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EFFECT OF HOLE SIZE
ON PLATE EFFICIENCY
IN A
PERFORATED PLATE DISTILLATION COLUMN

by

Frank Leonard Kuchinski

A THESIS

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This thesis is accepted and approved in partial fulfillment of the requirements for the degree of Master of Science in Chemical Engineering.

Oct 11, 1955
Date

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Alan S. Foust
Head of the Department

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ABSTRACT

This report presents the results of distillation runs made in an effort to determine the effect of plate hole size, 1/4, 3/16, and 1/8 inch diameter on Murphree plate efficiency at variable mass flow rate. The system methyl alcohol-water was studied. In the distillation runs, all salient plate design variables and experimental operating conditions were maintained constant.

Mass flow rates ranged from 50-200 lb/hr-ft^2, 100-170 lb/hr-ft^2, and 130-150 lb/hr-ft^2 for plates having 1/4, 3/16, and 1/8 inch hole diameters, respectively.

Results show no observed effect of hole diameter or mass flow rate on Murphree plate efficiency for plates 1 and 2 within the range of data obtained in this study. However, for the bottom plate, 3, mass flow rate constant, Murphree plate efficiency increased from 44 percent for the 1/4 inch hole diameter plates to 96 percent for the 1/8 inch hole diameter plate indicating higher plate efficiency for smaller hole diameter perforations.

Correlation of average dry and wet pressure drop data indicate the equation $P = R (P + S)$ to be accurate within 5 percent for predicting total plate pressure drop.

INTRODUCTION

Recent research studies made on perforated plate-distillation columns have clearly indicated the numerous advantages this type of tray offers as compared to the conventional bubble-cap plate column. The misbelief of the overall inferiority of the perforated tray is partially attributed to conclusions based on meager design information previously contained in the literature. Chief among these misbeliefs were the limited range of vapor and liquid flows permissable, general lower plate efficiency, and difficulty of operating the column.

Results of Mayfield et al(8), Marsh(7), Ragatz(11) have shown perforated trays are more economical and more efficient for many applications as compared to bubble-cap plates. These studies have also resulted in data whereby the design of the perforated plate column can be made with more confidence.

The purpose of this investigation was the design and construction of a perforated plate distillation column with the object of studying the effect of changes in mechanical design features of a perforated plate on plate efficiency. Since the number of mechanical design features which could be studied are rather numerous it is the scope of this report to study the effect of perforation size, 1/4, 3/16, and 1/8 inch diameter, on plate efficiency while maintaining other plate variables constant. Methyl alcohol and water was the system selected to be studied.

LITERATURE REVIEW

Distillation is the separation of the more volatile component of a liquid mixture by a series of vaporizations and subsequent condensations of the more volatile component. Rectification of the more volatile component takes place in the following manner. Vapor in a column passes countercurrently to liquid reflux on a tray and condenses giving up its heat of condensation to the liquid on the tray, resulting in the vaporization of the lower boiling component in the liquid. These series of vaporizations and condensations take place on each tray of the distillation column until finally the vapors leaving the top tray are totally condensed in a condenser. Part of the distillate from the condenser is usually removed as product and the remainder returned to the top of the column as liquid reflux.

The process of distillation is usually performed in a bubble cap, perforated plate or packed type column. A perforated plate differs very little from a bubble cap plate in its operation. Both have the primary function of more efficienty effecting mass transfer of the lower boiling component from the liquid to the vapor and the higher boiling component from the rising vapor to the descending liquid. In a perforated plate vapor bubbles through a large number of small holes through a liquid whereas in a bubble-cap plate vapor bubbles through a series of caps containing slots submerged in the liquid. Vapor bubbling through the perforated tray must support the liquid above it; otherwise liquid will drain to the plate below. In the perforated plate the height of the liquid on the tray is governed by the weir height; in the bubble cap plate liquid height is maintained by the height of the downcomer pipe and does not drain the tray of liquid in the event vapor velocity is not great enough to support it. It is obvious that a minimum gas velocity (weep point) and pressure drop exists, depending on the perforated plate design and operating conditions, below which efficient operation of the perforated plate ceases. For perforated and bubble-cap plates an upper limiting vapor velocity (flood point) exists where the liquid seal in the downcomer is broken resulting in the flooding of the plate and column with liquid.

In the usual procedure for designing a distillation column for a desired rectification, the theoretical (or perfect) plates can be readily calculated by known methods(10,4). The design engineer is handicapped by the unknown relationship existing between the theoretical and actual plates, i.e., overall plate efficiency of the column.

Plate efficiency is usually expressed as overall column efficiency, and Murphree Plate efficiency. The overall column efficiency is defined as the ratio of the number of theoretical plates necessary to produce a given separation to the number of actual plates used to achieve the same separation under similar reflux conditions. The Murphree efficiency is defined by the ratio of the actual change in composition, which the vapor undergoes in passing through a plate to the change in composition which it would undergo if it left the plate in equilibrium with the liquid which leaves the plate. A method of relating Murphree plate efficiency to overall column efficiency is given by Perry(10).

In equation form Murphree plate efficiency is expressed as follows:

$$E_m = \frac{y_n - y_{n-1}}{y_n^* - y_{n-1}} \quad (100)$$

where: E_m = Murphree plate efficiency (percent)

y_n = mol fraction of vapor leaving plate n

y_{n-1} = mol fraction of vapor leaving plate n-1

y_n^* = mol fraction of vapor in equilibrium with liquid leaving plate n

Murphree plate efficiencies more than 100 percent can be obtained when the vapor leaving the plate is richer than the vapor in equilibrium with the liquid leaving the plate. This is possible, especially in large diameter columns, if the vapor leaving the plate is incompletely mixed and is in equilibrium with the liquid leaving the liquid on the inlet weir.

Two of the major factors affecting overall plate efficiency are the mechanical design features of the plate and column, and the physical properties of the mixture being distilled.

Various methods of correlating the physical properties of the mixture with plate efficiency have been presented in the literature. The bases of these correlations are rather varied indicating the complexity of predicting overall plate efficiency. O'Connell's$^{(4)}$ correlation uses the product of the liquid viscosity and the relative volatility at the average column temperature; Drickamer and Bradford$^{(4)}$ correlation is based on the viscosity of the feed; Geddes$^{(4)}$ uses surface tension, density of liquid, density of vapor and diffusivities of the phases in the mixtures as well as the area of the bubble-cap slot. Gunnes$^{(4)}$ uses vapor pressure of feed stock, i.e., properties of the feed. The most recent of these correlations deserving further study is the method of Gerster et al$^{(3)}$ using separate gas and liquid film resistances to predict plate efficiency.

Considerable progress has been made recently in determining the effect of the various mechanical design features of a perforated plate. Umholtz$^{(13)}$ in his study of overall plate efficiency in a 1.83 and 3 inch diameter perforated plate distillation columns has studied the effect of free space area, percent downcomer area, pitch/diameter ratio of perforations, and perforation size. Nandi and Karim$^{(9)}$ using a 2-1/4 inch diameter column studied the effect of distillation rate, free space area, plate spacing, weir height, and perforation size on overall plate efficiency. Arnold et al(1) using a 15 inch diameter column studied pressure drop behavior of perforated trays (using air and water) by varying weir height, perforation size, free space area, and L/G ratios. Mayfield et al(8) using a 6 inch diameter column studied pressure drop behavior of perforated plates (using air) by varying the perforation size. In addition using a 6.5 foot diameter column data were obtained (using air-water) relating pressure drop, weir height, and L/G ratios.

In spite of these recent studies previously mentioned, attempts to correlate mechanical plate design features with the physical properties of the liquid in an effort to predict plate efficiency have as yet not been successful. The main reason being the complex nature of mass transfer between the vapor and liquid phases as a result of the changing physical phenomena of the vapor-liquid interaction as the mass flow rate is varied.

This vapor-liquid interaction can best be described as the foaming or frothing which a liquid undergoes on and above a plate as the mass flow rate is varied. Visual observation of a distillation tray in stable operation indicates three zones of different foam density. A highly agitated foam layer through which bubbles form and rise through the liquid, a zone of dense droplets, and finally a zone composed of a fine mist.

Mass transfer in these three zones is dependent upon the vapor and liquid film resistances, concentration gradient and surface area between the two phases. Gerster et al(3) has attempted to relate foam height with mass flow rate and the overall mass transfer coefficient in an effort to predict overall plate efficiency. West et al(14) elaborating on the equation proposed by Gerster et al(3) expressed the foam height in terms of fraction voids in the foam. In addition interfacial area was expressed as a function of volume of foam. It is believed that if plate efficiency data for a variety of liquids be obtained as a function of mass flow rate and utilizing the proposed methods of Gerster et al and West et al plate efficiency for any system could be calculated with relative ease and accuracy.

EXPERIMENTAL PROCEDURE

The object of this study was the design and construction of a perforated plate distillation column to determine the effect of plate hole size, 1/4, 3/16, and 1/8 inch diameter on Murphree plate efficiency. Methyl alcohol and water was the system selected to be studied in lieu of published equilibrium, density and refractive index data which was readily available.

Plans were to hold all salient plate design variables constant, except hole size, and perform distillation runs under constant operating conditions while varying the mass flow rate through the column. Plate design variables which efficiency is believed to be affected by and which were maintained constant are:

1. Weir height ------------------ 1/2 inch
2. Weir length ------------------ 5 inches
3. Column diameter, I.D. -------- 8.25 inches
4. Plate spacing ---------------- 9.5 inches
5. Perforated plate thickness --- .029 inches
6. Pitch/diameter of holes ------ 3.8
7. Percent downcomer area ------- 0.826
8. Baffle arrangement ----------- 3/8 inch above perforated plate and 5/16 inches from weir. Height of baffle 5 inches.
9. Percent free space area ------ 1.17

Conditions at which the experiments were performed are: concentration of kettle charge 25 percent methanol-75 percent (weight %) water, atmospheric pressure, and constant L/G ratio, i.e., total reflux. The temperature of the reflux was maintained constant ±0.5°F and 2-3 degrees below the boiling point of methanol.

The glass portions of the distillation column were wrapped with two layers of glass wool asbestos, with the exception of approximately four square inches of the front of each glass section. This unwrapped section was covered with a piece of plexiglass through which plate distillation phenomena could be easily observed.

Samples of liquid and vapor leaving each plate were taken after steady state column operating conditions were reached. Using a refractometer all samples were analyzed for percent methanol, from these data Murphree plate efficiency could be readily calculated.

APPARATUS AND MATERIALS

The apparatus employed in this investigation consists mainly of the perforated plate distillation column and steam boiler. Only essential information relative to the auxiliary apparatus will be listed below:

Pyrex Glass Cylinders. Four required, 8.25 inch inside diameter, 0.25 inches thick, 9.5 inches in height. Originally purchased from Hawshaw Scientific Glass Co. as battery jars. Battery jars were cut to size by Pittsburgh Plate & Glass Co., Allentown, Pa.

Brass Plate Holders. Five required, 12 x 12 x 3/8 inches. Brass sheet 0.029 inches thick, used for the distillation plate. Purchased from Whitehead Metals Co., Philadelphia, Pa.

Plate Gaskets. Teflon impregnated asbestos-3 ply, 9 inch outside diameter, 8 inch inside diameter. Manufactured by Johns-Manville Corp., Manville, N. J.

Distillation Kettle. Glass lined, 18 inch I.D., 25 inches in height, 75 psig jacket pressure, total heating surface 10 ft^2. Manufactured by Pflauder & Co., Rochester, N.Y.

Refractometer. Abbe 56, Manufactured by Baush & Lomb, Rochester, N. Y.

Steam Boiler. Gas fired, 5 H.P. Mfgs. Steam Rating. Used to supply steam to jacket of distillation kettle. Manufactured by Mears-Kane-Ofeldt, Inc., Philadelphia, Pa.

Methanol. Technical grade. Purchased from Lehigh Valley Chemical Co., Easton, Pa.

PERFORATED PLATE COLUMN DESIGN

The ideal column for this experimental study should be simple in construction to allow frequent changes in plate design, small enough to obtain reasonable boilup rates, permit close observation of the distillation process, and yet not be too costly. In Figure I, page 15, is shown a flow diagram and Figure II, page 16, a photograph of the perforated plate column and apparatus used in the experimental study.

SELECTION OF COLUMN SIZE

A four-plate column (2), 8-1/4 inch I.D. pyrex glass cylinders, 9-1/4 inches in height was selected and found to be commensurate with the boilup expected using a steam jacketed glass-lined kettle and heat available from the steam main.

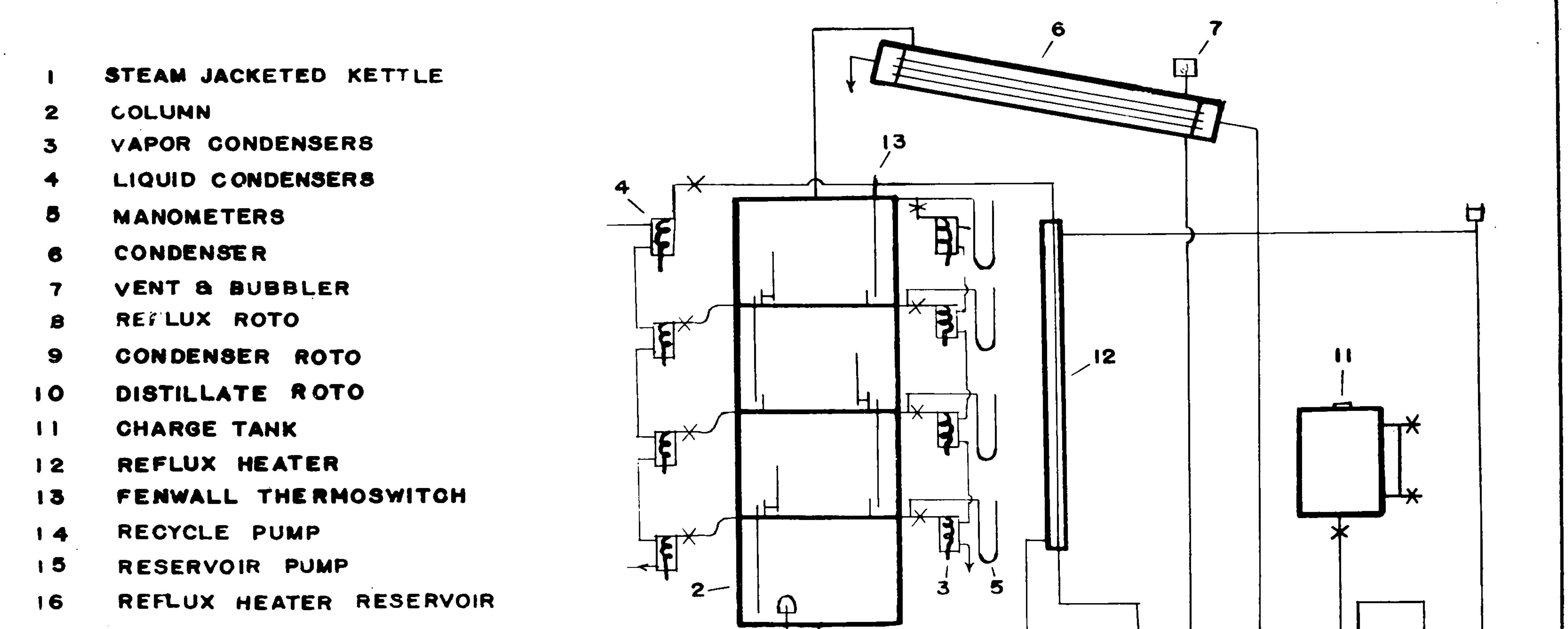
CONDENSER SPECIFICATIONS

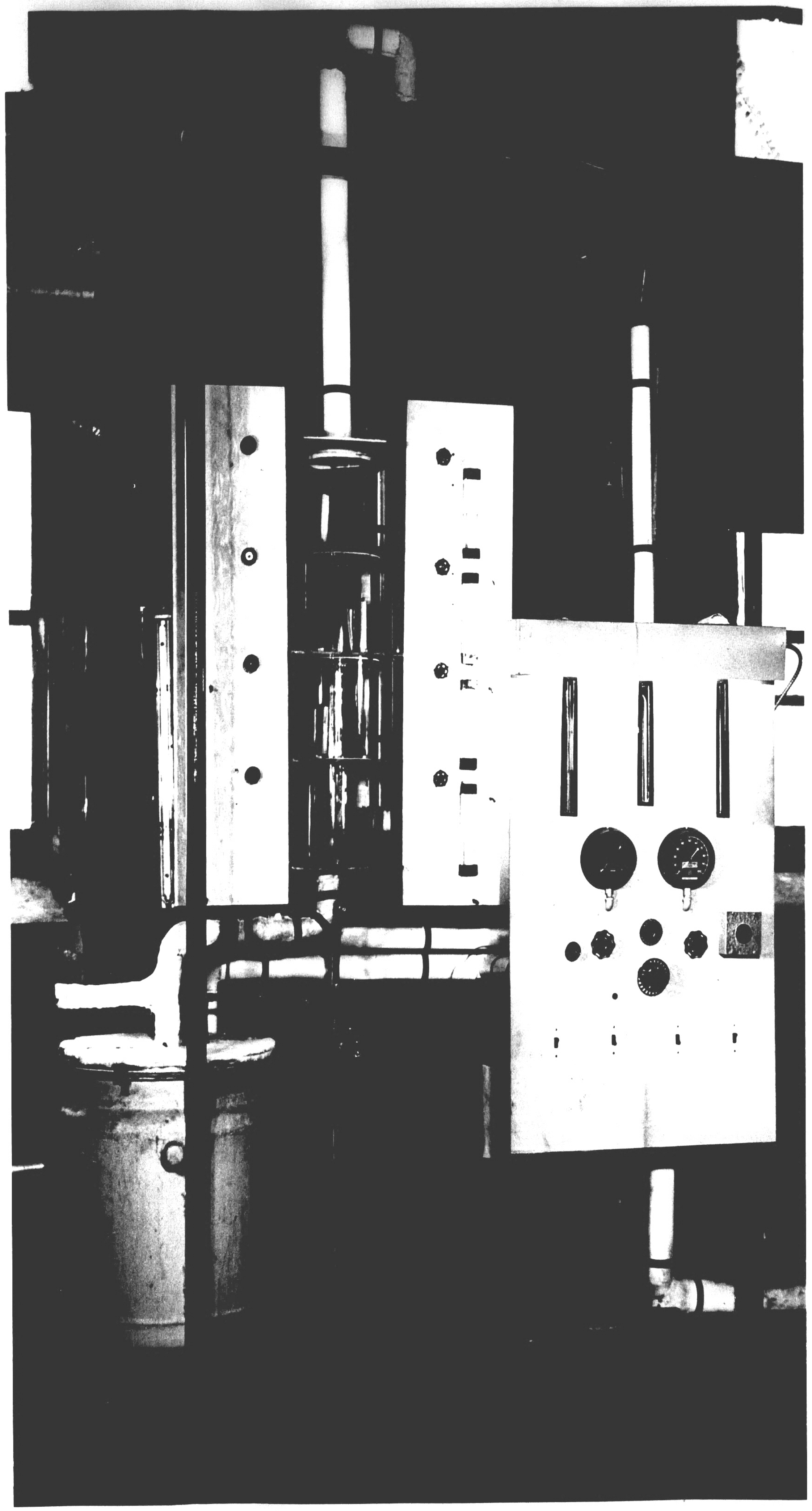
On the basis of the boilup of methanol expected from a 5 HP steam boiler and using a steam jacketed glass-lined kettle a condenser was designed. The specifications of this condenser are as follows: horizontal, single pass, counterflow, with vapor condensing in the shell and water flowing through the copper tubes. Tubes, eight 5/8 inch O.D., shell I.D. 2-3/4 inches, tube length 32 inches, total tube surface area 3.50 ft^2.

REFLUX TEMPERATURE CONTROL SYSTEM

An automatic reflux temperature control system was

FIGURE I. DIAGRAM OF EQUIPMENT





designed to maintain the reflux temperature ± 0.5°F and to within 2-3°F of the boiling point of methanol. Temperature of the reflux was controlled by a fenwall thermoswitch located at the top of the column. By means of the fenwall thermoswitch (13) and a relay, a pump and an immersion heater (1000 watts) were turned on. The reservoir pump (15) recycled hot water from the reflux heater reservoir to a constant temperature reservoir in which was located a heat exchanger. Another pump (14) recycled water through the heat exchanger located in the constant temperature reservoir to the heat exchanger through which the reflux flowed. When the temperature of the reflux was up to the desired temperature, the fenwall thermoswitch through the relay turned the reservoir pump (15) and immersion heater off, the recycle pump being in continual operation throughout the distillation run.

PLATE HOLDER DESIGN

Selection of the 12 x 12 x 3/8 inch brass plate was made on the basis of the ease at which brass could be machined. In the layout of the plate holders, calculations were based on obtaining maximum perforation area. With cross flow of liquid and using weirs, 58.2 percent perforation area was obtained. Figures III and IV, pages 18 and 19, show the details of construction of the five plate holders. Plate holder four is identical to plate holder two and is not shown in the drawings.

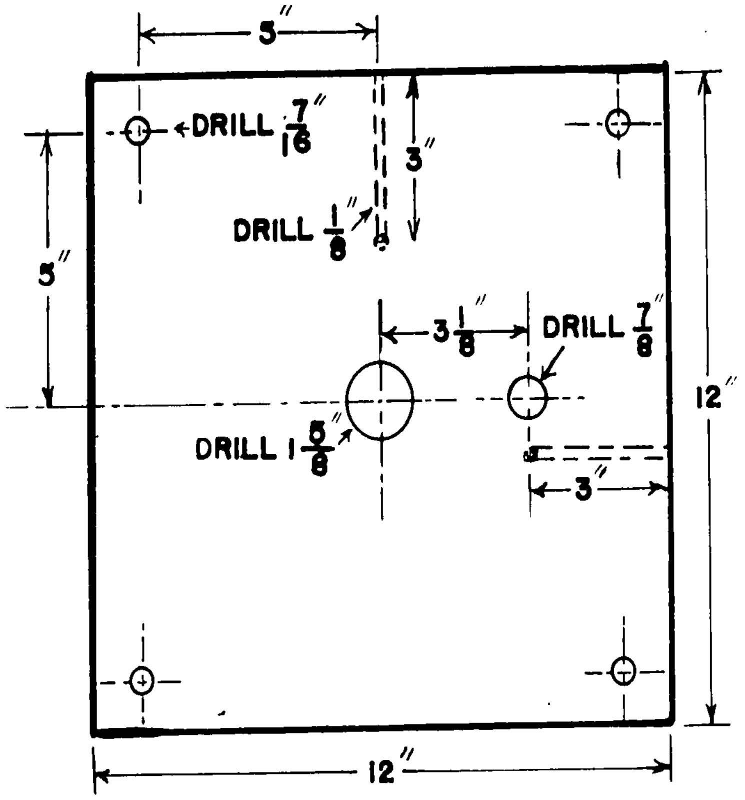


PLATE HOLDER -1

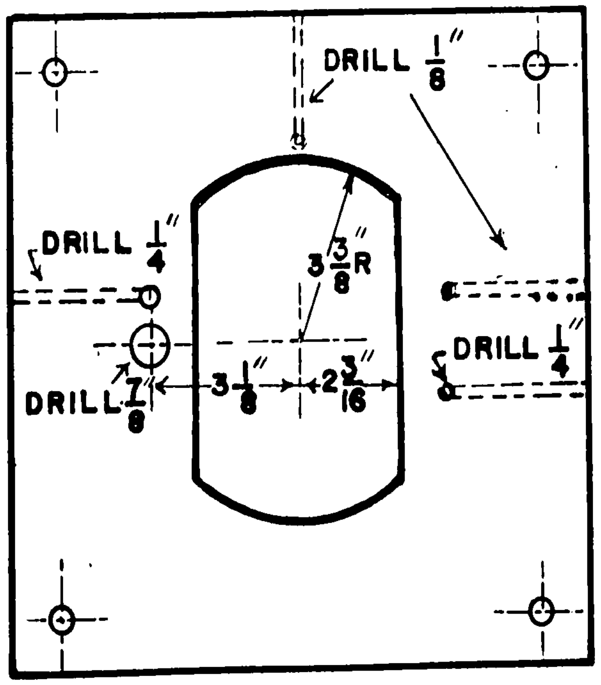


PLATE HOLDER -2

FIGURE III.

SCALE 1 = 4

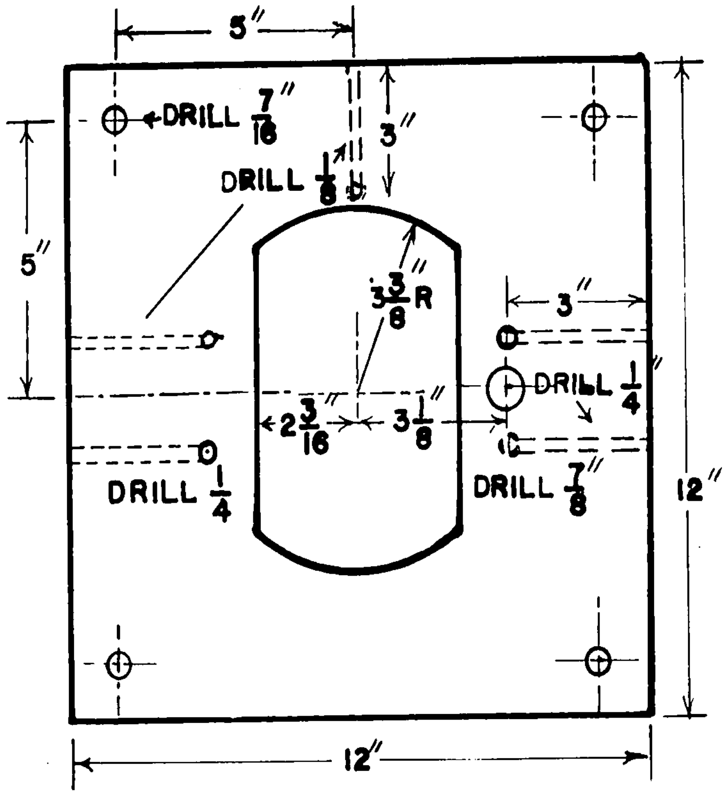


PLATE HOLDER—3

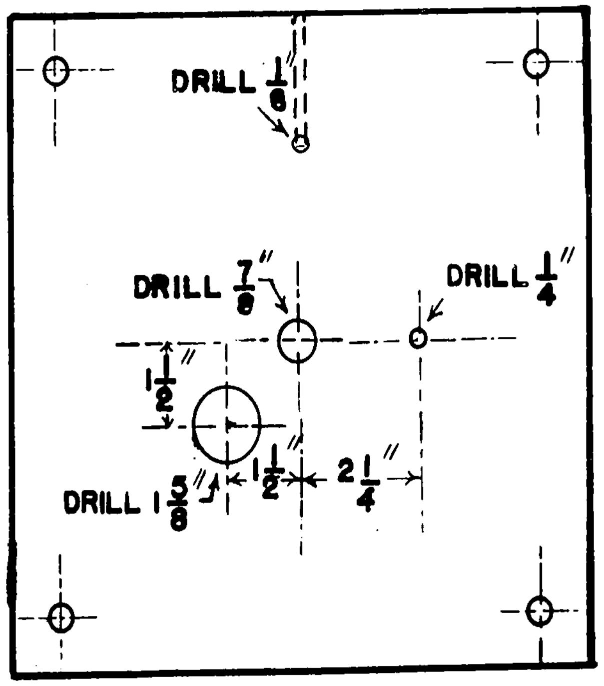


PLATE HOLDER—5

FIGURE IV.

SCALE 1 = 4

PLATE DESIGN

Selection of the percent free space area (1.17) was made by calculating the maximum mass flow rate, G, and computing the pressure drop across each tray by fixing the weir height, weir length, percent downcomer area, and baffle arrangement.

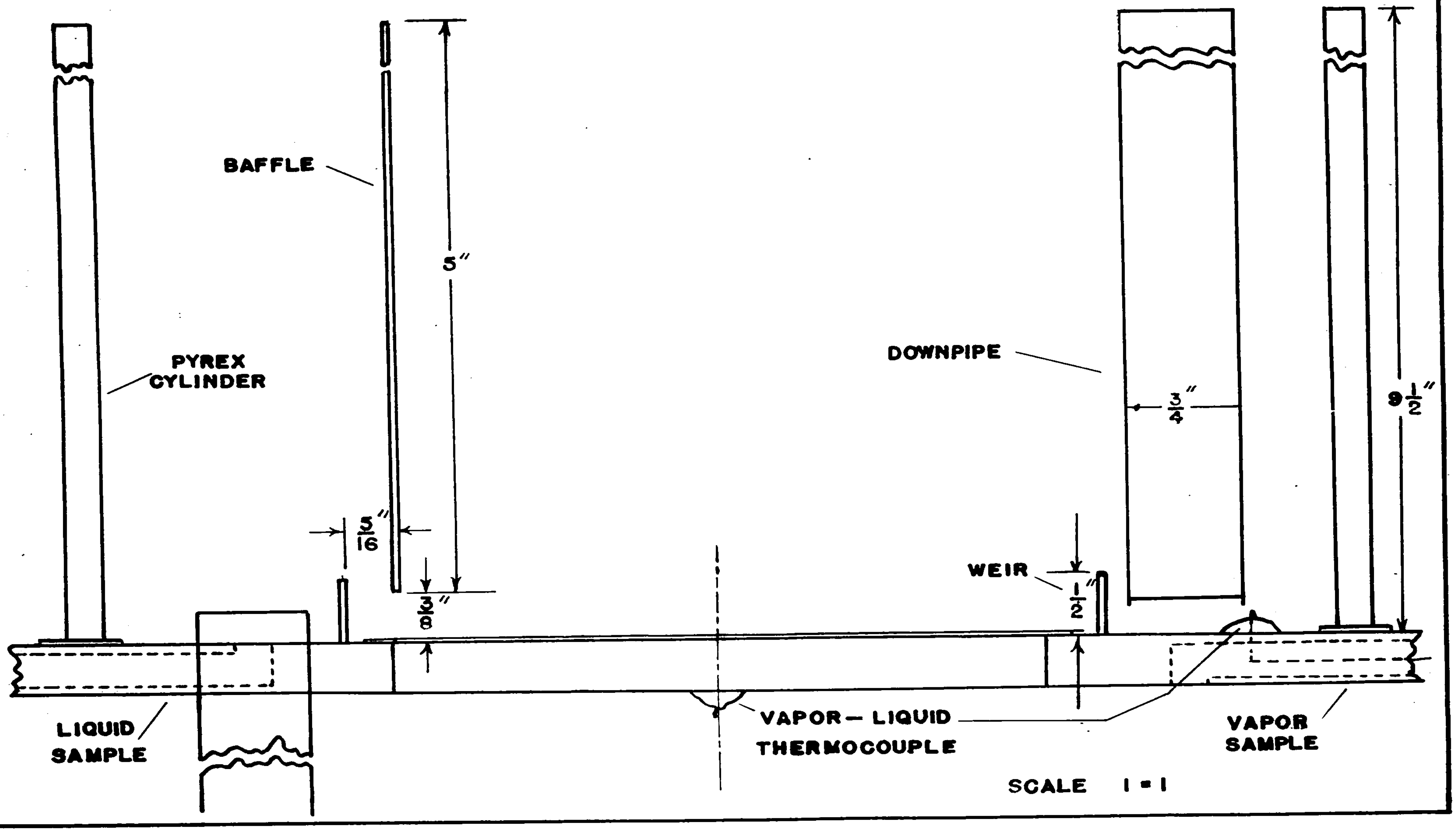
The pressure drop calculation consists of two (main) components, namely, the pressure drop as a result of vapor flowing through the perforations and the height of the liquid on the tray. To the liquid depth above the tray, the height of calculated weir head is added a correction called an "aeration factor"(2). The aeration factor being defined as the ratio of the observed pressure drop through the liquid on the tray to the calculated clear liquid on the tray. For purposes of design of this plate an aeration factor of 1.0 was used. Essentially this aeration factor takes into consideration the foaming characteristics of the liquid. To calculate aeration factors, the observed pressure drop through the liquid is obtained as the difference between the total observed pressure drop and the observed dry-tray pressure drop at the same air rate.

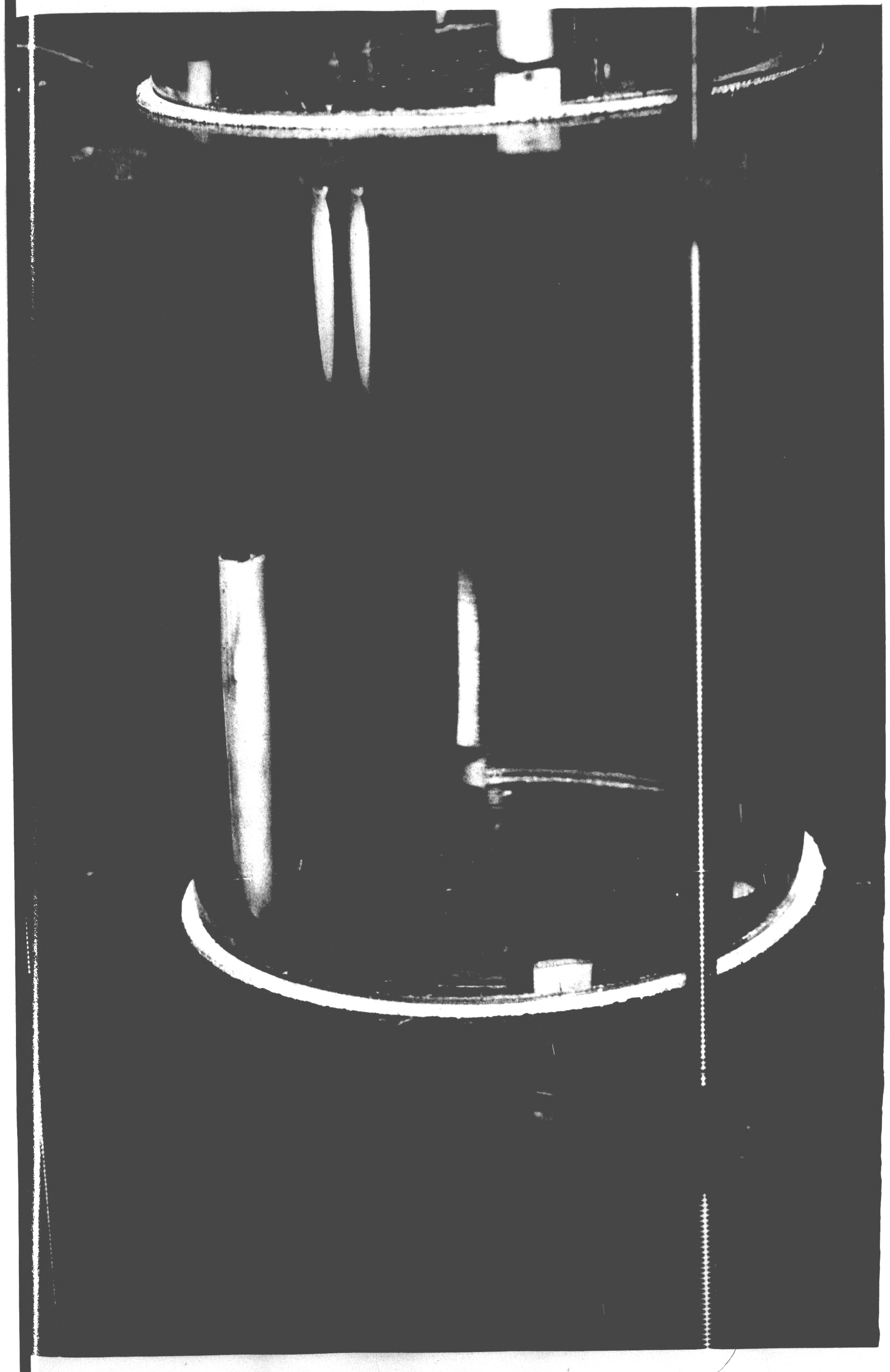
The computed pressure drop calculated in the above manner would have to be sufficiently large to overcome the resistance the vapors would be subjected to in passing through the perforations in addition to supporting the liquid above

FIGURE V.

PERFORATED PLATE

FRONT VIEW





each perforation. In addition, the upper limit of the pressure drop should be sufficiently low enough that the downcomer backup is not excessive and the column as a result, flooded. Consideration of the hole size was also made as the operating range of the column, i.e., range between the weep and flood point, is considerably reduced with an increase in hole size. In the design of any column it is desirable that the pressure drop be a minimum and yet the range between the weep and flood point be a maximum.

PLATE LAYOUT

The perforated plate was punched with the desired hole diameter punch on equilateral triangular centers with a pitch/diameter hole ratio of 3.8. Calculations for the plate layout are included in the Appendix C , page 41. The perforated plates were soldered to the plate holder. Figure VI, page 22, shows a 3/16 inch hole diameter perforated plate assembly.

VAPOR AND LIQUID SAMPLING SYSTEM

Referring to Figure IV, page 19, showing the details for the perforated plate holders, holes 1/4 inch diameter, were drilled through each side of the 3/8 inch brass plate holders. One hole was drilled through the bottom of the plate for the vapor sample and the other through the top to obtain a liquid sample; refer Figure V. Small condensers for cooling the liquid sample and condensing the vapor sample were located at each sampling outlet.

TEMPERATURE MEASUREMENT

Temperature of the liquid and vapor of each plate in the column were obtained using copper-constantan wire, an eleven terminal thermocouple selector and a Leeds-Northrup potentiometer. Referring to Figure V, page 21, the vapor and liquid thermocouple locations are shown. Holes 1/8 inch diameter was drilled into the sides of the plate holder, Figure IV, page 19.

DISTILLATION OR BOILUP RATE CONTROL

Control of boilup rate was accomplished by regulating the steam pressure in the steam jacket of the glass-lined kettle.

PRESSURE MEASUREMENT

The 1/4 inch holes drilled in plate holders used for obtaining vapor samples were also used to obtain the pressure below and above each tray.

MISCELLANEOUS COLUMN FEATURES

A charge tank was installed in which the concentration of the kettle charge could be easily increased or decreased as desired by regulating flow of the reflux and product streams.

A valve system was also installed for continuous distillation studies or for air water studies.

VAPOR RATE MEASUREMENT

A sharp edge orifice, flange type, 0.75 inch hole diameter was designed and calibrated. The calibration curve for this orifice is included in Appendix E, Figure VIII.

DATA AND RESULTS

The distillation runs and sample data obtained in this study are listed in Table IV of the Appendix F.

WET AND DRY PLATE PRESSURE DROP

Prior to starting the distillation runs using the binary, methyl alcohol and water, data were taken to obtain the resistance (pressure drop in inches of water), the 1/4 and 3/16 inch holes offered to the flow of gas. This was accomplished by recording the pressure below and above each plate when air at varying rates was pumped through the column. The difference in pressure was recorded as the pressure drop across the plate. A summary of these data recorded as dry plate pressure drop is shown in Table I, page 28.

Pressure drop data were also obtained during the distillation runs that were made in the course of the experimental study. These data for 1/8, 3/16, and 1/4 inch holes are recorded as wet plate pressure drop and are listed in Table II, page 29. Wet pressure drop data includes the resistance the gas, methanol vapor, encounters in passing through the holes in addition to supporting the liquid above it.

A graph showing the relationship between dry and wet pressure drop data for the 1/4 and 3/16 inch diameter holes is shown in Figure VII, page 30.

MURPHREE PLATE EFFICIENCY

In Table III, page 31, is a summary of all distillation runs listing the calculated Murphree plate efficiencies with the corresponding mass flow rate. A sample calculation showing the manner in which the results were calculated from experimental data are given in Appendix C, page 41.

TABLE I

Summary of Dry Plate Pressure Drop Data for 1/4, 3/16 Inch Diameter Holes Plate Series

No. of Reading	Orifice Reading In.-H_2O	P_1	P_2	P_3	P_4	P_1-P_2	P_2-P_3	P_3-P_4	Avg. P In.H_2O	G $\frac{\text{Lb.}}{\text{Hr-Ft}^2}$	Hole Diameter Inches
		In. Red Oil				In. H_2O					
1	2.50	2.90	3.35	4.0	4.18	1.33	1.03	1.40	1.25	168	1/4
2	1.95	2.25	2.50	2.90	3.20	0.738	1.18	0.885	0.93	148	1/4
3	1.00	1.20	1.35	1.55	1.70	0.442	0.59	0.442	0.49	107	1/4
4	0.41	0.60	0.61	0.75	0.83	-	0.44	0.222	0.22	68	1/4
5	1.85	2.10	2.40	2.75	3.00	0.885	1.03	0.736	0.88	145	1/4
6	3.80	4.25	4.85	5.53	6.15	1.77	2.01	1.83	1.87	209	1/4
7	0.20	0.30	0.31	0.40	0.44	0.030	0.265	0.127	0.14	48	1/4
8	3.80	4.35	4.90	5.55	6.25	1.62	1.92	2.07	1.87	209	1/4
1	2.60	3.45	3.90	4.33	4.73	1.33	1.27	1.18	1.26	171	3/16
2	0.58	0.80	0.90	1.00	1.12	0.295	0.295	0.354	0.32	80	3/16
3	0.20	0.33	0.35	0.43	0.45	0.074	0.22	0.074	0.56	48	3/16
4	0.20	0.30	0.35	0.43	0.45	0.148	0.22	0.074	0.15	48	3/16
5	0.78	1.10	1.20	1.35	1.50	0.295	0.443	0.443	0.39	94	3/16
6	1.65	2.30	2.60	2.95	3.28	0.885	1.03	0.974	0.96	137	3/16
7	2.80	3.85	4.38	4.90	5.47	1.565	1.535	1.68	1.62	179	3/16
8	3.25	4.45	5.10	5.70	6.30	1.92	1.77	1.77	1.82	193	3/16
9	0.20	0.30	0.35	0.40	0.45	0.147	0.147	0.147	0.15	48	3/16

TABLE II

Summary of Wet Plate Pressure Drop Data for 1/4,3/16,1/8 Inch Hole Diameter Plate Series

Run No.	Time	Plate In. Red Oil 1	2	3	4	Pressure In. H_2O P_2-P_1	P_3-P_2	P_4-P_3	Drop Avg. P	G Calc lb/hr-ft^2	Hole Diameter
6	1400	0.3	0.8	1.4	1.8	1.48	1.77	1.18	1.47	149	1/8
6	1530	0.3	0.8	1.35	1.7	1.48	1.62	1.03	1.35	137	1/8
6	1730	0.3	0.8	-	1.7	1.48	1.62	1.03	1.37	137	1/8
7	1545 1745	0.2	0.75	1.35	1.85	1.62	1.77	1.48	1.62	137	1/4
7	1815	0.3	1.20	1.85	2.35	2.66	1.92	1.48	2.02	165	1/4
7	1940	0.85	1.70	2.70	3.70	2.51	2.95	2.95	2.80	194	1/4
8	1700	0.35	1.30	2.4	3.3	2.80	3.24	2.67	2.90	185	1/4
8	1810	0.35	1.25	2.1	3.1	2.51	2.51	2.95	2.66	175	1/4
9	1840	0.35	1.00	1.75	2.2	1.92	2.21	1.33	1.82	157	1/4
9	1930	0.28	0.90	1.60	2.0	1.77	2.07	1.18	1.67	150	1/4
10	1445	0.10	0.40	0.70	1.0	0.89	0.89	0.89	0.89	108	3/16
10	1840	0.20	0.95	1.6-	2.2	2.21	1.92	1.77	1.97	166	3/16
10	2000	0.95	1.90	2.8	3.65	2.80	2.66	2.51	2.70	192	3/16
11	1946	0.10	0.4	0.7	1.0	0.89	0.89	0.89	0.89	108	3/16
11	1700	0.2	0.9	1.6	2.2	2.06	2.06	1.77	1.96	166	3/16

FIGURE VII.

AVERAGE DRY AND WET PLATE PRESSURE DROP

FOR $\frac{1}{4}$, $\frac{3}{16}$ INCH DIAMETER HOLES

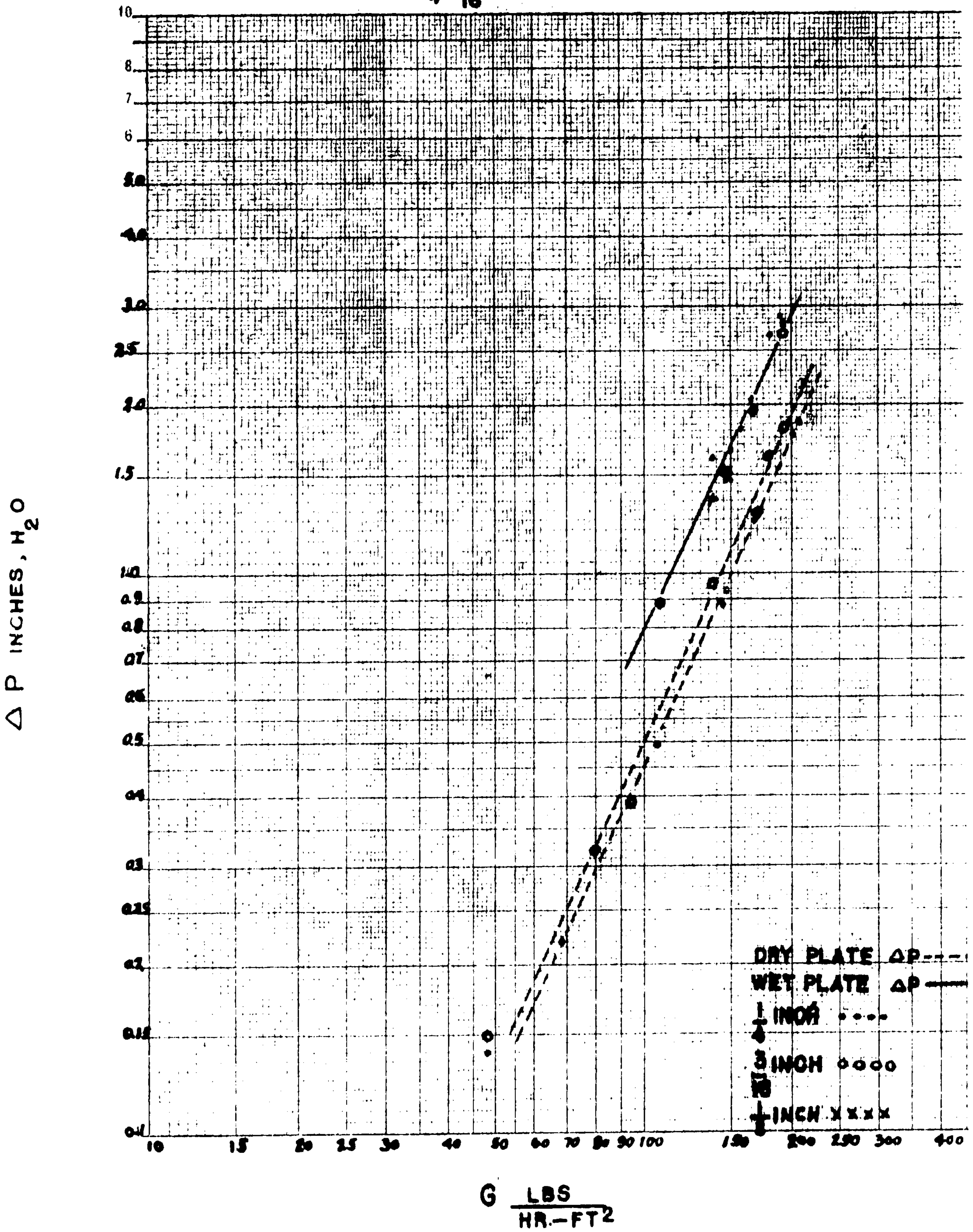


TABLE III

Murphree Plate Efficiency - Summary of Distillations Runs for 1/8, 3/16, 1/4 Hole Diameter Plate Series

				Murphree Plate Efficiency Percent		
Run No.	Time	G $\frac{\text{lbs}}{\text{hr-ft}^2}$	Hole Diameter	Plate 1	Plate 2	Plate 3
6	1400	149	1/8	126.0	89.6	99.0
6	1530	137	1/8	108.5	88.0	94.6
6	1730	137	1/8	155.0	81.6	94.3
7	1545	137	1/4	168.0	82.9	70.9
7	1715	137	1/4	156.0	94.3	86.2
7	1950	165	1/4	125.0	89.4	41.4
7	2015	188	1/4	184.0	100.0	42.5
8	1700	185	1/4	117.0	89.7	55.3
8	1810	175	1/4	117.0	93.6	54.9
9	1230	ca. 50	1/4	131.0	79.4	79.1
9	1330	ca. 50	1/4	139.0	122.0	95.5
9	1820	157	1/4			49.8
9	1930	150	1/4	97.2	95.7	40.1
10	1630	99	3/16	105.5	92.4	86.6
10	1945	166	3/16	129.0	97.3	78.5
11	1730	166	3/16	118.0	92.5	40.5
11	1945	99	3/16	89.6	97.5	78.0

DISCUSSION OF RESULTS

The calculated results of this investigation are presented in Table I to Table III. A graphical presentation of dry and wet plate pressure drop as a function of mass flow rate are presented in Figure VII.

Average dry plate pressure drop data for the 1/4 inch diameter holes as calculated by the method of least squares resulted in the following equation, $P = .0000838 G^{1.873}$. Average deviation of the experimental data from this equation was 3.13 percent. An increase in pressure drop with decreasing hole diameter, as shown by the graphical presentation of data was to be expected as the equation is essentially the standard orifice equation, namely $V_o = C(2g H)^{1/2}$.

Analysis of the average wet plate pressure drop data show little observed change in pressure drop with variable hole diameter in the narrow range in which data were obtained. The equation, $P = R(P + S)$(1) for predicting total plate pressure drop gave results which deviated approximately 5 percent from the data obtained in this report. The empirical constant R used in this equation takes into account the effect of aeration and height of liquid above the plate.

Results of the distillation runs show no observed effect of hole diameter or mass flow rate on Murphree plate efficiency for plate 1 and 2 within the range of data obtained in this study. However, for the bottom plate, 3, mass flow rate constant, Murphree plate efficiency increased from 44 percent for the 1/4 inch hole diameter plate to 96 percent for the 1/8 inch hole diameter plate indicating higher plate efficiency for smaller hole diameter plate perforations. In addition, Murphree plate efficiency for all distillation runs increased from the bottom plate to the top plate of the column.

The data obtained in this study on the methanol-water distillation run can be explained using the gas film and liquid film plate efficiency concept of mass transfer as outlined by Gerster et al$^{(3)}$.

According to the data of Gerster et al$^{(3)}$ the gas vapor rate had very little effect on the N_G, the number of gas transfer units as a result of the other counteracting variables involved, namely the degree of "aeration" of the liquid. The effect of vapor rate, N_G, is readily explained as follows: an increase of gas flow increases the degree of "aeration" and as a result increases the contact time between the gas and liquid, in addition to increasing the interfacial area available for mass transfer. These phenomena are counteracted by the decreased time available for mass transfer as a result of decreased contact time between gas and liquid.

Gerster et al has also pointed out the increase in the gas film resistance with increasing methanol concentration. At a liquid plate concentration of 90 mole percent methanol, 90 percent of the resistance to mass transfer is in the gas film, and at 10 mole percent methanol only 55 percent of the resistance is in the gas film. This increasing effect of liquid film resistance with decreasing plate concentration indicates the importance of considering plate design variables affecting liquid film resistance in distillation runs where low plate concentrations are involved. Data as obtained by Gerster et al also shows a decrease in Murphree plate efficiency with increase in plate concentration.

A plate design variable which does show effect on Murphree efficiency as indicated by results obtained in this study is plate hole size. A decrease in hole size, 1/4" to 1/8" diameter gave higher Murphree plate efficiencies for the bottom plate which is what would be expected as a result of the increase in the degree of "aeration". This same increase in plate efficiency was not readily observed in the top plate as a result of the higher gas film resistance due to the higher concentration of methanol on the plate. In other words, the increase in the degree of aeration as a result of the smaller hole size was counteracted by the higher gas film resistance, hence no observed increase in Murphree plate efficiency was obtained.

In the lower perforated plate, 3, the decrease in plate concentration did lower the Murphree plate efficiency as is indicated by data in all distillation runs. However, the effect of the increased hole size as a result of changing the froth density above the plate was the controlling factor and resulted in decrease in plate efficiency, more than was obtained at constant hole diameter.

LIMITATIONS

The limitations of this experimental study are summarized as follows:

1. Accuracy of pressure drop data ± 0.5 inches of water.
2. Comparison of percent methanol obtained from refractive index data (using the refractometer) with density measurement (Westphal balance) showed a deviation of 0.91%.
3. Refractometer readings differed in the majority of readings in the fourth decimal plate ± 1. This could result in an error of 0.5 percent methanol by weight.

RECOMMENDATIONS

Insofar as the range of study was limited by the steam supply available, it is suggested that additional data be obtained at higher mass flow rates.

Calculation of the heat transfer coefficient for the distillation kettle used in this study indicate that a mass flow rate of 700 lb/hr-ft^2 is capable of being obtained, (U_{calc} = 147). To efficiently handle this vapor rate a new condenser would need to be designed as the present condenser was designed to handle a vapor rate of ca. 300 lb/hr-ft^2.

Construction of a new thermocouple housing on the plate holders is suggested as the sauerisen used was easily chipped when the distillation column was dismantled.

APPENDIX A. Nomenclature

G = Mass flow rate: lbs/hr-ft^2

E_m = Murphree plate efficiency

y_1 = Mol fraction vapor leaving plate 1

y_2 = Mol fraction vapor leaving plate 2

y_3 = Mol fraction vapor leaving plate 3

y_4 = Mol fraction vapor leaving plate 4

x_1 = Mol fraction liquid leaving plate 1

x_2 = Mol fraction liquid leaving plate 2

x_3 = Mol fraction liquid leaving plate 3

y_n^* = Mol fraction of vapor in equilibrium with liquid leaving plate n, where n is the plate number starting from the top of the column.

P/D = Pitch/diameter ratio. Ratio of the length of one side of an equilateral triangle to the hole diameter.

P = Pressure drop, inches, H_2O

$$\text{Percent Free Space Area} = \frac{\text{Perforation area}}{\text{Column cross section area}}(100)$$

Thermocouple designations:

1. = water exit overhead condenser
2. = reflux entering top of column
3. = reference thermocouple maintained at 0°C
4. = vapor -- plate 1
5. = liquid -- plate 2
6. = vapor -- plate 2
7. = liquid -- plate 2

8. = vapor -- plate 3

9. = liquid -- plate 3

10. = vapor -- plate 4

11. = distillate exit overhead condenser

APPENDIX B. Summary of Design Information for the Perforated Distillation Column

Column diameter, I.D., inches	8.25
Distance between perforated plates, inches	9.50
Number of perforated plates	3
Column cross section area, ft^2	0.3718
Maximum perforated plate area/plate, ft^2	0.216
<u>1/8 inch hole diameter series</u>:	
Number of holes/plate	51
Distance between hole centers, inches	0.475
Hole arrangement	6,7,8,9,8,7,6
<u>3/16 inch hole diameter series</u>:	
Number of holes/plate	24
Distance between hole centers, inches	0.712
Hole arrangement	4,5,6,5,4
<u>1/4 inch hole diameter series</u>:	
Number of holes/plate	13
Distance between hole centers, inches	0.950
Hole arrangement	4,5,4

APPENDIX C. Murphree Plate Efficiency Sample Calculation

Run VIII, Plate 2, time 1730:

$$E_m = \frac{y_2 - y_3}{y_2^* - y_3}(100) \qquad = \frac{0.846 - 0.640}{0.870 - 0.640}(100) = 89.7\%$$

$$y_2 = \frac{\frac{90.7}{32}}{\frac{90.7}{32} + \frac{9.3}{18}} = 0.846$$

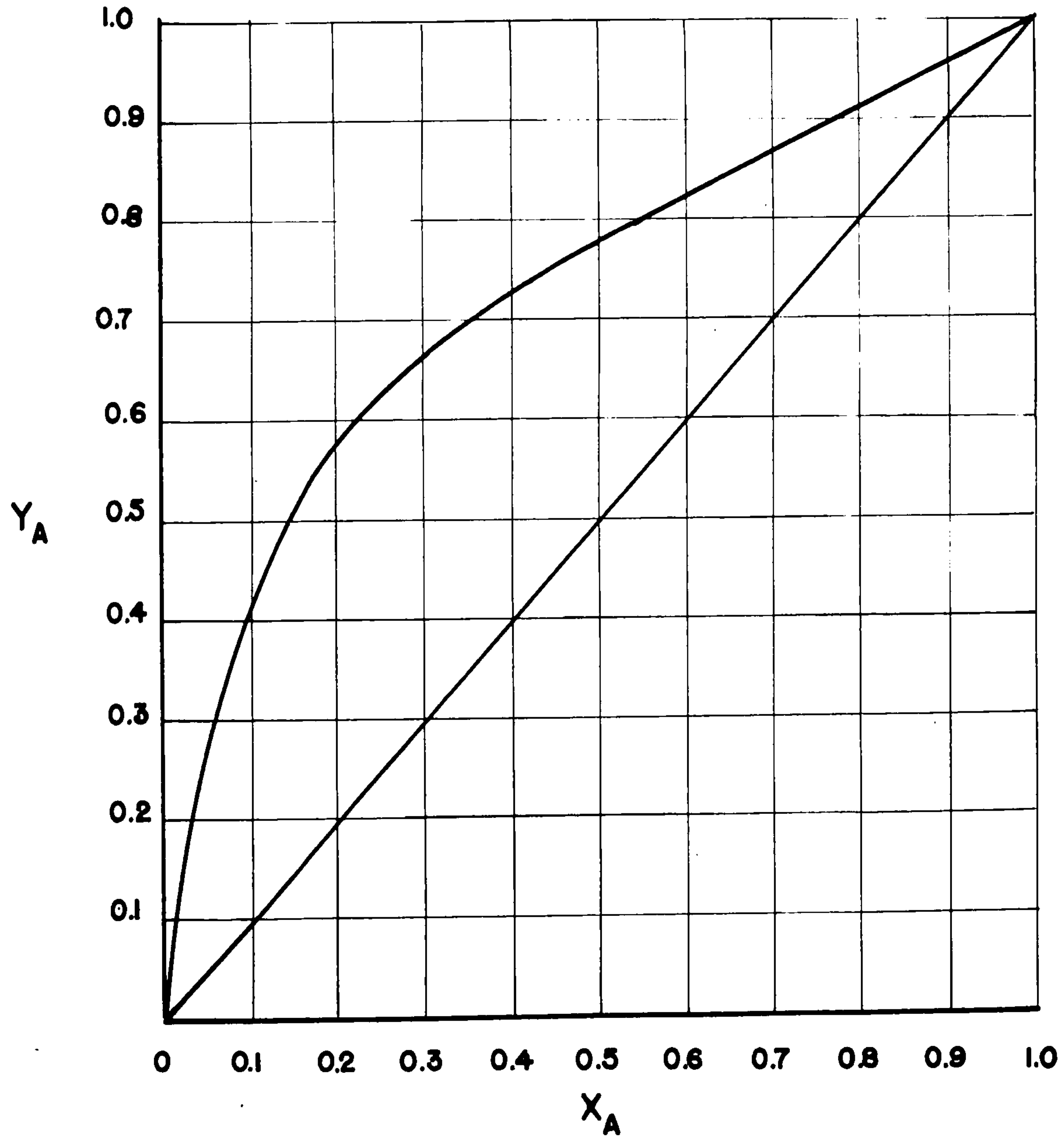
$$y_3 = \frac{\frac{76.0}{32}}{\frac{76.0}{32} + \frac{24.0}{18}} = 0.640$$

$$y_2^* = \frac{\frac{80.6}{32}}{\frac{80.6}{32} + \frac{13.35}{18}} = 0.700$$

From equilibrium curve pg a value of 0.870 is obtained

EQUILIBRIUM CURVE#

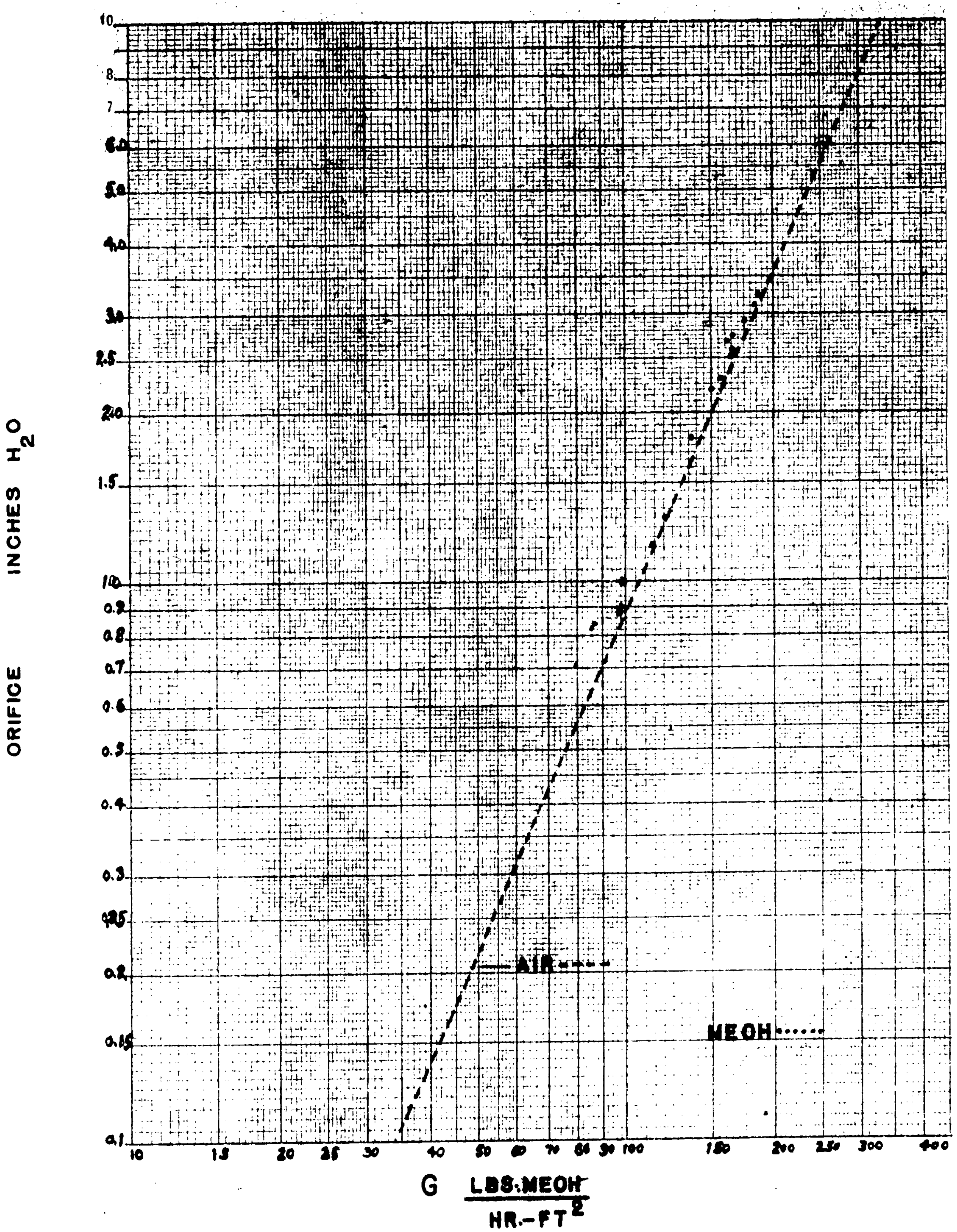
MEOH — H_2O



\# JU CHIN CHU
DATA
1 ATM.

FIGURE VIII.

E. ORIFICE CURVE



APPENDIX F. TABLE IV

RUN IV DATA

Time	Temperature °F Thermocouple Designation										Pressure Plate In. Red Oil				Orifice In. Red Oil	Kettle Jacket (steam) psig	Roto Rdg.	
	1	2	4	5	6	7	8	9	10	11	1	2	3	4			Reflux	Condenser
1500	Steam to kettle																	
1545	117.0	113.8	155.4	171.1	135.5	171.9	177.5	177.5	177.5	102.3	0.1	0.45	0.9	1.3	0.25	10	-	79.5
1645	118.4	145.9	149.8	149.6	151.8	151.8	-	173.4	154.4	95.2	0.1	0.35	0.7	1.0	0.28	5	2.8	54.0
1730	120.0	145.9	148.8	148.8	151.8	151.8	-	173.8	157.2	101.3	0.1	0.30	0.65	1.0	0.25	5	2.85	51.0
1815	111.8	135.5	145.5	145.5	150.6	150.3	-	173.8	156.4	90.0	0.1	0.30	0.65	1.0	0.24	5	2.50	52.0

RUN IV. SAMPLE DATA

TIME	SAMPLE DESIG.	N_D 25°C	DENSITY 20°C	% MEOH N_D	% MEOH DENSITY	MOL FRACT MEOH N_D
1700	Dist	1.3280	0.795	99.5	98.90	
1735	x-3	1.3383	0.870	74.00	70.50	0.615
1800	x-2-L	1.3312	0.817	93.75	91.20	0.894
1810	x-1	1.3285	0.799	98.65	97.50	0.977
1815	Dist	1.3270		100.00		
1830	y-4	1.3387		72.50		0.597
1837	y-3	1.3320		92.25		0.870
1842	y-2	1.3291		97.50		0.949
	y-1			99.7		0.901

RUN V. DATA

Kettle Charge
Hole Diameter
1.17% Free Space Area

Time	Temperature °F Thermocouple Designation 1	2	4	5	6	7	8	9	10	11	Pressure Plate In. Red Oil 1	2	3	4	Orifice In. H_2O	Kettle Jacket (steam) psig	Roto Rdgs. Reflux	Condenser
1235	Steam to kettle																	
1400	118.4	136.4	144.2	144.2	144.2	147.6	-	172.6	153.5	84.5	0.1	0.30	0.60	1.0	-	10	2.5	54
1545	93.3	135.5	148.0	148.0	151.0	149.9	-	181.5	153.9	118.9	0.3	1.0	1.40	1.8	-	30	7.7	
1600	84.9	132.1	147.2	153.4	152.5	151.4	-	181.8	156.0	98.2	0.3	0.8	1.4	1.85	-	30	7.6	
1715	86.7	126.9	149.2	146.4	152.2	151.0	-	184.8	156.8	96.5	0.3	0.8	1.4	1.85	-	30	7.0	
1805	Steam off																	

RUN V. SAMPLE DATA

TIME	SAMPLE DESIG.	N_D 25°C	% MEOH	MOL FRACT. MEOH
1410	x-4	1.3390	71.30	0.583
	x-3	1.3380	75.20	0.630
	x-2-L	1.3319	92.50	0.874
	x-1	1.3285	98.65	0.979
	y-4	1.3386	72.35	0.596
		1.3392	70.60	0.596
	y-3	1.3320	92.25	0.887
	y-2	1.3283	99.00	0.982
	y-1		90	0.982
1600	x-4			
	x-3	1.3392	70.50	0.573
	x-2	1.3350	86.40	0.783
	x-1	1.3300	96.10	0.975
	y-4	1.3395	69.40	0.561
	y-3	1.3340	88.53	0.870
	y-2	1.3290	97.70	0.958
	y-1	1.3279	99.70	0.994
	Dist.	1.3275	100.00	1.000
1735	x-4			
	x-3	1.3392	70.5	0.562
	x-2-L	1.3335	89.65	0.830
	x-1	1.3308	94.45	0.907
	y-4	1.3395	69.40	0.561
	y-3	1.3348	86.80	0.786
	y-2	1.3286	98.40	0.974
	y-1	1.3279	99.70	0.994
	Dist.	1.3272	100.00	1.000

RUN VI. DATA

Kettle Charge - Mol Fraction

Time	Temperature °F Thermocouple Designation										Pressure Plate In. Red Oil				Orifice In. H_2O	Kettle Jacket (steam) psig	Roto Rdg.	
	1	2	4	5	6	7	8	9	10	11	1	2	3	4			Reflux	Condenser
1200	Steam to kettle																	
1400	100.8	148.0	148.4	148.4	152.6	150.8	-	179.9	157.2	130.5	0.3	0.8	1.4	1.8	-	15	7.0	
1530	102.3	147.2	148.8	148.8	151.8	149.5	-	178.0	156.8	127.3	0.3	0.8	1.35	1.7	-	15	6.1	
1645	106.4	148.0	149.5	149.5	153.9	150.3	-	179.0	157.6	129.3	0.25	0.6	-	1.6	-	17	6.1	
1730	107.3	146.4	149.2	149.2	153.4	151.8	-	-	158.4	128.9	0.3	0.8	-	1.7	-	19	6.0	
1740	Steam off																	

RUN VI. SAMPLE DATA

TIME	SAMPLE DESIG.	N_D 25°C	% MEOH	MOL FRACT. MEOH
1445	x-4	1.3402	66.5	0.528
	x-3	1.3397	68.6	0.553
	x-2	1.3338	89.0	0.822
1455	x-1	1.3309	94.25	0.903
1500	Dist.	1.3285	98.6	
1507	y-4	1.3345	87.5	0.797
	y-3	1.3340	88.8	0.814
1515	y-2	1.3306	94.7	0.910
	y-1	1.3283	98.5	0.973
1610	x-4	1.3406	64.5	
	x-3	1.3400	67.5	
	x-2	1.3333	89.9	
1625	x-1	1.3306	94.8	
1628	Dist.	1.3290	97.2	
	y-4	1.3401	67.0	
	y-3	1.3350	86.4	
1640	y-2	1.3310	94.1	
1643	y-1	1.3289	98.0	
1655	x-4	1.3407	64.0	0.50
1658	x-3	1.3401	67.0	0.533
	x-2	1.3339	88.8	0.818
	x-1	1.3307	94.6	0.9085
1710	Dist.	1.3288	98.1	
1713	y-4	1.3402	66.7	0.533
1716	y-3	1.3350	86.4	0.778
	y-2	1.3310	94.1	0.902
1725	y-1	1.3279	99.6	0.992
1727	x-2	1.3339	89.0	
1730	Dist.	1.3286	98.4	

RUN VII. DATA

Kettle Charge Mol Fraction MEOH
1/4" Diameter Holes
1-17% Plate Free Space Area

Time	Temperature °F Thermocouple Designation										Pressure Plate In. Red Oil				Orifice In. H_2O	Kettle Jacket (steam) psig	Roto Rdg.	
	1	2	4	5	6	7	8	9	10	11	1	2	3	4			Re-flux	Condenser
1430	Steam to pot																	
1545	105.9	148.2	159.2	158.4	131.7	161.6	161.2	186.4	164.4	114.6	0.2	0.75	1.35	1.85	1.8	10.0	6.0	
1645	105.1	148.2	158.4	158.4	130.9	161.2	164.0	187.2	165.0	114.6	0.2	0.75	1.35	1.85	1.8	12.5	6.0	
1745	105.1	148.0	159.2	158.0	130.9	161.2	164.0	187.2	165.0	116.4	0.2	0.75	1.35	1.85	1.8	13.5	6.0	
1815	105.5	148.8	158.8	158.4	130.9	159.6	161.2	190.0	164.8	142.2	0.3	1.20	1.85	2.35	2.7	20.0	8.0	
1930																	8.0	
1940											0.85	1.70	2.7	3.70	3.3	30.0	9.7	
2015	99.0	149.2	161.2	159.6	110.3			193.5	166.4	139.4	1.50		2.3	3.50		35	9.3	

RUN VII. SAMPLE DATA

TIME	SAMPLE DESIG.	N_D 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1615	x-4	1.3401	67.0	0.533
1618	x-3	1.3392	70.5	0.574
1623	x-2-L	1.3331	90.25	
1625	x-1	1.3318	92.10	0.869
1628	Dist.	1.3289	97.80	
1630	x-2-R	1.3350	86.40	0.783
1635	y-4	1.3388	72.00	0.591
1638	y-3	1.3351	86.00	0.747
1640	y-2	1.3317	92.75	0.878
1643	y-1	1.3281	99.25	0.986
1645	Dist.	1.3286	98.40	
1717	x-4	1.3400	67.5	0.539
1720	x-3	1.3391	70.90	0.578
1722	x-2-L	1.3313	90.25	
1724	x-1	1.3313	93.50	0.889
1726	Dist.	1.3290	97.70	
1728	x-2-R	1.3350	86.40	0.783
1731	y-4	1.3385	73.25	0.608
1732	y-3	1.3350	86.40	0.783
1738	y-2	1.3311	93.90	0.898
1740	y-1	1.3285	98.60	0.979
1850	x-4	1.3411	61.4	0.474
1853	x-3	1.3380	75.2	0.630
1855	x-2-L	1.3339	88.75	0.816
1858	x-1	1.3319	92.40	0.874
1910	y-4	1.3392	70.50	0.573
1915	y-3	1.3379	76.00	0.640
1920	y-2	1.3324	91.50	0.859
1923	y-1	1.3288	98.00	0.967
1925	x-2-R	1.3359	83.50	0.74
1930	Dist.	1.3288	98.00	
2005	Dist.	1.3291	97.5	
2025	x-4	1.3409	62.75	0.486
2027	x-3	1.3402	66.5	0.528
2030	x-2-L	1.3353	85.5	0.767
2033	x-1	1.3330	90.5	0.845
2036	y-4	1.3405	65.1	0.513
2039	y-3	1.3380	75.2	0.630
2042	y-2	1.3310	94.05	0.899
2044	y-1	1.3292	97.40	0.956
2045	Dist.	1.3298	96.25	

RUN VIII.

Kettle Charge Mol Fraction MEOH
1/4" Diameter Holes
1.17% Plate Free Space Area

Time	Temperature °F Thermocouple Designation 1	2	4	5	6	7	8	9	10	11	Pressure Plate In. Red Oil 1	2	3	4	Orifice In. H_2O	Kettle Jacket (steam) psig	Roto Rdg. Reflux	Condenser
1500	Steam to pot																	
1700	94.7	148.4	154.4	156.4	132.9	161.0	156.0	188.8	163.6	130.5	0.35	1.30	2.4	3.3	3.1	35	9.1	
1810	94.2	148.4	158.0	156.0	-	160.0	169.0	190.8	163.2	120.5	0.35	1.25	2.1	3.1	2.9	40	8.4	
1900	Steam off																	

RUN VIII. SAMPLE DATA

TIME	SAMPLE DESIG.	N_0 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1730	Dist.	1.3296	96.6	
	x-4	1.3409	62.75	
	x-3	1.3401	67.00	
	x-2-L	1.3349	86.65	
	x-2-R	1.3366	80.60	
	x-1	1.3328	90.80	
	y-4	1.3404	65.60	
	y-3	1.3378	76.00	
	y-2	1.3329	90.70	
	y-1	1.3295	96.75	
	Dist.	1.3295	96.75	
1830	x-4	1.3410	62.2	0.482
1833	x-3	1.3400	67.5	0.540
1837	x-2-L	1.3350	86.2	0.776
1839	x-2-R	1.3368	79.4	0.684
1841	x-1	1.3330	90.5	0.844
1843	y-4	1.3404	65.60	0.518
1845	y-3	1.3372	78.25	0.670
1849	y-2	1.3328	90.8	0.848
1852	y-1		96.75	0.944
1855	Dist.	1.3300	95.8	

RUN IX.

Kettle Charge Mol Fraction MEOH
1/4" Diameter Holes
1.17% Plate Free Space Area

Time	Temperature °F Thermocouple Designation										Pressure Plate In. Red Oil				Orifice In. H_2O	Kettle Jacket (steam) psig	Roto Rdg	
	1	2	4	5	6	7	8	9	10	11	1	2	3	4			Reflux	Condenser
1130	Steam to pot																	
1230	131.7	147.9	160.4	158.4	135.9	162.4	163.6	190.8	164.8	101.3	0.2 -0.3	0.6 +0.1	1.0 +0.3	2.0 +0.5	0.52	10	1.5 2.5	48
1330	127.3	149.0	160.0	157.6	138.2	161.6	162.8	192.3	164.4	98.7					0.50	12	1.5 3.0	48
1615															1.10	21	4.5	69
1645	121.9	149.0	160.8	158.8	133.7	162.4	163.6	196.3	166.0	121.5	0.3	0.85	1.4	1.7	1.30	25	5.0	96
1800	114.6	148.6	160.8	158.8	-	162.4	-	199.1	166.4	125.5		0.75	1.2	1.6	1.30	27	5.0	97
1840											0.35	1.00	1.75	2.2	2.3	42	7.8	
1945											0.28	0.90	1.60	2.0	2.2	44	7.0	
1955	Steam off																	

RUN IX. SAMPLE DATA

TIME	SAMPLE DESIG.	N_D 25°C	% MEOH	MOL FRACT. MEOH
1330	Dist.	1.3294	97.0	
1335	x-4	1.3409	62.75	0.487
	x-3	1.3401	67.00	0.533
	x-2-L	1.3345	87.60	0.687
1425	x-2-R	1.3361	82.75	0.630
	x-1	1.3321	92.10	0.870
	y-4	1.3401	67.00	0.533
	y-3	1.3360	83.20	0.736
	y-2	1.3312	93.70	0.894
	y-1	1.3290	97.60	0.957
1500	Dist.	1.3290	97.6	
1505	x-4	1.3410	62.00	0.479
	x-3	1.3440	67.50	0.795
	x-2-L	1.3345	87.60	0.799
1517	x-2-R	1.3361	82.75	0.730
1525	x-1	1.3320	92.25	0.870
	y-4	1.3401	67.00	0.533
	y-3	1.3350	86.40	0.783
	y-2	1.3310	94.10	0.901
1546	y-1	1.3290	97.6	0.958
1548	Dist.	1.3290	97.6	
1715	x-4	1.3408	63.5	0.494
	x-3	1.3410	62.2	0.482
1721	x-2-L	1.3359	83.5	0.640
1728	x-2-R	1.3380	75.2	0.630
1730	x-1	1.3336	89.3	0.824
1739	y-4	1.3410	62.2	0.482
1743	y-3	1.3380	75.2	0.630
1747	y-2	1.3336	89.3	0.824
1749	Dist.	1.3300	95.8	
1758	y-1	1.3296	96.6	0.943
1820	x-3	1.3410	62.2	0.482
	x-2-L	1.3362	82.15	0.723
	x-2-R	1.3380	75.20	0.630
	x-1	1.338	89.00	0.820
1835	y-4	1.3410	62.20	0.482
	y-3	1.3381	74.80	0.625
1900	x-3	1.3412	60.50	0.464
1910	x-2-R	1.3390	71.3	0.583
1913	x-1	1.3348	86.8	0.786
1914	y-4	1.3410	62.2	0.482
1922	y-3	1.3397	68.65	0.553
	y-2	1.3344	87.75	0.801
1926) 1939)	Dist.	1.3308	94.20	0.902

RUN X.

Kettle charge
3/16" Diameter Holes
1.17% Plate Free Space Area

Time	Temperature °F Thermocouple Designation										Pressure Plate In. Red Oil				Orifice In. H_2O	Kettle Jacket (steam) psig	Roto Rdg.	
	1	2	4	5	6	7	8	9	10	11	1	2	3	4			Reflux	Condenser
1315	Steam to pot																	
1445	115.4	145.8	150.6	150.0	126.9	152.2	154.2	178.5	-	128.9	0.1	0.4	0.7	1.0	0.9	6.6	4.0	77
1650	113.0	148.1	150.3	149.6	126.4	151.8	155.2	179.9	-	121.9	0.1	0.4	0.7	1.0	0.9	8.0	4.0	76
1700																		
1815	98.0	146.8	151.4	150.0	127.3	153.4	159.6	184.0	-	129.7	0.6	1.45	2.1	2.75	2.5	23.0	8.0	
1845											0.2	0.95	1.6	2.2	2.43	23.5	8.0	
1945		148.4	151.4	150.0	128.0	152.6	153.0	187.6			0.2	0.95	1.6	2.2	2.5	26.0	8.0	
											0.95	1.90	2.8	3.65	2.7	38.0	9.6	
2030																	10.5	

RUN X. SAMPLE DATA

TIME	SAMPLE DESIG.	N_D 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1415	Dist.	1.3290	97.75	
1425	Dist.	1.3290	97.75	
1515	Dist.	1.3290	97.75	
1527		1.3290		
1536	x-4	1.3403	66.00	0.523
1538		1.3403		
1559	x-3	1.3392	70.50	0.583
		1.3392		
1601	x-2-L	1.3339	88.85	0.813
1604		1.3339		
1606	x-1	1.3311	93.85	0.895
1609		1.3311		
1613	y-4	1.3400	67.25	0.537
1616		1.3401		
1619	y-3	1.3352		
1621		1.3351	86.00	0.775
		1.3351		
1629	y-2	1.3310	94.10	0.899
		1.3311		
	y-1		97.75	0.958
1636	Dist.	1.3291	97.50	
	x-2-R		80.75	
1840	Dist.	1.3290	97.75	
1855	x-4	1.3410	62.00	0.523
1859		1.3410		
	x-3	1.3404	66.00	0.523
		1.3403		
1906	x-2-L	1.3349	86.65	0.778
		1.3349		
1912	x-1	1.3320	92.25	0.872
1915		1.3320		
1918	y-4	1.3410	62.00	0.480
		1.3410		
1933	y-3	1.3362	82.15	0.720
		1.3361		
1938	y-2	1.3321		
1945		1.3320	96.25	
		1.3321		
1950	Dist.	1.3298	96.25	
		1.3299		
	y-1	1.3285	98.00	0.968
		1.3288		
2005	Dist.	1.3299	96.25	
	x-2-R		80.90	0.704

RUN XI.

Kettle Charge
3/16" Hole Diameter
1.17% Free Space Area

Time	Temperature °F Thermocouple Designation										Pressure Plate In. Red Oil				Orifice In. H_2O	Kettle Jacket (steam) psig	Roto Rdg. Reflux	Roto Rdg. Condenser
	1	2	4	5	6	7	8	9	10	11	1	2	3	4				
1500	Steam to pot																	
1645	93	147.8	152.2	149.9	132.8	154.8	-	190.0	-	-	0.2	0.9	1.6	2.2	2.5	38	8.0	
1730											0.2	0.95	1.6	2.2	2.5	39	8.0	
1900	96.5	140.0	152.6	150.6	133.7	154.8	-	195.9	-	-	0.19	0.9	1.6	2.2	2.4	43	7.5	
1917																		
1945	118.4	147.0	153.4	151.8	135.0	156.8	-	195.9	-	-		0.1	0.4	0.7	1.0	24	4.0	
2130	Steam off																	

RUN XI. SAMPLE DATA

TIME	SAMPLE DESIG.	N_D 25°C	WEIGHT % MEOH	MOL FRACT. MEOH
1700	Dist.	1.3299	96.1	
1730	x-4	1.3405		
		1.3406	64.7	0.508
		1.3407		
1735	x-3	1.3408	63.3	0.492
1745		1.3409		
1753	x-2-L	1.3361		
1758		1.3361	82.5	
		1.3361		
1805	x-1	1.3332	90.2	0.837
1807		1.3332		
1809	x-2-R	1.3382	74.4	0.621
1815		1.3382		
1821	y-4	1.3409	62.75	0.487
1824		1.3409		
1827	y-3	1.3385	73.0	0.603
1830		1.3386		
1837	y-2	1.3340	88.6	0.815
		1.3340		
1847	Dist.	1.3304	95.1	
1900		1.3305		
1852	y-1	1.3292	97.1	0.95
1915		1.3294		
1946	Dist.	1.3305	95.0	
1940				
1944				
1955	Dist.	1.3305	95.0	
2014		1.3305		
	x-3	1.3405	65.0	0.512
		1.3405		
2025	x-2-L	1.3381	74.75	
		1.3380		
2040	x-2-R	1.3396	69.00	0.554
2045		1.3396		
2047	x-2-L	1.3381	74.75	
	x-1	1.3345	87.60	0.798
2055	y-4	1.3399	68.00	0.553
2057		1.3399		
2100	y-3	1.3396	69.00	0.554
2105		1.3396		
	y-2	1.3346	87.30	0.795
		1.3346		
2112	Dist.	1.3310	94.10	0.899
		1.3310		
	y-1		94.1	0.899

APPENDIX G. BILBIOGRAPHY

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