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## I - INTRODUCTION

The process of fractional distillation has been performed for many years, but has only within the past half century become a true science. Sorel<sup>(69)</sup> in 1893, was the first to give a theoretical basis for predicting the operating characteristics of a fractionating column. Later work by Lewis<sup>(43)</sup>, Peters<sup>(53)</sup>, McCabe and Thiele<sup>(48)</sup>, and others, <sup>(50)</sup>,<sup>(7)</sup>,<sup>(75)</sup>, has resulted in the theoretical principles of modern distillation practice.

Even though considerable work has been done relating to fractional distillation, there are still questions as to the conditions existing in a bubble cap column and the variables that affect the efficiency of such a column. Because of the many variables encountered and the uncertainty of their effect on column efficiency, the design of commercial columns has long been a major engineering problem. Among the variables that affect column efficiency are: vapor rate, reflux ratio, nature of the components being distilled, position of the feed plate, and finally, the construction of the column.

Plate and overall column efficiencies, as given in the literature vary widely, and it is thought possible that the non-uniformity of results obtained is due to poor sampling and to the column not being at equilibrium when the samples are taken. Recent work by

Dorsey<sup>(21)</sup> tends to substantiate this theory and also to show that overall column efficiency is not a proper measure of the number of plates required in a column. His work indicated that in a column in which isopropyl alcohol and water were being fractionated, all of the separation took place on four plates near the center of the column. These four plates approached 100 per cent efficiency while the others were near zero per cent efficiency. However, Vilbrandt et al,<sup>(77)</sup> in a study of the distillation of ethyl alcohol and water in a three plate bubble-cap column of the same construction, found plate efficiencies relatively constant over the column.

The object of this investigation is to determine the operating characteristics of a fifteen plate fractionating column (8" diameter), using the minimum boiling mixture of isopropyl alcohol - water, and the normal boiling mixture of toluene - ethylene dichloride. Attempt will be made to clarify the effects of the operating variables, vapor rate, concentration of the feed, nature of the components being distilled, and position of the feed plate, upon the efficiency of the column. In addition, the distillation characteristics of the binary mixtures used will be investigated by determining the actual operating lines and comparing them with the McCabe and Thiele operating lines for the rectifying and stripping sections of the column.

## II - LITERATURE REVIEW

Definitions.-Distillation is the separation of the constituents of a liquid mixture by partial vaporization of the mixture and separate recovery of vapor and residue. The more volatile constituents of the original mixture are obtained in increased concentration in the vapor; the less volatile in greater concentration in the liquid residue. (52)

Rectification or fractional distillation is a distillation carried out in such a way that the vapor rising from a still comes in contact with a condensed portion of vapor previously evolved from the same still. A transfer of material and an interchange of heat results from this contact, thereby securing a greater enrichment of the vapor in the more volatile components than could be secured with a single distillation operation using the same amount of heat. The condensed vapors, returned to accomplish this object, are termed reflux. (52)

### History

The Early History.-The early history of the art of distillation goes back to the beginnings of alchemy, for distillation was

an operation of fundamental importance in the practice of alchemy. Underwood<sup>(74)</sup> states that one of the earliest applications of distillation was in the preparation of mercury and the treatment of metals with mercury. Dioscorides describes the preparation of mercury by heating cinnabar in an iron pot and condensing the vapor on an earthenware cover. Dioscorides, Pliny, and Galen<sup>(74)</sup> describe the preparation of zinc oxide by heating calamine and condensing the vapors similarly on the cover of the vessel. Underwood<sup>(74)</sup> states that the distillation of mare's milk was practiced in 1000 A.D. A primitive method of distillation is described by Pliny<sup>(24)</sup> for obtaining oil of turpentine from rosin. The vapor from heated rosin was collected on fleeces of wool spread over the pots, and the condensed liquid extracted from it by expression. Dioscorides first employed the principle of dephlegmation in the first century A.D. One of the important ancient authors is Zosimus, who by the end of the third century had compiled a chemical encyclopaedia and had written twenty-eight books on alchemy. Zosimus describes copper still heads with three receivers, air-cooled condensers, and heat controlling devices. The Arabs adopted distillation from the Greeks and used it in preparing many perfumery and pharmaceutical products.

Later Advances in Distillation.—The discovery of alcohol or spirits of wine about 1050 A.D. was of great importance in the advancement of distillation. From the eleventh to the eighteenth century advances were made in condensing and cooling the vapor, more

rapid rates of distillation, and methods of heating<sup>(25)</sup>. It was at the latter end of the eighteenth century that there commenced a series of fundamental changes in still construction that completely revolutionized still design<sup>(74)</sup>. The first important step was the invention of the preheater by Argand in 1780. The next important advance in still design was Adam's still which introduced the principle of the enrichment of a relatively poor vapor by contacting it with a liquid to evolve a richer vapor. Berard, in 1805, patented a still employing partial condensation of the vapors from the kettle to obtain a more concentrated distillate. The next great advance in still design was due to Cellier-Blumenthal<sup>(1)</sup> who patented a continuous still in 1813. A means of regulating steam supply was achieved by Savalle's invention of the steam regulator about 1857. Perrier in 1822, was granted a patent for a still in which baffles caused the wash to follow a lengthy path and steam was blown through it by means of small tubes, similar in action to bubbler caps. The great practical advance in steam heated stills came with the still invented by Coffey in 1830, which contained perforated plates so that the vapor could pass upward through them but the liquid could not pass downward. During the period covered above, distillation was an art. Its transformation into a science may be said to date from the last decade of the nineteenth century.



Modern Developments in Distillation.—When Sorel<sup>(69)</sup> published his fundamental work on the quantitative application of physical principles to still design in the latter part of the nineteenth century, the science of distillation was born. With Lewis<sup>(43)</sup> application of theoretical principles to distillation and fractionation in 1922, a new period in the history of distillation processes was initiated. Carey<sup>(15)</sup> in summarizing distillation in 1934, stated that the great advances made in increasing efficiency and lowering operating costs of distillation units was largely due to scientific control and continuous regulated operation.

### Theory of Distillation

Equilibrium Data.—The basic requirement for a separation of substances by distillation is that the composition of the vapor be different from the composition of the liquid from which it is formed. Thus, the basic data of any distillation problem are the equilibria between the liquid and vapor phases of the system to be distilled. This relationship is expressed as an equilibrium diagram. Tables and curves giving equilibrium data are available in handbooks<sup>(52)</sup> and in other literature, or the vapor - liquid equilibrium diagrams can be constructed from the boiling point curves of the mixtures under consideration. For substances which follow Raoult's Law<sup>(3)</sup> it is possible to compute the boiling point diagram from vapor pressure data of the pure components. Most combinations used in practice deviate more

or less widely from this law.

The Fractionating Column.-Instead of laborious and time-consuming fractional distillations involving methods closely allied to simple distillation, commercial practice has developed the use of continuous countercurrent rectification carried out in fractionating columns. Essentially, a fractionating column is a device for bringing a stream of vapor and a stream of liquid into intimate countercurrent contact. Provision is made for intimate contact by either lumps of inert material, plates and bubble caps, or perforated plates. The plate on which the feed is introduced is called the feed plate. The part of the column above the feed plate is called the rectifying section, and that below and including the feed plate is called the stripping section. The vapor coming from the top of the column is completely condensed. Part of it is withdrawn as the product; and the rest, called reflux, is put back into the top of the column to provide the down coming stream of liquid. The ratio of reflux to product is called the reflux ratio.

Early Applications of Theory.-Sorel<sup>(69)</sup>, who was the first to apply theoretical considerations to a rectifying column, applied energy and material balances from plate to plate throughout the column to calculate the enrichment of the vapor. He made the assumption that the column was composed of ideal or equilibrium plates. (An ideal plate fulfills the requirement that the vapor rising from the plate is in equilibrium with the liquid descending from the plate). The

method suggested by Ponchon<sup>(55)</sup> and applied by Savarit<sup>(62)</sup> and Thiele<sup>(71)</sup> simplified Sorel's method by solving his equations graphically to determine the number of equilibrium plates required to effect a given separation, and the composition of liquid on each plate. Because of the complexity of Sorel's method, the calculations are usually modified by making the following simplifying assumptions<sup>(59)</sup>:

1. Heat losses from the column are negligible.
2. The heats of mixing in the vapor and liquid phases are negligible.
3. The components being separated are of the same class; i.e., both are associating or both are non-associating such that their heats of vaporization and other thermal properties are similar.

Lewis' Method of Calculation.-Lewis<sup>(43)</sup> assumed that the change in vapor concentration from plate to plate was infinitely small and that perfect rectification was obtained. His approach is a modification of Sorel's method, simplifying computations and making it possible to present results graphically. He derives basic equations of general applicability for calculating theoretical rate of rectification within any column and amount of overflow, assuming Raoult's Law, constant molal overflow, and perfect equilibrium. The calculations are accurate for columns with a large number of plates but an error is introduced in a column of few plates by assuming constant stepwise conditions. During his work, Lewis developed the first

method offered for determining at what point feed should enter a continuous column. The methods of Leslie<sup>(42)</sup> and Van Nuys<sup>(76)</sup> are similar to that of Lewis, and are correspondingly limited in their use. Peters<sup>(53)(54)</sup> also assumed a column with a large number of plates in his modification of Lewis' basic equation.

McCabe-Thiele Method of Calculation.-Shirk and Montonna<sup>(64)</sup> found the method of McCabe-Thiele<sup>(48)</sup> the most accurate and practical of any in popular use in 1927. Assuming conditions of constant molal overflow, Raoult's Law, straight line operating conditions, and that the overall column efficiency is known, this method is a simple means of calculating the number of plates required from the equilibrium diagram by graphical means. Baker and Stochhardt<sup>(4)</sup> have modified this method by lowering the equilibrium curve to correspond to the column efficiency and thus, giving results in terms of the actual plates required for a given separation in a given column. A graphical method similar to McCabe and Thiele's had been introduced previously by Rodebush<sup>(61)</sup>, but was too complicated to meet with popular approval.

Murphree Column Calculations.-Murphree<sup>(49)(50)</sup> treated rectification as a special case of absorption and applied equations for graphically determining the number of plates required for a given separation. The graphical solution, which checks exactly with the McCabe and Thiele method, involves lengthy calculations. Murphree was the first to prove that the required number of plates is a minimum

when the feed is introduced on the plate at which the liquid composition is nearest that of the feed. Murphree also derived a method of calculating plate efficiency (Murphree Plate Efficiency) which is the ratio of the actual enrichment occurring on a plate to the equilibrium enrichment on that plate.

Other Calculation Methods.—Other methods of calculation fall into four main classes:

1. Development and/or modification of the methods of Lewis and McCabe and Thiele. Under this class are included the modifications of Dodge and Huffman<sup>(20)</sup>, W. K. Lewis, Jr.<sup>(44)</sup>, Smoker<sup>(68)</sup>, Baker and Lindsay<sup>(5)</sup>, and Mains<sup>(46)</sup>. These methods apply to calculations involving such variables as entrainment, more than one feed or drawoff, and distillation of partly miscible liquids.
2. Simplification of known methods of calculation. Among the methods in this group are those of Keyes, Soukup, and Nichols<sup>(38)</sup>, Branden<sup>(7)</sup>, Brown and Martin<sup>(10)</sup>, and Gilliland<sup>(29)</sup>. Charts and nomographs have been developed by Smoker<sup>(67)</sup>, Faasen<sup>(26)</sup>, Underwood<sup>(75)</sup>, and Edgeworth-Johnstone<sup>(23)</sup>.
3. Treatment of distillation as a diffusional process. Chilton and Colburn's HTU method for diffusional processes<sup>(17)</sup> is a general method whereby operations of a diffusional nature can be calculated on a common basis. Modifications by Colburn<sup>(18)</sup> and by Withrow, Koffolt, and Gerster<sup>(79)</sup> extended the method to the design and correlation of the operating characteristics of plate column

still. Other methods include those by Randall and Longtin<sup>(57)</sup> and by Scheibel and Othmer<sup>(63)</sup>.

4. General distillation equations. Fenske<sup>(27)</sup> presented an equation of this type for distillation at total reflux, while Smoker<sup>(68)</sup> derived a similar equation to be applied to binary distillation at any reflux. The system of distillation equations presented by Harbert<sup>(30)</sup> is the most complete of any yet published; included is the general equation for the case of any number of components and any reflux ratio.

#### Column and Plate Efficiency Factors

Plate Efficiency.-In practice equilibrium between vapor and liquid is never reached<sup>(78)</sup>. It is therefore necessary to convert the number of theoretical plates to the number of plates actually needed to perform a given separation. Two kinds of plate efficiency are recognized i.e., overall column efficiency and individual plate efficiency. The number of plates theoretically necessary for a given enrichment, divided by the number of plates actually required, is called the overall column efficiency of the column. The second type of plate efficiency considers the individual plates and is postulated by Murphree<sup>(49)</sup>. This efficiency is defined by the equation:

$$E_o = \frac{Y_n - Y_{n+1}}{Y_{*n} - Y_{n+1}}$$

Where:  $E_o$  = Murphree Plate Efficiency  
 $Y_n$  = Composition of vapor leaving the plate  
 $Y^*_n$  = Composition of vapor in equilibrium with the liquid  
leaving the plate  
 $Y_{n+1}$  = Composition of vapor rising to the plate

Murphree efficiency is not very practical in its original form because of the difficulty involved in measuring vapor concentrations. Underwood<sup>(75)</sup> has expressed this efficiency in an equation involving liquid concentrations as follows:

$$E_o = \frac{X_{n-1} - X_n}{X^*_{n-1} - X_n}$$

Where:  $E_o$  = Murphree Plate Efficiency  
 $X$  = Composition of liquid on the plate  
 $X^*$  = Composition of liquid, resulting from the condensation of vapor, in equilibrium with liquid on the plate  
 $n$  = Plate under consideration  
 $n-1$  = Plate above the plate under consideration

As shown by Holbrook and Baker<sup>(33)</sup>, the overall plate efficiency may be more or less than the Murphree individual plate efficiency, depending on the nature of the equilibrium curve and operating line. They are equal only when the equilibrium curve and operating line are straight and parallel or when the individual plate efficiency is unity.

### Design Factors Affecting Column Efficiencies

Plate Spacing.-Vapor velocity and plate spacing are the major factors influencing entrainment according to Siegel<sup>(66)</sup>, who reports that decreasing the plate spacing by one-half causes a ten-fold increase in entrainment. Commercial columns are made with their plates spaced from six to thirty-six inches apart. Plate spacing seems to affect only the amount of entrainment in a column and is dependent upon this value and the vapor velocity.

Downspout Area.-Pyott, Jackson and Huntington<sup>(56)</sup> find that increasing the downspout area to a certain critical point yields the most benefit for the least cost. Souders and Huntington<sup>(70)</sup> report that a limitation of the column capacity may occur when the lack of sufficient downspout area causes the column to become flooded. Walker, Lewis, McAdams, and Gilliland<sup>(78)</sup> say that the perimeter of the downspout is more important than the area because most of the time the entire area of the downcomer is not filled with the liquid. Siegel<sup>(66)</sup> advocates that the width of the weir be three times the height of the liquid over the weir.

Slot Area and Design.-Bubble caps have been made with several different types of slots, among which are the original V-notch without gables, the V-notch with gables, rectangular notches, and punched holes in the horizontal skirt of a cap. Siegel<sup>(66)</sup> reports that the slotted-edge caps are better than those with a serrated edge with the shape of the slots of little importance. Carey, Griswold,



Lewis, and McAdams<sup>(14)</sup>, Siegel<sup>(66)</sup>, and Keyes<sup>(37)</sup>, all agree that the narrower the slot, the smaller are the bubbles and thus, the more contact between liquid and vapor. An optimum slot width must be sought, for below this value the entrainment within the column becomes excessive.

Liquid Depth on the Plates.-Carey, Griswold, Lewis, and McAdams<sup>(14)</sup> state that an increase in liquid depth on the plates increases the column efficiency and also the entrainment. Siegel<sup>(66)</sup>, however, states that there is no advantage in the vertical rising of the bubbles and that deep submergence is not conducive to bubble formation. He advises that from one to three inches is preferable, stating that a higher value decreases column capacity. The necessity of evenness of the liquid level is stressed by Siegel<sup>(66)</sup>, Peavy and Baker<sup>(51)</sup>, and Chillas and Weir<sup>(16)</sup>, so that channeling will be kept at a minimum.

#### Operating Factors Affecting Column Efficiency

Vapor Velocity.-Vapor velocity or rate of distillation is important for its effect on entrainment, and consequently on the efficiency. Siegel<sup>(66)</sup> and Thomson<sup>(72)</sup> found that entrainment increases slowly with the vapor velocity up to a certain point, after which, the amount of entrainment increases rapidly with the vapor velocity. Shirk and Montonna<sup>(64)</sup> indicate that plate efficiency

varies inversely as some function of the rate of distillation. Peavy and Baker<sup>(51)</sup> report that the prediction of allowable vapor rates on the basis of entrainment is justifiable for low plate spacings at least. Carey, Griswold, Lewis, and McAdams<sup>(14)</sup> state that plate efficiency should be influenced but little by vapor rate. Byman and Keyes<sup>(13)</sup>, in a study of plate efficiencies in alcohol distillation, noted that low distillation rates gave somewhat higher efficiencies than those at moderate vapor velocities, but found no marked and continued variation of efficiency with rate of distillation. Vilbrandt et al<sup>(77)</sup>, in a study of the distillation of ethyl alcohol and water in a three plate bubble-cap column, found that plate efficiencies decreased with increased vapor velocity from 87 per cent at 0.4 feet per second vapor velocity to 70 per cent at 0.8 feet per second vapor velocity. Knit copper cloth packing above the bubble-cap increased the efficiency of the plate, but lowered the limiting superficial column velocity. Rhodes and Slachman<sup>(58)</sup>, operating a three plate column, found that in the distillation of ethyl alcohol and water, the Murphree efficiency was substantially independent of the rate of distillation; with benzene and toluene, the increased rate of flow appeared to cause a slight increase in the Murphree plate efficiency. Sherwood and Jenny<sup>(65)</sup> concluded that column capacities may be limited by pressure drop rather than entrainment.

Reflux Ratio.-Byman and Keyes<sup>(13)</sup> report that a marked change in reflux had little effect on plate efficiencies, although the higher ratio, in general, yielded somewhat higher efficiencies.

Robinson<sup>(60)</sup>, in his study of the plate efficiency of a continuous alcohol still, found no correlation of plate efficiency and reflux ratio. Dorsey<sup>(21)</sup>, in a distillation study of isopropyl alcohol and water, found maximum efficiency to exist at a reflux ratio of 0.93, when the column was one hundred per cent efficient. The column was sixty-seven per cent efficient under a reflux ratio of 1.13, and thirteen per cent efficient under a reflux ratio of 0.39. Shirk and Montonna<sup>(64)</sup> found that plate efficiency increased almost linearly with the reflux ratio when the rate of distillation remained constant.

Nature of the Components and Feed Concentration.-Plate efficiencies apparently are affected by the materials being distilled, although the exact relationships involved are still unknown<sup>(3)</sup>. Peters<sup>(54)</sup> shows that the efficiency of any column depends on the materials being separated. He also shows that feed composition affects column conditions through its effect on heat consumption. Carey, Griswold, Lewis, and McAdams<sup>(14)</sup> state however, that plate efficiency should be relatively independent of the nature of the constituent or the character of the vapor-liquid equilibrium for components of roughly the same molecular weight and liquid velocity. For a comparison of column efficiencies for different mixtures, we may refer to Table I.

Byman and Keyes<sup>(13)</sup> comment that one set of factors affecting plate efficiency include such physical properties as viscosity and surface tension. These investigators suggest that probably of more significance are such factors as diffusivity, partial and total

TABLE I

AVERAGE MURPHREE PLATE EFFICIENCIES FOR  
SEVERAL BINARY MIXTURES<sup>(52)</sup>

<u>Apparatus</u>	<u>Mixture</u>	<u>Plate Efficiency</u> (%)
1	Benzene and Toluene	57
1	Carbon Tetrachloride and Toluene	47
2	Benzene and Toluene	51.2
2	Ethanol and Water	72.6
3a(77)	Ethanol and Water	87.0
3b	Ethanol and Water	70.0

Apparatus 1 was a seven plate column, eight inches inside diameter, with a one-foot plate spacing, and one, four inch diameter, bubble-cap per plate; operated at vapor velocities between 0.7 and 1.2 feet per second.

Apparatus 2 was a single plate column, six inches inside diameter, with one bubble-cap of two and three-eighths inches inside diameter; operated at vapor velocities from 0.21 to 0.3 feet per second.

Apparatus 3 was a three plate column, eight and three-eighths inches inside diameter, with one, four and one quarter inch bubble-cap per plate, and plate spacing of five and one eighth inches. Test 3a was operated at vapor velocity of 0.4 feet per second and test 3b at vapor velocity of 0.8 feet per second.

pressures, effective film thickness, and temperature. They showed maximum plate efficiency to exist in the same region of liquid composition as maximum viscosity. Drickamer and Bradford<sup>(22)</sup> found a correlation between viscosity and plate efficiency from test data on fifty-four refinery fractionating columns.

Benedict<sup>(80)</sup> says that when feed composition is between 30 and 70 per cent, ordinary distillation run at minimum heat input has a fairly high thermodynamic efficiency, but at very low or very high feed concentrations, ordinary distillation is very inefficient.

### The Binary Mixtures

Equilibrium Data for the System Isopropanol - Water.-Isopropanol and water form a minimum boiling point mixture, the exact composition of the isotrope having long puzzled investigators. The most recent work by Langdon and Keyes<sup>(40)(41)</sup>, (Table II, Figure 1), indicates that the isotrope has a composition of 68.35 mo. per cent alcohol at atmospheric pressure. Vapor-liquid equilibrium data in the literature include the results of investigations by Lebo<sup>(34)</sup>, Evans<sup>(52)</sup>, and Brunjes and Bogart<sup>(11)</sup>, in addition to that of Langdon and Keyes. The data of Langdon and Keyes was obtained using improved techniques and is considered reliable.

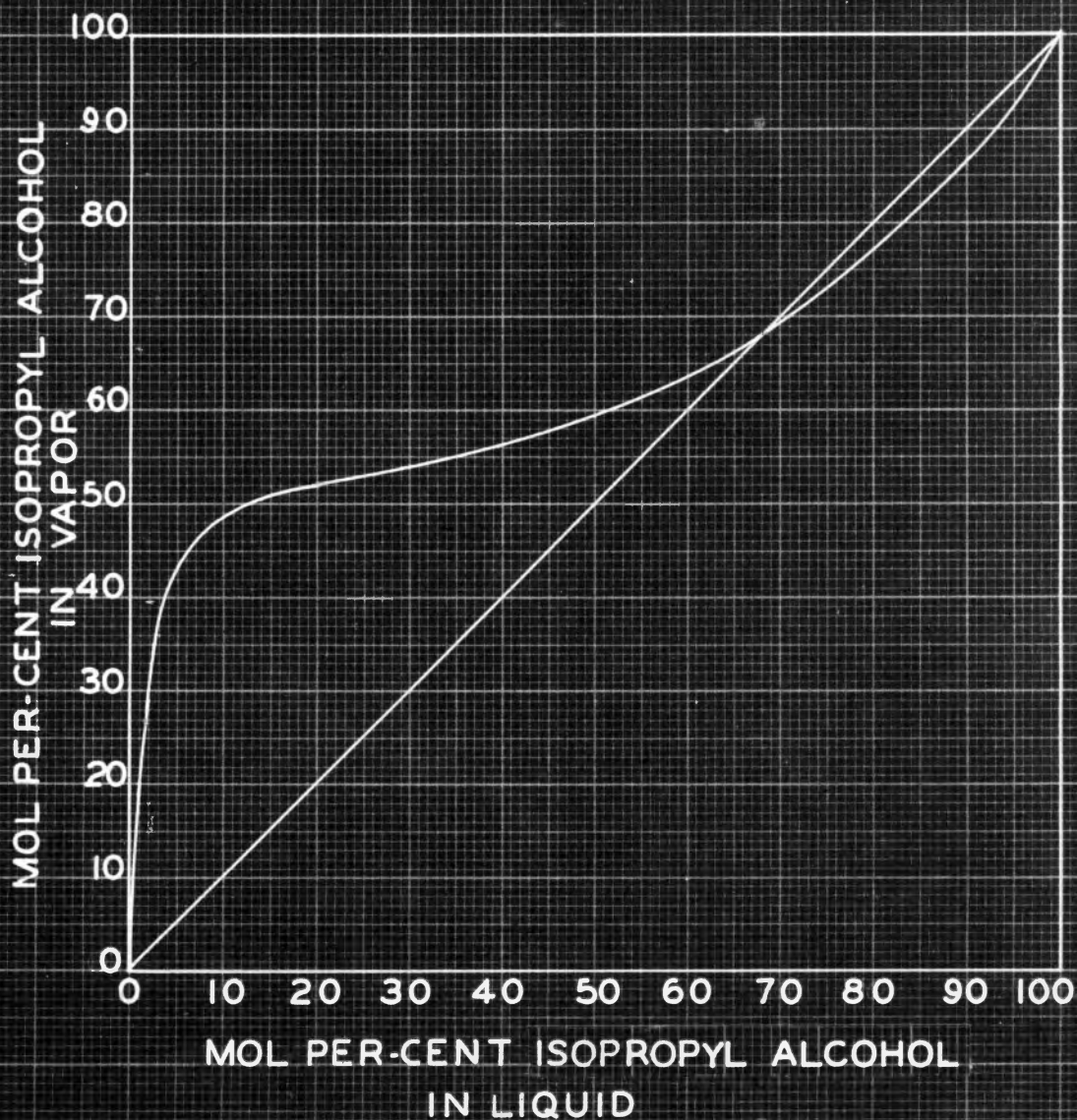
Distillation Characteristics of Isopropanol.-Langdon and Keyes<sup>(39)</sup> made a detailed study of the continuous rectification of mixtures of isopropanol and water in a four plate bubble cap column.

TABLE II  
EQUILIBRIUM DATA FOR ISOPROPYL ALCOHOL AND WATER AT 760 MM. (40)

X	Y*	X	Y*	X	Y*	X	Y*	X	Y*
1	18.0	21	52.25	41	56.50	61	64.35	81	77.9
2	29.2	22	52.45	42	56.80	62	64.85	82	78.8
3	36.0	23	52.65	43	57.10	63	65.35	83	79.75
4	40.6	24	52.85	44	57.45	64	65.90	84	80.55
5	43.45	25	53.05	45	57.80	65	66.45	85	81.5
6	45.65	26	53.30	46	58.10	66	67.0	86	82.5
7	47.0	27	53.50	47	58.45	67	67.6	87	83.55
8	47.7	28	53.70	48	58.75	68	68.15	88	84.05
9	48.25	29	53.95	49	59.05	69	68.70	89	85.75
10	48.75	30	54.15	50	59.40	70	69.30	90	86.85
11	49.2	31	54.35	51	59.75	71	69.90	91	88.05
12	49.6	32	54.55	52	60.10	72	70.6	92	89.30
13	50.0	33	54.75	53	60.5	73	71.35	93	90.6
14	50.35	34	55.0	54	60.95	74	72.10	94	91.9
15	50.7	35	55.2	55	61.4	75	72.90	95	93.25
16	51.05	36	55.4	56	61.85	76	73.70	96	94.60
18	51.60	38	55.8	58	62.85	78	75.35	98	97.30
19	51.85	39	56.0	59	63.35	79	76.20	99	98.65
20	52.05	40	56.25	60	63.85	80	77.05	100	100.00

X = Composition of liquid, mol per cent isopropyl alcohol  
 Y\* = Composition of vapor in equilibrium with liquid, mol per cent isopropyl alcohol

FIG. 1  
VAPOR-LIQUID EQUILIBRIUM DATA  
FOR ISOPROPYL ALCOHOL & WATER(40)



Their results may be summarized as follows:

1. Plate efficiencies showed nearly constant values over the composition range of 18-55 mol per cent alcohol but decreased rapidly above fifty-five per cent.
2. Changes in reflux ratio and superficial vapor velocity affected these efficiencies very little.
3. The parallel trends of plate efficiency and viscosity - liquid composition curves seemed to emphasize the significance of the liquid in plate column fractionations.

Manowitz<sup>(47)</sup> investigated the isopropanol - water system in a fifteen plate bubble-cap column under conditions of continuous rectification in a closed system. The following general conclusions may be made from his data:

1. Overall column efficiency increases with the reflux ratio.
2. The purity of the final product increases with the reflux ratio.

Dorsey<sup>(21)</sup>, investigating the isopropanol - water mixture in the same column, under similar conditions arrived at the following conclusions:

1. Operation of the column with only a rectifying section caused the feed to the column to be diluted on the lower plates, often not reaching its initial concentration until the eleventh and twelfth plates had been reached.



2. Higher reflux ratios resulted in a more pure product and tended to cause rectification to take place lower in the column.
3. Four adjacent plates seemed to do most of the enriching of the alcohol solution in the column, their location depending on the reflux ratio and on the steam pressure. These four plates operated at approximately one hundred per cent efficiency; the remaining plates, with the exception of the plates immediately above and immediately below the four plates, operated at almost zero efficiency.

Equilibrium Data for Toluene - Ethylene Dichloride.-Experimental vapor - liquid equilibrium data on this mixture are given by Jones, Schoenborn, and Colburn<sup>(35)</sup>, and by Colburn, Schoenborn, and Shilling<sup>(19)</sup>, (Table III, Figure 2). Both groups of investigators agree that the system toluene - ethylene dichloride is ideal and follows Raoult's Law closely.

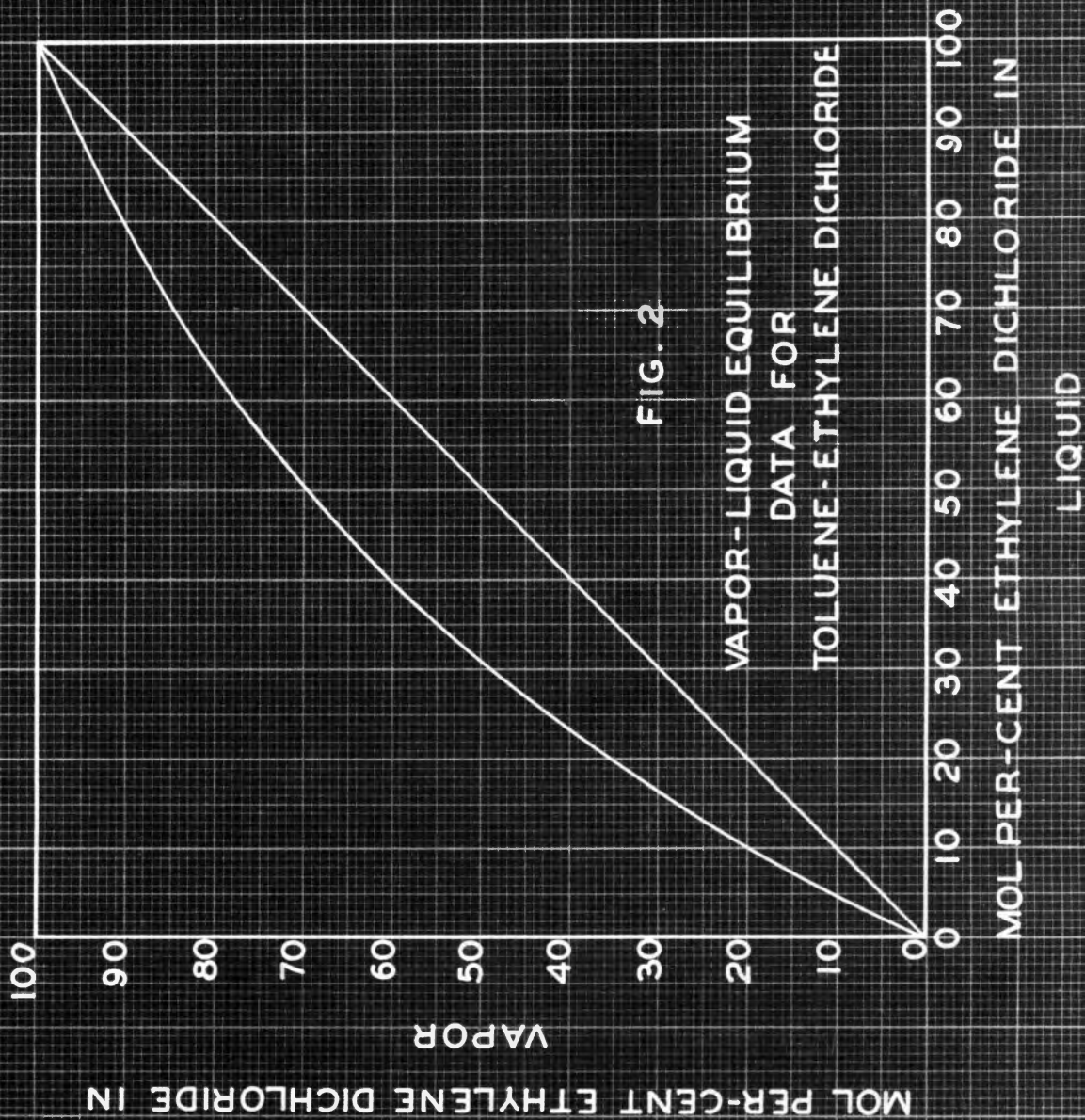
Index of Refraction as a Means of Analysis  
of Plate Compositions

Methods of Analysis.-Methods of plate composition analysis include specific gravity tests, boiling point determinations, and chemical analyses. The chief disadvantage of methods of these types is in the comparatively large quantity of sample needed for the

TABLE III

VAPOR - LIQUID EQUILIBRIUM DATA FOR TOLUENE -  
ETHYLENE DICHLORIDE AT 760MM. PRESSURE<sup>(35)</sup>

Temperature Degrees Centigrade	Mol Fraction Ethylene Dichloride in	
	Liquid <u>X</u>	Vapor <u>Y</u>
87.1	0.812	0.909
87.7	0.784	0.893
90.2	0.700	0.844
92.2	0.585	0.765
92.6	0.568	0.750
95.9	0.479	0.674
96.0	0.446	0.645
96.8	0.415	0.614
97.8	0.375	0.578
99.3	0.365	0.565
100.8	0.252	0.424
102.0	0.235	0.401
107.0	0.100	0.197
108.4	0.045	0.095



determination of the compositions. The removal of samples of this size from any plate is enough to destroy equilibrium conditions within a small column. Widely varying values of plate efficiencies have been obtained in the Unit Operations Laboratory of the Chemical Engineering Department of the Virginia Polytechnic Institute when data obtained from the analysis of 20 - 50 ml. samples were used in such calculations. The index of refraction method provides a convenient means for analyzing small volumes (two ml. or less) and thus avoiding the creation of any serious change in equilibrium conditions.

Aqueous Isopropanol Analysis.-Bennett and Garrett<sup>(6)</sup> made use of the index of refraction as a measure of volume per cent of isopropanol in aqueous solution, but since distillation calculations require the use of weight or mol per cent, their data are not easily applied. Bryan, Cooper, and Dorsey<sup>(12)</sup> developed this method for correlating refractive index with weight per cent. Kelly, Bennett, et al<sup>(36)</sup> further developed this method (Table IV, Figure 3) and found it to be simple, rapid, and accurate for analyzing aqueous isopropanol solutions from the plates of a fifteen plate fractionating column.

Toluene - Ethylene Dichloride Analysis.-Jones, Schoenborn, and Colburn<sup>(35)</sup> used a Spencer Abbe refractometer in analyzing compositions of this mixture from an equilibrium still. They found a regular variation in refractive index with mol per cent of ethylene dichloride present. The indices of refraction vary from 1.4939 at 0 mol per cent ethylene dichloride to 1.4423 at 100 mol per cent ethylene dichloride, (Table V, Figure 4).

TABLE IV

INDEX OF REFRACTION - COMPOSITION DATA  
FOR AQUEOUS ISOPROPANOL (36)  
(SECTION A)

Refractive Index	Weight Per Cent Isopropanol
------------------	-----------------------------

1.3330	0
1.3348	2
1.3418	10
1.3506	20
1.3582	30
1.3634	40
1.3678	50
1.3715	60
1.3743	70
1.3765	80
1.3774	90
1.3776	100

(SECTION B)

Isopropanol Sample Diluted:  
2 Volumes of Sample to 1 Volume of Distilled Water

1.3670	75
1.3680	80
1.3691	85
1.3701	90
1.3710	95
1.3718	100

FIG. 3  
REFRACTIVE INDEX-COMPOSITION DATA FOR ISOPROPYL ALCOHOL  
AND WATER(36)

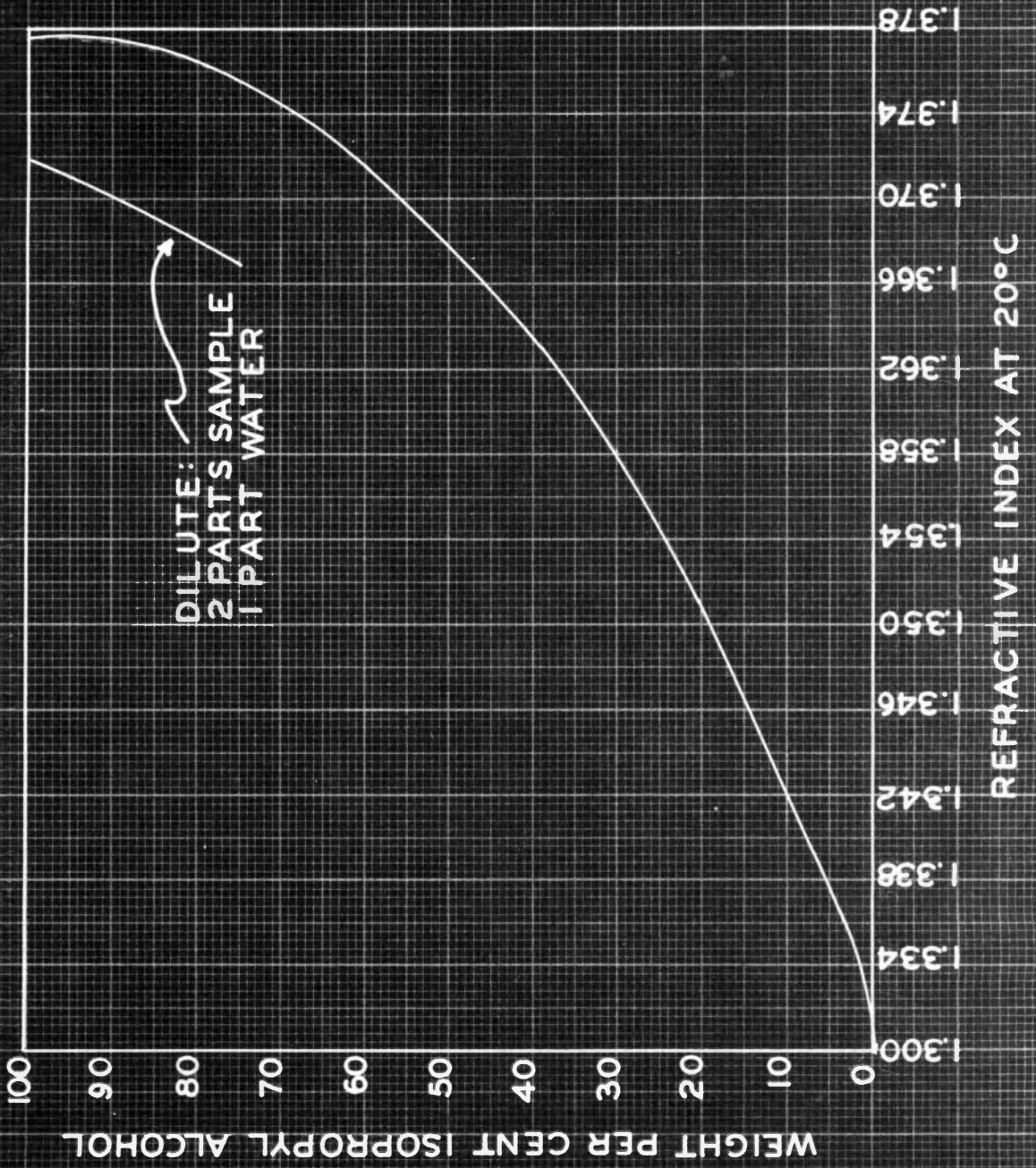
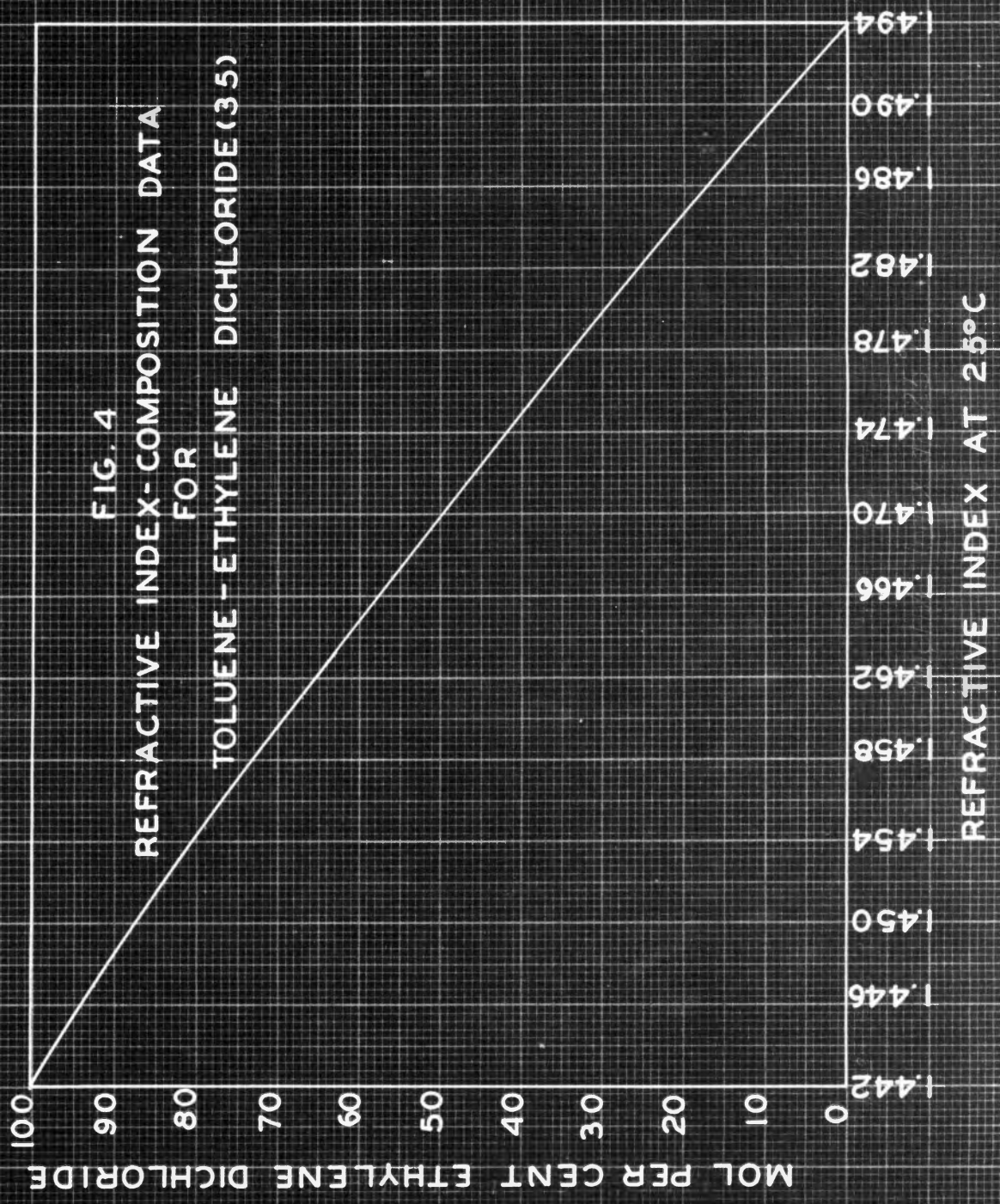


TABLE V

REFRACTIVE INDEX - COMPOSITION DATA FOR  
ETHYLENE DICHLORIDE - TOLUENE AT 25°C<sup>(35)</sup>

Mol Fraction Ethylene Dichloride	Refractive Index
0	1.4939
0.1221	1.4881
0.2144	1.4841
0.3230	1.4790
0.4663	1.4719
0.5600	1.4670
0.6699	1.4611
0.7900	1.4547
0.9250	1.4469
1.0000	1.4423





### III - EXPERIMENTAL

#### Purpose of Study

The purpose of this study is as follows: (1) to determine the operating characteristics of a fifteen plate fractionating column, operated in continuous distillation with the feed introduced on an intermediate plate; (2) to clarify the effects of the operating variables on column operation and plate efficiency; and (3) to determine the distillation characteristics of the systems isopropyl alcohol - water and toluene - ethylene dichloride.

#### Plan of Investigation

The plan to be used in conducting this investigation is as follows:

Redesign of Column Assembly.-The present column assembly will be redesigned and the necessary accessory equipment added to enable the introduction of feed at its boiling point to any intermediate plate in the column, to maintain a constant rate of distillation, and to determine the effects of operating conditions. In order that the column assembly will meet the above specifications the following accessory equipment will be installed:

(1). Introduction of feed at its boiling point. - A feed heater and feed lines to all intermediate plates will be installed.

(2). Constant Rate of Distillation. - A steam pressure regulator in the steam line to the column steam chest, an electric heater in the column steam chest, and a constant head tank and feed weir in the feed line will be installed for this purpose. A pump will be installed to supply recycled feed to the constant head tank.

(3). Control Devices. - Control devices in addition to the steam regulator and feed weir to be installed are: a manometer indicating steam pressure to the column steam chest, manometers indicating column pressure drop, a thermometer indicating steam chest temperature and a thermometer indicating feed temperature. Thermometers on each plate and in the reflux heater are a part of the present column assembly.

Analysis of Plate Concentrations.-The further development of the refractive index method of analysis of plate concentrations will be undertaken. Dorsey<sup>(21)</sup> has previously shown the relationship between the refractive index and the composition of aqueous isopropyl alcohol solutions. These results will be checked and the work further developed to enable the analysis of alcohol concentrations above 55 mol per cent. The data given in the literature<sup>(35)</sup> for the refractive index - composition data of Toluene and Ethylene Dichloride will be investigated for accuracy.

Operation of the Column.-The column will be operated under conditions of constant reflux ratio (3 to 1) and constant feed rate. The rate of distillation will be varied from 0.2 to 0.5 feet per second and the feed concentration from 3 to 10 mol per cent for the two binary systems isopropyl alcohol - water and toluene - ethylene dichloride. The reflux will be returned to the column at the temperature of the top plate and the feed will be introduced as a liquid at its boiling point in each test. The feed will be introduced at the proper feed plate in the column as determined by the feed concentration and the liquid concentrations on the plates.

Evaluation of Results.-The distillation characteristics of the binary mixtures will be shown by a plot of the plate concentration gradients. Murphree Plate Efficiencies will be calculated and compared for the individual tests. The actual operating lines followed in the distillation will be calculated and compared with the McCabe - Thiele operating lines.

Materials

The materials used in the investigation were as follows:

Alcohol, Isopropyl, "Petrohol". (55 gallons)

Specifications:

Specific Gravity 20/20 ..... 0.789  
Purity ..... 99.3%  
Distillation - Initial ..... 81.7°C.  
                  Final ..... 82.4°C.

Use - Portion of Distillation Mixture Obtained from the Standard Alcohol Company, Linden, New Jersey.

Water, tap, refractive index at 20°C. 1.3330. Used for binary mixture and condenser cooling. Obtained from the water lines in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute.

Toluene, purified toluol, (20 gallons)

Specifications:

Specific Gravity 25/4 ..... 0.862  
B.P. at 760 mm ..... 110.6°C.  
Refractive Index at 25°C ..... 1.4939

Use - Used in preparing binary mixture

toluene - ethylene dichloride.

Manufactured by J. T. Baker Chemical Company, Phillipsbury, New Jersey.

Obtained from Phipps & Bird, Inc., Richmond, Virginia.

Ethylene Dichloride, technical grade, (20 gallons).

Specifications:

Specific Gravity 25/4 ..... 1.245  
B.P. at 760 mm ..... 83.6°C.  
Refractive Index at 25°C ..... 1.4423

Use - Used in preparing binary mixture

toluene - ethylene dichloride

Obtained from Carbide and Carbon Chemicals Corporation, Charleston,  
West Virginia.

Steam, process, high (50 p.s.i.g.) and low (15 p.s.i.g.)  
pressure. Used for heating feed, reflux, and steam chest. Obtained  
from the lines of the Chemical Engineering Laboratory of the Virginia  
Polytechnic Institute.

Insulation, pipe. Magnesia, 85% Johns - Manville. Obtained  
from Tidewater Supply Company, Roanoke, Virginia.

Gaskets, Cork, 1/8" thick. Used over the entire column  
assembly. Obtained from Auto Spring and Bearing Company, Roanoke,  
Virginia.

Apparatus

The apparatus used in this investigation consisted of a fifteen plate bubble-cap column and accessories.

Column, bubble-cap - 15-plate. The column (Figure 5) consisted of fifteen bronze plates, each containing a tangentially opposed flow type bubble-cap, a downcomer, an overflow weir, and taps for a vapor space thermometer and a sampling outlet. Plate specifications were as follows: (See Figure 6).

Number of Plates .....	15
Number of bubble-caps per plate .....	1
Plate spacing .....	5-7/8 in.
Plate I.D. ....	8-3/8 in.
Liquid depth on plates .....	1-3/8 in.
Bubble-cap O.D. ....	3-7/8 in.
Slots per bubble-cap .....	32
Slot width .....	1/8 in.
Slot height .....	7/8 in.
Slot area per cap .....	3.4 sq/in.
Vapor Riser I.D. ....	2 in.
Downcomer I.D. ....	1-1/16 in.

The column contained 84 inches of 1/2 inch copper tubing (142 sq. in. heating surface) in the steam chest, carrying low pressure steam.

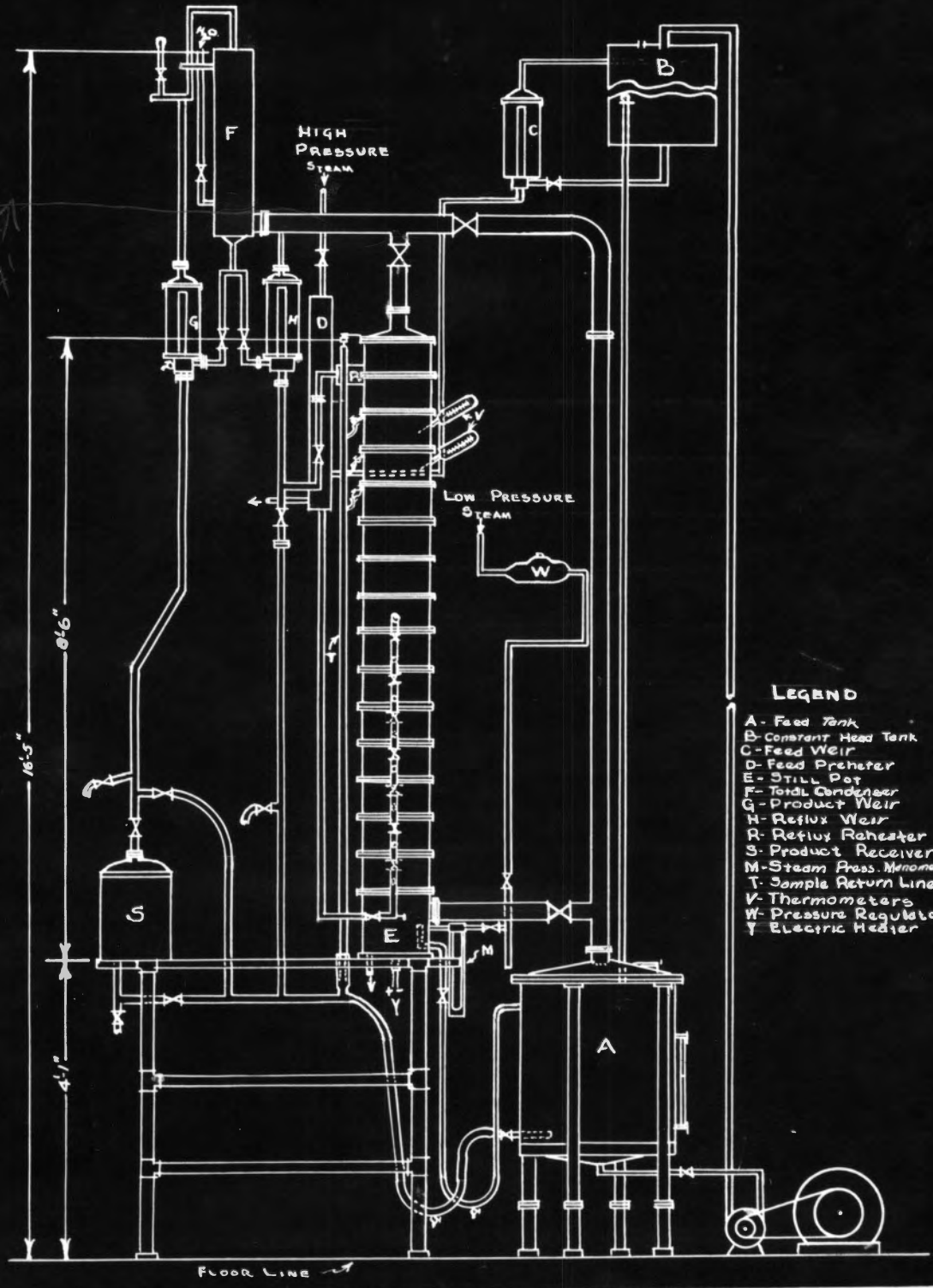
Obtained from the Vulcan Copper and Supply Company, Cincinnati, Ohio.

DEPARTMENT OF CHEMICAL ENGINEERING  
 VIRGINIA POLYTECHNIC INSTITUTE  
 BLACKSBURG, VA.

FRACTIONATING COLUMN  
 &  
 ACCESSORIES

DRAWN: *WJB*  
 CHECKED: *WJB*  
 APP'D.:

DATE: 2-20-47  
 SCALE:  $\frac{3}{4}'' = 1'0''$   
 FIG. No. 5



LEGEND

- A - Feed Tank
- B - Constant Head Tank
- C - Feed Weir
- D - Feed Preheater
- E - Still Pot
- F - Total Condenser
- G - Product Weir
- H - Reflux Weir
- R - Reflux Reheater
- S - Product Receiver
- M - Steam Press. Manometer
- T - Sample Return Line
- V - Thermometers
- W - Pressure Regulator
- Y - Electric Heater

Stillpot, copper, 50 gallon capacity. Contained 36 feet of 3/4 copper coil through which cooling water was circulated. Used only as a collecting tank. Manufactured by the Vulcan Copper and Supply Company, Cincinnati, Ohio.

The following accessories were used in conjunction with the column:

Condenser, total. Consisted of an 18 gage copper cylinder, 6-1/2 inches O.D. and 31 inches long. Condensing surface was furnished by 21 copper tubes 1/4 inch in diameter and 27 inches long, cooling surface 442 sq. in. Used for condensing the overhead distillate from the column. Constructed in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute.

Weirs, product and reflux. Consisted of two 5-1/2 inch I.D., 12 inches in length, pyrex glass cylinders, with suitable vents and flanged copper connections. Used for measuring product and reflux flow. Manufactured by the Vulcan Copper and Supply Company, Cincinnati, Ohio.

Weir, feed. Consisted of a 12 inch section of 6 inch galvanized iron pipe, topped by a 5 inch section of 5-1/2 inch I.D. pyrex glass, with bolted copper flanged connections. Used to measure the feed to the column. Manufactured by the Vulcan Copper and Supply Company, Cincinnati, Ohio.



Tank - Head, Constant. Made from a standard 35 gallon drum, 18-1/2 inches O.D. and 29 inches in height. Used to maintain a constant head of feed. Constructed in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute.

Pump, Eco single impeller 1/2 inch gearless, bronze. Used to supply feed to the constant head tank. Obtained from the Eco Engineering Company, Newark, New Jersey.

Motor, 1/4 Hp., 60 cycle, 1750 R.P.M., Model 1155618. Used to drive the Eco pump. Obtained from Sears Roebuck and Company, Philadelphia, Pennsylvania.

Regulator, Steam Pressure, 1/8 to 15 pound capacity, bronze. Used to regulate low pressure process steam to the column steam chest. Manufactured by Kieley and Mueller, Inc., North Bergen, New Jersey.

Heater, Electric, 2000 Watt, 230 Volt, Serial Number 15 x 831. Used to aid in controlling the temperature of the steam chest. Manufactured by General Electric Corp., Schenectady, New York.

Control, Thermostat. (One) 220 Volt A.C., range 176 - 240°F. Used to control the electric heater. Manufactured by General Electric Corp., Schenectady, New York.

Trap, Steam, 150 pounds, bucket type. (One). Used on high pressure steam line from the feed heater. Manufactured by the Strong-Carlisle Manufacturing Company, Cleveland, Ohio.

Trap, Steam, 15 pounds, type Sarco 87. (Two). Used on low pressure steam lines from the steam chest and reflux heater. Manufactured by the Sarco Manufacturing Company, New York, New York.

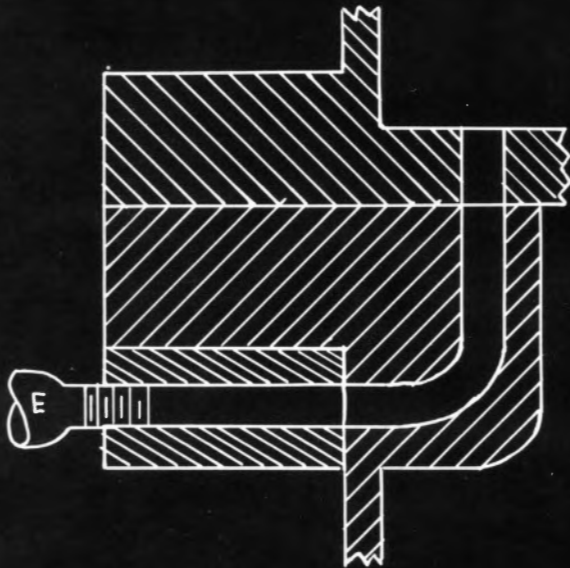
Heater, Reflux. (One). Countercurrent exchanger, copper, 18 inches long, 2 inch copper pipe concentric with 1/2 inch copper pipe. Heated by low pressure (15 psig.) steam with a 1/8 inch thermometer well in the end of the heater. Used to heat the reflux returned to the column. Constructed in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute.

Heater, Feed. (One). Copper pipe 52 inches long, 2 inch diameter, concentric with 3/4 inch copper pipe. Feed line entered 6 inches from the bottom and flowed in the same direction as the high pressure (50 psig.) steam. Used to heat the feed to the column. Constructed in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute.

Separator, Entrainment. (One). Constructed of standard 4 inch iron pipe, 12 inches in height with top and bottom of 1/4 inch sheet iron. Steam was bubbled through a water trap to improve the quality before entering the column steam chest. Constructed in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute.

System, Sample Return. (One). (See Figure 5a). Used to allow samples to be taken continuously from the plates of the column without disturbing the equilibrium. Constructed in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute.

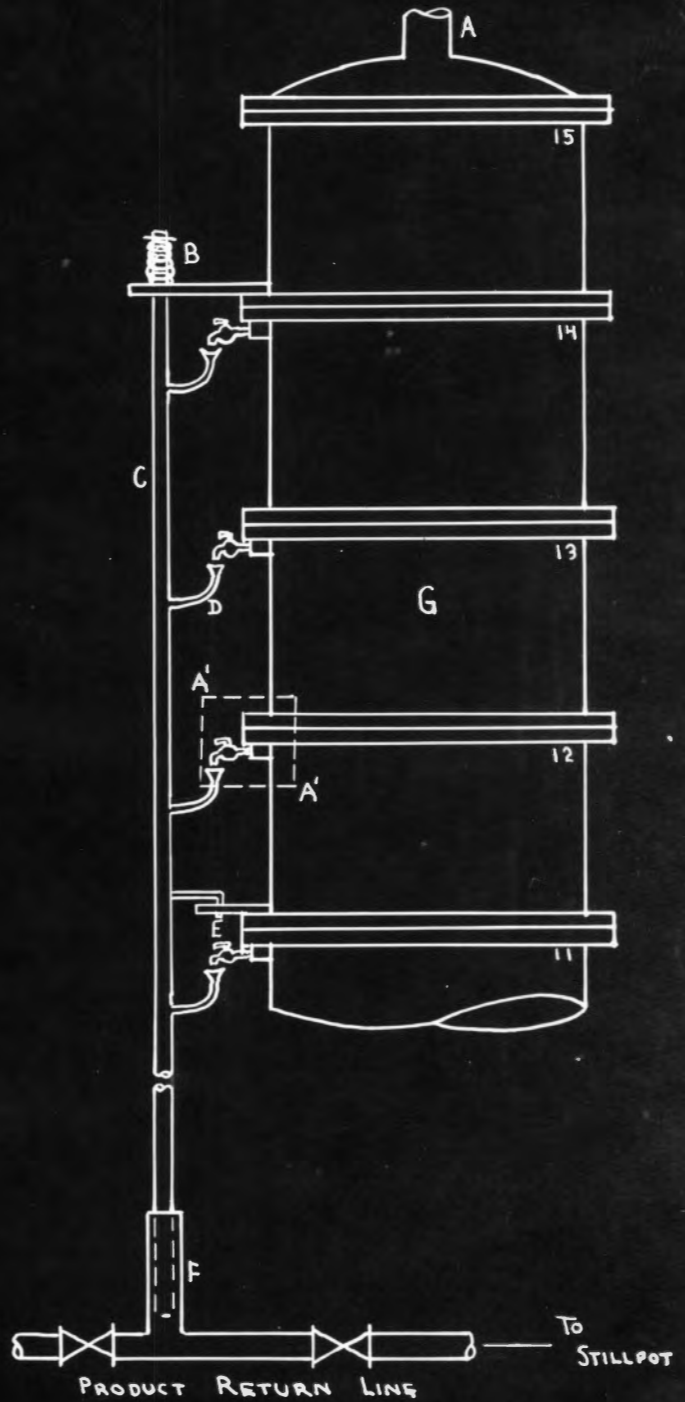
Manometers, Mercury, 19 inches high. (Three). Used to indicate steam pressure to the column steam chest and pressure drop over the column. Constructed during the course of the investigation.



SECTION A-A  
SAMPLING PETCOCK INSTALLATION

LEGEND

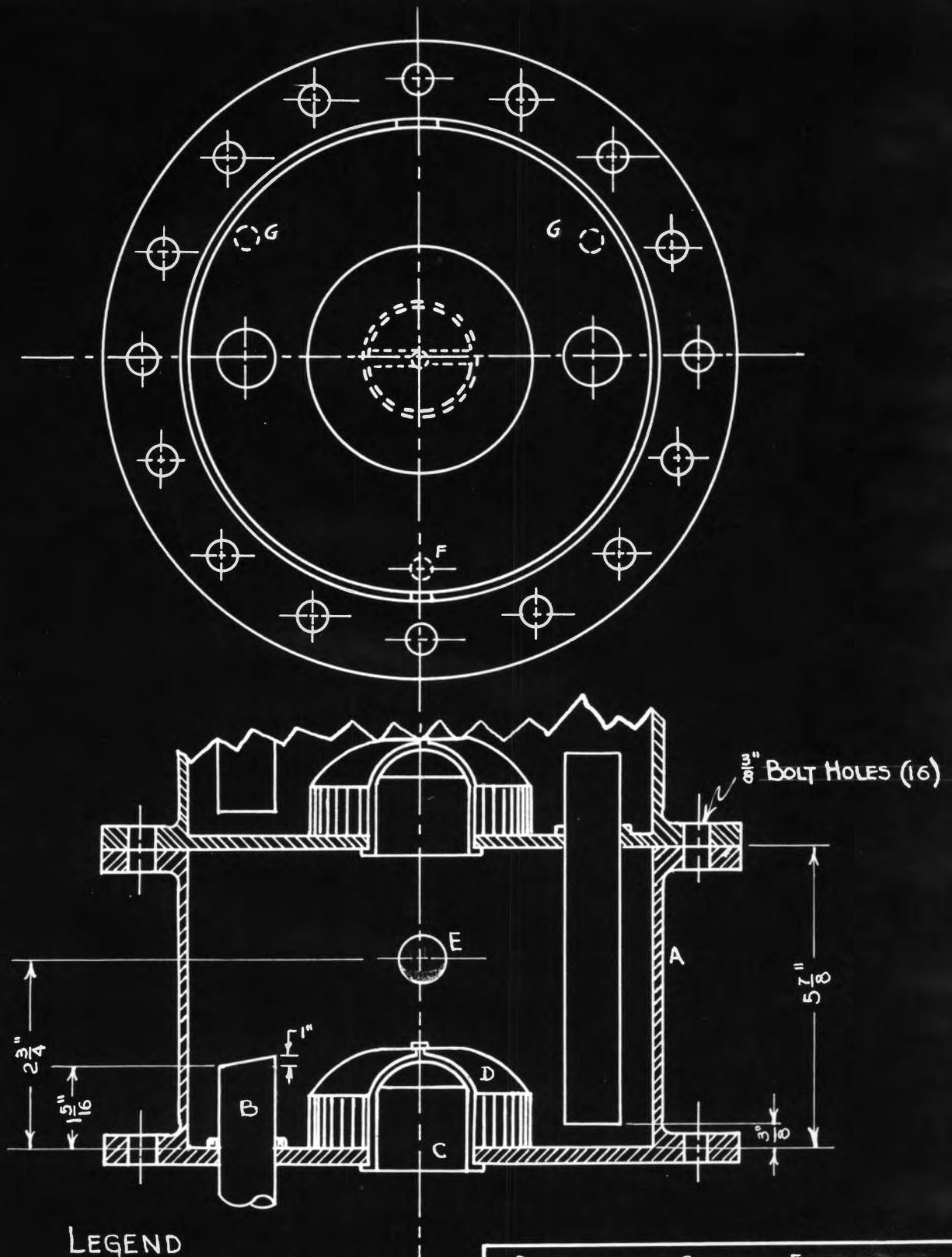
- A - VAPOR LINE TO CONDENSER
- B - COIL SPRING (IN TENSION)
- C - 3/4" COPPER TUBING
- D - 1/4" COPPER TUBING (FLARED AT END)
- E - 1/4" PETCOCK
- F - 1" COPPER TUBING
- G - EXPERIMENTAL COLUMN



NOTE: COIL SPRING (B) HOLDS RECEIVING TUBES (D) AGAINST MOUTH OF PETCOCKS. RETURN TUBE (C) LOWERED WHEN SAMPLES ARE TAKEN.

NOTE: TUBE (C) SUPPORTED FROM PLATES 4, 8, 11, 14; GROOVES IN SUPPORT FROM PLATE 14 FIT KEYS ON (C) TO PREVENT TURNING.

DEPARTMENT OF CHEMICAL ENGINEERING VIRGINIA POLYTECHNIC INSTITUTE BLACKSBURG, VIRGINIA	
DETAILS OF SAMPLING SYSTEM	
DRAWN BY: C.L.D.	DATE: 1-30-46
INKED BY: C.L.D.	SCALE: NONE
APPVD BY: <i>[Signature]</i>	FIG. No: 5a



LEGEND

- A - BRONZE CYLINDER, 8 3/8" I.D.
- B - DOWN PIPE, 1 1/16" I.D.
- C - VAPOR PIPE, 2" I.D.
- D - VULCAN BUBBLE CAP, 3 7/8" O.D.
- E - THERMOMETER INLET
- F - SAMPLING TUBE INLET, 5/16" D.
- G - SAMPLING TUBE INLETS - PLATE 2

DEPARTMENT OF CHEMICAL ENGINEERING VIRGINIA POLYTECHNIC INSTITUTE BLACKSBURG, VIRGINIA	
DETAILS OF <b>EXPERIMENTAL PLATE</b>	
DRAWN BY: <i>AJB</i> CHECKED BY: <i>AJB</i> APPVD. BY:	DATE: 2-20-47 SCALE 3/8" = 1'0" FIG. No. 6

Thermometers, Precision. (Twelve). Range 40 - 240°F., increments 2°F. Used to determine plate temperatures. Manufactured by the Fisher Scientific Company, Pittsburg, Pennsylvania.

Thermometers, Weston, metal. (Four). Range 0 - 220°F. and 20 - 250°F., increments 2°F. Used to determine reflux, steam chest and feed temperatures. Manufactured by the Weston Electrical Instrument Corp., Newark, New Jersey.

Meter, water, "Niagara". (One). Serial Number 1013790. Used to meter the water to the total condenser. Manufactured by the Buffalo Meter Company, Buffalo, New York.

The following items were used in analyzing samples and in determining refractive index - composition data:

Refractometer, Spencer Abbe Model 547, range 1.3000 to 1.7100, with water bath and thermometer 0 - 100°C. Used to determine sample concentrations. Manufactured by the Spencer Lens Company, Buffalo, New York.

Constant Temperature Assembly. Consisted of a 5 gallon steel constant head tank, heated by a 115 Volt A.C. electric heater; controlled by a Model B, 115 Volt, 60 cycle, Precision Temperature Regulator, manufactured by the Eastern Engineering Company, New Haven, Connecticut; and actuated by mercury immersion bulb in the constant head tank. Used to control the temperature of the refractometer within  $\pm 0.5^\circ\text{C}$ .

Bottles, glass, 5 ml. (Twenty-five). Used in collecting samples from the plates of the column. Obtained from the stockroom of the Chemical Engineering Department of the Virginia Polytechnic Institute.

Straws, paper, drinking. (Five hundred). Used for pipetting samples onto the refractometer lens. Obtained from Rose's Department Store, Blacksburg, Va.

Balance, analytical. (One). Used to weigh pycnometers. Obtained from the Chemical Engineering Department of the Virginia Polytechnic Institute.

The following equipment was obtained from the Chemistry and Chemical Engineering stockrooms and used in determining refractive index - composition data for isopropyl alcohol and water:

Pycnometers. (Two). Twenty-five ml., unadjusted Gay - Lussac, with ground-in perforated glass stoppers. Used to determine the density of the original isopropyl alcohol. Manufactured by the Fisher Scientific Company, Pittsburg, Pennsylvania.

Burettes. (Two). Fifty ml., glass.

Stands. (Two). Iron ring, with burette clamps.

Graduate, glass, fifty ml.

Thermometers. (Two). Mercury, 0 - 220°F. Increments 2°F.

Breakers. (Two). Two hundred and fifty ml., glass.

Stirring Rod. (One). Glass, 10 inches long.

Pipette. (One). Glass, two ml.

Bottles. (Twenty). Two hundred and fifty ml. glass, with ground glass stoppers. Used to contain standard solutions of aqueous isopropyl alcohol.

#### Method of Procedure

The first step in the procedure was to further develop the method<sup>(21)</sup> for determining the percentage of isopropyl alcohol in aqueous solution by its refractive index. It was decided to make up samples of isopropyl alcohol and water from 0 - 100 weight per cent isopropyl alcohol. Then, the refractive index at 20°C. could be determined and a relationship between refractive index and composition plotted. This was done by Kelly, Bennett, et al<sup>(36)</sup> in the following manner:

Density Determination of Original Isopropyl Alcohol.-In order to make aqueous solutions of isopropyl alcohol of known concentrations it was first necessary to know the original concentration of the isopropyl alcohol. Twenty-five ml. Gay - Lussac pycnometers were used in making the density determination and the test was made in duplicate as a check on the results. The pycnometers were weighed at 25°C. while empty and while filled with distilled water. The density of the water as reported in the International Critical Tables, was used as basis for further computations. The same procedure of weighing the

pycnometer filled with the isopropyl alcohol was followed and the ratio of the weight of the isopropyl alcohol to the weight of the distilled water, gave the density of the isopropyl alcohol. From density - weight percentage tables<sup>(52)</sup>, the weight percentage of the original alcohol was determined to be 99.3 per cent.

Preparation of Samples.-Duplicate samples of isopropyl alcohol solutions from 0 - 95 weight per cent were prepared. Calculations showed the exact volumes of alcohol and water required to prepare 100 ml. of the desired concentration. The required quantities of isopropyl alcohol and distilled water to give these concentrations were introduced through 50 ml. burettes into 250 ml. glass bottles which were then sealed with glass stoppers.

Calibration of Refractometer.-A Spencer Abbe Refractometer, Model Number 547 was calibrated by the use of distilled water at the lower range and by a glass standard at the middle range. The refractometer was calibrated at 20°C. to the refractive index of distilled water (1.3330) and to the refractive index of the glass standard (1.5108). The readings checked and the calibration of the instrument was assumed to be correct.

Analysis of Samples.-The duplicate 250 ml. samples were placed in a water bath to bring them to approximately 20°C. The refractometer was then adjusted by means of the constant temperature assembly until the temperature was at 20°C  $\pm$  0.5. Samples were taken from each of the 250 ml. sample bottles by means of paper drinking



straws and placed on the lens of the refractometer. Two readings for each sample were made and if the readings for the duplicate samples were not within 0.0002, the samples were prepared again and the refractive indices redetermined. A plot of the results gave the relationship shown in Figure 3.

Refractive Index of Diluted Samples.-For concentrations between 80 and 100 weight per cent isopropyl alcohol the refractive index change was very small (1.3770 - 1.3776). It was decided to dilute the samples with distilled water in order to increase the accuracy in the range from 75 - 100 weight per cent isopropyl alcohol. Twenty-five ml. samples were taken from the known sample of 75 - 100 weight per cent in the 250 ml. bottles and diluted with an equal volume of distilled water. The range of refractive index was improved very little and so new samples were made by diluting 25 ml. of the known samples with half their volume of distilled water. The range of the refractive index from 80 - 100 weight per cent became 1.3680 to 1.3718 by using this procedure. A plot of the results is shown in Figure 3. Accuracy in the range of 80 - 100 weight per cent was  $\pm 2$  weight per cent isopropyl alcohol.

The second step in the procedure was to redesign the fifteen plate column assembly in the Chemical Engineering Laboratory of the Virginia Polytechnic Institute. (See Figure 5). During the previous investigation by Dorsey<sup>(21)</sup> the column was operated as a closed system

with feed introduced as vapor to the bottom plate, and hence the column operated with only a rectifying section. In order to introduce feed at its boiling point on intermediate plates and to maintain control of operating variables, accessory equipment was constructed and installed. This equipment is listed and described as follows.

Constant Head Tank: To insure a constant rate of feed to the column a constant head tank was constructed from a standard 35 gallon steel drum 18 1/2 inches O.D. and 29 inches in height. Provisions were made for the liquid to be pumped to the top of the tank and the overflow to be returned to the collecting tank on the ground floor through 3/8 inch galvanized iron pipe. A 1/2 inch galvanized iron pipe leading from the bottom of the tank enabled the withdrawal of feed to the feed weir.

Feed Pump: A pump to supply the constant head tank with recycled feed was installed on the ground floor. The pump was an Eco single impeller, gearless, bronze pump manufactured by the Eco Engineering Company. It was driven by a 1/4 Hp., 110 Volts, 1750 R.P.M. motor, obtained from Sears, Roebuck and Company. The diameter of the pump pulley was 1 inch and that for the motor was 2 inches. The pump was packed with lead packing to prevent leakage around the bearing. All fittings and piping to and from the pump were of 3/8 inch galvanized iron.

Entrainment Separator: A steam - water separator was constructed to improve the quality of low pressure steam furnished to

the column steam chest. The separator was constructed of standard 4 inch iron pipe, 12 inches in height, with top and bottom of 1/4 inch standard sheet iron. Provisions were made for the steam to bubble through a 3 inch water trap to remove entrained water. This separator was installed in the low pressure steam line to the column steam chest. The use of this device increased the quality of the steam from approximately 70 to 95 per cent.

Reflux Heater.-Since the reflux heat exchanger had a heating area of only 35 square inches this was insufficient to maintain the reflux at the proper temperature (approximately 170°F). A new heat exchanger having a heating area of 50 square inches was constructed of 2 inch copper pipe concentric with the 1/2 inch copper pipe carrying the reflux. Heating was effected by low pressure steam (15 psig.) countercurrent to the reflux flow. A 1/8 inch thermometer well was bronzed into the end of the 1/2 inch copper pipe to allow for a thermometer to indicate reflux temperature.

Feed Heater.-The feed heat exchanger, which was a part of the assembly before this investigation was begun, consisted of a vertical 52 inch length of 2 inch copper pipe concentric with a 3/4 inch copper pipe heated with low pressure steam. The feed entered at the top of the heater and flowed by gravity in the annular space between the two pipes in the same direction as the steam. It was found that the more volatile component in the feed flashed back into the feed weir when the feed was heated to near its boiling point.

This difficulty was solved by welding a copper nipple into the side of the 2 inch pipe, 6 inches from the bottom. The feed was introduced into the heat exchanger by a 1/2 inch galvanized iron pipe through this connection and the former entrance at the top was sealed by a 1/2 inch copper plug. It was found that the decreased heating surface was insufficient with low pressure steam (15 psig.) and therefore high pressure steam (50 psig.) was used. A Strong, bucket type, steam trap, 150 pounds capacity, was inserted in the condensate line from the feed heater in the place of a Sarco Number 87 low pressure steam trap. The feed was therefore forced into the column by its own vapor pressure as well as by gravity.

Feed Lines.-Feed lines from the feed heater to the fifth through the eighth plates from the bottom of the column were a part of the original column assembly. These lines were of 1/2 inch copper pipe and all connections were made to the plates from one side of the column only. This meant that feed was placed on every odd-numbered plate at the overflow weir instead of at the downcomer from the plate above. This condition was corrected, and provisions made for feed entrance on lower plates at the proper positions on plates 1 through 4. By the use of a 1/2 inch copper tee, the original feed line was changed so that a length of 1/2 inch copper pipe extended up each side of the column connecting alternate plates. One-half inch copper globe valves were installed in the feed line to each plate, enabling the feed to be introduced on any plate from 1 to 8, numbering from the bottom of the column.

Heating Coil.-It was necessary to install a copper coil, for heating the column with steam, into the column steam chest beneath the bottom plate. This was done by dismantling the column and hoisting the entire column with a chain hoist so that access to the steam chest could be obtained. Six feet of standard  $3/8$  inch copper tubing (heating surface 56.4 sq. in.) was wound into a 9 inch coil and placed in the steam chest. Two  $3/8$  inch holes were drilled on opposite sides of the chest to accommodate the coil connections to the steam supply. These connections were secured by means of copper flanged fittings. The coil was connected to the low pressure process steam line by means of  $1/4$  inch black iron pipe and the condensate was exhausted through a Sarco Number 87 steam trap to the basement drain. It was found that the heat furnished by this coil was insufficient for operation of the column with feed rates above 250 gms/min. and therefore it was replaced with a coil consisting of 84 inches of  $1/2$  inch copper tubing (heating surface 132 sq. in.).

Pressure Regulator.-A pressure regulator was installed in the low pressure steam line to control the steam pressure to the coil in the steam chest. This was a bronze regulator with capacities from  $1/8$  to 15 pounds pressure and connections for  $1/2$  inch iron pipe. The connections were made as shown in Figure 5.

Electric Heater.-In order to give more accurate control of steam chest temperature an electric heater was also installed in the steam chest. A 2-inch diameter hole was drilled in the base of the column, the heater was secured in place, and the connection sealed

with cork and lead gaskets. The heater was thermostatically controlled. The bulb which actuated the thermostat was placed in the vapor space in the steam chest through a  $3/8$  inch hole. The copper lead line from the bulb to the thermostat was sealed in the  $3/8$  inch opening by means of a cork stopper which was held in position by a copper flanged fitting. The heater and thermostat were connected to the 220 Volt lines in the laboratory. While the column was dismantled a  $1/8$  inch thermometer well was bronzed into the steam chest to enable the insertion of a thermometer for indicating vapor space temperature.

Water Line to Collecting Tank.-Since the waste returning to the collecting tank was at the temperature of the steam chest ( $190 - 230^{\circ}\text{F.}$ ) there was a possibility that a back pressure might be built up in the tank. It was therefore necessary to cool this liquid as it entered the tank. This tank is normally used as a stillpot and contained a 36 foot length of copper coil through which water could be circulated to cool the incoming waste. Valves were so placed that either steam or cooling water could be circulated through the coil.

Feed Weir.-A feed weir was installed in the pipe line from the constant head tank to the feed heater. The weir consisted of a 5 inch section of  $5\ 1/2$  inches I.D. pyrex glass cylinder, on the top of a 12 inch section of 6 inch galvanized iron pipe. Liquid drained from the weir through a  $7/64$  inch hole drilled in the copper standpipe. The height of liquid in the weir was controlled by a  $1/2$  inch globe valve.

Lagging: The column, reflux heater, feed heater, steam lines, and all lines carrying hot feed to the column were lagged with 1" Johns Manville pipe insulation and with 85 per cent magnesia.

Manometers: Mercury manometers were installed on the low pressure steam line to the steam chest, and on lines from the vapor spaces in the steam chest and the top plate. The former was to indicate the steam pressure to the steam chest and the latter were to indicate the pressure drop over the column.

The third stage in the procedure was the calibration of weirs and thermometers.

Weir Calibrations: The feed weir was calibrated by weighing the quantity of feed flowing through the weir at different settings for three minute intervals. The liquid was collected by removing the bonnets on the valves leading to the feed plates and allowing it to flow into a glass jar. It was found that the differences in weight of feed for the same weir setting between the first and eighth plates were negligible. Thereafter for the feed weir calibration, the feed was removed through the valve bonnet on the feed line to the fourth plate.

The product and reflux weirs were calibrated with hot distillate (157°F.) in the same way as the feed weir. Calibration curves were made for isopropyl alcohol and water and are shown in Figure 8.

Since only two tests were made with the system toluene - ethylene dichloride it was considered unnecessary to make complete weir calibrations for this system, i.e., the weirs were calibrated for the one flow rate used in the two tests.

Thermometer Calibrations.-The thermometers measuring the temperatures of the reflux, top plate, steam chest, and feed were calibrated against a standard glass mercury thermometer by simultaneous immersion in a water bath.

The fourth stage in the procedure was the operation of the column assembly. All letters in the following description refer to Figure 5.

Preliminary.-The binary mixture of isopropyl alcohol with a concentration approximately four weight per cent greater than that finally desired was placed in the collecting tank (A) and the feed pump started to fill the constant head tank (B). It was necessary to make the original concentration greater than that finally desired since hold-up in the column and weirs decreased the percentage of the more volatile component in the feed and make-up liquid was not added during a test. Steam lines to the steam chest (E) and the feed heater (D) were drained of condensate and then adjusted to the desired pressure. The current to the electric heater (Y) was turned on and the thermostat adjusted to the temperature desired in the steam chest (E). The feed line from the constant head tank (B) was opened



and the feed weir (C) adjusted to the desired rate of feed; the temperature of the feed was adjusted by the valve controlling the high pressure steam line to the feed heater (D). The feed was placed on some intermediate plate by opening the valve in the feed line to that plate. Cooling water to the condenser (F) and to the collecting tank (A) was then turned on, the former at the rate of approximately 8 gallons per minute and the latter at the rate of approximately 1 gallon per minute. Barometric pressure was determined and recorded.

Operation.-As soon as the vapor from the column began to condense (condenser F), the steam pressure to the steam chest was readjusted by the pressure regulator (W) to give the desired temperature in the steam chest vapor space. The weirs (G & H) were adjusted to give a reflux ratio of 3 to 1. The low pressure steam to the reflux heater (R) was adjusted to maintain the reflux return to the column at the same temperature as the top plate. The petcocks on each plate were opened to give a flow of approximately 20 drops a minute from each into the sample return line (T).

After these steps had been taken, the proper temperature to which the feed should be heated to raise it to the temperature of a liquid at its boiling point, and the proper feed plate could be determined. Samples of 2 ml. each from the feed, the feed plate, and the two plates above and below the feed plate were taken in 5 ml. glass bottles with screw caps. The feed sample was taken by collecting it from the overflow from the constant head tank (B) at

the point at which it flowed into the collecting tank (A). The other samples were taken from the petcocks on the plates. The refractive indices of the samples were determined and this data gave the concentration of the feed and of the liquid on the plates. Using this data, the feed was placed on that plate on which the liquid concentration most closely approximated the concentration of the feed, and the steam pressure to the feed heater (D) adjusted to heat the feed to its boiling point.

The column was operated at steady state conditions for a minimum of two hours. Samples were taken from the top plate, feed plate, bottom plate, and feed every half hour. If an analysis of the samples showed that the feed plate location should be changed this was done. All other operating variables, steam pressure, feed rate, feed temperature, and reflux ratio were maintained constant. When these operating variables had remained constant for an hour, and the analysis of the samples showed no change in concentration it was assumed that equilibrium had been reached.

Collection of Data: Samples of from 2 - 4 ml. were collected from each plate, the product, the feed, and the waste in 5 ml. glass sample bottles fitted with screw caps. The product sample was taken from a petcock on the product weir (G), the waste sample from a 1/4 inch valve in the waste line, and the other samples as previously described. These samples were placed in a water bath (20°C.) to cool. Other data then taken consisted of: plate, steam chest, feed,

and reflux temperatures, feed weir reading, product and reflux weir readings, and manometer readings on the steam line to the steam chest and on lines to the vapor spaces of the top plate and the steam chest.

Shut Down.-After all data had been taken the operation of the still was ended by first closing the valves on all steam lines and turning off the current to the electric heater. The feed pump was then turned off and the liquid from the constant head tank allowed to drain through the column into the collecting tank (A). All cooling water was then turned off and all valves checked to be sure that they were closed tightly.

### Data and Results

The data and the results which have been obtained from the experimental tests may be found according to the following outline:

Table VI: "Feed Weir Calibration".

Figure 7: "Feed Weir Calibration".

Table VII: "Reflux and Product Weirs Calibration".

Figure 8: "Reflux and Product Weirs Calibration".

Tables VIII - XII: "Distillation of Isopropyl Alcohol and Water".

Figures 9 - 11: "Concentration Gradients, Distillation of Isopropyl Alcohol and Water".

Table XIII: "Murphree Plate Efficiencies, Distillation of Isopropyl Alcohol and Water".

Figures 12 - 14: "Murphree Plate Efficiencies, Distillation of Isopropyl Alcohol and Water".

Table XIV: "Coordinates of Actual Operating Lines, Distillation of Isopropyl Alcohol and Water".

Figures 15 - 16: "Theoretical and Actual Operating Lines, Distillation of Isopropyl Alcohol and Water".

Table XV - XVI: "Distillation of Toluene - Ethylene Dichloride".

Figure 17: "Concentration Gradients, Distillation of Toluene - Ethylene Dichloride".

Table XVII: "Murphree Plate Efficiencies, Distillation of Toluene - Ethylene Dichloride".

Figure 18: "Murphree Plate Efficiencies Distillation of Toluene - Ethylene Dichloride".

Table XVIII: "Coordinates of Actual Operating Lines, Distillation of Toluene - Ethylene Dichloride".

Figures 19 - 20: "Theoretical and Actual Operating Lines, Distillation of Toluene - Ethylene Dichloride".

TABLE VI

FEED WEIR CALIBRATION  
ISOPROPYL ALCOHOL AND WATER  
10 MOL PER CENT 70°F

Weir Reading Inches	Collection Interval Seconds	Grams Collected	Flow Rate Gms/Min
0	120	588	294
1	120	612	306
2	120	626	313
3	120	642	321
4	120	660	330
5	120	682	341
6	120	702	351

FIG. 7  
FEED WEIR CALIBRATION  
ISOPROPYL ALCOHOL AND WATER  
10 MOL % 70°F

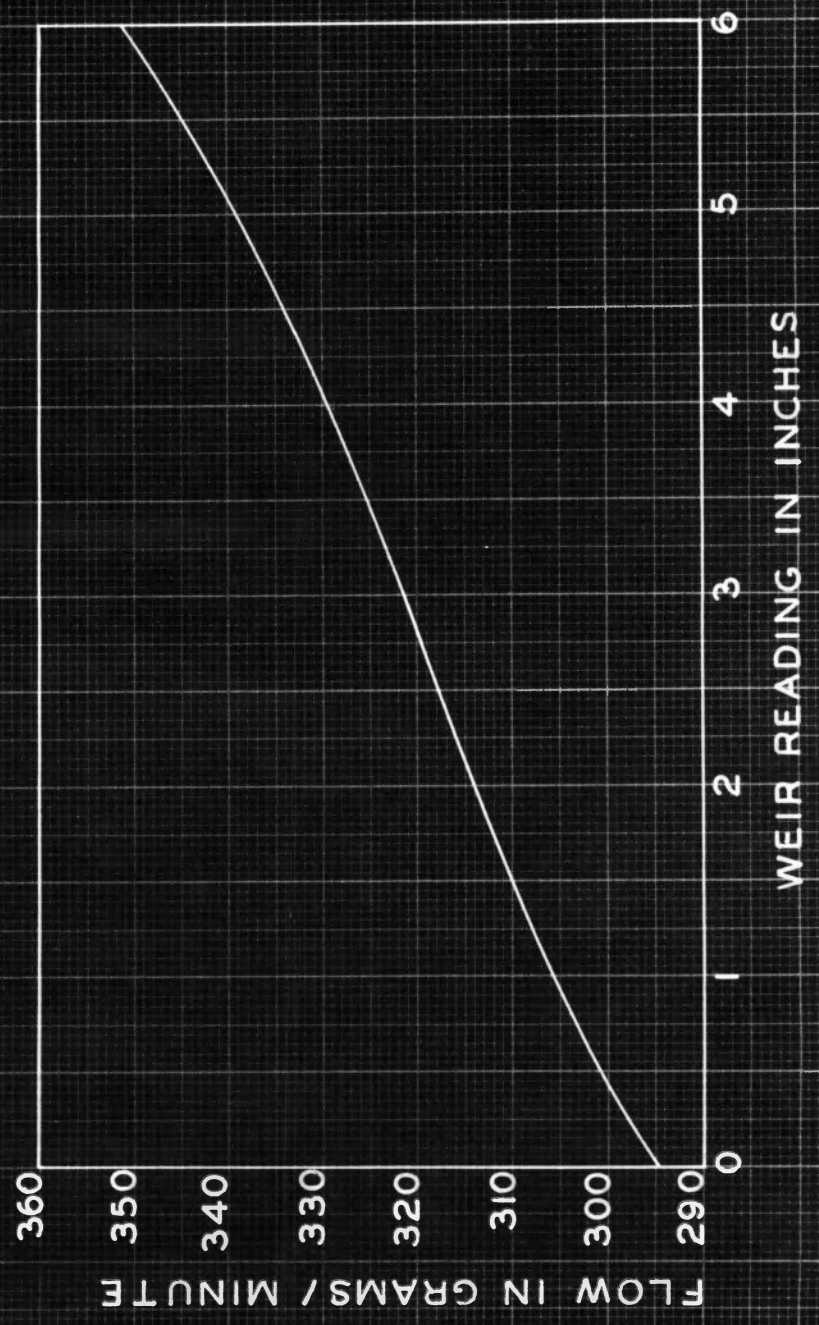


TABLE VII

REFLUC AND PRODUCT WEIRS CALIBRATION  
60 MOL PER CENT ISOPROPYL ALCOHOL 157°F

Weir Reading Inches	Product Weir Flow Rate Gms/Min	Reflux Weir Flow Rate Gms/Min
0	---	82
0.5	40	139
1	71	182
2	110	249
3	143	318
4	171	379
5	195	---

FIG. 8  
REFLUX AND PRODUCT WEIRS  
CALIBRATION  
60 MOL% ISOPROPYL ALCOHOL  
157° F

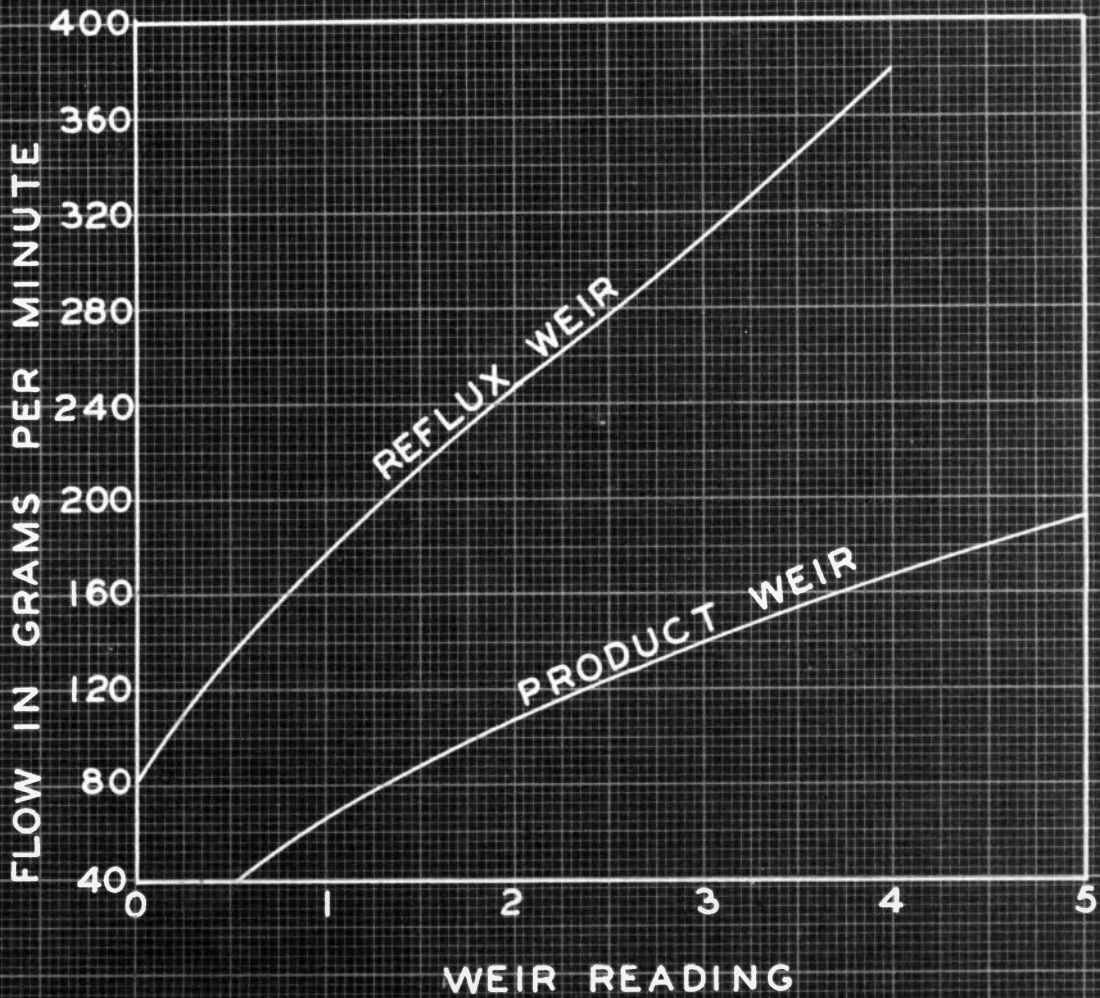




TABLE VIII

DISTILLATION OF ISOPROPYL ALCOHOL AND WATER

DISTILLATION RATE: 362 GMS./MIN.

FEED CONCENTRATION: 10.5 MOL PER CENT

Position of Sample	$N_d^{20}$	Wgt. Per Cent Isopropyl Alcohol	Mol Per Cent Isopropyl Alcohol	Maximum Deviation Mol Per Cent
Waste	1.3366	3.7	1.3	0.3
Plate 1	1.3539	24.1	8.9	0.1
Plate 2*	1.3627	38.4	16.0	0.6
Plate 3	1.3746	71.1	42.1	3.4
Plate 4	1.3756	75.5	48.3	4.5
Plate 5	1.3766	80.4	55.7	1.3
Plate 6	1.3768	81.7	57.6	1.1
Plate 7	1.3769	82.5	58.9	1.1
Plate 8	1.3770	83.3	59.9	1.0
Plate 9	1.37705	84.1	61.2	1.2
Plate 10	1.3771	84.5	61.7	1.6
Plate 11	1.37715	85.1	62.8	2.2
Plate 12	1.3772	85.7	63.7	2.4
Plate 13	--	-	-	-
Plate 14	--	-	-	-
Plate 15	--	-	-	-
Product	1.37725	86.2	64.6	2.4
Feed	1.3567	27.6		0.2

Feed Rate ..... 331 gms./min.  
 Product Rate ..... 91 gms./min.  
 Reflux Rate ..... 272 gms./min.  
 Reflux Ratio ..... 3.0  
 Steam Pressure ..... 9.5 in. Hg gage  
 Atm. Pressure ..... 715 mm. Hg  
 Temp. top plate ..... 169.5°F.  
 Temp. reflux ..... 169.5° F.  
 Temp. steam chest ..... 200° F.  
 Temp. feed ..... 179.5° F.

\* Feed Plate

Note: This table is an average of the data from Experiments 1, 2, and 3.

FIG. 9  
CONCENTRATION GRADIENTS  
EXPERIMENTS 1, 2, & 3

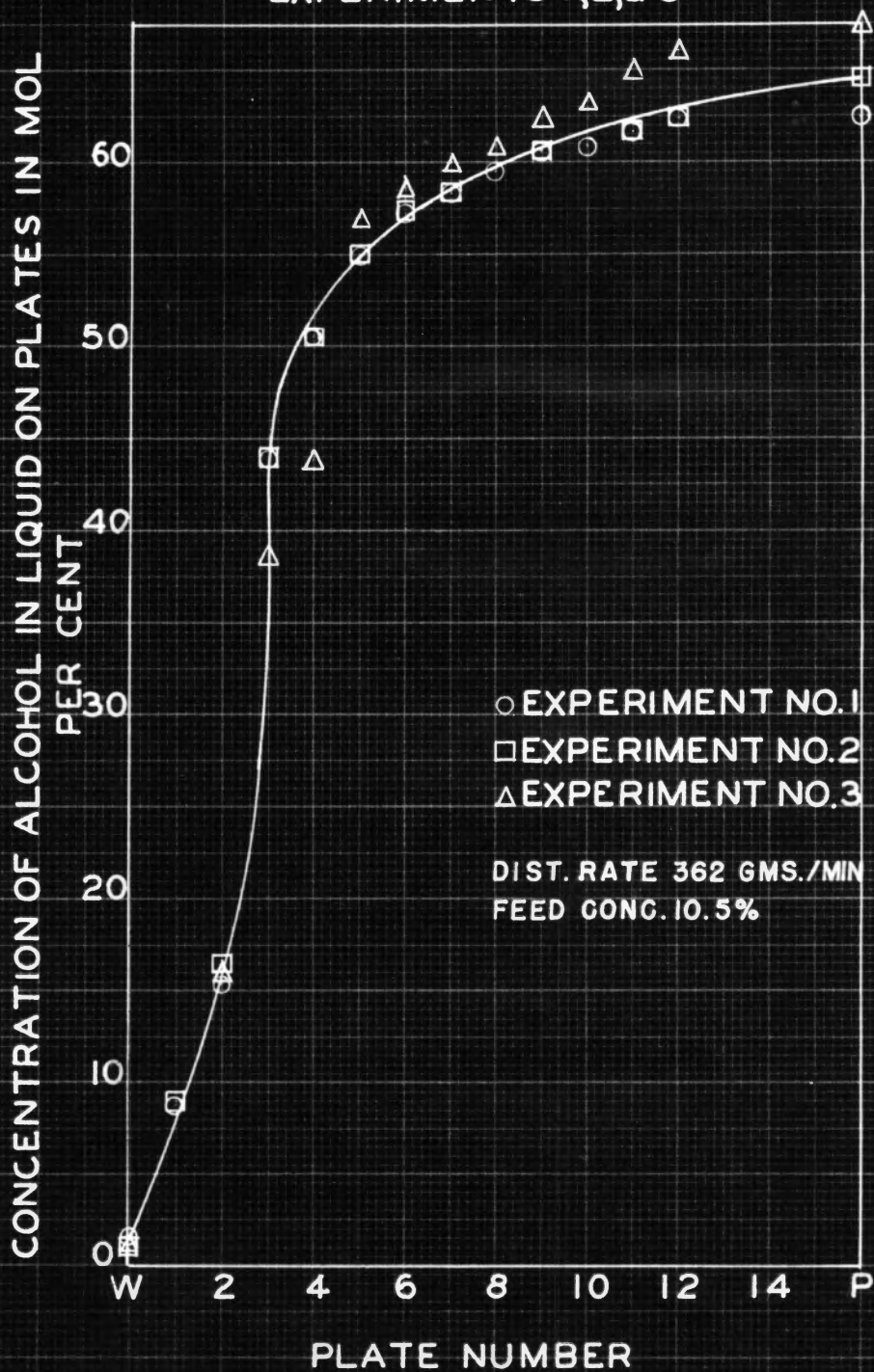


TABLE IX

DISTILLATION OF ISOPROPYL ALCOHOL AND WATER  
 DISTILLATION RATE: 372 GMS./MIN.  
 FEED CONCENTRATION: 9.6 MOL PER CENT

Position of Sample	$n_d^{20}$	Wgt. Per Cent Isopropyl Alcohol	Mol Per Cent Isopropyl Alcohol	Maximum Deviation Mol Per Cent
Waste	1.3338	0.9	0.2	0.1
Product 1	1.3432	11.7	3.9	0.8
Plate 2*	1.3604	34.0	13.6	0.8
Plate 3	1.3745	70.7	41.9	1.0
Plate 4	1.3760	77.5	51.1	0.2
Plate 5	1.3766	80.5	55.8	1.8
Plate 6	1.3768	82.1	58.1	1.2
Plate 7	1.3769	83.0	59.4	1.4
Plate 8	1.3770	83.5	60.2	1.4
Plate 9	1.3771	84.0	60.9	1.0
Plate 10	1.37715	84.4	61.4	1.5
Plate 11	--	-	-	-
Plate 12	--	-	-	-
Plate 13	--	-	-	-
Plate 14	--	-	-	-
Plate 15	--	-	-	-
Product	1.3772	85.1	62.8	2.2
Feed	1.3553	25.7	9.6	0.2

Feed Rate ..... 330 gms./min.  
 Product Rate ..... 93 gms./min.  
 Reflux Rate ..... 279 gms./min.  
 Reflux Ratio ..... 3.0  
 Steam Pressure ..... 11.7 in. Hg gage  
 Atm. Pressure ..... 710 mm. Hg  
 Temp. top plate ..... 169° F.  
 Temp. reflux ..... 169° F.  
 Temp. steam chest ..... 205° F.  
 Temp. feed ..... 180° F.

\* Feed Plate

Note: This table is an average of the data from Experiments 4, 5, and 6.

FIG. 10  
CONCENTRATION GRADIENTS  
EXPERIMENTS 4,5,& 6

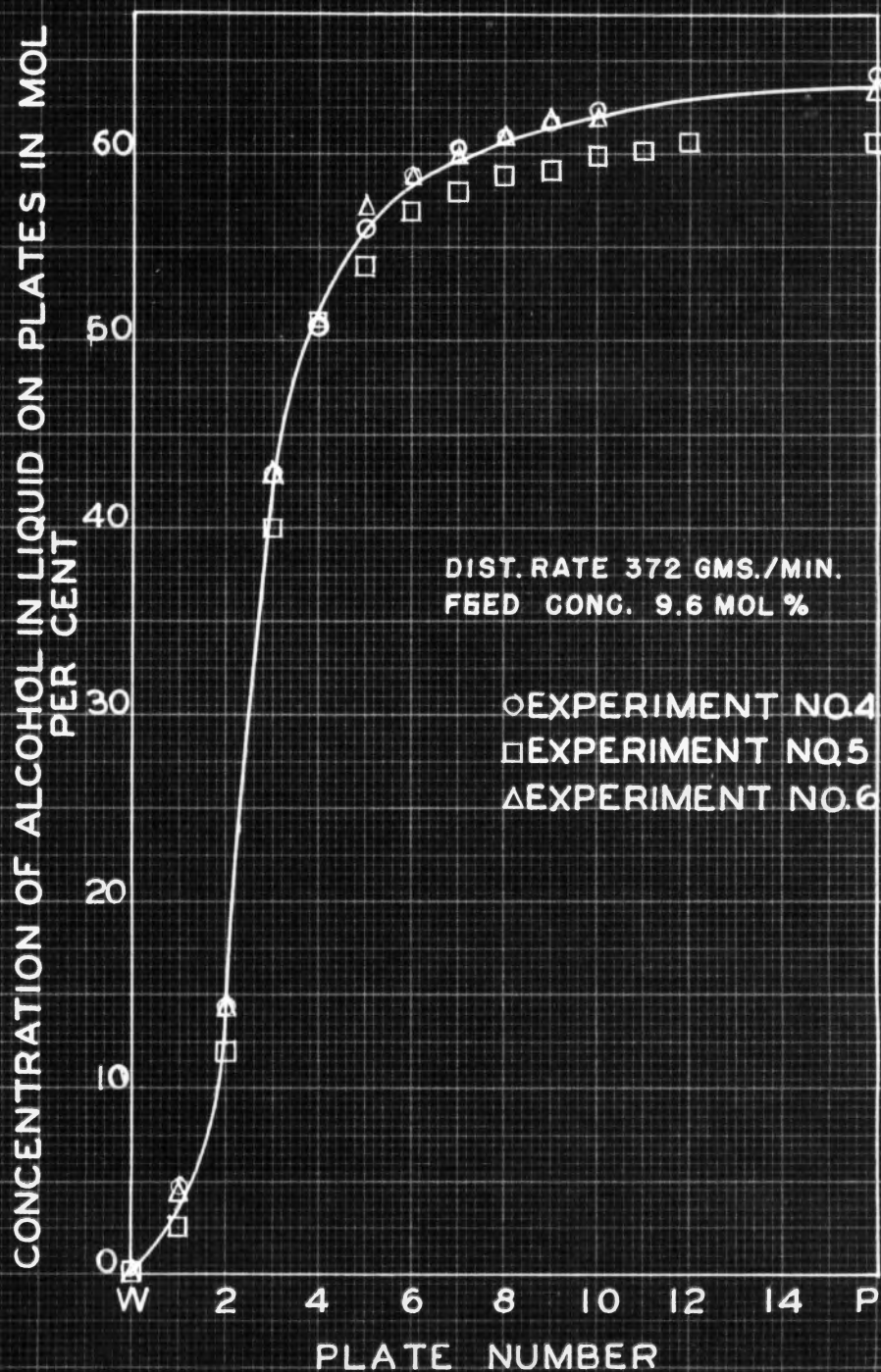


TABLE X

DISTILLATION OF ISOPROPYL ALCOHOL AND WATER

EXPERIMENT NO. 7

DISTILLATION RATE: 235 GMS./MIN.

FEED CONCENTRATION: 8.8 MOL PER CENT

Position of Sample	$n_d^{20}$	Wgt. Per Cent Isopropanol	Mol Per Cent Isopropanol
Waste	1.3404	8.2	2.7
Plate 1*	1.3591	31.7	12.4
Plate 2	1.3678	50.0	23.2
Plate 3	1.3759	76.7	48.0
Plate 4	1.3762	78.2	52.0
Plate 5	1.3764	79.7	54.7
Plate 6	1.3766	80.2	55.5
Plate 7	1.3767	81.5	57.2
Plate 8	1.3768	82.0	58.0
Plate 9	1.3770	83.5	60.2
Plate 10	1.3771	83.7	60.5
Plate 11	1.37715	84.7	62.1
Plate 12	1.3772	85.5	63.6
Plate 13	---	---	---
Plate 14	---	---	---
Plate 15	---	---	---
Product	1.37725	86.0	64.4
Feed	1.3540	24.0	8.8

Feed Rate ..... 331 gms/min  
 Product Rate ..... 59 gms/min  
 Reflux Rate ..... 176 gms/min  
 Reflux Ratio ..... 2.98  
 Steam Pressure ..... 2.5 in. Hg gage  
 Atm Pressure ..... 715 mm. Hg  
 Temp. top plate ..... 168.5°F  
 Temp. reflux ..... 168.5°F  
 Temp. steam chest ..... 190°F  
 Temp. feed ..... 180°F  
 Pressure Drop ..... 0.95 in. Hg

\* Feed Plate

TABLE XI

DISTILLATION OF ISOPROPYL ALCOHOL AND WATER  
 EXPERIMENT NO. 8  
 DISTILLATION RATE: 171 GMS./MIN.  
 FEED CONCENTRATION: 4.0 MO. PER CENT

Position of Sample	N <sub>d</sub> <sup>20</sup>	Wgt. Per Cent Isopropanol	Mol Per Cent Isopropanol
Waste	1.3339	1.0	0.3
Plate 1	1.3383	6.0	1.9
Plate 2*	1.3499	19.2	6.8
Plate 3	1.3711	58.7	29.7
Plate 4	1.3749	72.2	43.8
Plate 5	1.3762	78.2	52.2
Plate 6	1.3767	81.2	57.0
Plate 7	1.3769	82.5	58.8
Plate 8	1.3770	83.5	60.1
Plate 9	1.37705	84.0	60.9
Plate 10	1.3771	84.5	61.7
Plate 11	---	---	---
Plate 12	---	---	---
Plate 13	---	---	---
Plate 14	---	---	---
Plate 15	---	---	---
Product	1.3772	85.7	63.9
Feed	1.3432	11.7	4.0

Feed Rate ..... 331 gms/min  
 Product Rate ..... 43 gms/min  
 Reflux Rate ..... 128 gms/min  
 Reflux Ratio ..... 2.98  
 Steam Pressure ..... 9.5 in. Hg gage  
 Atm Pressure ..... 711 mm. Hg  
 Temp. top plate ..... 169°F  
 Temp. reflux ..... 169°F  
 Temp. steam chest ..... 205°F  
 Temp. feed ..... 185°F  
 Pressure Drop ..... 0.8 in. Hg

\* Feed Plate

TABLE XII

DISTILLATION OF ISOPROPYL ALCOHOL AND WATER  
 EXPERIMENT NO. 9  
 DISTILLATION RATE: 157 GMS./MIN.  
 FEED CONCENTRATION: 3.1 MOL PER CENT

Position of Sample	N <sub>d</sub> <sup>20</sup>	Wgt. Per Cent Isopropanol	Mol Per Cent Isopropanol
Waste	1.3330	0	0
Plate 1	1.3330	0	0
Plate 2	1.3339	1.0	0.3
Plate 3	1.3366	4.0	1.2
Plate 4*	1.3452	13.7	4.7
Plate 5	1.3719	61.4	31.6
Plate 6	1.3758	76.2	49.3
Plate 7	1.3764	80.0	55.0
Plate 8	1.3768	82.0	58.0
Plate 9	1.3770	83.5	60.1
Plate 10	1.3771	84.0	61.0
Plate 11	1.37715	85.0	62.5
Plate 12	1.3772	85.5	63.6
Plate 13	---	---	---
Plate 14	---	---	---
Plate 15	---	---	---
Product	1.3773	87.0	66.1
Feed	1.3414	9.5	3.1

Feed Rate ..... 330 gms/min  
 Product Rate ..... 40 gms/min  
 Reflux Rate ..... 117 gms/min  
 Reflux Ratio ..... 2.9  
 Steam Pressure ..... 13.0 in. Hg gage  
 Atm Pressure ..... 710 mm. Hg  
 Temp. top plate ..... 169°F  
 Temp. reflux ..... 169°F  
 Temp. steam chest ..... 209.5°F  
 Temp. feed ..... 186°F  
 Pressure Drop ..... 0.9 in. Hg

\* Feed Plate

FIG. 11  
CONCENTRATION GRADIENTS  
EXPERIMENTS 7,8,9

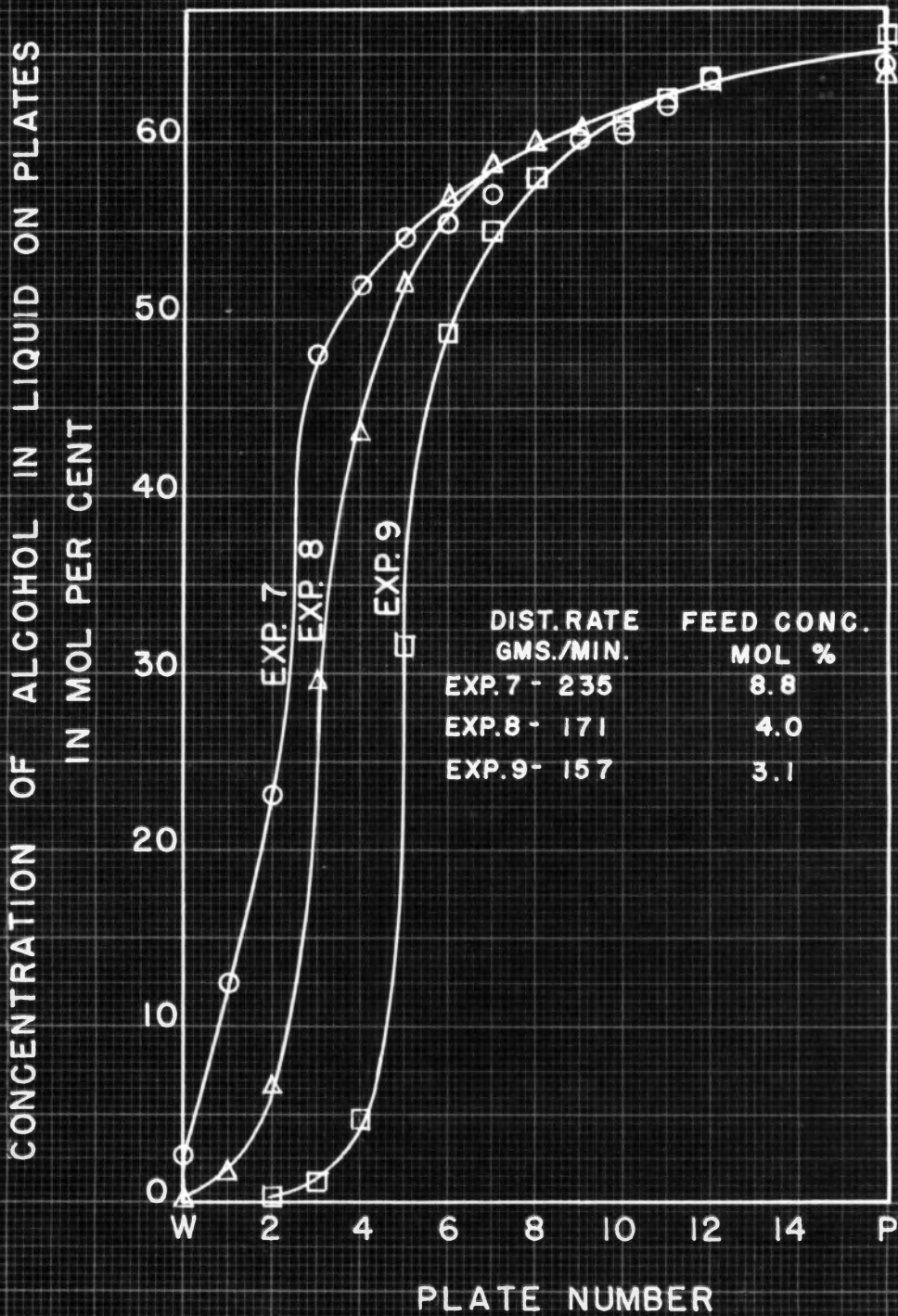




TABLE XIII

MURPHREE PLATE EFFICIENCIES  
DISTILLATION OF ISOPROPYL ALCOHOL - WATER

## MURPHREE PLATE EFFICIENCY (%)

Plate No.	Exp. No. 1	Exp. No. 2	Exp. No. 3	Exp. No. 4	Exp. No. 5	Exp. No. 6	Exp. No. 7	Exp. No. 8	Exp. No. 9
1	19.3	23.4	22.0	31.3	41.0	33.0	52.2*	32.3	---
2	83.6*	82.2*	70.5*	73.3*	81.8*	83.8*	88.5	86.8*	86.4
3	29.5	32.7	16.0	38.0	47.4	45.0	23.5	42.1	63.4
4	35.2	41.7	47.0	50.5	17.1	68.2	23.0	50.0	96.6*
5	24.8	22.1	30.4	39.6	25.4	18.7	7.8	54.0	83.5
6	12.5	21.2	27.7	25.0	11.5	20.0	20.2	25.0	60.0
7	16.0	15.6	22.0	10.9	9.0	17.6	10.5	21.0	46.9
8	18.2	20.7	50.0	15.4	3.4	17.0	36.0	14.3	42.0
9	3.2	5.6	34.6	14.9	9.5	0	5.2	15.4	21.4
10	13.3	16.3	100.0	0	4.2	0	32.7	0	41.7
11	12.5	17.8	100.0	0	4.5	0	39.5	0	36.7
Average	24.4	27.2	47.3	27.2	23.2	27.6	30.8	30.9	52.3

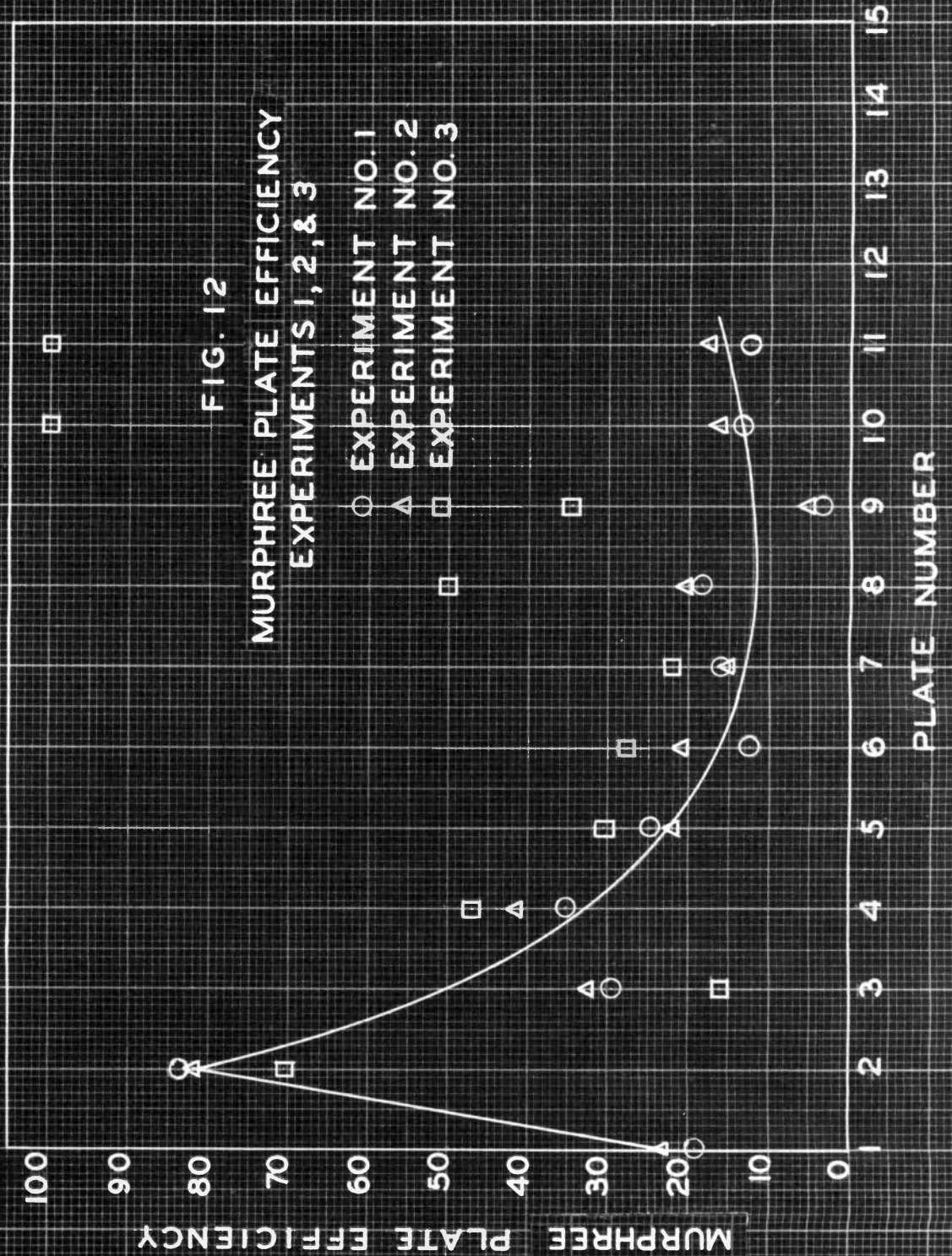
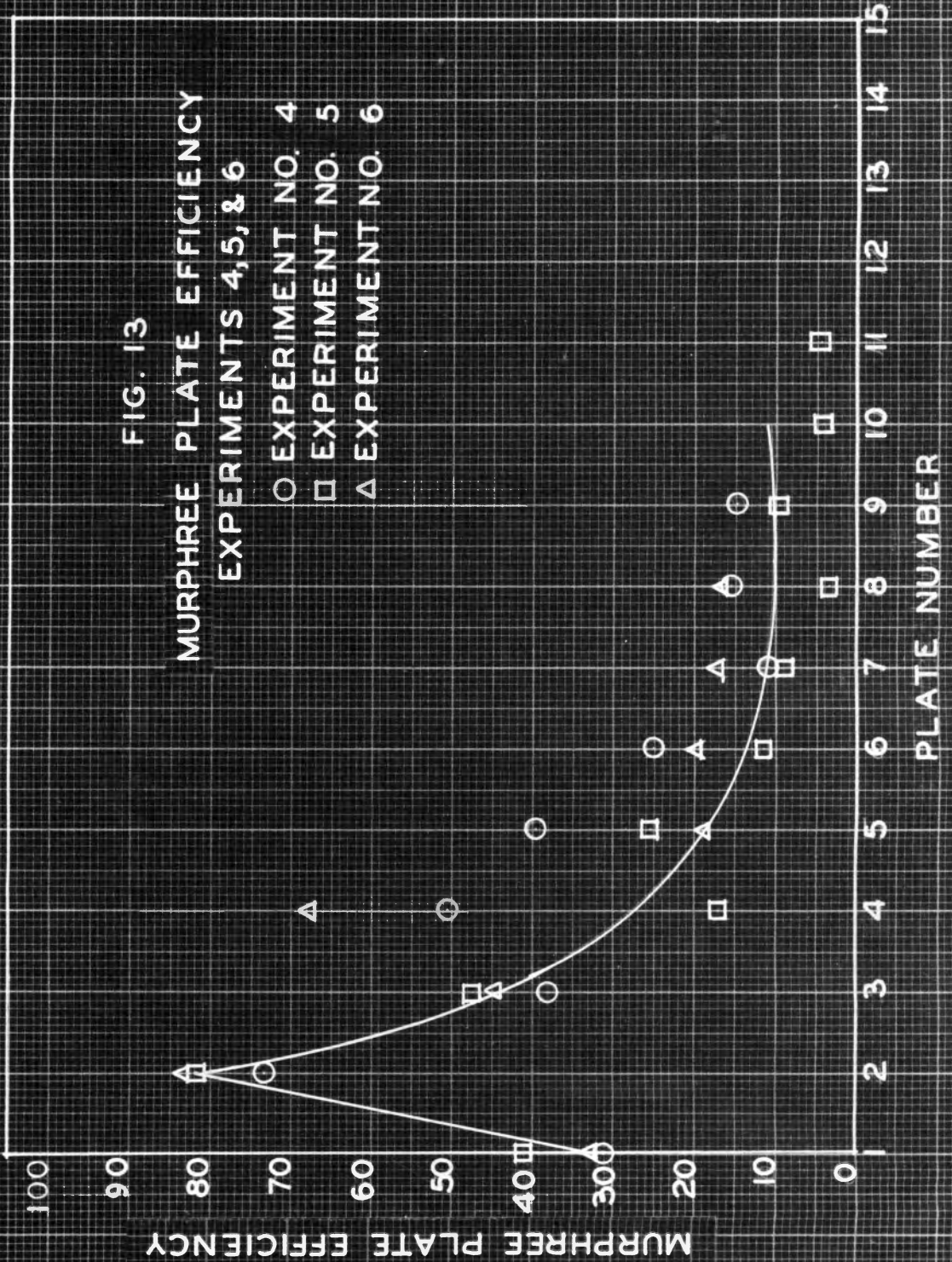


FIG. 12  
MURPHREE PLATE EFFICIENCY  
EXPERIMENTS 1, 2, & 3

FIG. 13  
MURPHREE PLATE EFFICIENCY  
EXPERIMENTS 4, 5, & 6

- EXPERIMENT NO. 4
- EXPERIMENT NO. 5
- △ EXPERIMENT NO. 6



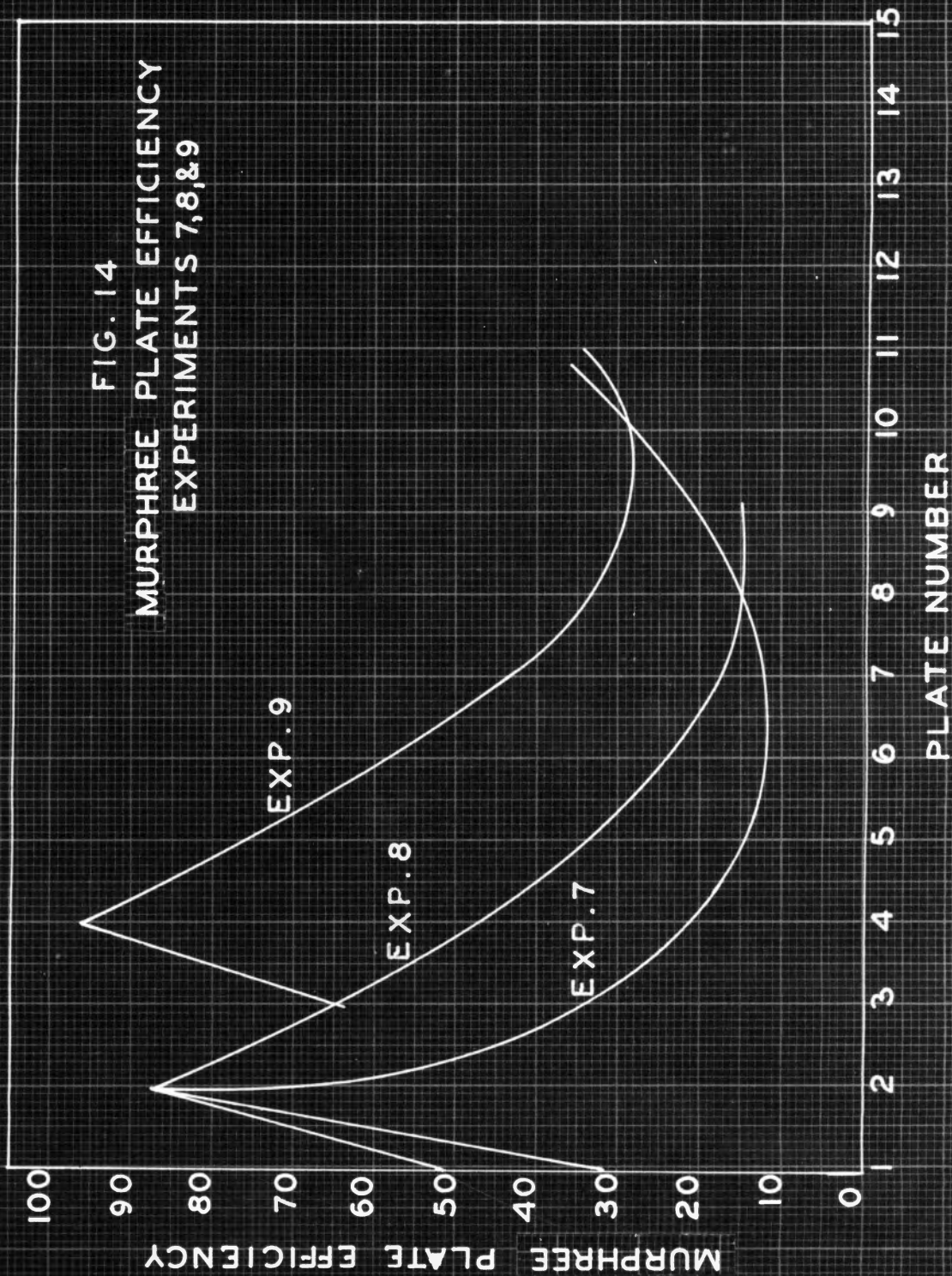


TABLE XIV

COORDINATES OF ACTUAL OPERATING LINES  
DISTILLATION OF ISOPROPYL ALCOHOL - WATER

Exp. No. 1		Exp. No. 2		Exp. No. 3		Exp. No. 4		Exp. No. 5		Exp. No. 6		Exp. No. 7		Exp. No. 8		Exp. No. 9	
X <sup>1</sup>	Y <sup>2</sup>	X	Y	X	Y	X	Y	X	Y	X	Y	X	Y	X	Y	X	Y
8.8	24.7	9.0	16.2	8.8	21.5	4.7	7.0	2.6	14.2	4.4	7.0	12.4	34.5	1.9	9.0	1.2	9.0
15.4	48.0	16.6	48.1	16.0	48.0	14.4	42.5	12.0	33.5	14.4	41.2	23.2	49.8	6.8	28.9	4.7	22.0
43.9	50.7	43.8	51.0	38.7	51.1	42.9	50.2	40.0	49.5	42.8	50.3	48.0	52.7	29.7	46.4	31.6	43.0
50.6	57.2	50.5	57.2	43.8	55.9	50.8	56.9	51.2	56.0	51.3	56.8	52.0	58.7	43.8	54.0	49.3	54.4
55.0	59.5	55.0	59.5	57.0	57.5	56.0	59.9	54.0	59.8	57.3	59.8	54.7	60.4	52.2	57.4	55.0	59.3
57.3	61.2	56.9	61.4	58.7	62.6	58.8	61.8	56.9	61.0	58.7	62.5	55.5	61.5	57.0	60.5	58.0	61.7
58.3	62.5	58.4	62.3	60.0	63.4	60.3	63.2	58.0	62.2	60.0	63.2	57.2	62.0	58.8	62.6	60.1	63.1
59.5	63.0	59.4	63.0	60.9	64.0	60.9	64.0	58.8	62.8	61.0	63.7	58.0	62.7	60.1	63.5	61.0	64.0
60.7	63.5	60.6	63.5	62.4	64.5	61.7	64.2	59.1	63.2	61.9	64.2	60.2	63.1	60.9	64.0	62.5	64.5
60.9	64.2	60.9	64.0	63.3	65.2	62.4	64.7	59.9	63.4			60.5	64.0	61.6	64.5	63.6	65.2
61.7	64.3	61.7	64.3	65.0	65.7			60.2	63.7			62.1	64.3				
62.4	64.7	62.5	64.6	66.1	66.5			60.6	63.9			63.6	65.0				

1 - Mol Per Cent Isopropyl Alcohol in Liquid  
2 - Mol Per Cent Isopropyl Alcohol in Vapor

FIG. 15  
THEORETICAL AND ACTUAL OPERATING LINES  
EXPERIMENTS 1, 2, & 3

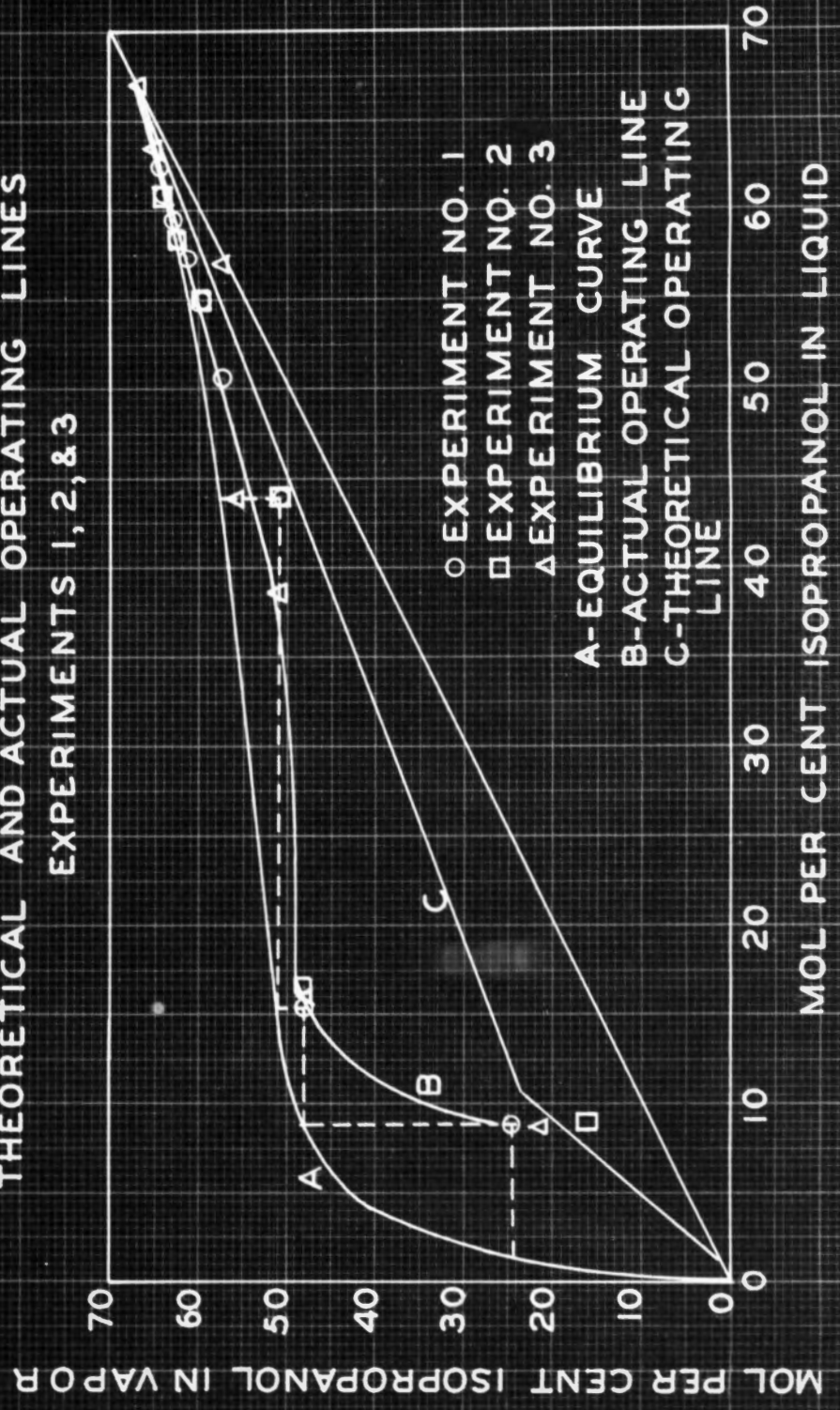


FIG. 16  
THEORETICAL AND ACTUAL OPERATING LINES  
EXPERIMENTS 4, 5, & 6

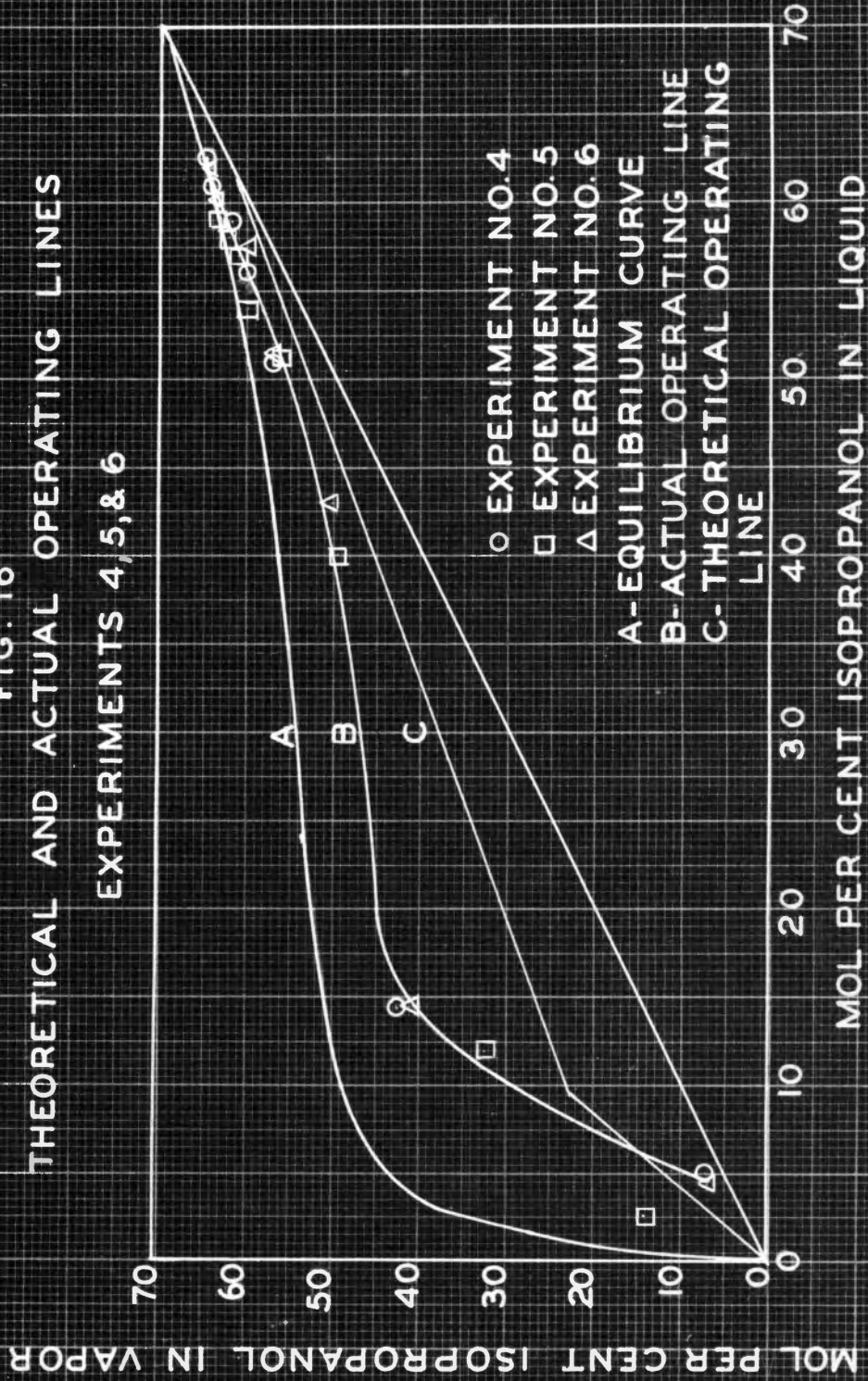


TABLE XV

DISTILLATION OF TOLUENE - ETHYLENE DICHLORIDE  
 EXPERIMENT NO. 10  
 DISTILLATION RATE: 203 GMS./MIN.  
 FEED CONCENTRATION: 41.4 MOL PER CENT

Position of Sample	$n_D^{25}$	Mol Per Cent Ethylene Dichloride
Waste	1.4863	16.4
Plate 1	1.4839	21.5
Plate 2	1.4778	34.5
Plate 3*	1.4750	40.5
Plate 4	1.4697	50.8
Plate 5	1.4650	59.6
Plate 6	1.4595	70.0
Plate 7	1.4550	78.4
Plate 8	1.4511	85.5
Plate 9	1.4490	89.1
Plate 10	1.4463	93.7
Plate 11	1.4452	95.3
Plate 12	1.4441	97.2
Plate 13	1.4435	98.0
Plate 14	1.4430	98.8
Plate 15	1.4427	99.3
Product	1.4425	99.7
Feed	1.4745	41.4

Feed Rate ..... 138 gms/min  
 Product Rate ..... 42 gms/min  
 Reflux Rate ..... 161 gms/min  
 Reflux Ratio ..... 3.9  
 Steam Pressure ..... 21.6 in. Hg gage  
 Atm Pressure ..... 699 mm. Hg  
 Temp. top plate ..... 172°F  
 Temp. reflux ..... 172°F  
 Temp. steam chest ..... 219°F  
 Temp. feed ..... 204°F  
 Pressure Drop ..... 0.9 in. Hg

\* Feed Plate



TABLE XVI

DISTILLATION OF TOLUENE - ETHYLENE DICHLORIDE  
 EXPERIMENT NO. 11  
 DISTILLATION RATE: 196 GMS./MIN.  
 FEED CONCENTRATION: 39.7 MOL PER CENT

Position of Sample	$N_d^{25}$	Mol Per Cent Ethylene Dichloride
Waste	1.4892	10.1
Plate 1	1.4869	15.0
Plate 2	1.4820	25.3
Plate 3*	1.4795	31.3
Plate 4	1.4742	42.2
Plate 5	1.4729	44.7
Plate 6	1.4687	52.7
Plate 7	1.4629	63.5
Plate 8	1.4587	71.5
Plate 9	1.4540	80.2
Plate 10	1.4508	85.8
Plate 11	1.4482	90.5
Plate 12	1.4462	93.8
Plate 13	1.4450	95.6
Plate 14	1.4440	97.2
Plate 15	1.4431	98.7
Product	1.4427	99.3
Feed	1.4754	39.7

Feed Rate ..... 138 gms/min  
 Product Rate ..... 39 gms/min  
 Reflux Rate ..... 157 gms/min  
 Reflux Ratio ..... 4.0  
 Steam Pressure ..... 22.5 in. Hg gage  
 Atm Pressure ..... 699 mm. Hg  
 Temp. top plate ..... 172°F  
 Temp. reflux ..... 172°F  
 Temp. steam chest .... 221°F  
 Temp. feed ..... 206°F  
 Pressure Drop ..... 0.9 in. Hg

\* Feed Plate

FIG. 17  
CONCENTRATION GRADIENTS  
DISTILLATION OF TOLUENE-ETHYLENE DICHLORIDE

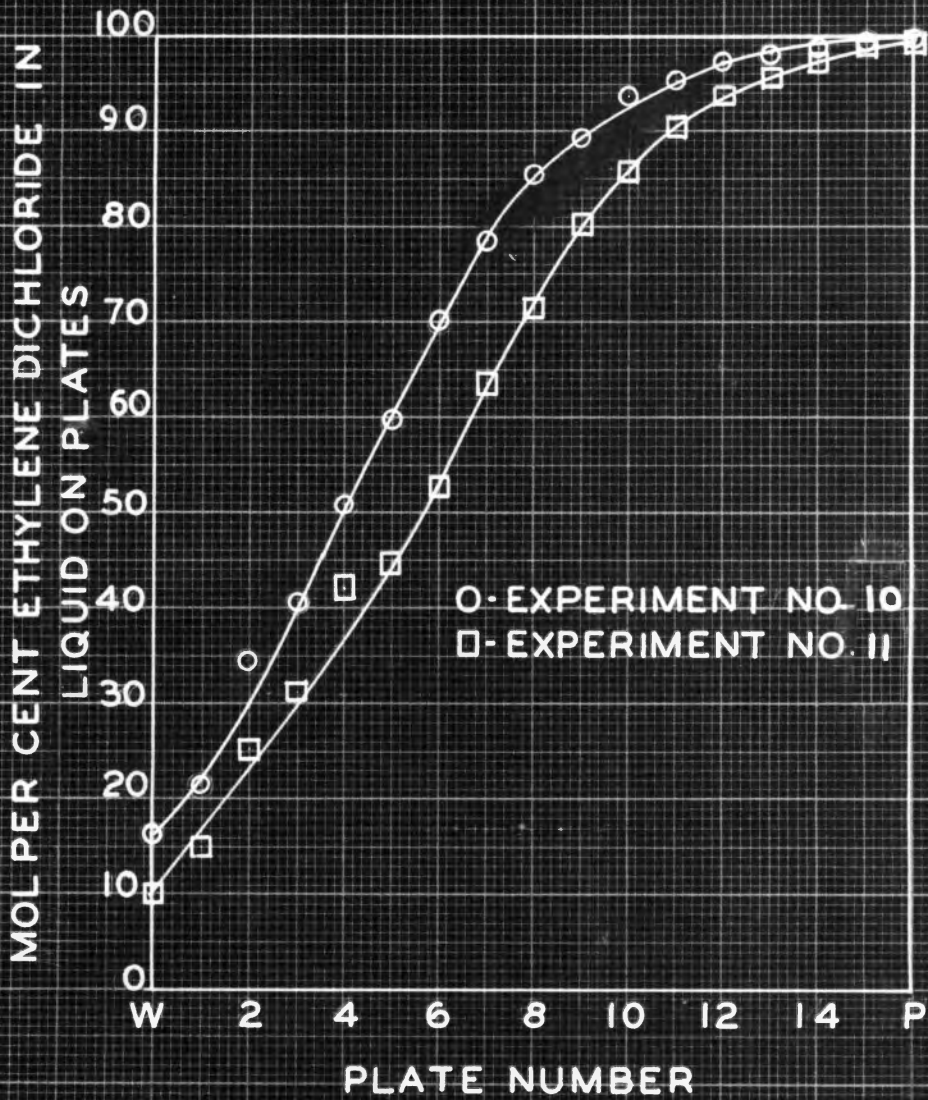


TABLE XVII

MURPHREE PLATE EFFICIENCIES  
DISTILLATION OF TOLUENE - ETHYLENE DICHLORIDE

MURPHREE PLATE EFFICIENCY			
Plate No.	Exp. No. 1	Exp. No. 2	Average
1	93.5	91.0	92.2
2	121.0	116.0	118.5
3*	98.1	73.0	85.5
4	75.8	84.6	80.2
5	83.2	74.1	78.6
6	74.3	89.3	81.8
7	79.7	66.1	72.9
8	50.7	80.5	65.6
9	93.8	63.8	78.2
10	44.5	77.1	60.8
11	79.2	73.3	76.2
12	44.5	48.7	46.6
13	61.5	64.0	62.7
14	71.5	100.0	85.7
15	80.0	85.8	82.9
Average	76.7	79.1	

\* Feed Plate

FIG. 18  
MURPHREE PLATE EFFICIENCIES  
DISTILLATION OF TOLUENE-ETHYLENE DICHLORIDE

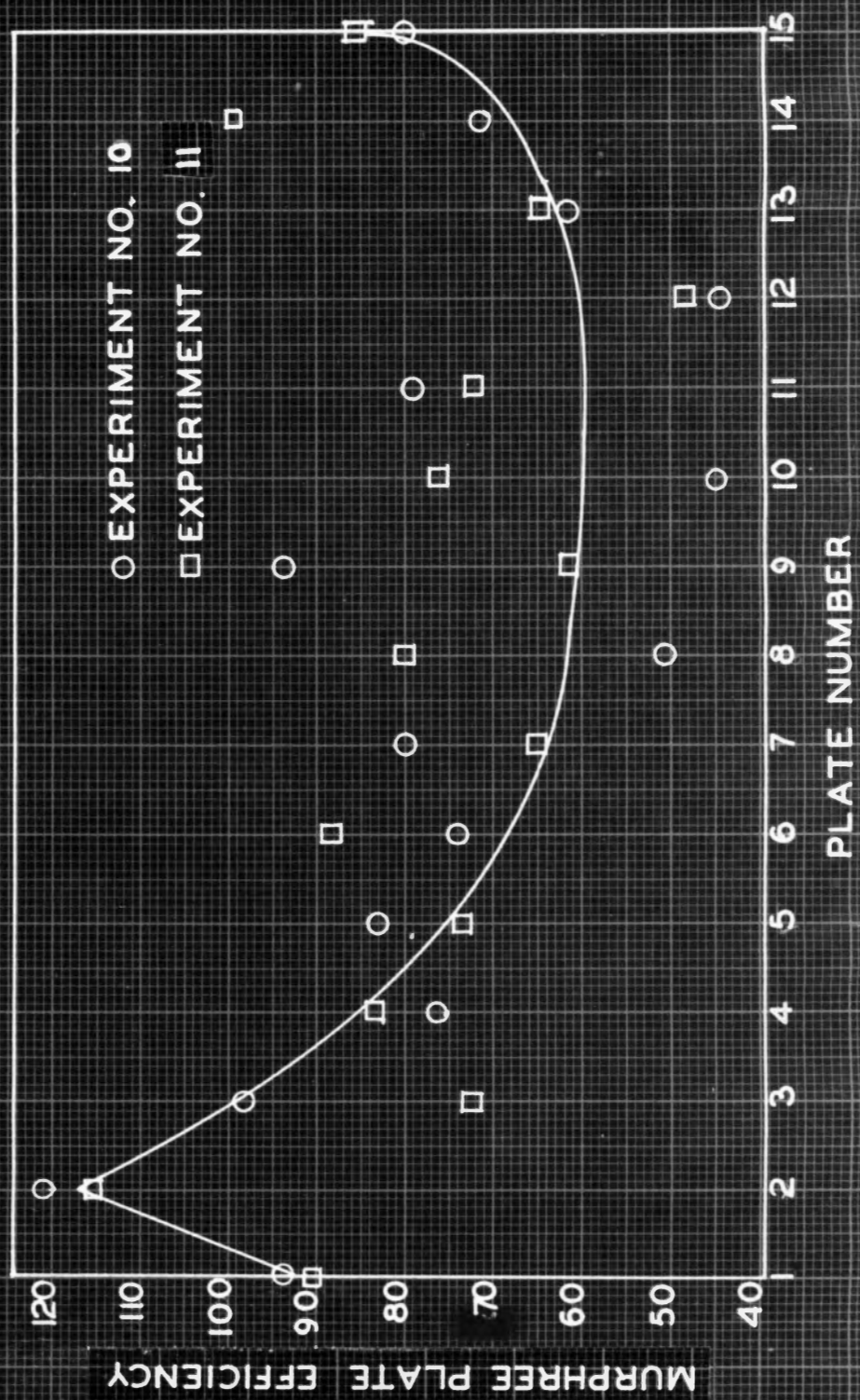


TABLE XVIII

COORDINATES OF ACTUAL OPERATING LINES  
DISTILLATION OF TOLUENE - ETHYLENE DICHLORIDE

Experiment No. 1		Experiment No. 2	
$x^1$	$x^2$	X	Y
21.5	29.8	15.0	19.8
34.5	37.0	25.3	27.5
40.5	54.3	31.3	42.5
50.8	60.5	42.2	50.2
59.6	70.1	44.7	62.2
70.0	77.2	52.7	64.5
78.4	84.5	63.5	71.7
85.5	89.2	71.5	80.2
89.1	93.0	80.2	85.3
93.7	95.0	85.8	90.2
95.3	97.2	90.5	93.2
97.2	97.9	93.8	95.6
98.0	98.8	95.6	97.1
98.8	99.2	97.2	98.0
99.3	99.5	98.7	98.9
99.7	99.7	99.3	99.4

1 - Mol Per Cent Ethylene Dichloride in  
Liquid

2 - Mol Per Cent Ethylene Dichloride in  
Vapor

FIG. 19  
THEORETICAL AND ACTUAL OPERATING LINES  
DISTILLATION OF TOLUENE-ETHYLENE DICHLORIDE  
EXPERIMENT NO. 10

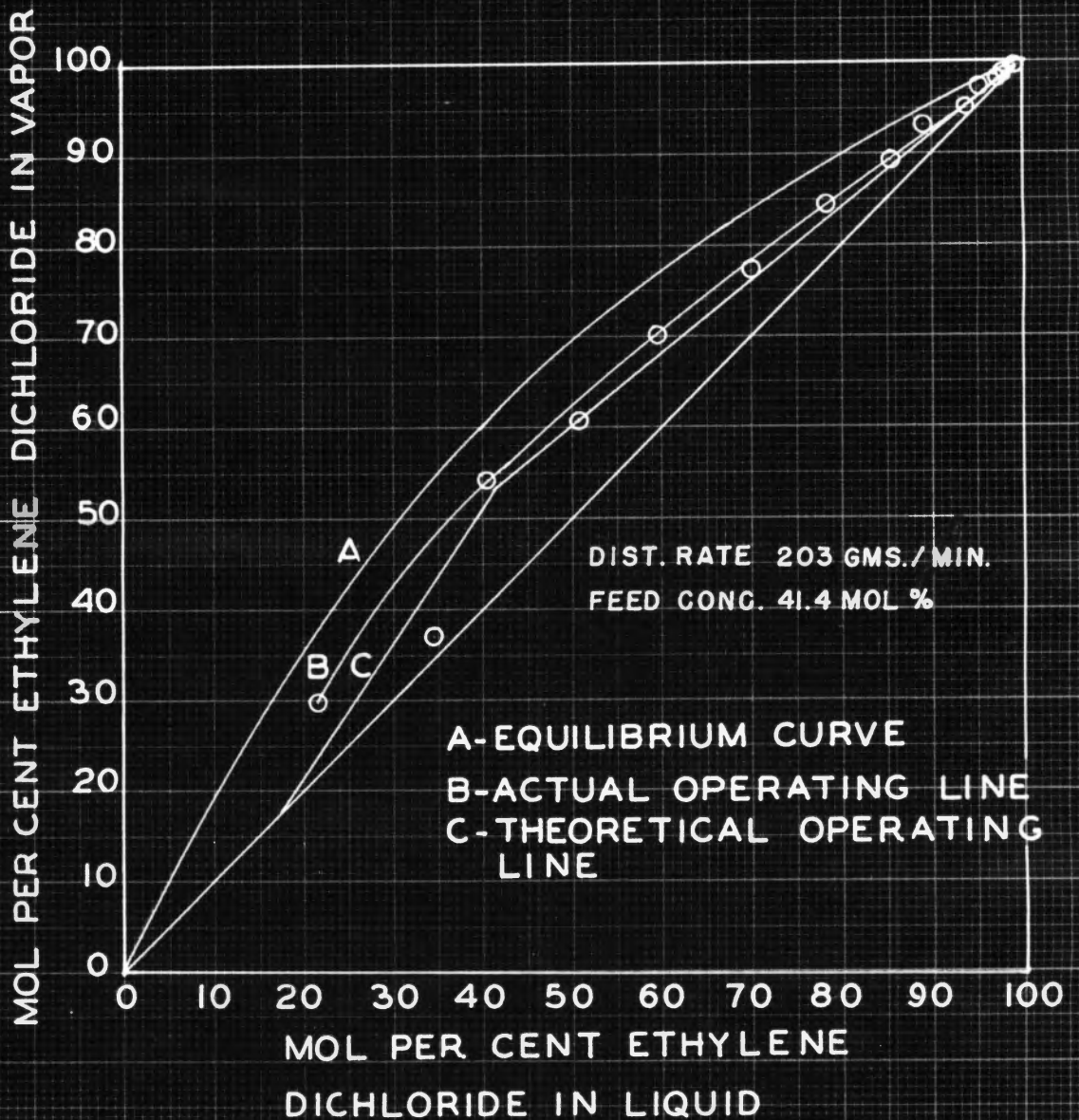
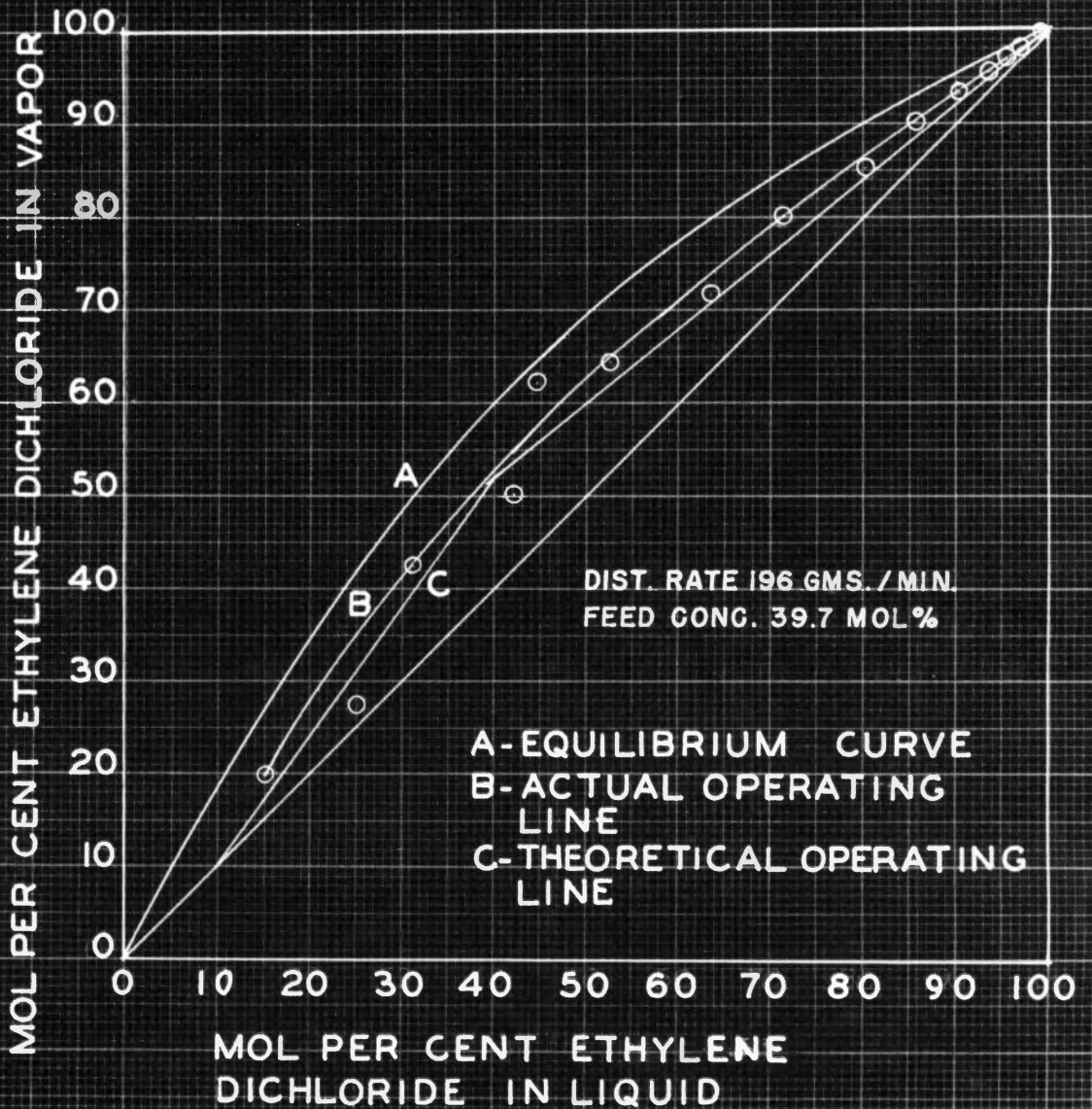


FIG. 20  
THEORETICAL AND ACTUAL OPERATING LINES  
DISTILLATION OF TOLUENE-ETHYLENE DICHLORIDE  
EXPERIMENT NO. II



#### IV - DISCUSSION OF RESULTS

General: Any results in studies of distillation in plate columns are apt to be inaccurate due to the difficulties of controlling the external effects on the column such as rate of feed, concentration of the feed, location of the feed plate, rate of distillation, reflux ratio, and temperature of the reflux. Added to the difficulties of investigation caused by these factors are the internal effects within the column. The internal effects causing inaccuracy may be: lack of equilibrium due to any one of the above factors, poor mixing on the plates, hydraulic gradients on the plates, unequal molal overflow, and other diffusional factors such as surface tension, viscosity, and bubble size.

The external effects have been controlled as accurately as possible while operating this column assembly. Tests of as long as ten hours duration have been made in an attempt to obtain true equilibrium within the column and the general similarity of results show that some success has resulted. Realizing that any one set of results may be somewhat inaccurate, attempt has been made to make several tests with the same operating conditions and average the results obtained.



Sampling of Plate Concentrations: Liquid samples from each plate of the column, the waste, the product and the feed were taken after the column had operated at steady conditions for at least two hours. The small volume taken from each plate (4 ml. or less) seemed to have little effect on the operation of the column as indicated by no apparent change in rate of distillation, although the removal of even this small volume probably had some effect on equilibrium conditions.

In order to determine the effectiveness of mixing on the plates, samples were taken from three points on Plate 2 during each test. The location of these three points were as shown in Figure 6 and were located on the periphery of the plate near the downcomer from Plate 3, near the overflow weir to Plate 1, and centered on the periphery parallel to the cross flow of liquid. The concentrations of the samples from any one test varied from within 0 to 4 mol per cent of each other. The samples taken nearest the downcomer and overflow weir were, in general, approximately 1 mol per cent lower in concentration than the sample taken at the center of the plate. The samples taken nearest the overflow weir were usually slightly lower (0 - 0.8 mol per cent) than the concentration of the samples taken nearest the downcomer. Apparently, mixing was not altogether complete on the plates since the sample taken at the center of the plate should never be higher than the sample taken at the downcomer. This apparent incomplete mixing would probably contribute to errors

in determining equilibrium concentration on the various plates. The plots of concentration gradients against plate numbers (Figures 9, 10, 11, and 17) show that all points do not follow a smooth curve and further bears out the possibility of incomplete mixing as affecting plate concentrations.

Analysis of Plate Samples.—The refractive index method of analysis is highly recommended for the analysis of plate samples because it permits the analysis to be made on such a small volume of sample. A limitation was found in the higher concentrations of isopropyl alcohol where the degree of variance of the index of refraction with concentration was very small. (See Figure 3). The index of refraction at 20°C. varied from 1.3770 at 60 mol per cent to 1.3776 at 100 mol per cent isopropyl alcohol. An attempt was made to overcome this limitation by preparing a curve relating the refractive index at 20°C. with the concentration after the samples of above 50 mol per cent isopropyl alcohol had been diluted with one-half their volume of distilled water. This increased the accuracy of analysis to some extent but slight errors in diluting the samples with water and maintaining the correct temperature gave an accuracy only within the limits of  $\pm 1$  mol per cent. This however, was not a sufficient accuracy above 55 mol per cent alcohol where changes of less than 1 mol per cent in plate concentrations apparently took place.

The refractive index - composition data for the toluene - ethylene dichloride mixtures gave a much more regular curve and con-

centrations could be easily read from 0 to 100 mol per cent ethylene dichloride. (See Figure 4).

Column Operation: The column was found to be quite sensitive to any change in operating conditions and since a change in one variable often affected the other operating conditions, constant operation was difficult to maintain. A change in the rate of distillation might affect the feed plate location, reflux ratio, and, of course, the plate concentrations.

Variance in steam pressure, and consequently, the rate of distillation, caused the most trouble, but the irregularities in steam pressure were overcome to some extent by the use of a steam pressure regulator which held the steam pressure constant to within one-eighth of a pound of steam, except when the main line pressure became very erratic. The use of an electric heater in the steam chest, controlled automatically to  $\pm 2^{\circ}\text{F.}$ , aided in maintaining an even temperature in the steam chest. By close supervision during a test, the steam chest temperature was held within  $\pm 1^{\circ}\text{F.}$

Constant effort was necessary to stop any leakage from the pump which returned the waste and product liquid to the constant head tank. Since it was necessary to use a glandless, self-lubricating pump in order that the binary mixture would not become contaminated with grease, and since the only pump of this type available was practically worn out, considerable difficulty was encountered. The pump was finally packed with lead packing and this was tightened several

times during each test. Serious changes in feed concentration were avoided by holding a large volume of feed (15 gal.) in the constant head tank.

The feed heater, as first used, consisted of a brass pipe heated by an interior pipe carrying low pressure (15 lbs.) steam concurrent with the flow of the feed, which was introduced at the top of the heat exchanger. It was found that the volatile constituent in the feed tended to flash back into the feed weir. By introducing the feed into the exchanger 6 inches from the bottom, closing the entrance at the top of the heat exchanger, and using high pressure steam (40 - 75 lbs.) as the heating medium, to give the added heat needed, the feed was forced into the column by its own vapor pressure as well as by gravity. (See Fig. 5). Since the feed had to be under a slight pressure in order to force it into the column it was probably heated slightly beyond its boiling point. However, as can be realized by an inspection of the equilibrium diagrams at different feed conditions, the temperature of the feed had little effect on column operation or plate efficiency.

The proper temperature of the feed and the proper feed plate had to be determined after the column had been in operation for two or three hours time. During this time the concentration of the feed slowly decreased, usually 2 to 3 mol per cent, and the liquid concentration on the plates became stable. Plates and feed concentration analyses then showed which was the proper feed plate

and readjustments could be made. This resulted in making the proper operation of the column, time-consuming and difficult. However, once a test had been made under one set of conditions, the results could be duplicated in another test in about half the original time.

Experiments 1, 2, and 3. Distillation of Isopropyl Alcohol - Water: These three experiments (Table VIII) were run under as nearly the same operating conditions as possible. Experiments 1 and 2 gave products of 62.4 and 64.4 mol per cent isopropanol at 9.5 and 10.1 inches of mercury steam pressure; while in Experiment 3 a product of 67 mol per cent at 9 inches of mercury steam pressure was obtained. The rates of distillation were respectively 366, 368, and 353 gms./min. The difference in product purity is not considered significant and is probably due to inaccuracies in analysis and to small variances in column operation. Since in these three tests, under the same operating conditions, comparable results were obtained, the data were used in calculating Murphree plate efficiencies, and determining the actual operating line for the distillation.

Experiments 4, 5, and 6. Distillation of Isopropyl Alcohol - Water: In this series of experiments, (Table IX), it was attempted to maintain the steam chest temperature at 205°F. as compared with a temperature of 200°F. in Experiments 1, 2, and 3. By maintaining all other operating conditions constant the effect of the rate of distillation could be studied. A rate of distillation of 372 gms./min. was obtained, or approximately 10 gms./min. higher than

in Experiments 1, 2, and 3. As a result of increasing the steam chest temperature from 200°F. to 205°F., the concentration of the alcohol in the waste was lowered from approximately 1 mol per cent to 0.2 mol per cent isopropyl alcohol. In comparison with results in Experiments 1, 2, and 3, no significant changes in product purity could be noted.

Experiments 7, 8, and 9. Distillation of Isopropyl Alcohol - Water: In these experiments, (Tables XI, XII, and XIII), it was intended to use a lower concentration of feed and maintain the rate of distillation within the limits used in Experiments 1 through 6. It was found that the rate of distillation could not be increased materially above 235, 171, and 157 gms./min. for feed concentrations of respectively 8.8, 4.0, and 3.1 mol per cent isopropyl alcohol. The purity of the final product (approximately 65 mol per cent) and the temperature of the top plate (169°F.) could not be changed by increasing the steam chest temperature from 190°F. to 209.5°F. It was found that the composition of the waste could be changed as before and that the location of the feed plate could be moved up or down by increasing or decreasing the steam pressure to the steam chest. In Experiment 9, separation was not being accomplished below Plate 2, and yet, the product purity was just as high as in all other distillations, (approximately 65 mol per cent). Perhaps this apparent inconsistency is due to the fact that analysis of samples in the upper ranges of concentration is at fault. In all nine ex-

periments the majority of the distillation seemed to occur on the first seven plates and at the seventh plate the plate concentrations were within three mol per cent of each other.

Experiments 10 and 11. Distillation of Toluene - Ethylene Dichloride: These experiments (Tables XV and XVI) were run at practically identical rates of distillation (203 and 196 gms./min. respectively). However, the feed concentration changed from 41.4 mol per cent in Experiment 10 to 39.7 mol per cent in Experiment 11. In Experiment 10 a product concentration of 99.7 mol per cent was obtained, as compared to 99.3 mol per cent for Experiment 11 and the waste compositions were 16.4 and 10.1 mol per cent for Experiments 10 and 11 respectively. The lower concentration of feed and slightly higher steam chest temperature both apparently contributed to the differences in waste and product compositions. The deduction is further complicated by the fact that in Experiment 11 the feed composition of 39.7 mol per cent was more closely approximated on plate 4 (42.2 mol per cent) rather than on Plate 3 (31.3 mol per cent) which was the feed plate used.

Concentration Gradients: Both binary mixtures gave typical S - shaped concentration gradient curves. In the distillation of isopropyl alcohol and water, Figures 9 - 11, the gradient curves were the steepest between 10 and 50 mol per cent alcohol which corresponds to the section of the equilibrium curve bounding the greatest area between itself and the  $45^\circ$  diagonal. The concentration gradient curves

in the distillation of toluene and ethylene dichloride, shown in Figure 17, had less slope than those for the distillation of isopropyl alcohol and water. Since the equilibrium diagrams of the two systems differ greatly, some difference in concentration gradient would be expected. The system toluene - ethylene dichloride gradient curves were the steepest between 30 and 80 mol per cent ethylene dichloride, corresponding again to the section of the equilibrium curve bounding the greatest area between itself and the  $45^\circ$  diagonal.

The equilibrium curve for the system toluene - ethylene dichloride is a normal curve and therefore the concentration gradient curves from 0 - 100 mol per cent should approach a perfect S - shaped curve. The lower portion of the concentration gradient curves for isopropyl alcohol and water were more curved at steam chest temperatures of 200 - 210°F. and tended to become straight at a temperature of 190°F. This effect could not be noted in concentration gradient curves for toluene - ethylene dichloride. In other words, separation was more complete at low rates of distillation, but this effect was apparent only on the first two plates. The upper part of the curve seemed to slope off more rapidly and at a slightly lower concentration at the higher rates of distillation. However, this rule was not followed in Experiment 9, Figure 11, where the final concentration was higher than in Experiments 7 and 8.



Murphree Plate Efficiency: Murphree Plate Efficiencies were subject to rather high error as shown by the scattered points in Figures 12, 13, 14, and 18. This was to be expected since an error in sampling or analysis of one plate concentration caused a corresponding error in the efficiency for that plate and for the plate above or below. In Figure 18 the efficiency for the seventh plate of Experiment 10 was 79.7 per cent; for the eighth plate the efficiency decreased to 50.7 per cent, and increased to 93.8 per cent for the ninth plate. At the higher concentrations a very small error in analysis or sampling caused a serious error in plate efficiency due to the small concentration changes used in calculating the efficiencies.

Plate efficiencies tended to increase to a peak in the lower section of the column, drop sharply in the center portion, and then began to rise again at the top of the column. The abrupt rise in the lower part of the column was where the equilibrium curve was the greatest distance from the  $45^\circ$  diagonal and corresponded to the straight portion of the concentration gradient curves. (See Figures 9, 10, 11, and 17).

Average Murphree Plate Efficiencies for isopropyl alcohol and water varied from 23.2 to 52.3 per cent while those for toluene - and ethylene dichloride were 76.7 and 79.1 per cent respectively. Individual Murphree Plate Efficiencies varied from 0 to 88.5 per cent for isopropyl alcohol and water while efficiencies for toluene and

ethylene dichloride varied from 44.5 to 121 per cent. It seems inaccurate to take an average efficiency because obviously, the plate efficiency varies with the concentration on the plate. The reason that plate efficiencies for toluene and ethylene dichloride are more consistent than for isopropyl alcohol and water due to the fact that the theoretical operating line for the former is a much closer approach to the actual than is the theoretical to the actual operating line for isopropyl alcohol and water. (See Figures 15, 16, and 19).

The fact that plate efficiencies for both binary mixtures reached a peak on Plate 2 is probably a coincidence. In the distillation of toluene and ethylene dichloride the operating lines for the stripping and rectifying sections intersected near the concentration of ethylene dichloride on Plate 2, coming the closest to the equilibrium curve and thus giving an apparent high efficiency. The apparent high efficiency on Plate 2 for isopropyl alcohol and water corresponds to the flat portion of the equilibrium curve where the horizontal distance between the equilibrium curve and the actual operating line is nearly the same as the distance between the equilibrium curve and the theoretical operating line.

Theoretical and Actual Operating Lines: Murphree Plate Efficiency is based on straight line rectification and obviously this can not be true as shown by Figures 12, 13, 14, and 18, where the efficiency varies as the plate concentration. Since all of the plates were of the same construction, each should have given the same plate

efficiency. Therefore, plate efficiency based on the McCabe - Thiele operating line is incorrect due to the assumption of a false operating line. For the distillation of toluene and ethylene dichloride the theoretical operating line is a fair average of the actual one.

(Figures 19, 20). Thus, Murphree Plate Efficiencies vary somewhat but are an average of the column performance. This will hold true as long as the equilibrium curve is an ideal one and the theoretical operating lines intersect near the center of the equilibrium curve to give an approximation of a curve. The odd but characteristic shape of the equilibrium curve for isopropyl alcohol and water however, is such that the theoretical operating lines is not a good approximation of the true operating line. (Figures 15 and 16). Therefore, Murphree Plate Efficiencies became meaningless when the McCabe - Thiele operating line is used for non-ideal solutions. Average Murphree Plate efficiency as calculated at present from straight line operating lines will depend on the number of plates in the column, i.e., the degree of separation obtained.

Figures 15, 16, 19, and 20 are plots of the actual and McCabe - Thiele operating lines shown in relation to the equilibrium curves. The actual operating lines were determined by plotting the plate concentrations step-wise so that the steps fell along the equilibrium curves rather than along the McCabe - Thiele operating lines. The coordinates of the lower corners of the plates were then taken as the coordinates of the actual operating lines. These lines

have been plotted on the basis that the plates are all 100 per cent efficient. This may or may not be true, but it may be assumed that since the plates are constructed the same, there will be a constant efficiency for all the plates. Therefore, if the plate efficiency is less than 100 per cent, the entire operating line will be lowered the required amount.

It may be seen from a reference to Figures 15, 16, 19, and 20, that the actual operating lines are, in effect, parallel to the equilibrium curves over the center sections and intersect the equilibrium curve at 0 and 100 mol per cent concentration or at the constant boiling mixture concentration in the case of isopropyl alcohol and water.

An inspection of Figure 15 shows the reason for the apparent discrepancies in Murphree Plate Efficiency. Referring to Experiment 1, it may be seen that the liquid concentration was actually increased from 8.8 to only 15.4 mol per cent isopropyl alcohol on Plate 1, while theoretically according to the McCabe - Thiele operating line, the composition should have been increased from 1.6 to 15.4 mol per cent isopropyl alcohol. When these data were used in calculating the efficiency, assuming the theoretical operating line, low values were obtained. Plate 2 increased the liquid concentration from 15.4 mol per cent to 43.9 mol per cent. This compares favorably with calculations made from the theoretical operating line and thus, Plate 2 gave an apparently high plate efficiency. When the liquid concen-

tration passes 40 mol per cent, the relative horizontal discrepancy between the actual and theoretical operating lines becomes large again and Murphree Plate Efficiencies decrease rapidly. As the concentration nears the constant boiling mixture, the horizontal discrepancy between the theoretical and the actual operating line becomes small and the Murphree Plate Efficiency rises. The location of this end of the actual operating line for isopropyl alcohol and water is not clear due to the previously stated difficulties of analysis. Apparently, however, the theoretical and actual operating lines became closer together and finally identical. This explains the increase in the apparent plate efficiency at high place concentrations.

Figures 19 and 20 show that the actual operating lines for the distillation of toluene and ethylene dichloride closely parallel the equilibrium curve. Discrepancies in Murphree Plate Efficiency can be explained by noting the discrepancy between the actual operating lines and the McCabe - Thiele operating lines.

Effect of Rate of Distillation: The rate of distillation seemed to have little effect on plate efficiency and product purity over the range of 157 to 376 gms./min. of distillate in the distillation of isopropyl alcohol and water. These rates of distillation correspond to superficial vapor velocities of 0.18 and 0.44 ft. per sec. The calculated maximum advisable vapor velocity<sup>(52)</sup> is approximately 1.2 ft. per sec. Since plate efficiencies have been reported

relatively constant over large rates of distillation, both of these rates are probably well within this constant efficiency range. Pressure drops were negligible, ranging from 0.9 in. Hg to 1.1 in. of Hg at the extremes of distillation rates. An increase in the rate of distillation however, did give a lower concentration of the more volatile component in the waste from the column.

Effect of Feed Concentration: The feed concentration was found to have little effect on plate efficiency and product purity over the range of feed concentrations of 4.0 to 10.6 mol per cent isopropyl alcohol. As might be expected waste concentrations were lowered when the feed concentrations were lowered. In the distillation of isopropyl alcohol and water, Experiment 9, the feed concentration was 3.0 mol per cent alcohol. The average overall Murphree Plate Efficiency was 52.3 per cent, or approximately 20 per cent higher than in all other distillations. A possible explanation for this is that the driving force (temp. gradient) was greater than in other tests. While the rate of distillation was low (157 gms./min.) the temperature drop from Plate 1 to the top of the column was high (210 - 169°F.).

Effect of Feed Plate Location: The feed plate location was found to be extremely important. In some preliminary experiments not reported in this paper, the introduction of the feed at a plate in the column on which the liquid concentration was higher than the feed concentration caused the entire stripping section to be inoperative.

The efficiency of the feed plate was also lowered but no effect could be noted on the rectifying section of the column. The effect of introducing the feed too low in the column was to increase the efficiency of the stripping section and feed plate, and to decrease the efficiency of the rectifying section of the column.

It was found that the feed plate location could be changed by increasing or decreasing the steam pressure to the column. This changed the temperatures in the bottom of the column, and thus changed the plate concentrations. Therefore, it was possible in Experiment 7 to introduce feed of 8.8 mol per cent isopropyl alcohol concentration on Plate 1 using a steam pressure of 2.5 in. of Hg, while in Experiment 9 a feed concentration of 3.1 mol per cent isopropyl alcohol was introduced on Plate 4 by using a steam pressure of 13.0 in. of Hg.

### Recommendations

System Toluene - Ethylene Dichloride.-It is recommended that a further study be made of the distillation characteristics of this system with a view to determining the location of the actual operating line under different conditions of column operation, i.e., feed concentrations from 5 to 35 mol per cent ethylene dichloride, rates of distillation from 0.2 to 1.5 feet per second, and reflux ratios from 0.5 - 1 to total reflux.

Study of Actual Operating Lines.-In view of the commercial importance of the ability to predict with accuracy the number of plates required for a given separation, it is recommended that efforts be made to determine the actual operating lines for other mixtures over a range of operating conditions. These true operating lines would not only enable plate calculations to be made with accuracy but would also permit the comparison of various types of plate construction.

The Column Assembly.-Since poor mixing on plate #2 of the column was noted, it is recommended that copper baffles or other devices be installed on the plates to increase the efficiency of mixing. The purchase and installation of a suitable feed pump is recommended, as well as the replacement of all galvanized iron pipe and apparatus contacting the mixture being distilled, with copper material in order to decrease the formation of iron rust. The feed heater should be lowered at least one foot and the feed introduced



one foot higher into the heater in order to obtain an increased heating area. A rotameter should be installed in the waste line from the column to enable a true material balance over the column to be made.

#### Limitations

The results from the distillation of isopropyl alcohol and water are based only on rates of distillation from 0.18 to 0.44 feet per second, feed concentrations from 3.1 to 10.8 mol per cent isopropyl alcohol, a reflux ratio of 3 to 1, and a feed rate of 330 gms./min.

The results from the distillation of toluene and ethylene dichloride are based only on two tests made with feed concentrations of 41.4 and 39.7 mol per cent ethylene dichloride, distillation rate of approximately 200 gms./min., a reflux ratio of 4 to 1, and a feed rate of 138 gms./min.

Allowances for differences in column construction must be made in using this data for calculating column performance. It is applicable only to a continuous fractionator, with feed at its boiling point, introduced on an intermediate plate.

## V - CONCLUSIONS

### The System Isopropyl Alcohol - Water

From the operation of a fifteen plate bubble-cap column (8-3/8" I.D., one 3-7/8" O.D. bubble-cap per plate, and plate spacing of 5-7/8") in the continuous fractionation of aqueous isopropyl alcohol under the following operating conditions: feed introduced at its boiling point at concentrations from 3.1 to 10.8 mol per cent isopropyl alcohol, feed rate of 300 gms./min., reflux ratio of 3 to 1, and superficial vapor velocities from 0.18 to 0.44 feet per second, the following conclusions may be made:

(1). The assumption of the McCabe - Thiele operating line is probably incorrect. The actual operating line for this mixture is a curve, approximately parallel to the equilibrium curve and joins the equilibrium curve at its intersections with the 45° diagonal.

(2). Murphree Plate Efficiencies varied from 0 - 100 per cent. Between 2.5 and 45 mol per cent alcohol concentrations these efficiencies increased from approximately 30 per cent to a maximum of 88.5 per cent; between 45 and 60 mol per cent alcohol, plate efficiencies decreased to a minimum ranging from 3.2 to 40 per cent; and for concentrations above 60 mol per cent alcohol the plate ef-

iciencies tended to increase again, efficiencies ranging from 4.2 to 100 per cent. These efficiencies can be shown to vary with the relative deviation of the actual and the McCabe - Thiele operating lines from the equilibrium curve.

(3). Rate of distillation had little effect on product purity since overhead distillates of 376, 171, and 157 gms./min. gave product concentrations of 64.3, 63.9, and 66.1 mol per cent respectively.

(4). Feed concentration had little effect on product purity. Product concentrations of 64.3, 64.4, 63.9, and 66.1 mol per cent isopropyl alcohol were obtained when feed concentrations of 9.8, 8.8, 4.0, and 3.1 respectively were used.

(5). Improper mixing caused an error on plate #2 of as high as 4 mol per cent in the determination of the plate equilibrium concentrations.

(6). Product concentrations of from 66 to 67 mol per cent and waste concentrations of 0 mol per cent isopropyl alcohol may be obtained from the column.

The System Toluene - Ethylene Dichloride

From the results obtained in the study of fractional distillation of the system toluene - ethylene dichloride in the column previously described and under the following operating conditions: feed concentrations, 41.4 and 39.7 mol per cent ethylene dichloride, introduced at the boiling points of the feed (204°F. and 206°F. respectively), feed rate of 138 gms./min., reflux ratio of 4 to 1, and distillation rates of 203 and 196 gms./min. respectively, the following conclusions may be made:

(1). The McCabe - Thiele operating line approximates the actual operating line for this mixture which is a curve, nearly parallel to the equilibrium curve, and joins it at its intersections with the 45° diagonal.

(2). Murphree Plate Efficiencies varied from 44.5 to 121 per cent. Between 85 and 98 mol per cent ethylene dichloride, the plate efficiencies decreased to a minimum of 44.5 per cent, and above concentrations of 98 mol per cent ethylene dichloride, the efficiencies increased to approximately 100 per cent. These efficiencies varied with the relative deviation of the actual and the McCabe - Thiele operating lines from the equilibrium curve.

(3). Improper mixing on plate #2 of this column caused an error of two (2) mol per cent in the determination of the equilibrium concentration on the plate.

(4). Product concentrations of from 99.3 to 99.7 mol per cent ethylene dichloride may be obtained from the distillation of this mixture in the column.

## VI - SUMMARY

The process of fractional distillation has been performed for many years but has only within the past half century become a true science. However, because of the many variables involved and the uncertainty of their effect upon fractionating column efficiency, the design of columns has long been a major engineering problem. Plate and column efficiencies, as given in the literature, vary widely and it was thought possible that the inconsistencies of the results reported might be due to poor equilibrium within the column, caused in part by faulty sampling.

In order to determine the operating characteristics of a fifteen plate bubble-cap column (8-3/8" I.D., one 3-7/8" bubble-cap per plate, and plate spacing of 5-7/8"), the effects of the operating variables, and the distillation characteristics of two different types of binary mixtures, the column assembly was redesigned for continuous distillation and provisions made for the introduction of feed at its boiling point to any one of the bottom eight plates. The binary mixtures distilled were isopropyl alcohol - water (distillation rates 157 - 376 gms./min., feed concentrations 3.1 - 10.8 mol per cent iso-

propyl alcohol, reflux ratio 3:1, and feed rate of 330 gms./min.), and toluene - ethylene dichloride (distillation rates 203 and 196 gms./min., feed concentrations 41.4 and 39.7 mol per cent ethylene dichloride, reflux ratio 4:1, and feed rate of 138 gms./min.) In order to reduce the possibility of disturbances within the column by removal of large plate samples, the refractive index method of analysis was used which required maximum samples of only 4 ml.

Results of the experiments made indicated that the assumption of the McCabe - Thiele operating line is probably incorrect. In the case of the isopropyl alcohol - water fractionation, a considerable divergence (largest between 10 and 40 mol per cent isopropyl alcohol) between the McCabe - Thiele and the actual operating line was noted, the actual operating line being a curve approximately the shape of the equilibrium curve. Murphree Plate Efficiencies for the mixture varied from 0 to 100 per cent, depending primarily on the relative deviations of the two operating lines from the equilibrium curve. The McCabe - Thiele operating line for the system toluene - ethylene dichloride closely approximated the actual operating line. Murphree Plate Efficiencies varied from 44.5 to 121 per cent, but the individual plate efficiencies were more consistent with the average efficiency. The rate of distillation over the range of 157 to 376 gms./min. and feed concentration over the range of 3.1 to 10.8 mol per cent isopropyl alcohol, at a reflux ratio of 3:1 had negligible effect on product purity which ranged from approximately 62 to 67 mol per cent isopropyl alcohol.

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#### ADDENDUM

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