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A distillation study : II. factors affecting plate efficiency : III. properties of laminated wood glue lines.

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UNIVERSITY OF LOUISVILLE

I A DISTILLATION STUDY
II FACTORS AFFECTING PLATE EFFICIENCY
III PROPERTIES OF LAMINATED WOOD GLUE LINES

A Thesis
Submitted to the Faculty
of the Graduate School
of the University of Louisville
in Partial Fulfillment
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for the Degree of

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

Jack H. Nichols
1947

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Approved by the Examining Committee.

Director G. C. Williams

R. C. Ernst

W. R. Barnes

December, 1947

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ABSTRACT

An extended evaluation of a previously advanced correlation is presented with extensive data obtained from fractionation of a variety of binary systems in a single column. The correlation is shown to be capable of predicting plate efficiencies well within the limits of mechanical design over wide ranges of relative volatility and viscosity.

A survey of the literature concerning the extent to which various factors affect fractionation plate efficiencies is presented. Equipment is designed and methods suggested for correlations which may be evolved from existing data and from future experimental data.

The results are presented for an investigation of the physical properties and characteristics of laminated white oak glue films. Some data are included which show the resistance of Penacolite G-1124 glue films to liquid diffusion and suggestions are included for correlations on the basis of fabrication and curing conditions.

I A DISTILLATION STUDY

INTRODUCTION

The use of distillation as a separational process for refinement of materials has been one of the most important operations practiced by manufacturing industries. The principles of distillation, i. e. thermal separation by differential volatilization, are incorporated in each of its many forms.

One of the most used forms of distillation is that known as plate fractionation. A factor of importance in the design of plate-fractionating columns is the determination of the number of plates required in the column. Theoretical relationships evolved by numerous investigators (1, 2, 3, 4) have suggested calculations for the number of plates necessary to produce a specified degree of separation of components. These methods, however, produce a result based on the premise that a complete and unitized interchange of components takes place on the plates. Since this ideal situation does not exist, the resulting "theoretical number of plates" must be modified to predict the actual number of plates required for a given operation. The design for plate-fractionating columns includes the determination of the type, size, and spacing of plates, placement and nature of weirs, orifices, heating coils, etc. which will give optimum results with the least expenditure. The determination of the relation between actual number of plates required and the theoretical number of plates has, however, been a factor of great concern to designers.

Attempts have been made to correlate the theoretical calculations with the results obtained in the field, and these have produced a factor known as the plate efficiency. This factor for many distillation systems may be found in the literature, as obtained under specific conditions, but the references are too meager for the expanding field of distillation design. There have been many attempts to correlate this plate efficiency factor with the many variables which might affect its magnitude. These correlations have resulted in only a few procedures which give promise of reasonable accuracy.

Realizing the inadequacies of these attempts at correlation, O'Connell (5) expanded the work of other investigators to show a correlation of an overall plate efficiency in fractionating columns as a function of the relative volatility and viscosity of the key components. Using O'Connell's work as a basis, Stigger (6) produced a partial evaluation of this correlation based on experimental data obtained on a single column.

This paper presents the results of a continuation and expansion of the research project initiated by Stigger and reported in his "Correlation of Plate Efficiencies in Fractionating Columns", (6).

HISTORICAL

One of the oldest technical practices in existence is that of distillation. This field has been adopted by chemical engineers and incorportated in their philosophy as one of the unit operations of chemical engineering. For many years there was no real engineering design of fractionation equipment. Results of the first worthwhile attempt to rationalize column design were presented by Sorel (4) at the end of the nineteenth century. Sorel worked from equilibrium relationships between liquid and vapor mixtures. If it had been possible to obtain vapor-liquid equilibrium in a column, Sorel would have solved the question of column design from the standpoint of the number of plates required for a given separation. In practically every case, however, the attainment of equilibrium is impossible, and a field was opened for investigators to find a definite basis for the difference between the theoretical and the actual number of necessary plates.

The discrepancy between actual and theoretical plates, termed efficiency, required a mathematical description. This expression took three general forms: (1) point efficiency, (2) Murphree plate efficiency, and (3) overall efficiency.

Point efficiency is described as the efficiency at one point on any plate in a column, and is the ratio of the enrichment obtained to the enrichment under equilibrium conditions at the designated point.

Murphree plate efficiency is essentially the same as point efficiency except that the enrichment is based on the average composition of vapor entering and that leaving a plate, in comparison to the average liquid leaving the plate.

Overall efficiency is the ratio of the theoretical number of plates to the actual number of plates required for a given separation.

For engineering calculations overall efficiency values are the easiest to use, the most consistent, and in most cases the least difficult to obtain.

Early workers, noting that different columns gave different operating characteristics for similar materials, made an effort to relate the column efficiency to mechanical design aspects of the column.

Peavy and Baker (7) investigated the effect of vapor velocity on plate efficiency, and reported that plate efficiency increases with increasing velocity, becoming fairly constant over a considerable range at moderate velocities. At high velocities, efficiency drops as a result of entrainment and liquid carry-over to the next plate. In order to determine the optimum velocity on the basis of maximum efficiency, a method was developed by Brown and Souders (8) and expanded by Brown and Lockhart (9).

Carey et all (10) determined the effect of slot submergence and slot velocity on plate efficiency. They concluded that the efficiency increased as slot submergence increased; and, for normal operating velocities, the efficiency was essentially independent of slot velocity.

In order to insure stable plate operation, the liquid load must be maintained below the value which would produce a greater liquid head at the entrance to the plate than at the overflow from the plate. Allowable liquid loads are predictable according to the method discussed by Good et al (11).

O'Connell (5) has stated that the diameter of the column does not affect plate efficiency if diameters are less than five feet. However, if columns are larger than seven feet, the length of the liquid path(cross flow effect) must be taken into account.

The effect of mechanical design factors on plate efficiency has been confirmed in practice. Yet, when a column is designed for optimum efficiency, based on these mechanical factors, a considerable divergence of results may be obtained when the column is operated with different systems. This variation suggested the possibility that physical properties of the process materials might affect the efficiency of the separation.

One of the first physical properties investigated with regard to its effect on plate efficiency was viscosity. Walter and Sherwood (12) have shown that in the absorption of various materials the absorption efficiency increased with decreasing viscosity of the absorbant. Moreover, Keyes and Byman (13) presented evidence of viscosity affecting plate efficiency in laboratory distillations of ethanol-water mixtures.

If viscosity affects absorption efficiency, as has been shown by Walter and Sherwood, then it follows that there should be a similar effect in fractionation, since both operations are diffusional processes. This conclusion is based on the postulation that the major portion of the enrichment during fractionation, is a result of selective counter-diffusion of the key components in the froth above the plate (5).

Most of the work on viscosity involved the use of laboratory data and was thereby limited as to commercial application. Drickamer and Bradford (14) published a definite correlation of plate efficiency as a function of viscosity (Figure 1) wherein the efficiency was shown to increase with decreasing viscosity. The data for this graph were obtained from commercial operations, of systems of hydrocarbons having low relative volatility between the key components.

Gerster, Koffolt, and Withrow (15) presented evidence that the actual number of plates required for a given separation was a function of the slope of the equilibrium curve. From this, and the previous literature O'Connell (5) deduced that the limitations of the correlation of Drickamer and Bradford could be reduced by correlating plate efficiency as a function of the viscosity and

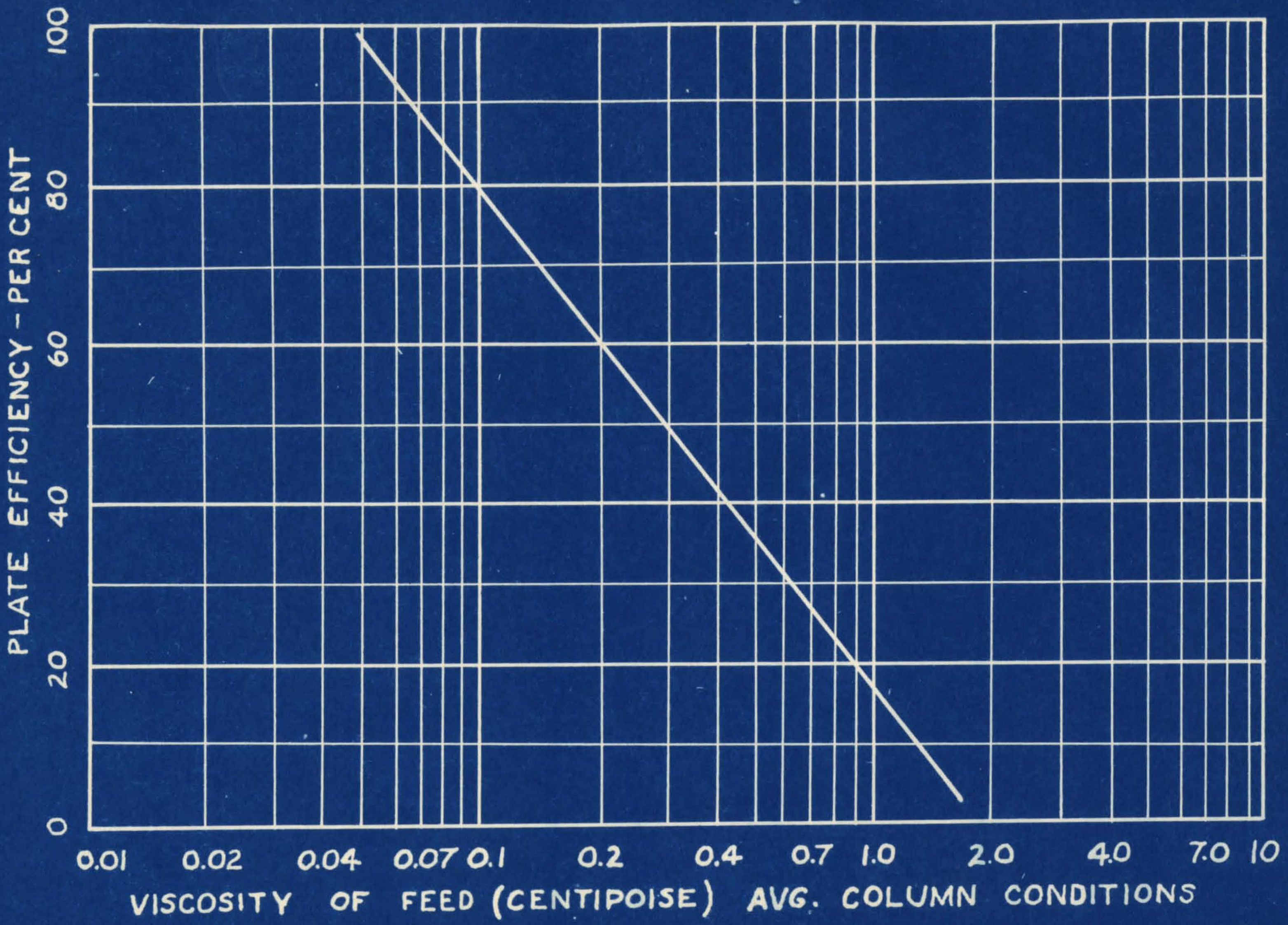


FIG. I — EFFECT OF VISCOSITY ON PLATE EFFICIENCY

(CORRELATION OF DRICKAMER AND BRADFORD)(6) the relative volatility of the key components.

His correlation (Figure 2) advances the relationship as a simple plate efficiency vs. viscosity-relative volatility product and was estimated from the results of actual experiments with numerous hydrocarbon, and non-hydrocarbon systems.

This correlation was presented as a non-specific function, but was not fitted with equations. It was considered to be well within the limitations of normal column calculations, and has been substantiated by some results from commercial units subsequent to its presentation.

Stigger (6) presented evidence to support O'Connell's correlation in the form of data which were obtained from rectification of binary mixtures. He used a single column in his work and thus eliminated the effects of variation in mechanical design. His investigation included the separation of five binary mixtures of widely variant properties. Stigger's data supported the correlation of O'Connell in rectification work in certain ranges. This work was used as a basis for the extended evaluation presented later in this report.

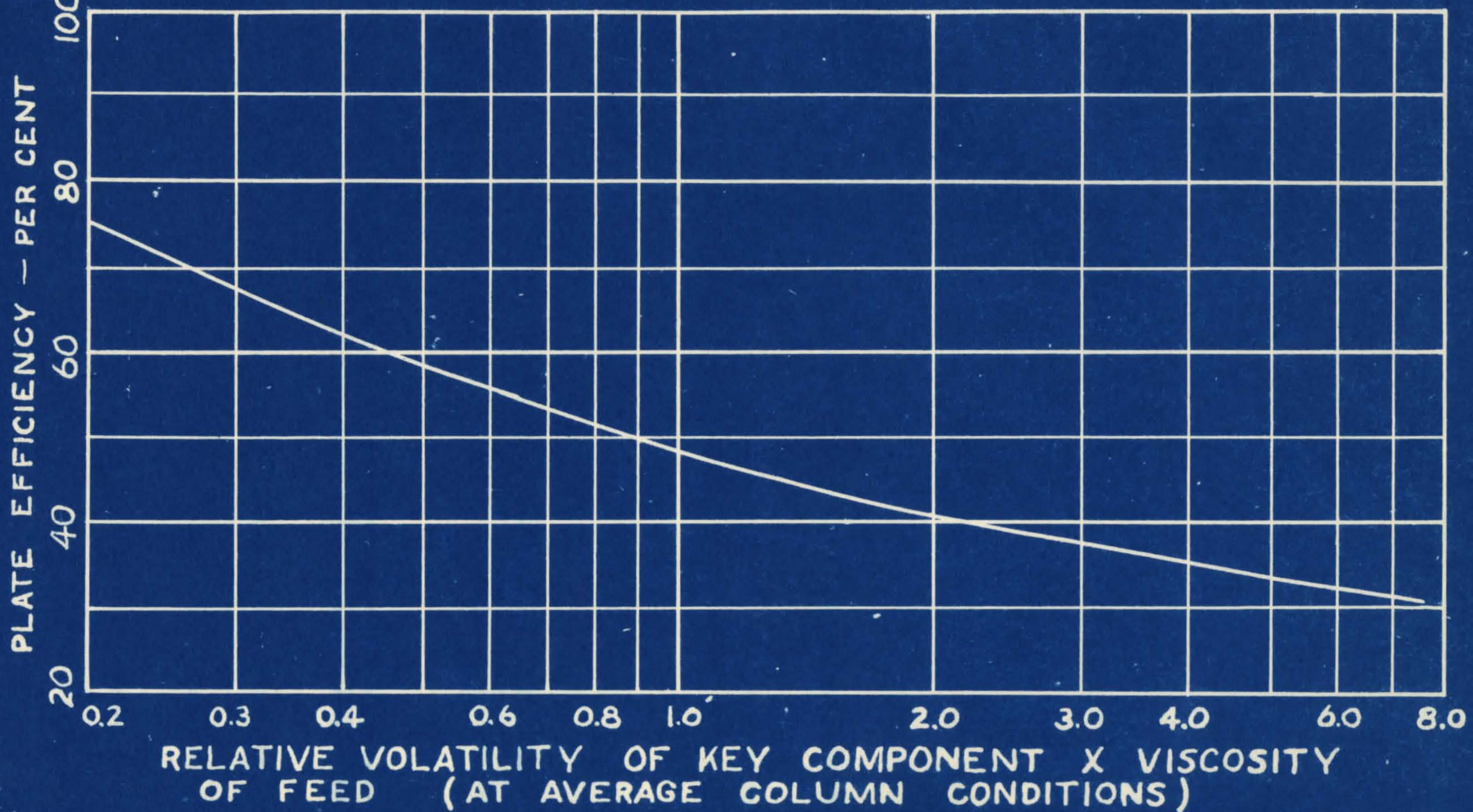


FIG. 2 — EFFECT OF RELATIVE VOLATILITY AND VISCOSITY ON PLATE EFFICIENCY

(CORRELATION OF O'CONNELL) (6)

THEORETICAL

EFFECT OF VISCOSITY ON PLATE EFFICIENCY

The theoretical background for the effect of viscosity on plate efficiency was summarized by Stigger as follows (6).

First, the Stokes-Einstein equation for diffusivity,

$$k = \frac{RT}{6\pi N \mu r} \qquad (1)$$

presents diffusivity, k, as an inverse function of viscosity, μ.

where k = diffusivity, square feet per hour

R = Universal gas constant

T = Temperature, degrees Kelvin

N = number of mols transferred

μ = viscosity

The equation for mass transfer developed by Chilton and Colburn (16) indicates that mass transfer, the coefficient "K", under turbulent conditions is an inverse function of viscosity, μ.

$$K = \left(\frac{G\rho}{M}\right)\left(\frac{k}{\mu}\right)^{0.67} \qquad (2)$$

where K = mass transfer coefficient

G = Mass velocity of gas

M = mean molecular weight

ρ = density

Further evidence was presented by von Karman (17) in his equation for film thickness;

$$s = \frac{(\text{constant})(\mu)}{(G)\,\frac{f}{2}} \qquad (3)$$

where s = film thickness

f = fanning friction factor.

Here the film thickness, s, is shown to be a direct function of the viscosity, μ.

Sherwood and Walter (12) developed an equation for liquid mass transfer,

$$K_La = \frac{3.4}{(\mu)^{0.58}} \qquad (4)$$

where K_La = the product of the mass transfer coefficient and the wetted area, used as a unit.

Here there is an inverse relationship between the liquid mass transfer coefficient, K_La and viscosity, μ.

Although none of these equations indicates a constant mathematical relationship between viscosity and plate efficiency, they all show that viscosity enters into the value of numerous factors which do affect the degree of separation attainable through fractionation.

EFFECT OF RELATIVE VOLATILITY ON PLATE EFFICIENCY

In the work of Gerster, Kofolt and Withrow (15) it was pointed out that the slope of the equilibrium curve had a direct bearing on the actual number of plates required per transfer unit. If the number of transfer units is equal to, or has a direct relation to, the number of theoretical plates required, then the plate efficiency may also be considered a function of the slope of the equilibrium curve. If the relative volatility is a function of the slope of the equilibrium curve, or, more precisely, a mathematical representation of the slope, then it follows that there should be a connection between plate efficiency and relative volatility.

Although no theoretical explanation for the relationship is available, O'Connell (5) presents the following in support of such a conclusion:

> "The effect of the relative volatility was shown by the results of a test on an ethylene dichloride stabilizer. In this column, the feed contained both water and ethyl chloride which had to be reduced to low concentrations in the ethylene dichloride bottoms. The plate efficiency based on water as the low boiling key component (ethylene dichloride the high boiling key) was 29% and the relative volatility between water and ethylene dichloride was 16. The plate efficiency based on ethyl chloride as the low boiling key component was 57%, and the relative volatility between ethyl chloride and ethylene dichloride was 3.1."

A product of viscosity and relative volatility can have no physical significance, nor can this product be justified on strictly theoretical lines. However the previous references do indicate some such function which for brevity and ease is presented in this form.

ANALYSIS OF PROBLEM

EQUIPMENT

As previously stated it was desired in this project to evaluate a predetermined relationship between the plate efficiency of a distillation system and the viscosity-relative volatility product of the same system. Plate efficiencies have been shown to vary with different columns, different flow rates and other mechanical factors (7, 8, 9, 10). If the correlation is to hold in general, it should hold even more rigidly for a single column operated under definite and specific conditions. It was proposed to utilize one column for this investigation, using different systems, but with as closely constant operating conditions as possible.

It was desirable that the column correspond as closely as possible to commercial size and operation. The unit chosen for the work was a copper laboratory experimental column with the following specifications (18).

Number of plates	20 (plus reboiler)
Diameter	10 inches
Plate spacing	8 inches
Vapor risers	1-5/8 inch dia. (4 per plate)
Downcomers	½ inch dia. (2 per plate)
Weirs	6 inch X 1 in. (2 per plate)
Bubble caps	2½ in. dia. X 1-3/4 in. high (4 per plate)
Slots	5/8 in. X 1/8 in. (31 per cap)

Calandria (Reboiler)	12 in. dia. X 12 in. long (shell) 24 1 in. tubes with auxiliary open steam connection
Feed arrangement	Liquid feed inlets on plates 1, 3, 5, ----- 15, 17 (from top) Vapor feed inlets on plates 2, 4, 6, ----- 18, 20 (from top) liquid feed delivered by gear pump through a rotameter
Feed pot	60 gallon capacity
Overheads	Two condensers (tube and shell) Splitter box arranged to produce any desired portion or overheads as reflux to top plate Product and reflux flow by gravity through rotameters
Sampling	sample cocks arranged to produce samples of the liquid off plates 1, 3, 5, --- 17, 19 Samples of feed, waste, and overheads easily obtainable
Temperatures	Plate temperatures obtained by copper-constantan thermocouples in the liquid on plates 1 through 20

The column was designed for a maximum feed rate of twenty gallons per hour, and for satisfactory operation on the systems, ethanol-water, benzene-toluene, and acetic acid-water at vapor velocities between 0.75 and 1.25 feet per second. (A sketch of the column and details of plate construction are shown in Figures 30 and 31, Appendix.)

PREVIOUS WORK

The data used by O'Connell in the original placement of his correlation fell within the limits of 27% and 90% plate efficiency and 0.10 and 7.5 viscosity-relative volatility product expressed in terms of centipoises. It was desired that this investigation extend the range of the viscosity-volatility product to as low and as high a value as possible, while at the same time covering intermediate points with a variety of systems.

Mathematically the same product of viscosity and volatility may be obtained by an increase of one value if the other decreases correspondingly. This lent a further degree of variance in the choice of systems.

Examination of the literature for binary systems with a variety of molecular structures, viscosities, volatilities, and known vapor-liquid diagrams, disclosed approximately two dozen systems falling within the permissible temperature and pressure range of the apparatus. Of these systems many were discarded because of hazardous operation, similar nature, inability to obtain easy and accurate analysis, or presence of abnormalities in the equilibria.

The systems chosen and investigated by Stigger (6) are as follows:

System	Range of $\mu\alpha$, (centipoise)
Ethanol-water	1.0 - 2.0
Methanol-Water	1.0 - 2.5
Benzene-Toluene	0.69 - 0.72
Benzene-Carbon Tetrachloride	0.38 - 0.44
Acetaldehyde-Water	5.0 - 50

Average results of the work of Stigger are shown in figure 3. These data cover a fairly wide range of the correlation in question and are represented comparatively well by it. Figure 3, however, indicates that the correlation is not applicable for the stripping efficiencies as presented. In an attempt to partially explain the non-applicability of the correlation for stripper operations Stigger presented the data for the system methanol-water as a function of the reflux ratio as is shown in figure 4. This curve indicates a relation between the plate efficiency and the reflux ratio in rectifying sections, where the reflux ratio is below one. In stripping sections, however, the ratio of liquid to vapor is greater than one. If the relation of figure 4 can be extended to include stripping sections then an explanation for high efficiencies in stripping operations will have been found.

To complete the evaluation of the correlation of O'Connell it was decided to include the study of three additional systems which would fill in the ranges not covered previously and at the same time would extend the correlation to a high value of the viscosity relative volatility product. The systems chosen are shown in Table I.

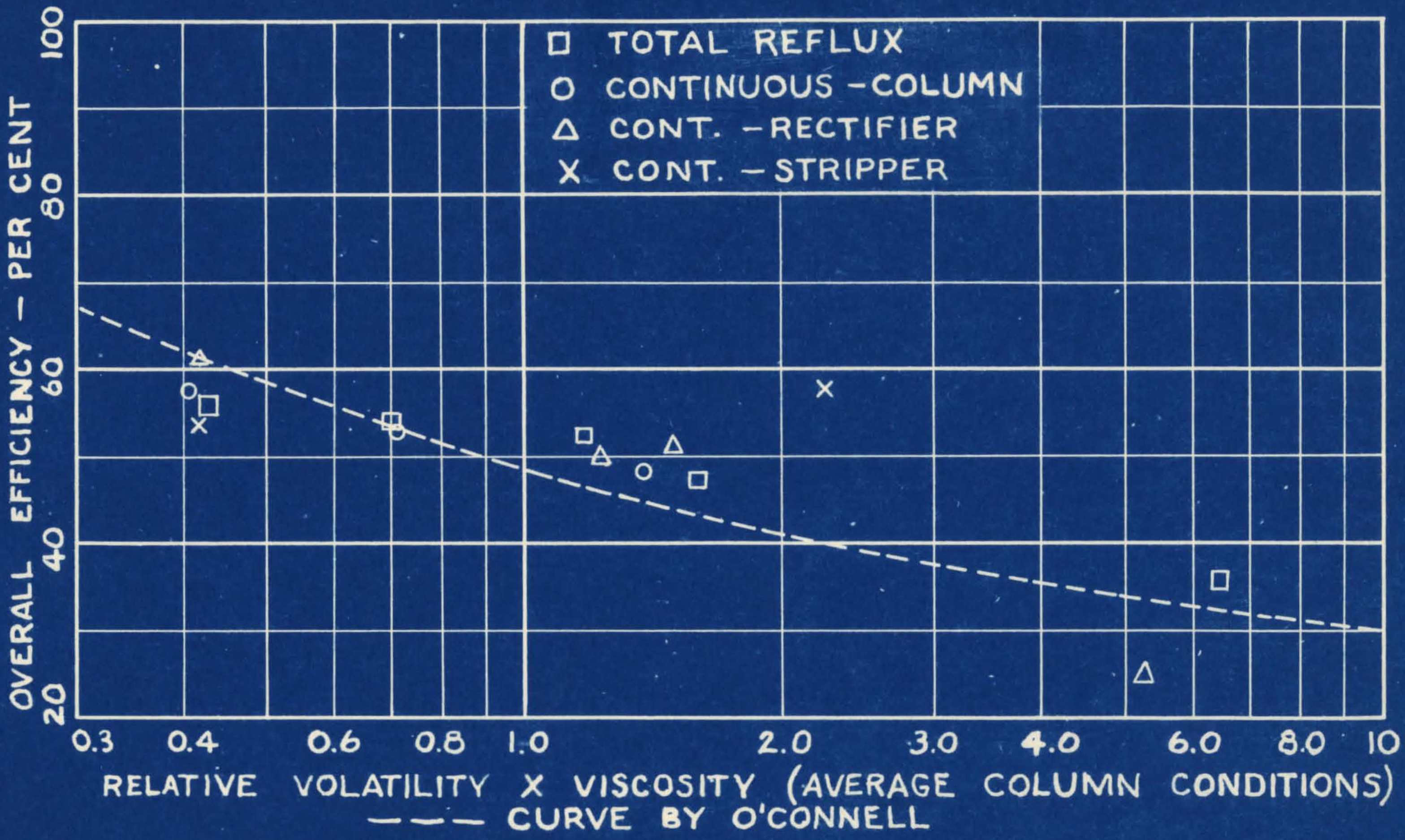


FIG. 3 – EFFECT OF RELATIVE VOLATILITY AND VISCOSITY ON OVERALL EFFICIENCY

(AVERAGE RESULTS BY SYSTEMS)(6)

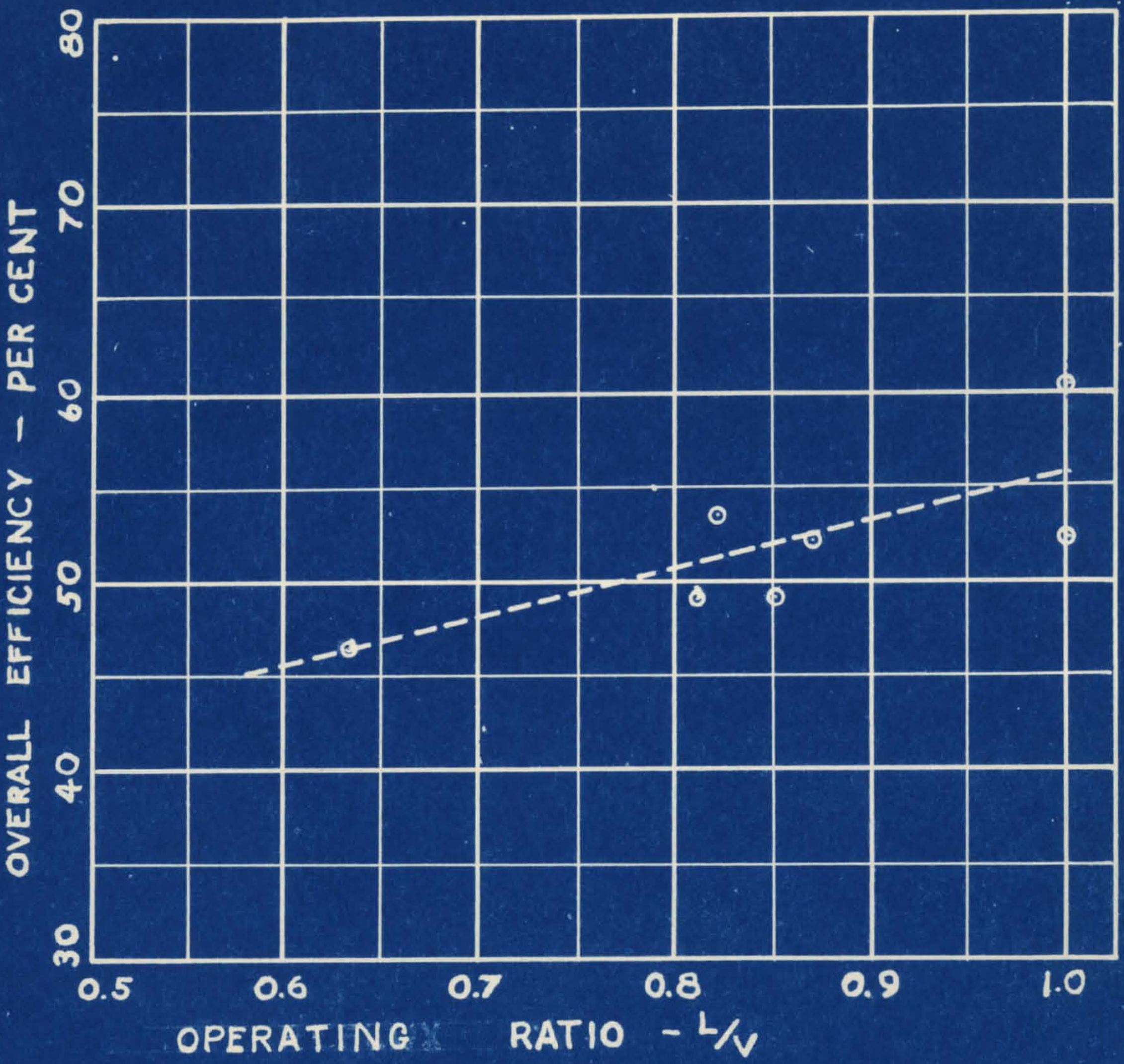


FIG. 4 - EFFECT OF REFLUX RATIO ON OVERALL EFFICIENCY OF RECTIFYING SECTION (6)

SYSTEM: METHANOL-WATER

TABLE I. SYSTEMS INVESTIGATED

No.	System	Boiling point range	μ, Centipoises
1	Acetone-water	56.1 - 100°C	0.4 - 9.0
2	MEK-water	73.8 - 100°C	10 - 20
3	Pentane-water	36.7 - 110.7°C	1.5 - 2.5

EXPERIMENTAL

GENERAL EXPERIMENTAL PROCEDURE

A general outline of experimental procedure which was sufficiently flexible to accommodate the different systems and various conditions of operations to be encountered was developed for this investigation. This general procedure was used as a basis for four types of operations:

1. Total reflux operations.
2. Continuous overall column operation, with feed near the center of the column and both rectification and stripping data being obtained.
3. Continuous fractionation with the greater part of the column under stripping conditions, and feed near the top of the column. This type of operation gave data on the characteristics of the system being studied under stripping conditions.
4. Continuous fractionation, with the greater part of the column acting as a rectifier and feed near the bottom of the column. Data obtained from this type of run for the most part covered rectification characteristics.

In preparation for a total reflux run, approximately fifty gallons of material were placed in the still feed tank. A quantity of the mixture was placed in the reboiler of the still so that the steam heating tubes were covered. Cooling water was turned into the condensers and steam was admitted to the reboiler. Partial distribution of components was attained as vapors rose in the column, heating the entire column to operating temperature. As the vapor rose in the column and the liquid level in the reboiler fell, additional feed mixture was pumped into the column. The flow of feed was manually controlled to maintain the desired level in the reboiler, until condensed vapors reentered the top of the column as total reflux. The feed was shut off and the column allowed to operate at total reflux until equilibrium conditions were approached.

The approach of the column conditions to those of equilibrium was observed by taking thermo-couple plate temperature readings at frequent intervals. When these readings became constant, it was assumed that the column had reached equilibrium. If, on total reflux runs, it was desired to adjust the range of component distribution, the change was made by the addition of feed and the removal of either distillate or waste.

The temperature of the liquid on each plate was recorded, later to be averaged for determining viscosity and relative volatility values in the column. Samples were withdrawn from alternate plates (1, 3, 5---17, 19), from the distillate, and from the reboiler. These samples were usually about 25 - 30 ml. in volume and were obtained in the shortest possible time so that the equilibrium conditions of the column would not be adversely affected. The quantity of distillate could be observed at any time during operation by readings on a tapered-tube reflux rotameter.

Temperature measurements were made with copper-constantan thermocouples inserted in the liquid close to the center of each plate. The readings were direct with the use of a Leeds & Northrop automatically compensating potentiometer.

Continuous column operations were started in the same method as that for total reflux runs. At the point where total reflux conditions would have been attained, continuous feed was introduced at a metered rate onto the plate containing a mixture most nearly corresponding to the concentration of the feed. The distillate was continuously split into product and reflux with the product rate adjusted to remove the proper amount of low boiler from the system at this point and at the same time to produce the desired reflux ratio.

Two methods were used to effect a change in reflux ratio:

(1) Feed and product rates were held constant while the heat input to the reboiler was changed.

(2) Heat input to the reboiler was held constant while the feed and/or product rates were changed.

In each case the result was a shift in the component distribution and this necessitated a corresponding period for column readjustment.

The bottoms rate was adjusted to maintain a constant liquid level in the reboiler. Continuous feed, product, and bottoms rates were carefully controlled for a period sufficient to produce steady operation as indicated by constant plate temperatures. Samples and data were obtained as during total reflux, with the addition of a feed sample, and product flow rate. The bottoms rate was not metered; but it was assumed that the maintenance of constant feed and product rates and a constant liquid level in the reboiler would render the bottoms rate calculable. This assumption required that column holdup be essentially constant; and this should occur under normal operating conditions. Sample operational data sheets for experimental work are presented in figures 5, 6, and 7.

Samples were analyzed by methods chosen for the system under investigation, and the data were used in calculation of the efficiency of fractionation.

Efficiency, as used in this report is defined as the ratio of the number of theoretical plates required for a given separation, to the actual number of plates used for that separation. The calculation of the theoretical number of plates was accomplished by the step-by-step procedure of McCabe and Thiele (19) on a plot of the vapor liquid equilibria. Vapor-liquid equilibria were plotted on 20" x 20" cross section paper except when accuracy in low-composition ranges required the use of a plot on log-log paper (20). Prior to efficiency calculations on each run a plot was made of the plate sample composition versus its placement

LABORATORY EXPERIMENTAL DATA

Run No. 50 System Acetone - Water

Type of Operation Continuous Column Date 11 Feb., 1947

Started Column @ - - - .M. Continuation of Run #49

Total Reflux @ ______ .M.

Feed to plate No. 9 @ 12:00 A.M.

Product over @ 12:05 P.M., bottoms over 12:05 P.M.

Samples and temperature readings @ 1:10 P.M.

Steam pressure 10 p.s.i. gage

Reflux Rotameter 62 mm; Rate 120 #/hr @ 25°C

Product Rotameter 90 mm; Rate 80 #/hr @ 25°C

Feed Rotameter 200 mm; Rate 115 #/hr @ 50°C

Sample	Specific Gr.	Mol % light key
P	0.7946	95.5
1	.7979	92.0
3	.8006	90.0
5	.8063	84.0
7	.8172	75.0
9	.8889	35.5
11	.9962	0.5
13	.9975	0.5
15	.9975	↓
17	.9975	
19	.9976	
B	.9978	0.5
F	.8907	35.0

Plate	Temp. °C.
1	56
2	↓
3	
4	
5	
6	
7	
8	56
9	60
10	62
11	78
12	92
13	98
14	100
15	100
16	↓
17	
18	
19	
20	100

Figure 5 Typical Data Sheet, Acetone-Water

LABORATORY EXPERIMENTAL DATA

Run No. 70 System MEK - water

Type of Operation Continuous Stripping Date 15 April, 1947

Started Column @ 1:40 P.M.

Total Reflux @ 2:10 P.M.

Feed to plate No. 3 @ 2:21 P.M.

Product over @ 2:25 P.M., bottoms over 2:25 P.M.

Samples and temperature readings @ 3:15 P.M.

Steam pressure 8 p.s.i. gage

Reflux Rotameter ____ mm; Rate ____

Product Rotameter 58 mm; Rate 49 #/hr.

Feed Rotameter ____ mm; Rate 39.1 #/hr. @ 74°C

Sample	Freezing Pt.	Mol % light key
P	Vol. Est.	95
1		
3	-2.06	1.89
5	-2.03	1.86
7	-0.94	0.80
9	-0.11	0.10
11	-0.01	0.01
13	0	0
15		
17		
19		
B		
F	-1.75	1.58

Plate	Temp. °C.
1	75
2	78
3	78
4	76
5	79
6	79
7	79
8	79
9	84
10	90
11	95
12	98
13	100
14	
15	
16	
17	
18	
19	
20	100

Figure 6 Typical Data Sheet, MEK-Water

LABORATORY EXPERIMENTAL DATA

Run No. 85 System Pentane - toluene

Type of Operation Continuous Stripping Date 29 April, 1947

Started Column @ 3:15 P.M.

Total Reflux @ 3:40 P.M.

Feed to plate No. 9 @ 3:46 P.M.

Product over @ 3:55 P.M., bottoms over 3:55 P.M.

Samples and temperature readings @ 4:20 P.M.

Steam pressure 18 p.s.i. gage

Reflux Rotameter 25 mm; Rate 23 #/hr

Product Rotameter 20 mm; Rate 18 #/hr

Feed Rotameter 200 mm; Rate 110 #/hr @ 25°C

Sample	Specific Gr.	Mol % light key
P	0.6232	99.2
1	.6246	98.7
3	.6272	97.2
5	.6323	95.0
7	.6518	86.2
9	.7748	37.2
11	.7717	34.0
13	.7836	29.4
15	.8267	13.0
17	.8554	2.5
19	.8612	0.5
B	.8624	>0.5
F	.8146	17.7

Plate	Temp. °C.
1	
2	36
3	36
4	
5	36
6	38
7	40
8	41
9	56
10	56
11	58
12	58
13	60
14	62
15	72
16	84
17	96
18	104
19	107
20	

Figure 7 Typical Data Sheet, Pentane-Toluene

in the column so that abnormalities in distribution, feed mismatch etc. could be detected and dealt with properly.

Where feed mismatch was evident the abnormal data of adjacent plates were not used in calculation of rectifier and stripper efficiencies. Data on runs where feed was excessively mismatched were discarded completely. A check on the method was made by the calculation of two-plate efficiencies throughout the length of the column. Where the two-plate efficiency deviated widely from the average, the data were not included in calculation of the overall column efficiency. This was especially evident in stripping sections. By this method data from the section of the column over which there was good distribution were selected and used in efficiency determinations.

For correlation of results a psuedo molal viscosity was employed as suggested by Drickamer and Bradford (14). Their method assumed that the viscosities of mixtures may be determined by adding the viscosities of the components at a given temperature in proportion to the mol fraction of each present. Viscosity values for pure components as a function of temperature were obtained from standard references. The viscosity for correlation purposes was taken at an average column temperature.

The value of the relative volatility for use in the correlation was obtained by a weighted integration of the actual values on plates over which results are reported.

DATA AND RESULTS

ACETONE-WATER

Investigation of the system acetone-water would place data in the same range as those of the methanol-water and ethanol-water systems. However, since the system is of a different type than those studied by Stigger (6), it was included to improve the value of the resulting correlation.

Initial work on the system proceeded in the same manner as that previously described. One run was made at total reflux, and a number of continuous column operations were made with a central feed. Separation in the stripping section was so rapid and the system so sensitive to slight temperature and pressure changes that mismatch was frequent and stripping data were useless for initial runs. To complete the study of the system, several runs were made with continuous feed to the top plate, and the entire column under stripping conditions. During these last operations the rapid separation caused difficulties in obtaining a wide distribution in the column, but efforts were made to increase the distribution by increasing the liquid-vapor ratio to the limit of column operation. Near this upper limit, a distribution of acetone was obtained over as many as seven plates.

Over the range of compositions to be encountered in investigation of the acetone-water system, it was found that Westphal balance density measurements would give an accuracy of 0.25 mol per cent. Density composition data were determined in the laboratory, (Table II) and used subsequently in sample analysis.

For the initial runs this analysis was satisfactory, but for continuous stripping runs, where the acetone content of samples was very low, the precision was inadequate. It was decided then to use a 25 ml. pyonometer with a thermometer and side arm, as specified by ASTM standard D-153-39 for density determinations. These were made at 20° C. with an accuracy of 0.02 mol per cent, which was sufficient for this part of the investigation.

Calculations were made for the initial runs on acetone-water by the method of McCabe and Thiele (19), from equilibrium data calculated by Carlson and Colburn (21) and presented in Table III. However, because of the very low acetone content in stripping section samples, an extension of the method of McCable and Thiele was necessary for calculation of stripping efficiencies. For these operations the applicable section of the equilibrium curve was transferred to log-log coordinate paper and calculations completed (20).

The results for the system are presented in Table IV and shown graphically in Figure 8.

The overall and rectification efficiencies appear to be in good agreement. The overall efficiency of run No. 50 is somewhat higher than the average result, but it is noted that the efficiency of the rectifying section of this same run agrees closely with the average. The fact that a short but very high efficiency of the stripper section is included

TABLE II. ANALYTICAL DATA, ACETONE-WATER

Sample	Wt. % Acetone	Mol Per Cent Acetone	Specific Gravity
0	0.00	0.00	0.9986
1	16.50	5.75	.9756
2	27.10	10.32	.9600
3	38.30	16.20	.9438
4	46.00	21.00	.9262
5	54.20	26.90	.9111
6	58.40	30.40	.9007
7	62.60	34.30	.8913
8	71.50	43.70	.8702
9	76.00	49.50	.8502
10	85.30	64.20	.8340
11	90.20	73.90	.8197
12	95.00	85.60	.8057
13	97.50	92.40	.7974
14	100.00	100.00	.7910

TABLE III. VAPOR LIQUID EQUILIBRIA, ACETONE-WATER

x_A	y_A	t_b	α
0.00	0.000	100.0	28.50
0.01	0.225	93.5	28.80
.02	.370	88.1	28.80
.05	.591	77.8	27.40
.10	.723	69.3	23.50
.20	.797	63.4	15.70
.30	.817	61.7	10.42
.40	.826	61.0	7.14
.50	.834	60.4	5.05
.60	.847	59.8	3.68
.70	.866	58.9	2.78
.80	.894	57.9	2.12
.90	.939	56.9	1.70
.95	.966	56.5	1.51
1.00	1.000	56.1	1.30

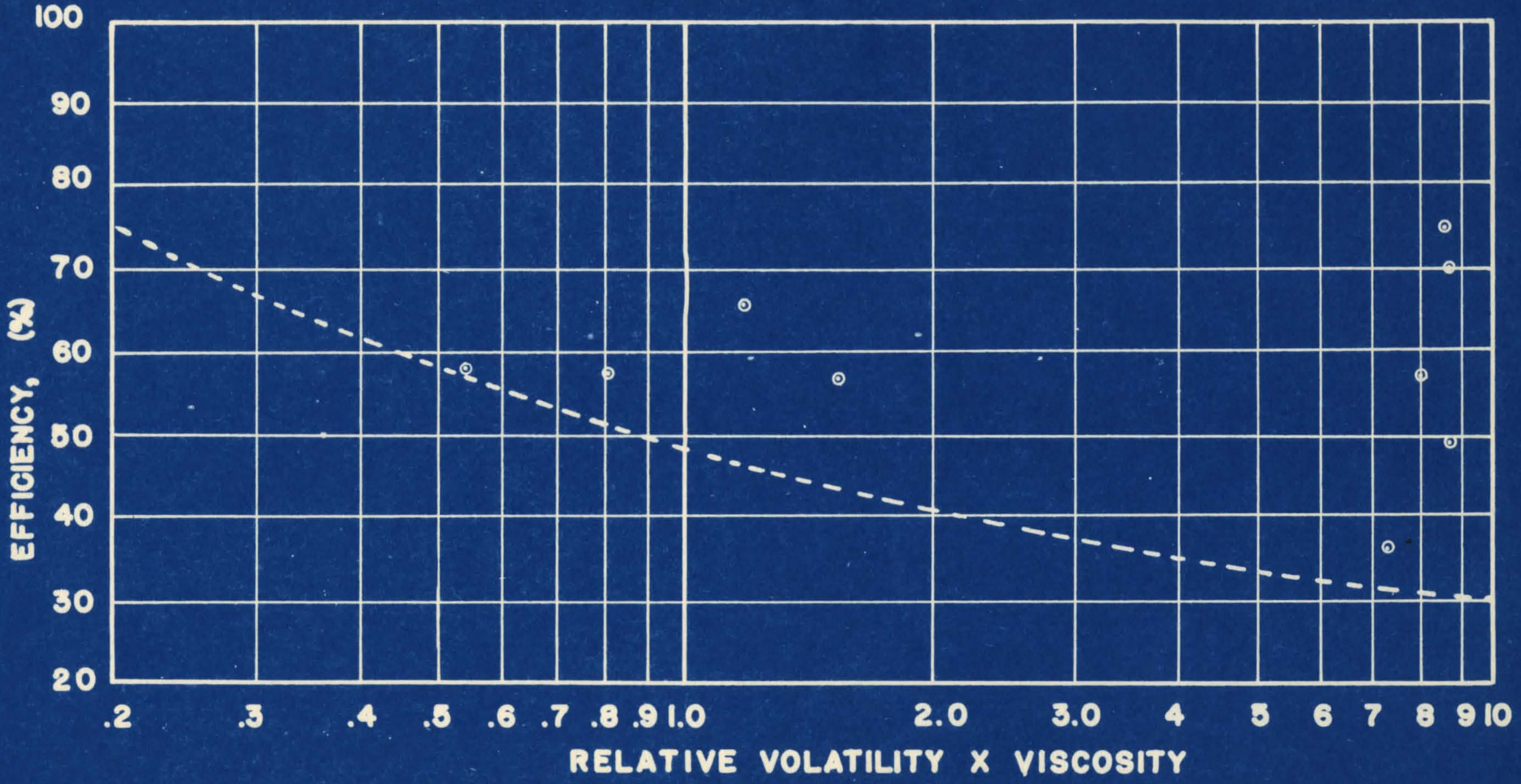


Figure 8 Effect of Relative Volatility and Viscosity on Plate Efficiency, Acetone-Water System

TABLE IV. ACETONE-WATER SUMMARY DATA

Run No.	Section	Vapor Velocity	$\frac{L}{V}$	Actual Plates	No. of Plates	Theo. Plates	Overall Efficiency	μ	α	$\mu\alpha$
49	Col.	0.86	1.00	9-1	8	4.52	57	0.33	4.7	1.54
50	Rect.	0.79	0.60	9-1	8	4.60	58	.30	2.7	0.81
50	Col.		0.60	11-1	10	6.60	66	.32	3.8	1.22
52	Rect.	1.00	0.90	9-3	6	3.48	58	.27	2.0	0.54
56	Strip.	0.49	6.40	7-1	6	3.40	57	.28	28.5	8.00
57	Strip.	0.50	8.90	13-3	10	7.00	70	.30	28.5	8.70
58	Strip.	0.37	8.70	11-7	4	1.95	49	.30	28.5	8.70
61A	Strip.	0.72	3.57	9-3	6	2.17	36	.30	23.8	7.26
62	Strip.	0.39	10.07	7-5	2	1.20	75	.33	26.15	8.63

in the overall figure may be the cause of the deviation. The average of overall and rectifier efficiencies was not as closely represented by the proposed correlation as were the results of Stigger's experimentation (6), but it is in the same region and, when compared with the previous data, falls within allowable limits of engineering accuracy. There is no correlation between stripping efficiencies and viscosity-relative volatility product except at low reflux ratios as in run No. 61-A. This fact substantiates the previous supposition that the effect of the $\frac{L}{V}$ ratio is of importance in determining plate efficiency.

METHYL ETHYL KETONE-WATER

For the inclusion in the correlation of a partially miscible system of a different type, and with a viscosity-relative volatility product in the uppermost region of the range to be covered, the system methyl ethyl ketone-water was chosen.

Because of the low solubility it was not possible to operate the column under rectification conditions without entering into a double liquid phase relationship. This necessitated the confinement of operations to stripping runs alone. Here again it was necessary to operate at a high feed rate in order to obtain good column distribution of components. This raised the liquid vapor ratio to a high figure, but under these conditions distribution was obtained over as many as eight plates.

In operation of the column on this system equilibrium conditions were extremely difficult to obtain because of the high sensitivity of the system to slight changes in calandria steam pressure. A slight drop in steam pressure caused depletion of liquid on the plates, while a slight upward variation caused build-up in the column. A high feed rate limited the time allowed for obtaining equilibrium conditions to not more than one hour, and made it necessary to discard much of the data for runs adversely affected by steam pressure variations.

In the low methyl ethyl ketone concentrations there was such a short range of variation that a highly accurate method of analysis was found necessary. Therefore, a freezing point method, using the Beckman freezing point apparatus and affording an accuracy of 0.02 mol per cent, was chosen and employed for analyses using data determined in the laboratory, Table V.

TABLE V. ANALYTICAL DATA, METHYL ETHYL KETONE-WATER

Sample	Weight Per Cent MEK	Mol Per Cent MEK	Freezing Point, °C.
1	0	0	0
2	3.22	0.83	-0.97
3	6.50	1.71	-1.89
4	9.84	2.66	-2.80
5	13.22	3.67	-3.67

The product was composed of two immiscible layers; its composition was estimated volumetrically for purposes of obtaining a material balance on each run. Actual product concentration, for purposes of calculating efficiencies, was calculated from a material balance over the column, from known feed composition and rate, and from effulent rates.

The low concentration of methyl ethyl ketone in the samples necessitated the use of the log-log extension (28) of the McCabe Thiele diagram for calculation of theoretical plates. Equilibria data for these determinations were calculated by the method of Carlson and Colburn (21) for immiscible systems, Table VI.

TABLE VI. VAPOR LIQUID EQUILIBRIA, METHYL ETHYL KETONE-WATER

X_A	Y_A	t_b	α
0.00	0.000	100.0	----
0.01	0.355	88.5	54.50
0.02	0.513	81.5	51.60
0.03	0.594	77.2	47.20
0.04	0.637	74.6	42.20
0.10	0.650	73.8	16.70
0.20	0.650	73.8	7.43
0.40	0.650	73.8	2.79
0.60	0.650	73.8	1.23
0.70	0.650	73.8	0.79
0.80	0.710	74.4	0.61
0.90	0.813	76.0	0.48
0.95	0.891	77.7	0.43
1.00	1.000	79.6	----

No consistent trend is indicated by the data, Figure 9, when presented on a viscosity-volatility plot, nor do the results of the system when so plotted approach O'Connell's correlation except in low liquid-vapor ratios. Therefore, one might infer that if rectification operation (where $\frac{L}{V}$ is less than one) were possible with this system, the correlation would be more nearly applicable (page 61).

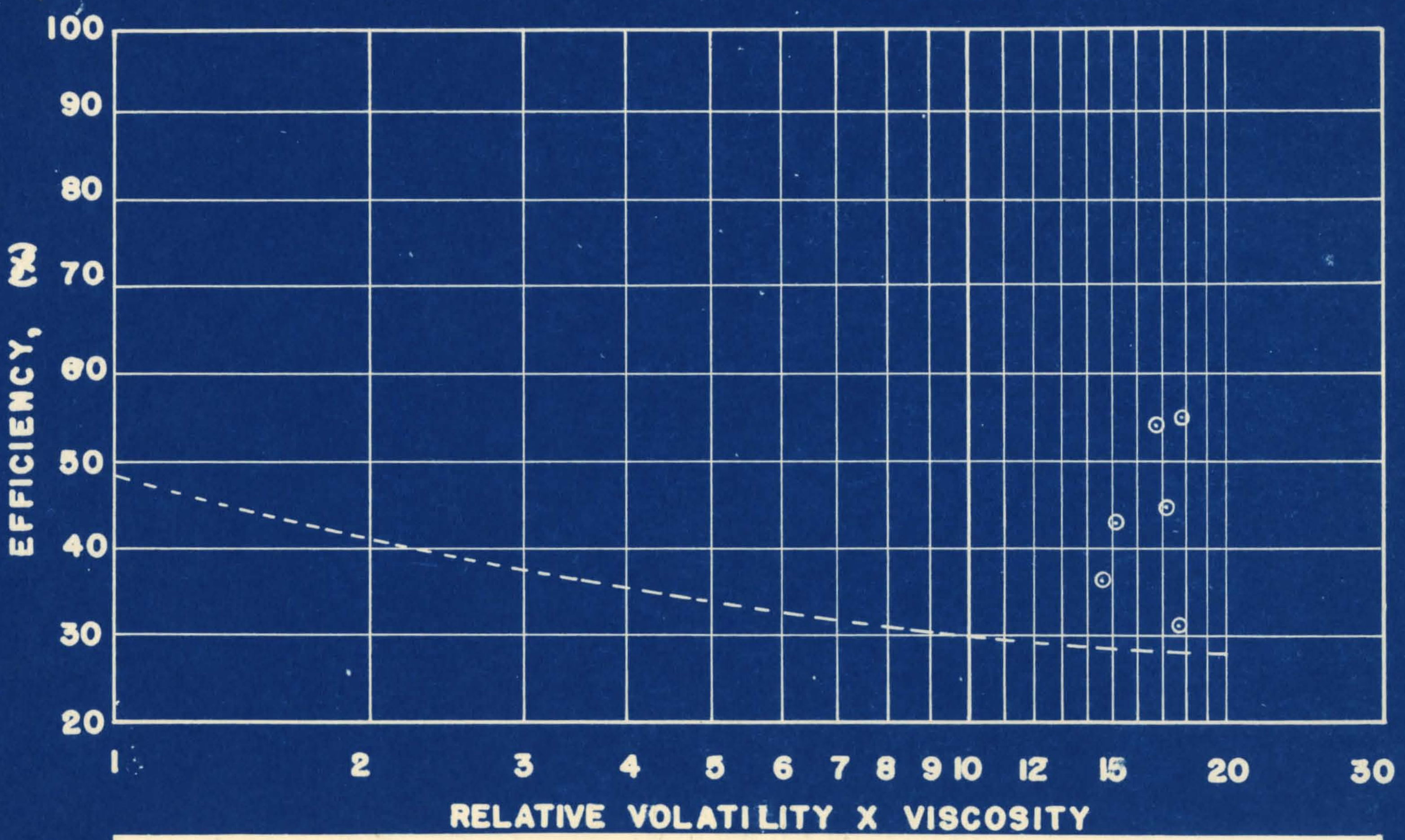


Figure 9 Effect of Relative Volatility and Viscosity on Plate Efficiency, Methyl Ethyl Ketone-Water System

TABLE VII. METHYL ETHYL KETONE-WATER, SUMMARY DATA

Run No.	Section	Vapor Velocity	$\frac{L}{V}$	Actual Plates	Number of Plates	Theo. Plates	Overall Efficiency	μ	α	$\mu\alpha$
63	Strip.	0.71	3.11	5-1	4	1.85	36.2	0.290	50.0	14.6
67	Strip.	0.29	12.60	11-3	8	5.60	55.0	.328	54.4	17.8
69	Strip.	0.71	4.52	9-5	4	1.45	30.8	.328	54.0	17.7
70	Strip.	0.64	7.47	11-5	6	3.40	45.0	.325	53.2	17.3
71	Strip.	0.46	7.95	7-3	4	2.83	54.3	.310	54.5	16.9
74	Strip.	0.20	5.8	9-3	6	2.87	43.3	.310	48.8	15.1

PENTANE-TOLUENE

This system was selected to represent the correlation in medium-high ranges of the relative volatility-viscosity product. It also joins an aliphatic and an aromatic compound and thus extends the type coverage.

Experimental work on the system was begun with a total reflux run. Subsequent runs were made at total reflux and at continuous column operation. Feed plate location, feed composition, effluent rates, etc. were adjusted as has been explained in the general experimental procedure. A high sensitivity to slight steam variations as was also encountered with the methyl ethyl ketone-water system, made it difficult to obtain equilibrium in each run. Feed plate mismatch was frequent and much resulting data were discarded; however several successful runs were completed.

By inspection of physical properties from data obtainable in the literature, it was evident that the simplest method of analysis which would afford an accuracy of 0.2 mol per cent was by density measurement with the Westphal balance. A density composition plot was made from data obtained in the laboratory, Table VIII, and used in the determination of sample compositions.

Calculations were based on equilibrium data calculated by the method of Lewis and Kay (21) using fugacities of the pure components and boiling point compositions estimated from Raoult's law, Table IX. The method of McCabe and Thiele (19)

TABLE VIII. ANALYTICAL DATA, PENTANE-TOLUENE

Sample	Wt. % Pentane	Mol Per Cent Pentane	Specific Gravity @ 22 Deg. C
1	0.00	0.00	0.8620
2	12.30	13.5	.8256
3	20.80	22.7	.7999
4	26.90	29.2	.7839
5	29.55	32.0	.7778
6	31.40	33.7	.7716
7	37.30	39.9	.7562
8	44.90	47.7	.7399
9	49.60	52.3	.7288
10	54.60	57.2	.7163
11	61.90	64.5	.7026
12	74.60	76.7	.6722
13	82.90	84.6	.6553
14	90.50	91.5	.6434
15	100.00	100.0	.6210

was used for determination of overall efficiencies using a 20" x 20" plot of the equilibria data.

TABLE IX. VAPOR LIQUID EQUILIBRIA, PENTANE TOLUENE

X_a	Y_a	t_b	α
0.0017	0.0126	110	6.65
.0520	.2710	100	7.10
.1080	.4730	90	7.45
.1910	.6400	80	7.85
.2820	.7640	70	8.17
.4100	.8550	60	8.52
.6030	.9280	50	8.70
.8580	.9790	40	7.13

Results for the system are presented in Table X and are shown graphically in Figure 10. With the exception of runs 79 and 83 the overall column efficiencies were in excellent agreement. It is further evident that these two runs have the greatest deviation from the correlation of O'Connell. It will be noted that these defficiencies are higher than for the other runs and represent runs in which the average $\frac{L}{V}$ ratio was correspondingly high. Rectifier efficiencies are well represented by the correlation, but except for run No. 84, stripping efficiencies deviate widely.

TABLE X, PENTANE-TOLUENE SUMMARY DATA

Run No.	Section	Vapor Velocity	$\frac{L}{V}$	Actual Plates	No. of Plates	Theo. Plates	Overall Eff.	μ	α	$\mu\alpha$
77	Col.	2.35	1.000	(11-3)	8	3.50	43.8	0.26	8.65	2.25
78	Col.	0.25	1.000	(13-7)	6	2.93	49.0	0.20	7.80	1.56
79	Col.	1.39		(19-3)	16	8.40	52.5	.27	8.75	2.36
79	Rect.		0.460	(13-3)	10	4.00	40.0	.22	6.90	1.52
79	Strip.		2.380	(19-13)	6	4.00	66.6	.27	7.35	1.98
83	Col.	2.90		(11-1)	10	5.60	56.0	.28	8.60	2.45
83	Rect.		0.596	(7-1)	6	2.70	45.0	.26	8.40	2.18
83	Strip.		1.730	(11-7)	4	2.75	69.0	.28	7.45	2.12
84	Col.	0.70		(19-5)	14	5.00	35.7	.27	8.60	2.36
84	Rect.		0.640	(13-5)	8	3.70	46.2	.22	7.30	1.64
84	Strip.		1.520	(19-17)	2	1.90	45.0	.29	8.40	2.48
85	Col.	1.14		(19-1)	18	7.70	42.7	.27	8.75	2.36
85	Rect.		0.561	(9-1)	8	3.20	40.0	.31	7.00	1.64
85	Strip.		2.25	(19-13)	4	2.7	67.5	.31	7.50	2.32

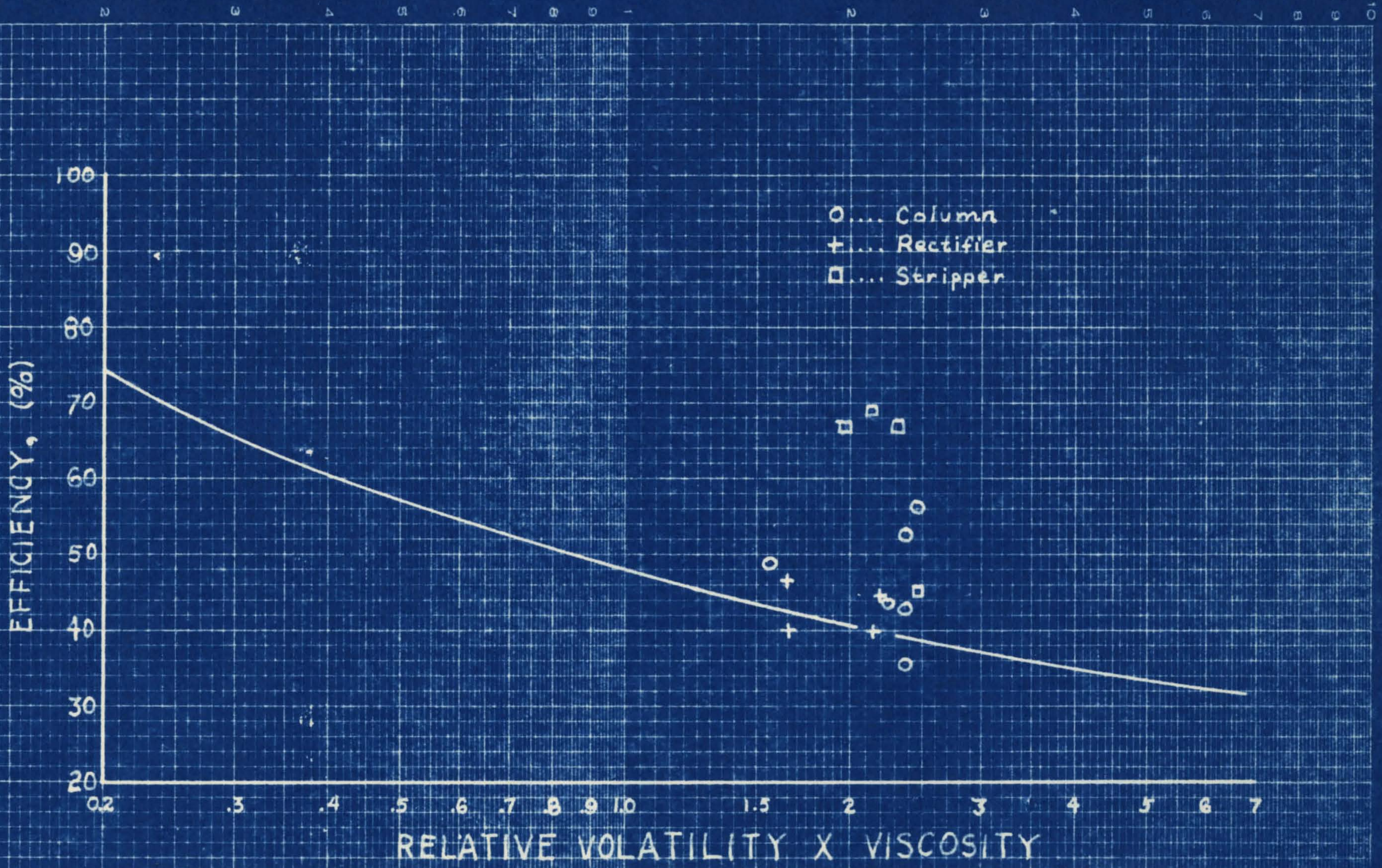


Figure 10 Effect of Relative Volatility and Viscosity on Plate Efficiency, Pentane-Toluene System

CORRELATION AND DISCUSSION OF RESULTS

Data have been collected from all the systems studied, both by Stigger and by this investigator, and presented in Figure 11 for comparison with the correlation developed by O'Connell. The range of the correlation has been extended to include relative volatility-viscosity products from 0.2 to 40.0.

The most widely divergent results represent operation at stripping conditions, and at overall column operation wherin large stripping sections were contained. This divergence has been repeatedly observed during the investigation of the various systems. On stripping sections the liquid-vapor ratio varied from one to values as high as 15, with a resulting proportionate increase in the divergence of results from those of rectification, where the reflux ratio was in the neighborhood of one. This observation led to the conclusion that the magnitude of the liquid-vapor ratio had a profound influence on plate efficiencies in distilling operations.

To illustrate this conclusion Figure 12 was plotted from data taken from rectifying sections of runs made on each system. The liquid-vapor ratio varied on these operations from 0.6 to 1.0, a narrow range as compared to that for stripping operations. The agreement of the results from data obtained from rectifying sections is excellent. It is approximated very closely by the curve of the correlation developed by O'Connell.

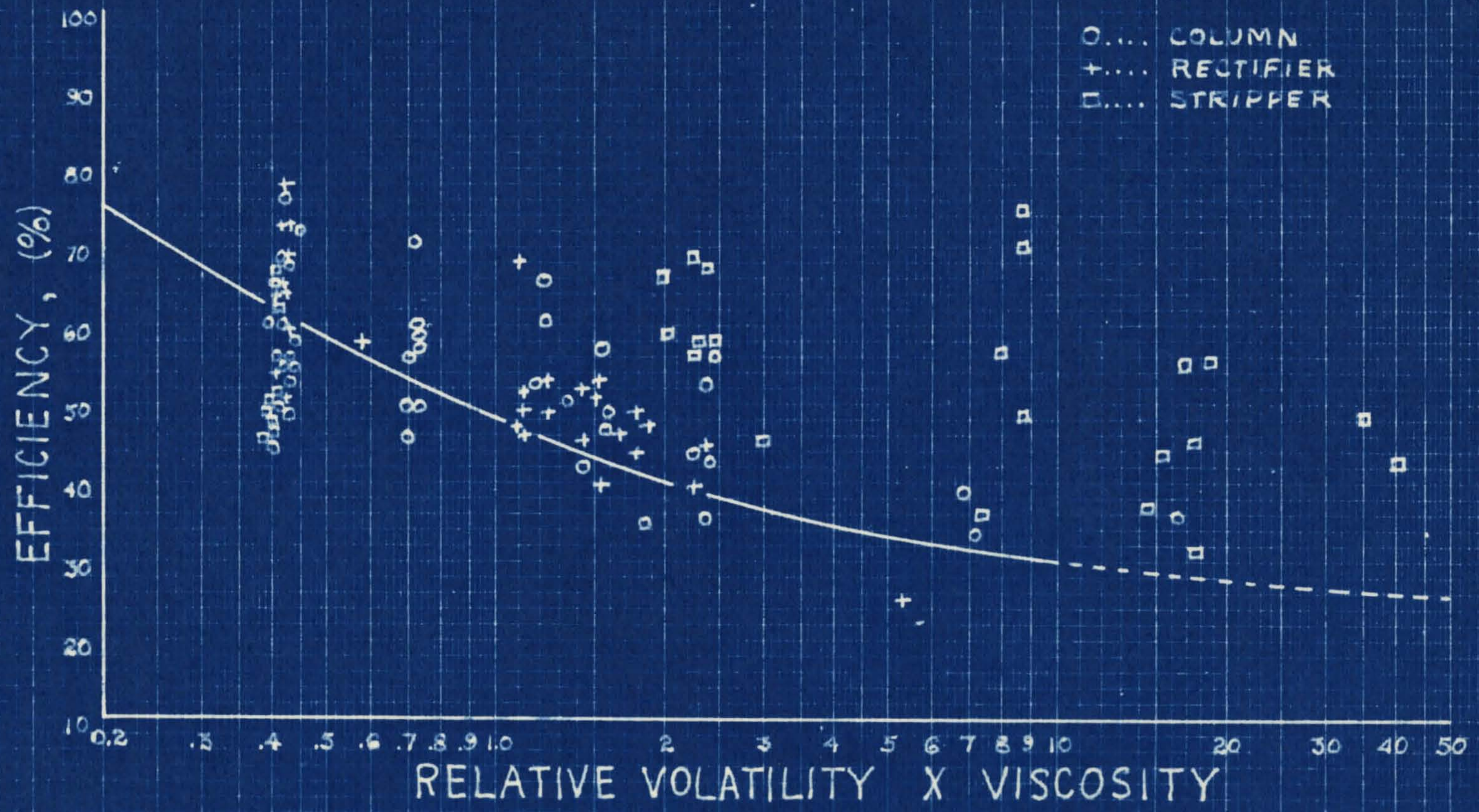


Figure 11 Effect of Relative Volatility and Viscosity on Efficiency, All Data, All Systems

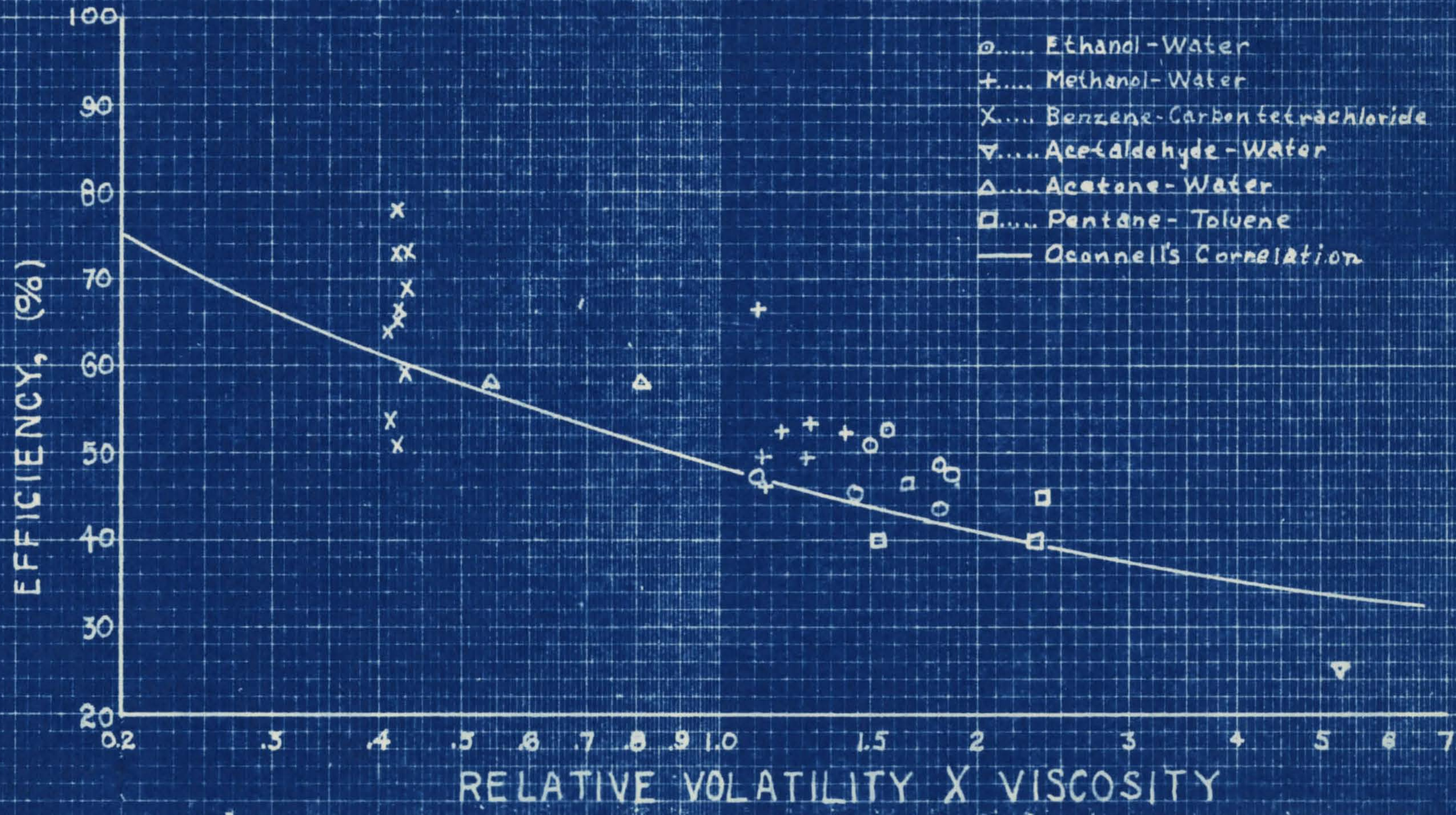


Figure 12 Effect of Relative Volatility and Viscosity on Rectifier Efficiency, All Data, All Systems

In contrast with these results Figure 13 shows no agreement between stripping efficiencies for all runs and the correlation proposed by O'Connell. This graphic representation showing the non-agreement of stripping efficiencies and their deviation from the correlation which holds for rectification efficiencies is striking evidence of the effect of the liquid-vapor ratio in fractionation efficiencies.

In order to determine the consistency of the effect of $\frac{L}{V}$ on the systems used in this investigation, it was decided to analyze the data for the five systems investigated by Stigger, as well as those included in this report. There follows a summary of the results of this analysis for those systems which yielded applicable data.

Recalculation of the results presented for methanol-water yielded liquid-vapor ratios for stripping sections as well as for the rectifier. This procedure made it possible to extend the relation, Figure 4, to include liquid-vapor ratios greater than one. The resulting plot of the efficiency as a function of $\frac{L}{V}$, Figure 14, indicates conclusively that it is quite definitely a factor in the determination of plate efficiencies. The data are not sufficient for a plot in which the $\mu\alpha$ product is held constant, but a trend is evident over the small range in these runs.

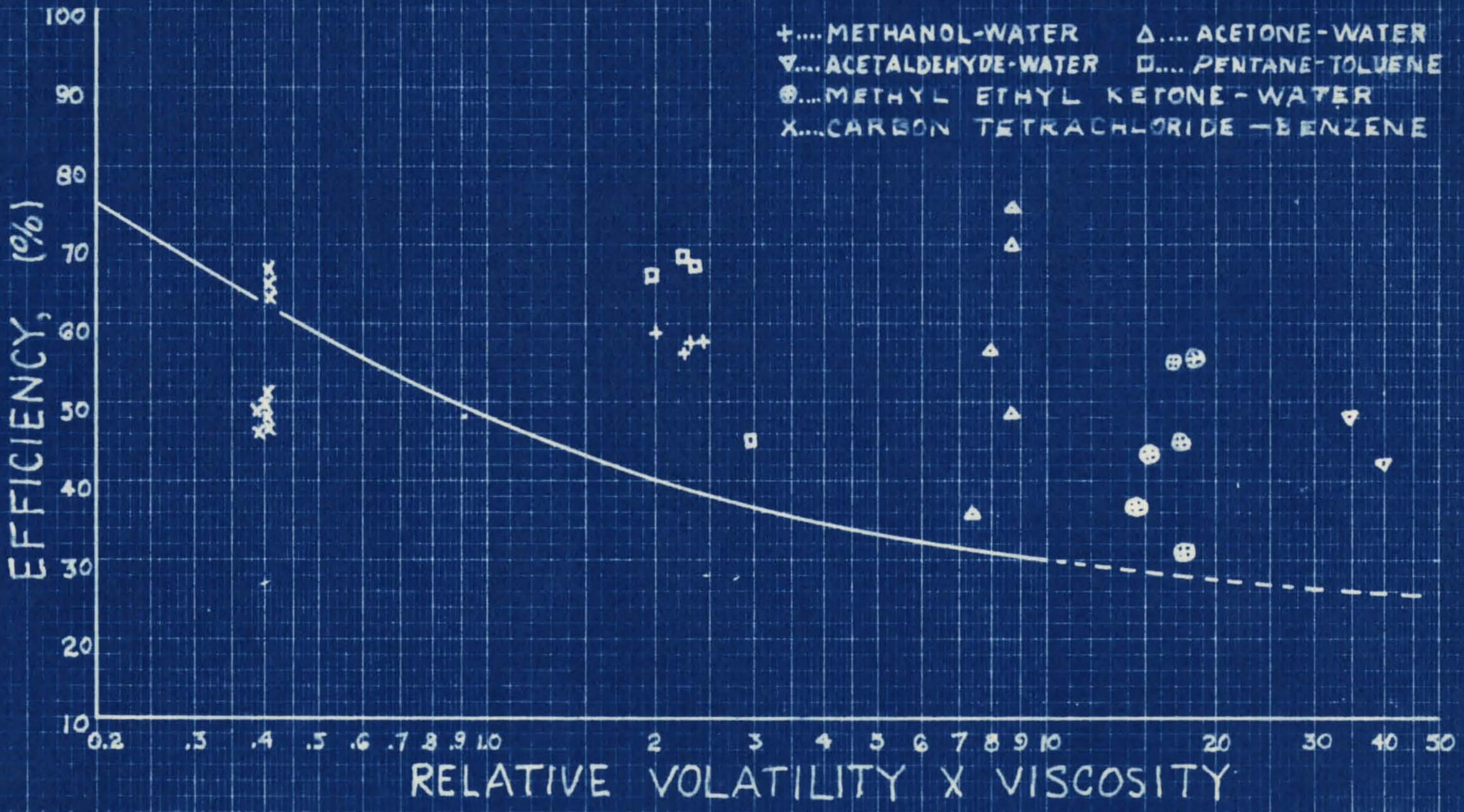


Figure 13 Effect of Relative Volatility and Viscosity on Stripper Efficiency, All Data, All Systems

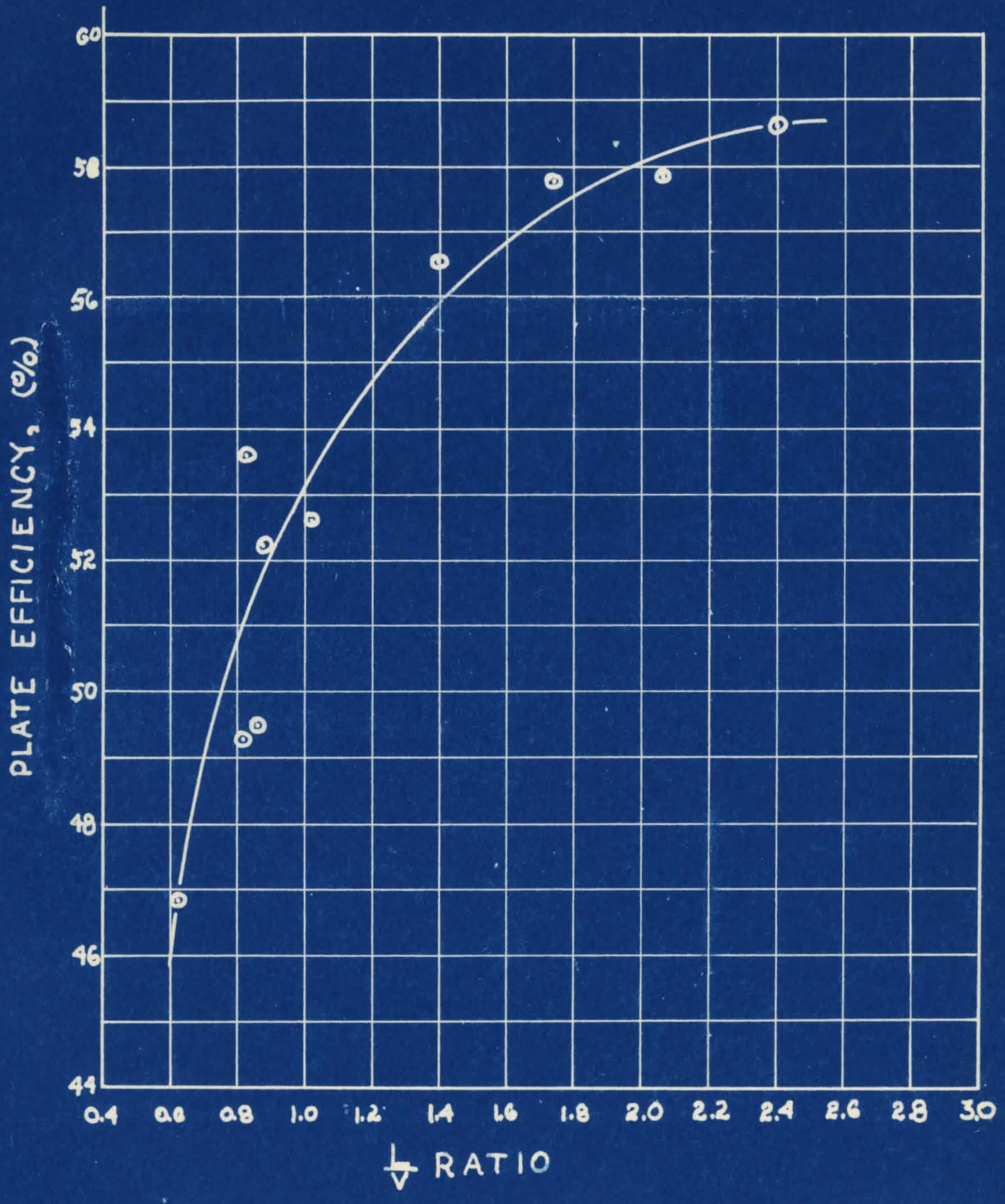


Figure 14 Effect of Liquid Vapor Ratio on Plate Efficiency, Methanol-Water

Although the magnitude of $\frac{L}{V}$ varied considerably with operation of the benzene-toluene system, no relation could be determined between this factor and plate efficencies.

Deviations from O'Connell's correlation were present in the results of the carbon tetrachloride-benzene system. Here there is a relation between these deviations and the $\frac{L}{V}$ ratio, but in an opposite direction from that of methanol-water. Confirmation of this effect is shown by the plot in Figure 15 where efficiency is represented as an inverse function of the $\frac{L}{V}$ ratio. Actual results for stripping conditions are below those for rectification, and an extension of the plot of Figure 15 would further substantiate this trend.

Extreme deviations were evidenced in the high $\mu\alpha$ ranges covered by data from the acetone-water system. In an attempt to find an explanation of the deviations, a plot is presented in Figure 16 of stripping efficiency versus liquid-vapor ratio in which a good correlation is obtained with the exception of run Number 58.

Because of the wide-spread range of the viscosity-volatility product over which the data were observed, no finite relation can be determined from the plot, but it is evident that the liquid vapor ratio does have a controlling influence on resulting column efficiencies in this $\mu\alpha$ range. The relation is of the same type

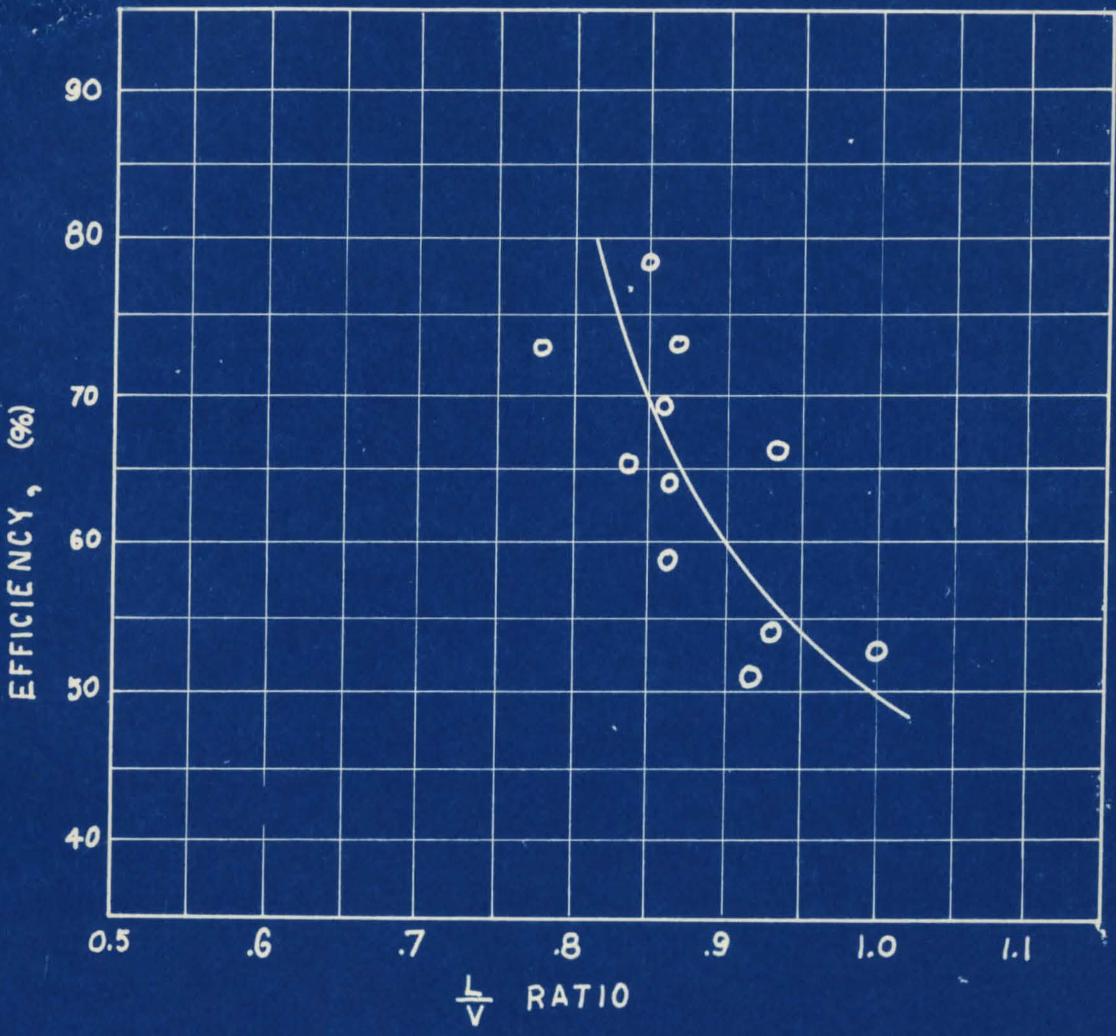


Figure 15 Effect of Liquid Vapor Ratio on Plate Efficiency, Carbon-Tetrachloride-Benzene

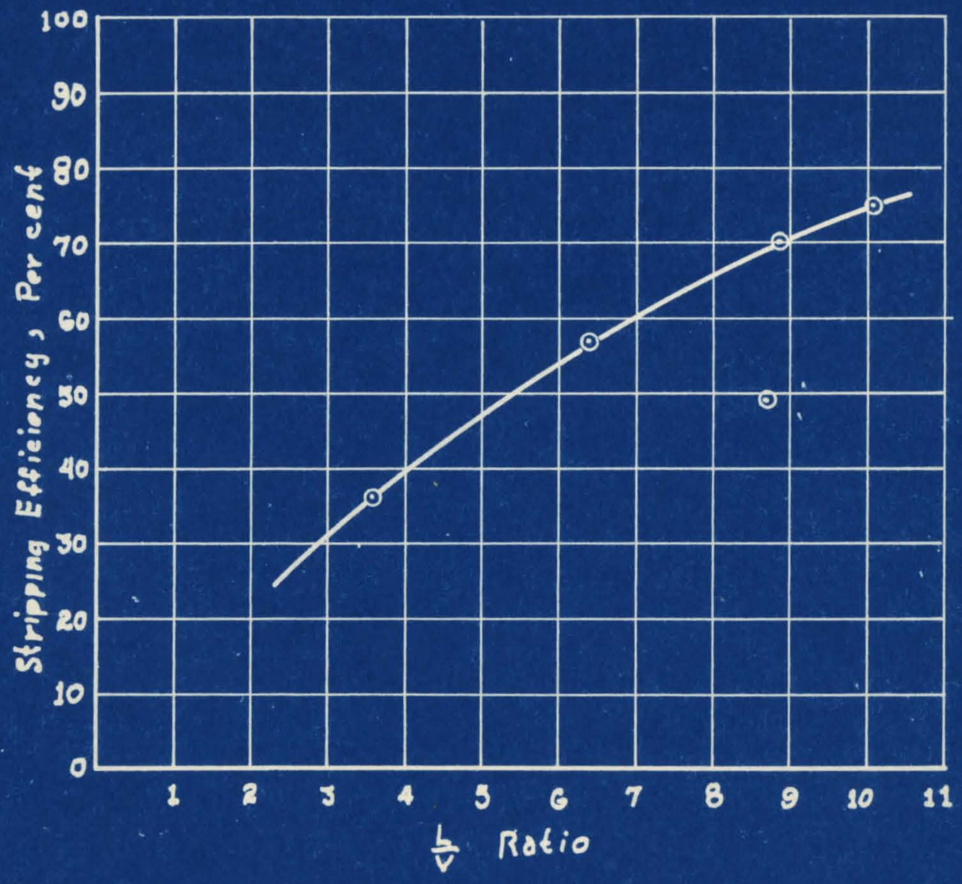


Figure 16 Effect of Liquid vapor Ratio on Stripping Efficiency, Acetone-Water System

as that for methanol-water. Again, the deviation of results of the methyl ethyl ketone-water system from the correlation of O'Connell was accompanied by a change in the liquid-vapor ratio. A plot in Figure 17 of stripping efficiency as a function of the liquid-vapor ratio shows a direct relation similar to those of methanol-water and acetone-water. In this case the range of variance of $\mu\alpha$ is small enough to be considered constant and the efficiency is therefore shown as a direct function of the liquid-vapor ratio. For this system no rectifier sections were included, and therefore all values of $\frac{L}{V}$ are greater than one, but it is interesting that an extrapolation of the $\frac{L}{V}$ curve to values of 1-1.5 result in estimated efficiencies of 20-30 per cent, a range of values which is quite in keeping with the original correlation, but not possible of demonstration by the experiments with methyl ethyl ketone-water.

For the system, pentane-toluene, a plot of efficiency versus liquid vapor ratio is presented in Figure 18. The data are inconclusive, but again follow the trend which has been shown in other high $\mu\alpha$ product materials.

The magnitude of the product of viscosity and relative volatility appears to have a direct bearing on the effect which $\frac{L}{V}$ exerts. That is, in the low ranges of $\mu\alpha$, below 0.7 centipoises, the effect of an increase

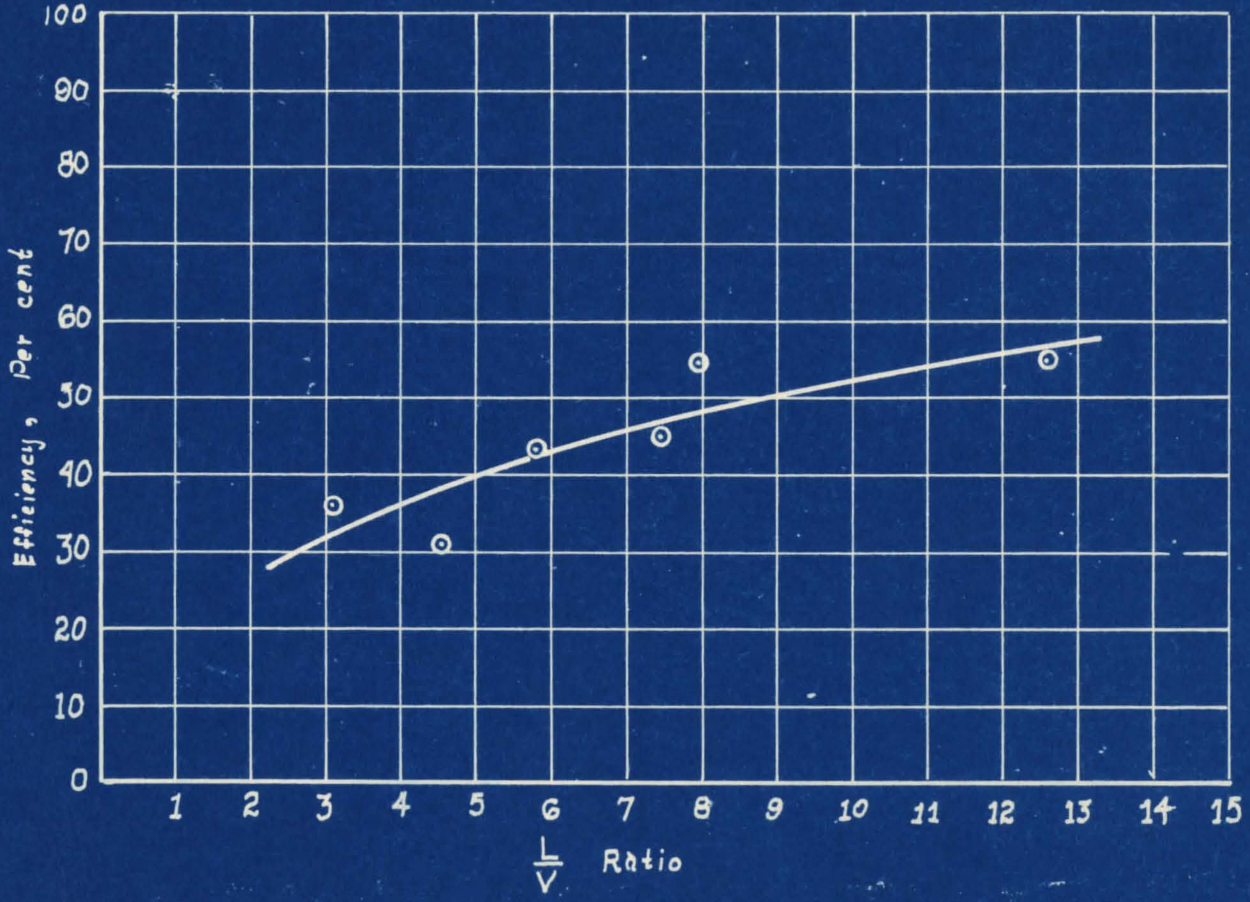


Figure 17 Effect of Liquid Vapor Ratio on Stripping Efficiency, Methyl Ethyl Ketone-Water System

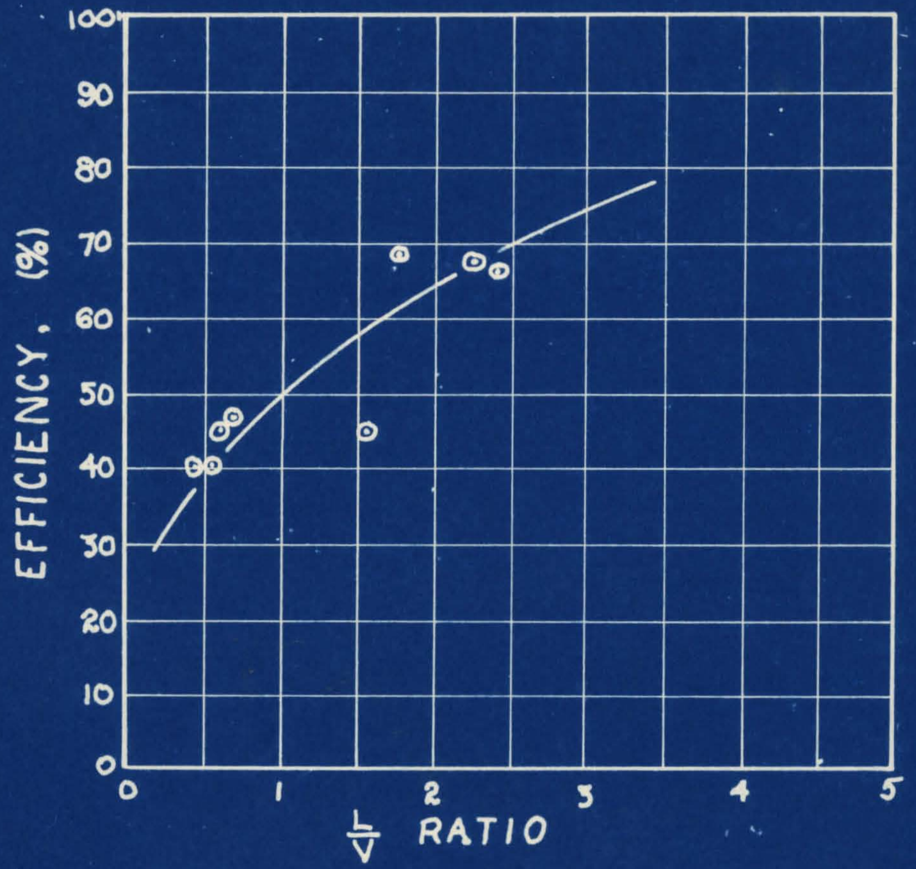


Figure 18 Effect of Liquid Vapor Ratio on Stripping Efficiency, Pentane-Toluene System

in $\frac{L}{V}$ may decrease the efficiency (benzene-carbon tetrechloride system). Then in the region where $\mu\alpha$ equals 0.7 the effect of $\frac{L}{V}$ may be negligible (benzene-toluene system). However for $\mu\alpha$ products above 0.7 the effect of an increase in $\frac{L}{V}$ is to increase the efficiency (all other systems in which $\frac{L}{V}$ varied considerably).

In view of the varied effect of $\frac{L}{V}$ on different types of binary mixtures and the wide range of the viscosity-volatility product involved, an attempt at a correlation in which the effect of $\frac{L}{V}$ is included is beyond the scope of the investigation.

In Figure 19 is shown a plot of overall column efficiencies for the systems studied. The plotted runs include rectifier and stripper sections. Agreement between these results is not as close as that between rectifier efficiencies, nor are they as well represented by the correlation of O'Connell. However, the data are in good agreement and are represented closely enough for engineering accuracy by the proposed correlation. The cause of the deviation of most of the results is that stripping section data were included in the calculations, since there in little agreement between stripping section efficiencies and the correlation of O'Connell, Figure 13.

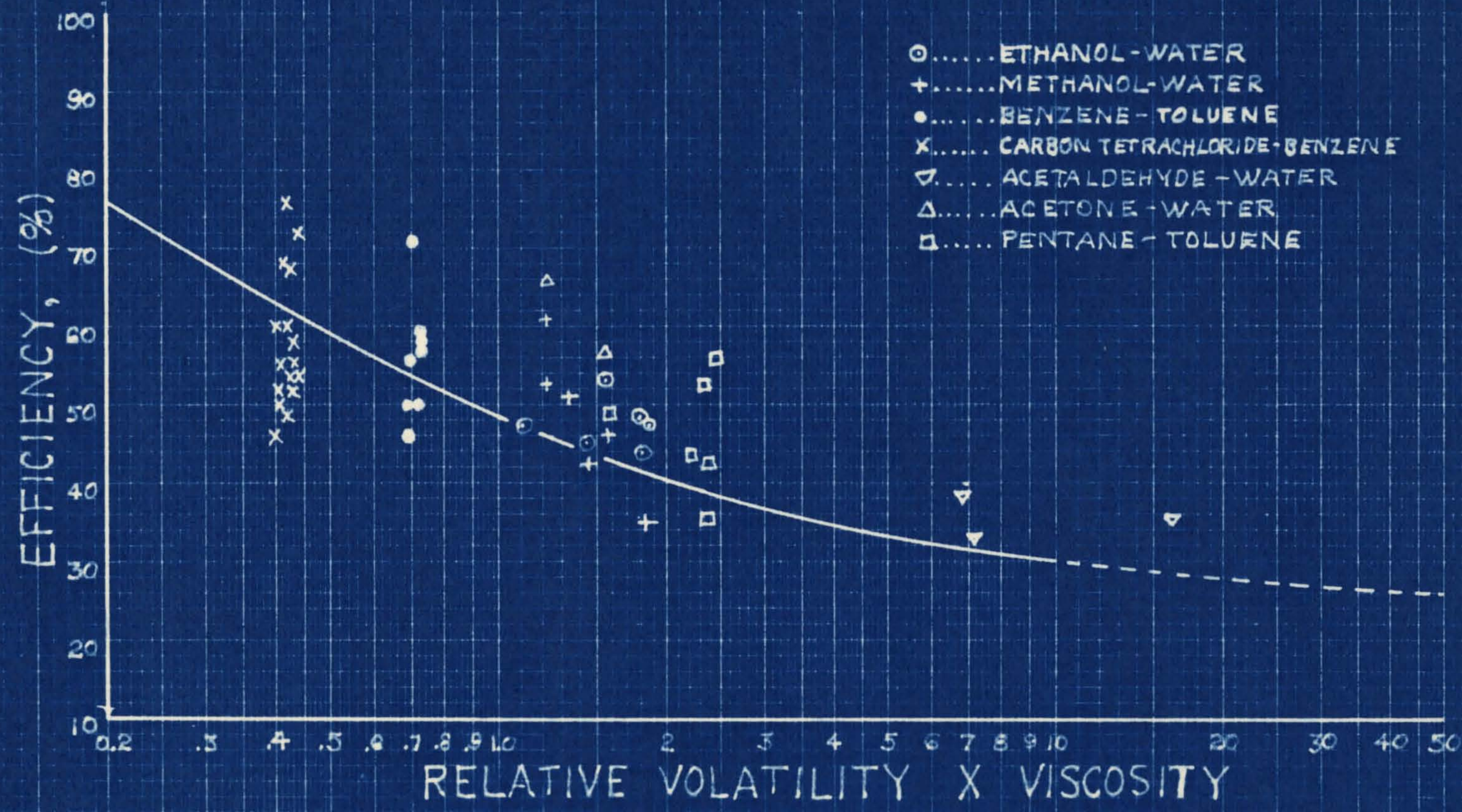


Figure 19 Effect of Relative Volatility and Viscosity on Overall Column Efficiency, All Data, All Systems

SUMMARY AND CONCLUSIONS

Overall plate efficiencies for fractional distillation of three different systems have been presented. Rectifier, stripper, and overall column efficiencies during total reflux and continuous operation were correlated on the basis of the viscosity-relative volatility product, calculated at an average temperature and composition in the respective portion of the column involved. Data obtained from these operations were used to complete an evaluation initiated by Stigger (6) of a correlation of column efficiency as a function of the relative volatility-viscosity product proposed by H. E. O'Connell (5). All fractionations were carried out in a twenty-plate, ten inch, bubble cap column, with four multiple slot caps per plate. Use of the single column minimized the effect of mechanical design factors on the results. The column was sufficiently large to be successfully used for pilot plant work and in many industrial applications.

Results of the investigation indicate that the correlation of O'Connell, although semi-empirical, may be used to predict overall efficiencies in commercial fractionators with certain limitations. For viscosity-relative volatility products of from 0.2 to 10.0, efficiencies of rectifier sections in binary distillation can be predicted within 25 per cent by this correlation. When used for prediction of overall column efficiencies between the above limits the result can be expected to be within a 35 per cent error. For predictions under conditions outside these limits, the correlation should be used with discretion. It cannot be used for stripping efficiency work where the value of the liquid-vapor ratio is high, without some modification to compensate for the influence of these higher ratios. This was indicated by the number and size of deviations in the value of the ratio $\frac{L}{V}$.

Definite relationships between plate efficiencies and liquid-vapor ratio have been established for all the systems studied in this investigation. Graphic representations have been given for these relationships. Analysis of the results reported by Stigger produced interesting relationships between $\frac{L}{V}$ and plate efficiencies which were different for different systems, and seemed to be a function of the $\mu\alpha$ product.

Though the correlation as presented has been found satisfactory for use in prediction of results on rectifier and overall column operations, there is an evident need for a modification on the basis of $\frac{L}{V}$ for stripping section work. Such a modification would also increase the accuracy of the predictions of rectifier and overall column efficiency and might result in a finished correlation of considerably greater value.

The correlation proposed by O'Connell has been evaluated and the types of systems have been expanded. Although the entire investigation involved the use of binary systems the expansion to include multi-component fractionations is practical, since the original development of the correlation included multi-component separations.

II. FACTORS AFFECTING PLATE EFFICIENCY

INTRODUCTION

Chemical engineering and the design of chemical equipment have been treated summarily in the literature, in accordance with the relative importance of the various branches. Even so, the individual branches of equipment design have not received the theoretical consideration commensurate with the thoroughness they deserve. One reason for the insufficient treatment of the all-important field of distillation is that manufacturing requirements have kept commercial practice well ahead of theory.

The purpose of this investigation is to combine and clarify some of the information presented in the literature and to present a method of attack for future research on the subject.

The necessity for an investigation of the factors which affect plate efficiencies obtained from bubble plate fractionators has been brought about by the increasing disagreement between investigators as to the effect of these factors, and the consequent lack of absolute knowledge. It is evident that the present information is insufficient to permit designs which would produce optimum results for distillation operations.

To alleviate this situation it was apparent that the necessary investigation should begin with a study of the fundamental principles of distillation and their application to practice.

The broad scope of the problem indicated that the work should include a thorough search of the literature to determine the points at which there is most disagreement as well as to obtain a general knowledge of data as obtained by other investigators and to facilitate the correlation of future results. It was the plan of the author that the conclusions of the literature survey be utilized in the design and operation of a basic, single slot bubble plate to be used for collection of experimental data.

HISTORICAL AND THEORETICAL

Presented in this section are discussions of the findings of investigators on a number of the factors affecting bubble plate fractionation efficiencies. For simplification and ease of reference these findings are presented under the descriptive title of each factor discussed. The factors investigated are presented in Table XI, but the position in the sequence is not dictated by relative importance or interrelationships.

TABLE XI, FACTORS AFFECTING PLATE EFFICIENCIES

1. Hydraulic Action
2. Concentration Gradient
3. Entrainment and Plate Spacing
4. Vapor Load (Velocity)
5. Reflux Ratio
6. Submergence
7. Liquid Concentration
8. Viscosity of Liquid
9. Relative Volatility
10. Mass Transfer Phenomena

HYDRAULIC ACTION

The design of any distillation unit in which there is to be liquid moving over a plate and simultaneously subjected to contact with vapor by a bubbling action, requires a careful consideration of the hydraulic factors incurred. However, keeping within the limits of good hydraulic stability, there are factors of design and of operation, which can be manipulated to give widely variant results.

Davies (23) recommended that a tray be designed initially on the basis of the mechanical factors. He derived the equation

$$\sqrt{\Delta}\left\{\Delta\left[3\left(\frac{n}{2}-1\right)+\frac{2}{1+\frac{1}{4}\beta^{2}}\right]+3n\left[d_o+s(\varphi-1)\right]\right\} = \frac{n\sqrt{n}\,\sqrt{1+\frac{1}{4}\beta^{2}}\,L_H}{2.4\,C_d\,l_1} \qquad (5)$$

where Δ = liquid gradient, difference in clear liquid levels between inlet and outlet sides of tray, inches.

n = number of rows of caps perpendicular to liquid flow.

$\beta = \left(\frac{b}{w}\right)_a$ = average of ratios of distances between caps and between risers.

b = distance between adjacent caps in a row normal to flow of liquid, inches.

d_o = total clear liquid depth adjacent to overflow weir, inches

s = cap skirt clearance distance of bottom edge of cap above tray floor, inches

$\varphi = \frac{l_2}{l_1}$

ℓ_2 = total free space between risers normal to liquid flow, average of various rows, inches

ℓ_1 = total free space between caps normal to liquid flow, average of various rows, inches

L_H = hot liquid quantity flowing across tray, gallons per minute at flowing conditions

C_d = liquid gradient constant at section d.

He correlated the data of Good and associates (11) by use of equation 5.

The plot of his correlation closely fits the data of Good, and predicts a rise of liquid above the normal quiescent liquor level. These data, from experiments on 3½ in. D. caps, four caps per row and six rows in a rectangular tray, with air and water, indicated that the hydraulic gradient across the plate was proportional to the liquid rate and also to the vapor rate. Good does not develop any equations for the phenomena but photographs show the results. When the hydraulic gradient becomes very great, the caps at the inlet side of the tray pass less and less vapor and finally become inoperative, allowing back flow of liquid through the bubble cap risers. Good likewise showed that the hydraulic gradient was higher for lower skirt clearance and a low seal, and that it was proportional to the number of rows of caps normal to liquid flow, i. e., to the baffle resistance offered by the caps and their positions on the plate.

Carey and associates (10) found that the slot opening (effective slot opening through which vapor is passing) increased with the rate of vapor flow. They plotted their results from rectification of binary mixtures as slot opening versus vapor rate on log-log coordinate paper and graphically obtained a slope of 2/3 in the range above one cubic foot per inch of slot width. Their work further showed that the depth of slot submergence varied directly with the vapor rate until the point was reached where the liquid "cones" back from the slots.

Williams and associates (24), from experiments on air and water with a single bubble cap, showed that frothing above the liquid was a direct function of the vapor velocity. Their work showed further that there was a point of vapor velocity (proportional to superficial column velocity) at which the actual velocity through the slot reached a maximum. At this same point the per cent of actual slot open to vapor flow increased sharply and the pressure of vapor in the bubble cap decreased. These phenomena were closely followed by a "coning" back of the liquid and consequently a reduced liquid head.

Peavy and Baker (25) showed that a splashing effect due to the velocity of the vapor was always present, and that at low liquid rates it actually decreased the liquid head by splashing over the outlet weir. To correct this effect they installed baffles above the outlet weir in the vapor area. These baffles prevented the splash-over and permitted the liquid to attain normal flow levels. Accompanying this was the formation of froth extending above the top of the weir, which has little effect on the clear liquor heights.

The area of contact of the bubbles is of importance in that it has a direct effect on the coefficient of mass transfer. Carey (10) predicted that a decrease in slot width would produce more and smaller bubbles and thus a greater surface contact area per unit volume. His concept was supported by experiments with ethanol-water distillation. Litwin (26) in other experiments reported an improved surface contact obtained in a cascade type column. Geddes (27) in his calculations of plate efficiencies, makes simplifying assumptions of bubble size, surface area, and time of contact. For bubble size, he assumes perfect spheres of a size dependent upon

Surface tension
Vapor density
Liquid density
Slot width
Slot openings

Where the bubble radius, $a = 0.78\left[\frac{\sigma(h_s + \omega)}{\rho_L - \rho_V}\right]^{1/3}$

He also assumes time of contact to be =

$$\frac{(\text{mean slot submergence})}{4a^{0.37}}$$

These references indicate the necessity of a study of the effect of slot height, slot width, submergence, liquid path, baffling, the radius of action of bubbles from the slot, turbulence, frothing etc., both on the mechanical aspects of gas-liquid contact and the kinetics of local efficiencies.

It is highly probable that these factors are mutually dependent, but the prime elements cannot be ascertained as yet. Until more experimental data have been obtained, either from the literature or by experimental work, the method of Geddes for determination of bubble size, surface area, and time of contact must suffice.

CONCENTRATION GRADIENT

The existence of this factor in bubble cap distillation work has been mentioned by many investigators and studied by a few. The concentration gradient between liquid entering and liquid leaving the plate has been shown to be the cause of plate efficiencies above 100 per cent (28). Liquid leaving the plate has a low-boiler content much less than the average of the total liquid on the plate and thus indicates a greater enrichment than that predicted from the vapor-liquid equilibrium of the downcomer liquid. This is the reason for calculated Murphree plate efficiencies above 100 per cent.

Kirschbaum (29) reported the existence of this factor and determined the magnitude of its effect. He pointed out that the most desirable liquid path over the plate is a cross flow, i.e., across the vapor flow, with as long a path of travel and as little mixing as possible. Lewis (30) presented a mathematical expression for the difference in efficiency in binary rectification resulting from various degrees of mixing of plate liquids.

$$E_o = \frac{\gamma - 1}{\lambda - 1} \qquad (6)$$

Where: γ is a function of the concentration gradients across the plate in both liquid and vapor phases and is determined by a graphical analysis.

λ is a function of the slope of the operating line and the slope of the equilibrium line

$$\lambda = \frac{\text{slope of equilibrium line}}{\text{slope of operating line}}$$

E_o = overall efficiency

From this he derived a relation between the overall efficiency and the number of required plates:

$$S = \frac{\ln \lambda}{\ln(1 + E_o[\lambda - 1])} = \frac{\text{no actual plates}}{\text{no perfect plates}}$$

Where: S = Number of plates required in a small section of McCabe-Thiele diagram divided by number of plates required in the same section when $E_o = 1$.

Peavy and Baker (25), observing plate efficiencies as high as 120 % in ethanol-water separation, measured the composition of the liquid at several points on the plate. They used an 18 in., seven bubble cap plate and from this calculated and plotted a curve of the composition of vapor rising from different points on the plate. Gerster and associates (15) estimated the magnitude of the concentration gradient by their "transfer unit" method of calculation. Langdon and Keyes (31) in a 5 in., 1 bubble cap column found plate efficiencies as high as 220%, and attributed this to the effect of the concentration gradient.

O'Connell (32) suggested an outline of a rigorous method of calculating column performances based on several factors of which concentration gradient was of prime importance. This method included a determination of liquid submergence over each bubble cap, and determination of the composition of vapor leaving the caps near the liquid exit weir, and a step by step calculation of vapor compositions over the plate by material balances based on semiemperical relationships which can be experimentally determined. These relationships would be evolved from data collected and correlated as suggested by Geddes (27). O'Connell further suggested that a correction factor for interchange of components between vapor leaving the plate and entrained liquid be incorporated, and that corrections for the degree of mixing and for carry over be included as well. This outline requires a large quantity of data not at present in an available form, and further requires correlations which can be applied in mass transfer predictions. At present the method is unwieldy and impractical.

ENTRAINMENT AND PLATE SPACING

The calculations usually made on the entrainment phenomena assume that entrained liquid leaving with the vapor is of the same composition as that leaving in the downtake. This is possible, but is not probably the case, i.e. (1) the mass of liquid on the plate has a composition higher in low boiler component than that leaving in the downcomer; (2) the liquid in the upper levels of the mixture on the plate can have a lower composition in the light key component than that in lower layers; (3) the liquid leaving with the vapor decreases in light key composition as it is carried aloft with the vapor.

Many investigators have shown the effect of entrainment on plate efficiency and have mads efforts to calculate its extent; Colburn (32) derived and presented the equation:

$$E_a = \frac{E_v}{1 + \frac{eE_v}{R}} \qquad (8)$$

Where E_a = apparent plate efficiency = $\frac{Yn - Y(n+1)}{Yn^* - Y(n+1)}$

E_v = dry vapor efficiency = $\frac{yn-y(n+1)}{yn^*-y(n+1)}$

y_n = mole fraction of low boiler leaving Nth plate

y^*n = mole fraction of low boiler in equilibrium with X_n

Yn = apparent composition of vapor = Yn-e(Xn+1-Xn)

e = moles entrained liquid per mole dry vapor.

He concluded that, from an economical standpoint, there is a small amount of entrainment that can be tolerated. The carry over data of Peavy and Baker (25) support Colburn's (33) findings.

Carey and associates (10) showed that the entrainment ratio was a function of the vapor velocity, and that increased distance between plates reduced its effect by lowering the carry over.

The entrainment was found to increase directly proportionally to the increase in vapor velocity. Plate spacings of 7 inches, 9 inches, and 15 inches were used for this investigation.

Gerster, Koffolt & Withrow (13) produced a correlation which suggests a logical basis for determining optimum plate spacing from these factors. A plot of $(PTU)_{ov}$ versus the vapor velocity, with the average slope of the vapor-liquid equilibrium curve as the parameter, gave a simple correlation of results at a constant vapor-liquid ratio. This method can be used to advantage in determining the allowable entrainment and thus plate spacing. Using a slightly different approach, Souders and Brown (8) developed their equation for allowable vapor velocity based on the quantity of entrainment that can be tolerated.

$$E = 1 - \left[\frac{(K)(1-f) + \left(\frac{e}{V}\right)(Kf-1)}{K - \left(\frac{x_p}{x_n}\right)\left(1 - \frac{L}{V}\right) - \left(\frac{L}{V}\right)}\right] \qquad (9)$$

where:

f = a factor which represents the degree of an approach toward equilibrium between dry vapor and liquid overflow leaving the plate, and is dependant upon a large number of variables including the mechanical design of the plate.

V = total mols of mixture of vapor and entrained liquid rising from plate to plate

e = total mols of entrained liquid carried with dry vapor from plate to plate.

VAPOR LOAD (VELOCITY)

A factor which has been extensively studied with respect to its effect on column performance is the vapor velocity. Carey and associates (10) as mentioned above, found that the slot opening (to vapor flow) increased with the rate of vapor flow. They observed that the liquid depth on the plates was a direct function of vapor rate, until liquid "coning" appeared. These findings were substantiated by Williams and associates (24), who found also that the radius of action of bubbles from the slot depended on the vapor rate. Then Souders and Brown (8) presented their equation predicting the maximum velocity allowable, equation (9).

Peavy and Baker (25) plotted efficiency versus vapor velocity for ethanol-water separation, indicating a pronounced effect. With velocity, the efficiency rose rapidly to a maximum, was constant for a period, and then dropped off. A maximum buildup of froth was evidenced in the maximum efficiency range. Brown and Lockhart (9) recommended allowable gas velocities according to the equation of Souders and Brown (8)

$$G = C\left[\rho_v\left(\rho_L - \rho_v\right)\right]^{\frac{1}{2}} \qquad (10)$$

Where G = mass velocity, lb/hr sq. ft.

C = empirical factor, a function of plate spacing and surface tension

ρ_v = density of vapor lb/cu. ft. and

ρ_L = density of liquid lb/cu. ft.

but states that values are essentially constant over a range from 0.4 to 1.2 times this value. The results agree with those of Peavy and Baker who reported constant efficiencies over a velocity range of 1.0 to 3 ft/sec. The work of Byman and Keyes (34) and Langdon and Keyes (31) showed parallel results over a range of 0.5 to 1.5 feet/sec. Stigger (6) found no effect of efficiency in vapor velocity variations between 0.7 and 1.0 feet per second.

The velocity of rise of bubbles through the liquid on the plate was calculated by Geddes (27) in his exploration of plate efficiencies. He used the equation of O'Brien and Gosline (35):

$$v = \frac{4a^{0.37}}{\mu^{0.08}} \tag{11}$$

Where a = the bubble radius, equivalent to a sphere, feet

v = velocity of rise in feet per second

μ = viscosity of liquid, centipoises

There is an apparent need for extended work on this factor, particularly with the effect of slot width and column vapor velocity on the velocity of rise of bubbles and the consequent time of contact with the liquid.

REFLUX RATIO

The effect of reflux ratio has generally been considered to have little or no effect on plate efficiency at values below one (8, 25, 31, 34). Contrary to this, the work of Gerster and associates (15) indicated that the number of plates equivalent to a transfer unit is a function of the reflux ratio.

An attempt was made to correlate the results of plate-to-plate Murphree efficiencies for typical runs, as given in their data, but it was not successful because of the scattering of points. The Plate Transfer Unit (PTU) however, when plotted against $\frac{V}{L}$ gave a smooth correlation, indicating an increase in efficiency with the reflux ratio below 1 (total reflux). This was not the only factor of importance because of the other variables, but its effect is startlingly apparent.

In stripping sections of the column, where the liquid to vapor ratio is greater than one, this investigator found a direct relation between $\frac{L}{V}$ and the plate efficiency, especially apparent with mixtures having high relative volatility (pages 52 to 64).

SUBMERGENCE

The effect of this factor on efficiency has been found to be of great importance. Most investigators have observed an increase in efficiency with increased depth of liquid, and Geddes (27) attributes this to a longer time of contact with the vapor bubble.

Equation (5) shows that the time of contact with the liquid is a direct function of the submergence because of a constant velocity of bubble rise.

Carey and associates (10) and later Peavy and Baker (25) obtained data showing increased efficiencies with liquid submergence. Good (11) showed that the submergence was a function of the liquid rate and vapor rate and the path of liquid flow.

Above the liquid is a layer of foam which aids in the separation process (25). Since this upper layer has a different consistancy from that of the liquid, it is conceivable that some degree of stagnation is present. The effect of this would be to present horizontal layers of liquid (and foam) having progressively lower low boiler contents nearer the top. This effect has not been determined and in fact has not been mentioned by many investigators. It will take some further study to find a method of determining the extent of this factor.

LIQUID CONCENTRATION

There is disagreement among investigators as to the effect of the concentration of the mixture being rectified on the efficiency of separation. Peavy and Baker (25) found no effect of concentration on efficiency between 5 and 55 mol % ethanol in ethanol-water separation. The work of Carey and associates (10) gave parallel results, and Johnstone and Pigford (36) observed a negligible effect of concentration.

These investigators found, however, that at the extremes of concentration, very high and very low, there was a decrease in efficiency. Inspection of the work of this investigator (pages 35 to 46) shows that, for mixtures of high relative volatility, stripping action was so high that data could not be obtained in stripping sections of the column except at extremely low concentrations of the more volatile component. This experience indicates that efficiency of separation is lower with low concentrations of more volatile components, at least in the high relative volatility range.

On the other hand, the data of Byman and Keyes (34) for ethanol-water fractionation and of Langdon and Keyes (31) for iso-propanol-water indicated a marked effect of concentration on the efficiency. Their maximum efficiency is obtained at approximately 20 mol per cent alcohol, with gradual to sharp decrease at higher and lower concentrations. Furnas and Taylor (37) observed a marked effect of concentration on efficiency in a packed column. An increased concentration was accompanied by an increased efficiency. They tentatively explained this as a result of an increased wetted area, caused by decreased interfacial tension in the liquid with greater concentrations.

Griswold and Stewart (38) obtained data from fractionations of benzene-toluene mixtures from which they concluded that the efficiency of fractionation was a function of the concentration. They found a decrease in efficiency of 0.6 per cent for an increase of one mol per cent benzene in the liquid.

There is an obvious need for a careful and exact determination of the effect of concentration on efficiency of fractionation. The one-slot method of examination is admirably suited for this type of study.

VISCOSITY

The background for a study of the effect of viscosity on plate efficiency was summarized by Drickamer and Bradford (14), and is shown on pages 13 to 14.

There is some controversy between investigators as to the observed effect of viscosity on efficiency. Byman and Keyes (34) obtained data on ethanol-water separation which indicated that the efficiency increased with increasing viscosity. Further data, obtained by Langdon and Keyes (31), gave the same results. Where maximum viscosity prevailed, the efficiency was also at its maximum.

Contrary to this, Walter and Sherwood (12) evolved a relation which indicated that the efficiency was inversely proportional to the viscosity to the 0.68 power, equation 12. In this the gas and liquid film resistances were proportional to the 0.68 power of the viscosity.

$$E_{mv} \quad 1 - e^{-\left[\frac{h}{\left(2.5+\frac{0.37}{HP}\right)\mu^{0.68}\,w^{0.33}}\right]} \qquad (12)$$

Where: E_{mv} = Murphree point vapor efficiency - $\frac{yi - yo}{yi - y^{*}}$

h = liquid depth from middle of slot to top of weir

H = Henry's law coefficient $\frac{\text{lb. mols}}{(\text{cu. ft.})\ (\text{atm})}$

P = slot width, inches

μ = viscosity of liquid, centipoise

w = slot width, inches

Drickamer and Bradford (14) developed a relation between viscosity and plate efficiency from data of 54 refinery fractionating columns. They presented their correlation of overall plate efficiency as a straight line function of the logarithm of the viscosity of the feed. In order to make this correlation more useful in design application, O'Connell (5) introduced the effect of relative volatility in interpreting the efficiency of fractionation. His correlation presented the efficiency as a function of the product of viscosity and relative volatility. That this correlation was satisfactorily representative of the data is shown by this investigator in part I of this report (Figure 19).

This factor again presents a promising field for investigation of effect on plate efficiency. Its effect can be studied with a fair amount of accuracy in a single plate, single slot, local efficiency investigation.

RELATIVE VOLATILITY

As stated by Othmer (39), "The simple operation of distillation is not related to diffusion in any important sense. It is the vaporization of volatile materials, usually to separate one from another. Equilibrium is static, diffusion dynamic." It seems that even in this stultified outlook the separation which theoretically can be obtained by distillation is ultimately influenced by the relative volatility of the components involved. The resistance to diffusion must enter to prevent attainment of this theoretical separation. O'Connell (5) and this investigator have shown, however, that the relative volatility of the components also affects the degree of attainment of theoretical separation (100 per cent efficiency). The O'Connell correlation presents efficiency as a function of the relative volatility and viscosity.

This effect may be explained as follows: Since there is little reason to suppose a change in the diffusional resistance coefficient with changes in relative volatility, it may be assumed that no change occurs. It may also be assumed that the time for diffusion of components to and from the vapor bubble will remain substantially the same regardless of the volatility. In a given time of contact a given number of moles of material are transferred and a definite separation is obtained. Now, with greater relative volatility the theoretical separation obtainable is greater, and the ratio of actual separation to theoretical (efficiency) is lower. Then if the relative volatility is lower, the possible separation less and the ratio of the actual separation (which has remained substantially unchanged) to the theoretical, is greater.

The data do not indicate that this effect is wholly realized, but the correlation of O'Connell supports the conclusion. That the effect is not as pronounced as the explanation indicates is probably because of the widely differing volatility of the components as they come in contact.

In the work of Gerster and associates (15), it was pointed out that the slope of the equilibrium curve had a direct bearing on the actual number of plates required per transfer unit. If the number of transfer units is equal to or has a direct relation to the number of theoretical plates required, then the plate efficiency may also be considered a function of the slope of the equilibrium curve, and hence the relative volatility, which is a mathematical representation of the slope.

MASS TRANSFER CONCEPT

The physical mechanism by which equilibrium conditions are approached is necessarily that of interdiffusion between the liquid and vapor phases. The method of molecular transfer between phases has long been assumed to occur across an interface (40) composed of a film of vapor in contact with a film of the liquid. In distillation work the net condensation of the less volatile component from the vapor within the interface and the net evaporation of the more volatile component within the interface takes place almost instantaneously.

There is, however, an opposition to the approach of molecules to the interface represented by the resistances presented by the liquid and vapor films. The net movement of the more volatile molecules to the interface through the liquid film and after vaporization through the gas film into the bubble is effected by the resistance of the respective films and to the driving force which causes transfer. This driving force is a direct function of the concentration differences across the films. The less volatile component is moving in a direction opposite to that of the more volatile and as the less volatile molecules condense at the interface they give up the heat required to evaporate a corresponding number of molecules of the more volatile component. It appears, therefore, that pure diffusion characteristics must be somewhat modified by the necessity of heat transfer.

These phenomena progress as a function of time and do not allow equilibrium to be attained in the short period of contact experienced by a bubble rising through a shallow tray of liquid.

FILM RESISTANCE

In the application of this factor to the determination of fractionation efficiency, it will be necessary to follow a method outlined by Geddes (27) and expanded by the data of other investigators. The data on film resistance in rectification are so meager in bubble cap work as to present serious difficulties. It will therefore be necessary to explore the data for other means of estimation and to apply methods to adapt these data to bubble cap work.

Geddes initiated this procedure and laid a valuable base from which to project the investigation.

Othmer (39) made attempts to measure the driving forces present in the actual film but was not successful because of the microscopic thickness of that film.

Other empirical and semi theoretical methods to develop a relation between film resistances and other variables have been attempted as follows: Walter and Sherwood (12) empirically fitted an equation to their data for bubble cap absorption. Their equation has been reported under "Viscosity".

Peck and Wagner (41) present a method for the determination of the relative resistance presented by liquid and vapor in distillation. Since their graphical analysis requires an operating line with a slope of one, their method of calculation confines their work to operations of total reflux.

Their results indicate that the resistance of the gas film is 50 per cent in bubble plate columns and 100 per cent in wetted wall towers. The data presented for rectification of Methanol-water, and isopropanol-water in a wetted wall tower check those obtained by Johnstone and Pigford (36) for distillation in a wetted wall column. There is no reason why different systems should have identical $\frac{K_g}{K_L}$ ratios, and the results should be accepted with caution.

Johnstone and Pigford (36) tried to allow in their correlation for the turbulence in the gas which is in contact with the liquid and for the wave motion and laminar flow in the liquid phase. With this purpose in mind they correlated their results on fractionation of four different binary systems on the basis of the average Reynolds number of the vapor stream relative to the moving interface. When correlated in this manner the data fell between the equations of Von Karman and Sherwood (42) and Gilliland and Sherwood (43) and the equation of Chilton and Colburn (44) for the diffusional phenomena in absorption work. A plot of

$$\frac{(HTU)_{ov}\left(\frac{\mu}{\rho D}\right)^{2/3}}{d}$$

is found to be a straight line function of the log. of the Reynolds number. (Where d is the diameter of the column.)

Taylor and Furnas (37) utilized the equation

$$K_L a = \frac{k_L a}{1 + \frac{k_L a}{m k_G a}} \qquad (13)$$

in an attempt to separate the resistances of the two films in packed tower distillation. Their results were not conclusive but indicated that the resistance resided primarily in the liquid film. This result is contrary to that of other investigators.

Surowiec and Furnas (45) used the same tower as used by Taylor (37) but without packing. For determining the resistance in the gas film they used the equation of Chilton and Colburn (16):

$$(HTU)_G = 10.9 \cdot d \left(\frac{dv\rho}{\mu}\right)_G^m \cdot \left(\frac{\mu}{\rho D}\right)_G^n \qquad (14)$$

and derived exponents from the data of Gilliland and Sherwood (43), producing $m = 0.17$, $n = 0.56$, where d is the diameter of the column in feet. They used for the liquid film their own derived equation analagous to Nusselt's work and adapted to mass transfer:

$$k_L = \frac{B'}{M} \left(\frac{D_L \rho_L^{5/3} \, g^{1/3} \, \Gamma^{1/6}}{3^{4/3} \, H^{\frac{1}{2}} \, \mu_L^{1/3}}\right)^{2/3} \qquad (15)$$

where:

B' = a constant

g = acceleration due to gravity, $ft/(hr)^2$

Γ = peripheral liquid rate,
lb/hr ft of wetted perimeter

D = diffusivity in square ft/hr.

H = height of column, feet.

Using this equation they determined that the resistance was largely in the gas phase as compared with the reverse results of Taylor (37). They reconcile this apparent disagreement by use of equation 14 which indicates that the gas film resistance is inversely proportional to the 1.17 power of the diameter of a given circular conduit. Thus the effective diameter of the packed tower is lessened by the presence of packing. These assumptions and rationalizations must also be accepted only as opinions in variance.

Colburn (46), in a study of diffusional processes, recognizes the 2-film resistance concept and presents an analysis using an HTU method of calculation. The overall HTU is equal to the gas film HTU plus the (product of the liquor film HTU and the ratio of the equilibrium curve to the slope of the operating line). This method has been applied to absorption calculations but will necessitate careful revision for use on plate distillation work. This procedure was followed to a great extent by Geddes (27) in application to his work.

MASS TRANSFER COEFFICIENT

The work of Walter and Sherwood (12) indicated a relation between the transfer coefficient and the ratio of the slot velocity to its area. Thus as the ratio $\frac{V}{A}$ increased, the coefficient of mass transfer increased proportionately. The magnitude of the increase is limited by the maximum value which $\frac{V}{A}$ can reach because of hydraulic considerations (23, 24). The data obtained on the effect of this factor were used in the evolution of equation 12.

Litwin (26) observed a material increase in the mass transfer coefficient when an increased surface area is exposed to the liquid in the cascade type still. The turbulence affected by the violent action is also a probable influencing factor. The increase in the value of the liquid film coefficient observed by Taylor and Furnace (37) is attributed to an increased area caused by an increased wetting of packing.

There is needed much explanation of these phenomena. A careful and planned research should bring into relief some of the factors which affect the mass transfer coefficient in bubble plate distillation work. The mass transfer coefficient is probably the most important part of this investigation, for it determines to what degree the theoretical separation of the operation will be obtained. A determination of the governing factors may show a way for new design which will materially increase the efficiency now obtainable.

DESIGN OF EXPERIMENTAL EQUIPMENT

From an analysis of the foregoing survey of the literature, it is evident that much is to be desired in the intelligent application of the material available for predicting operating characteristics in design of distillation equipment. In order to form a concise, understandable, and logical pattern from which an explanation of the exact effects of the various factors can be evolved, it is desirable to approach the problem from the standpoint of the basic relations of distillation. It has been suggested that such basic relations be evolved from experimental observation of operations on a single plate, single slot, bubble type rectifier. A design for such an apparatus was developed and is explained in the following paragraphs.

Because of the fundamental nature of the investigation of factors affecting plate efficiencies it became evident that special equipment would be necessary for the collection of experimental data. Accordingly, from suggestions evolving from discussions of the problem, models of two possible types of equipment were constructed of corrugated cardboard. Each of these models, figures 20 and 21, has a tray bounded by a vertical wall which separates the liquid and vapor. In the lower section of this wall is a slot through which vapor is allowed to pass. Liquid is to flow from the downcomer into the horizontal tray on one side of the vapor-liquid separating wall, past the slot, and over the level-control weir at the outlet end of the

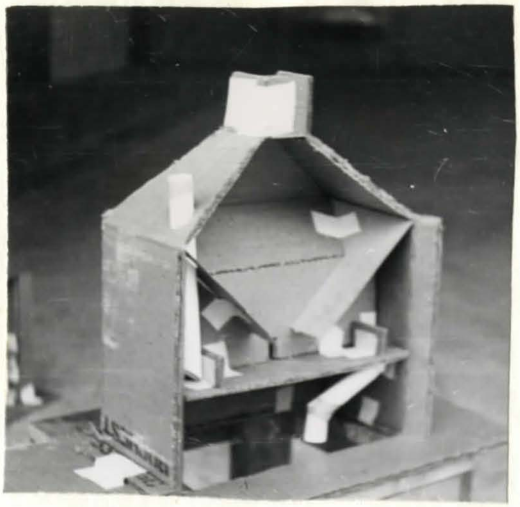
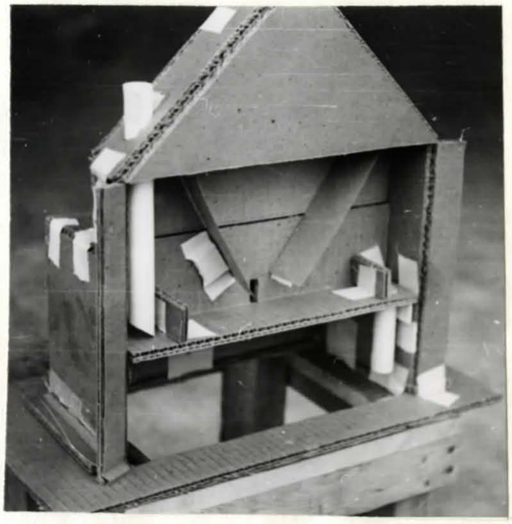


Figure 20 One plate, Single Slot Rectifier Number 1

Figure 21 One Plate Single Slot Rectifier Number 2



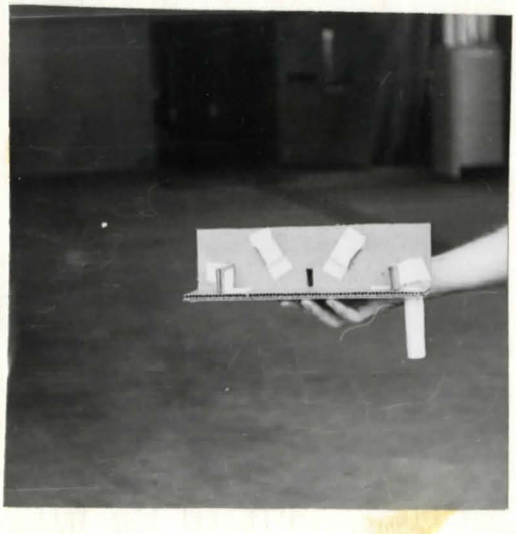


Figure 22 Removable Plate of Rectifier Number 2

tray. Vapor from the calandria is to flow through the slot and bubble through the liquid. It is to pass up from the surface and be collected in the uptake for subsequent condensing.

Above the surface of the liquid on the tray are two baffles whose purpose is to confine the vapor from the slot so that it does not come in contact with a liquid surface larger than that normally existing in a bubble plate column. In this way the effect of vapor and liquid contact at the liquid surface will more nearly approximate that in industrially designed trays. Inclusion of baffles will also eliminate the added effect of this surface contact and should make analysis of the resulting data simpler. It is planned to close the front of the experimental plate with a clear glass plate. This plate will be sealed to the column with a chemically resistant sponge rubber or other material, and clamped in place.

Liquid level will be controlled by an adjustable clip on the outlet weir. Slot dimensions can be changed by inserting different plates, Figure 22. The baffles can be adjusted to conform to changes in liquid level and to frothing characteristics.

DISCUSSION

The need for concentrated research on several of the factors which affect plate efficiencies is quite apparent. On the other hand, the determination of the effect of some factors has been fairly well completed.

The effect of variation in hydraulic factors is in some cases well established, but in other respects needs further study. For instance the effect of the hydraulic gradient, the bubble action, etc. on the degree of mixing, and consequently the concentration gradient, has not been determined. In addition to this it seems desirable to make a study of these factors for determination of a vertical concentration gradient extending into the foam above the liquid.

The exact effect of entrainment and of other factors on entrainment has not been determined from the standpoint of the nature of the mixture of entrained liquid and vapor.

There is a definite need for a study of the effect of reflux ratio, both in rectifier and in stripper operations. A study of the effect of submergence on the mass transfer coefficients and on the efficiency is also desired. The widely disputed effect of concentration of the liquid calls for an especially intense study. Of almost as great importance is the disputed effect of the viscosity of liquid on the plate.

A method of physical attack on the evaluation of mass transfer factors has not as yet been formulated. A mathematical approach is indicated, however, in order to predict the results to be obtained by experimentation.

The one-plate, single-slot rectifier proposed for an experimental investigation of effects of operating variables should be well suited to the nature of the work involved. It appears that such an investigation should proceed along the lines suggested by Geddes, in his paper presented on the factors which effect local efficiencies on bubble plates. The information and equations obtained from the literature can be analyzed and adapted for use with bubbling phenomena. One factor of considerable importance is the effect of turbulence in the bubble. This effect will be reflected by the magnitude of the transfer coefficients. It is conceivable that in a series of controlled experiments, simultaneous equations may be developed and some of the non-measurable quantities thereby calculated.

SUMMARY

A survey of the literature has been made to determine to what extent the data on factors affecting plate efficiencies can be correlated. Variables studied were, hydraulic relationships, liquid submergence, entrainment and plate spacing, concentration gradients across the plate, the average concentration of components in the liquid, vapor velocity and vapor-liquid ratio, average viscosity of the liquid, relative volatility of components, and the basic mass transfer relationships involved.

Consideration of the results of the survey led to the design of an experimental unit which can be used for obtaining basic data for prediction of local efficiencies from a carefully controlled manipulation of construction variables and operating variables and precisely measured physical characteristics. Photographs of models of the unit so designed have been presented.

III. PROPERTIES OF LAMINATED WOOD GLUE LINES

INTRODUCTION

The use of wood as a material of construction has been known to man as long as the art of construction itself has been practiced. There are several reasons for its wide use, among which are its inexpensiveness and availability, its versatility and adaptability, and its durability and resistance to weathering conditions. As a consequence of the high usability of wood, and its plentifulness in the past, there has been a great waste of valuable raw materials because of the haste of man to obtain the choice sections of the tree. In recent years constant efforts have been made to utilize more economically the total wood content of the tree, to improve the properties of the products made from wood, and to find new uses for these improved wood products.

One of the most valuable uses of wood is in the manufacture of laminated products. These are manufactured by the bonding of layers of wood by a suitable adhesive. Laminated wood has been made in many forms and shapes. It has been made in varying degrees of strength from thin, three-ply plywoods, one eithth of an inch thick, to multi-ply beams of greater than a foot thick. Under suitable conditions it can be formed into various and sundry curved shapes.

The use of laminated white oak veneers as a material for barrel staves was recently conceived and has been started commercially. These laminated members are designed to be used as a substitute for solid oak staves in the manufacture of barrels for the ageing of whiskey.

A factor which might limit the economical use of barrels, made by the above method, for the ageing of whiskey, is the variation in "outage". The "outage", or loss of liquid through the walls of the barrel, was found under certain conditions to be over twice that of the loss from a barrel constructed from solid oak staves. Since this factor was undesirable and placed a limit on the useability of the product, it was decided to make an investigation of the problem to obtain a more uniformly desirable product.

Extensive research had produced a bonding glue which was not harmful to the contents of a whiskey barrel. Since it was desired to continue the use of this adhesive, Penacolite G-1124, this investigation is directed toward the improvement of the physical properties of the glue line formed between wood veneers.

HISTORICAL

The properties of wood have been greatly enhanced by developing bonding glues and by their intelligent application. One of the most important advances in wood utilization as a structural material can be credited to the improvement in glues and gluing techniques.

It is important that the glue bond of laminated structures be as strong as the wood which is being held together throughout the service life of the material (49). A number of glues have been developed which fulfill these conditions under adverse conditions (50). Phenol-formaldehyde and urea-formaldehyde resins have come into use. The resorcinol-formaldehyde resin glues are rapidly coming to the fore (51, 52).

One of the resorcinol-formaldehyde resins, Penacolite G-1124 (53), was chosen for the fabrication of barrel staves because of its favorable behaviour when subjected to strenuous extraction tests (48). Under the established fabrication conditions, however, the product was not completely satisfactory from the standpoint of its permeability to water.

The permeability of wood to the flow of water was studied by Erickson and associates (55). They found that the rate of flow of water through wood decreased with continued flow and approached equilibrium after a definite period. Stamm and associates (56) studied the diffusion of water vapor and liquid through plywood specimens. Their investigation resulted in the conclusion that if the veneers are pre-im-pregnated with resin that there is a reduction in permeability of as great as 90 per cent.

It was suggested that the structure of the glue line itself be investigated in order to determine the reasons for the high permeability found in Penacolite-bonded white oak veneers.

Demarco and Smith (57) presented a method for the determination of the penetration of resin from the glue lines into the surface of veneers by color indicators. Farrow and associates (58) developed a more practical method in which the wood veneer is removed completely by digestion in an acid bath, leaving the glue film untouched for visual inspection.

THEORETICAL

The structure of wood has been found to be micellular. The cell size and density, the pit membranes and grain orientation vary with the type of wood and with its climatic environment (47). The normal trunk is swollen when green, with dead heartwood at its center and active fibrous cells in the outer part (59). The seasoning of wood removes this moisture and changes radically the physical properties of the seasoned product. The dry, cured wood adsorbs water, and is so swelled by it that its structure expands, allowing more surface for continued adsorption of water (47). Wood has been known to adsorb as much as 300 per cent of its dry weight.

Stamm (60) showed that moisture is carried through wood in three ways: (a) as a liquid, above the fiber saturation point, (b) as bound water within the cell walls, (c) as vapor, due to humidity gradients.

In resin-bonded veneers this flow of moisture is impeded to a greater or lesser extent by the presence of glue lines as barriers. At first glance it might seem that a resin, insoluble in water, and forming a continuous sheet at right angles with the flow of water, would present an impenetrable barrier to the flow. That this is not the case, however, is the reason for this investigation.

EXPERIMENTAL

STRUCTURE OF GLUE FILM

In an investigation of this type it was found desirable to make preliminary studies of the physical characteristics of the glue bond which is formed between veneers in the fabrication of laminated sections. Because the most reliable and the most emphatic method of presenting evidence is by visible means it was decided to use the method of Farrow (58) for visual examination of glue line structure. The procedure is given by Farrow in the following:

> "The present standard practice is to suspend the test specimen in hot chromic acid solution (100 grams of chromic acid in 100 cc. of water) with about 2.5 cm. (1 inch) immersed. The remainder of the specimen forms the support. Digestion is carried on at 80 to 85 deg. C. for about 20 minutes, which is sufficient to expose completely the glue lines in 3-ply 1/16-inch birch veneers. After careful digestion, thorough washing with hot water is necessary to remove the chromic acid and the caramel-like products of oxidation, which, if present, obscure glue line detail. The specimen is then rinsed with acetone and allowed to dry. Though complete removal of this sludge is very difficult in the case of fiber casts, normal washing and drying are rapid and permit immediate examination of the resin."

Two-ply sections of laminated rotary cut white oak veneer 3/16 inch thick were fabricated in a heated press at 240 deg. F. and 150 pounds per square inch pressure for eight minutes. The adhesive was Penacolite G1124 in four-to-one proportions of the prepared resorcinol resin and of formaldehyde catalyst, as recommended by the manufacturer. Specimens were cut from these sections and digested in chromic acid by the method of Farrow.

After nine hours digestion in 50 per cent chromic acid at 85 deg. C. it became apparent that no more than 50 per cent of the wood layers would be removed in this run. The hot acid attacked the outer layers of soft spring wood and removed them quickly; the hard summer wood was so slowly reacted that it was not removed even with nine hours digestion. The specimen was removed and placed in a bath of fresh 50 per cent chromic acid at 85 deg. C. This procedure caused all remaining wood to be removed from the glue film in less than one hour.

As a variation of the procedure a solution of chromic acid made up from fresh chromium trioxide in a ratio by weight of 40 parts to 60 parts of water. This solution was placed in a container, a new specimen suspended in the bath as before, and a total reflux condenser attached to the container. The bath was then heated to the boiling point and maintained at this temperature until all wood veneer was removed from the glue film. The time required for this and subsequent runs varied from 40 to 90 minutes.

All glue film specimens from the digested sections showed similar construction. There were long thin projections of resin, lying in planes parallel to the grain of the wood. These projections are indication of the penetration of the adhesive into the cracks inherent in rotary cut veneer. In the film itself were small, longitudinal perforations indicative of "starved" spots in the otherwise continuous film.

Quantitative data are not practical by this method except as to the depth of penetration of the adhesive. The projections showed that this depth varied from very slight penetration to depths equaling the thickness of the veneer itself.

PENETRATION OF GLUE FILM

At this stage of the investigation it became desirable to make a more nearly exact evaluation of penetration of the adhesive into the rotary cut white oak veneers being used in fabrication of laminated sections. Accordingly, another method of visual analysis of penetration was adopted. This method involved the use of a carpenter's plane for removing successive layers of veneer and the estimation, by visual inspection, of the area occupied by the adhesive in these successive layers. Thus a quantitative estimation of the degree of penetration could be obtained from knowledge of the adhesive present in adjacent planes of the veneer parallel to the glue line.

Specimens for investigation by planing tests were cut from five-ply, laminated staves fabricated at Gamble Brothers' plant in Louisville. These specimens were cut ten inches long and one inch wide from staves which were three feet long and one foot wide, having a radius of curvature of 1.5 feet. They were cut from various positions in different staves as is shown in Table XII.

Results of planing tests are shown by the curve of Figure 23. Though the utilization of a logarithmic plot for the representation of glue concentration at different planes exaggerates to some extent the accuracy of the estimation of low absolute glue concentrations, it enables an expression which most nearly represents actual conditions.

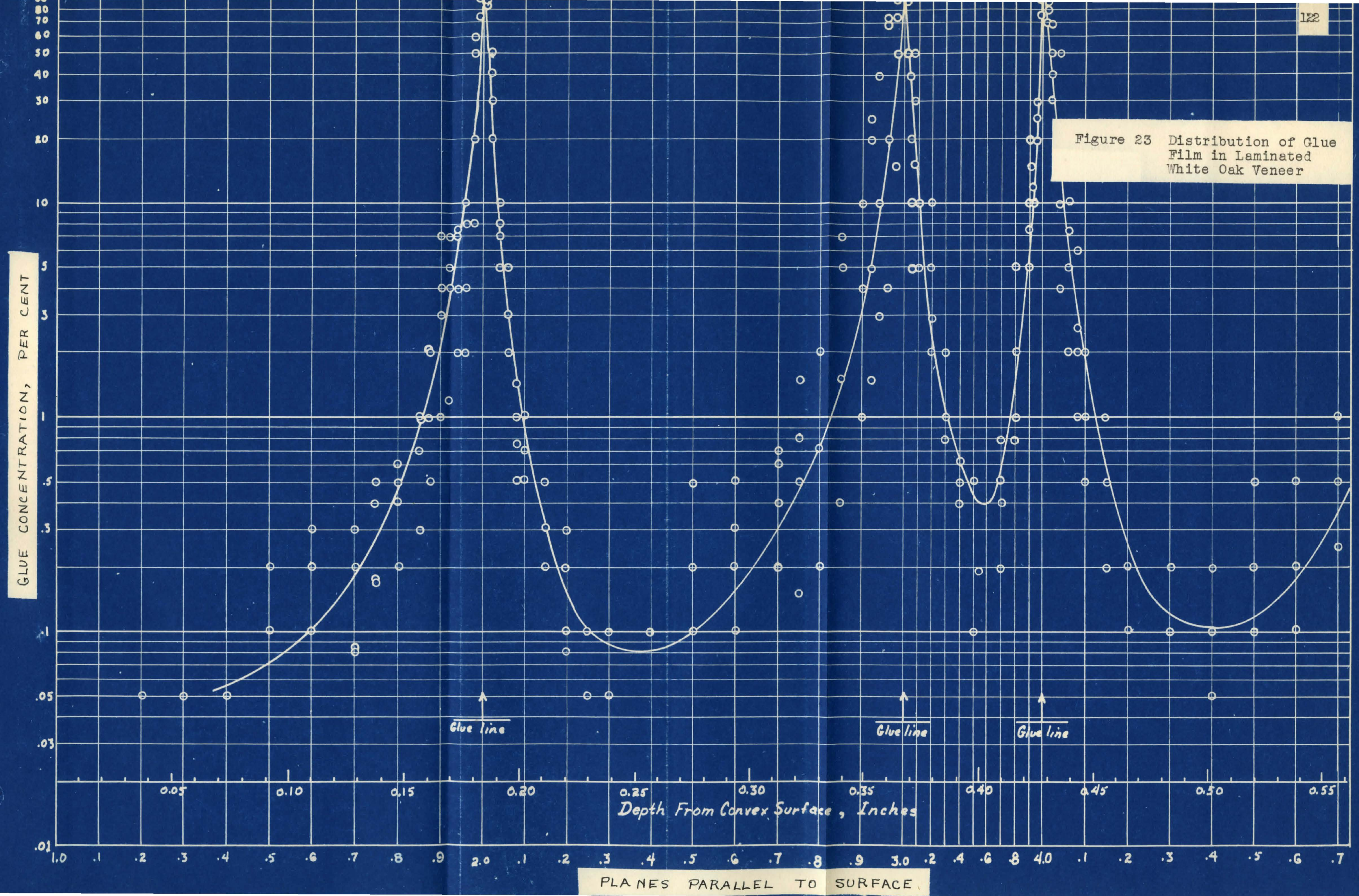


Figure 23 Distribution of Glue Film in Laminated White Oak Veneer

Furthermore this method of presentation produces a more easily interpreted relation. The laminated sections were divided into planes equidistant from one another, and are so represented in Figure 24. Veneers were numbered one to five, starting at the convex surface. Each veneer was equally divided into ten parallel sections which were further divided into ten sections each, all such sections being designated in figure 23 by their decimal equivalents. The center veneer, which is 1/16 inch thick as compared to the 3/16 inch thickness of the other veneers, is designated on the figure in like manner.

Data, represented by points on the plot, are the result of investigation of six specimens, each from a different stave (Table XII). Analysis of the plot shows that penetration of glue into the surface is small in quantity, but that the depth of penetration is extensive. From inspection of the data obtained on either side of the first glue line it is evident that glue penetration into the concave, open face of the first veneer was slightly greater than that into the smooth convex surface of the second veneer. In inner veneers penetration was slightly greater than that evidenced in those at the surface. The center veneer, which has its grain at right angles to that of the other veneers showed greater penetration into its convex surface than into its concave surface.

In the production of rotary cut veneer the cutting

TABLE XII. CHARACTERISTICS OF PLANING SPECIMENS

Specimen	Position in Stave
1	Center
2	Side at center
3	Side at center
4	Center at end
5	Center at end
6	Center

blade which removes the sheet from the rotating log causes a roughened and cracked surface on the concave side of the product. This is the surface which was more easily penetrated by adhesive (Figure 23). This was a logical development which has thus been substantiated by observation. On the other hand, the thin cross-grained veneer in the center, had its convex side in tension and its concave side in compression. One would expect a greater penetration to occur in the side which was stretched than in the other. That this was the case was substantiated by the data and is shown in Figure 23.

The original data indicated that greater penetration of adhesive is experienced in veneer sections at the center of the barrel stave than at outer edges. This variation may be due to slightly different curing conditions existing in different parts of the stave, to heat losses at the edges, and to the tendency of the resin to be squeezed out the edges of the stave while being pressed. These original data were considered too voluminous to be included in this work in tabular form, but have been shown graphically in figure 24.

RESISTANCE TO LIQUID DIFFUSION

Having determined the general nature of the physical structure of the resin film existing in glued white oak laminates it became desirable to make a quantitative determination of the rate of diffusion of liquid water through a number of specimens. In order to determine the relative resistances to diffusion presented by specimens fabricated under different conditions, it was planned to carry out diffusion measurements under similar conditions for each specimen.

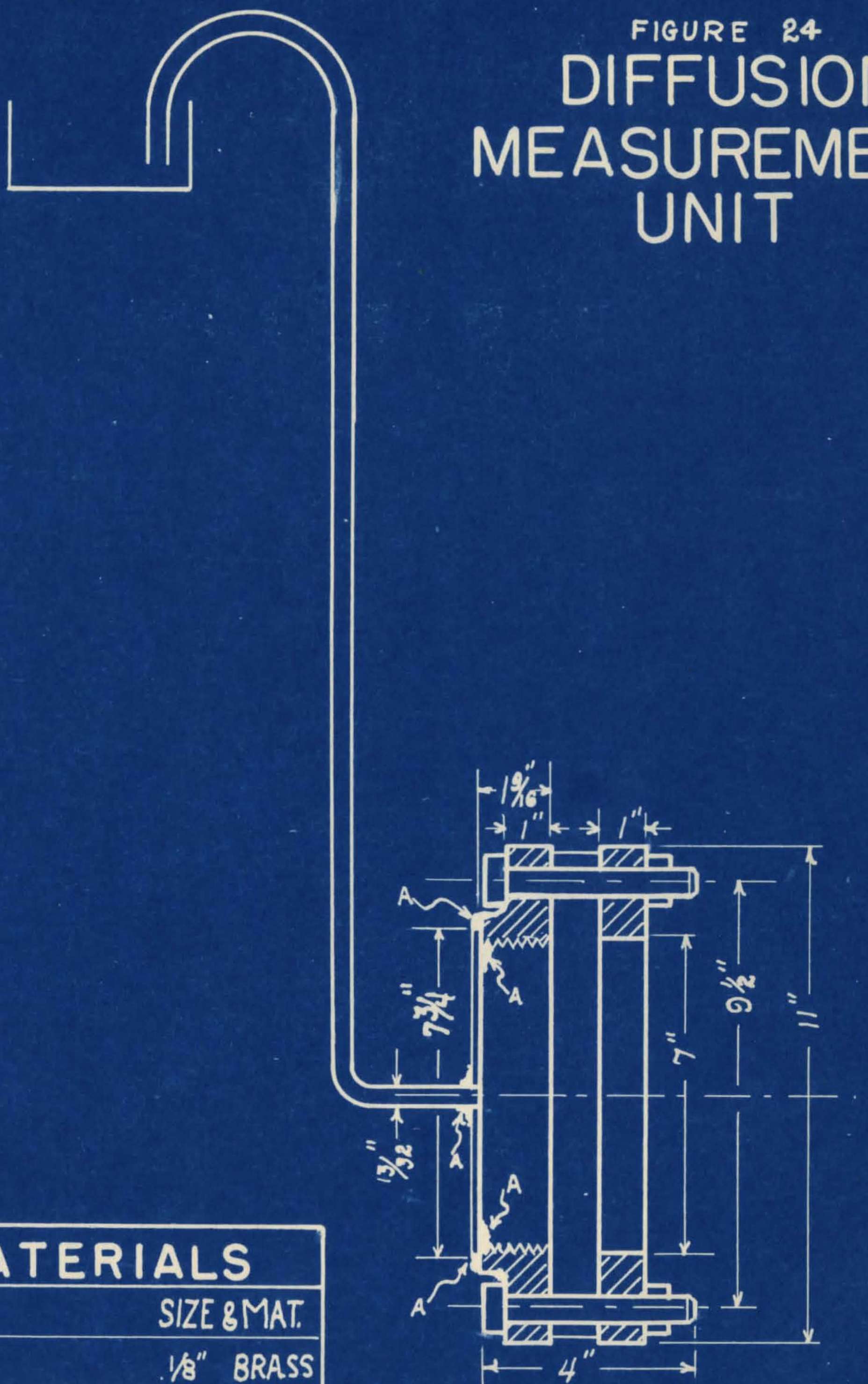
An experimental unit was designed and constructed as shown in Figure 24 for measurement of diffusion of a liquid through a flat section of laminated veneer into the atmosphere. The liquid pressure exerted against the surface of the specimen was fixed by the heighth of liquid in the unit above the plane of the specimen.

Procedure.

In preparation for a test of glue film diffusional resistance, a specimen was fabricated under a set of conditions as is shown in Table XIII. For runs number one and two, the specimen was placed in the diffusion unit head and sealed by application of a glyceryl phthalate resin. This method proved unsatisfactory because the air-drying resin took an excessively long drying period. For subsequent runs the specimen was edge sealed with three or more coats of

FIGURE 24

DIFFUSION MEASUREMENT UNIT



LIST OF MATERIALS

NO.	NAME	SIZE & MAT.
LTH.	PIPE	1/8" BRASS
1	SCREWED FLANGE (25 POUND), DRILLED & FACED	6" CI
1	PLATE	1/8" BP
8	BOLTS	3/8" I
1	FLANGE RING	6" CI

POINTS "A" TO BE BRAZED

SCALE: 1/4" = 1"

TABLE XIII. DIFFUSION TEST SPECIMENS

Specimen Number	Precure Time Hours	Curing Conditions			Wood Rays per inch
		Pressure psi	Temperature Degrees F.	Time Min.	
1	0	150	240	8	
2	0	50	250	8	
3	0	50	250	8	
4	0	50	250	8	
5	0	50	250	8	
6	24	50	250	8	
7	0	50	250	8	12.5
8	0	50	250	8	14.5

the glyceryl phthalate resin with a low-temperature (180 deg. F.) cure between each application. To prevent subsequent dimensional changes in the specimen after being placed in the unit and while it was being saturated with water, the specimen was next immersed in water for a pre-soak period of approximately 24 hours. The specimen was then placed between the flanges of the diffusion unit head and sealed with a coarse parafin-impregnated thread. The diffusion unit was then assembled as shown in Figure 24 and filled with water; all air was vented from the system and the syphon started. The volume of liquid was then brought up to the design level. Flow measurements were made by observation of the amount of decrease in the liquid content of the system during a definite time interval. At the end of each time interval (usually one day periods) the volume of liquid in the unit was increased to its initial level by admission of a measured quantity of water from the burrette. This measured quantity (after temperature corrections) represented the flow of liquid through the exposed surface of the specimen during the elapsed time interval. Such data were recorded at the end of each interval. After sufficiently constant flow was reached, the run was considered complete and the data were used in flow rate calculations.

Results were calculated by setting up a weight material balance across the system, including corrections for changes in density of the water (causing erroneous volumetric readings) with temperature variation. The following equation was utilized:

$$W_1 \quad w_2 = W_2 \quad w_D \qquad (16)$$

Where W_1 is the weight of water placed in the unit initially

w_2 is the weight of water added at time of reading to replenish the total contents to the initial volume

W_2 is the weight of water in the unit after w_2 has been added

w_D is the weight of water diffused through the specimen during the elapsed time interval

From this equation the average rate of diffusion on an incremental basis was calculated by the following relation:

$$D_1 = \frac{w_D}{(t_2-t_1)\ (A)} \qquad (17)$$

Where D_1 = Average diffusion rate for the interval t_1 to t_2, pounds per hour per square foot

t_1 = time of reading

t_2 = time of reading at end of first interval, (t_2-t_1) is the period over which diffusion w_D occurred, hours

A = cross sectional area of specimen through which diffusion occurred, square feet.

DATA AND RESULTS

Each specimen subjected to diffusion tests was fabricated under the conditions listed in Table XIII, and was cut from a two-ply section of 3/16 inch white oak veneer, laminated with grains parallel. The adhesive used exclusively was Penacolite G-1124 in a ratio of four parts of resin to one part of formaldehyde catalyst. Each specimen was flat and octagonally shaped, measuring seven inches across.

Results of the diffusion tests were plotted as a function of time. Figure 25 shows a typical curve for the accumulative flow of water throughout the duration of the run. This plot represents the data obtained from tests of specimen Number 5. The slope of the straight portion of the curve represents the average rate of flow in pounds per square foot per hour for the duration of the run in question. Figure 26 shows accumulative results of all runs plotted in the same manner as were the data for specimen Number 5. The data are now shown by individual points on the plot to avoid any unnecessary confusion.

Figure 27 is the result of a plot of incremental flow rates determined at the time of each reading. These data also represent tests on specimen Number 5 and show the relative deviation of each result from the average rate of flow. Shown in Figure 28 are the average rates of flow for all specimens tested. Here again the individual data are not shown in order to avoid confusion.

Three runs were made with specimen number one which was fabricated under conditions given in Table XIII. The first two of these runs utilized a three foot heat of water. The method of flow measurement for these first two runs was not as accurate as that with subsequent runs because of initial difficulties in setting up the apparatus. Furthermore the amount of diffusion under the three foot pressure head was too small to magnify any experimental errors. Therefore, the data were inconsistent, and the reported flow rates of .00009 and .00035 pounds per square foot per hour should be considered as approximate. The third run with specimen number one was carried out under a 13-foot head of water and the standardized method of measurement using the apparatus as shown in Figure 24. The average flow rate for this run was 0.00078 (Figure 26).

Also represented in Figures 25 and 26 are the results for subsequent specimens whose curing conditions are shown in Table XIII. Average results of all diffusion tests are recorded in Table XIV.

The consistency of the data obtained for each run is excellent in view of the several factors which might cause deviation. (a) Although the calculations included the effect of temperature variation on the volume of fluid in the unit, they did not compensate for the variation in the volumetric capacity of the unit itself with temperature

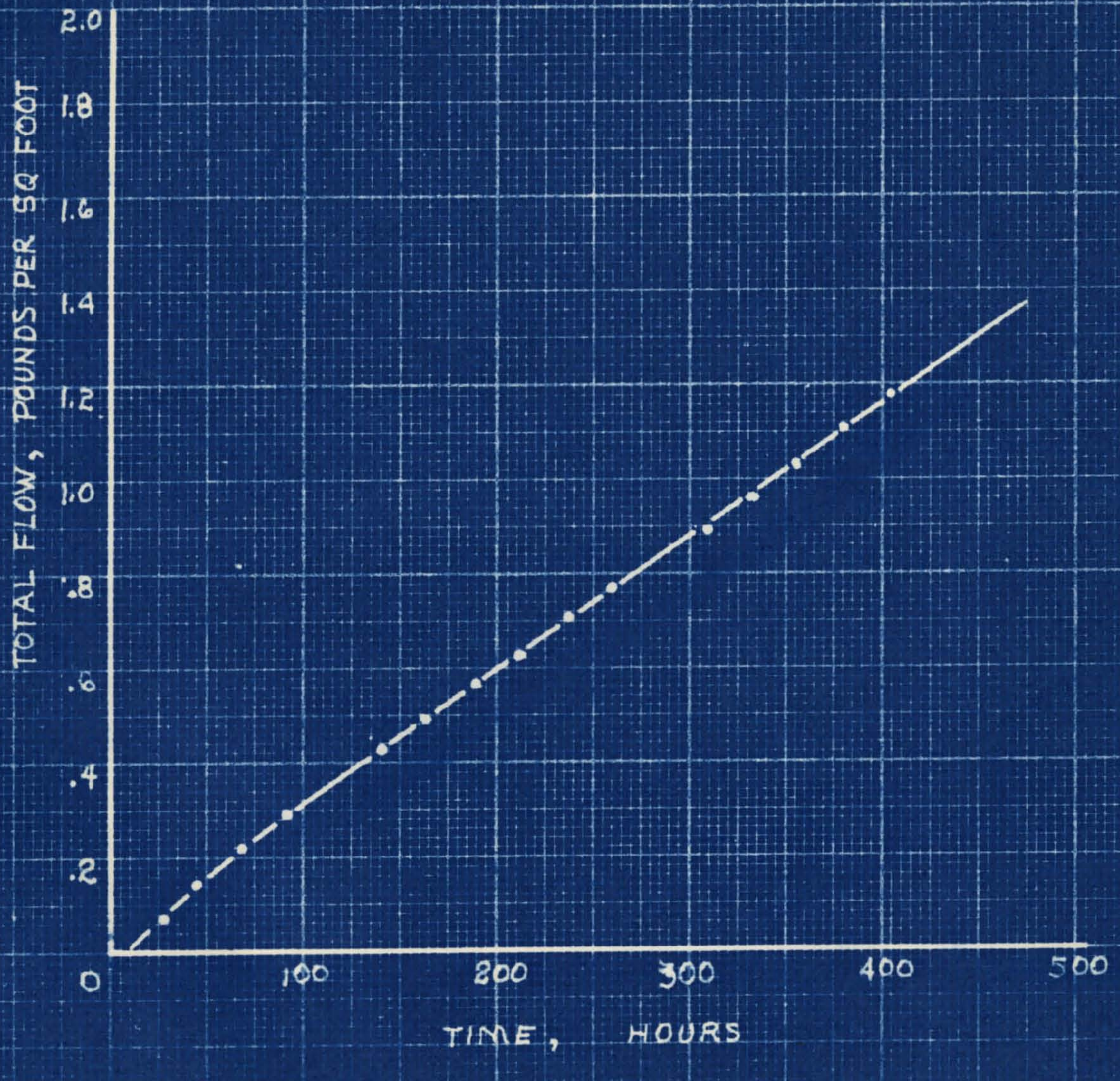


FIGURE 25 ACCUMULATIVE FLOW
SPECIMEN NO.5

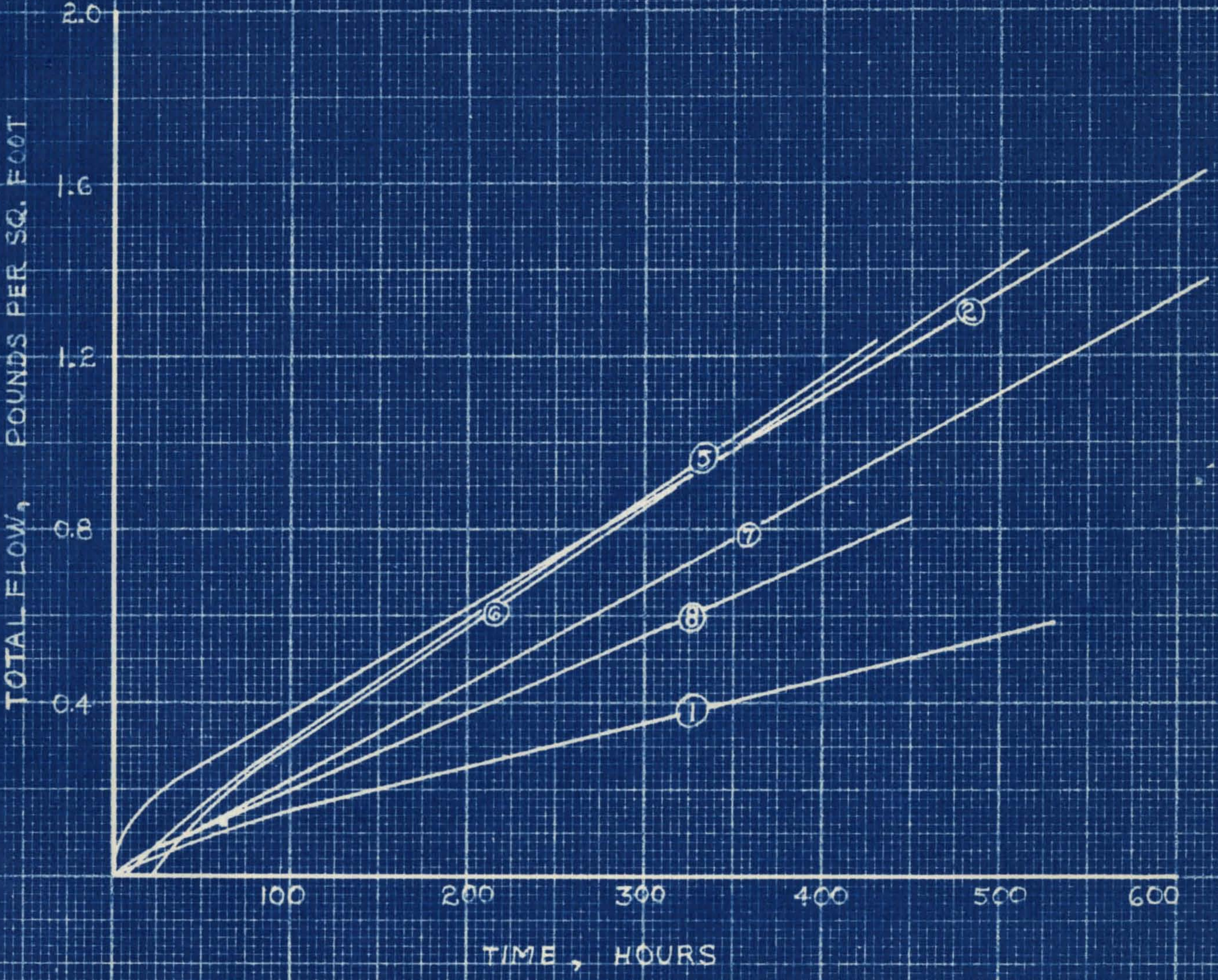


Figure 26 Comparison of Accumulative Flows, All Specimens

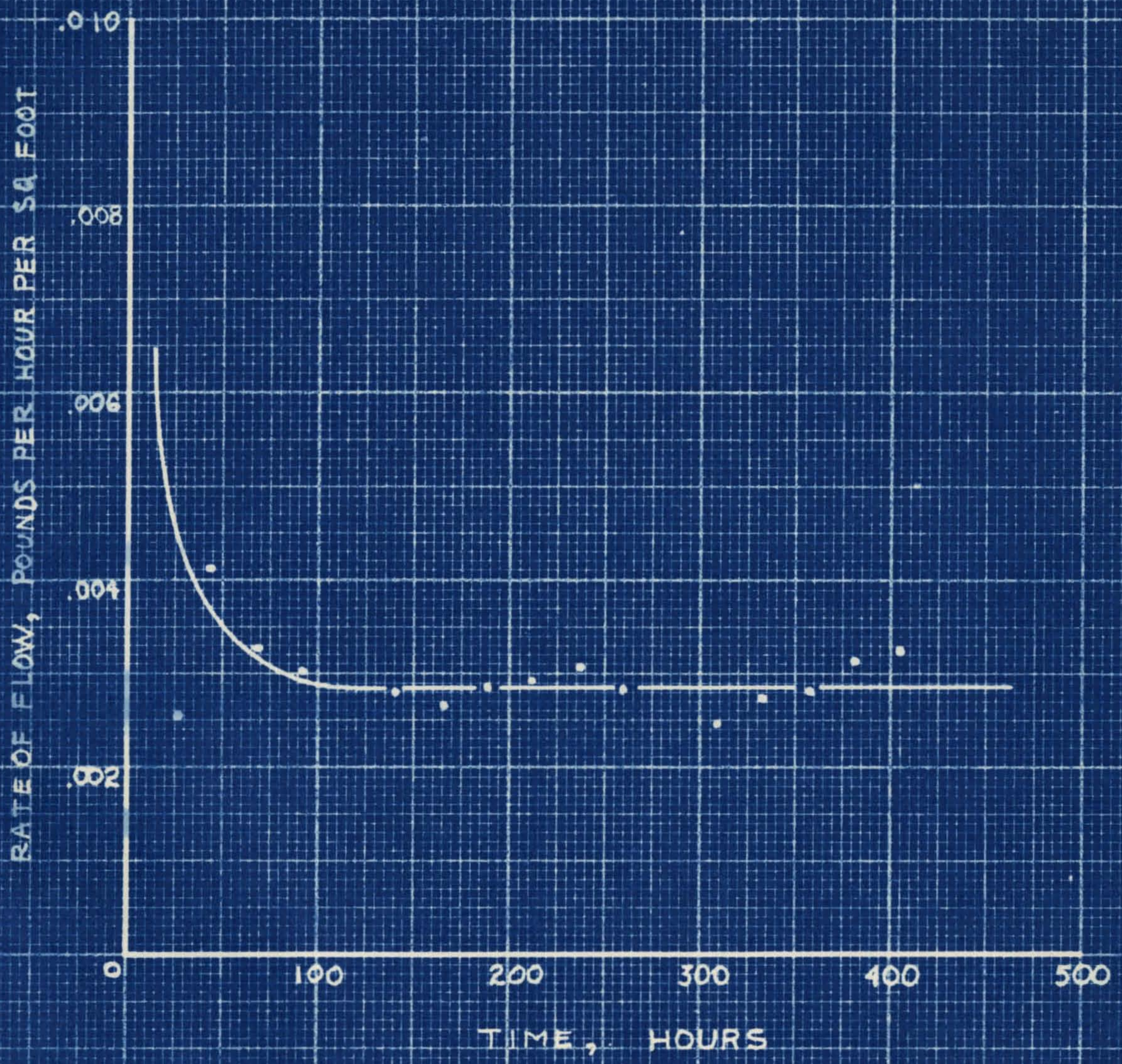


FIGURE 27 FLOW RATE
SPECIMEN NO. 5

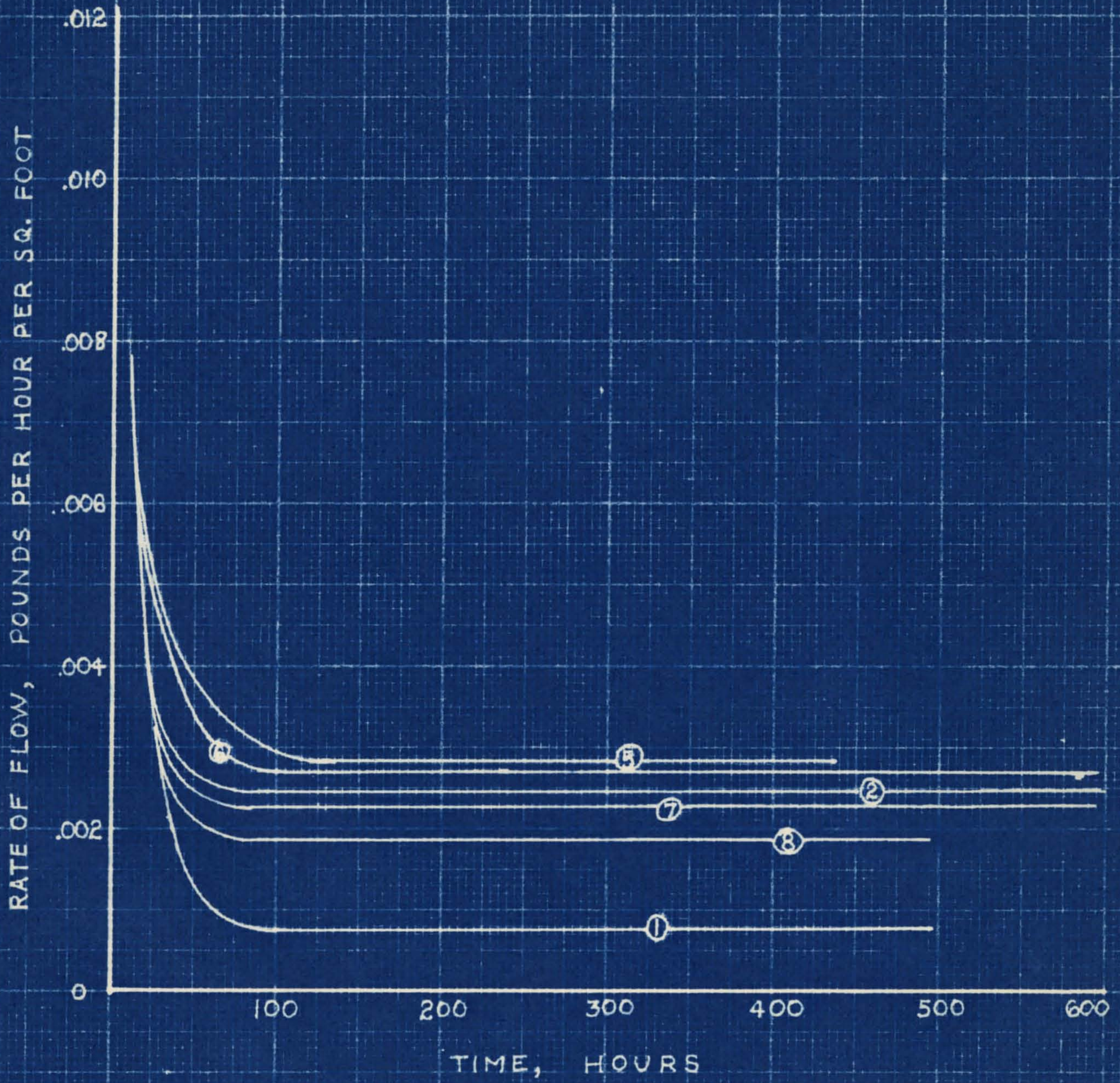


Figure 28 Comparison of Rates of Flow, All Specimens

TABLE XIV. LIQUID DIFFUSION THROUGH LAMINATED VENEERS

Specimen Number		Pressure, Feet of Water	Constant Flow Period, Hours	Flow Rate lbs./(sq ft) (hr)
1 Run	1	3	Indet.	.00009
	2	3	Indet.	.00035
	3	13	300	.00078
2		13	550	.00244
3		13	Grain Cracked	
4		13	During Pre-soak	
5		13	350	.00283
6		13	425	.00284
7		13	800	.00224
8		13	400	.00185

changes. This factor should not affect the average flow rate determined from the data because it would cancel a complete cycle of temperature change. However, it would cause an apparent deviation of individual readings from the average. (b) The humidity changes in the surrounding atmosphere have not been taken into consideration. This effect is not considered of great importance in this investigation because it simulates actual conditions to be encountered in industrial use of the product. It would, however, cause deviations of individual flow readings from the average. (c) A slight error necessarily present in reading such small amounts of flow may have caused deviations, but this error, too, is statistically unimportant as all liquid additions were made in sequence in the same tube.

No relation has as yet been discovered between the rates of diffusion of the various specimens and the curing conditions used in fabrication.

In a further attempt to correlate the results the physical structure of the oak veneer was considered. In the manufacture of rotary cut veneer an oak log is rotated against a doctor blade which continually slices away a sheet of veneer. In its action this blade folds the sheet of veneer back away from the log, tending to break the surface and generally roughen it. The degree to which this cracking occurs is a function of the angle through which the blade bends the veneer from its original position, this in turn being determined by the original radius of curvature of that veneer. Extending radially are medullary wood rays, which are necessarily more closely packed near the center of the log than in the outer sections. A count of the number of these rays in a given width of the product should then be a measure of the original placement of the veneer in the log and consequently the degree of cracking caused by the cutting blade.

The data are not sufficient for a correlation of results on this basis, but the method is suggested as a possible means of future attempts at developing a practical relation.

SUMMARY AND CONCLUSIONS

Results of a study of the characteristics of glue lines present in laminated 3/16 inch rotary cut, white oak veneers have been presented. The investigation was confined to the physical characteristics of films of the resorcinol-formaldehyde resin Penacolite G-1124. The investigation took three forms. First, the removal of all wood from the glue line structure by destructive digestion in chromic acid produced an undisturbed glue film which was studied by visual means. The glue film showed that great depth of penetration had occurred in open crevices in the wood veneers. On the other hand some areas were very thin and a great number of perforations were in evidence.

Secondly, the depth and quantity of penetration of resin into the veneers was determined by a progressive removal of small thicknesses of wood and glue from five-ply laminated sections. A quantitative estimation of glue present in successive layers of wood veneer led to a graphical plot of glue penetration into the various veneers.

Lastly the relative resistance to the diffusion of liquid water of several sections of two-ply-veneer fabricated under controlled conditions was determined. Present data for this study indicate very little influence of curing conditions and of the "looseness" or "tightness" of veneer used on the diffusional resistance of the finished product.

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APPENDIX

NOMENCLATURE

a	Surface per unit volume, sq. ft./cu. ft.
C_p	Molal heat capacity of liquid
D	Column diameter, feet
F	Feed rate, lb. mols/hr.
HTU	Height of transfer unit, ft.
K_G	Overall mass transfer coefficient, based on gas film
K_L	Overall mass transfer coefficient, based on liquid film
L	Liquid flow in column, mols/hour
M	Mean molecular weight
N	Number of mols transferred
P	Product rate, lb. moles/hour
T	Absolute temperature, degrees Kelvin
V	Vapor flow in column lb. mols/hour
W	Waste, bottoms, lb. mols/hour
X	Mol fraction of component in liquid
y	Mol fraction of component in vapor
Y^*	Mol fraction of component in vapor at equilibrium
Z	Viscosity, pounds per foot hour
k	Mass transfer coefficient, individual
q	Factor determining slope of line joining feed composition and operating line intersection in McCabe - Thiele calculation of theoretical plates.
t	Temperature, degrees Centigrade

α	Relative volatility, $\alpha = \frac{y_A/(1-y_A)}{X_A/(1-X_A)}$
μ	Psuedo molal viscosity, centipoises
λ	Molal latent heat of vaporization
ρ	Density, pounds per cubic foot

Subscripts

A	More volatile component
B	Less volatile component
b	Boiling point
d	Distillate
f	Feed
p	Product
w	Waste, bottoms

GLOSSARY

Actual Number of Plates

The number of plates in the section considered over which the data were of sufficient precision to use in calculations - not necessarily the number of plates between feed and product or between feed and waste. The number of plates which produced enrichment from X_m to X_n.

Average Column Temperature

The average temperature of the liquid on the plates being considered.

Liquid-vapor Ratio

The ratio of mols of liquid descending to the mols of vapor rising at any point in the column.

Overall Efficiency

The ratio of the number of theoretical plates to the number of actual plates. Overall rectifier efficiency includes only the section of the column above the feed plate; overall stripper efficiency, the section below the feed plate, and overall column efficiency, the entire section including the feed plate.

Psuedo Molal Viscosity

A psuedo viscosity based on a direct composition relationship.

Operating Ratio

The liquid-vapor ratio in tower sections.

Relative Volatility

The ratio of the mol fractions of more volatile components in the vapor to the less volatile component in the vapor divided by the ratio of the more volatile component in the liquid to the less volatile component in the liquid.

Theoretical Number of Plates

The number of plates necessary to produce a given separation when ideal interchange exists between the phases, calculated by the method of McCabe and Thiele (26) or a modification thereof.

Vapor Velocity

The superficial velocity of the vapor rising in the column, based on the diameter D of the column and expressed in feet per second.

Reflux Ratio

Molal ratio of reflux to distillate at top of column.

SAMPLE CALCULATIONS (19)

These calculations are for Run No. 85, Pentane-toluene.

Vapor Velocity

Vap. Vel. $$\frac{W_v\,(359)\,T_1\,(4)}{(3600)\,M\,(273)\,(\pi)\,D^2}$$

where W_v = Pounds vapor/hour

T_1 = Temperature of plate 1. deg. K.

D = Diameter of column, feet

$$\text{Vap. Vel.} = \frac{(23\quad 18)\,(359)\,(36\quad 273)\,(144)\,(4)}{(3600)\,(72)\,(273)\,(\pi)\,D^2}$$

Vap. Vel = 0.12 ft./sec

Operating Ratio

$$\frac{L}{V} = \frac{\text{moles liquid reflux/unit time}}{\text{moles vapor/unit time}}$$

$$\frac{L}{V} = \frac{(23)\,M}{(23+18)M}$$

$$\frac{L}{V} = 0.56$$

Material Balance

Material balance is on acetone fraction

$$\text{Balance} = \frac{100\ (\text{output})}{(\text{input})}$$

Acetone in feed = (F/M_f) (wt % Acetone)

= (110)(.163)

= 17.9 pounds/hour

Acetone in product = (P/M_p) (wt % Acetone)

= (18) (.992)

= 17.8 pounds/hour

Acetone in waste = negligible

$$\text{Balance} = \frac{(100)\ (17.8)}{(17.9)}$$

$$= 95.5\%$$

Slope of q line, $\frac{q}{q-1}$

Specific heat of pentane = 37 cal/gm mol

Specific heat of toluene = 40 cal/gm mol

Mean specific heat $= C_p = X_f(37)-(1-X_f)(40)$

$C_p = (.176)(37) - (.824)(40)$

$C_p = 38.5$

Molal latent heat $= \lambda = 7700$ cal./gm. mol

$$q = \frac{\text{latent heat of feed at feed temperature}}{\text{latent heat of feed at boiling point}}$$

$$q = \frac{7700 + (82025)\ (38.5)}{7700}$$

$q = 1.278$

$$\text{Slope} = \frac{q}{q-1} = \frac{1.278}{0.278} = 4.60$$

Theoretical Plates

The number of theoretical plates was calculated by the method of McCabe - Thiele. The equilibria data were plotted (figure 27) and the rectifier operating line drawn from X_p at a slope of L/V. From the X equal Y line at X_f, the q line was constructed with a slope of 4.60 to intersect the operating line. From this intersection to X_w, the stripper operating line was drawn. By a stepwise calculation from X19 to X1, the number of theoretical plates was found to be 7.7.

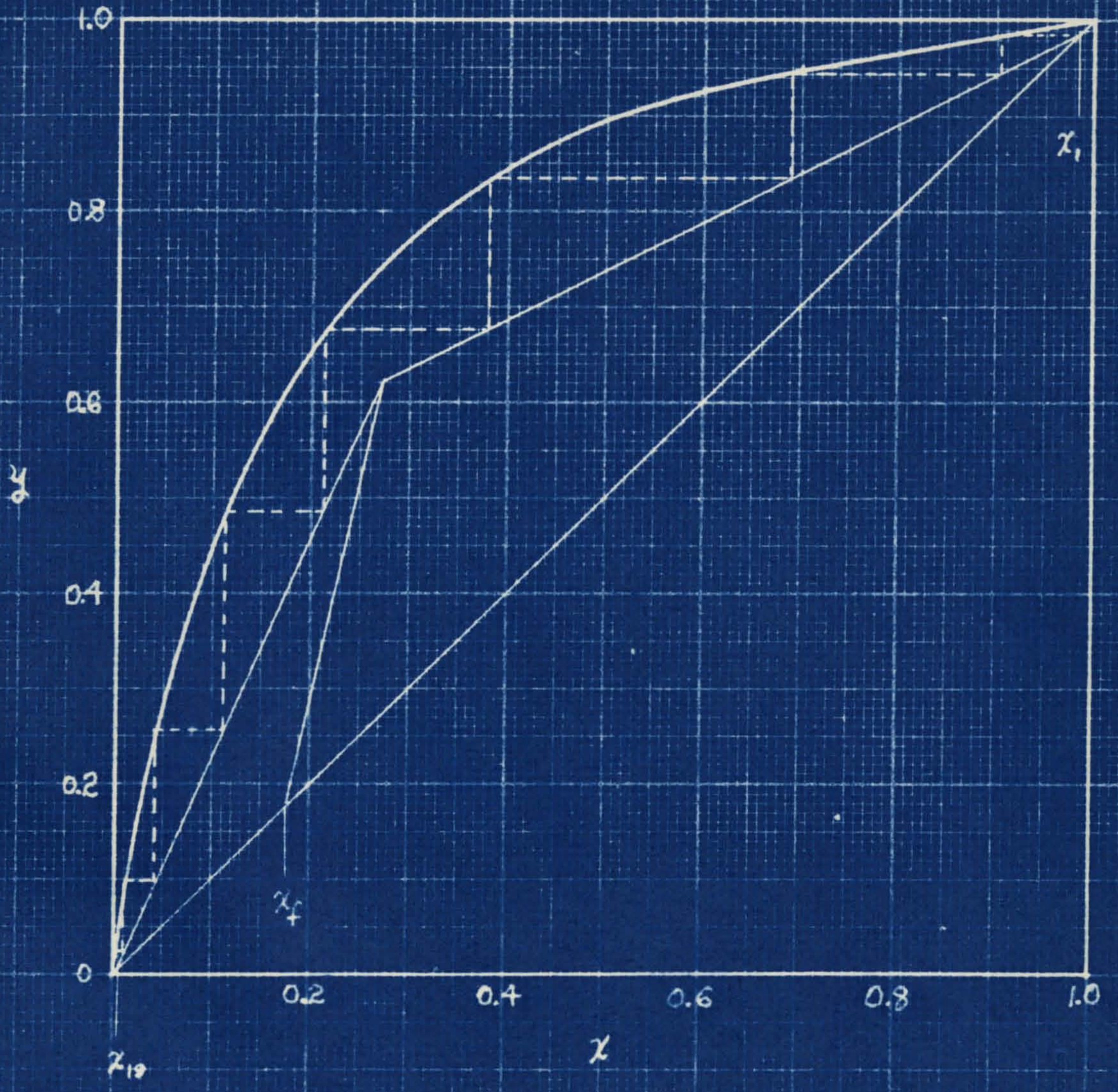


Figure 27 Sample Calculation of Theoretical Plates

Overall Efficiency

$$E = \frac{(100)\text{ theoretical plates required}}{\text{actual plates used}}$$

$$E = \frac{(100)\ (7.7)}{18}$$

$$E = 42.7\ \%$$

Stripper and rectifier efficiencies were calculated in the same manner, using the appropriate end values for X.

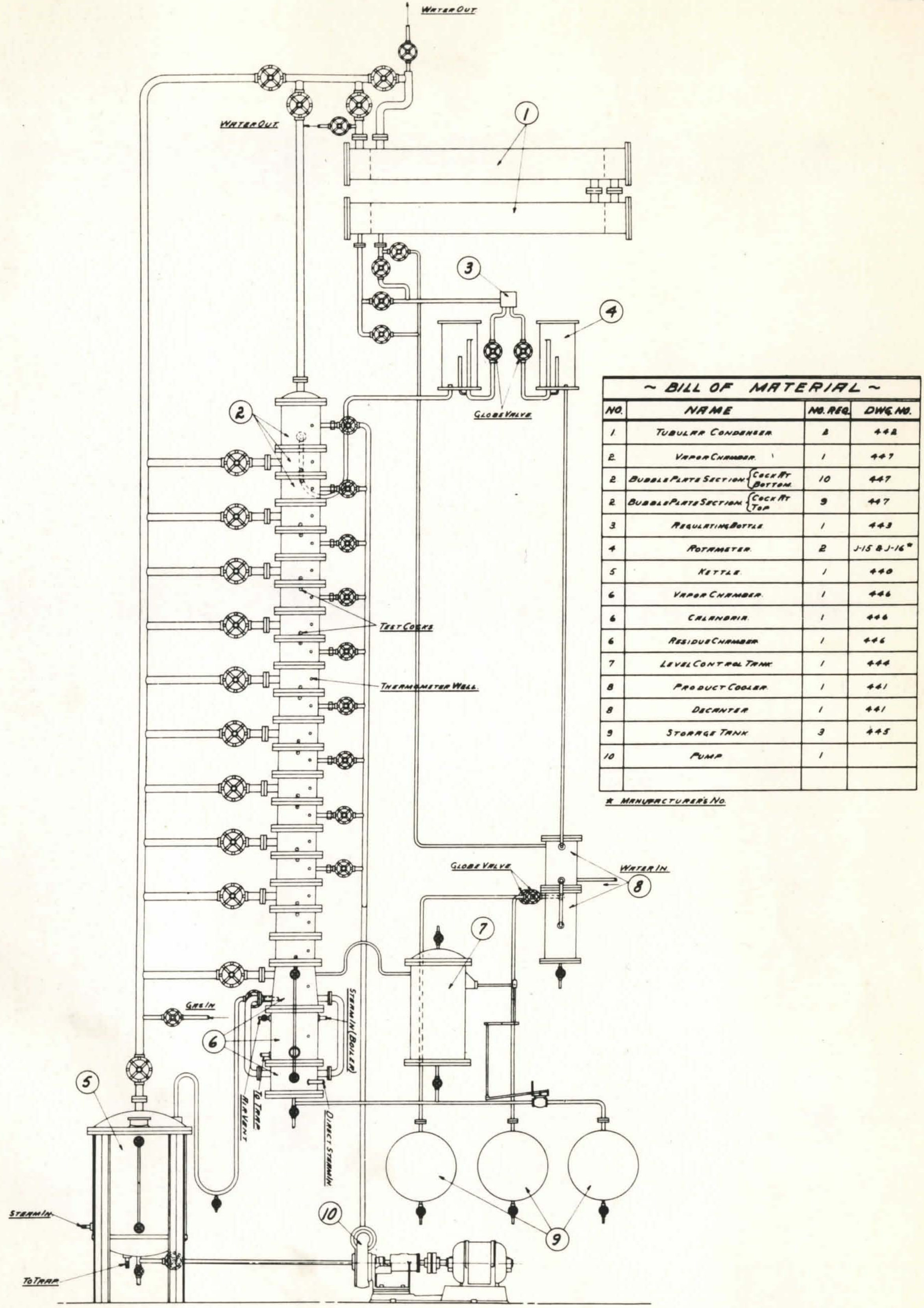
Relative Volatility and Viscosity

Using the average composition in the column α is determined from a plot of vs. composition.

$\alpha = 8.75$

$\mu = 0.31$

$\mu\alpha = 2.71$



~ BILL OF MATERIAL ~

NO.	NAME	NO. REQ.	DWG. NO.
1.	TUBULAR CONDENSER	2	442
2.	VAPOR CHAMBER	1	447
2	BUBBLE PLATE SECTION {COCK AT BOTTOM	10	447
2	BUBBLE PLATE SECTION {COCK AT TOP	9	447
3	REGULATING BOTTLE	1	443
4	ROTAMETER	2	J-15 & J-16*
5	KETTLE	1	440
6	VAPOR CHAMBER	1	446
6	CALANDRIA	1	446
6	RESIDUE CHAMBER	1	446
7	LEVEL CONTROL TANK	1	444
8	PRODUCT COOLER	1	441
8	DECANTER	1	441
9	STORAGE TANK	3	445
10	PUMP	1	

* MANUFACTURER'S NO.

Figure 30 Details of Experimental Fractionating Column

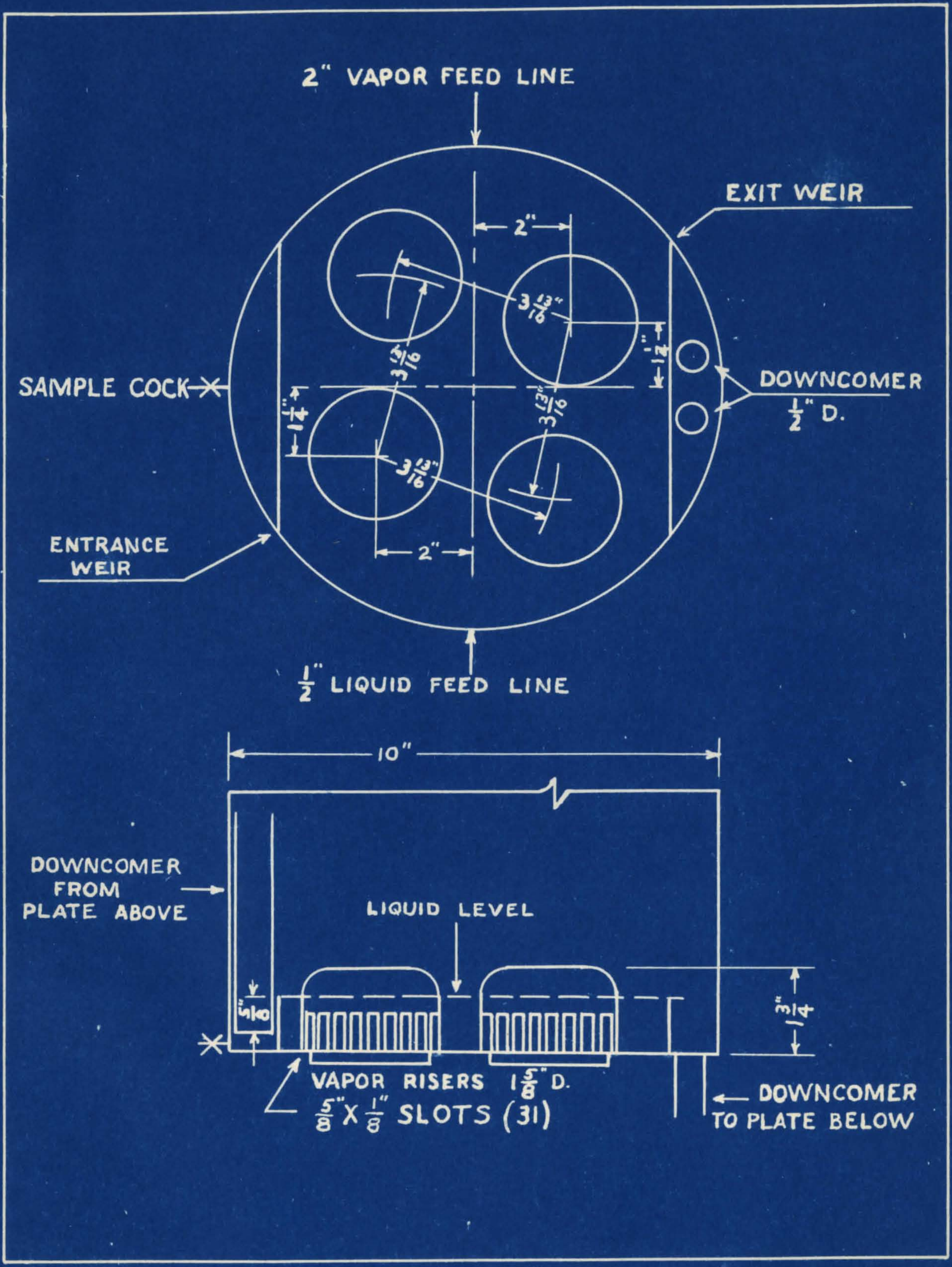


FIG. 31 DETAILS OF PLATE CONSTRUCTION

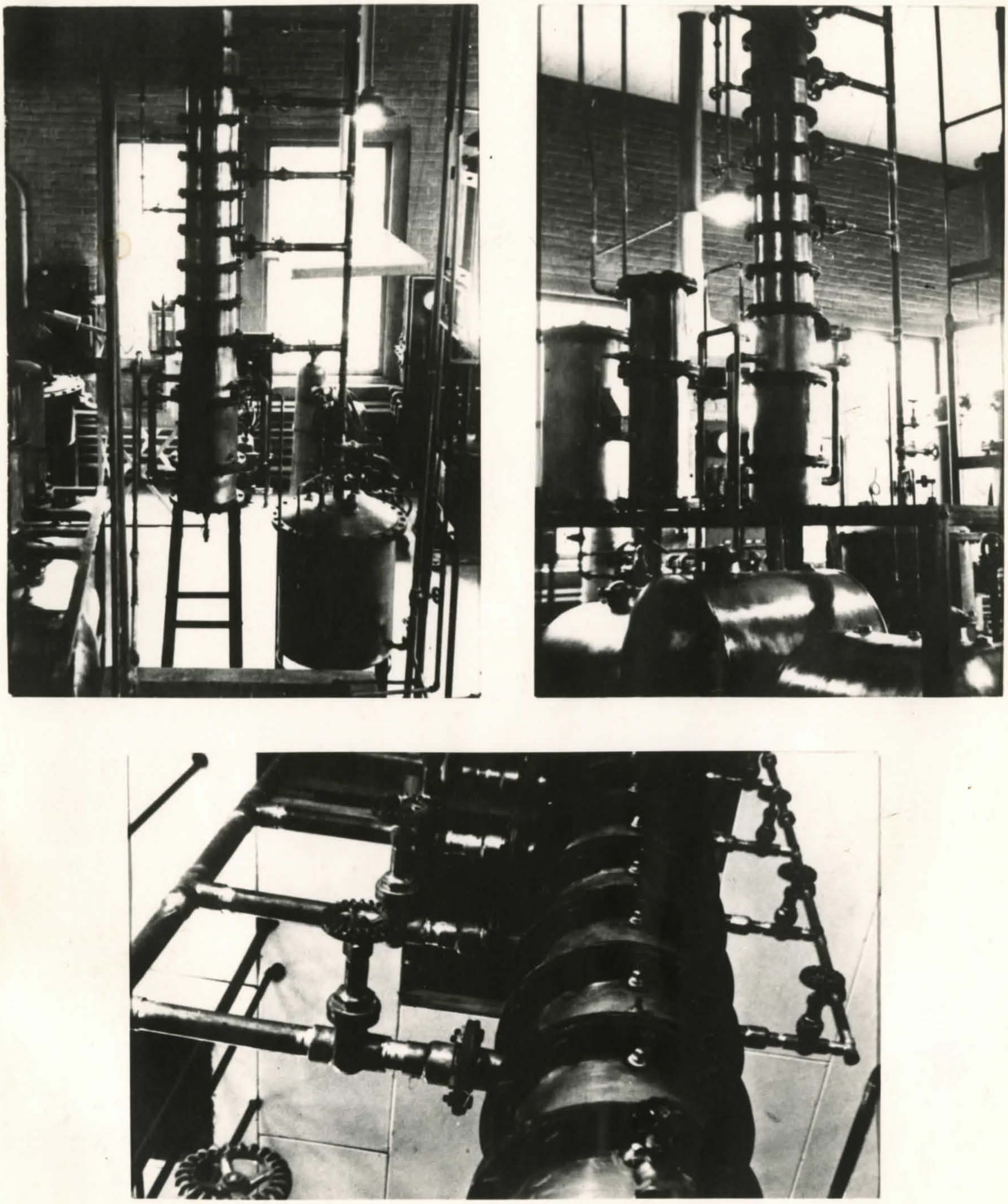


Figure 32 General Views of Fractionating Column

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VITA

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