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THE EFFECT OF COLUMN DIAMETER  
ON THE EFFICIENCY OF PACKED  
FRACTIONATING TOWERS

BY

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A THESIS

PRESENTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE

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Newark, New Jersey  
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## ABSTRACT

### THE EFFECT OF COLUMN DIAMETER ON THE EFFICIENCY OF PACKED FRACTIONATING TOWERS

The object of this work was to study the effect of packed column diameter on the fractionation efficiency so that a more reliable engineering approach could be employed in the scaling up of fractionating columns.

The height equivalent to a theoretical plate (HETP) was employed to compare column efficiencies in this study. The HETP was calculated for several gas mass velocities through columns of one-inch, two-inch and three-inch diameter, packed with one-quarter inch Berl saddles. All data were collected at total reflux conditions employing the standard n-heptane-methylcyclohexane binary.

The results indicated that the efficiencies of the one-inch and two-inch diameter columns were essentially equal and constant throughout the entire range of gas mass velocities studied. However, the efficiency of the three-inch diameter column was approximately 35% that of the smaller diameter columns at low gas mass velocities and increased to a maximum near the flood point where the efficiencies of all three columns were essentially identical.

It is concluded that the use of larger diameter columns results in substantially lower efficiencies at low gas mass velocities, but the effect of column diameter on the efficiency of packed fractionating towers at gas mass velocities near the flood point is negligible.

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## CONTENTS

Chapter	Page
1. Introduction	1
2. Background	2
3. Experimental Procedure	4
4. Discussion of Results	7
5. Conclusions	10
 Appendix	
I. Description of Equipment	12
II. Column Evaluation System	14
III. Relative Volatility Calculation	15
IV. The Effect of Composition on Refractive Index	17
V. Reflux Rotameter Calibration	19
VI. Rotameter Correction Factor	21
VII. Boil-up Correction Factor	23
VIII. Tabulation of Data	26
IX. Analysis of Data	40
X. Sample Calculation	50
XI. The Relationship Between Height Equivalent to a Theoretical Plate and Height of a Transfer Unit	61
XII. Quadratic Regression Equation	64
XIII. References	66

## FIGURES

1. HETP as a Function of Gas Mass Velocity.
- A-1. Equipment Diagram.
- A-2. The Effect of Composition on Refractive Index.
- A-3. Rotameter Calibration.
- A-4. Rotameter Calibration.
- A-5. The Effect of Composition on Specific Gravity.
- A-6. The Effect of Composition on the Rotameter Correction Factor.



## TABLES

- I. Data.
- II. Composition of the Samples as a Function of Refractive Index ( $N_D$ ).
- III. Summary of Calculations.

## CHAPTER 1

### Introduction

It is generally known throughout industry that the diameter of a packed fractionating column affects the efficiency of the fractionation. As the diameter of the column is increased in order to increase throughput, the packed height must also be increased to obtain the same degree of fractionation.

Attempts have been made to minimize this phenomenon by the installation of reflux redistributors throughout the length of the column in an effort to prevent channelling of the reflux down the column wall. This approach has met with some success, but in most cases the decrease in efficiency must be compensated for by longer packed sections.

In addition to the higher cost of installing higher fractionating towers, the increased pressure drop resulting from the greater packed depth can sometimes be intolerable, especially in high vacuum operations.

The object of this work is to study the effect of packed column diameter on the fractionating efficiency so that a more reliable engineering approach can be employed in the scaling up of fractionating columns.

## CHAPTER 2

### Background

The height equivalent to a theoretical plate (HETP) or efficiency of packed towers is a function of the following factors:

#### 1. Packing

Packing surface area, void space, and physical dimensions affect the degree of fractionation. Theoretically, if every molecule of liquid contacted every molecule of vapor within the column, the degree of fractionation would be at a maximum. However, this would require infinite packing surface to provide the required contact area. Column packings, therefore, are designed to provide maximum surface area and maximum void space in an attempt to compromise between fractionating efficiency and pressure drop.

#### 2. Tower Size

The column height and diameter also affect fractionating efficiency. As the diameter of a packed column is increased, the fractionating efficiency decreases because of poorer vapor-liquid contact. This is probably caused by channelling and can be improved by the use of liquid redistributors at the expense of pressure drop.

As the height of a packed column is increased, the HETP also tends to increase. This is probably caused by changes in the relative volatility and density of the material being fractionated as a result of the higher pressure drops incurred by the use of taller columns.

### 3. Mass Velocities

Gas mass velocity and liquid mass velocity also affect fractionating efficiency. Gas and liquid mass velocities of zero obviously provide no fractionation and velocities above the flood point of the column also result in no fractionation. An optimum gas and liquid mass velocity exists between these two extremes at which the vapor-liquid contact is at a maximum and HETP is at a minimum.

### 4. System Being Fractionated

The physical characteristics of the material being fractionated, such as molecular weight, diffusivity, viscosity, density, and relative volatility, all affect the efficiency of fractionation. These factors control the rate of mass transfer between the liquid and the gas.

In this work, all the above factors were held constant with the exception of tower diameter and gas and liquid mass velocities. The HETP and fractionating efficiency were therefore dependent only upon the intimate contact of the vapor and liquid within the column.

## CHAPTER 3

### Experimental Procedure

The effect of column diameter on fractionating efficiency was studied in laboratory equipment as described in Appendix I. The basic equipment consisted of a kettle, column, condenser and reflux system. The design of the system permitted interchange of columns of one-inch, two-inch, or three-inch diameter.

The binary system employed for column evaluation was n-heptane-methylcyclohexane, as described in Appendix II. All data and samples were collected at total reflux conditions.

The method of operation was as follows:

1. One of the three columns was selected, packed with one-quarter-inch Berl saddles, and installed in the system.
2. The binary mixture was charged to the kettle.
3. Heat was applied to the kettle by means of a rheostat controlled electric heating mantle or through steam coils wound inside the kettle.
4. The system was brought to total reflux and allowed to come to steady state conditions for a minimum of three hours.
5. After steady state was attained, the boil-up rate was determined by metering the reflux through pre-calibrated rotameters

(Appendix V) and then by correcting for density (Appendix VI) and condensate temperature (Appendix VII).

6. Samples were obtained at the top and base of the column. The composition of the binary was determined from the refractive index of the samples (Appendix IV).

7. From the composition of the samples and the relative volatility of the binary (Appendix III), the height equivalent to a theoretical plate was calculated from the Fenske Equation (Appendix X).

8. Boil-up conditions were then changed; and after steady state had again been obtained, the operation was repeated.

A series of 119 runs was made employing one-half inch and one-quarter inch Berl saddles. Only the data obtained with the use of the one-quarter inch Berl saddles were correlated because of the apparent unreliability of the data with the larger packing in the smaller diameter columns. In most cases, two runs were made during each day at identical conditions of boil-up in order to provide check results. After it was found that three to four hours at total reflux conditions were more than adequate to reach steady state, boil-up rates were sometimes changed between runs of the same day. All the data collected are tabulated in Appendix VIII.

An attempt was made to provide reliable data by a series of checks and rechecks as follows:

1. The columns were evaluated in the order: one-inch, two-inch, and three-inch; and then the one-inch and two-inch diameter columns were re-evaluated.

2. On many occasions, conditions were held constant for two runs on the same day in order to check reproducibility of results.

3. The operating conditions of the columns were duplicated on different days in order to check reproducibility of results.

4. Operating conditions were changed randomly rather than systematically in order to minimize the affect of uncontrollable variables.

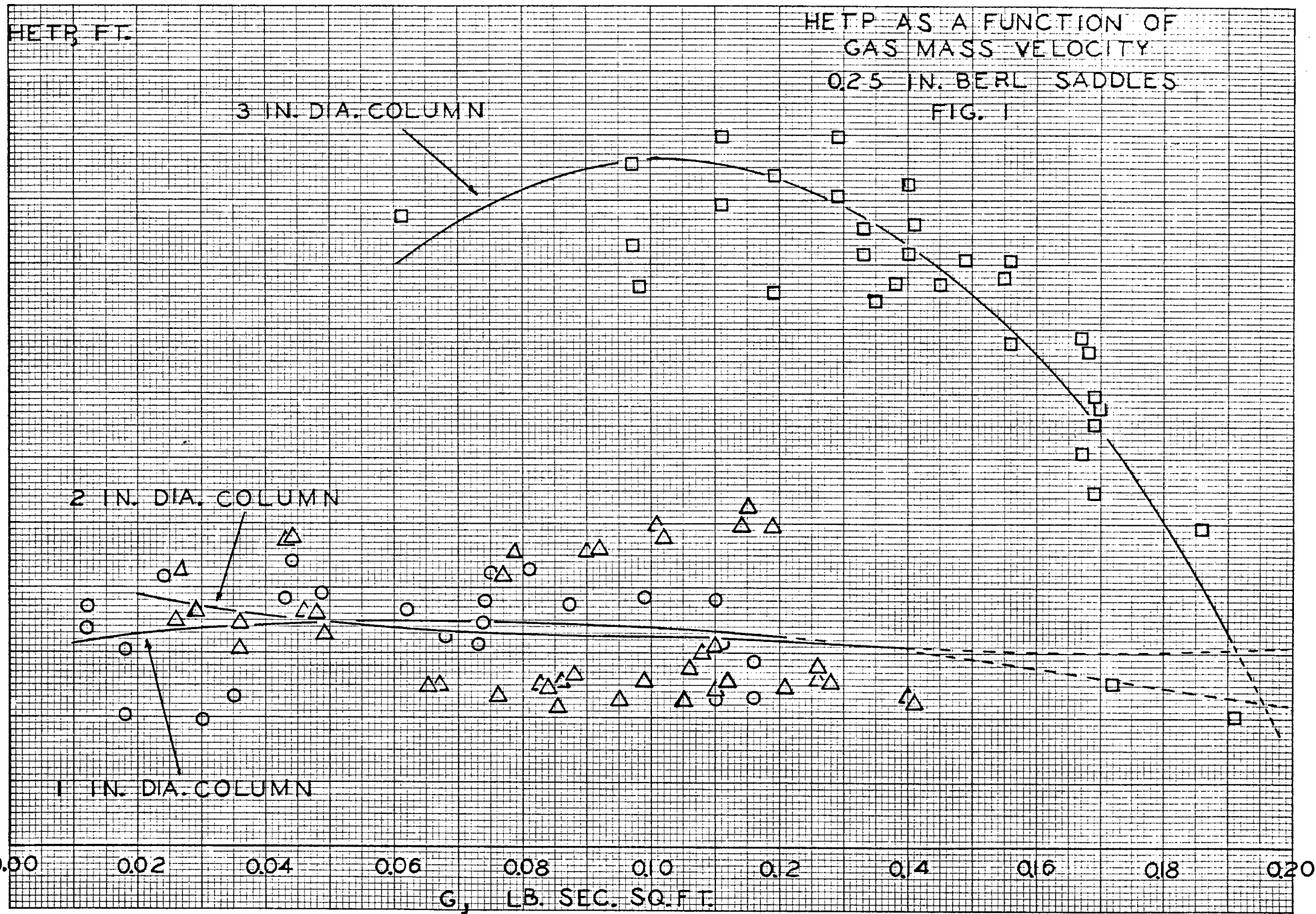
## CHAPTER 4

### Discussion of Results

In order to compare the efficiencies of the three columns studied, the height equivalent to a theoretical plate (HETP) was calculated at several gas mass velocities for each column. As the HETP increases, the column efficiency decreases because a greater packed height is required to obtain the same degree of fractionation. Although in current distillation theory the height of a transfer unit (HTU) appears to be a more reliable method of comparing fractionating efficiency than HETP, in cases involving low relative volatilities and laboratory size columns such as those employed in this study, the results are identical. The relationship between HETP and HTU is further described in Appendix XI.

The analyses of all the samples are tabulated in Appendix IX and the calculated HETP are tabulated in Appendix X. All the calculated results are presented in Figure 1, which shows the effect of column diameter and gas mass velocity on HETP. A quadratic regression equation was employed to obtain the best fit of the curves with the data (Appendix XII). As indicated in Figure 1, the HETP of the three-inch diameter column is approximately three times that of the one-inch and two-inch diameter columns at low gas mass velocities. In terms of efficiency, the three-inch diameter column is 35 percent as efficient as the smaller diameter columns at this gas mass velocity. This result is typical of that which occurs in industry, although probably more pronounced.





No significant difference in HETP occurs between the one-inch and two-inch diameter columns, nor does the HETP of these columns vary greatly throughout the entire range of gas mass velocities. It appears from this result that at low gas mass velocity rates, a maximum column-diameter-to-packing-size ratio exists, above which the HETP is increased considerably and the efficiency therefore is much decreased. This correlation, however is beyond the scope of this work.

The most startling and valuable result of the entire study is the fact that the HETP of all the columns are nearly equal and at a minimum near the flood point (approximately  $0.20 \text{ lb./sec-ft}^2$ ), indicating that maximum and equal efficiencies can be obtained from all diameter columns if operated at their maximum allowable gas mass velocity rates.

From this work it is apparent that at low gas mass velocities the vapor-liquid contact is relatively poor in larger diameter columns. This phenomenon becomes exaggerated in the case of the one-inch and three-inch diameter columns where the column cross sectional areas differ by a factor of nine.

The efficiency of packed columns is seriously impaired by the channelling of vapor and liquid which prevents effective interaction between the vapor and the liquid. Channelling is caused by the tendency of the liquid to pass down one side or the walls of the column while the vapor passes up the other side or the center of the column. The use of liquid redistributors has been employed in industry to minimize channelling and increase fractionation efficiency, but they

result in increased pressure drop. In addition, when a sufficient number of redistributors are employed to maximize efficiency, the tower will have become almost equivalent to a sieve-tray column and the packing itself will no longer be required.

Packed columns are usually operated by industry at approximately 90% of the flood point. The results of this work indicate that the columns should be operated even closer to the flood point to obtain maximum efficiency without liquid redistribution. From a control standpoint, this is very difficult to do because the column is inherently unstable under these conditions. In addition, for any particular column, the flood point varies with reflux ratio and the material being fractionated. However, it is possible and practical to purposely flood the column with each material to be fractionated to determine the flood point and then set the instrumentation to control the column just below this point. The value of this technique is twofold:

1. Laboratory fractionation data can be scaled up directly to plant equipment without purposely overdesigning to compensate for inefficiencies.

2. Shorter columns with higher throughputs can be employed, thus decreasing the original cost of the column and increasing productivity.

## CHAPTER 5

### Conclusions

1. The HETP and efficiency of the one-inch and two-inch diameter columns are nearly equal and constant throughout the entire range of gas mass velocities employed.

2. The HETP of the one-inch and two-inch diameter columns is approximately 35 percent that of the three-inch diameter column at low gas mass velocities. Similarly, the efficiency of the three-inch diameter column is approximately 35 percent that of the smaller diameter columns at low gas mass velocities.

3. The efficiency of the three-inch diameter column varies greatly with gas mass velocity, approaching a minimum at 0.10 lbs./sec-ft.<sup>2</sup> and a maximum near the flood point (0.20 lbs./sec-ft.<sup>2</sup>).

4. Near the flood point, the efficiency of all three columns are essentially equal and at a maximum.

APPENDIX

## APPENDIX I

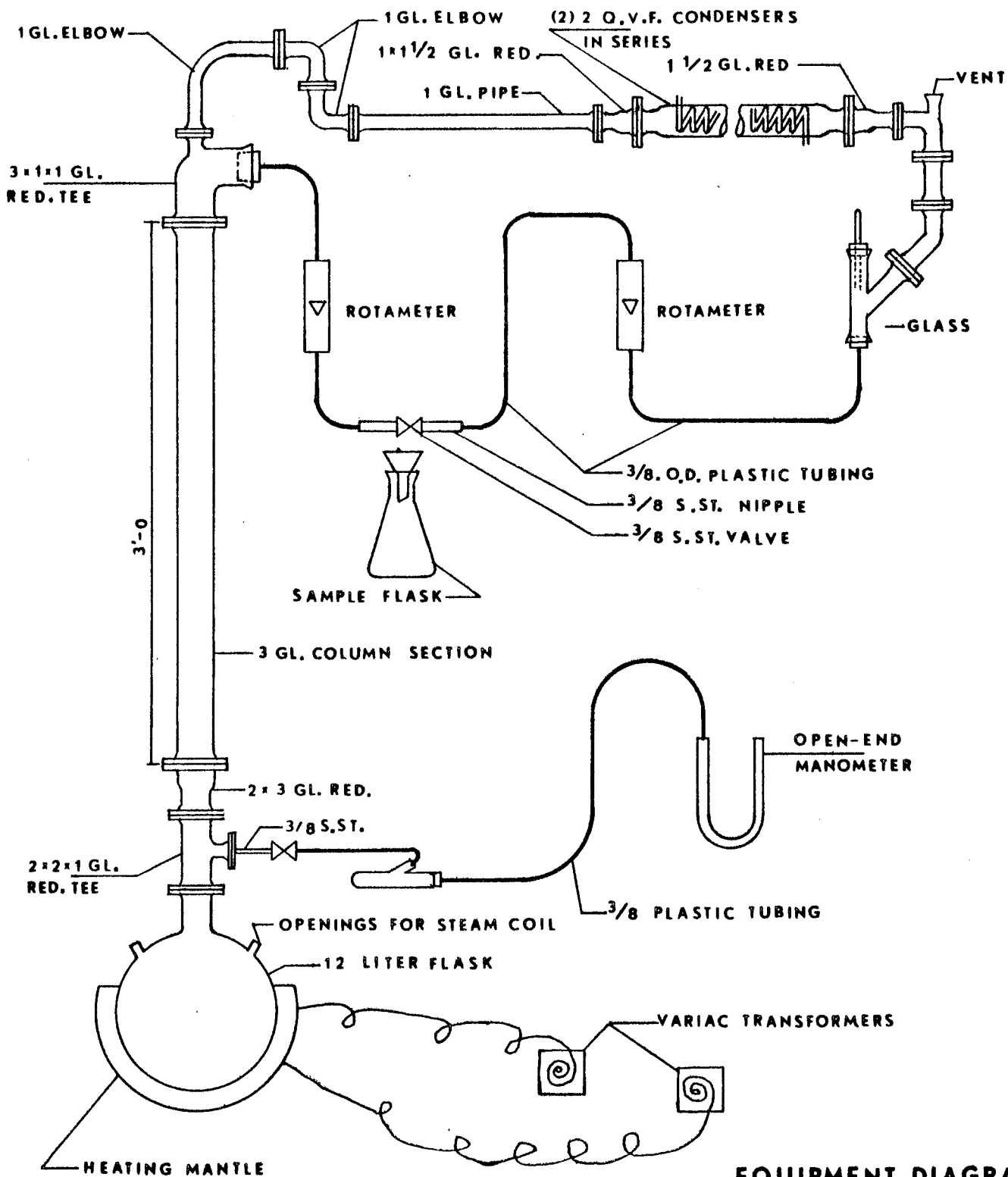
### Description of Equipment

The basic fractionation equipment consists of a kettle, column, condenser, and reflux system as shown in Figure A-1.

The kettle is a twelve-liter flask with three top outlets. The flask is heated by an electric heating mantle with two 1000-watt electrical elements. Each element is controlled individually by two rheostats which regulate the current from zero to full power. Auxiliary heat is provided by several coils of one-quarter inch copper tubing located inside the flask. Steam is fed to the coil through a pressure regulator and a one-quarter inch needle valve.

Flanged to the main outlet of the flask is a Pyrex tee to which a combination sample tap and pressure tap is attached. The pressure tap is connected to a thirty-inch water manometer through Mylar tubing via a glass condensate trap. Samples are taken by temporarily removing the tubing.

A three-foot-long column is also flanged to the tee. A system of Pyrex reducers is installed at this point so that columns of one-inch, two-inch, and three-inch diameters can be employed as required. The column is packed with one-quarter inch Berl saddles to a packed height of three feet. A wire-mesh mist entrainer is employed as both the packing support and reflux distributor at the extremities of the column.



**EQUIPMENT DIAGRAM  
FIGURE NO. A-1**

Through a series of Pyrex reducers, ells, and tees, the column is piped to two laboratory water cooled condensers connected in series. The system is vented after the condensers.

The condensate is piped back to the column through Mylar tubing and is totally returned above the wire mesh as reflux. A thermometer is installed in the condensate line to indicate the degree of sub-cooling. A sample tap is also installed in this line. The condensate is passed through two Fischer-and-Porter rotameters connected in series. The range of these rotameters is 0 to 1.4 gal./hr. and 1.0 to 6.0 gal./hr. water, respectively.

The entire system, with the exception of the reflux return, is insulated with layers of aluminum foil, asbestos cloth, one-half inch glass wool blanket, one-inch-thick standard pipe insulation and a covering of aluminum sheeting.



## APPENDIX II

### Column Evaluation System

The system normal heptane-methylcyclohexane was employed as the binary to evaluate the column and packing under study. This binary was selected because the molecular weight, boiling point, and latent heat of evaporation of the components are very similar. In addition, the refractive indexes of the components are quite different, thus providing a useful tool for the evaluation of samples.

The physical characteristics of the components are as follows:

	<u>Normal-Heptane</u>	<u>Methylcyclohexane</u>
Formula	$\text{CH}_3(\text{CH}_2)_5\text{CH}_3$	$\text{CH}_3(\text{CHC}_5\text{H}_{10})$
Molecular Weight, lb./lb. mole	100.20	98.18
Specific Gravity, 20°C./4°C.	0.684	0.769
Melting Point, °C.	-90.6	-126.3
Boiling Point, °C.	98.4	101.0
Refractive Index, 20°C.	1.3876	1.4230
Latent Heat, BTU/lb.	137.7	138.5
Heat Capacity, BTU/lb. °F.	0.507	0.883

### APPENDIX III

#### Relative Volatility Calculation

The relative volatility of the binary system normal-heptane-methylcyclohexane is calculated from the vapor-liquid equilibrium data published in the literature. The definition of relative volatility is

$$\alpha_{hm} = \frac{Y_h X_m}{X_h Y_m}, \text{ where}$$

$\alpha_{hm}$  = relative volatility of normal-heptane to methylcyclohexane.

$X_h$  = weight fraction of normal-heptane in the liquid.

$Y_h$  = weight fraction of normal-heptane in the vapor.

$X_m$  = weight fraction of methylcyclohexane in the liquid.

$Y_m$  = weight fraction of methylcyclohexane in the vapor.

The calculation of the relative volatility is as follows, wherein the data for  $X_h$ ,  $Y_h$ , and the temperatures were extracted from the literature.

<u><math>X_h</math></u>	<u><math>Y_h</math></u>	<u><math>X_m</math></u>	<u><math>Y_m</math></u>	<u>T, °C.</u>	<u><math>\alpha_{hm}</math></u>
0.0000	0.0000	1.000	1.0000	101.0	
0.0310	0.0350	0.9690	0.9650	100.7	1.133
0.0580	0.0620	0.9420	0.9380	100.6	1.073
0.0950	0.1030	0.9050	0.8970	100.5	1.093
0.1330	0.1430	0.8670	0.8570	100.4	1.087
0.1800	0.1920	0.8200	0.8080	100.3	1.083
0.2160	0.2290	0.7840	0.7710	100.2	1.077
0.2715	0.2890	0.7285	0.7110	100.0	1.092

$X_h$	$Y_h$	$X_m$	$Y_m$	$T, ^\circ C.$	$hm$
0.3170	0.3330	0.6830	0.6670	100.0	1.076
0.3630	0.3810	0.6370	0.6190	99.9	1.080
0.4010	0.4200	0.5990	0.5800	99.8	1.082
0.4560	0.4750	0.5440	0.5250	99.6	1.081
0.5010	0.5210	0.4990	0.4790	99.3	1.083
0.5590	0.5780	0.4410	0.4220		1.080
0.5990	0.6180	0.4010	0.3820	99.0	1.083
0.6470	0.6660	0.3530	0.3340	98.9	1.090
0.7090	0.7280	0.2910	0.2720	98.8	1.100
0.7560	0.7710	0.2440	0.2290		1.085
0.7960	0.8100	0.2040	0.1900	98.6	1.093
0.8430	0.8535	0.1570	0.1465	98.6	1.084
0.8790	0.8900	0.1210	0.1100		1.115
0.9060	0.9130	0.0940	0.0870		1.087
0.9310	0.9400	0.0690	0.0600	98.5	1.162
0.9540	0.9625	0.0460	0.0375		1.225
0.9800	0.9860	0.0200	0.0140	98.4	1.438
1.000	1.0000	0.0000	0.0000	98.4	

The relative volatility between the limits of  $X_h = 0.058$  to  $X_h = 0.843$  is relatively constant and averages 1.085. A relative volatility of 1.085 is therefore employed in all the calculations in this work.

APPENDIX IV

The Effect of Composition on Refractive Index

Synthetic blends of pure normal heptane and methylcyclohexane were prepared in the laboratory, each component being weighed to the nearest ten-thousandth gram. The refractive index of each sample was measured at 20°C. to the nearest ten-thousandth unit. The data collected are as follows:

<u>N-Heptane</u>	<u>Methyl- cyclohexane</u>	<u>Mass, grams</u> <u>Total</u>	<u>WT.%</u> <u>N-Heptane</u>	<u>WT.%</u> <u>Methyl- cyclohexane</u>	<u>Refractive</u> <u>Index, 20°C.</u>
2.8945		2.8945	100.0	0.0	1.3947
2.8945	0.1780	3.0725	94.3	5.7	1.3965
2.5999	0.6013	3.2012	81.2	18.8	1.3997
2.5350	1.0429	3.5779	70.9	29.1	1.4029
2.4889	1.4472	3.9361	63.3	36.7	1.4048
2.4157	2.4663	4.8820	49.4	50.6	1.4081
2.3648	4.5209	6.8857	34.4	65.6	1.4122
2.3360	6.8325	9.1685	25.5	74.5	1.4146
2.3101	9.3892	11.6993	19.7	80.3	1.4164
NIL	3.2464	3.2464	0.0	100.0	1.4217
0.7569	3.2464	4.0033	18.9	81.1	1.4167
1.6854	3.1868	4.8722	34.6	65.4	1.4123
3.1067	3.1209	6.2276	49.9	50.1	1.4082
5.4689	3.0758	8.5447	64.0	36.0	1.4040
8.2891	3.0388	11.3279	73.2	26.8	1.4020

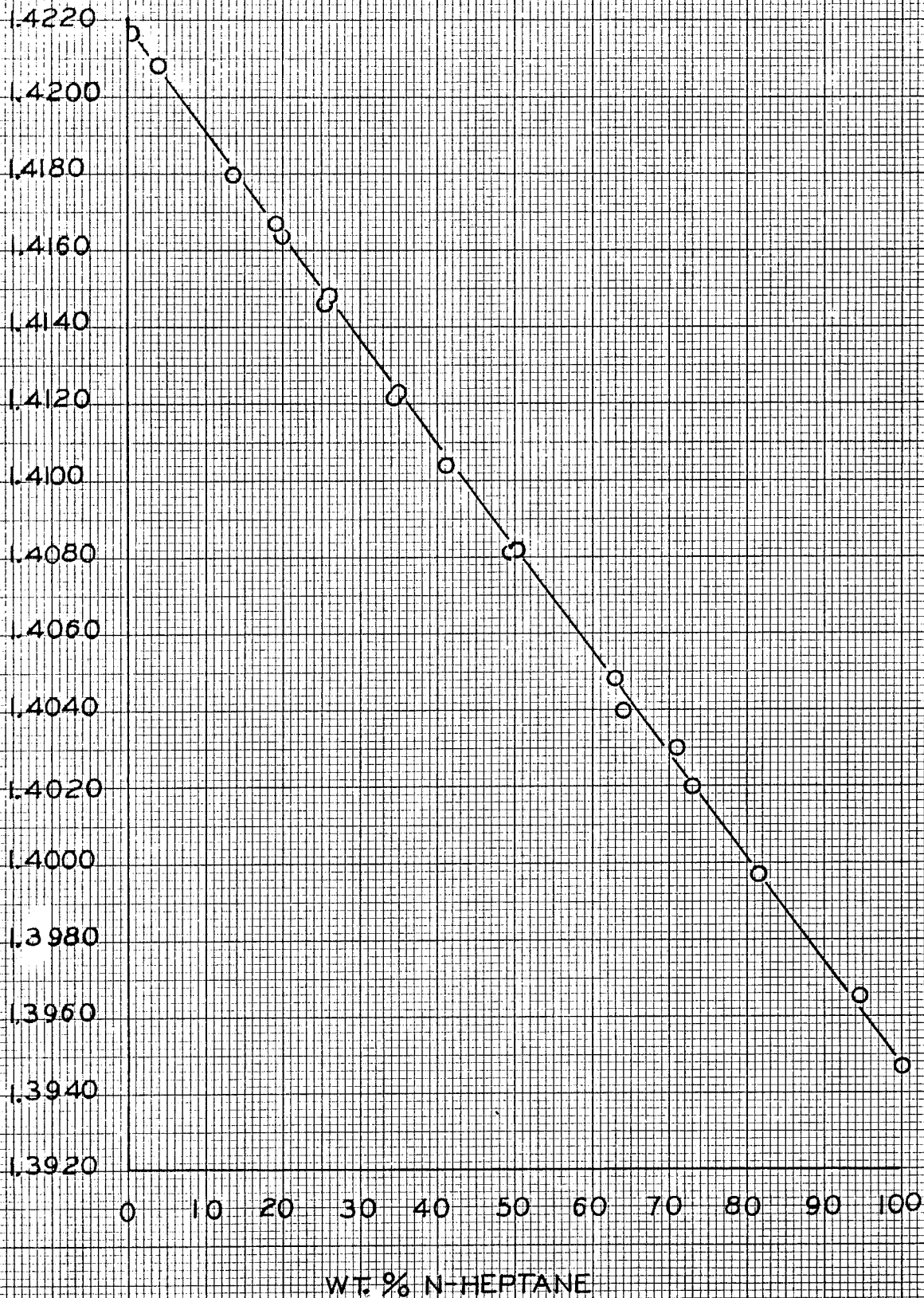
<u>Mass, grams</u>			<u>Wt. %</u>		<u>Refractive Index, 20°C.</u>
<u>N-Heptane</u>	<u>Methyl-cyclohexane</u>	<u>Total</u>	<u>N-Heptane</u>	<u>Methyl-cyclohexane</u>	
NIL	6.2651	6.2651	0.0	100.0	1.4217
0.2817	6.2651	6.5468	4.3	95.7	1.4208
0.9723	6.1807	7.1530	13.6	86.4	1.4180
2.1615	6.0869	8.2484	26.2	73.8	1.4148
4.3019	5.9630	10.2647	41.9	58.1	1.4104

These data are plotted in Figure A-2.

THE EFFECT OF COMPOSITION ON REFRACTIVE INDEX  
N-HEPTANE — METHYLCYCLOHEXANE

FIGURE A-2

REFRACTIVE INDEX, 20°C.



APPENDIX V

Reflux Rotameter Calibration

Two Fischer and Porter rotameters were employed in series to measure the reflux rate. The ranges of the rotameters overlapped, so that low flow rates employing the one-inch diameter column and high flow rates employing the three-inch diameter column could be accurately measured.

The rotameters were calibrated with water, the results of which are tabulated below:

<u>ROTAMETER SCALE READING</u>		<u>WATER RATE, gal/hr.</u>
<u>F&amp;P No. 1/8-22/G-4/42</u>	<u>F&amp;P No. 1/4-25-G-5/81</u>	
0.00	0.00	0.000
2.00	0.25	0.016
4.00	0.50	0.079
6.00	0.75	0.191
8.00	1.00	0.341
10.00	1.25	0.492
12.00	1.50	0.643
14.00	1.75	0.802
16.00	2.00	0.961
18.00	2.25	1.113
20.00	2.50	1.271
22.00	2.75	1.429
-	3.00	1.588
-	4.00	2.225
-	6.00	3.490

<u>ROTAMETER SCALE READING</u>		<u>WATER RATE, gal/hr.</u>
<u>F&amp;P No. 1/8-22-G-1/42</u>	<u>F&amp;P No. 1/4-25-G-5/81</u>	
-	8.00	4.760
-	10.00	6.040
-	12.00	7.310
-	14.00	8.570
-	16.00	9.840

These data are plotted in Figure A-3 and Figure A-4.



SCALE READING

ROTAMETER CALIBRATION

FP 1/8 22 G 4/42

FIG. NO. A-3

22

20

18

16

14

12

10

8

6

4

2

0

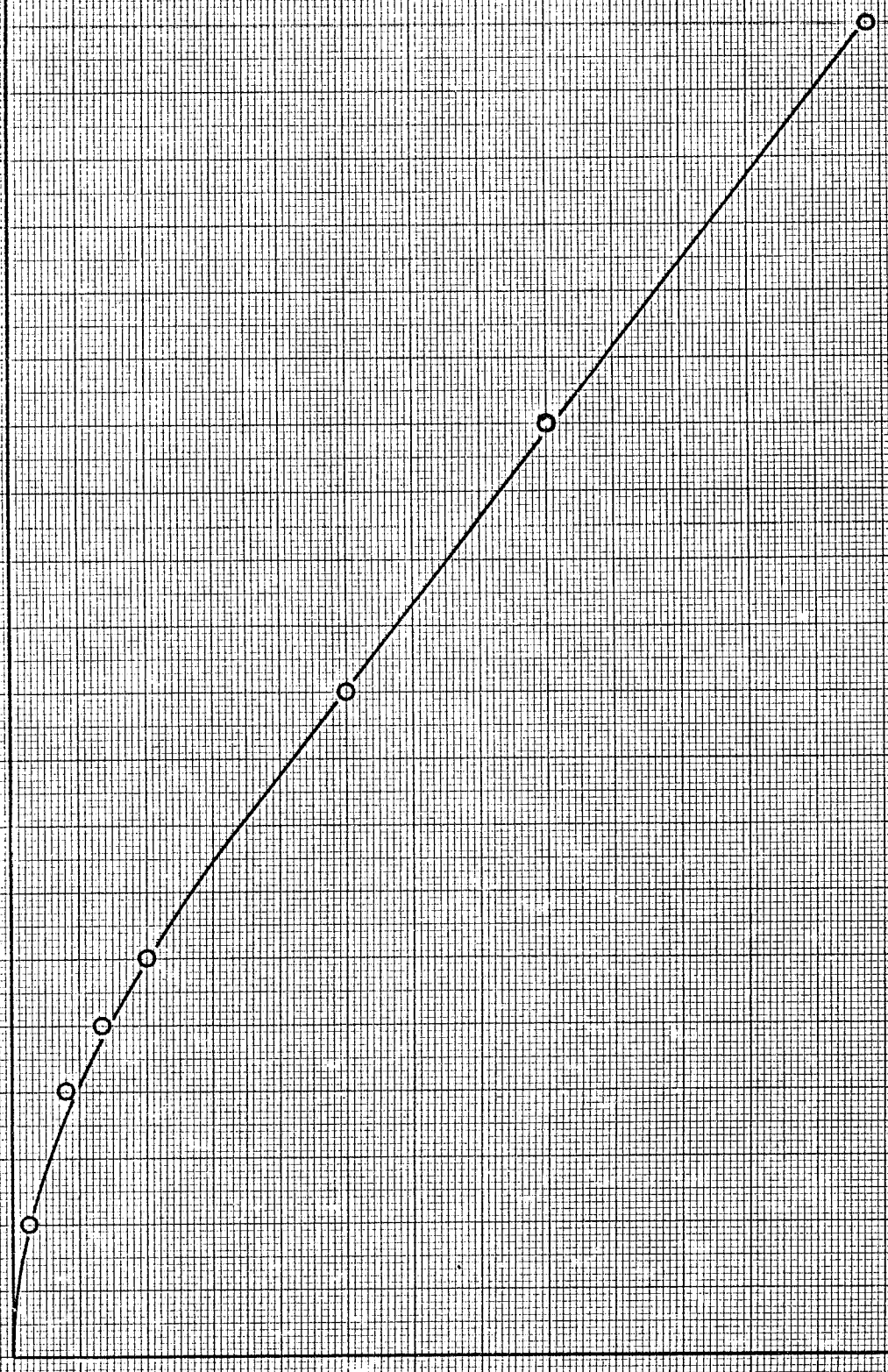
0.0

0.5

1.0

1.5

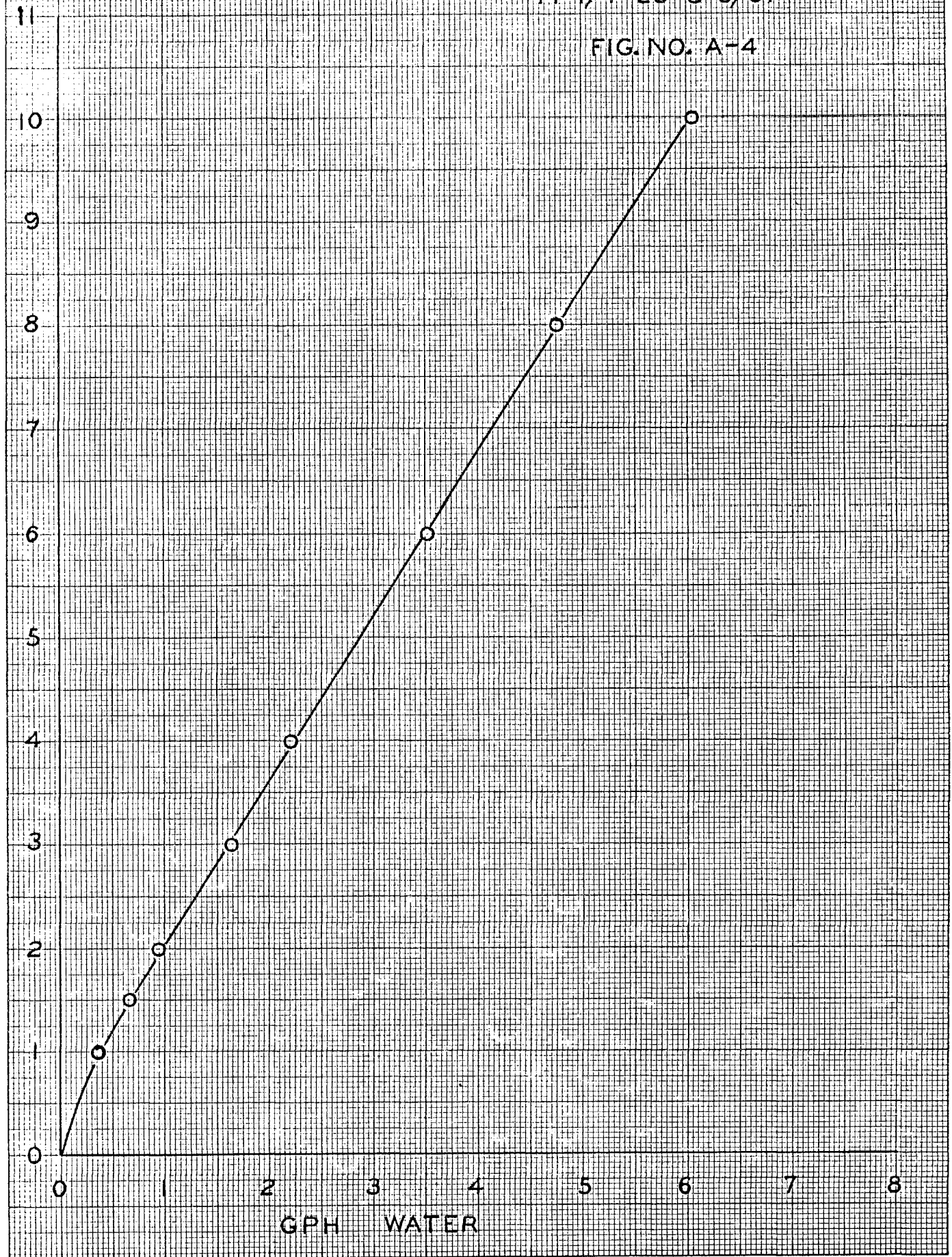
GPH WATER



SCALE READING

ROTAMETER CALIBRATION  
FP 1/4 25 G 5/81

FIG. NO. A-4



## APPENDIX VI

### Rotameter Correction Factor

Since the rotameters were calibrated with water, the readings must be corrected for the actual density of the reflux. In addition, the density of the reflux changes with composition.

The rotameter correction factor is expressed as <sup>1</sup>

$$\left[ \frac{(8.02 - \rho)(1.0)}{(8.02 - 1.00)(\rho)} \right]^{1/2},$$

where  $\rho$  = specific gravity of the reflux.

The specific gravity of the reflux is expressed as follows:

$$\frac{\text{WT.\% n-heptane}}{0.684} + \frac{100 \text{ WT.\% methylcyclohexane}}{0.769},$$

where,

specific gravity of n-heptane = 0.684  
specific gravity of methylcyclohexane = 0.769.

The solution of these equations at various reflux compositions are given in the table below:

Weight%	$\rho$ , g/cc	Rotameter
<u>N-Heptane</u>	<u>SpG of Reflux</u>	<u>Correction Factor</u>
0.0	0.769	1.160
10.0	0.759	1.169
20.00	0.750	1.180
30.0	0.741	1.183

<sup>1</sup>The Fischer & Porter Handbook, Fischer & Porter Co.

Weight%	$\rho$ , g/cc	Rotameter
<u>N-Heptane</u>	<u>SpG of Reflux</u>	<u>Correction Factor</u>
40.0	0.732	1.191
50.0	0.723	1.200
60.0	0.716	1.204
70.0	0.707	1.215
80.0	0.699	1.222
90.0	0.692	1.229
100.0	0.684	1.235

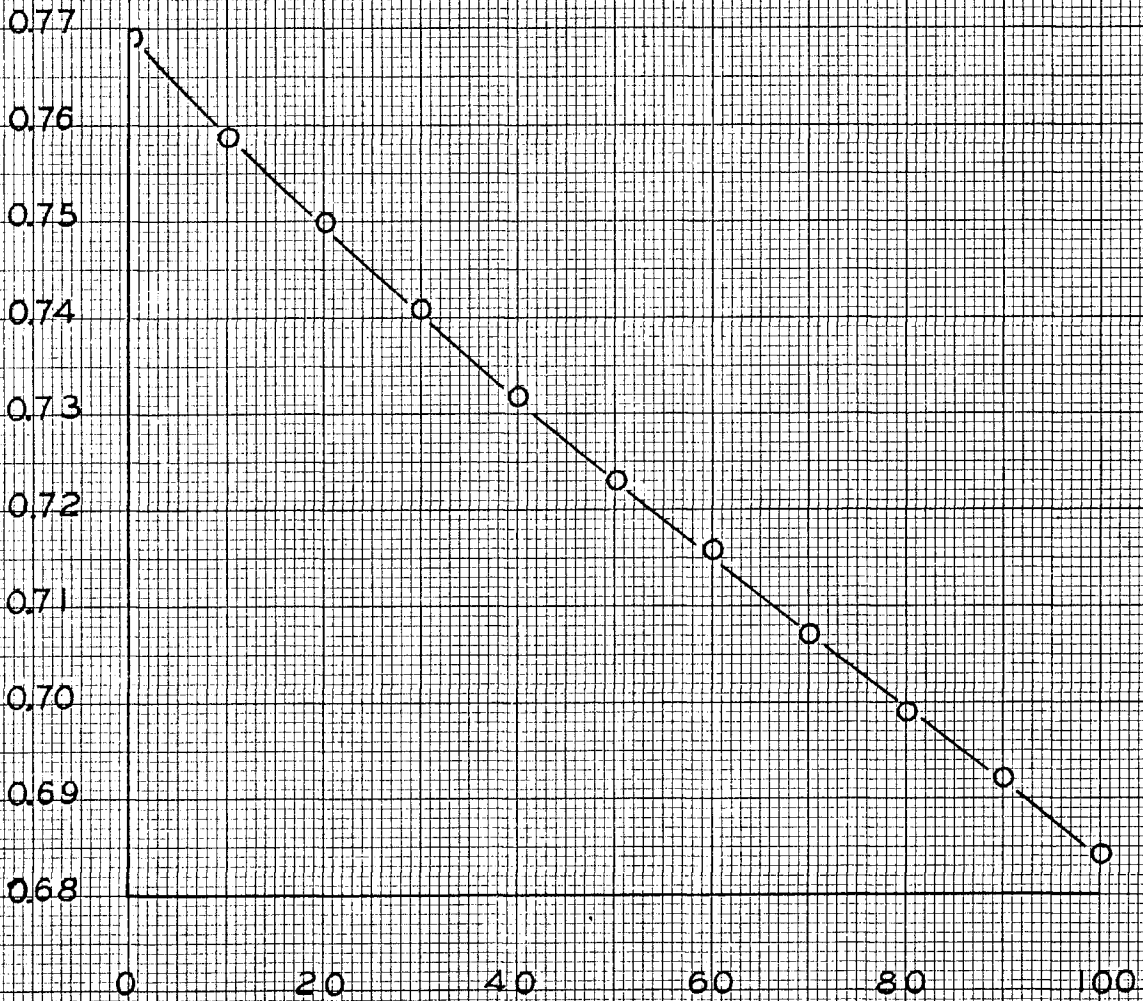
These data are plotted in Figure A-5 and A-6.

THE EFFECT OF COMPOSITION ON SPECIFIC GRAVITY

N-HEPTANE—METHYLCYCLOHEXANE

FIGURE A-5

SPECIFIC GRAVITY



WT.% N - HEPTANE

THE EFFECT OF COMPOSITION ON THE  
ROTAMETER CORRECTION  
FACTOR

N-HEPTANE — METHYLCYCLOHEXANE

FIGURE A-6

CORRECTION  
FACTOR

1.24

1.23

1.22

1.21

1.20

1.19

1.18

1.17

1.16

1.15

0

20

40

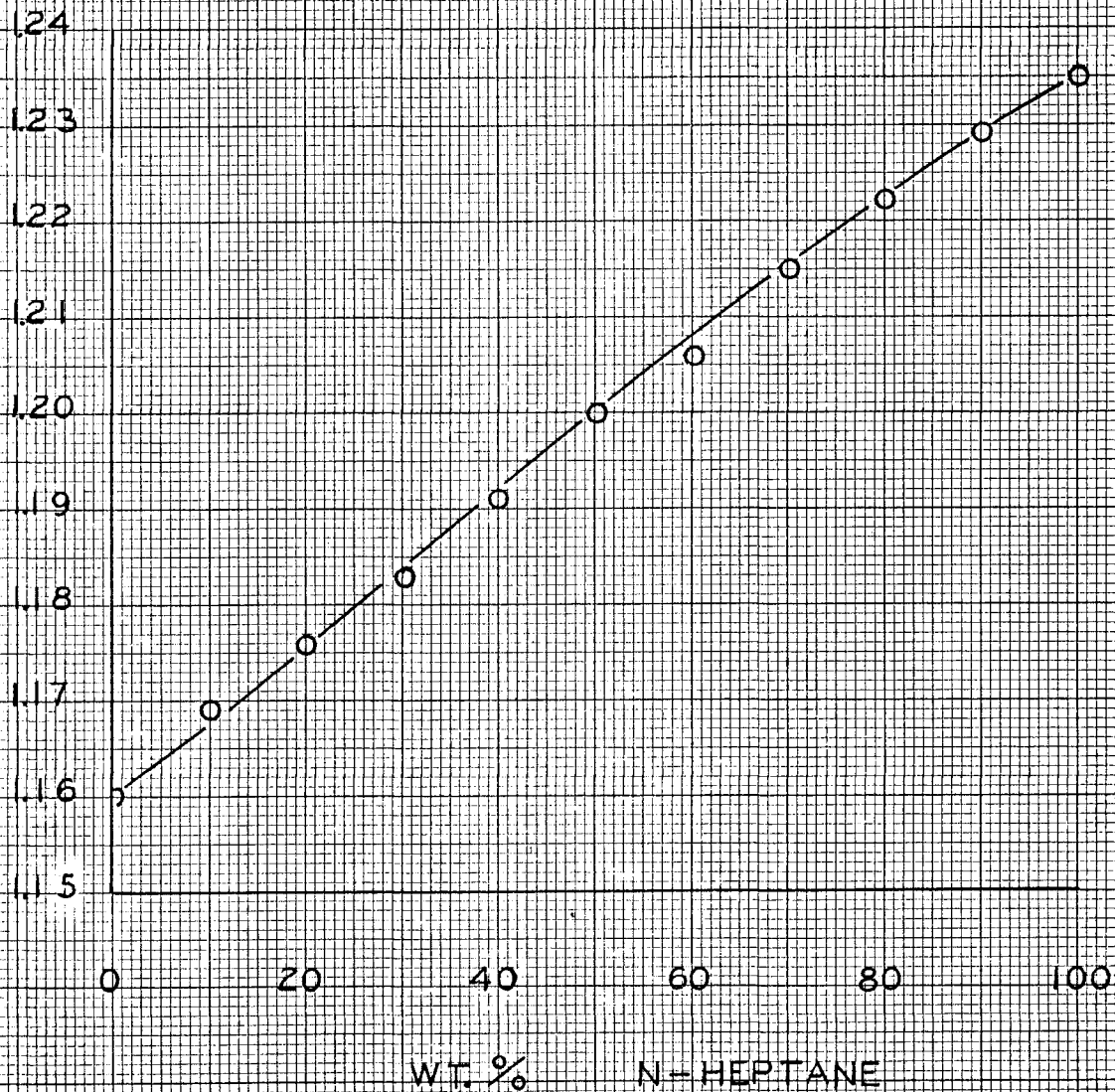
60

80

100

WT. %

N-HEPTANE



## APPENDIX VII

### Boil-Up Correction Factor

Since the reflux is returned to the column at a temperature below its boiling point, the actual boil-up through the column is equal to the quantity of reflux plus that quantity of vapor which is condensed by the cold reflux. Therefore, the reflux rate must be corrected for temperature to determine the actual boil-up through the column. This correction is made by calculating the heat and weight balances at the top of the column as follows:

#### Material balance at the top of the column

$$V_2 + L_1 = V_1 + L_2$$

where,

$V_2$  = lb./hr. vapor to condenser

$L_2$  = lb./hr. liquid from condenser

$V_1$  = lb./hr. boil-up through column

$L_1$  = lb./hr. reflux down column

Since the vapor rate to the condenser is equal to the liquid rate from the condenser,  $V_2 = L_2$ , and substitution in the above equation reveals that  $V_1 = L_1$ .

#### Heat balance at the top of the column

The heat content of the vapor to the condenser is

$$V_2(\Delta H + C_p \Delta T)$$

The heat content of the liquid from the condenser is

$$L_2 C_p \Delta T,$$

The heat content of the reflux down the column is

$$L_1 C_p \Delta T,$$

The heat content of the boil up through the column is

$$V_1 (\Delta H + C_p \Delta T),$$

where,

$\Delta H$  = average latent heat of evaporation, BTU/lb.

$C_p$  = average heat capacity, BTU/lb. °F.

$\Delta T = T_1 - T_2$

$T_1$  = temperature of vapor to condenser, °F.

$T_2$  = condensate temperature, °F.

The heat balance, therefore is as follows:

$$V_1 (\Delta H + C_p \Delta T) + L_2 C_p \Delta T = V_2 (\Delta H + C_p \Delta T) + L_1 C_p \Delta T$$

Combine weight and heat balances

$$V_1 = L_2 + \frac{L_2 C_p (T_1 - T_2)}{\Delta H}$$

where,

$\Delta H$  (n-Heptane) = 137.7 BTU/lb.

$\Delta H$  (Methylcyclohexane) = 138.5 BTU/lb.

$\Delta H$  (average) = 138.1 BTU/lb.

$T$  (n-Heptane) = 209.2°F.

$T$  (Methylcyclohexane) = 213.8°F.

$T_1$  (average) = 211.5°F.

$C_p$  (n-Heptane) = 0.507 BTU/lb. °F.

$C_p$  (Methylcyclohexane) = 0.883 BTU/lb. °F.

$C_p$  (average) = 0.695 BTU/lb. °F.



Substitution,

$$V_1 = L_2 + \frac{L_2 (0.695)(211.5 - T_2)}{138.1}$$

$$V_1 = L_2 (2.0644 - 0.005033 T_2).$$

## APPENDIX VIII

### Tabulation of Data

Any one of the one-inch, two-inch, or three-inch diameter columns was installed on the apparatus and packed with one-quarter inch Berl saddles. Some of the initial work was done with the columns packed with one-half inch Berl saddles. It soon became obvious that this packing was too large for the one-inch diameter column, so all the work with saddles larger than one-quarter inch was discontinued.

Approximately three liters of normal-heptane and three liters of methylcyclohexane were charged to the kettle and heated to the bubble point employing the electric heating mantle. In cases when the three-inch diameter column was employed, the internal steam heated coils were also required to attain maximum boil-up. The column was always flooded to wet the packing thoroughly and then allowed to reach steady state conditions for a minimum of three hours.

After steady state conditions were attained, readings were noted of the date, time, packing size, column diameter, reflux rotameter, reflux temperature, and the base column water manometer pressure. A five cc. sample of the reflux was then obtained after taking an approximately 100 cc. line flush to insure a significant sample. A five cc. sample of the vapor at the base of the column was also taken in the same manner. The samples were numbered in succession and the numbers were noted.

The boil-up through the column was then altered by changing the heat-put into the kettle by adjusting the rheostat to the heating mantle. Two sets of samples and readings were taken each day.

All the data collected are summarized in Table I.

TABLE IData

<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
Start	3-4-64	1600	0.50	2.0					
1	3-4-64	1740	0.50	2.0	1	2	0.93	15.5	0.75
Start	3-5-64	730	0.50	2.0					
2	3-5-64	1130	0.50	2.0	3	4	0.93	16.0	0.75
3	3-5-64	1600	0.50	2.0	5	6	0.93	14.5	0.75
Start	3-6-64	730	0.50	2.0					
4	3-6-64	1215	0.50	2.0	7	8	1.05	15.5	0.75
5	3-6-64	1615	0.50	2.0	9	10	1.05	15.0	0.75
Start	3-9-64	830	0.50	2.0					
6	3-9-64	1200	0.50	2.0	11	12	1.45	17.0	1.38
7	3-9-64	1610	0.50	2.0	13	14	1.45	19.0	1.38
Start	3-10-64	800	0.50	2.0					
8	3-10-64	1130	0.50	2.0	15	16	1.57	18.5	1.75

Table I (Continued)

29.

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
9	3-10-64	1600	0.50	2.0	17	18	1.50	18.0	1.75
Start	3-16-64	830	0.50	1.0					
10	3-16-64	1110	0.50	1.0	19	20	0.14	18.0	0.63
11	3-16-64	1630	0.50	1.0	21	22	0.16	18.0	0.63
Start	3-17-64	800	0.50	1.0					
12	3-17-64	1115	0.50	1.0	23	24	0.27	16.0	0.75
13	3-17-64	1600	0.50	1.0	25	26	0.33	16.0	1.13
Start	3-18-64	730	0.50	1.0					
14	3-18-64	1115	0.50	1.0	27	28	0.50	16.0	1.63
15	3-18-64	1530	0.50	1.0	29	30	0.52	16.0	1.63
Start	3-24-64	730	0.25	1.0					
16	3-24-64	1620	0.25	1.0	31	32	0.05	30.0	1.75
Start	3-25-64	300	0.25	1.0					
17	3-25-64	1115	0.25	1.0	33	34	0.06	32.0	7.50
18	3-25-64	1515	0.25	1.0	35	36	0.03	32.0	2.37
Start	3-26-64	730	0.25	1.0					

Table I (Continued)

30.

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
19	3-26-64	1200	0.25	1.0	37	38	0.04	30.0	8.50
Start	3-30-64	730	0.25	1.0					
20	3-30-64	1530	0.25	1.0	39	40	0.03	29.0	4.25
Start	3-31-64	800	0.25	1.0					
21	3-31-64	1140	0.25	1.0	41	42	0.02	28.5	3.50
22	3-31-64	1430	0.25	1.0	43	44	0.02	30.0	9.00
Start	4-6-64	730	0.25	2.0					
23	4-6-64	1130	0.25	2.0	45	46	0.54	18.0	0.75
24	4-6-64	1600	0.25	2.0	47	48	0.53	18.0	0.75
Start	4-7-64	800	0.25	2.0					
25	4-7-64	1115	0.25	2.0	49	50	0.69	16.0	1.25
26	4-7-64	1430	0.25	2.0	51	52	0.71	16.0	1.50
Start	4-8-64	730	0.25	2.0					
27	4-8-64	1130	0.25	2.0	53	54	0.79	16.0	1.75
28	4-8-64	1515	0.25	2.0	55	56	0.80	18.0	1.88
Start	4-9-64	730	0.25	2.0					

Table I (Continued)

<u>Data</u>	<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
						<u>Base Column</u>	<u>Reflux</u>			
	29	4-9-64	1100	0.25	2.0	57	58	0.56	18.5	0.63
	30	4-9-64	1515	0.25	2.0	59	60	0.53	18.5	0.63
Start		4-10-64	730	0.25	2.0					
	31	4-10-64	1130	0.25	2.0	61	62	0.68	17.5	1.25
	32	4-10-64	1500	0.25	2.0	63	64	0.69	17.0	1.50
Start		4-13-64	730	0.25	2.0					
	33	4-13-64	1130	0.25	2.0	65	66	0.17	24.5	0.50
	34	4-13-64	1630	0.25	2.0	67	68	0.19	26.0	0.25
Start		4-15-64	800	0.25	2.0					
	35	4-15-64	1500	0.25	2.0	69	70	0.31	23.5	0.38
Start		4-16-64	715	0.25	2.0					
	36	4-16-64	1115	0.25	2.0	71	72	0.17	22.5	0.25
	37	4-16-64	1530	0.25	2.0	73	74	0.17	20.5	0.25
Start		4-17-64	730	0.25	2.0					
	38	4-17-64	1115	0.25	2.0	75	76	0.27	20.0	0.38
	39	4-17-64	1500	0.25	2.0	77	78	0.28	20.0	0.38

Table I (Continued)Data

<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
Start	4-20-64	730	0.25	2.0					
40	4-20-64	1110	0.25	2.0	79	80	0.50	21.0	0.75
41	4-20-64	1530	0.25	2.0	81	82	0.50	19.5	0.75
Start	4-21-64	715	0.25	2.0					
42	4-21-64	1230	0.25	2.0	83	84	0.58	21.0	1.25
43	4-21-64	1600	0.25	2.0	85	86	0.57	21.5	1.25
Start	4-22-64	715	0.25	2.0					
44	4-22-64	1200	0.25	2.0	87	88	0.70	21.0	1.50
45	4-22-64	1430	0.25	2.0	89	90	0.71	21.5	1.50
Start	4-23-64	715	0.25	2.0					
46	4-23-64	1115	0.25	2.0	91	92	0.74	18.5	2.00
Start	4-24-64	715	0.25	2.0					
47	4-24-64	1110	0.25	2.0	93	94	0.71	19.5	2.50
48	4-24-64	1430	0.25	2.0	95	96	0.72	20.0	2.75
Start	5-4-64	730	0.25	3.0					



Table I (Continued)

Data

Run No.	Date	Time	Packing Size, in.	Column Dia., in.	Sample No.		Rota. R'd'g gal/hr.	Reflux Temp., °C.	Base Tower Press. in. H <sub>2</sub> O
					Base Column	Reflux			
49	5-4-64	1145	0.25	3.0	97	98	0.95	20.0	0.375
50	5-4-64	1500	0.25	3.0	99	100	0.97	21.0	0.375
Start	5-5-64	730	0.25	3.0					
51	5-5-64	1145	0.25	3.0	101	102	0.88	22.0	0.250
52	5-5-64	1500	0.25	3.0	103	104	1.41	25.0	0.750
Start	5-6-64	730	0.25	3.0					
53	5-6-64	1150	0.25	3.0	105	106	1.41	24.5	0.750
54	5-6-64	1500	0.25	3.0	107	108	1.42	25.0	0.750
Start	5-7-64	730	0.25	3.0					
55	5-7-64	1145	0.25	3.0	109	110	1.90	26.5	1.000
56	5-7-64	1500	0.25	3.0	111	112	1.90	27.5	1.000
Start	5-8-64	730	0.25	3.0					
57	5-8-64	1200	0.25	3.0	113	114	2.08	29.0	1.250
58	5-8-64	1500	0.25	3.0	115	116	2.08	29.0	1.250
Start	5-11-64	730	0.25	3.0					
59	5-11-64	1145	0.25	3.0	117	118	2.35	31.5	1.500
60	5-11-64	1500	0.25	3.0	119	120	2.25	31.5	1.500

Table I (Continued)

34.

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
Start	5-12-64	730	0.25	3.0					
61	5-12-64	1145	0.25	3.0	121	122	2.55	33.0	2.000
62	5-12-64	1515	0.25	3.0	123	124	2.55	34.0	3.25
Start	5-13-64	730	0.25	3.0					
63	5-13-64	1200	0.25	3.0	125	126	2.55	33.0	3.50
64	5-13-64	1500	0.25	3.0	127	128	2.55	31.0	3.00
Start	5-14-64	730	0.25	3.0					
65	5-14-64	1145	0.25	3.0	129	130	1.75	26.0	1.25
66	5-14-64	1500	0.25	3.0	131	132	1.75	26.0	1.25
Start	5-15-64	730	0.25	3.0					
67	5-15-64	1145	0.25	3.0	133	134	2.10	29.0	1.75
68	5-15-64	1500	0.25	3.0	135	136	2.10	29.0	1.75
Start	6-15-64	715	0.25	3.0					
69	6-15-64	1105	0.25	3.0	137	318	2.70	41.0	5.70
70	6-15-64	1730	0.25	3.0	139	140	2.70	41.0	4.50
Start	6-18-64	715	0.25	3.0					

Table I (Continued)

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Pressure in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
Start	7-1-64	700	0.25	3.0					
82	7-1-64	1140	0.25	3.0	163	164	2.07	32.0	1.50
83	7-1-64	1615	0.25	3.0	165	166	2.07	38.0	1.50
Start	7-2-64	705	0.25	3.0					
84	7-2-64	1135	0.25	3.0	167	168	2.07	37.0	1.00
85	7-2-64	1500	0.25	3.0	169	170	2.07	37.5	1.00
Start	7-23-64	700	0.25	1.0					
86	7-23-64	1030	0.25	1.0	171	172	0.18	25.0	4.0
87	7-23-64	1545	0.25	1.0	173	174	0.19	26.0	6.0
Start	7-24-64	700	0.25	1.0					
88	7-24-64	1045	0.25	1.0	175	176	0.19	25.0	4.5
89	7-24-64	1430	0.25	1.0	177	178	0.18	25.0	6.0
Start	7-27-64	700	0.25	1.0					
90	7-27-64	1105	0.25	1.0	179	180	0.11	25.0	2.0
91	7-27-64	1445	0.25	1.0	181	182	0.12	26.5	2.0
Start	7-28-64	700	0.25	1.0					

Table I (Continued)

35.

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
71	6-18-64	1105	0.25	3.0	141	142	3.48	64.0	10.00
Start	6-24-64	700	0.25	3.0					
72	6-24-64	1100	0.25	3.0	143	144	2.08	32.0	1.50
73	6-24-64	1545	0.25	3.0	145	146	2.70	40.0	2.50
Start	6-25-64	700	0.25	3.0					
74	6-25-64	1105	0.25	3.0	147	148	3.10	48.0	5.5
75	6-25-64	1535	0.25	3.0	149	150	3.43	56.0	10.0
Start	6-26-64	715	0.25	3.0					
76	6-26-64	1100	0.25	3.0	151	152	2.15	38.0	1.75
77	6-26-64	1445	0.25	3.0	153	154	2.27	39.0	1.75
Start	2-29-64	710	0.25	3.0					
78	6-29-64	1145	0.25	3.0	155	156	2.45	39.0	2.25
79	6-29-64	1415	0.25	3.0	157	158	2.45	39.0	2.25
Start	6-30-64	700	0.25	3.0					
80	6-30-64	1040	0.25	3.0	159	160	1.70	34.0	1.00
81	6-30-64	1500	0.25	3.0	161	162	1.70	34.0	1.00

Table I (Continued)

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
92	7-28-64	1120	0.25	1.0	183	184	0.07	25.0	1.25
93	7-28-64	11400	0.25	1.0	185	186	0.08	27.0	1.25
Start	7-29-64	700	0.25	1.0					
94	7-29-64	1050	0.25	1.0	187	188	0.16	25.5	3.00
95	7-29-64	11400	0.25	1.0	189	190	0.18	27.0	3.25
Start	7-30-64	700	0.25	1.0					
96	7-30-64	1110	0.25	1.0	191	192	0.10	23.0	1.75
97	7-30-64	1145	0.25	1.0	193	194	0.12	25.0	2.00
Start	8-5-64	700	0.25	1.0					
98	8-5-64	1010	0.25	1.0	195	196	0.07	20.5	1.50
99	8-5-64	1110	0.25	1.0	197	198	0.12	26.5	2.00
Start	8-6-64	700	0.25	1.0					
100	8-6-64	1030	0.25	1.0	199	200	0.16	25.0	3.50
101	8-6-64	1125	0.25	1.0	201	202	0.14	24.5	2.50
Start	8-7-64	700	0.25	1.0					

Table I (Continued)

38.

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
102	8-7-64	930	0.25	1.0	203	204	0.13	24.5	2.75
103	8-7-64	1400	0.25	1.0	205	206	0.12	24.0	2.00
Start	8-10-64	700	0.25	2.0					
104	8-10-64	1030	0.25	2.0	207	208	0.55	25.5	0.80
105	8-10-64	1415	0.25	2.0	209	210	0.50	25.0	0.75
Start	8-11-64	700	0.25	2.0					
106	8-11-64	1155	0.25	2.0	211	212	0.63	25.5	1.00
107	8-11-64	1420	0.25	2.0	213	214	0.70	27.5	1.25
Start	8-12-64	700	0.25	2.0					
108	8-12-64	1020	0.25	2.0	215	216	0.44	25.5	0.75
109	8-12-64	1310	0.25	2.0	217	218	0.43	25.5	0.70
Start	8-13-64	700	0.25	2.0					
110	8-13-64	1020	0.25	2.0	219	220	0.32	24.5	0.50
111	8-13-64	1515	0.25	2.0	221	222	0.30	24.0	0.50
Start	8-14-64	700	0.25	2.0					

Table I (Continued)

<u>Data</u>									
<u>Run No.</u>	<u>Date</u>	<u>Time</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	<u>Sample No.</u>		<u>Rota. R'd'g gal/hr.</u>	<u>Reflux Temp., °C.</u>	<u>Base Tower Press. in. H<sub>2</sub>O</u>
					<u>Base Column</u>	<u>Reflux</u>			
112	8-14-64	945	0.25	2.0	223	224	0.23	23.0	0.40
113	8-14-64	1345	0.25	2.0	225	226	0.23	23.0	0.40
Start	8-17-64	705	0.25	2.0					
114	8-17-64	1050	0.25	2.0	227	228	0.65	25.5	1.50
115	8-17-64	1545	0.25	2.0	229	230	0.70	26.0	1.50
Start	8-18-64	710	0.25	2.0					
116	8-18-64	1050	0.25	2.0	231	232	0.80	26.5	2.50
117	8-18-64	1345	0.25	2.0	233	234	0.85	27.5	2.50
Start	8-19-64	700	0.25	2.0					
118	8-19-64	1100	0.25	2.0	235	236	0.93	26.0	3.00
119	8-19-64	1620	0.25	2.0	237	238	0.93	27.0	6.00

## APPENDIX IX

### Analysis of Data

The refractive index at 20°C. was measured on all samples obtained. The composition of each sample was then determined from the refractive index vs. composition curve, Figure A-2, Appendix IV.

The results of the analyses are summarized in Table II.



TABLE II

Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column		Sample No.	Reflux			
		$N_D$ (20°C.)	Wt.% n-Heptane		Wt.% Methylcyclohexane	$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane
1	1	1.4078	51.5	48.5	2	1.4024	71.5	28.5
2	3	1.4093	46.0	54.0	4	1.4033	68.0	32.0
3	5	1.4081	50.5	49.5	6	1.4029	69.5	30.5
4	7	1.4088	48.0	52.0	8	1.4001	69.0	31.0
5	9	1.4088	48.0	52.0	10	1.4030	69.2	30.8
6	11	1.4093	46.0	54.0	12	1.4035	67.5	32.5
7	13	1.4094	45.5	54.5	14	1.4037	66.5	33.5
8	15	1.4109	40.0	60.0	16	1.4039	66.0	34.0
9	17	1.4098	44.0	56.0	18	1.4040	65.5	34.5
10	19	1.4100	43.5	56.5	20	1.4048	62.5	37.5
11	21	1.4105	41.5	58.5	22	1.4052	61.0	39.0
12	23	1.4109	40.0	60.0	24	1.4055	60.0	40.0
13	25	1.4110	39.5	60.5	26	1.4053	61.0	39.0
14	27	1.4114	38.0	62.0	28	1.4061	58.0	42.0

Table II (Continued)

Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column			Sample No.	Reflux		
		$N_D$ (20°C.)	Wt. % n-Heptane	Wt. % Methylcyclohexane		$N_D$ (20°C.)	Wt. % n-Heptane	Wt. % Methylcyclohexane
15	29	1.4115	37.5	62.5	30	1.4065	56.5	43.5
16	31	1.4103	42.0	58.0	32	1.3975	89.5	10.5
17	33	1.4105	41.5	58.5	34	1.3986	85.5	14.5
18	35	1.4107	40.5	59.5	36	1.3978	88.5	11.5
19	37	1.4117	37.0	63.0	38	1.4040	65.5	34.5
20	39	1.4133	31.0	69.0	40	1.4029	69.5	30.5
21	41	1.4138	29.0	71.0	42	1.4046	63.5	36.5
22	43	1.4140	28.5	71.5	44	1.4055	60.0	40.0
23	45	1.4095	45.0	55.0	46	1.3977	89.0	11.0
24	47	1.4093	46.0	54.0	48	1.3984	86.5	13.5
25	49	1.4097	44.5	55.5	50	1.3985	86.0	14.0
26	51	1.4100	43.5	56.5	52	1.3984	84.0	16.0
27	53	1.4107	40.5	59.5	54	1.3994	82.5	17.5
28	55	1.4104	42.0	58.0	56	1.3998	81.0	19.0

TABLE II (Continued)

Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column			Sample No.	Reflux		
		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane
29	57	1.4108	40.5	59.5	58	1.3998	81.0	19.0
30	59	1.4109	50.0	60.0	60	1.3996	82.0	18.0
31	61	1.4110	39.0	61.0	62	1.4010	76.5	23.5
32	63	1.4115	37.5	62.5	64	1.4015	75.0	25.0
33	65	1.4119	36.0	64.0	66	1.4029	69.5	30.5
34	67	1.4117	37.0	63.0	68	1.4029	69.5	30.5
35	69	1.4120	35.5	64.5	70	1.4043	64.5	35.5
36	71	1.4125	34.0	66.0	72	1.4050	62.0	38.0
37	73	1.4125	34.0	66.0	74	1.4050	62.0	38.0
38	75	1.4128	33.0	67.0	76	1.4060	58.0	42.0
39	77	1.4130	32.0	68.0	78	1.4065	56.5	43.5
40	79	1.4138	29.0	71.0	80	1.4064	57.0	43.0
41	81	1.4138	29.0	71.0	82	1.4070	54.5	45.5
42	83	1.4140	28.5	71.5	84	1.4073	53.5	46.5

TABLE II (Continued)

Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column			Sample No.	Reflux		
		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane
43	85	1.4142	27.5	72.5	86	1.4077	52.5	47.5
44	87	1.4147	26.0	74.0	88	1.4086	48.5	51.5
45	89	1.4150	24.5	75.5	90	1.4089	47.5	52.5
46	91	1.4152	24.0	76.0	92	1.4093	46.0	54.0
47	93	1.4157	22.0	78.0	94	1.4100	43.0	57.0
48	95	1.4155	22.5	77.5	96	1.4102	42.5	57.5
49	97	1.4101	43.0	57.0	98	1.4071	54.0	46.0
50	99	1.4101	43.0	57.0	100	1.4070	54.5	45.5
51	101	1.4102	42.5	57.5	102	1.4068	55.0	45.0
52	103	1.4105	41.5	58.5	104	1.4074	53.0	47.0
53	105	1.4110	39.5	60.5	106	1.4075	52.5	47.5
54	107	1.4110	39.5	60.5	108	1.4073	53.5	46.5
55	109	1.4110	39.5	60.5	110	1.4078	51.5	48.5
56	111	1.4110	39.5	60.5	112	1.4080	50.5	49.5

TABLE II (Continued)Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column			Sample No.	Reflux		
		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane
57	113	1.4118	36.5	63.5	114	1.4083	49.5	50.5
58	115	1.4123	35.0	65.0	116	1.4091	46.5	53.5
59	117	1.4129	32.5	67.5	118	1.4092	46.0	54.0
60	119	1.4132	31.5	68.5	120	1.4096	44.5	55.5
61	121	1.4133	31.0	69.0	122	1.4091	46.5	53.5
62	123	1.4133	31.0	69.0	124	1.4092	46.0	54.0
63	125	1.4110	39.5	60.5	126	1.4055	60.0	40.0
64	127	1.4110	39.5	60.5	128	1.4062	57.5	42.5
65	129	1.4105	41.5	58.5	130	1.4073	53.5	46.5
66	131	1.4107	40.5	59.5	132	1.4070	54.5	45.5
67	133	1.4106	41.0	59.0	134	1.4071	54.0	46.0
68	135	1.4106	41.0	59.0	136	1.4071	54.0	46.0
69	137	1.4114	38.0	62.0	138	1.4055	60.0	40.0
70	139	1.4112	39.0	61.0	140	1.4062	57.5	42.5

TABLE II (Continued)Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column			Sample No.	Reflux		
		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane
71	141	1.4130	32.0	68.0	142	1.4010	76.5	23.5
72	143	1.4122	35.0	65.0	144	1.4085	49.0	51.0
73	145	1.4123	34.5	65.5	146	1.4077	52.0	48.0
74	147	1.4129	32.5	67.5	148	1.4064	56.5	43.5
75	149	1.4142	27.5	72.5	150	1.4000	80.4	19.6
76	151	1.4125	34.0	66.0	152	1.4089	47.5	52.5
77	153	1.4125	34.0	66.0	154	1.4088	47.5	52.5
78	155	1.4129	32.5	67.5	156	1.4088	47.5	52.5
79	157	1.4129	32.5	67.5	158	1.4093	46.0	54.0
80	159	1.4128	33.0	67.0	160	1.4099	43.5	56.5
81	161	1.4130	32.0	68.0	162	1.4099	43.5	56.5
82	163	1.4130	32.0	68.0	164	1.4098	44.0	56.0
83	165	1.4130	32.0	68.0	166	1.4096	45.0	55.0
84	167	1.4130	32.0	68.0	168	1.4098	44.0	56.0

TABLE II (Continued)

Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column			Sample No.	Reflux		
		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane
85	169	1.4130	32.0	68.0	170	1.4097	44.5	55.5
86	171	1.4145	26.5	73.5	172	1.4046	63.5	36.5
87	173	1.4157	22.0	78.0	174	1.4052	61.0	39.0
88	175	1.4144	27.0	73.0	176	1.4013	75.5	24.5
89	177	1.4149	25.0	75.0	178	1.4020	73.0	27.0
90	179	1.4143	27.5	72.5	180	1.4047	63.0	37.0
91	181	1.4146	26.0	74.0	182	1.4048	62.5	37.5
92	183	1.4148	25.5	74.5	184	1.4068	55.0	45.0
93	185	1.4152	24.0	76.0	186	1.4075	52.5	47.5
94	187	1.4158	21.5	78.5	188	1.4083	49.5	50.5
95	189	1.4163	20.0	80.0	190	1.4088	47.5	52.5
96	191	1.4157	22.0	78.0	192	1.4076	52.0	48.0
97	193	1.4157	22.0	78.0	194	1.4073	53.5	46.5
98	195	1.4158	21.5	78.5	196	1.4094	45.5	54.5
99	197	1.4160	21.0	79.0	198	1.4089	47.5	52.5

TABLE II (Continued)

Composition of the Samples as a Function of Refractive Index ( $N_D$ )

Run No.	Sample No.	Base Column			Sample No.	Reflux		
		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane		$N_D$ (20°C.)	Wt.% n-Heptane	Wt.% Methylcyclohexane
100	199	1.4163	20.0	80.0	200	1.4090	47.0	53.0
101	201	1.4166	19.0	81.0	202	1.4092	46.5	53.5
102	203	1.4166	19.0	81.0	204	1.4102	42.5	57.5
103	205	1.4166	19.0	81.0	206	1.4101	43.0	57.0
104	207	1.4113	38.5	61.5	208	1.3997	81.5	18.5
105	209	1.4115	37.5	62.5	210	1.3991	83.5	16.5
106	211	1.4117	37.0	63.0	212	1.3991	83.5	16.5
107	213	1.4118	36.5	63.5	214	1.3991	83.5	16.5
108	215	1.4118	36.5	63.5	216	1.4000	80.5	19.5
109	217	1.4118	36.5	63.5	218	1.4000	80.5	19.5
110	219	1.4118	36.5	63.5	220	1.4023	72.0	28.0
111	221	1.4118	36.5	63.5	222	1.4031	69.0	31.0
112	223	1.4120	36.0	64.0	224	1.4028	70.0	30.0
113	225	1.4120	36.0	64.0	226	1.4018	73.5	26.5



TABLE II (Continued)Composition of the Samples as a Function of Refractive Index ( $N_D$ )

<u>Run No.</u>	<u>Sample No.</u>	<u>Base Column</u>			<u>Sample No.</u>	<u>Reflux</u>		
		<u><math>N_D</math> (20°C.)</u>	<u>Wt.% n-Heptane</u>	<u>Wt.% Methylcyclohexane</u>		<u><math>N_D</math> (20°C.)</u>	<u>Wt.% n-Heptane</u>	<u>Wt.% Methylcyclohexane</u>
114	227	1.4125	34.0	66.0	228	1.4008	77.5	22.5
115	229	1.4125	34.0	66.0	230	1.4014	75.0	25.0
116	231	1.4125	34.0	66.0	232	1.4003	79.0	21.0
117	233	1.4125	34.0	66.0	234	1.4008	77.5	22.5
118	235	1.4127	32.5	67.5	236	1.4001	80.0	20.0
119	237	1.4131	31.5	68.5	238	1.4003	79.0	21.0

## APPENDIX X

### Sample Calculation

For each set of samples, the height equivalent to a theoretical plate and the superficial vapor velocity were calculated. A sample calculation follows:

#### Calculation of HETP, ft.

$$N = \frac{\log \left[ \frac{X_h}{X_m} \right]_D \left[ \frac{X_m}{X_h} \right]_B}{\log \alpha} \quad 2$$

Where,

- N = number of theoretical plates
- $X_h$  = weight % normal heptane in liquid
- $X_m$  = weight % methyl cyclohexane in liquid
- D = distillate
- B = still pot
- $\alpha$  = relative volatility

For samples No. 1 and 2, 3-4-64,

$$\begin{aligned} \left[ \frac{X_h}{X_m} \right]_D &= 71.5 && \text{(Appendix IX)} \\ \left[ \frac{X_m}{X_h} \right]_D &= 28.5 && \text{(Appendix IX)} \\ \left[ \frac{X_h}{X_m} \right]_B &= 51.5 && \text{(Appendix IX)} \\ \left[ \frac{X_m}{X_h} \right]_B &= 48.5 && \text{(Appendix IX)} \\ \alpha &= 1.085 && \text{(Appendix III)} \end{aligned}$$

$$N = \frac{\log \left[ \frac{71.5}{28.5} \right] \left[ \frac{48.5}{51.5} \right]}{\log 1.085} = 10.55 \text{ Plates}$$

$$\text{HETP} = \frac{\text{Column height, ft.}}{N} = \frac{3.00 \text{ ft.}}{10.55} = 0.284 \text{ ft.}$$

Calculation of G, lb/sec ft.<sup>2</sup>  
for Samples No. 1 and 2, 3-4-64

<sup>2</sup> Teller, A. J., Binary Distillation, McGraw-Hill Publishing Co., New York, 1964.

Rotameter reading = 0.93 gal/hr. (Appendix VIII).

Weight % normal-heptane in the reflux = 71.5% (Appendix IX).

Rotameter correction factor = 1.216 (Figure A-6).

Corrected rotameter reading = (0.93)(1.216) = 1.131 gal/hr.

Specific gravity of reflux = 0.706 (Figure A-5)

Reflux rate = (1.131 gal/hr.) (0.706)(8.33 lb/gal.) = 6.66 lb/hr.

Condensate temperature = 15.5°C. = 59.9°F. (Appendix VIII).

Boil-up rate = 6.66  $\left[ 2.0644 - 0.005033 (59.9) \right]$   
 = 11.73 lb/hr. (Appendix VII).

Column cross sectional area =  $\frac{(1.0)^2}{144}$  = 0.0218 ft.<sup>2</sup>

$$G = \frac{(11.73 \text{ lb/hr.})}{(3600 \text{ sec/hr.})(0.0218 \text{ ft.}^2)} = 0.149 \text{ lb/sec. ft.}^2$$

Table III summarizes the results of all the calculations:

TABLE IIISummary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. ft.<sup>2</sup></u>	<u>HETP ft.</u>
1	1, 2	0.50	2.0	0.25	0.149	0.284
2	3, 4	0.50	2.0	0.25	0.149	0.268
3	5, 6	0.50	2.0	0.25	0.151	0.305
4	7, 8	0.50	2.0	0.25	0.169	0.278
5	9, 10	0.50	2.0	0.25	0.170	0.275
6	11, 12	0.50	2.0	0.46	0.232	0.275
7	13, 14	0.50	2.0	0.46	0.229	0.281
8	15, 16	0.50	2.0	0.58	0.249	0.229
9	17, 18	0.50	2.0	0.58	0.239	0.277
10	19, 20	0.50	1.0	0.21	0.089	0.317
11	21, 22	0.50	1.0	0.21	0.102	0.309
12	23, 24	0.50	1.0	0.25	0.174	0.301
13	25, 26	0.50	1.0	0.38	0.212	0.280
14	27, 28	0.50	1.0	0.54	0.323	0.301

TABLE III (Continued)Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. ft.<sup>2</sup></u>	<u>HETP ft.</u>
15	29, 30	0.50	1.0	0.54	0.335	0.317
16	31, 32	0.25	1.0	0.58	0.030	0.099
17	33, 34	0.25	1.0	2.50	0.035	0.116
18	35, 36	0.25	1.0	0.79	0.018	0.101
19	37, 38	0.25	1.0	2.83	0.024	0.209
20	39, 40	0.25	1.0	1.41	0.018	0.151
21	41, 42	0.25	1.0	1.117	0.012	0.169
22	43, 44	0.25	1.0	3.00	0.012	0.185
23	45, 46	0.25	2.0	0.25	0.085	0.107
24	47, 48	0.25	2.0	0.25	0.084	0.122
25	49, 50	0.25	2.0	0.63	0.110	0.121
26	51, 52	0.25	2.0	0.75	0.112	0.128
27	53, 54	0.25	2.0	0.88	0.126	0.130
28	55, 56	0.25	2.0	0.63	0.126	0.138

TABLE III (Continued)Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. ft.<sup>2</sup></u>	<u>HETP ft.</u>
29	57, 58	0.25	2.0	0.21	0.088	0.133
30	59, 60	0.25	2.0	0.21	0.086	0.128
31	61, 62	0.25	2.0	0.42	0.108	0.150
32	63, 64	0.25	2.0	0.50	0.110	0.152
33	65, 66	0.25	2.0	0.17	0.026	0.175
34	67, 68	0.25	2.0	0.08	0.029	0.181
35	69, 70	0.25	2.0	0.13	0.048	0.180
36	71, 72	0.25	2.0	0.08	0.027	0.212
37	73, 74	0.25	2.0	0.08	0.027	0.212
38	75, 76	0.25	2.0	0.13	0.043	0.237
39	77, 78	0.25	2.0	0.13	0.044	0.239
40	79, 80	0.25	2.0	0.25	0.077	0.208
41	81, 82	0.25	2.0	0.25	0.079	0.227
42	83, 84	0.25	2.0	0.42	0.092	0.231

TABLE III (Continued)Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. Ft.<sup>2</sup></u>	<u>HETP ft.</u>
43	85, 86	0.25	2.0	0.42	0.090	0.229
44	87, 88	0.25	2.0	0.50	0.111	0.249
45	89, 90	0.25	2.0	0.50	0.112	0.238
46	91, 92	0.25	2.0	0.67	0.119	0.247
47	93, 94	0.25	2.0	0.83	0.114	0.249
48	95, 96	0.25	2.0	0.92	0.115	0.261
49	97, 98	0.25	3.0	0.125	0.067	0.553
50	99, 100	0.25	3.0	0.125	0.068	0.529
51	101, 102	0.25	3.0	0.083	0.061	0.487
52	103, 104	0.25	3.0	0.250	0.097	0.528
53	105, 106	0.25	3.0	0.250	0.097	0.464
54	107, 108	0.25	3.0	0.250	0.098	0.432
55	109, 110	0.25	3.0	0.333	0.129	0.503
56	111, 112	0.25	3.0	0.333	0.129	0.548

TABLE III (Continued)Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. Ft.<sup>2</sup></u>	<u>HETP ft.</u>
57	113, 114	0.25	3.0	0.417	0.140	0.458
58	115, 116	0.25	3.0	0.417	0.140	0.511
59	117, 118	0.25	3.0	0.500	0.155	0.440
60	119, 120	0.25	3.0	0.500	0.149	0.452
61	121, 122	0.25	3.0	0.667	0.168	0.381
62	123, 124	0.25	3.0	1.083	0.167	0.393
63	125, 126	0.25	3.0	1.167	0.167	0.302
64	127, 128	0.25	3.0	1.000	0.169	0.346
65	129, 130	0.25	3.0	0.417	0.119	0.519
66	131, 132	0.25	3.0	0.417	0.119	0.428
67	133, 134	0.25	3.0	0.583	0.141	0.482
68	135, 136	0.25	3.0	0.583	0.141	0.482
69	137, 138	0.25	3.0	1.900	0.169	0.274
70	139, 140	0.25	3.0	1.500	0.169	0.327



TABLE III (Continued)Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. Ft.<sup>2</sup></u>	<u>HETP ft.</u>
71	141, 142	0.25	3.0	3.333	0.172	0.127
72	143, 144	0.25	3.0	0.500	0.135	0.423
73	145, 146	0.25	3.0	0.833	0.170	0.339
74	147, 148	0.25	3.0	1.833	0.186	0.247
75	149, 150	0.25	3.0	3.333	0.191	0.103
76	151, 152	0.25	3.0	0.583	0.138	0.435
77	153, 154	0.25	3.0	0.583	0.145	0.435
78	155, 156	0.25	3.0	0.750	0.156	0.388
79	157, 158	0.25	3.0	0.750	0.156	0.429
80	159, 160	0.25	3.0	0.333	0.111	0.548
81	161, 162	0.25	3.0	0.333	0.111	0.497
82	163, 164	0.25	3.0	0.500	0.137	0.637
83	165, 166	0.25	3.0	0.500	0.132	0.611
84	167, 168	0.25	3.0	0.333	0.133	0.477

TABLE III (Continued)Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. Ft.<sup>2</sup></u>	<u>HETP ft.</u>
85	169, 170	0.25	3.0	0.333	0.133	0.459
86	171, 172	0.25	1.0	1.333	0.111	0.156
87	173, 174	0.25	1.0	2.000	0.116	0.143
88	175, 176	0.25	1.0	1.500	0.116	0.115
89	177, 178	0.25	1.0	2.000	0.110	0.117
90	179, 180	0.25	1.0	0.667	0.068	0.163
91	181, 182	0.25	1.0	0.667	0.073	0.157
92	183, 184	0.25	1.0	0.417	0.043	0.192
93	185, 186	0.25	1.0	0.417	0.049	0.195
94	187, 188	0.25	1.0	1.000	0.099	0.192
95	189, 190	0.25	1.0	1.083	0.110	0.190
96	191, 192	0.25	1.0	0.583	0.062	0.182
97	193, 194	0.25	1.0	0.667	0.074	0.174
98	195, 196	0.25	1.0	0.500	0.044	0.220

TABLE III (Continued)Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. Ft.<sup>2</sup></u>	<u>HETP ft.</u>
99	197, 198	0.25	1.0	0.667	0.074	0.189
100	199, 200	0.25	1.0	1.167	0.099	0.193
101	201, 202	0.25	1.0	0.833	0.087	0.187
102	203, 204	0.25	1.0	0.917	0.081	0.213
103	205, 206	0.25	1.0	0.667	0.075	0.210
104	207, 208	0.25	2.0	0.267	0.083	0.125
105	209, 210	0.25	2.0	0.250	0.076	0.115
106	211, 212	0.25	2.0	0.333	0.095	0.114
107	213, 214	0.25	2.0	0.417	0.105	0.113
108	215, 216	0.25	2.0	0.250	0.067	0.124
109	217, 218	0.25	2.0	0.233	0.065	0.124
110	219, 220	0.25	2.0	0.167	0.049	0.163
111	221, 222	0.25	2.0	0.167	0.046	0.181
112	223, 224	0.25	2.0	0.133	0.036	0.172

TABLE III (Continued)

60.

Summary of Calculations

<u>Run No.</u>	<u>Sample No.</u>	<u>Packing Size, in.</u>	<u>Column Dia., in.</u>	$\frac{\Delta P}{\text{in. H}_2\text{O}}$ <u>ft.</u>	$\frac{G}{\text{Lb.}}$ <u>Sec. Ft.<sup>2</sup></u>	<u>HETP ft.</u>
113	225, 226	0.25	2.0	0.133	0.036	0.153
114	227, 228	0.25	2.0	0.500	0.099	0.129
115	229, 230	0.25	2.0	0.500	0.106	0.139
116	231, 232	0.25	2.0	0.833	0.121	0.123
117	233, 234	0.25	2.0	0.833	0.128	0.129
118	235, 236	0.25	2.0	1.000	0.141	0.111
119	237, 238	0.25	2.0	2.000	0.140	0.116

## APPENDIX XI

### The Relationship Between Height Equivalent to a Theoretical Plate and Height of a Transfer Unit

The interaction between vapor and liquid in packed towers is continuously countercurrent, rather than stepwise-countercurrent as in bubble plate towers. Calculations involving this continuous countercurrent process should be treated differentially rather than stepwise.

The following equation represents the rigorous design equation for mass transfer operations involving equimolar counterdiffusion as occurs in the packed fractionating columns employed in this work:

$$\int_0^z dz = \int_{y_1}^{y_2} \frac{d(Vy)}{K'_y a S (y^* - y)}$$

where

- z = tower height
- v = vapor rate
- y = more volatile component in vapor phase
- a = interfacial area per unit volume of packing
- s = empty tower cross-sectional area
- y\* = equilibrium composition
- y<sub>1</sub> = composition at bottom of tower
- y<sub>2</sub> = composition at top of tower
- K'<sub>y</sub> = mass transfer coefficient

Assuming that the vapor rate and mass transfer coefficients are constant, the above equation reduces to

$$\int_0^z dz = \frac{v}{K'_y a S} \int_{y_1}^{y_2} \frac{dy}{(y^* - y)}$$

Where the integral term is defined as the number of transfer units (N) and the quantity outside the integral is defined as the height of a transfer unit (H.). The value of H, of course, can also be determined by dividing the column height by N.

As is shown in Appendix III, the relative volatility of the binary system employed in this work is substantially constant. In addition, the column was operated at total reflux so that the operating line coincides with the 45° diagonal and  $y = x$ . By performing the proper substitutions into the integral term of the above equation, the equation for the number of transfer units integrates to

$$N = \frac{1}{\alpha - 1} \ln \frac{y_2(1-y_1)}{y_1(1-y_2)} + \ln \frac{1-y_1}{1-y_2} .$$

The number of theoretical plates is calculated from the Fenske equation:

$$n = \frac{\log \left[ \frac{x_2}{1-x_2} \right] \left[ \frac{1-x_1}{x_1} \right]}{\log \alpha} = \frac{\ln \left[ \frac{x_2}{1-x_2} \right] \left[ \frac{1-x_1}{x_1} \right]}{\ln \alpha}$$

As the value of the relative volatility approaches unity,  $\ln \alpha$  approaches  $\alpha - 1$  (the actual relative volatility for the binary employed in this work is 1.085). Since the relative volatility is close to unity, it can be assumed that the composition of liquid and vapor are nearly equal at the extremities of the column. The Fenske equation, therefore, reduces to the following:

$$n = \frac{1}{\alpha - 1} \ln \frac{y_2(1-y_1)}{y_1(1-y_2)}$$

Since the term,  $\ln \frac{1-y_1}{1-y_2}$  is small compared to  $\frac{1}{\alpha - 1} \ln \frac{y_2(1-y_1)}{y_1(1-y_2)}$ ,

because  $\frac{1}{\alpha - 1}$  is large, n is nearly equal to N.

In order to prove the validity of the above assumptions, several runs were selected at random and the height of a transfer unit was calculated for each. The results of these calculations are presented in the following table:

<u>Run No.</u>	<u>H</u>	<u>HETP</u>	<u>% Deviation</u>
16	0.098	0.099	1.0
28	0.136	0.138	1.5
42	0.235	0.231	1.7
56	0.554	0.548	1.1
66	0.434	0.428	1.4

The above deviations are well within experimental error; therefore, the application of the Fenske equation was entirely satisfactory for the calculations of this work.

## APPENDIX XII

### Quadratic Regression Equation

Although extreme precautions and care were employed in obtaining and correlating the data, considerable scatter of the data resulted when the height equivalent to a plate was plotted against the superficial vapor velocity. For this reason, the non-linear regression equation was employed to correlate the data. The use of this equation assumes that all or part of the curve follows the equation.

$$\text{HETP} = a + bG + cG^2$$

where,

HETP = height equivalent to a theoretical plate, ft.

G = superficial vapor velocity, lb/sec. ft.<sup>2</sup>

a, b, c = constants of the equation

This in fact, proved to be an excellent equation.

No attempt will be made herein to detail the calculations, however, the method employed is as follows:<sup>3</sup>

1. All the HETP and G data for each of the one-inch, two-inch, three-inch diameter columns are tabulated.

2. The products, (G)(HETP), (G<sup>2</sup>)(HETP), G<sup>2</sup>, G<sup>3</sup>, and G<sup>4</sup>, and the summations of these products are included in this table as shown below:

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<sup>3</sup> Goulden, C. H., Methods of Statistical Analysis, John Wiley and Sons, Inc., New York, 1956



$$\begin{array}{ccccccc}
 \frac{G}{\Sigma G} & \frac{\text{HETP}}{\Sigma \text{HETP}} & \frac{(G)(\text{HETP})}{\Sigma (G)(\text{HETP})} & \frac{(G^2)(\text{HETP})}{\Sigma (G^2)(\text{HETP})} & \frac{G^2}{\Sigma G^2} & \frac{G^3}{\Sigma G^3} & \frac{G^4}{\Sigma G^4}
 \end{array}$$

3. These summations are then substituted into the following three equations and the equations are solved simultaneously for the constants a, b, and c:

$$an + b\Sigma G + c\Sigma G^2 = \Sigma \text{HETP}$$

$$a\Sigma G + b\Sigma G^2 + c\Sigma G^3 = \Sigma (G)(\text{HETP})$$

$$a\Sigma G^2 + b\Sigma G^3 + c\Sigma G^4 = \Sigma (G^2)(\text{HETP})$$

where,

n = number of data points

The solutions of these equations are as follows:

for the one-inch diameter column

$$\text{HETP} = 0.156 + 0.513G - 3.808 G^2$$

for the two-inch diameter column

$$\text{HETP} = 0.207 - 0.659G + 1.984 G^2$$

for the three-inch diameter column

$$\text{HETP} = 0.051 + 9.411G - 46.122 G^2$$

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