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A STUDY OF DEPHLEGMATOR PERFORMANCE

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A STUDY OF DEPHLEGMATOR PERFORMANCE

By

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PREFACE

Condensing of the vapor is one of the major items of consideration in a distillation process. It is customary, on large scale equipment, to have two or more condensers connected in series rather than one large one. Sufficient heat is abstracted in the first condenser to condense only part of the total vapor entering it, the remaining vapor being condensed in the second condenser. In most setups of this type, the condensate from the first condenser is returned to the column as reflux and the output of the second condenser is withdrawn as product. The first condenser has been variously called the "reflux condenser", "partial condenser", or "dephlegmator".

If a mixture of vapors be partially condensed, relatively less of the lower boiling point components is condensed than those of higher boiling point. That is, the vapor passing out will be richer in more volatile components than the condensate returned as reflux. This is fractionating action, but the mechanism is different, except in special cases, from that which takes place in the column.

Although partial condensers are used quite widely in distillation processes, their main function, in recent years, has been to condense the vapor. Any fractionating action which occurs in the reflux condenser is quite incidental. This is due probably to the small amount of fractionation which occurs in this condenser in comparison with that which takes place in the column of the still. Also due to the uncertainty in predicting the performance of a partial condenser as a fractionating device from its design data, no reliance whatsoever is placed on the condenser for such function,

and any fractionation which does occur is looked upon as a factor of safety.

It would be of value, however, to have some basis for determining beforehand what the performance of a dephlegmator would be. It is the purpose of this investigation to study the effects of several variables on such performance in connection with a dephlegmator of engineering laboratory scale.

MEMPHIS ARCHIVE

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INTRODUCTION

Considerable controversy has existed over the value of dephlegmators as fractionating devices. Hausbrand (2) uses the term "reflux condenser", stating that the term "dephlegmator" implies separation, which is not its function, the actual function being to condense some of the vapor and to return it to the column as reflux. Young (13) places the function of fractionation, or separation on nearly equal importance with that of providing reflux. He states that in many plants the function of separation is far more important. Carey (7) states that the use of a partial condenser as part of a fractionating system is less efficient than when all the fractionation takes place in the column. The exact significance of the term "efficient" in this case is not stated. Robinson and Gilliland (9) state that partial condensers are widely used, especially in cases where the vapor is difficult to completely condense, that is, if very low boiling components are present as well as those of higher boiling point.

Hausbrand (2) raises the question of the possibility of separating the low boiling components of a mixture by condensation in a single stage and concludes that

"the separation of liquids by continuous, gradual condensation is, up to a certain point, much less efficient than separation by the use of a good rectifying column in which the liquids are repeatedly brought to boiling temperature".

Some early investigators believed that it would be possible to effect complete separation of two components by passing the mixed vapor through a chamber maintained at the boiling point of the lower component. This fallacy is explained by Hausbrand (2) and in present

methods of treating distillation problems is not encountered.

The degree of separation of two components effected in a partial condenser is usually referred to as the efficiency of the partial condenser. The first expression of partial condenser efficiency as an equivalent to a fraction of a theoretical plate was by Underwood (10), stating that this is the only logical basis. One theoretical plate produces vapor in equilibrium with the liquid on that plate. The vapor leaving a partial condenser may possibly be either richer or poorer in the more volatile component than that called for by equilibrium conditions, making the efficiency greater or less, respectively, than one theoretical plate. Hausbrand implies that greater than one theoretical plate separation is not obtained in the reflux condenser by stating that the weakest (lowest in more volatile component) reflux is not weaker than the liquid on the top plate of the column. The fact is brought out by Underwood that simple (equilibrium) condensation produces performance equal to that of one theoretical plate.

It has been found with all ordinary mixtures that the degree of enrichment in a dephlegmator is less if the entering vapor contains the more volatile component in large percent, than if the two components are present in nearly equal quantities. Young, Hausbrand, and others have made this observation. The x-y equilibrium diagrams immediately furnishes an explanation for this in that near the center of the diagram, the equilibrium curve is farther above the 45 degree line than it is near the end. Thus a theoretical plate will produce a greater enrichment near the center of the diagram than the same plate near the end, where operating line and equilibrium curve

are close together. A dephlegmator might reasonably be expected to perform similarly and such has been observed as stated above. It has not been definitely known whether or not the efficiency in terms of theoretical plate equivalent is the same throughout the range.

It is conceivable that the mechanism of condensation can be differential, that is, as soon as any condensate is formed, the liquid is immediately removed from further interaction with the remaining vapor. Under most conditions, this means that the liquid is to be removed from actual contact with the vapor. However, if the liquid could be cooled at once to a low temperature, the rate of diffusion, and consequently that of interaction would be reduced, resulting in a more near approach to the condition of equilibrium condensation. Under such conditions, a dephlegmator can possibly produce separation equivalent to that of more than one theoretical plate. The mathematical expression for this type of condensation is

$$\ln \frac{W}{W_0} = - \int_{y_0}^y \left(\frac{dy}{y - x} \right)$$

where W is the weight of vapor, x the composition of liquid and y the composition of the vapor, and the subscript 0 refers to initial conditions. This is quite similar to the Rayleigh equation for differential vaporization, which is really an exactly analogous process in the reverse direction. Underwood (10) cites Kirschbaum's results (3) of laboratory experiments in which results agree with the above equation for differential condensation. It is apparent, however, that conditions approaching this type of condensation are not readily achieved except with apparatus of special construction.

The most usual condenser for distillation application consists of a bundle of tubes in a shell, so arranged that the cooling medium can pass through the tubes, the vapor condensing on the outside of the tubes. The flow of liquid through the tube bundle may be divided into two or more passes, which allows the overall length of the condenser to be kept within limits and still have efficient heat transfer. Baffles are sometimes placed in the vapor space so that the entire tube surface available is utilized most efficiently. The condenser can be placed so that the tubes are either horizontal or vertical, the latter position being more generally used except in very large installations. In some cases the cooling medium is passed outside the tubes, and the vapor condenses inside. In these instances the tubes must be placed vertically in order that the condensate will drain from them. One of these condensers of simple construction is now generally used as a partial condenser where such operation is maintained.

In the past, numerous dephlegmators of unusual design have appeared, the main object being to increase the separation efficiency. In many cases more effort and stress were placed on the dephlegmator than on the still column. The purpose of most of these special condensers was to bring the condensate and remaining vapor into more intimate contact so that a nearer approach to equilibrium could be realized, with resulting better efficiency. Attempts have been made to produce differential condensation performance, but this is more difficult to approach. Some of the pieces of equipment which have been called dephlegmators really function similarly to a part of the

column, the vapor bubbling through a pool of condensate as in a bubble plate. The modern trend has been toward condensers of simple construction because it is found to be more economical to build a few more plates on top of the column than to construct a complicated dephlegmator that will not effect any greater separation.

Young (13) gives the following specifications for most effective separation in a partial condenser: (1) rate of vapor flow through the condenser should be as low as possible, (2) temperature difference between cooling liquid and vapor should be as small as possible, (3) vapor should rise and cooling liquid should fall in the condenser, (4) a large proportion of the vapor must be condensed, (5) the condensate should be kept in as small drops as possible. Hausbrand's views (2) on the proper design of a reflux condenser are in conflict with the above on several points. He maintains that the temperature of the cooling agent in the condenser is unimportant so long as it is below the boiling point of the vapor passing through. He implies further that the cooling liquid is always to enter at the bottom, stating that experiments in which the cooling liquid was led in at the top were unsuccessful in every case. Just what was meant by "unsuccessful" was not stated. Underwood (10) states that the difference in results between laboratory and industrial scale apparatus is due to differences in cooling water temperature and rate of vapor flow through the condenser. The warmer cooling medium used in industry would tend to allow more interaction between condensate and remaining vapor, as previously explained, and the generally higher vapor velocities used industrially would have the same effect. Higher vapor velocity might tend to promote better interaction by the

resulting increased convection, but the reduced time of contact due to the greater rate of flow would tend to nullify any effect in that direction.

Data on the performance of dephlegmators has not appeared in large quantities. Underwood reports work of Dehnicke (1) on a laboratory scale partial condenser for which percentages of one theoretical plate obtained were 77, 71, and 66 using ethyl alcohol and water as the binary mixture. Kirschbaum (3) gives data on a condenser which are similar to those predicted by differential condensation, but the condenser in question was of somewhat complex design and the separation of components was attributed by Underwood to be due not entirely to the condensation. Webber and Bridger (12) give data on a dephlegmator giving 48, 60 and 65 percent performance of a theoretical plate on a methanol-water mixture. This dephlegmator consisted of a flat coil through which water was circulated above the top plate of the column and would not be expected to effect a great amount of separation because of its design.

EXPERIMENTAL

The binary mixture used in this investigation was methanol and water. The methanol was the synthetic product manufactured by Carbide and Carbon Chemicals Corporation and checked better than 99% pure by density measurement. The methanol was diluted to the desired strength with distilled water. This mixture was chosen because of ease of analysis, the fact that no azeotropes are formed, and a fairly large enrichment can be obtained by one theoretical plate or its equivalent.

A diagram of the apparatus used is shown in figure 1. It consisted mainly in the pot and condensers of a still manufactured by the Ansonia Company. The pot was copper lined and had a capacity of thirteen gallons. Heat was supplied by steam passing into a flat spiral coil near the bottom of the pot. The two condensers, one used as dephlegmator, and the other as final condenser were identical and had the dimensions shown in figure 2. They were of copper construction. Cooling water flowed through a single pass bundle of 56 $\frac{1}{4}$ " inside diameter tubes; the vapor condensed on the outside of the tubes. The condensers were arranged so that the vapor from the pot entered at the bottom of the dephlegmator, any uncondensed vapor left at the top of the dephlegmator and entered the top of the final condenser. Cooling water for the final condenser was taken directly from the city mains and discarded after flowing through the final condenser. Cooling water for the dephlegmator flowed from a fifty gallon reservoir mounted above the apparatus. This system of supply was used so that the water could be heated to a predetermined temp-

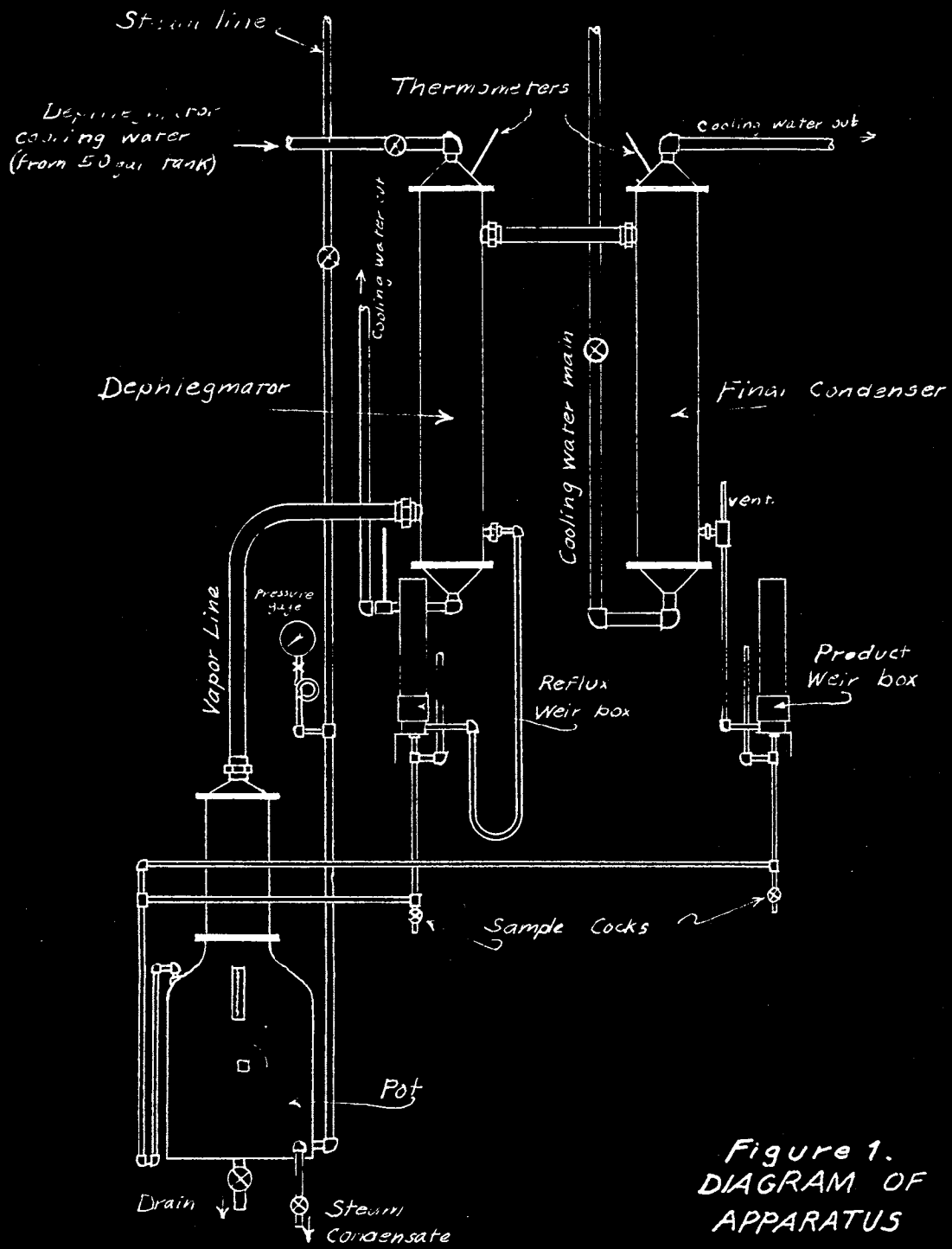


Figure 1.
DIAGRAM OF
APPARATUS

Approx. scale $\frac{3}{4}'' = 1'$

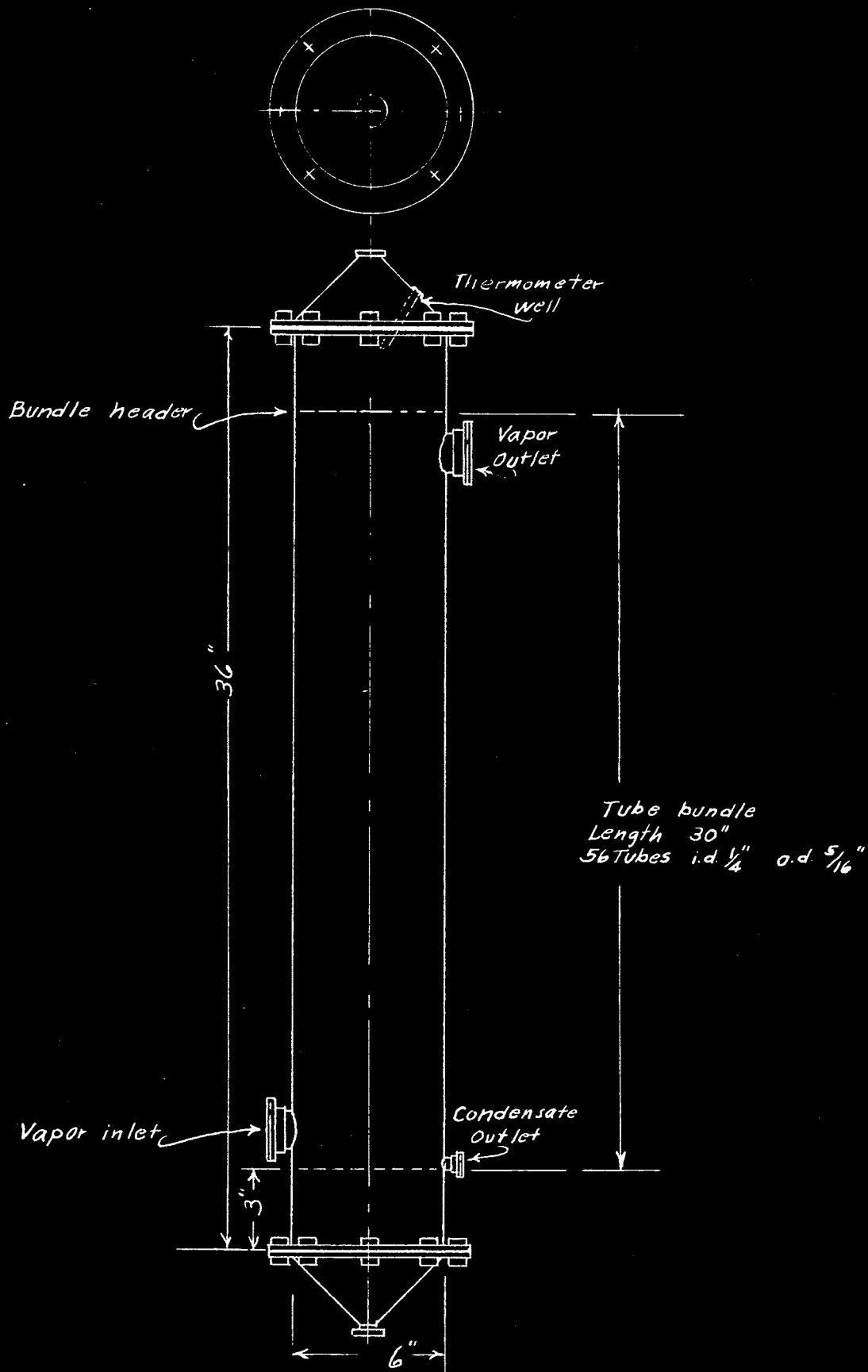


FIGURE 2. DEPHLEGMATOR

erature before being passed through the dephlegmator. Most of the experimental runs were made with the water entering the top of the dephlegmator, thus being countercurrent to the vapor flow. No particular precautions were taken to clean the condenser tubes, but a preliminary examination showed that although not bright, they were smooth and free of scale or encrustation.

Condensate from each condenser passed through a measuring weir. The construction of the weir boxes was as detailed in figure 3. They were made of readily obtainable pipe fittings and scrap materials. The flow was through a circular orifice discharging vertically. The size of the orifice was such that the rate of flow which could be produced in the still could be conveniently measured. The orifices were calibrated by measurement of time required for a given volume of water to flow through them at various heads. The height of liquid head was measured from the upper surface of the plate in which the orifice was drilled. Several check runs were made using methanol and substantially the same volumetric calibration obtained, so the calibrations for pure water were used for all the concentrations which were measured. Calibration curves for the orifices are given in figure 4. In a few cases, when the rate of flow was insufficient to obtain a reliable reading from the weir box, the rate was determined directly by measuring the time required for the delivery of 50 ml. of the distillate into a graduated cylinder.

The apparatus was arranged so that the condensates from both dephlegmator and final condenser could flow back into the pot, so that equilibrium could be established in operation. It was found

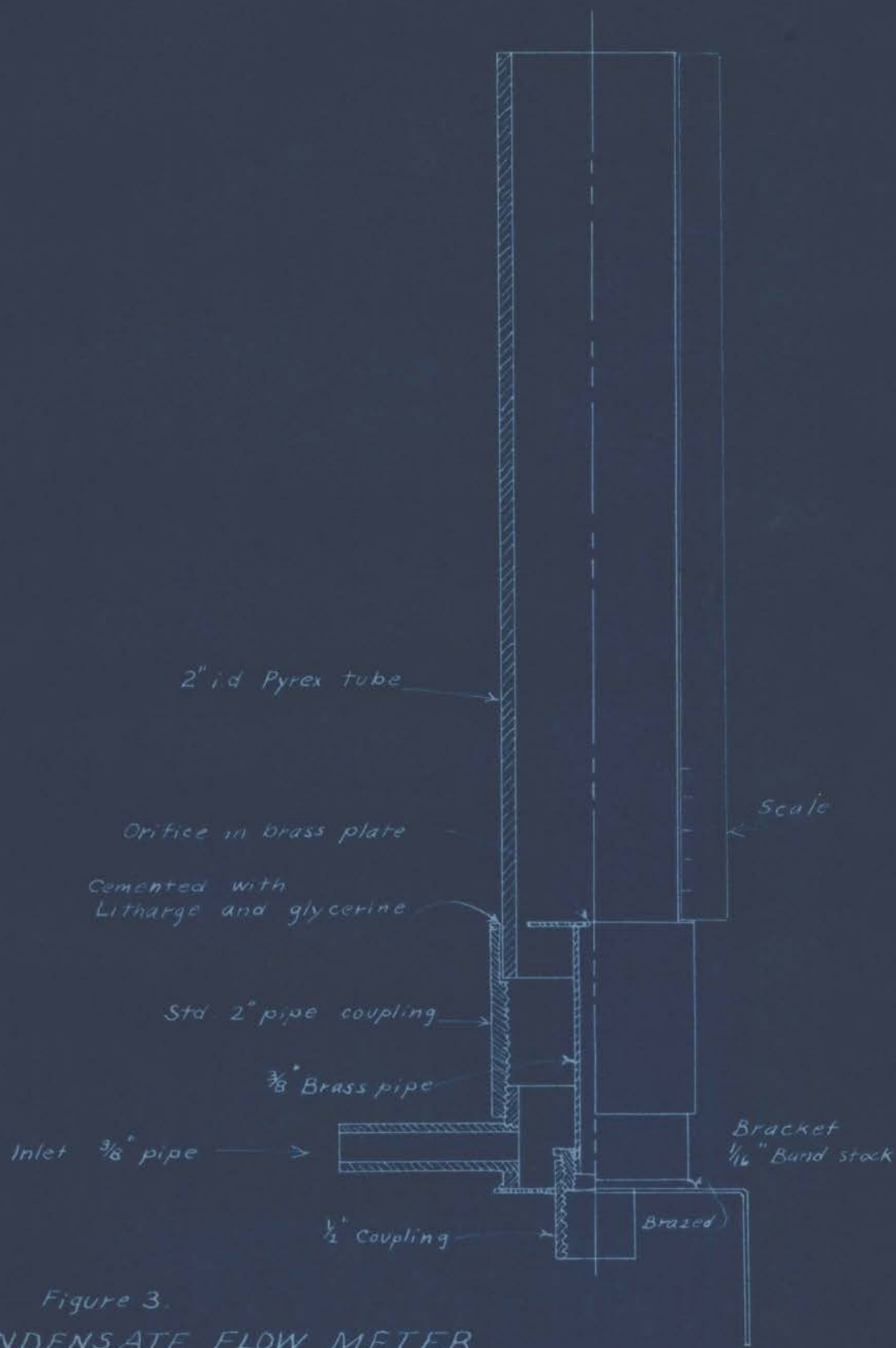


Figure 3.
CONDENSATE FLOW METER
(WEIR BOX)

half size

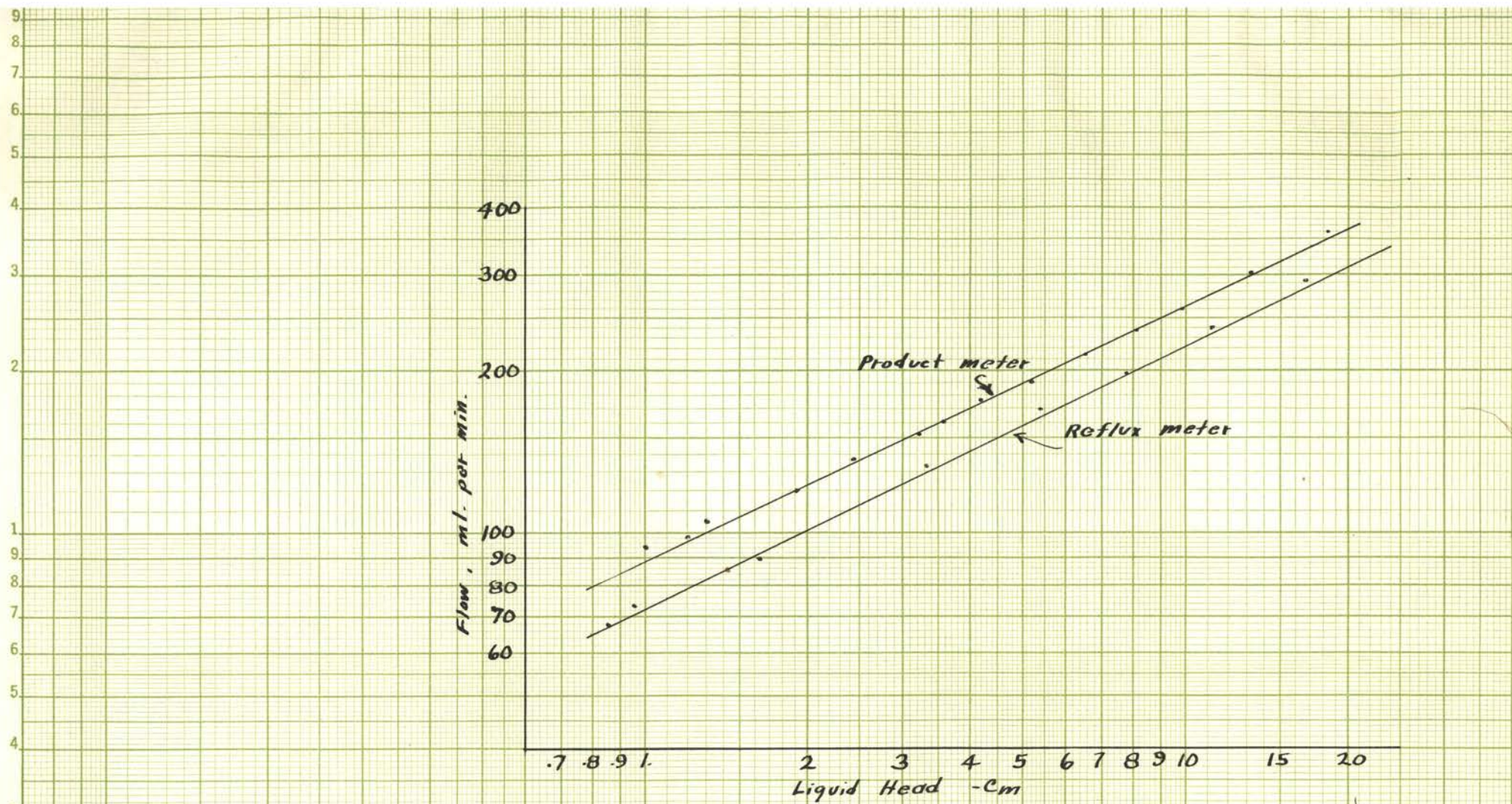


Figure 4.
ORIFICE CALIBRATION CURVES

that equilibrium was reached after about fifteen minutes. Arrangement was made to withdraw, at the proper time, samples of both reflux (partial condensate) and product for analysis. The amount of each sample required was 50 ml. After withdrawing a sample, the container was immediately stoppered and placed in a constant temperature bath.

Analyses for percent of methanol in the samples were made by specific gravity determinations. A standard analytical balance slightly modified for this type of measurement was used for all determinations. Specific gravities were obtained at 15° C., referred to water at 15° C., and converted to percent methanol by tables presented by Olsen (6) from values of Doroshevskii and Rozhdestvenskii.

Four different concentrations of methanol were used. About two gallons of solution was placed in the pot. A number of runs were made with each concentration, varying other conditions one at a time, in so far as possible. The fraction of vapor condensed in the dephlegmator was varied as widely as the measuring methods would permit. The fraction condensed was controlled by the rate of flow of cooling water through the dephlegmator. Cooling water entered the dephlegmator at a temperature between 30° and 40° in all the runs. The water had previously been heated in the fifty gallon reservoir by open steam. By the use of warm water for the dephlegmator, control of the amount of vapor condensed was much more precise and reliable than would have been possible with cold water. Also this is the condition which exists in many commercial dephlegmators, the water having previously been passed through another piece of equipment, usually the final condenser. According to Young's second

specification (p. 5) this should produce better fractionating action than the use of cold water.

Rate of vapor flow passing into the dephlegmator was varied several fold with one of the concentrations used.

A few runs were made with the cooling water entering at the bottom, making the flow parallel to that of the vapor in those cases.

Steam for vaporization of the mixture was condensed at atmospheric pressure in the heating coil. This temperature provided a sufficiently large rate of heat transfer for most of the runs. The temperatures of the boiling liquids ranged from about 70° C. to 80° C. depending on the percent of methanol. Precise regulation of the rate of steam flow was maintained by a standard one-half inch globe valve with an extension handle in the form of a lever about eight inches long.

In the cases during which cooling water was passed downward through the dephlegmator, precaution was taken to remove all entrapped air through a vent at the top of the condenser.

DATA AND CALCULATIONS

Rates of flow of the liquid from the two condensers as read from the calibration curves were converted to mass rate in grams per minute using the densities measured. The difference in density between the temperatures of 0° C. and 15° C. was neglected. Specific gravity was recorded to four decimal places, which was sufficiently accurate to determine the composition to well within 0.1% by weight.

Molecular weight of the methanol used throughout the calculations is a fictitious value, which as will be explained later must be used to give equal molal latent heats of vaporization for both the components of the mixture.¹ The molecular weight of water used is its true value, 18.02, and the value to use for methanol is calculated as follows: The average latent heat of vaporization of methanol between its boiling point (64.7° C.) and 100° C. is 252 cal./gm. The average value for water between these temperatures is 550 cal./gm. The average molal latent heat for water would then be $(550)(18.02) = 9911$ cal/gm. mol. Since the value per gram of methanol is 252 cal., the molecular weight must be $9911/252 = 39.3$. This value is used in these calculations wherever the molecular weight of methanol is required.

Vapor-liquid equilibrium data for the methanol-water system used were those of Bergström presented by Hausbrand (2). The values given were recalculated to the fictitious molecular weight basis and are given in the following table.

¹ This is essentially the same as using the "equivalent latent heat fraction" as suggested by Peters (8).

Mol % CH ₃ OH In Liquid	Mol % CH ₃ OH In Vapor	Mol % CH ₃ OH In Liquid	Mol % CH ₃ OH In Vapor
0.46	3.53	33.18	69.20
1.87	12.53	36.83	71.57
2.84	18.11	40.72	73.78
3.83	23.15	44.88	76.26
4.84	28.73	49.32	78.54
6.42	33.98	54.08	91.03 — 81.03
8.02	39.07	59.19	83.55
9.72	43.20	64.69	85.90
11.44	46.83	67.60	87.28
13.24	50.22	70.63	88.52
15.12	52.72	73.78	89.84
17.04	55.24	77.06	91.16
19.09	57.75	80.48	92.64
21.20	59.43	84.05	94.13
23.39	61.60	87.77	95.62
26.46	63.50	91.66	97.09
29.72	66.76	95.73	98.54

Calculation of the theoretical plate equivalent of the dephlegmator for each run was made by a modification of the method of McCabe and Thiele (4) suggested by Webber and Bridger (12). Since the composition of both reflux and product were known, the composition of the entering vapor could be calculated and the indirect method as used by Webber and Bridger was unnecessary.

SAMPLE CALCULATIONS: The method of calculation will be illustrated

using the data of run number 56:

Condensate from dephlegmator (reflux)
 Weight percent methanol = 81.8
 Mol percent methanol = 67.3
 Rate of flow: 257 ml./min. = 218 gm./min.
 Gram Mols per minute = 6.74

Condensate from final condenser (product)
 Weight percent methanol = 91.6
 Mol percent methanol = 83.3
 Rate of flow: 111 ml./min. = 91 gm./min.
 Gram mols per minute = 2.55

Entering vapor Weight percent methanol = 84.6
 Mol percent methanol = 71.6
 Rate of flow: 309 gm./min. = 9.29 gm. mols per minute.

Nomenclature: x = mol percent of CH_3OH in liquid
 y = mol percent of CH_3OH in vapor
 V = mols per minute of entering vapor
 R = mols of reflux per minute
 P = mols of product per minute

The reflux ratio, which is defined as the mols of reflux per mol of product is then $6.74/2.55 = 2.64$. The ratio of mols of reflux to mols of total vapor, which is the same as the slope of the operating line, to be used later, and also equal to the (mol) fraction of vapor condensed in the dephlegmator is $6.74/9.29 = 0.725$.

The theoretical plate equivalent, or efficiency of the dephlegmator is determined graphically. (4), (12). Referring to figure 5, composition of the product is located as point x_p . Where this composition intersects the 45 degree line is the point from which the operating line is drawn. The slope of the operating line, as calculated, is 0.725. The composition of the entering vapor y_1 , is projected horizontally until it intersects the operating line. The x value of this point is the composition of the liquid reflux $x_R = 67.3\%$. These points check to within a few tenths of a percent in nearly all the runs. The point on the equilibrium curve corresponding to $x =$

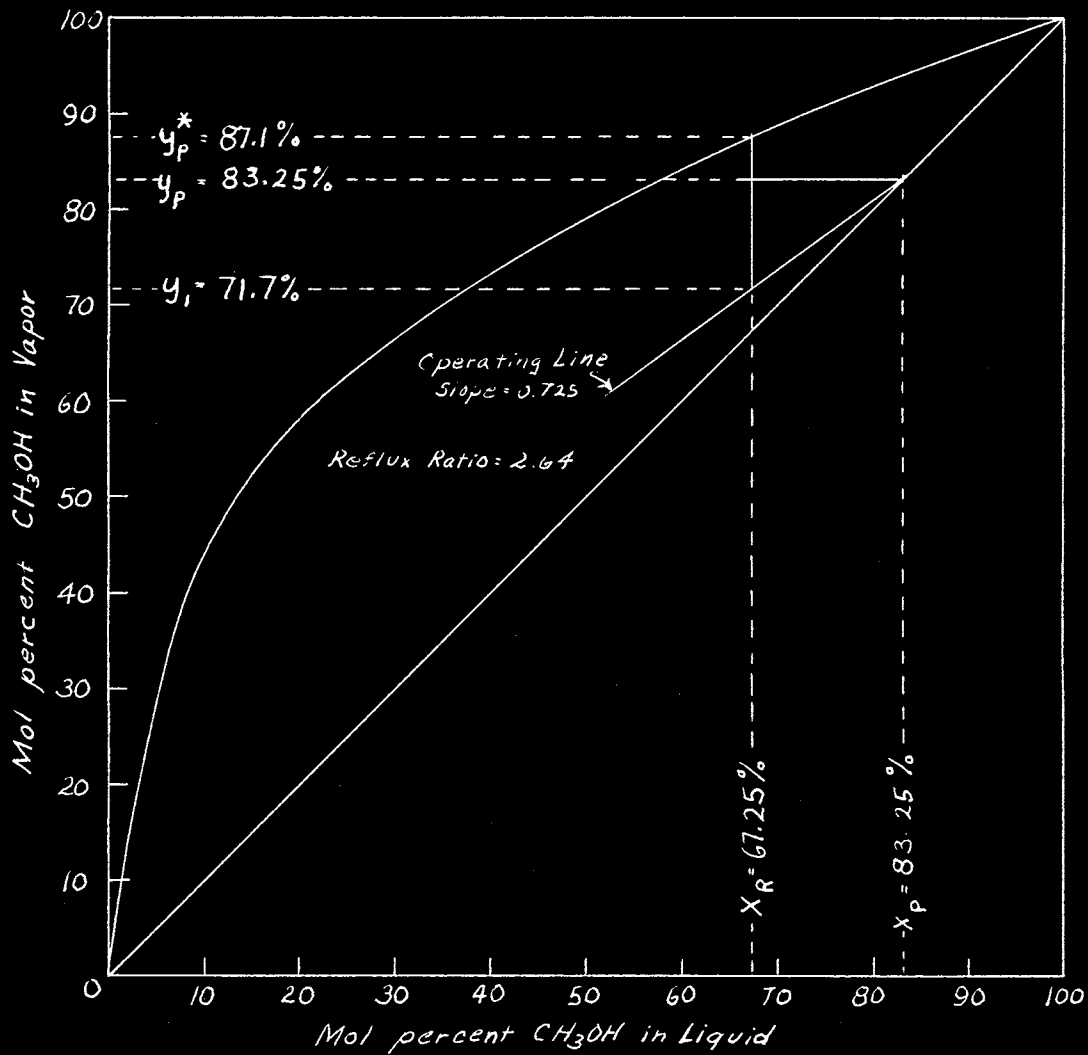


Figure 5.

Example of Determination of Theoretical Plate Equivalent of Dephlegmator.

(Experimental run No. 56.)

67.3% is y_p^* , or the composition of the vapor if one theoretical plate separation had taken place (vapor in equilibrium with the liquid). This value is 87.1%. The actual composition of the leaving vapor, $y_p = x_p = 83.3\%$ falls below the value of y_p^* . The fraction of a theoretical plate represented will thus be

$$\frac{y_p - y_1}{y_p^* - y_1} \quad \text{or} \quad \frac{83.3 - 71.7}{87.1 - 71.7} = 0.75$$

This is analogous to the Murphree plate efficiency (5) as applied to a rectifying column.

In cases when the value of y_p lies above that of y_p^* , it indicates that an efficiency of more than one plate is being attained, and y_p^* should be projected over to the operating line and then upward to the equilibrium curve again. This stepwise procedure is continued until the vertical projection reaches a value above y_p . The plate equivalent is then equal to the number of steps necessary plus the fraction of the last vertical step which is necessary to reach y_p .

A summary of the experimental and calculated data is presented in the following tables.

TABLE I

Run No.	Reflux (R)			Product (P)			Entering Vapor (V)			Reflux :Ratio : R/V	Theo. : Plate : Equiv.	
	:Percent CH ₃ OH:	Flow		:Percent CH ₃ OH:	Flow		:Percent CH ₃ OH:	Flow				
	:Weight: Mol	:Mols/min.:		:Weight: Mol	:Mols/min.:		:Weight: Mol	:Mols/min.:	R/P			
27	94.4	89.5	2.62	98.1	96.1	5.74	97.0	93.7	8.36	.456	.314	1.05
39	93.7	87.2	2.81	97.7	95.1	5.01	96.3	92.2	7.82	.561	.360	.93
28	94.9	89.5	3.06	98.1	96.1	5.37	96.8	93.3	8.43	.570	.363	1.09
33	94.8	89.2	3.45	98.3	96.3	5.00	96.8	93.3	8.45	.690	.408	1.03
29	94.9	89.5	3.65	98.3	96.3	5.00	96.3	92.3	8.65	.730	.422	1.00
40	94.8	89.3	3.60	98.1	95.9	4.36	96.6	92.9	7.96	.827	.452	.90
30	95.4	90.4	4.67	98.4	96.5	4.18	96.7	93.1	8.85	1.11	.527	.97
34	95.4	90.4	4.64	98.3	96.3	3.99	96.6	92.8	8.63	1.16	.538	.91
41	94.9	89.5	4.78	98.2	96.1	3.25	96.2	92.0	8.03	1.47	.595	.96
43	94.7	89.0	5.12	98.1	95.9	3.09	96.0	91.7	8.21	1.66	.624	.94
31	95.9	91.6	5.84	98.3	96.5	3.07	96.7	93.0	8.91	1.90	.655	.85
35	95.9	91.5	5.83	98.3	96.4	2.95	96.7	93.0	8.78	1.98	.664	.87
32	95.9	91.5	6.19	98.4	96.5	3.00	96.6	92.8	9.19	2.06	.674	.88
36	95.8	91.2	6.39	98.4	96.5	2.89	96.6	92.8	9.28	2.21	.689	.94
42	95.1	89.9	5.82	98.1	96.0	2.44	96.0	91.7	8.26	2.38	.705	.91
45	94.9	89.4	5.71	98.2	96.2	2.30	95.8	91.3	8.01	2.44	.700	.98
44	95.0	89.6	5.91	98.0	95.8	2.21	95.8	91.3	8.12	2.68	.728	.90
46	94.7	89.1	5.96	98.3	96.3	2.29	96.5	90.8	8.25	2.60	.722	1.06
47	94.6	88.9	6.29	97.2	94.0	1.97	95.3	90.3	8.26	3.20	.762	.66
38	96.2	92.1	7.31	98.4	96.5	1.96	96.6	92.8	9.27	3.73	.788	.91
37	96.2	92.1	7.63	98.4	96.5	1.48	96.6	92.8	9.11	5.15	.839	.83

TABLE II

Run No.	Reflux (R)			Product (P)			Entering Vapor (V)			Reflux	Ratio	Theo.
	Percent CH ₃ OH:	Flow		Percent CH ₃ OH:	Flow		Percent CH ₃ OH:	Flow		Ratio	Ratio	Plate
	Weight: Mol	Mols/min.		Weight: Mol	Mols/min.		Weight: Mol	Mols/min.		R/P	R/V	Equiv.
8	80.8	65.9	1.93	93.8	87.4	6.05	90.8	81.9	7.98	.319	.242	1.34
3	82.9	68.9	2.63	94.0	87.8	5.74	90.6	81.5	8.37	.448	.314	.99
7	81.9	67.5	2.59	94.1	88.0	5.59	90.3	81.2	8.18	.463	.316	1.29
17	83.3	69.5	3.67	94.6	88.9	5.00	89.9	80.4	8.67	.734	.423	1.15
6	83.8	70.3	3.62	95.2	90.1	4.80	90.5	81.4	8.42	.755	.430	1.20
5	84.3	71.1	3.73	94.8	89.3	4.75	90.4	81.2	8.48	.785	.440	1.10
4	82.9	68.9	3.83	94.0	87.8	4.62	90.6	81.6	8.47	.834	.455	.81
18	83.8	70.3	4.02	94.6	88.9	4.59	90.1	80.6	8.61	.875	.467	1.06
19	85.1	72.3	4.42	94.7	89.1	4.08	89.8	80.2	8.50	1.08	.520	1.00
14	85.8	73.5	4.51	94.6	88.9	4.02	90.3	81.0	8.53	1.12	.529	.90
15	85.9	73.6	4.51	94.6	88.9	4.03	90.2	80.8	8.54	1.12	.529	.90
10	85.3	72.6	4.78	95.8	91.3	3.90	90.3	81.0	8.68	1.23	.550	1.31
20	85.5	73.0	4.89	94.3	88.3	3.95	89.8	80.1	8.84	1.24	.554	.87
21	86.6	74.7	5.67	94.9	89.5	3.17	90.0	80.5	8.84	1.79	.641	.91
12	84.7	76.1	5.53	95.0	89.7	3.04	90.5	81.3	8.57	1.82	.645	.90
13	87.8	76.7	5.73	94.8	89.8	2.94	90.0	80.5	8.67	1.95	.661	.89
22	86.8	75.1	6.03	94.7	89.1	2.82	89.7	79.9	8.85	2.14	.681	.93
12	89.2	79.1	6.02	95.8	91.3	2.76	91.5	83.2	8.78	2.18	.685	.91
24	88.2	77.4	5.99	95.4	90.5	2.23	90.1	80.6	8.22	2.69	.730	.93
23	89.1	78.9	6.73	95.5	90.2	1.50	90.1	80.6	8.23	4.49	.816	.90
9	89.0	78.7	7.50	96.4	92.5	1.35	90.1	80.6	8.85	5.56	.849	1.12
25	89.2	79.1	7.13	95.5	90.1	1.28	90.0	80.5	8.41	5.57	.849	.83
26	89.3	79.3		95.7	91.0		90.0	80.5	8.52	12.10	.992	.90

TABLE III

Run No.	Reflux (R)			Product (P)			Entering Vapor (V)			Reflux	Ratio	Theo.
	Percent CH ₃ OH	Flow		Percent CH ₃ OH	Flow		Percent CH ₃ OH	Flow		Ratio	R/V	Plate
	Weight: Mol	Mols/Min.		Weight: Mol	Mols/Min.		Weight: Mol	Mols/Min.		R/P		Equiv.
48	75.2	58.2	3.93	94.6	89.0	5.31	87.2	75.7	9.23	.739	.425	.76
50	76.8	60.3	4.68	93.1	86.1	4.72	85.6	73.1	9.40	.99	.498	1.25
55	77.8	61.6	4.82	91.9	83.9	4.45	85.0	72.2	9.27	1.08	.520	.95
51	76.7	60.1	5.01	93.0	85.0	4.49	85.0	72.2	9.50	1.11	.527	1.02
52	78.4	62.4	5.30	93.0	85.0	4.25	85.5	73.0	9.55	1.25	.555	1.01
56	81.8	67.3	6.74	91.6	83.3	2.55	84.6	71.7	9.29	2.64	.725	.75
57	82.2	67.8	6.99	91.7	83.4	2.38	85.0	72.2	9.37	2.94	.747	.65
54	82.4	68.2	7.80	93.8	87.5	2.09	85.0	72.2	9.89	3.73	.788	1.00
53	82.8	68.8	7.68	93.7	87.2	2.03	85.2	72.5	9.71	3.78	.791	.93
58	82.4	68.1	7.57	92.0	83.0	1.97	84.5	71.4	9.54	3.84	.793	.73
49	84.8	71.9	10.07	91.6	83.4	1.17	85.7	73.3	11.24	8.60	.896	.65

TABLE IV

75	51.7	32.9	3.23	82.0	67.6	5.82	72.8	55.0	9.05	.555	.357	.91
84	50.5	31.8	5.27	80.9	66.0	6.00	68.6	50.0	11.27	.878	.467	.88
79	49.4	30.9	5.54	83.6	70.3	5.38	69.8	51.4	10.92	1.03	.508	1.11
76	60.2	40.9	5.72	84.3	71.1	3.85	71.1	52.9	9.57	1.49	.598	.87
85	58.1	38.8	7.16	78.4	84.3	4.40	68.6	50.0	11.56	1.63	.619	.83
80	56.3	37.1	6.96	83.3	69.5	3.32	66.8	47.9	9.28	2.10	.750	.88
81	58.2	38.9	7.51	83.4	69.6	2.67	65.9	46.9	10.18	2.81	.738	.87
86	63.0	43.8	8.34	78.3	84.4	2.97	68.7	50.3	11.31	2.81	.738	.74
77	65.5	46.7	7.52	83.8	70.4	2.35	70.0	51.7	9.87	3.20	.762	.74
82	60.3	41.0	8.06	82.2	68.8	2.25	66.1	47.2	10.31	3.58	.781	.81
78	65.1	46.1	8.20	83.9	70.5	1.30	70.0	51.7	9.50	6.31	.864	.73

TABLE V

Run No.	Reflux (R)			Product (P)			Entering Vapor (V)			Reflux	Ratio	Theo.
	Percent CH ₃ OH	Flow		Percent CH ₃ OH	Flow		Percent CH ₃ OH	Flow		Ratio	Ratio	Plate
	Weight: Mol	Mols/Min.		Weight: Mol	Mols/Min.		Weight: Mol	Mols/Min.		R/P	R/V	Equiv.
66	80.5	65.5	2.40	89.5	79.5	1.62	84.3	71.1	4.02	1.48	.597	.56
59	79.9	64.4	3.24	91.7	83.5	2.56	85.4	72.8	5.80	1.27	.559	.82
68	78.9	63.2	3.58	91.4	82.9	2.66	84.6	71.5	6.24	1.35	.574	.83
61	78.9	63.2	4.34	91.3	82.8	3.67	85.1	72.3	8.01	1.18	.541	.82
69	76.0	59.2	5.55	91.8	83.7	4.56	83.8	70.3	10.11	1.22	.548	1.02
71	77.2	60.8	8.75	92.4	84.7	4.92	83.2	69.4	13.67	1.78	.640	1.06

TABLE VI

87	50.9	32.2	4.44	77.9	61.8	6.05	67.9	50.3	10.49	.735	.424	.61
91	49.9	31.3	4.76	77.6	61.3	6.41	67.3	48.5	11.17	.742	.426	.64
88	54.1	35.0	6.80	84.2	71.0	4.10	67.3	48.5	10.90	1.66	.624	1.02
92	53.7	34.7	7.37	84.1	70.8	4.15	66.5	47.6	11.52	1.77	.638	1.03
93	58.7	39.4	8.68	87.5	76.3	3.08	67.8	49.1	11.76	2.82	.738	1.20
89	57.5	38.2	8.06	90.7	81.8	2.77	68.0	49.3	10.83	2.91	.745	1.59
90	58.8	39.5	8.40	88.0	77.0	2.48	66.8	47.9	10.88	3.38	.762	1.25
94	62.3	43.1	10.15	90.0	80.4	1.32	66.4	47.5	11.47	7.70	.885	1.34

The weight and mol percents of methanol in reflux, product, and entering vapor are given for each run, together with the rate of flow of each in gram mols per minute. Calculated values of the reflux ratios, R/V ratios, and the theoretical plate equivalents are also included.

Table I contains data for runs with an entering vapor composition of around 93 mol percent methanol, at a substantially constant rate of flow of between 8 and 9 gram mols per minute. The entries are arranged in ascending order of reflux ratio.

Conditions for the results tabulated in table II were the same as for those of table I except that the composition of the entering vapor was 81 mol percent methanol.

Data for table III were taken under similar conditions to those of tables I and II except that the composition of the entering vapor was changed to 72 mol percent.

The same applies to table IV with the vapor reduced to around 52 mol percent methanol. The rate of flow varied somewhat from run to run in this series.

Table V includes data for trials in which the rate of flow of entering vapor was varied, the composition and fraction condensed remaining practically the same.

All the data in tables I to V inclusive are for runs in which cooling water for the dephlegmator entered at the top and passed downward through the tubes, and the vapor passed upward outside the tubes.

For the set of runs tabulated in table VI, the flow of cooling water was changed to pass upward through the tubes in the condenser,

the reflux ratio being varied for this series.

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DISCUSSION

From observation of the tabulated data, it is seen that, in general, as the reflux ratio is increased, the composition of the reflux becomes richer in methanol and the composition of the product remains practically the same with a tendency toward an increase of concentration of methanol.

The definite rise in percent methanol in the reflux (partial condensate) with increasing fraction condensed might be expected from the following: The first drop of condensate will be poorer in methanol than the remaining vapor, thus increasing the concentration in the vapor. Thus the composition progresses infinitesimally up the equilibrium curve, where the second drop of condensate will, of course, be slightly richer than the first, assuming that the same degree of approach to equilibrium has been attained. Therefore the more vapor condensed, the richer will be the condensate. The composition of the condensate will approach the composition of the entering vapor, if all the vapor be condensed. From this it may be concluded that the mechanism of condensation has been, at least, partially differential, and even though the theoretical plate equivalent may be exactly unity, as was the case in some of these runs, the mechanism is not entirely one of equilibrium as it might first appear.

In the cases of countercurrent flow of liquid and vapor, there is a definite trend toward a lower value of theoretical plate equivalent as the reflux ratio increases. Discrepancies exist at some points but these are probably due to insufficient control of one or

more of the possible variables. The preceding discussion may be applied to the results in tables I, II, III, and IV.

The effect of concentration of the entering vapor on the dephlegmator performance may be observed by comparing runs which have similar reflux ratios in the first four tables. From such an examination it is seen that the percentage increase in vapor concentration in methanol is larger with the lower concentrations. For instance, in table IV is recorded an average increase in concentration from around 50% to about 70%, or an enrichment of 20 mol percent, while in table II the enrichment is from 81% to about 89% or only around 8% enrichment. This is to be expected, as explained in the introduction that the equilibrium curve is farther from the operating line near the center of the graph than at the upper end. However, by noting the values of plate efficiency, or theoretical plate equivalent for the corresponding runs in each table, it is seen that they are substantially the same. Thus it may be concluded that the effect of composition of vapor entering the dephlegmator is small for a particular mixture of vapors, with reference to the performance in terms of theoretical plate equivalent.

In the series of runs to test the effect of rate of vapor flow, the flow was varied from 4.02 to 13.67 mols per minute, a range of over three to one. It is found from table V that with increase in vapor velocity the separation efficiency rises markedly from about 0.6 to a little over 1.0 theoretical plate. This is in agreement with the statement of Underwood previously cited. It is believed that with a considerable increase in vapor velocity the efficiency would reach a maximum and then possibly decline. Due to the rapidity

with which the vapor passes, insufficient time will be allowed for complete interaction between liquid and vapor.

The results of the series of runs in which the cooling water was passed upward through the dephlegmator are tabulated in table VI. This arrangement showed a considerable rise in separation efficiency with increase in reflux ratio, which was not the case in the other runs. The cause of this difference probably can be attributed to the temperature gradient along the tubes of the condenser. With the water passing upward, the coldest part of the tubes is at the lower end. Condensate running down the tubes is cooled to below its boiling point and consequently does not react so readily with the ascending vapors, allowing a more near approach to ideal differential condensation. Thus for most efficient dephlegmator operation the cooling water should enter at the bottom.

All of the runs in this investigation were made with the same binary mixture and in the same piece of apparatus. Therefore the results can yield no information as to the effect of other combinations of substances and apparatus construction. It is known that the efficiency of a bubble plate varies with the mixture being distilled, so it is likely that the efficiency of a dephlegmator will vary in an analogous fashion, the separating performance depending on the properties of the mixture.

Although numerous dephlegmators of unusual design have appeared in the past, as has been discussed, most modern pieces of such equipment are of the same general design--- that of a tube bundle and shell as used in these experiments. The arrangement, size, and spacing of tubes and baffles as well as proportion of length to number

of tubes for the same surface area might be expected to influence the performance of the dephlegmator.

CONCLUSIONS

From the results of this investigation, the following conclusions are drawn:

A greater separating efficiency is obtained in a dephlegmator with parallel flow of liquid and vapor than with countercurrent flow. The parallel conditions were for both vapor and liquid flowing upward while countercurrent conditions were with liquid flowing downward.

The use of reasonably high vapor velocities gives better fractionation than very low vapor velocities.

For parallel flow of vapor and liquid (upward) the efficiency increases definitely with increasing reflux ratio.

For countercurrent flow of vapor and liquid (liquid downward) the efficiency decreases slightly with increasing reflux ratio.

A dephlegmator will produce nearly the same equivalent theoretical plate performance even though the composition of entering vapor may vary widely.

The separating efficiency of a dephlegmator of the general type used in this investigation is in the neighborhood of one theoretical plate. Variation in operating conditions slightly affect the performance. A safe assumption would be to assign an efficiency of one actual plate. This plate should be the same as the average plate in the column in conjunction with which the dephlegmator is used.

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