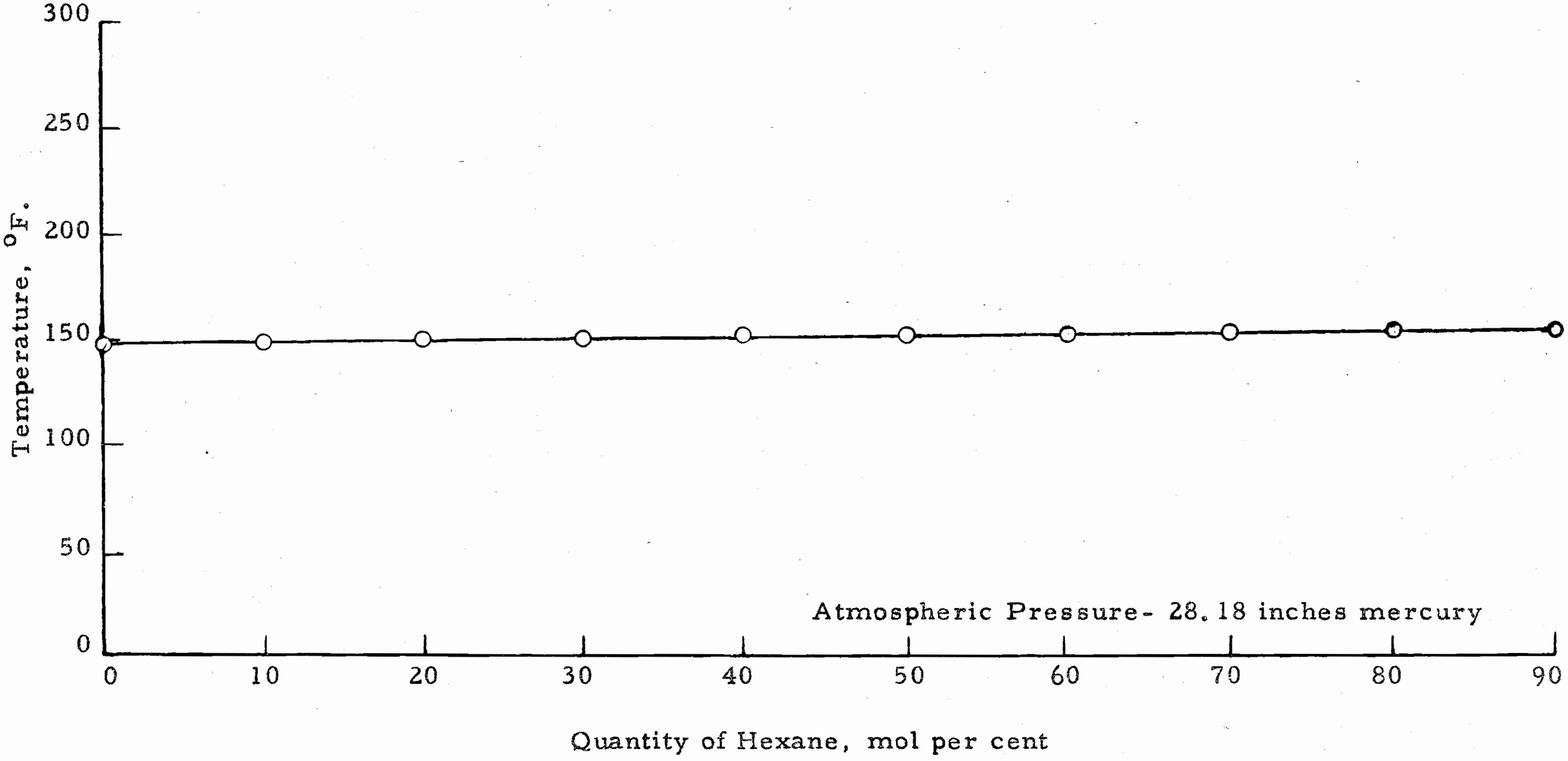

Figure 9. Distillation Curve for Commercial Grade Hexane

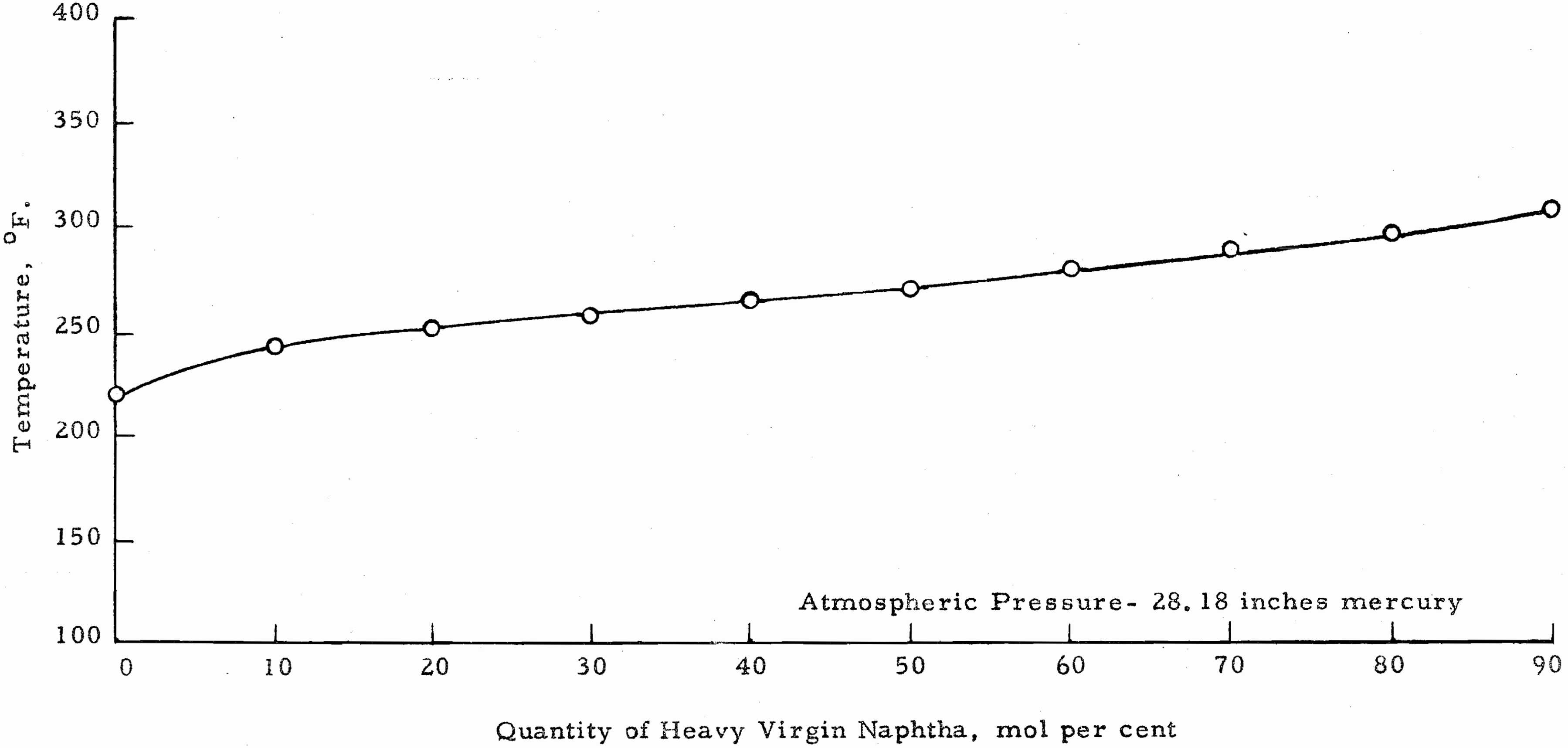

Figure 10. Distillation Curve for Heavy Virgin Naphtha

TABLE III

Calibration of Feed Flow Controller Setpoint and Bottoms, Product, and Reflux Flow Recorders for the Hydrocarbon Fractionating Unit

Feed		Bottoms		Product		Reflux	
Chart Value	Flow Rate lb/min	Chart Value	Flow Rate lb/min	Chart Value	Flow Rate lb/min	Chart Value	Flow Rate lb/min
10.0	15.63	14.0	5.44	22.0	7.85	15.0	9.78
20.0	17.00	22.0	6.81	36.0	11.46	27.5	12.50
30.0	18.13	29.5	8.00	42.0	11.67	40.5	14.85
40.0	19.00	40.0	9.25	46.0	12.23	41.5	15.07
50.0	20.00	51.0	10.88	55.5	13.32	50.5	16.50
60.0	20.50	56.0	11.25	62.5	14.22	61.0	18.00
70.0	22.13	70.5	13.69	72.0	15.27	74.5	20.00
80.0	22.50	86.5	14.88	80.0	16.00	99.5	22.52
--	--	--	--	88.0	16.78	--	--
--	--	--	--	91.0	17.03	--	--

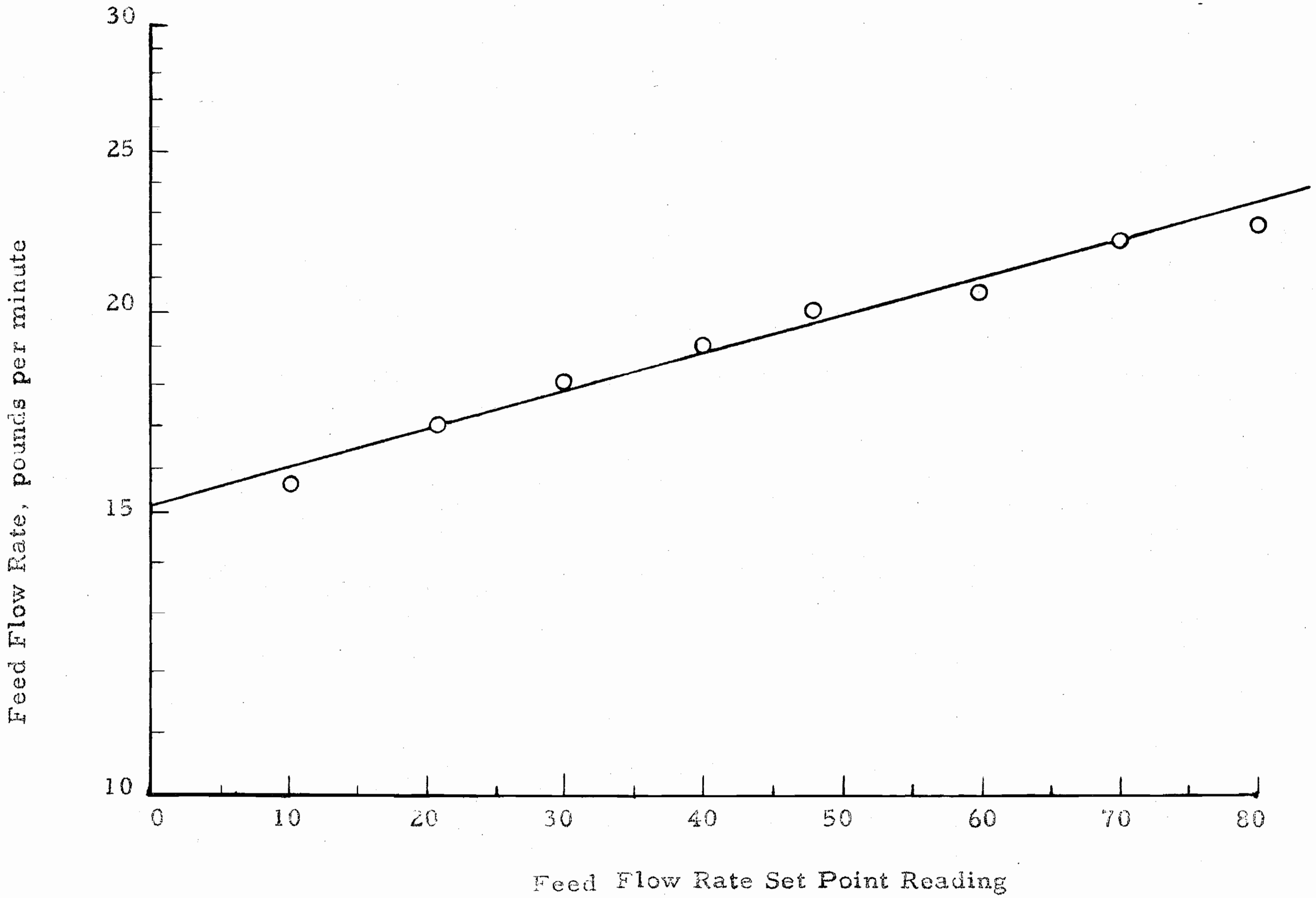

Figure 11. Calibration of Feed Flow Rate Controller Set Point

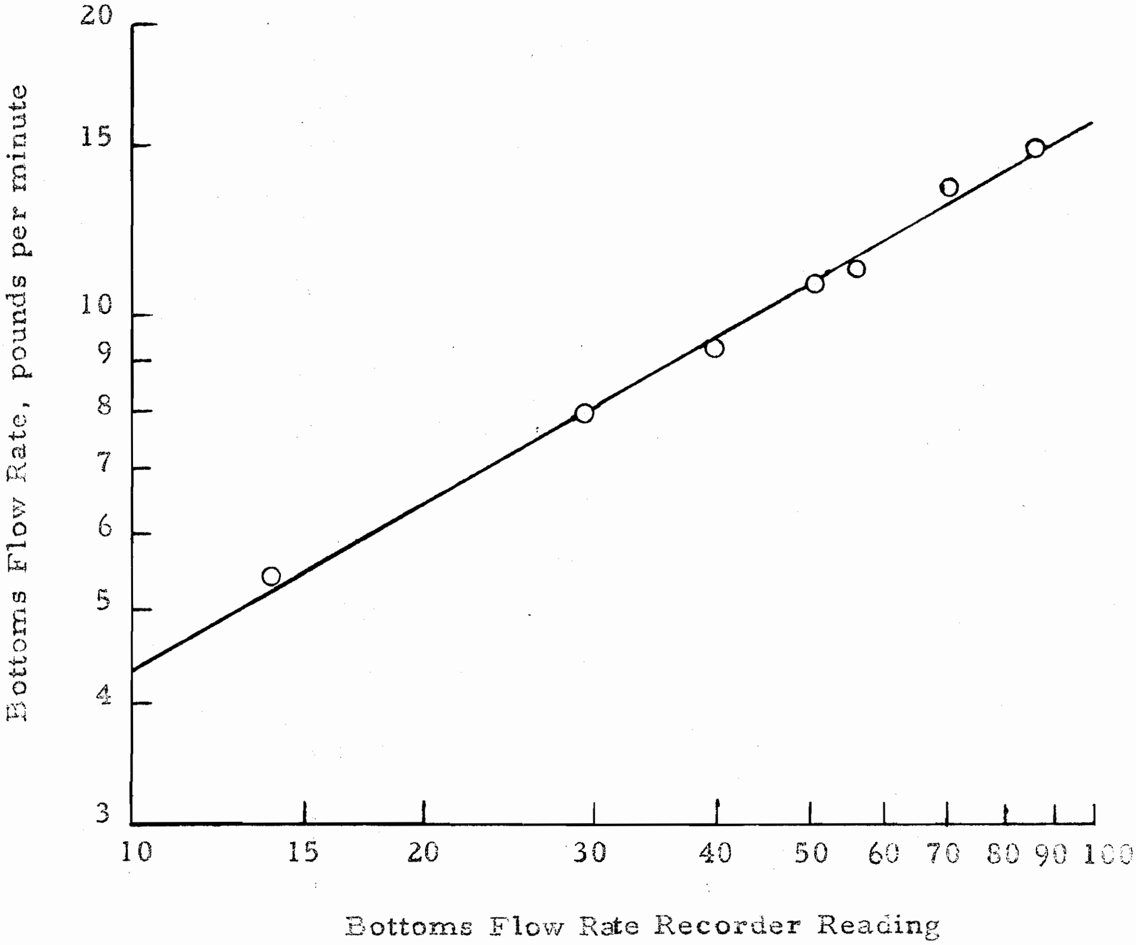

Figure 12. Calibration of Bottoms Flow Rate Recorder

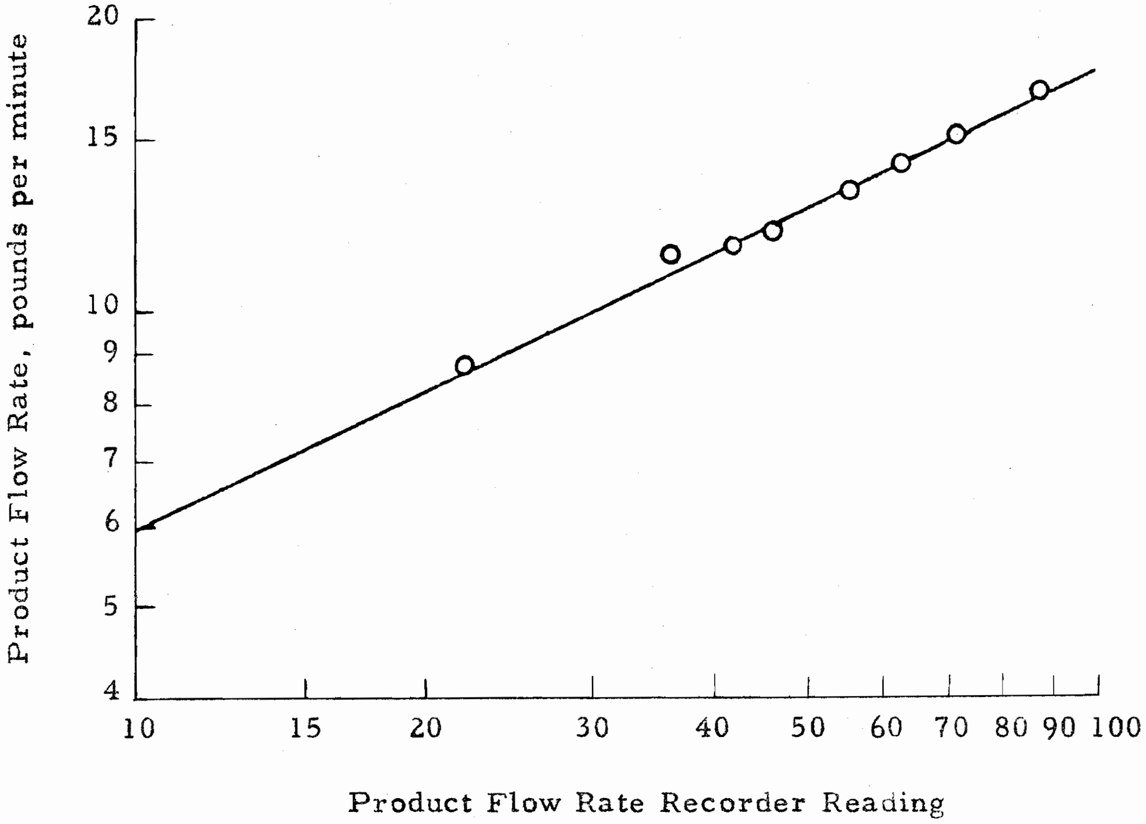

Figure 13. Calibration of Product Flow Rate Recorder

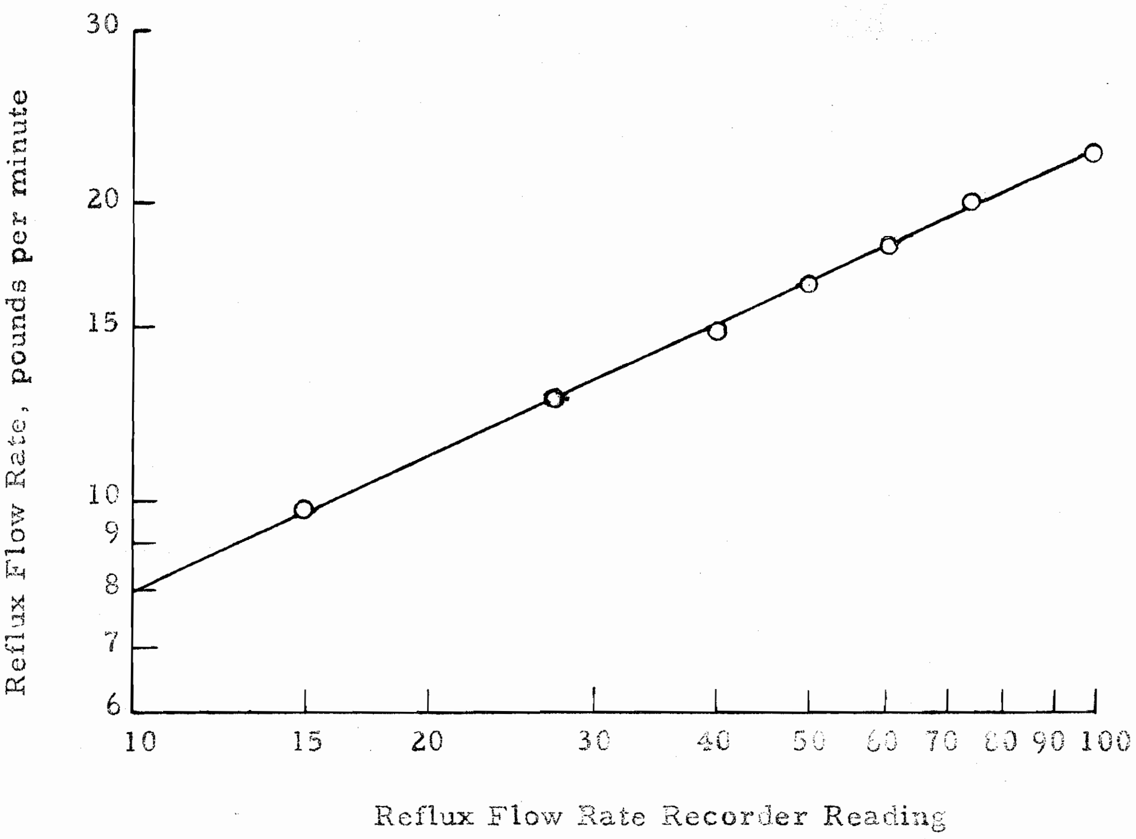

Figure 14. Calibration of Reflux Flow Rate Recorder

TABLE IV

Calibration of Feed, Bottoms, and Product Temperature Recorders for the Hydrocarbon Fractionating Unit

Temperature	Chart Value		
°F	Feed	Bottoms	Product
91	---	64	72
100	117	68	75
108	118	73	77
110	124	74	79
118	130	78	83
122	132	82	---*
131	135	86	87
149	145	95	97
158	150	99	102
165	156	103	107
168	158	106	108

* Thermocouple lead line broken

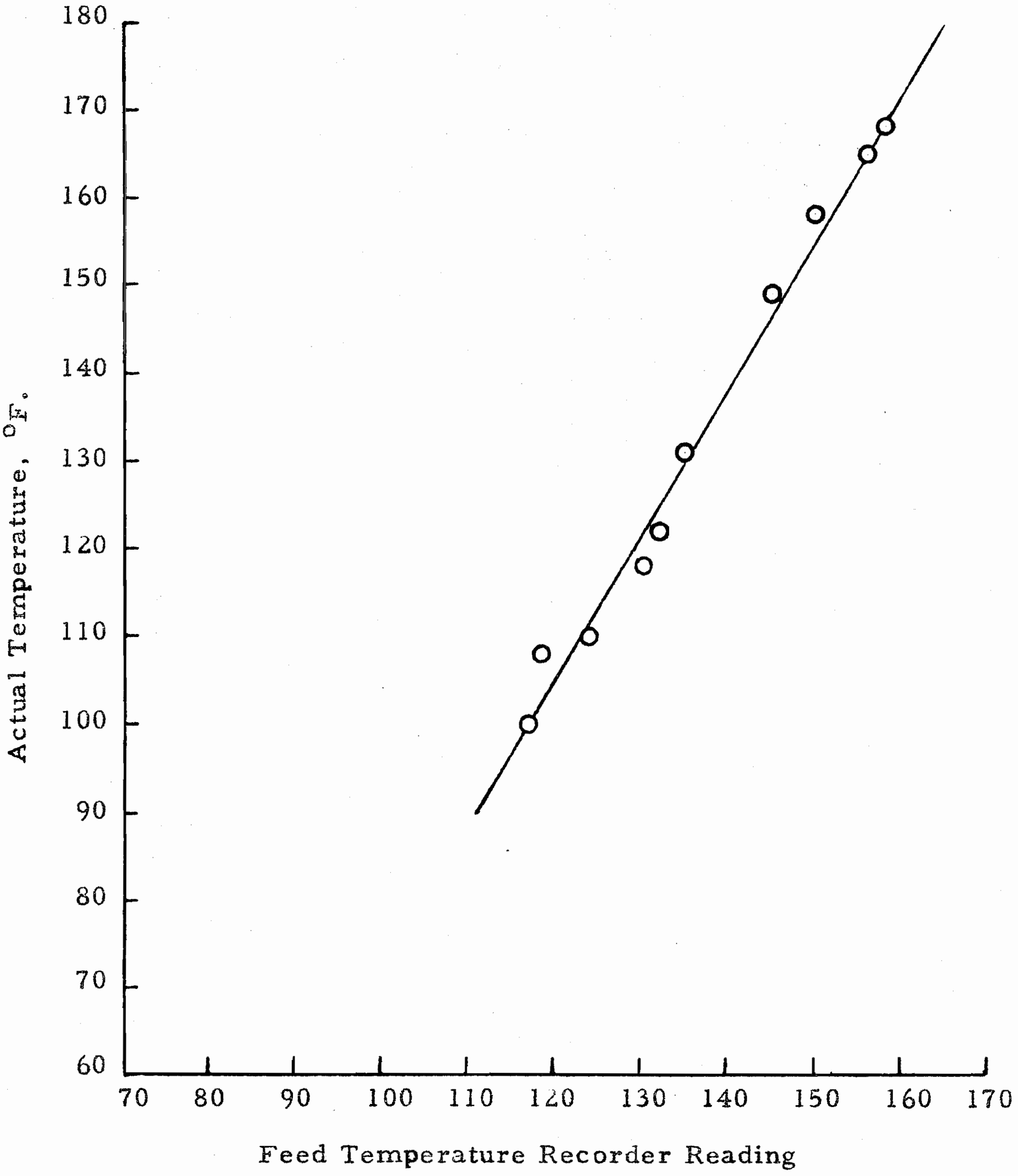

Figure 15. Calibration Curve for Feed

Temperature Recorder

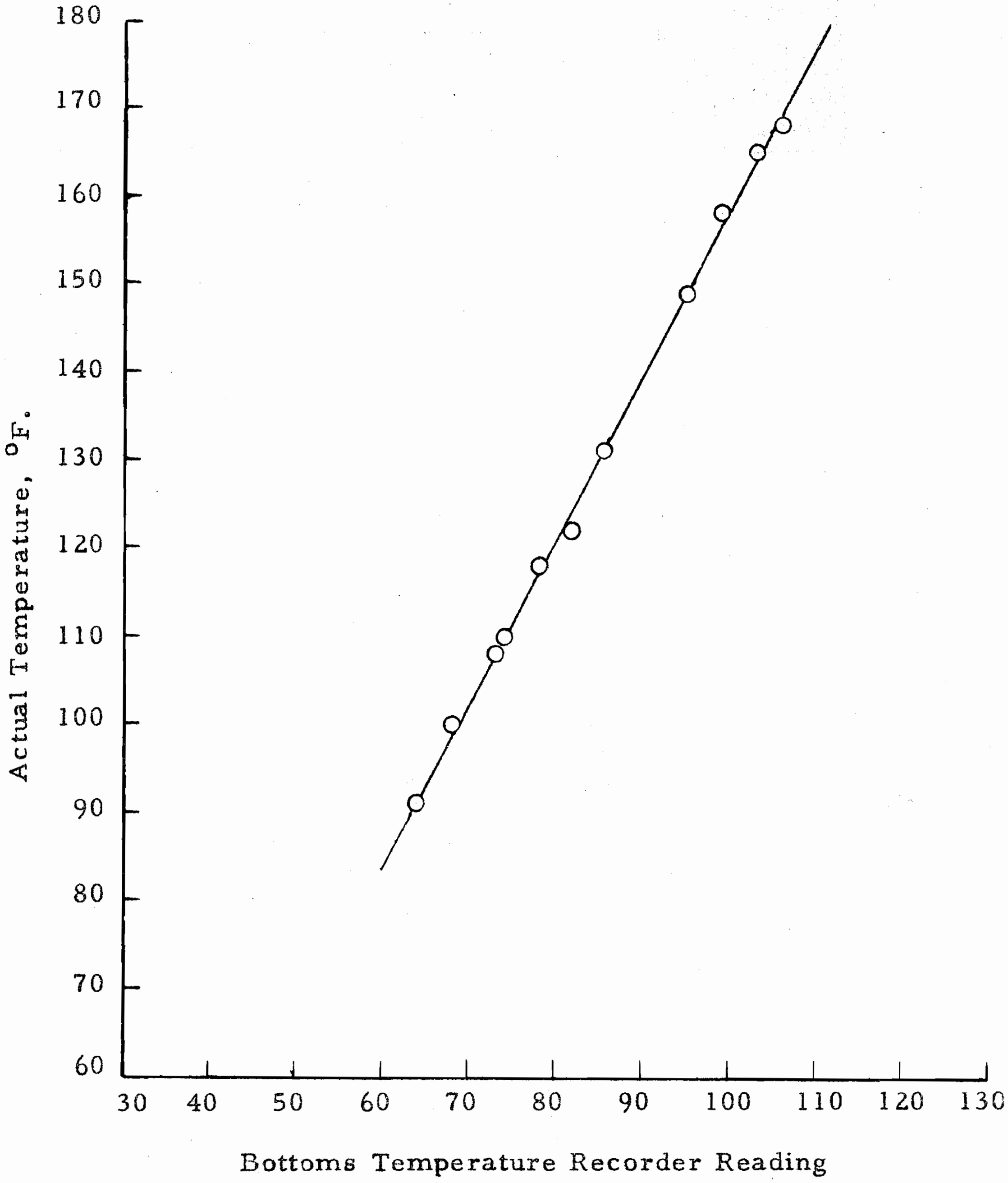

Figure 16. Calibration Curve For Bottoms Temperature Recorder

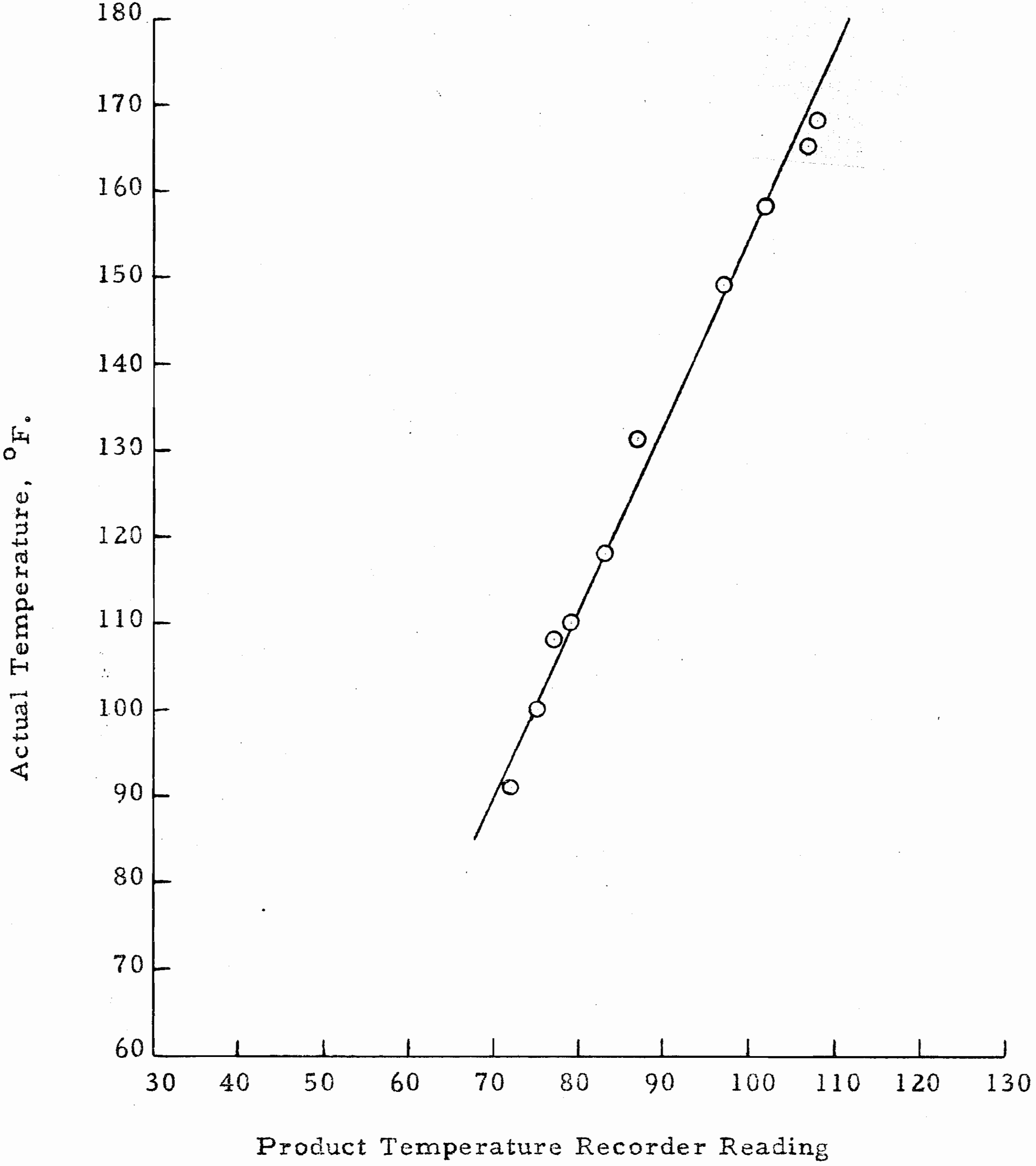

Figure 17. Calibration Curve for Product Temperature Recorder

TABLE V

Equilibrium Curve Data for Mixtures of Hexane and Heavy Virgin Naphtha

Temperature	Mol Per Cent Hexane	
°F	Liquid	Vapor
156	88.1	98.3
160	72.7	95.8
175	64.7	93.0
190	48.1	85.8
210	30.2	72.2
230	16.3	51.9
250	5.9	25.0
260	1.8	8.6

Atmospheric pressure, 0.94 atmosphere

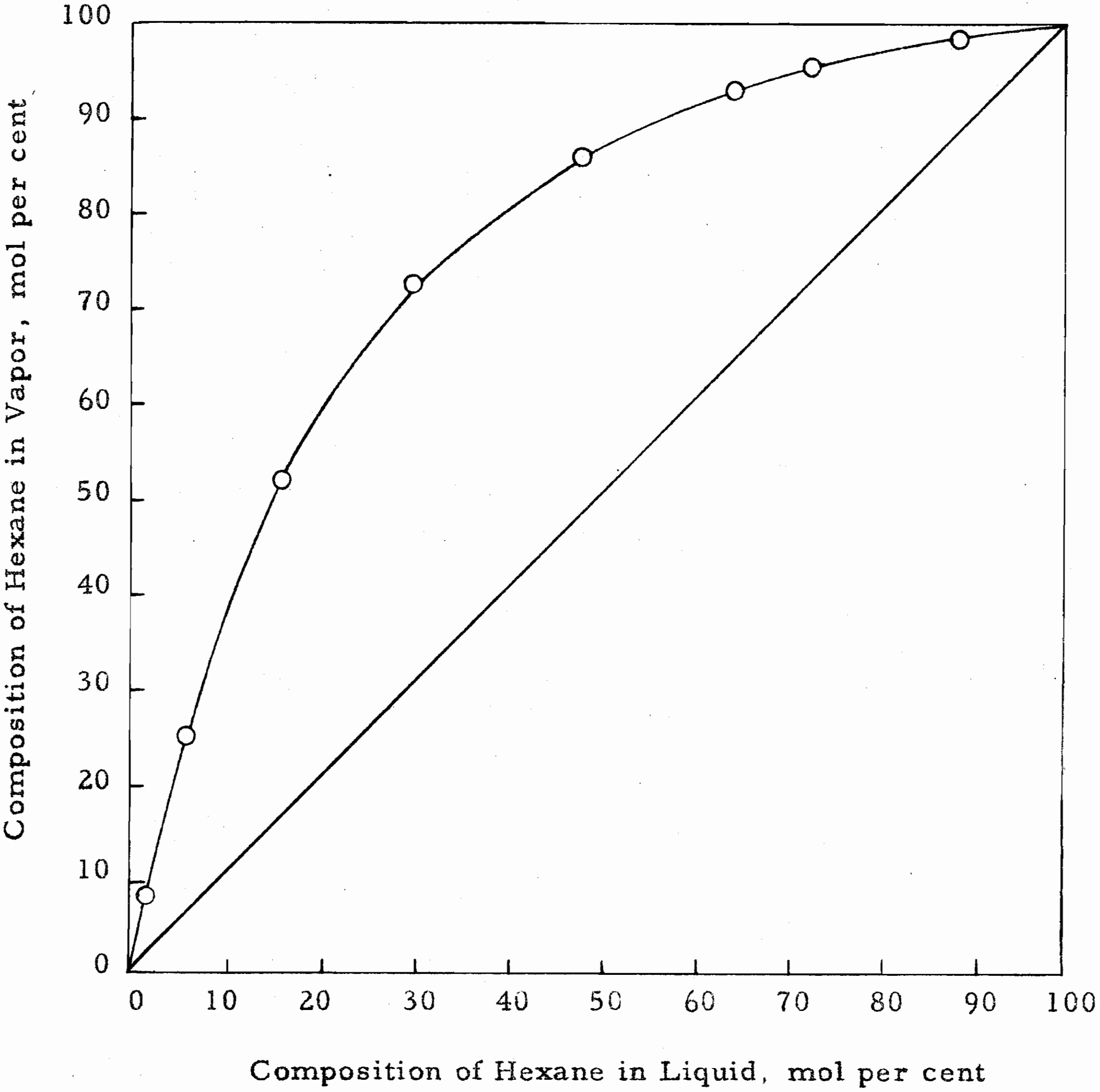

Figure 18. Equilibrium Curve for the System

Hexane and Heavy Virgin Naphtha

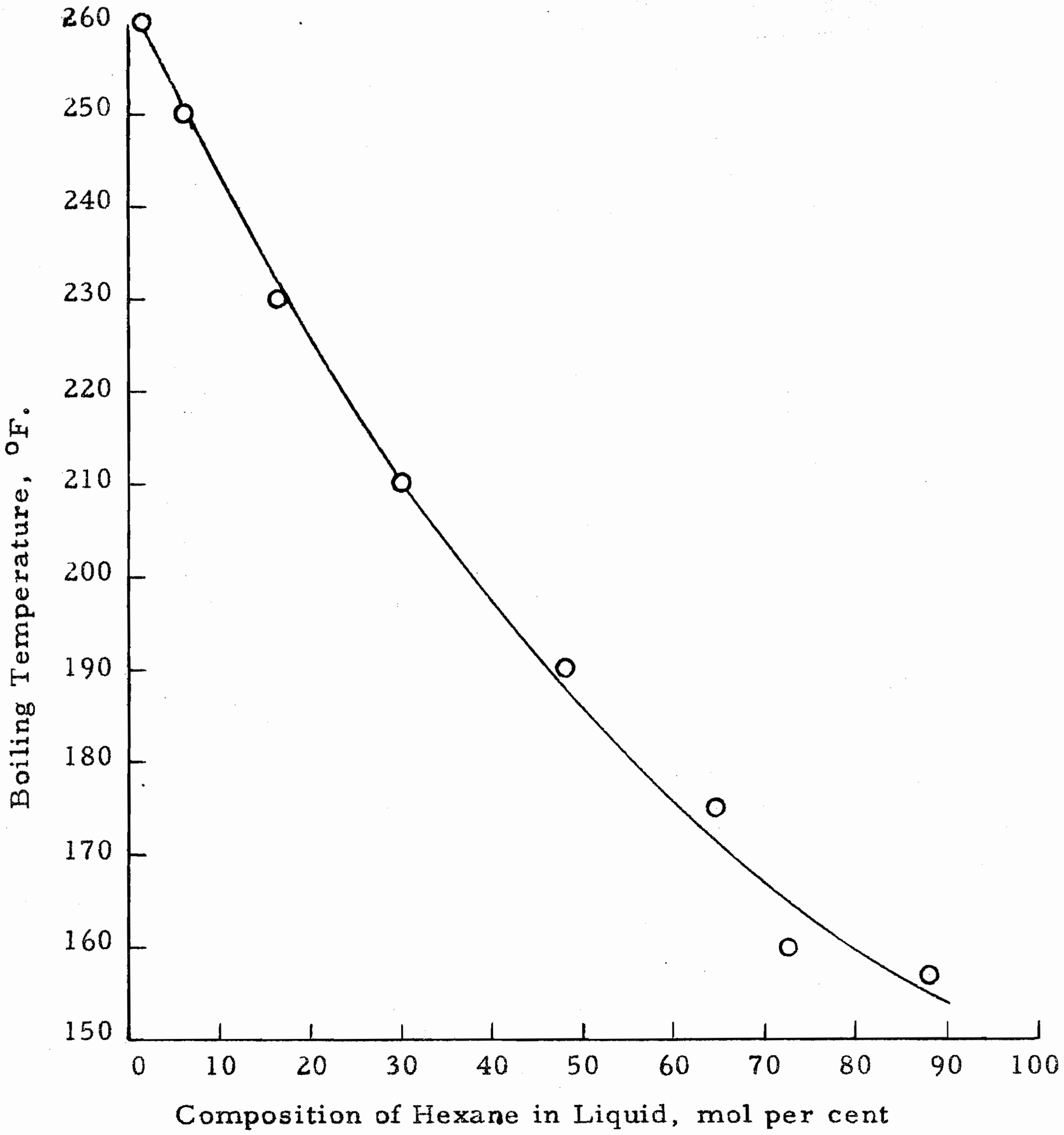

Figure 19. Boiling Temperature Versus Composition of Hexane for Mixtures of Hexane and Heavy Virgin Naphtha

TABLE VI

Operating Data and Results for the Hydrocarbon Fractionating Unit Using a Mixture of Hexane and Heavy Virgin Naphtha

	Test Number							
	1	2	3	4	5	6	7	8
Flow Rates								
mol/min x 10								
Feed	2.04	2.04	1.62	1.69	2.12	2.12	1.66	1.64
Bottoms	0.85	0.68	0.46	0.36	0.47	0.57	0.47	0.36
Product	1.14	1.26	0.99	1.38	1.61	1.54	0.98	1.28
Reflux	2.81	2.97	2.71	0.92	1.69	1.51	2.60	0.82
Temperature								
°F								
Feed	141	146	142	141	141	141	141	141
Distillate	148	151	147	145	146	146	146	146
Column Reboiler	257	275	275	275	275	257	257	257
Mol Per Cent Hexane								
Feed	46.0	47.0	48.5	59.8	55.7	55.5	56.0	51.5
Bottoms	5.3	2.5	0.3	0.0	0.3	2.0	0.0	5.5
Distillate	79.3	80.5	80.5	75.3	75.0	78.0	78.0	79.5
Hexane Balance Discrepancy								
%	+0.7	+7.6	+1.9	+3.0	+2.3	+3.3	-16.6	+23.0
Number Theoretical Plates	2.80	3.35	5.00	--	5.00	3.50	--	2.83
Overall Column Efficiency								
%	18.0	23.5	40.0	--	40.0	25.0	--	18.3
Product Valve Pressure								
lb/sq in., gage	13	13	13	15	15	15	13	15

TABLE VII

Compositions of Liquid Samples from Hydrocarbon Fractionating Unit

	Mol Per Cent Hexane							
Plate Number (2)	Test Number (1)							
	1	2	3	4	5	6	7	8
1	37.5	34.0	26.0	18.0	25.0	33.5	27.0	39.5
2	42.7	36.0	32.0	20.5	26.8	36.0	31.0	41.0
3	53.6	51.5	40.0	41.2	46.8	53.5	47.0	54.5
4	55.7	52.0	53.5	43.5	51.8	56.5	52.0	57.5
5	52.0	53.5	54.0	54.8	59.8	67.0	57.0	57.5
6	70.0	68.5	67.0	59.4	64.0	71.5	65.5	69.5
7	74.5	74.0	71.5	64.0	69.0	71.5	68.5	74.0
8	76.0	74.0	74.5	65.5	70.0	73.5	71.0	74.0
9	79.0	78.0	75.5	71.3	72.0	75.0	74.0	76.5
10	79.4	77.5	77.5	71.3	74.0	76.5	76.0	78.0

(1) Test conditions described in Table VI, page 95

(2) Plates numbered from the bottom of the column

TABLE VIII

Individual Plate Efficiencies for Hydrocarbon Fractionating Unit Using a Mixture of Hexane and Heavy Virgin Naphtha

	Murphree Plate Efficiencies %							
Plate Number (2)	Test Number (1)							
	1	2	3	4	5	6	7	8
1	19.0	8.3	25.1	12.0	9.5	10.2	18.6	6.1
2	34.2	30.1	30.2	75.0	67.5	55.6	51.7	51.0
3	6.4	1.6	41.8	7.4	15.7	9.4	15.1	10.5
4	-12.3	4.8	1.5	35.5	22.6	28.4	14.5	0.0
5	20.0	38.7	33.5	14.0	11.1	11.6	21.2	33.0
6	11.0	13.5	11.2	13.0	12.4	0.0	7.5	11.5
7	3.6	0.0	7.3	4.1	2.4	5.1	4.5	0.0
8	7.2	9.7	2.4	14.6	4.8	3.7	7.3	6.3
9	1.0	-1.2	4.8	0.0	4.8	3.7	4.9	3.7
10	0.0	7.2	7.2	9.4	2.4	3.6	4.8	35.7

(1) Test conditions described in Table VI, page 95

(2) Plates numbered from the bottom of the column

Sample Calculations

The following paragraphs contain samples of the calculations involved in determining the overall column and individual plate efficiencies for the hydrocarbon fractionating unit using a mixture of hexane and heavy virgin naphtha. The assumptions used in these calculations will be mentioned where applicable.

Equilibrium Curve. The equilibrium curve for the system hexane and heavy virgin naphtha was calculated by the use of Dalton's and Raoult's laws, assuming ideal gas behavior. The vapor pressures for hexane and heavy virgin naphtha were obtained from Maxwell(42), and the atmospheric pressure was found to be 0.94 atmosphere. A sample of the calculation is as follows:

By use of Dalton's law:

$$p^o_H \; x_H + p^o_{HVN} \; x_{HVN} = P_T$$

where:

p^o_H = vapor pressure of pure hexane at assumed temperature 156 °F, atm

p^o_{HVN} = vapor pressure of pure heavy virgin naphtha at assumed temperature 156 °F, atm

x_H = composition of hexane in liquid phase in equilibrium with vapor, mol fraction

x_{HVN} = composition of heavy virgin naphtha in liquid phase in equilibrium with vapor, mol fraction

P_T = total pressure of system, atm.

$$1.05 \; x_H + 0.125 \; x_{HVN} = 0.94$$

$$x_N = 0.88$$

By use of Raoult's law:

$$P_T \, y_H = p_H^o \, x_H$$

where:

P_T = total pressure of the system, atm

p_H^o = vapor pressure of pure hexane at assumed temperature 156 °F, atm

y_H = composition of hexane in vapor in equilibrium with liquid, mol fraction

x_H = composition of hexane in liquid in equilibrium with vapor, atm.

$$0.94 \, y_H = (1.05) \, (0.881)$$

$$y_H = 0.983$$

Correction Factor for Water Flow. In calibrating the reflux and product flow rate recorders, as tabulated in Table III, page 83, water was used in the place of hexane. A correction factor was needed to correct for the flow of hexane used during the tests on the fractionating tower. The equation used for calculating the factor was taken from Eckman(32).

$$q \rho_b = \frac{\pi}{4} K_o \beta^2 D^2 \sqrt{(r_m - r_f) \rho (2gh)}$$

where:

q = flow rate of fluid, cu ft/sec

ρ_b = density of flowing fluid, lb/cu ft

K_o = flow coefficient, dimensionless

D = inside pipe diameter, ft

β = diameter ratio, (d/D)

r_m = density of manometer fluid, lb/cu ft

r_f = density of fluid over manometer, lb/cu ft

ρ = density of flowing fluid, lb/cu ft

h = manometer differential, ft water

g = acceleration due to gravity, ft/sec^2

For all practical purposes all the terms in the right side of the equation are constant for both water and hexane, except the term

$$\sqrt{(r_m - r_f)\,\rho}\ .$$

Therefore:

$$\frac{(q\,\rho_b)_{water}}{(q\,\rho_b)_{hexane}} = \frac{\left(\sqrt{(r_m - r_f)\,\rho}\right)_{water}}{\left(\sqrt{(r_m - r_f)\,\rho}\right)_{hexane}}\ .$$

Since

$$w = (q\,\rho_b) = \text{flow rate, lb/sec}$$

then

$$w_{hexane} = \text{water}\ \frac{(13.6 - 0.684)\ 0.684}{(13.6 - 1)\ 1}$$

$$w_{hexane} = \text{water}\ (0.837)$$

Average Molecular Weight. For each test an average molecular weight was calculated so that the feed flow rates could be converted from pounds per minute to mols per minute. The data used in these calculations were taken from Tables II and VI, pages 80 and 95, respectively.

$$\text{Ave M W} = x_{H_f}(86) + x_{HVN_f}(120)$$

where:

x_{H_f} = composition of hexane in the feed, mol fraction

x_{HVN_f} = composition of heavy virgin naphtha in the feed, mol fraction.

$$\text{Ave M W} = (0.46)(86) + (0.54)(120)$$

$$\text{Ave M W} = 104.3$$

Feed Flow Rate Conversion. In order to calculate a hexane balance on the fractionating column, the flow rates from Table VI, page 95, had to be converted from pounds per minute to mols per minute.

$$F_f = f_f/(104.3)$$

where:

F_f = flow rate of feed, mol/min

f_f = flow rate of feed, lb/min.

$$F_f = (21.3)/(104.3)$$

$$F_f = 0.204$$

Overall Material Balance. Overall material balances for each test on the hydrocarbon fractionating unit were calculated by the following method using data from Table VI, page 95:

$$F = D + B$$

where:

F = feed flow rate, mol/min

D = product flow rate, mol/min

B = bottoms flow rate, mol/min.

$$0.204 \neq 0.085 + 0.114$$

$$0.204 \neq 0.199$$

Discrepancy = 3 per cent

Overall Hexane Balance. An overall hexane balance for each test on the hydrocarbon fractionating unit is shown in Table VI, page 95. This table was prepared by using data from the same table.

$$F\,x_F = D\,x_D + B\,x_B$$

where:

F = feed flow rate, mol/min

D = product flow rate, mol/min

B = bottoms flow rate, mol/min

x_F = composition of hexane in feed, mol fraction

x_D = composition of hexane in product, mol fraction

x_B = composition of hexane in bottoms, mol fraction.

$$0.204\ (0.460) \neq 0.114\ (0.793) + 0.085\ (0.053)$$

$$0.0938 \neq 0.0945$$

Discrepancy = 0.75 per cent

Average Latent Heat of Vaporization. In order to calculate the value of q necessary for constructing a q-line on the equilibrium curve for the system hexane and heavy virgin naphtha, an average value of the latent heat of vaporization is needed for the feed mixture. Using data from Tables II and VI, pages 80 and 95, respectively, the value is calculated as follows:

$$(\Delta H_v)_{ave} = (x_{H_f}(\Delta H_v)_H + x_{HVN_f}(\Delta H_v)_{HVN}$$

where:

$(\Delta H_v)_{ave}$ = average latent heat of vaporization of feed, Btu/mol

$(\Delta H_v)_H$ = latent heat of vaporization of hexane, Btu/lb

$(\Delta H_v)_{HVN}$ = latent heat of vaporization of heavy virgin naphtha, Btu/lb

x_{H_f} = composition of hexane in feed, mol fraction

x_{HVN_f} = composition of heavy virgin naphtha in feed, mol fraction.

$$(\Delta H_v)_{ave} = 0.46\ (138)(86) + 0.54\ (152)(120)$$

$$(\Delta H_v)_{ave} = 15,310$$

Average Specific Heat. The average specific heat of the feed is necessary in the calculation of the value of q used in constructing the q-line. Using the data from Tables II and VI, pages 80 and 95, respectively, the average value for the specific heat of the feed is calculated as follows:

$$c_{p_{ave}} = x_{H_f}(c_{p_H}) + x_{HVN_f}(c_{p_{HVN}})$$

where:

$c_{p_{ave}}$ = average specific heat of the feed, Btu/mol-°F

c_{pH} = specific heat of hexane, Btu/lb-°F

c_{pHVN} = specific heat of heavy virgin naphtha, Btu/lb-°F

x_{H_f} = composition of hexane in feed, mol fraction

x_{HVN_f} = composition of heavy virgin naphtha in feed, mol fraction.

$$c_{p_{ave}} = 0.46\ (0.565)(86) + 0.54\ (0.607)(120)$$

$$c_{p_{ave}} = 61.6$$

Feed Enthalpy Line. The calculation of the feed enthalpy line, or q-line, is dependent upon the condition of the feed as it enters the column and also upon the boiling point of the feed. The feed condition was obtained from Table VI, page 95, and the boiling point was obtained from Figure 19, page 94.

$$q = \frac{w\lambda + w\,c_p\,dt}{w\lambda}$$

where:

q = total amount of heat necessary to vaporize one mol of feed divided by the molal latent heat of vaporization of the feed, Btu/min

w = flow rate of feed, mol/min

λ = molal latent heat of vaporization of the feed, Btu/mol

c_p = specific heat of the feed, Btu/mol-°F

dt = temperature difference between entering feed and boiling point of feed, °F.

$$q = \frac{(0.204)(15{,}310) + (0.204)(61.6)(191.6-141.0)}{(0.204)(15{,}310)}$$

$$q = 1.20$$

$$\text{Slope of } q\text{-line} = \frac{q}{q-1}$$

$$\text{Slope} = 6$$

Enrichment Line. In order to calculate the theoretical number of plates required to perfect a given separation, an operating line for the section of the column above the feed plate has to be calculated. Using data from Table VI, page 95, the enrichment line was calculated as follows:

$$y = \frac{L}{L + D} x_n + \frac{D}{L + D} x_D$$

where:

y = composition of hexane in vapor rising from plate, mol fraction

x_n = composition of hexane in liquid leaving plate n, mol fraction

x_D = composition of hexane in product, mol fraction

L = reflux flow rate, mol/min

D = product flow rate, mol/min.

$$y = \frac{0.243}{0.243+0.102} x_n + \frac{0.102}{0.243+0.102} (0.793)$$

$$y = 0.704\, x_n + 0.235$$

Overall Column Efficiency. The number of theoretical plates required for each test on the hydrocarbon fractionating unit were calculated by the McCabe-Thiele method, as shown in Figure 20, page 112. The overall column efficiency for each test was calculated as follows:

$$\text{Eff} = \frac{n_t - 1}{n_a}(100)$$

where:

Eff = overall column efficiency, %

n_t = number of theoretical plates required to perfect a given separation, dimensionless

n_a = number of actual plates required to perfect a given separation, dimensionless.

$$\text{Eff} = \frac{2.80 - 1}{10}(100)$$

$$\text{Eff} = 18$$

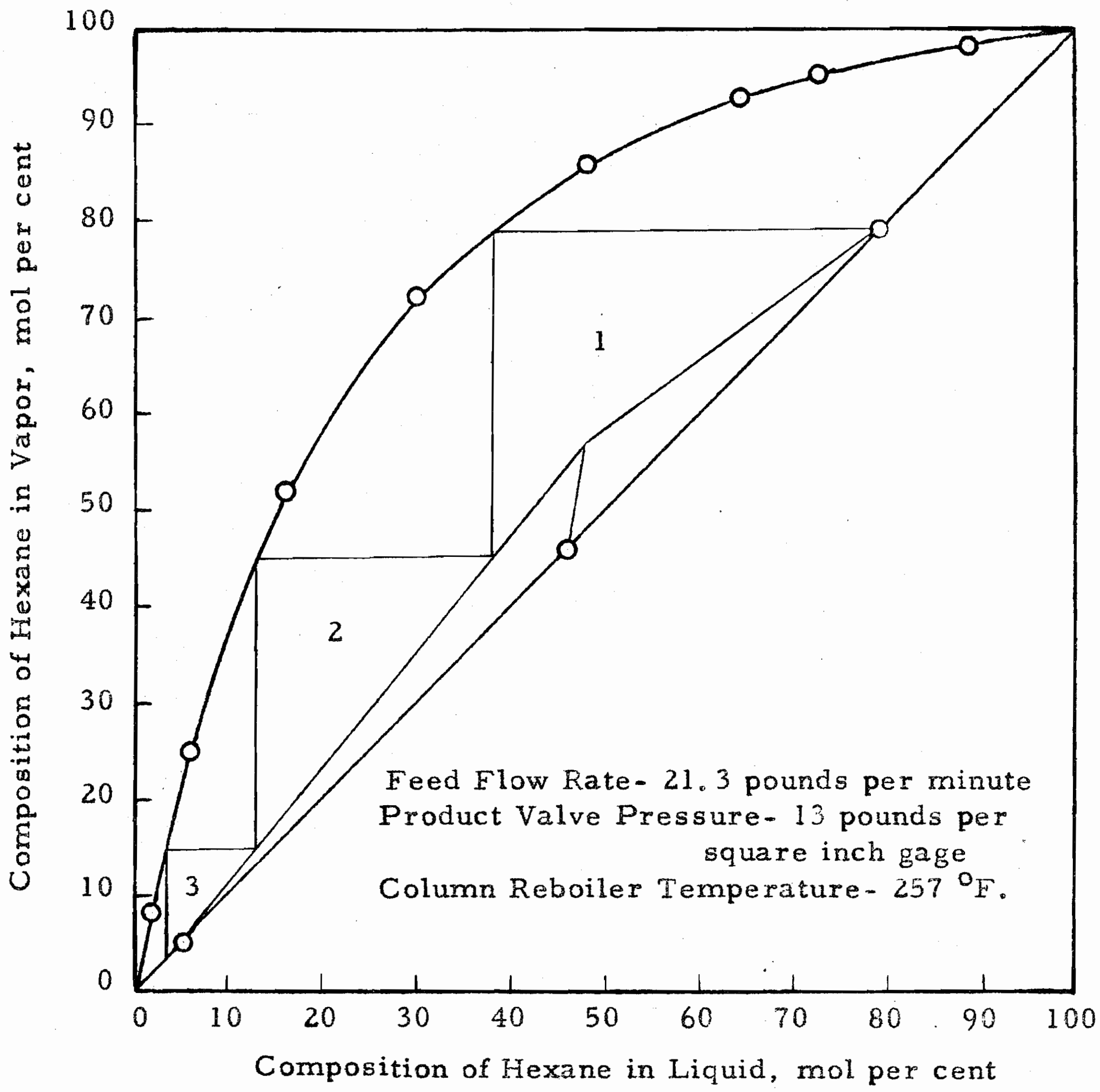

Figure 20. Calculation of Number of Theoretical Plates in Ten-Plate Bubble-cap Distillation Column Using the System Hexane and Heavy Virgin Naphtha

Murphree Plate Efficiency. The individual plate efficiencies for each test were calculated by the Murphree method and are tabulated in Table VIII, page 97. Using the data from Table VII, page 96, and Figure 18, page 93, the individual plate efficiencies were calculated as follows:

$$\text{Eff} = \frac{x_{n+1} - x_n}{x_{n+1} - x_n^*} (100)$$

where:

Eff = individual plate efficiency, %

x_{n+1} = actual composition of hexane leaving plate n+1, mol fraction

x_n = actual composition of hexane leaving plate n, mol fraction

x_n^* = theoretical composition of hexane leaving plate n, mol fraction.

$$\text{Eff} = \frac{79.4 - 79.0}{79.4 - 37.4} (100)$$

$$\text{Eff} = 1.0$$

IV. DISCUSSION

The following paragraphs contain a discussion of the modifications, instrumentation, and operation of the hydrocarbon fractionating unit, a listing and discussion of the recommendations for mechanical improvement and future operation of the unit, and a listing of the limitations imposed on the operation of the unit.

Discussion of Results

The following paragraphs contain a discussion of the modifications, instrumentation, and operation of the fractionating unit, and the results obtained from tests on the unit.

Modifications. During the preliminary tests on the column using water as a feed, it was observed that condensate would not flow from the condenser to the condensate pump, because the pump was at a level higher than the liquid level in the condenser and could not be primed. Therefore, in order to drain the condenser it was necessary to place the condensate pump below the level in the condenser. Since there was no liquid level controller on the condenser, it was decided that the accumulator drum should also be moved to a position below the condenser to make certain the condensed vapor had a free flow from the condenser. In the course of this change, the condensate pump was deleted from the entire system, and the accumulator drum was placed about two feet below the condenser. The product and reflux pump was then placed about one foot below the level in the accumulator drum to allow self-priming.

After the accumulator drum was relocated, it was observed that the instruments were not working properly and needed adjustments or replacements. The reflux controller was deleted from the system and the accumulator level controller was replaced, as was the column reboiler level controller. The other instruments were tested and adjusted to make certain they were in the range of column operation. Final adjustments were made when the petroleum fraction was used as the feed.

Before the petroleum mixture was put into the feed tank, it was found that the condenser, preheater, and bottoms cooler were without drains on the water side. Removable plugs were inserted in the exchangers so that during freezing weather the water could be drained out.

A water condenser, 40 inches high, was made from 1 3/4-inch and 3 1/2-inch steel pipe and placed on top of the feed tank to help condense the petroleum vapors that escaped from the drum during operation. One half inch copper tubing was used as water lines for the condenser.

Column Operation. The following is a discussion of the factors involved in the operation of the fractionating unit.

System. The hydrocarbon fractionating unit was designed for a system composed principally of hexane, heptane, and octane. The commercial grade hexane was obtained from Hercules Powder Company and the heavy virgin naphtha was obtained from Esso Standard Oil Company. The heavier of the two feed components was found to contain mainly paraffins with eight to ten carbon atoms, as well as substantial amounts of naphthenes (see Table VI, page 95). Since no particular compounds were found to be in large concentrations in the heavy virgin naphtha, a multicomponent distillation calculation was deemed impracticable. Therefore, the McCabe-Thiele method of calculation was performed for each test, assuming the two components to be hexane and heavy virgin naphtha.

Variables Studied. The variables that were studied included feed flow rate, column reboiler temperature, and product valve pressure. It was planned that reflux ratio was to be studied in place of the product valve pressure, but after discovering that the temperature recorder for the fourth plate from the top of the column was instrumentally faulty, the instrument was deleted from the system. This instrument was supposed to control the flow of reflux; however, after changing the reflux control valve to the product line, the flow of product was controlled manually, and two different valve pressures, 13 and 15 pounds per square inch, gage, were used for the tests.

There were many other variables that could have been studied, including:

(1) feed composition
(2) feed temperature
(3) feed plate
(4) reflux temperature
(5) bottoms temperature
(6) accumulator level
(7) column reboiler level.

Any or all of these variables should be studied in the future.

Equilibrium Data. The equilibrium data for the system hexane and heavy virgin naphtha were calculated by use of Dalton's and Raoult's laws of partial pressure. Check points which were calculated using fugacity functions of the hydrocarbons were found to correspond within one per cent of the original calculations.

Heat Balance. During the first test on the fractionating column using the petroleum fraction as the feed it was discovered that no thermocouples had been placed in the lines carrying water from the bottoms cooler, tops condenser, feed preheater, and column reboiler. It was impossible, without these temperature measurements, to obtain a heat balance for the fractionating unit. Therefore, no heat balance was calculated for any test.

Instrumentation and Control. The following paragraphs contain a discussion of some of the instruments used for recording and controlling the temperatures and flow rates of the terminal streams during tests on the hydrocarbon fractionating unit.

Bottoms Temperature Controller. The bottoms temperature was controlled by the amount of cooling water passing through the heat exchanger. During preliminary tests using the petroleum fraction, it was found that the automatic control in the instrument was faulty. Therefore, the instrument was operated manually for all subsequent tests.

Column Level Controller. The column level controller was one of the most critical instruments used, in that, with changes in the liquid level in the column, the boilup rate was altered, thereby changing the compositions on each plate. During each test the level had to be kept practically constant, as recorded on the chart, before equilibrium conditions could be attained. Column level fluctuations as much as two feet caused the temperatures on each plate to change approximately three degrees.

Reflux Accumulator Drum Liquid Level Controller. The liquid level controller for the reflux accumulator drum was excellent in its control features. During all the tests a straight line was plotted on the recorder chart indicating a constant level in the drum. Since the boilup rate in the fractionating column did not remain constant, the flow of material from the accumulator drum differed from time to time. The product flow rate was controlled by product valve pressure; therefore, the reflux flow rate varied with the boilup rate in the column. For each test an average value of reflux rate was taken for each 15 minute period and these values were averaged for the overall test flow rate.

<u>Orifice Meter Errors</u>. According to the literature(57), the probable overall tolerance for orifice meter installations should be ± 1.25 per cent. Even though normal precautions are taken, sometimes the accuracy attained in practice may not fulfill expectations, since minor errors may develop, and their causes may not always be apparent. One of the following conditions may occur to cause inaccuracies of this nature(57):

(1) gradual deposition of solids or collection of liquid condensates adjacent to the orifice

(2) entrapping of vapors in the upper portion of the flow line

(3) gradual change in the characteristics of the metered fluid

(4) reduction of flow rates, producing pen readings that are in the lower, less accurate region of the meter chart

(5) existence of pulsating flow which is not detected by the meter because of slowness of response

(6) slight differences between the gravities of the sealing liquid columns in the lines connecting seal pots and the meter

(7) gradual plugging of the two meter lead lines

(8) erosion of the orifice.

With these adverse conditions taken into account, accuracies of three per cent are generally accepted in industrial operations.

Sample Analysis. The analysis procedure for the samples from tests on the hydrocarbon fractionating unit were analyzed by using an Abbé refractometer. The reference samples for the refractive index curve included heavy virgin naphtha and 99.5 per cent pure hexane. The commercial grade hexane used in the feed mixture could not be used as a reference liquid because the weight per cent hexane in the material was only 56.3 per cent by mass spectrophotometry analysis. The distillation process upgraded the product to approximately 80 per cent hexane by refractive index analysis.

Number of Theoretical Plates. The number of theoretical plates for each test, tabulated in Table VI, page 95, ranged from 2.80 to 5.00 for all tests other than four and seven. Tests four and seven had a bottoms with no hexane component, which means that the number of theoretical plates was infinity. In performing the calculations, it was observed that, in the enriching section of the column, the number of theoretical plates for each test was less than one. It was also observed during sample collecting, that the condition of blowing was evident in that same section of the column.

Material Balance. The material balances for the eight tests were calculated using flow rates in mols per minute and compositions of hexane in mol fraction. The discrepancies of the hexane balances are tabulated in Table VI, page 95, and appear to be within the range of instrument calibration error and sample analysis error, with the exception of tests seven and eight. In these latter two tests the feed flow controller calandria section temperature controller, reflux temperature controller, and column reboiler level controller were not operating with the same precision of control that had been experienced with the earlier tests. Analysis of the terminal streams, however, indicated that the system was at equilibrium.

Recommendations

The following paragraphs contain a listing and discussion of the recommendations for future work on the hydrocarbon fractionating unit.

Study of Instrument Control. During the preliminary tests on the fractionating unit the reflux control instrument was removed from the system because of malfunctioning. The instrument should be replaced by a later model or repaired and reinserted into the control system of the unit. Further studies of the instrumentation control of the unit is recommended, using various settings of proportional band and reset rate.

Installation of Thermocouples. In order to make a complete heat balance for the fractionating unit, thermocouples must be inserted into the exit water streams of the calandria section, bottoms cooler, tops condenser, and feed preheater. The thermocouple leads could be placed in the indicating potentiometer along with the thermocouple leads from the plates in the column.

Application of Thermal Insulation. During the tests on the hydrocarbon fractionating unit, it was quite evident that considerable heat was being lost through the walls of the tower, tops condenser, and feed preheater. This condition makes an accurate heat balance impossible. It seems advisable that thermal insulation be applied to the column, at least up to the supporting lugs that mark the end of the calandria section, and to the outside surface of the tops condenser and feed preheater. In both the condenser and the preheater the shell side operates with the hot streams flowing through them.

Steam Calorimeter. The correct evaluation of the heat passing into the calandria section of the column and the feed preheater depends on the accurate knowledge of the quality of the steam used. A throttling type steam calorimeter installed in the main steam line would allow the determination of the quality of the steam during the operating periods.

Analysis Procedures. At present, the samples from the fractionating column are analyzed by means of refractive indices with the accuracy of approximately three per cent. The use of gas-liquid partition chromatography could possibly reduce the error to one per cent, provided suitable packing and instrument conditions can be determined to allow a reasonable resolution time for the samples.

Capacity Tests. It is recommended that after instrument and column modification, the feed rate be varied over a wide range in an attempt to determine the optimum operating region for the column. Flooding and blowing conditions and analyses of the tops and bottoms would indicate the points of optimum operation.

Installation of Glass Handhole. The installation of glass handholes on at least two plates would enhance the determination of good column operation because the conditions of dumping and blowing could be visually observed for those plates.

Pressure Drop Across Plates. There is now no way to determine the height of the liquid seal on the plates in the column. The liquid head on any one plate is an important factor in the operating efficiency of the unit; to obtain an indication of the liquid head, a differential pressure measuring device should be connected to taps through the column wall at each plate level and in the vapor space above each plate.

Construction of Fence Around Fractionating Unit. Because of the nature and location of the fractionating unit, a cyclone type fence at least six feet high should be constructed to increase general safety and protect the installation.

Limitations

The following paragraphs contain a listing of the limitations imposed upon the operation of the hydrocarbon fractionating unit.

Distillation Column. The fractionating column used in the distillation process contained ten plates, spaced one foot apart, was 25 feet high, two feet in diameter, and contained twenty-two 2-inch bubble caps per plate. The heating coils in the column were constructed of extra heavy one inch pipe and had a total heating area of 60 square feet.

Bottoms Cooler. The bottoms cooler in the fractionating unit was a four-pass shell and tube type exchanger having a contact surface of thirty-seven 3/4-inch tubes 70 inches long.

Tops Condenser. The tops condenser used in the fractionating unit was a single pass shell and tube type exchanger having a contact surface of eighty-nine 3/4-inch admiralty metal tubes 69 inches long.

Feed Preheater. The feed preheater used in the fractionating unit was a single pass shell and tube type exchanger having a contact surface of nineteen 5/8-inch copper tubes 45-1/2 inches long.

Pump Capacity. The two centrifugal pumps used to pump the feed and overhead vapor condensate streams had a capacity of 38 gallons per minute against a 40 pound head. The centrifugal pump used to pump the bottoms stream had a capacity of 30 gallons per minute against a 40 pound head.

Flow Line Piping. All flow line piping, with the exception of the cooling water lines, was installed with black iron pipe. The cooling water lines were 3/4-inch galvanized iron pipe. Steam supply line to the calandria was 1-inch while the steam line to the feed preheater was 1/2-inch. The feed line was 3/4-inch. The bottoms and product lines were 3/8-inch. The overhead vapor product line was 2-inch. The reflux line was 1/2-inch. The cooling water and steam condensate drain line was 2-inch.

Control Valves. All control valves were diaphragm motor valves with throttling action.

Air and Orifice Tap Lines. All air and orifice tap lines were 1/4-inch copper tubing with the exception of the main air supply line which was 1/2-inch galvanized iron pipe.

Steam. Steam at 80 pounds per square inch, gage, was used to supply heat to the calandria and feed preheater.

Air. Air from 40 to 65 pounds per square inch, gage, was filtered and throttled to 20 pounds per square inch, gage, for use in the instrumentation system.

Feed Rate Control. The feed rate was controlled at the preseat levels of 16.8 and 21.3 pounds per minute with a flow rate controller.

Calandria Section Temperature Control. The temperature of the calandria section was controlled at the preset temperatures of 257 and 275 °F, by use of a temperature controller.

Product Valve Pressure. The product control valve opening was controlled by the air pressure exerted on the diaphragm of the valve. The pressures used were 13 and 15 pounds per square inch, gage.

Feed Temperature. The feed to the distillation column had a constant temperature of 141 °F, controlled by means of a temperature controller on the feed preheater.

Reflux Temperature. The reflux temperature was maintained at a temperature of 145 to 151 °F by using a temperature controller on the tops condenser.

Feed Mixture. The feed was approximately a 50 to 50 weight per cent mixture of commercial grade hexane, boiling in the range from 148 to 152 °F, and heavy virgin naphtha, boiling in the range from 220 to 309 °F.

Atmospheric Pressure. The atmospheric pressure for the tests on the fractionating column was between 710 and 720 millimeters mercury for all tests.

Water. The cooling water for the bottoms cooler, and condensing water for the tops condenser was at a temperature of 60 °F.

V. CONCLUSIONS

An automatically controlled hydrocarbon fractionating unit two feet in diameter and 25 feet high, having ten plates, one foot apart, each plate containing twenty-two 2-inch bubble-caps, was operated under 0.94 atmosphere pressure using 50 weight per cent hexane and 50 weight per cent heavy virgin naphtha. The feed flow rates were 16.8 and 21.3 pounds per minute, the calandria section temperatures were 257 °F and 275 °F, and the product valve pressures were 13 and 15 pounds per square inch, gage. Feed and reflux temperatures were 141 °F and 146 °F, respectively. Reflux ratios ranged from 0.64:1 to 2.74:1 for the eight tests.

The conclusions drawn from the tests include:

1. The fractionating unit can be operated automatically, except for manual controlling the amount of product withdrawn.

2. The overall column efficiency is between 18 and 40 per cent.

3. The number of theoretical plates in the column is between 2.8 and 5.0.

4. The individual plate efficiencies vary from -12.5 to 75.0 per cent.

VI. SUMMARY

An automatically controlled hydrocarbon fractionating unit was operated using a petroleum fraction consisting of commercial grade hexane and heavy virgin naphtha as the feed. The distillation column was 25 feet high, two feet in diameter, and contained ten plates spaced at one foot intervals, each plate having twenty-two 2-inch bubble-caps.

Flow rates of the feed were 16.8 and 21.3 pounds per minute, calandria section temperatures were 257 °F and 275 °F, and product valve pressures were 13 and 15 pounds per square inch, gage. The feed temperature was 141 °F and the reflux temperature was 146 °F. The reflux ratio ranged from 0.64:1 to 2.74:1.

The feed was preheated in a shell and tube heat exchanger governed by a temperature controller. The feed flow was controlled by a flow controller. The hot bottoms stream was cooled in a shell and tube heat exchanger. The overhead vapor product was condensed in an automatically controlled condenser and flowed freely into an accumulator drum. The reflux and product streams were divided by the action of a manually controlled diaphragm motor valve on the product line, which determined the amount of product, the remainder of the stream being reflux. The bottoms discharge rate from the bottom of the column was controlled by a liquid level controller maintaining a constant level in the reboiler section of the column. The bottoms and product streams were remixed and used as the feed stream.

Temperatures of each plate in the column and the hydrocarbon streams from the three heat exchangers were indicated either by a precision potentiometer actuated by alumel-chromel thermocouples or the recording elements of the four temperature controllers in the instrumentation system. The flow rates in the cooling water lines, steam lines, product line, bottoms line, reflux line, and feed line were recorded by flow transmitter-recorders.

Eight tests were performed on the unit using a feed mixture of approximately 50 weight per cent hexane and 50 weight per cent heavy virgin naphtha. Preliminary tests were made on the distillation apparatus using water as a feed.

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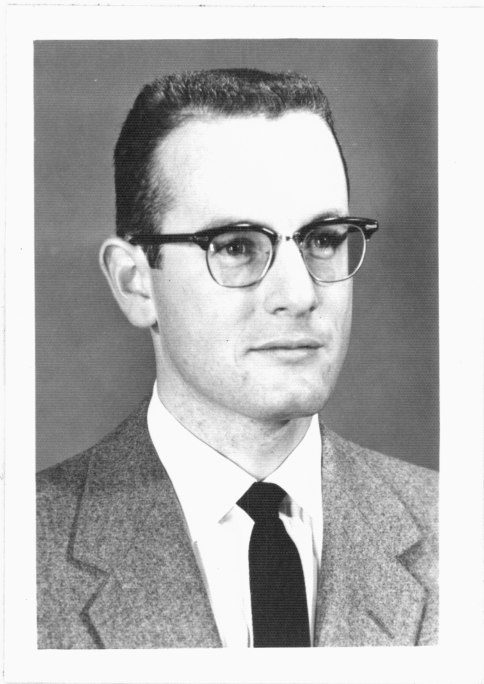
IX. VITA



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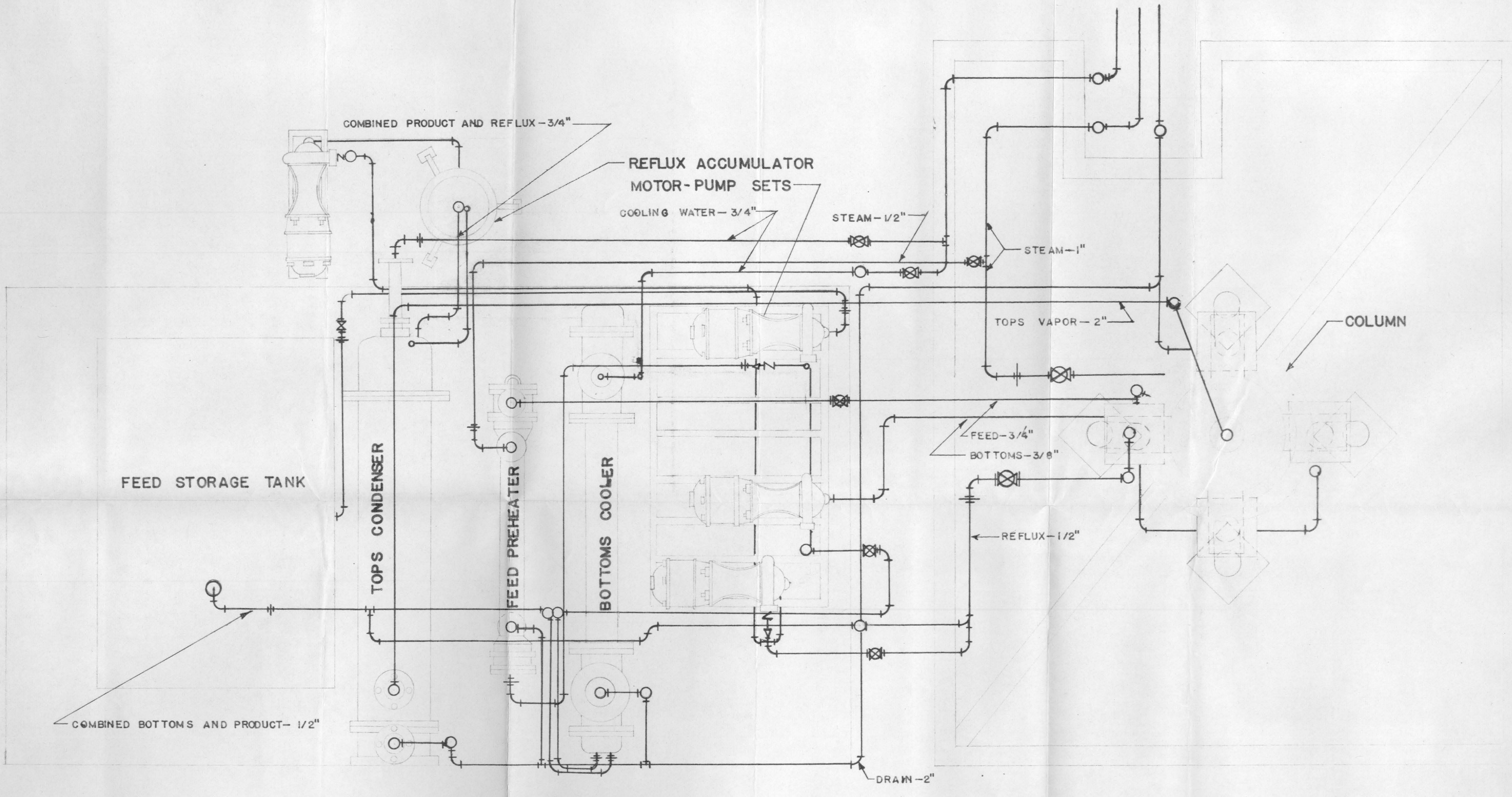
The author was a Graduate Assistant during his graduate studies.

Wayne Cammer Richard



NOTES

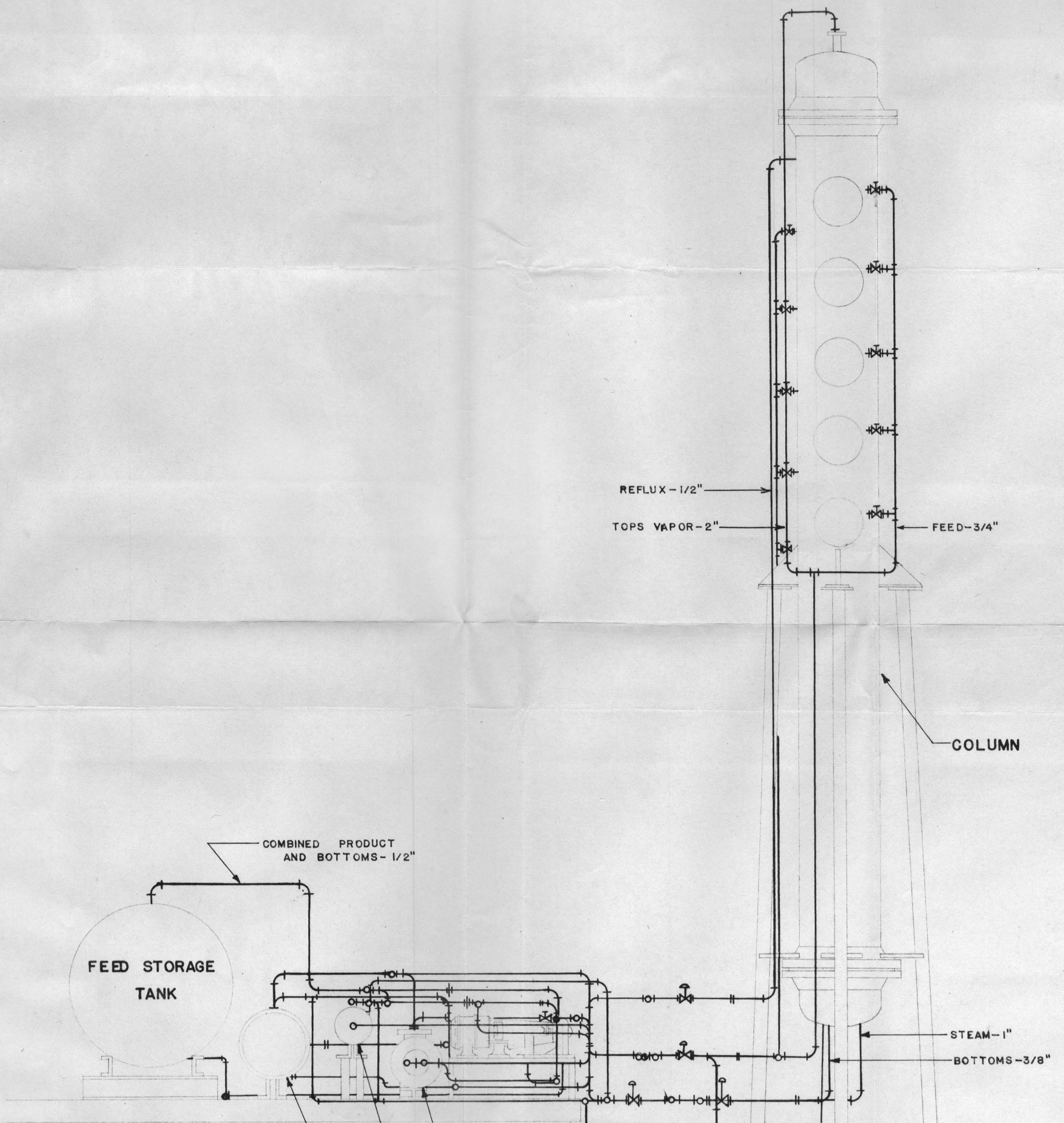
ALL PIPE BLACK IRON EXCEPT WATER LINES
ALL WATER LINES 3/4" GALVANIZED IRON
BYPASS LINES AROUND PUMPS AND CONTROL VALVES OMITTED FOR CLARITY
COLUMN ROTATED 90° COUNTER-CLOCKWISE FROM TRUE POSITION FOR CLARITY

DEPARTMENT OF CHEMICAL ENGINEERING
VIRGINIA POLYTECHNIC INSTITUTE
BLACKSBURG, VIRGINIA

PIPING DIAGRAM OF FRACTIONATING COLUMN
AND ACCESSORY EQUIPMENT

PLAN VIEW

SCALE: 3/4" = 1' 0" DATE: 6/20/57 CASE NO: 439
DRAWN BY: FILE NO: 57
CHECKED BY: W. C. Richard FIGURE NO: 6
APPROVED BY: Frank C. Vilbrandt 6/20/57



NOTES

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BLACKSBURG, VIRGINIA

PIPING DIAGRAM OF FRACTIONATING COLUMN
AND ACCESSORY EQUIPMENT
SOUTHWEST ELEVATION

SCALE: 1/2"=1'0" DATE: 6/26/57 CASE NO: 439
DRAWN BY: J. A. Silver FILE NO: 57
CHECKED BY: F. C. Vilbrandt FIGURE NO: 7
APPROVED BY: F. C. Vilbrandt 7/3/57