

71-24,911

MEAD, Richard Wilson, 1941-
THE EFFECT OF VARIATION OF DESIGN PARAMETERS
ON THE MASS TRANSFER EFFICIENCY OF PACKED
DISTILLATION COLUMNS.

The University of Arizona, Ph.D., 1971
Engineering, chemical

University Microfilms, A XEROX Company, Ann Arbor, Michigan

THE EFFECT OF VARIATION OF DESIGN PARAMETERS ON THE MASS
TRANSFER EFFICIENCY OF PACKED DISTILLATION COLUMNS

by

Richard Wilson Mead

A Dissertation Submitted to the Faculty of the
DEPARTMENT OF CHEMICAL ENGINEERING
In Partial Fulfillment of the Requirements
For the Degree of
DOCTOR OF PHILOSOPHY
In the Graduate College
THE UNIVERSITY OF ARIZONA

1 9 7 1

THE UNIVERSITY OF ARIZONA

GRADUATE COLLEGE

I hereby recommend that this dissertation prepared under my direction by Richard Wilson Mead entitled THE EFFECT OF VARIATION OF DESIGN PARAMETERS ON THE MASS TRANSFER EFFICIENCY OF PACKED DISTILLATION COLUMNS be accepted as fulfilling the dissertation requirement of the degree of Doctor of Philosophy

Thomas R. Lehm
Dissertation Director

16 Apr 71
Date

After inspection of the final copy of the dissertation, the following members of the Final Examination Committee concur in its approval and recommend its acceptance:*

<u>Thomas R. Lehm</u>	<u>16 APR 71</u>
<u>J. Richard M. Edwards</u>	<u>4/19/71</u>
<u>M. F. Burke</u>	<u>4/19/71</u>
<u>H. F. Freair</u>	<u>4/19/71</u>
<u>Neil D. Cox</u>	<u>4/19/71</u>

*This approval and acceptance is contingent on the candidate's adequate performance and defense of this dissertation at the final oral examination. The inclusion of this sheet bound into the library copy of the dissertation is evidence of satisfactory performance at the final examination.

STATEMENT BY AUTHOR

This dissertation has been submitted in partial fulfillment of requirements for an advanced degree at The University of Arizona and is deposited in the University Library to be made available to borrowers under rules of the Library.

Brief quotations from this dissertation are allowable without special permission, provided that accurate acknowledgment of source is made. Requests for permission for extended quotation from or reproduction of this manuscript in whole or in part may be granted by the head of the major department or the Dean of the Graduate College when in his judgment the proposed use of the material is in the interests of scholarship. In all other instances, however, permission must be obtained from the author.

SIGNED: Richard W. Mead

ACKNOWLEDGMENTS

I wish to thank The University of Arizona for their support of my research. Everything necessary was provided including computer time, chemicals, packings, and the research and analytical equipment. The departmental faculty was always willing to discuss anything that was causing trouble. The National Science Foundation supported last summer's work with a Summer Traineeship; this financial aid was of great assistance.

My adviser, Thomas R. Rehm, has provided aid and assistance throughout my academic career. In the eleven years I have known him, he has never been unavailable for consultation on any subject. His suggestions have led to solutions of many experimental problems and have given me insight necessary for practical evaluation of engineering problems.

TABLE OF CONTENTS

	Page
LIST OF ILLUSTRATIONS	vi
LIST OF TABLES	vii
ABSTRACT	viii
1. THE DESIGN OF PACKED DISTILLATION COLUMNS	1
Mass Transfer Evaluation	4
Capacity Design	10
2. SCALE-UP CORRELATIONS	15
Previous Scale-Up Correlations	17
Theoretical Plate Correlations	18
Transfer Unit Correlations	19
Proposed Scale-Up Correlation	22
Parameters Under Study	23
Multiple Linear Regression	24
3. EXPERIMENTAL TECHNIQUES	29
Binary Chemical Mixtures	29
The Benzene-Toluene Pair	29
The Isooctane-Toluene Pair	30
The Heptane-Methylcyclohexane Pair	30
Apparatus	34
The Column System	34
The Feed System	38
The Overhead System	38
The Reboiler System	39
Auxillary Systems	40
Procedure	42
Start-Up Procedure	42
Procedure for Continuous Operation	42
Shut-Down Procedure	44
Experimental Data Obtained	44
Experimental Problems	45
Feed Temperature	45
Overhead Condensation	45
Redistributors	46
Uncalculatable Data	48
Continuing Problems	48
Summary of Experimental Program	49

TABLE OF CONTENTS--Continued

	Page
4. EXPERIMENTAL RESULTS	50
Experimental Data	50
Preliminary Calculations	51
Temperature Conversions	51
Pressure Conversion	51
Weight Fractions	51
Overall Material Balance	52
Component Material Balance	52
Flooding Calculations	52
Physical Properties	52
Percent Flooding	53
Height Equivalent to a Theoretical Plate	54
Height of an Overall Gas Phase Transfer Unit	54
Summary of Experimental Results	56
Discussion of Experimental Results	56
5. CORRELATION OF EXPERIMENTAL DATA	66
Fitting Procedure	66
Summary of Correlation Equations	68
Discussion of Correlation Equations	72
6. DISCUSSION AND RECOMMENDATIONS	78
Discussion	79
Recommendations for Further Work	84
Additional Packings	85
Liquid Rate Study	85
Effect of Bed Depth	86
Enriching Section Verification	87
Generalized Pressure Drop Correlation	88
7. CONCLUSIONS	91
APPENDIX A: NOMENCLATURE	96
APPENDIX B: EXPERIMENTAL DATA	100
APPENDIX C: ENTHALPY-COMPOSITION DIAGRAMS	116
APPENDIX D: GENERALIZED PRESSURE DROP CORRELATION	125
LIST OF REFERENCES CITED	127

LIST OF ILLUSTRATIONS

Figure	Page
1. Packed Stripping Section with Flash Reboiler	6
2. Temperature-Composition Diagram for Vapor-Liquid Equilibrium of Benzene and Toluene at 1 atm.	31
3. Temperature-Composition Diagram for Vapor-Liquid Equilibrium of Isooctane and Toluene at 1 atm.	32
4. Temperature-Composition Diagram for Vapor-Liquid Equilibrium of Heptane and Methylcyclohexane at 1 atm.	33
5. Flow Diagram of the Upper Portion of the Experimental Apparatus	35
6. Flow Diagram of the Lower Portion of the Experimental Apparatus	36
7. Sample Gas-Liquid Chromatograph Record	41
8. Ponchon-Savarit Solution for Run 170	55
9. The Effect of Flow Conditions on Mass Transfer Efficiency . .	60
10. Trends Displayed by the Smoothed Four Inch Column Data . . .	62
11. Comparison of Empirical Fits and Smoothed Experimental Data at 65 Percent of Vapor Flooding in the Four Inch Column	76

LIST OF TABLES

Table	Page
1. Summary of Results Calculated from Experimental Data	57
2. Summary of Correlation Equations	69

ABSTRACT

The design of packed distillation columns may be separated into two areas: mass transfer design and capacity design. The capacity design deals with redistribution, throughput, and flooding. The mass transfer design guarantees that sufficient interfacial area to accomplish a desired amount of mass transfer will be provided. The mass transfer efficiency is a measure of the area provided and is characterized by the height of packing required to achieve the desired transfer. The more efficient the operation of the packed section is, the less packed height will be required. Either the transfer unit or the theoretical plate concept of mass transfer may be used as the basis for calculation of the height which expresses the mass transfer efficiency.

The current work investigates the relationship between the mass transfer efficiency and three general parameters indicating the operating conditions of the packed section: column diameter, type and size of the packing material, and the degree of flooding. Of these three parameters, only the degree of flooding is influenced by the physical properties of the fluids in the section. Previous correlations for mass transfer efficiency have been based on molecular properties of the components of the vapor and liquid phases. These properties are not readily available for the multicomponent mixtures generally encountered. Therefore, a correlation based on general

parameters will be more versatile as an expression for the design of packed distillation columns.

The mass transfer efficiency, using both mass transfer concepts, was determined for 107 experimental runs on packed stripping sections. The binary mixture used as feed for the runs consisted of either benzene-toluene or isooctane-toluene. The five foot high packed sections had diameters of 2, 4, and 6 inches. They were packed with eight ceramic packings of three types: Intalox saddles, Berl saddles, and Raschig rings, which covered a packing factor range of 98 to 750. The flow conditions were limited to 44 to 88% of vapor flooding. All runs were made at the same feed mass flow rate, 0.5 lb/sec-ft^2 .

Two families of equation forms were used in selecting the best equation to fit the experimental data. One family consisted of polynomial equations and the other was based on dimensionless ratios of the correlating parameters. Multiple linear regression was used to evaluate the coefficients of each specific equation tested as a correlating equation. The column diameter was found to have minimal effect on the mass transfer efficiency. The best fit of the data was obtained using a second order polynomial with packing factor and degree of flooding as the independent variables. The mass transfer efficiency increased as the degree of flooding increased. Repacking the section with a packing having an increased packing factor was found to decrease the mass transfer efficiency.

The empirical equations of this study, one each for HETP and HOG, may be used as scale-up design equations if a restriction is

placed on their use. Pilot plant experimental data obtained at a liquid rate identical to the rate proposed in a commercial stripping section must be used to evaluate the constant of the polynomial equation. This constant is influenced by the liquid feed rate to the packed section, a parameter not included in the current work. The interaction of liquid rate, packing factor, and degree of flooding are such that further work is required before an unrestricted scale-up design equation may be promulgated.

CHAPTER 1

THE DESIGN OF PACKED DISTILLATION COLUMNS

The use of distillation as a separation and purification process is an ancient one, first used to produce essential oils, elixers, and spirits. The initial large scale operation was, of course, the production of alcoholic beverages. The use of distillation was similarly an art from its beginnings, three or four thousand years ago, up to the start of the nineteenth century. At this point in time the chemical industry as we know it today began to develop. With this growth, existing distillation techniques were studied and new techniques and equipment were developed. This advancement has continued until the present day.

Distillation is a method of separating the components of a solution dependent upon the distribution of the substance between a liquid and a vapor phase. In absorption operations a new material is introduced into the system to provide a second phase; whereas in distillation the new phase is obtained from the original material by the application of thermal energy.

The chemical industries have developed many different types of vapor-liquid contactors to perform multistage distillation, but in general these fall into three main categories: tray devices, high efficiency contactors, and packed sections. Tray devices are of the

equilibrium-stage type while the other devices are differential contactors. In a contactor of the equilibrium-stage type, the principle mass transfer takes place over each increment of height and composition changes are continuous throughout the contacting section.

Tray contactors have been used for most industrial distillation columns with a diameter greater than two feet for a variety of reasons. The design of plate towers is more reliable because the hydrodynamics are understood and the fluid distribution is not as critical a parameter as it is in the other types of contactors. The operating range for plate towers is broad. The tray contactor is easier to clean if liquids containing dispersed solids or sludge are being processed. Tray contactors in general will retain a large volume of the liquid phase and are thus less sensitive to sudden upsets.

Packed towers are seldom designed with diameters greater than four feet although some proprietary work has been done up to eight feet. They are particularly useful in cases where the pressure drop across the contactor must be kept low. Operating situations which require a small pressure drop are, for example, the processing of heat sensitive materials and vacuum distillations. A small liquid hold-up, also characteristic of a packed section, may be essential to product purity when heat sensitive materials are being distilled. Packed towers prove to be cheaper and easier to construct especially if corrosive chemicals are being handled. A consideration which has hindered widespread useage of packed distillation columns is that the

design techniques are not nearly as reliable as those existing for plate columns.

Specialized high efficiency contactors are capable of providing the equivalent of many equilibrium stages in a few feet of column height. However, design of columns employing these devices is quite unreliable and actual experimentation must be used to determine the characteristics of the particular contacting device.

The design techniques for packed distillation columns are not nearly as reliable as those for plate towers as previously mentioned. An indication of the general feeling may be found in the literature. Jordan (16) states on page 444 that "no calculations, however elaborate, on a packed column should be taken seriously." Van Winkle (38) on page 625 makes the comment that "no design method is completely adequate to give accurate predictions for all packing systems and operating conditions." Treybal (37) simply states in Chapter 9 that more research is required in this area.

The design of distillation columns can be separated into two main sections: capacity design and mass transfer design. The mass transfer design assures that the column will have the ability to perform the desired separation, while the capacity design provides for the amount of product to be produced. These design techniques may be applied to an existing piece of equipment to characterize the operating conditions on the basis of the design parameters. A study of the design parameters in terms of the operating variables will provide

information necessary to improve the reliability of existing design methods.

Mass Transfer Evaluation

The amount of material that may be transferred across the phase boundary in a countercurrent mass transfer contactor is a function of both the interfacial area and the resistance to mass transfer of the two laminar films and the interface itself. In a packed column, the interfacial area is a function of the volume of the packing and an increase in the height of a constant diameter section will produce a corresponding increase in the mass transfer area. Common practice has dictated that the separating ability of a packed distillation column will be judged on the basis of feet of packed height (of a unit cross sectional area) necessary to accomplish a certain amount of mass transfer. If the packed section is represented as a differential contactor, as in truth it is, the unit of separating ability is called the height of a transfer unit. The packed section is also often represented as a hypothetical plate column for ease of calculation and the unit of separating ability is referred to as the height equivalent to a theoretical plate. These heights represent the mass transfer efficiency of the contactor and an increase in efficiency decreases the height required to achieve a given degree of separation. In both cases, unless samples are taken throughout a packed section, these terms are evaluated by determining the number of transfer units or theoretical plates necessary to accomplish the given separation and

dividing the packed height by this number to give a value averaged over the entire packed section.

Figure 1 represents a packed section operating as the stripping section of a continuous rectification column. A liquid feed, L_0 , is supplied to the packed section. The liquid passes down the column to the reboiler where a portion is drawn off; the remainder is vaporized and supplied as vapor feed, V_{N+1} , to the bottom of the packed section. The vapor stream rises through the packing and exits as an overhead product.

The transfer unit concept was first presented by Chilton and Colburn (2). The concept may be developed, using the nomenclature presented in Appendix A, by writing the mass transfer rate equation for the differential packed section, dZ . The rate of transfer of the most volatile species is

$$N_a = -d(Vy) = K_y (y^* - y) aSdZ = -d(Lx) = K_x (x - x^*) aSdZ \quad (1)$$

Equation 1 may be rearranged and integrated from the feed point to the bottom of the packed section to give the following:

$$Z = \int_0^Z dZ = \int_{y_1}^{y_{N+1}} \frac{-d(Vy)}{K_y aS (y^* - y)} = \int_{x_F}^{x_B} \frac{-d(Lx)}{K_x aS (x - x^*)} \quad (2)$$

The mass transfer coefficient, K , is, of course, a point value which is determined for a given concentration driving force. Chilton and Colburn assumed that the total molar liquid and vapor flows passing through the packed section were essentially constant and that an

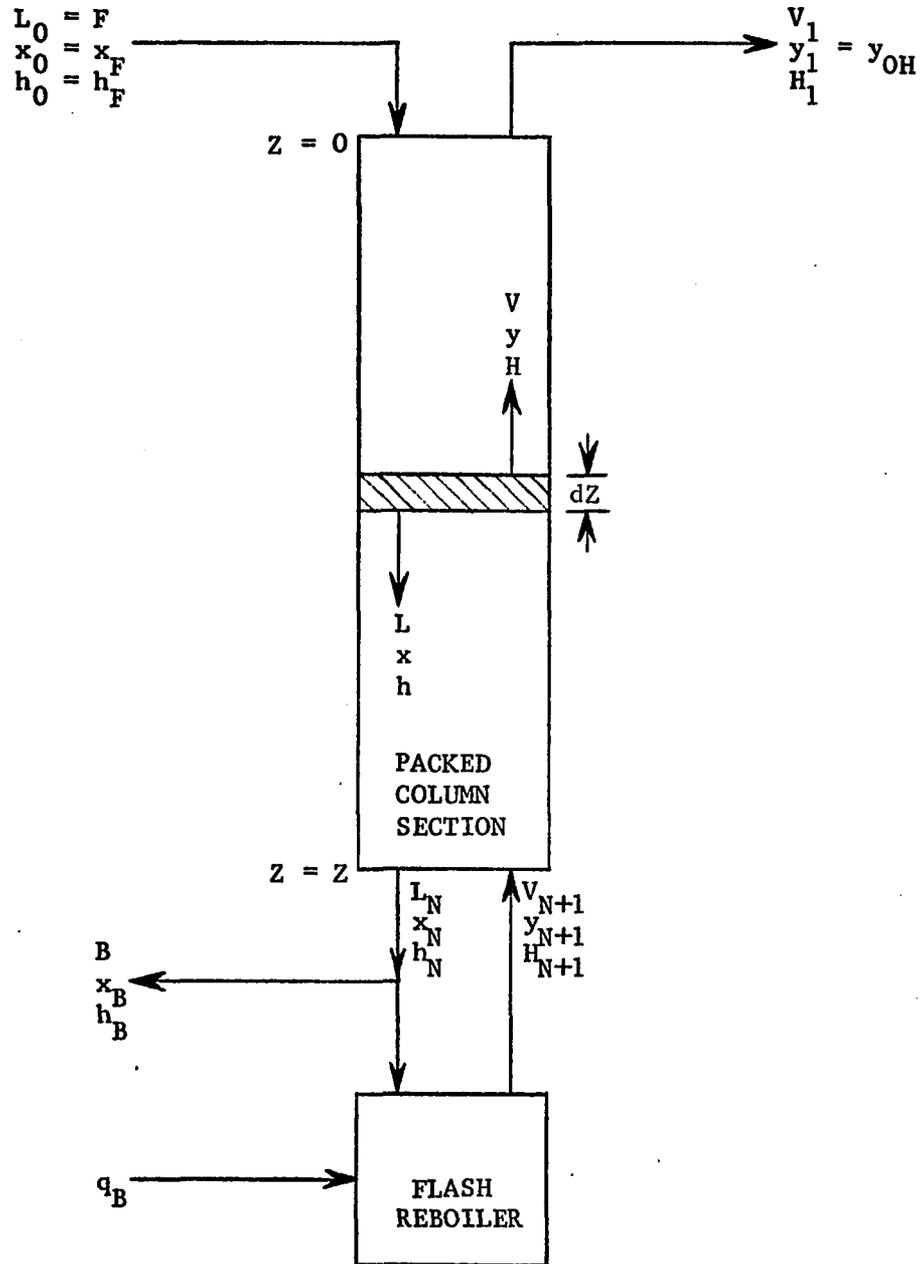


Figure 1. Packed Stripping Section with Flash Reboiler

average overall mass transfer coefficient, \bar{K} , could be determined for the column. They then proceeded to define the overall height and the number of both gas and liquid phase transfer units from the integrated form of Equation 2.

$$Z = HOG \cdot NOG = \frac{V}{\bar{K}_y a S} \cdot \int_{y_{N+1}}^{y_1} \frac{dy}{(y^* - y)} \quad (3a)$$

$$Z = HOL \cdot NOL = \frac{L}{\bar{K}_x a S} \cdot \int_{x_B}^{x_F} \frac{dx}{(x - x^*)} \quad (3b)$$

In Equation 3, V/S and L/S are the molar rates of flow (lb mole/hr-ft²) of the vapor and liquid streams. The transfer unit concept is applied to an existing piece of equipment by dividing the actual packed height by the number of transfer units to obtain the height of a transfer unit.

The height equivalent to a theoretical plate concept was suggested by Peters (28). He proposed that the differential contactor be represented by a number of equilibrium stages. In an equilibrium stage or theoretical plate liquid and vapor streams of different compositions are brought together and intimately mixed. The phases are allowed to equilibrate and are then separated. Again the packed height is divided by the number of theoretical plates to obtain the height equivalent to a theoretical plate.

In order to evaluate both the height of a transfer unit and the height equivalent to a theoretical plate, it is necessary to represent both the equilibrium relationship and the operating

conditions either by equations to obtain an analytical solution or by lines to obtain a graphical solution. The vapor-liquid equilibrium relationship may be easily stated as:

$$y^* = f(x) \quad (4)$$

Although many methods have been developed for the prediction of vapor-liquid equilibrium relationships, it is generally preferable to use experimental data if available. A simultaneous material and energy balance around the column provides the relationship which represents the operating conditions. At some point in the packed section, we may write the overall material balance,

$$L = B + V \quad (5)$$

the component material balance,

$$Lx = Bx_B + Vy \quad (6)$$

and the energy balance

$$Lh + q_B = Bh_B + VH \quad (7)$$

Rearrangement of Equation 7 yields the defining equation for the hypothetical enthalpy difference stream.

$$h_{\Delta} = h_B - q_B/B = (L/B)h - (V/B)H \quad (8)$$

Combination of Equations 5, 6, and 8 produces the following result:

$$\frac{L}{V} = \frac{y - x_B}{x - x_B} = \frac{H - h_{\Delta}}{h - h_{\Delta}} \quad (9)$$

The operating equation for the packed section is obtained by solving Equation 9 for the vapor concentration with the following result

$$y = x_B + \left(\frac{H - h_{\Delta}^-}{h - h_{\Delta}^-} \right) (x - x_B) \quad (10)$$

Equation 10 is complicated by the fact that the enthalpies of both phases are functions of the compositions of the phases. If one assumes that both phases are ideal with the exception of non-ideal mixing in the liquid phase, the following expressions may be written to describe the phase enthalpies:

$$h = [xC_{La}M_a + (1-x)C_{Lb}M_b](T_L - T_R) + \Delta H_{SOL} \quad (11)$$

$$H = y[C_{La}M_a(T_V - T_R) + \lambda_a M_a] + (1-y)[C_{Lb}M_b(T_V - T_R) + \lambda_b M_b] \quad (12)$$

The phase temperatures, T_L and T_V , are functions of composition and are obtained from temperature-composition data included with the equilibrium data represented by Equation 4. The integral heat of solution is also composition dependent. Therefore, when Equations 11 and 12 are substituted into Equation 10, a complex expression is obtained which requires detailed literature data concerning several thermodynamic quantities. It should be pointed out that fairly ideal behavior was assumed for the phases, if highly non-ideal behavior is assumed the operating line becomes extremely complex.

The solution of Equation 3a for the number of overall gas phase transfer units requires determination of the (y^*-y) term as a

function of y . This relationship is obtained by incrementing from x_0 to x_N . The value of x is supplied to Equation 4 to obtain the equilibrium value, y^* . The operating vapor composition is obtained by supplying the value of x to Equations 10 and 11 and then solving Equations 10 and 12 simultaneously for the value of y . The integration of Equation 3a may then be performed either numerically or graphically.

The number of theoretical plates are again determined from the above equations by moving down the column, one theoretical plate at a time. Given the vapor composition leaving the J^{th} plate, y_J , Equation 4 is used to find the exit liquid concentration, x_J . Equations 10, 11, and 12 are then solved to obtain the vapor composition entering the plate, y_{J+1} . This new composition is the starting point for the next iteration. If however, y_{J+1} is less than the bottom vapor composition, y_{N+1} , which is also the bottom liquid composition, x_B , it is necessary to define a partial plate to terminate the calculation.

$$\text{Partial Plate} = (y_J - x_B) / (y_J - x_J) \quad (13)$$

As before this procedure may be carried out either numerically or graphically utilizing the Ponchon-Savarit method presented in Treybal (37).

Capacity Design

The capacity design of packed distillation columns deals with such subjects as flooding, pressure drop, liquid hold-up, and the distribution (initial or intermediate) of the liquid phase. The techniques of capacity design have been summarized by Eckert (5), but

these techniques are primarily for the design of absorption towers. Eckert (6), Eckert and Walter (8), and Eckert, Foote, and Walter (9) have extended the design techniques for use in distillation with a series of related studies that were performed with the same piece of experimental equipment. The capacity design methods are summarized in the following discussion.

In 1938 Sherwood, Shipley, and Holloway (32) presented the initial form of a general empirical flooding correlation in which the type of packing was represented by the specific surface of the packing divided by the cube of the porosity, a_v/ϵ^3 . Lobo et al. (18) examined the pressure drop across the bed to determine the flood point, then empirically determined a packing capacity factor, P , to use in place of the calculated constant of Sherwood et al. Leva (17) determined that for a given degree of flooding, regardless of the flow rates, the pressure drop would be constant. He also found that the addition of a reciprocal specific gravity, ψ , of the liquid tended to give a more reliable correlation. Eckert (5) measured packing factors at pressure drops that were less than those observed at flooding conditions. The average packing factors found with this procedure were applicable over a wide range of operating conditions and thus tend to separate the flooding characteristics from the operating parameters. The correlation obtained from this experimental work is referred to as the generalized pressure drop correlation (GPDC). The correlation graphically relates the X term, $(L/V)(\rho_V/\rho_L)^{1/2}$, to the Y term, $(V/S)^2 P \psi^{0.2} / g_c \rho_V \rho_L$, and is presented in Appendix D. The flooding curve of the GPDC, relating

the vapor and liquid rates at flooding, was fit in the region of interest with a polynomial equation of the form

$$Y = c_0 + c_1/X + c_2/X^2 + c_3/X^3 \quad (14)$$

utilizing a least squares program developed by Simpson (34). Given the liquid rate and the compositions of both phases, it is possible to calculate the flooding vapor rate, V_F , by trial and error. It should be mentioned that the liquid rate is fairly constant in the stripping section and a change in the reboiler ratio will change the vapor rate, therefore, vapor flooding is considered. Correspondingly, the vapor rate is constant in the enriching section and a change in the reflux ratio causes a change in the liquid rate, hence, one considers liquid flooding. The degree of flooding in the stripping section is obtained by dividing the vapor rate, V , by the flooding vapor rate, V_F .

The pressure drop aspects of the GPDC have been studied and modeled by Prahl (30). He has reduced the curves to a series of algebraic expressions with four empirical constants for each packing. The packings treated are large in size but the method is applicable to the smaller packings used in pilot plant work.

The amount of liquid hold-up found on the packing is of little consequence when steady state analysis of the packed section is being made. However, when one studies the dynamics of the packed section, the liquid hold-up has a dampening effect on sudden upsets. Leva (17) presented an empirical equation to predict the volume of liquid hold-up per cubic foot of packing. This equation is:

$$L_H = 0.004 \mu_L^{0.1} \psi^{0.78} (L/d_p)^{0.6} (\sigma/73)^{(1.0-0.262 \log L)} \quad (15)$$

Otake and Okada (25), working in Japan, also derived an empirical equation to predict this quantity.

$$L_H = 1.295 a_v d_p \left(DL/\mu_L \right)^{0.676} \left(\mu_L^2/d_p^3 g \rho_L^2 \right)^{0.44} \quad (16)$$

Maldistribution tends to decrease the efficiency of the packed section. Porter and Templeman (29) found that up to fifty percent of the liquid fed to the column section would migrate to the wall if no redistribution was provided. Eckert (5) suggested practical guidelines to follow to minimize the effects of poor distribution. It is suggested that a redistributor should be placed in the packing at a height equivalent to 2 1/2 to 3 column diameters for a column packed with Raschig rings and every 5 to 8 column diameters for saddle packed columns. Generally, orifice-type plate redistributors placed in the bed are satisfactory.

The previous discussion has shown that one may determine both the mass transfer efficiency, HETP or HOG, and the degree of flooding, V/V_F , for a packed stripping section when the operating flow rates and compositions are known. The discussion has focused on the design of packed stripping sections, but similar procedures yield the corresponding design equations for packed enriching sections. The current work has investigated changes in mass transfer efficiency caused by variation of three operational parameters: column diameter, packing, and degree of flooding. The study of these parameters will yield information for

the development of a scale-up correlation which may be used to predict the mass transfer efficiency on the basis of the operational parameters.

CHAPTER 2

SCALE-UP CORRELATIONS

The distillation columns used in pilot plants are generally small packed columns. They find wide acceptance in this service primarily for economic reasons, but the low pressure drop and the small liquid hold-up also are in their favor. The optimum operating conditions of all process distillation columns are determined with the packed, pilot plant distillation columns as a part of the pilot plant experimental program. Since in many instances plate distillation columns rather than packed distillation columns will be used in the commercial plant, only a portion of the experimental information, that concerning the effect of reflux ratio on yield and concentration, is required for the design of the large plate column.

The design engineer may find that it is feasible to use a large packed distillation column instead of a plate column. In that case the design of the full size column must be based on the complete pilot plant data, and it is essential that accurate scale-up methods be available. It should be noted that Eckert (7) feels that accuracy in scale-up is not critical and the large scale column should be based on the capacity design methods only.

Two categories of information are experimentally obtained in a pilot plant distillation study: stream conditions and physical dimensions. The stream conditions include the temperature, composition,

and flow rate of each process stream. The type and size of packing and the column dimensions comprise the other experimental data. If this data is used in conjunction with known physical properties of the materials involved, one may determine the mass transfer efficiency and the degree of flooding for each pilot plant run.

Four design parameters are required for the design of a large scale packed distillation column; these are: the desired flow conditions, the mass transfer efficiency, the size of the column and packing, and the degree of separation required to produce the desired product. Of these parameters, only the last is unaffected by scale-up. Therefore, information concerning the scale-up behavior of the other parameters is necessary to obtain a reliable plant scale column design.

The capacity design methods, previously presented in Chapter 1, in combination with economic considerations are generally sufficient to scale-up to a column of a size capable of handling the desired flow rates. In general, packed distillation columns are designed to operate between fifty and eighty percent of flooding. Vivian, Brian, and Krukonis (39) have pointed out that the loading region generally begins at fifty percent of flooding and that the pressure drop and liquid hold-up are insignificant before the loading region is reached. Fair (12,13) has recommended that a packing with a high capacity and a size of two inches or less is preferable for packed distillation service. Clay, Clark, and Munro (3) evaluated the capacity design methods with industrial column data. They found that the flooding

range was wider than expected. They attributed this to the fact that the GPDC involves averaged packing factors over the entire range of the empirical relationship.

The scale-up of the height of the packed section to provide enough transfer units or theoretical plates to accomplish the desired separation comprises the mass transfer efficiency design method. The method is not well defined and is generally applied by multiplying the experimental mass transfer efficiency height by a safety factor. One hopes that the increase in height will guarantee that sufficient mass transfer will take place. This inaccurate procedure must be improved to obtain a reliable scale-up method.

Two different approaches are applicable to scale-up design. The first predicts the height on the basis of the physical properties of the fluids and constants based on the column and packing. The second approach is to base the height on a value determined with a pilot plant column which has previously performed the desired separation. Several different investigators have studied the problem using both approaches, but in all cases the studies are limited.

Previous Scale-Up Correlations

The previous studies may be separated into two types, those dealing with theoretical plates and those dealing with transfer units. The correlations involving the theoretical plate concept were developed first. Then, in about 1960, the transfer unit concept became of primary importance.

Theoretical Plate Correlations

In 1951, Hands and Whitt (14) presented the following expression for the prediction of HETP in columns packed with Raschig rings

$$\text{HETP} = 70 \left(d_p \mu_L \bar{M}/L \right)^{1/2} \quad (17)$$

The correlation was based on data obtained from the literature as well as experimental data obtained during the vacuum distillation of ethanalamines. The form of the equation was assumed so that only the coefficient was determined. More subtle effects were not investigated because of the inconsistency of the data from several sources.

Ellis (11) suggested an equation to predict HETP for columns packed with one or two inch Raschig rings. The equation,

$$\text{HETP} = [18d_p + 12m(V/L-1)](Z/10)^{1/2} \quad (18)$$

suggests that the mass transfer efficiency is dependent on column height.

Murch (22) combined the data of several authors to obtain a correlation. The equation was based on average physical properties and assumed a constant relative volatility. The height effect is based on the height between redistributors, z' , rather than the total bed depth. The equation obtained

$$\text{HETP} = c_1 (V/\bar{M})^{c_2} D^{c_3} (z')^{1/3} \left(\alpha \bar{\mu}_L / \bar{\rho}_L \right) \quad (19)$$

is awkward because no predictable behavior of the empirical constants was observed. Therefore, experimental determination of the constants would be required for each process case.

Eckert and Walter (8) found that none of these equations gave reliable predictions. The value obtained experimentally would, however, tend to fall in the region between the various predictions. More recent empirical equations have become more complex with the addition of further constants and are thus impractical for design use.

Transfer Unit Correlations

All correlations used to predict the height of overall gas phase transfer units are based on the prediction of the height of film transfer units, H_G and H_L , and their combination. The basis for the combination of the film transfer unit heights comes from the relationship of the mass transfer coefficients predicted by the Whitman two-film theory. Following a discussion in Treybal (37) one may separate the overall gas phase driving force into two portions, that is

$$(y^*-y) = (y_i - y) + (y^* - y_i) \quad (20)$$

Remembering that y^* is the vapor concentration in equilibrium with the bulk liquid concentration x , and that the interfacial values are in equilibrium, and assuming that the equilibrium relationship can be represented by a straight line in the region of interest, Equation 20 can be rewritten as

$$(y^*-y) = (y_i - y) + (dy^*/dx)(x-x_i) = (y_i - y) + m(x-x_i) \quad (21)$$

Defining the film mass transfer coefficients on the same basis as Equation 1 gives the expression

$$K_y a S (y^*-y) dZ = k_y a S (y_i - y) dZ = k_x a S (x-x_i) dZ \quad (22)$$

A simultaneous solution of Equations 21 and 22 yields an expression for the relationship between the mass transfer coefficients

$$\frac{1}{K_y aS} = \frac{1}{k_y aS} + \frac{m}{k_x aS} \quad (23)$$

Multiplication by the appropriate flow rate converts Equation 23 to the following

$$\frac{V}{K_y aS} = \frac{V}{k_y aA} + m(V/L) \frac{L}{k_x aS} \quad (24)$$

In Equation 3, the left-hand side of Equation 24 is defined as the height of an overall gas phase transfer unit, HOG. The terms containing the film mass transfer coefficients on the right-hand side of Equation 24 are defined as film transfer unit heights in a similar manner yielding the following result

$$HOG = HG + m(V/L) \cdot HL \quad (25)$$

It should be pointed out that the development was made on the basis of point mass transfer coefficients and appropriate averaging across the packed section is necessary to go from Equation 24 to Equation 25.

Morris (20) extended a design method for absorption columns that he developed with Jackson (21) to packed column distillation. The empirical equations developed were based on different standard one-inch reference columns: a falling-film column for the vapor film and a disc column for the liquid film. The vapor film equation is

$$HG = M \frac{V}{a} \frac{1}{v} \frac{1}{k'_p} \quad (26)$$

where

$$k'_p = 48.6 P'_G \rho_{RS} \left(\frac{\rho_{RG} v_r^3}{\pi S c_{RG}} \right)^{1/4} (T_R/T_f)^{0.56} (1-y)_{1m}^{-1} \quad (27)$$

and the liquid film equation is

$$HL = \bar{M}_L / a_v \rho_L k'_C \quad (28)$$

where

$$k'_C = 19.75 (Sc_{RL})^{-1/2} \left(\frac{\rho_{RL}}{\mu_{RL}} \right)^{0.03} \left(\frac{\mu_{RL} T_f}{\mu_L T_R} \right)^{1/2} \left(\frac{L_w}{L_w^*} \right)^{c_1} (L_w^*)^{0.7} \quad (29)$$

Morris assumed that there were no differences between absorption mass transfer (diffusion through a stagnant component) and distillation mass transfer (equimolar counterdiffusion).

Cornell et al. (4) developed empirical equations for the prediction of the film transfer unit heights of the following form:

$$HG = c_1 (Sc_V)^{1/2} D^{c_2} \left(\frac{z'}{10} \right)^{1/3} \left[(\bar{M}/L) (\rho_L)^{1.25} \left(\frac{1.005}{\mu_L} \right)^{0.16} \left(\frac{\sigma}{73} \right)^{0.8} \right]^{c_3} \quad (30)$$

$$HL = c_4 c_5 (Sc_L)^{1/2} \left(\frac{z'}{10} \right)^{0.15} \quad (31)$$

Equations 30 and 31 do take into account variations in column size. The column diameter affects only the gas film transfer unit and the index on the term averages approximately 1.2 for the Raschig ring and Berl saddle packings studied. The film transfer units heights are obtained by utilizing the values of the empirical constants read from graphs and then solving the above equations.

Yoshida and Koyanagi (41) followed a similar approach but did not investigate the effect of column diameter or packed height because their data was contradictory. The equations presented were:

$$HG = (c_1/a_e) \left(VA_p^{1/2} / \bar{M}\mu_V \right)^{0.41} (Sc_V)^{2/3} \quad (32)$$

$$HL = 1.9 d_p (Sc_L)^{1/2} \left(L/a_e \mu_L \right)^{1/2} \left(\mu_L^2 / d_p^2 g \rho_L \right)^{1/6} \quad (33)$$

The fact that effective mass transfer areas, a_e , are available only for selected packings makes widespread application of these equations very difficult.

The reliability of the three transfer unit design methods presented above has been studied by Eckert and Walter (8) and by Clay et al. (3). The method of Cornell et al. (4) yields predicted values of HOG similar to those obtained experimentally, however, large deviations do occur in several instances. The other methods were found to be highly unreliable.

Proposed Scale-Up Correlation

The desired result of the current study is the development of an empirical expression which would allow the design engineer to reliably design an industrial scale packed distillation column from data obtained with a pilot plant column. To develop this correlation a series of experiments will be performed investigating the effect of several different parameters on the mass transfer efficiency of a packed section. The experimental results will be fit with an empirical equation which will predict the behavior of packed sections in the

region of study. The empirical equation obtained may be used to predict the mass transfer efficiency in a packed stripping section of a commercial size. These predictions may be compared with results taken during the operation of large columns, subsequently reported in the literature, to establish the validity of the equation as an aid to scale-up design.

Parameters Under Study

Column operation will be represented by macro-scale parameters leading to an equation of the desired generality. Three major design parameters which influence the mass transfer efficiency are included in this study. The type and size of the packing are represented by the packing factor, P , an empirically determined value specific for each packing. The column diameter, D , takes into account heat losses and other wall effects. The two-phase, countercurrent flow conditions are represented by the degree of flooding in percent, F . The dependent variable of the correlation equation will be the mass transfer efficiency of the contactor, represented by either the height equivalent to a theoretical plate, HETP, or the height of an overall gas phase transfer unit, HOG.

The study of other parameters are beyond the scope of the experimental program and must be left to studies in further work. Two significant parameters were not studied: the depth of the packed bed and the overall liquid loading. In addition, only the stripping section was investigated, therefore, all results should be verified by future work in the enriching section.

Multiple Linear Regression

Graphical techniques for the determination of coefficients of empirical equations are extremely tedious when normal experimental scatter is encountered. A mathematical procedure called multiple linear regression allows for the evaluation of the coefficients of a proposed equation form using a multi-dimensional least squares technique. The use of the term 'linear' refers to the fact that the proposed equation must either be of a linear form or be transformable to a linear form.

Following the discussion of Chapter 17 of Neville and Kennedy (23), one may consider the case of a linear relation between the dependent variable, Y , and the independent variables, X_1 and X_2 .

$$Y = c_0 + c_1X_1 + c_2X_2 \quad (34)$$

Two independent variables are used for simplicity but the following relationships can be extended to any number of variables. The relationship for the sum of the squares of the residuals of n observations is given as

$$\Sigma \delta^2 = \Sigma [Y - c_0 - c_1X_1 - c_2X_2]^2 \quad (35)$$

Using the principle of least squares, one minimizes $\Sigma \delta^2$ with respect to the coefficients. This is accomplished by equating the partial derivatives with respect to the coefficients to zero. The resulting equations are

$$\begin{aligned}\frac{\partial(\Sigma\delta^2)}{\partial c_0} &= -2\Sigma[Y - c_0 - c_1X_1 - c_2X_2] = 0 \\ \frac{\partial(\Sigma\delta^2)}{\partial c_1} &= -2\Sigma[Y - c_0 - c_1X_1 - c_2X_2]X_1 = 0 \\ \frac{\partial(\Sigma\delta^2)}{\partial c_2} &= -2\Sigma[Y - c_0 - c_1X_1 - c_2X_2]X_2 = 0\end{aligned}\tag{36}$$

The three equations obtained may then be simplified, giving

$$\begin{aligned}\Sigma Y &= nc_0 + c_1\Sigma X_1 + c_2\Sigma X_2 \\ \Sigma X_1 Y &= c_0\Sigma X_1 + c_1\Sigma X_1^2 + c_2\Sigma X_1 X_2 \\ \Sigma X_2 Y &= c_0\Sigma X_2 + c_1\Sigma X_1 X_2 + c_2\Sigma X_2^2\end{aligned}\tag{37}$$

The solution of the set of simultaneous equations gives the values of the empirical coefficients.

Comparison of empirical equations of several different forms becomes necessary to determine which equation best predicts the experimental data. The best criterion is that the sum of the squares of the residuals will decrease as the form of the equation tested fits the data better. Three different statistical tests, all based on the sum of the squares of the residuals, may be used to indicate improvement in fit. The standard deviation, s , decreases as the fit improves and is defined as

$$s^2 = \frac{\Sigma\delta^2}{n_0 - n_k - 1}\tag{38}$$

where n_o is the number of observations and n_k is the number of independent variables. The correlation coefficient, r , indicates the fraction of the total variance contributed by the independent variables. The correlation coefficient is defined as

$$r^2 = 1 - (\Sigma\delta^2/\Sigma Y^2) \quad (39)$$

where ΣY^2 is the sum of squares of the observed values of the dependent quantity. As the variance is accounted for, the value of r approaches unity. The F-test compares the variance contributed by the regression and the standard deviation squared (error variance) to determine if the regression is significant. The value of the test

$$f = \left(\frac{n_o - n_k - 1}{n_k} \right) \left(\frac{\Sigma Y^2}{\Sigma \delta^2} - 1 \right) \quad (40)$$

is compared with a tabular value based on the number of observations and variables at a 99% level of significance. If f is greater than the value given in the table then the assumption that all coefficients are equal to zero is false.

Two different groupings of equation types will be used in the attempt to fit the experimental data. The first grouping is based on dimensionless ratios of the three major design parameters. Equation 41 presents a simple ratio equation as an example

$$\frac{HOG}{HOG_R} = c_0 \left(\frac{D}{D_R} \right)^{c_1} \left(\frac{P}{P_R} \right)^{c_2} \left(\frac{F}{F_R} \right)^{c_3} = c_0 (RD)^{c_1} (RP)^{c_2} (RF)^{c_3} \quad (41)$$

The equation may be transformed to a linear form by taking the logarithm of the equation.

$$\log(\text{HOG}/\text{HOG}_R) = \log(c_0) + c_1 \log(\text{RD}) + c_2 \log(\text{RP}) + c_3 \log(\text{RF}) \quad (42)$$

Multiple linear regression is then used to evaluate the coefficients. This type of equation allows the design engineer to select an experimental run as a reference and to determine the desired mass transfer efficiency, HOG, on the basis of the reference run efficiency, HOG_R , when the operational behavior of the reference and desired columns are represented by the macro-scale design parameters. Equations of a polynomial combination of the independent variables, the simplest of which is

$$\text{HOG} = c_0 + c_1 D + c_2 P + c_3 F \quad (43)$$

comprise the second group of equation types. Equations of this group require several experimental runs to verify the coefficients prior to their use as an aid to scale-up design.

The number of specific equations of the above groups is limited only by the imagination. The coefficients of any proposed equation may be determined by multiple linear regression as long as the conditions of the procedure are met. Since the author feels that a useful design equation must be relatively simple in form, highly complex forms will be omitted from this study. A further note of caution must be added, when a specific equation is said to be the best fit, the reference is only to the equation forms considered in the study and further studies might yield an equation of a better form.

In summary, the goal of this research is the development of a scale-up design equation that will allow the prediction of the mass

transfer efficiency of a packed distillation column section on the basis of general operating parameters. These macro-scale parameters are the column diameter, the packing factor, and the degree of flooding. Previous correlations have either been limited in scope or have contained variables such as effective mass transfer area, relative velocity, and diffusivity which make the equations difficult to use. This study will develop an empirical equation based on new experimental data which covers the normal variation of the design parameters.

CHAPTER 3

EXPERIMENTAL TECHNIQUES

A detailed experimental plan was necessary to study the interaction of the various experimental parameters comprehensively. In this chapter a description of the apparatus and procedure is included along with a discussion of experimental problems. The range of variation of the experimental parameters covered by this study is also mentioned in the appropriate sections.

Binary Chemical Mixtures

Three different binary pairs were used as feedstock. The properties of the pairs are discussed below. The contents of a fifty-five gallon drum of each material, obtained from Phillips Petroleum Company, were combined in a tank and mixed so that two drums of uniform composition were available for use. To assure that the composition of the feed remained constant, the overhead and bottoms products from the experimental apparatus were collected in the same drum, producing more feedstock for further operation.

The Benzene-Toluene Pair

The benzene-toluene mixture has a difference in normal boiling points of 55 F°. The material used experimentally was pure grade (a minimum of 99 m/o) and is referred to as Mixture I in tables in the

other sections of this work. In Figure 2 the experimental temperature-composition data of Todd (36) is compared with that predicted assuming ideal solution behavior (Raoult's law). The figure shows that the mixture exhibits nearly ideal behavior.

The Isooctane-Toluene Pair

The second pair was pure grade toluene and pure grade isooctane (2,2,4-trimethylpentane). Mixture II has a 20 F° difference in normal boiling points. Figure 3 demonstrates that deviations from ideal solution behavior do exist. The data by Thornton and Garner (35) shows that the envelope is quite symmetrical in the region of interest, i.e., high toluene concentrations.

The Heptane-Methylcyclohexane Pair

Mixture III, heptane and methylcyclohexane (MCH), has a 5 F° difference in normal boiling points. Pure grade materials were too expensive, so technical grade heptane and commercial grade MCH were used. The synthesis route for the production of MCH involves both heptane and toluene, therefore one of the contaminants was not harmful. The mixed feedstock did, however, contain 7 m/o toluene which boils 17 F° above the boiling point of MCH. Figure 4 compares the bubble point data of Bromiley and Quiggle (1) and the temperature-composition data of Hughes and Maloney (15) with temperature-composition data predicted from Raoult's law. The experimental data displays no consistent trends, therefore no conclusions may be drawn as to the ideality of the mixture.

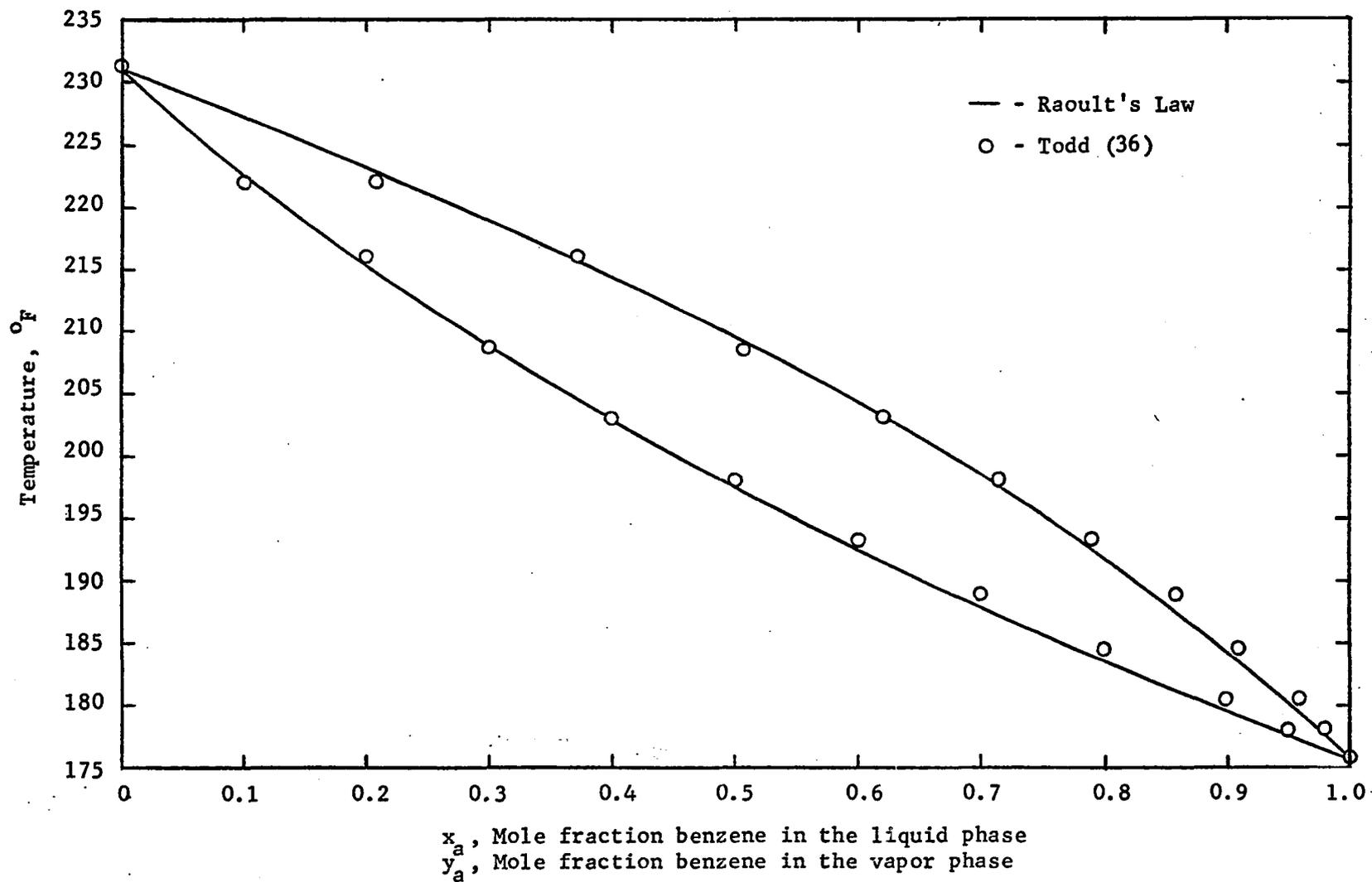


Figure 2. Temperature-Composition Diagram for Vapor-Liquid Equilibrium of Benzene and Toluene at 1 atm.

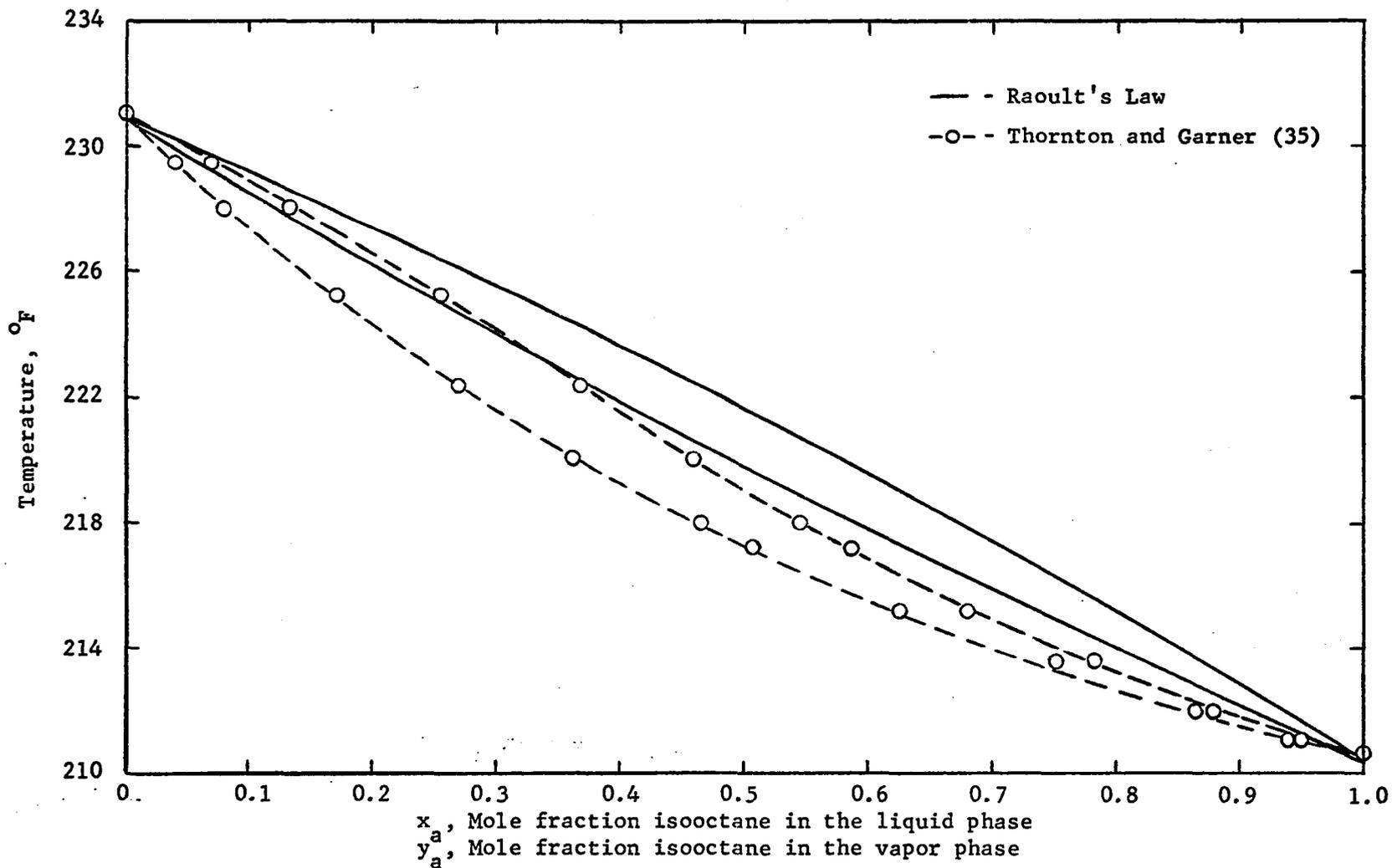


Figure 3. Temperature-Composition Diagram for Vapor-Liquid Equilibrium of Isooctane and Toluene at 1 atm.

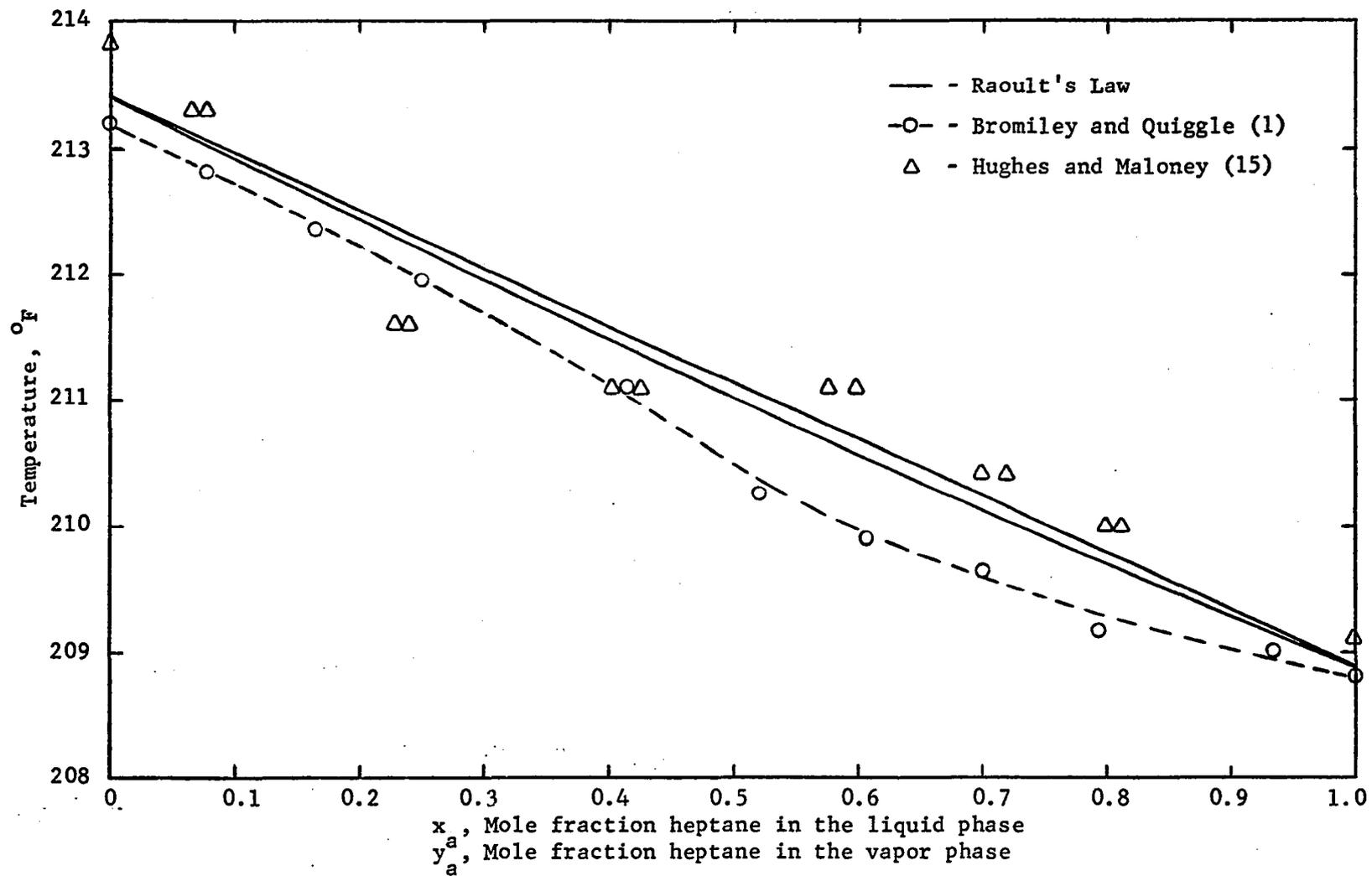


Figure 4. Temperature-Composition Diagram for Vapor-Liquid Equilibrium of Heptane and Methylcyclohexane at 1 atm.

The data from the literature for mixtures one and two was converted to temperature-composition information at 700 mm Hg using the method of Othmer and Gilmont (26). Temperature-composition relationships for the third mixture at ambient pressure were obtained using Raoult's law. All further calculations were performed using the results of these conversions to the average ambient pressure in Tucson of 700 mm Hg.

Apparatus

The experimental apparatus consisted of four major systems: the column system, the feed system, the overhead system, and the reboiler system. In addition, two auxiliary measuring devices were used. The equipment was located on the first and second levels of the University of Arizona Chemical Engineering Laboratory. The flow schematic for the experimental apparatus is presented in Figures 5 and 6. Figure 5 represents the feed system, the overhead system, and the upper section of the column system. Figure 6 portrays the lower section of the column system and the reboiler system. The operating systems were connected in such a way that the columns could be operated either at total reflux or as the stripping section of a continuous rectification column.

The Column System

The column system consisted of three packed column sections with feed and exit connections manifolded to phase separation chambers at the top and the bottom of the columns. Provisions were made to

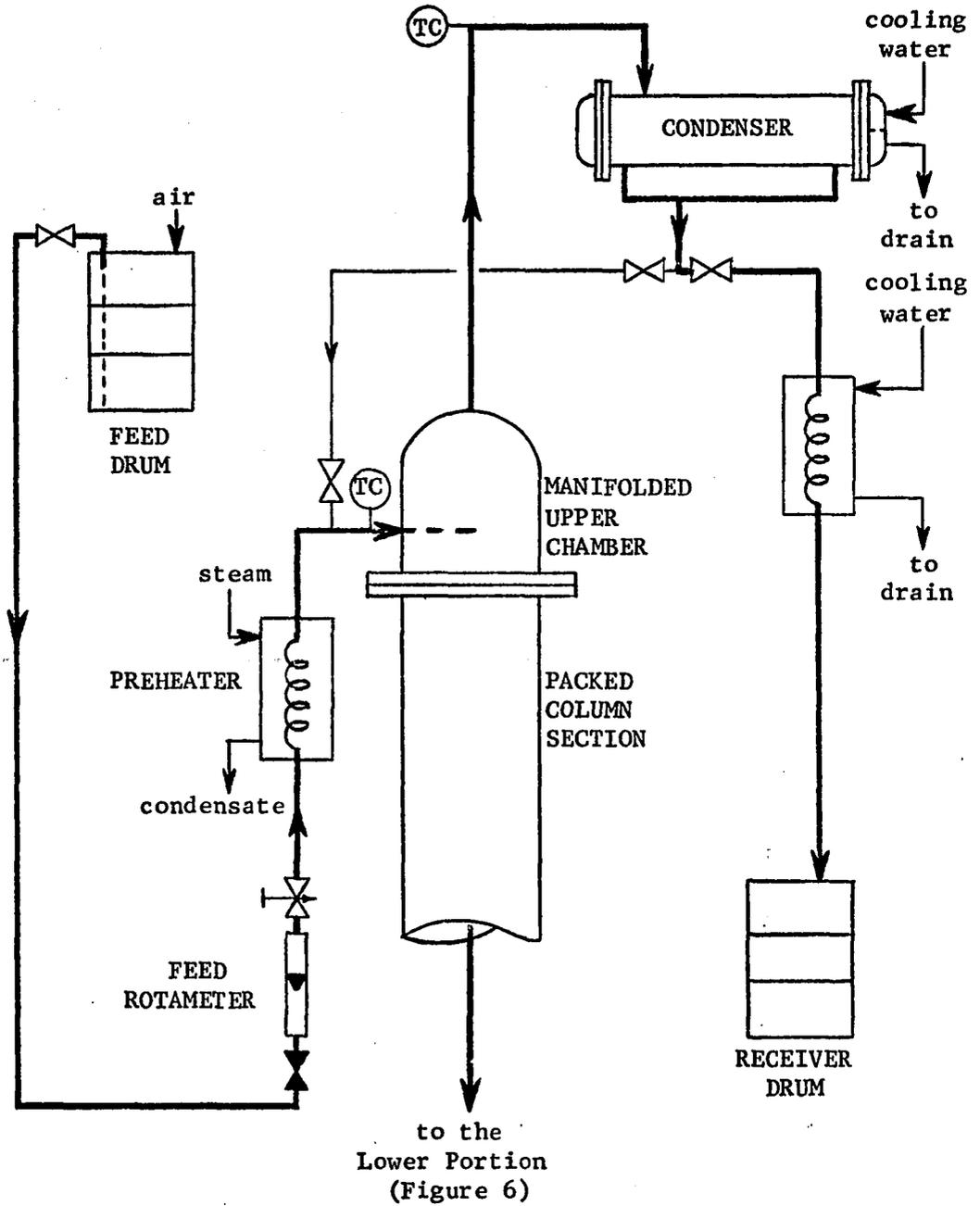


Figure 5. Flow Diagram of the Upper Portion of the Experimental Apparatus

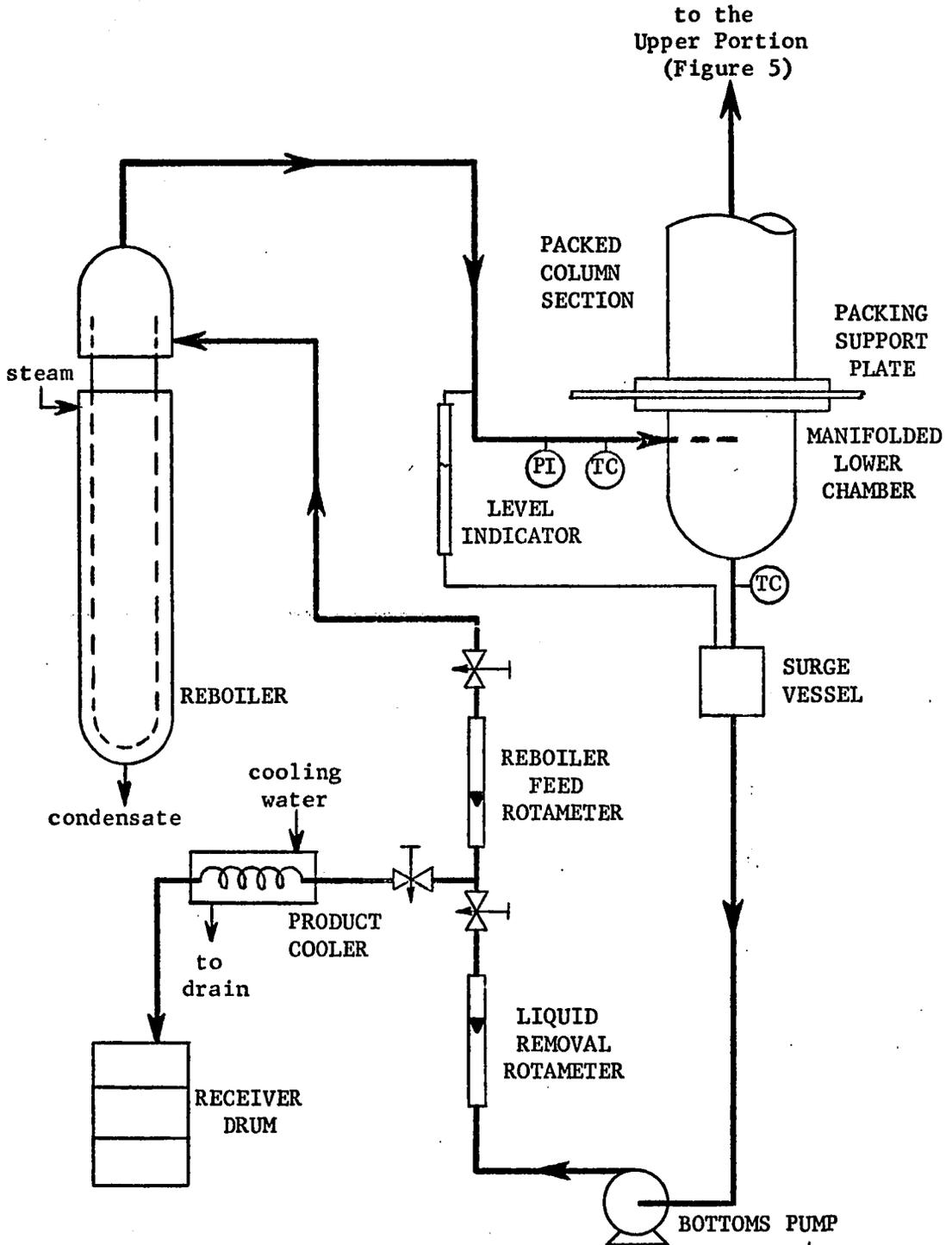


Figure 6. Flow Diagram of the Lower Portion of the Experimental Apparatus

measure the vapor temperature at the top and both the liquid and vapor temperatures at the bottom of the sections. The column sections were flanged pipe (Schedule 40) five feet in length and had diameters of two, four, and six inch nominal. These sections were mounted on three-eighths inch aluminum sheet in which holes were drilled for both the flange bolts and to provide a perforated plate for packing support. The perforated plate holes were drilled on triangular pitch so that the free flow areas for all column sizes were between 38 and 39 percent. One-quarter inch hardware cloth was placed above the perforations to prevent individual pieces of packing from plugging the holes.

Rubber gaskets were provided to seal all flanged connections. Fiberglass insulation, molded to a thickness of one and a half inches, was placed around the column sections to prevent heat losses. In addition the flanged joints were wrapped with foil-backed fiberglass insulation. Provisions were made to measure the pressure drop across the packed section with a water manometer.

The columns were wet-packed to prevent fracturing the ceramic packing material and to aid in achieving a random orientation of the packing pieces. Eight individual packings of three basic types were used: Intalox saddles (1/4, 1/2, 3/4, 1 inch), Berl saddles (1/2, 3/4 inch), and Raschig rings (1/2, 3/4 inch). It was not feasible to perform experiments with certain combinations of packing size and column diameter for reasons which were either economic or were due to non-uniformity of the packed bed caused by large packing in small columns. The combinations not used were: one inch packing in the

2 and 4 inch columns, three-quarter inch packing in the 2 inch column, and one-quarter inch packing in the 6 inch column. All packing used was inspected and broken pieces removed prior to placing the packing in the column.

The Feed System

The feedstock for a run was contained in two drums located on the first level some eight feet above the rest of the experimental equipment. Air at 5 psig pressure was applied to the feed drums to initiate and maintain flow to the remainder of the apparatus. Globe valves were adjusted so that a predetermined flow rate based on a mass flow rate of 0.5 lb/sec-ft^2 was indicated on the feed rotameter. A sample port before the feed rotameter provided for the acquisition of a feed sample.

The feed stream then entered a preheater where the material was raised to its bubble point. The preheater was fabricated from a twenty-five foot coil of three-eighths inch copper tubing placed in a jacket made from four inch pipe. Steam was supplied to the jacket to act as the heating medium. An electric tape heater was placed around the preheater exit pipe to allow for fine temperature adjustment. A thermocouple located just prior to entry into the column feed manifold was used to measure the feed temperature.

The Overhead System

The vapor leaving the upper chamber was carried to an American Standard BCF heat exchanger with 34 square feet of heat transfer area.

The lines from the columns were wrapped with electric heating tapes, a layer of molded fiberglass, and a layer of the foil-backed fiberglass building insulation to prevent condensation prior to the exchanger. The overhead product was condensed in the exchanger and drained into a drum through an additional heat exchanger which further cooled the product. Provision was also made to return the condensate to the feed manifold, which allowed for operation at total reflux. Total reflux operation was necessary for column start-up.

The Reboiler System

The basic piece of equipment in the reboiler system was the falling-film reboiler. The unit was designed so that a liquid feed greater than the maximum expected rate could be totally flashed. The unit had a heat transfer area of 3.8 square feet and consisted of a steam-jacketed length of four-inch Schedule 40 pipe with a distribution weir at the top. The fluid to be vaporized entered the weir and overflowed, running down the reboiler wall in a thin film. Rapid vaporization was obtained by maintaining a large temperature driving force between the bubble point of the liquid and the temperature of the steam condensing in the jacket. The vapor produced in the reboiler was then fed to the vapor manifold leading to the lower chamber.

The feed to the reboiler was obtained from a stream splitter which divided the fluid leaving the bottom of the packed section into a bottoms product stream and the reboiler feed. A sight glass was provided so that the liquid level in the lower chamber could be monitored as shown in Figure 6. The liquid was removed by a D-11 Eastern

pump and passed through a rotameter-needle valve combination where the rate was indicated and adjusted. This liquid stream then went to another rotameter-needle valve combination where a portion of the stream was metered into the reboiler weir and the remainder passed through a product cooler to the receiver. All lines in this area were well insulated to prevent condensation of vapor. It should be noted that the reboiler had a tendency to slightly superheat the vapor it produced which also aided in the prevention of partial condensation.

Auxillary Systems

In addition to the operating system, two auxillary measuring systems were used to obtain experimental data. A Perkin-Elmer 154D Vapor Fractometer was used to analyze the samples of the feed, overhead product, and bottoms product drawn from the operating apparatus. The instrument was operated as a capillary gas-liquid chromatograph (GLC) with a hydrogen flame detector. A 150 foot Golay column (0.01 inch ID) with squalene as the substrate was used to separate the sample into its components. The column was operated at 70°C with nitrogen carrier gas at a pressure of 15 psig. Figure 7 displays the order in which the components were eluted and detected by the instrument. Calibration curves, which related peak height fraction to mole fraction, for each of the binary pairs were prepared by Novinski (24).

A Leeds and Northrup sixteen-point temperature recorder was utilized to measure the temperature of the feed, overhead vapor, bottoms liquid, and reboiler vapor. It was series connected so that

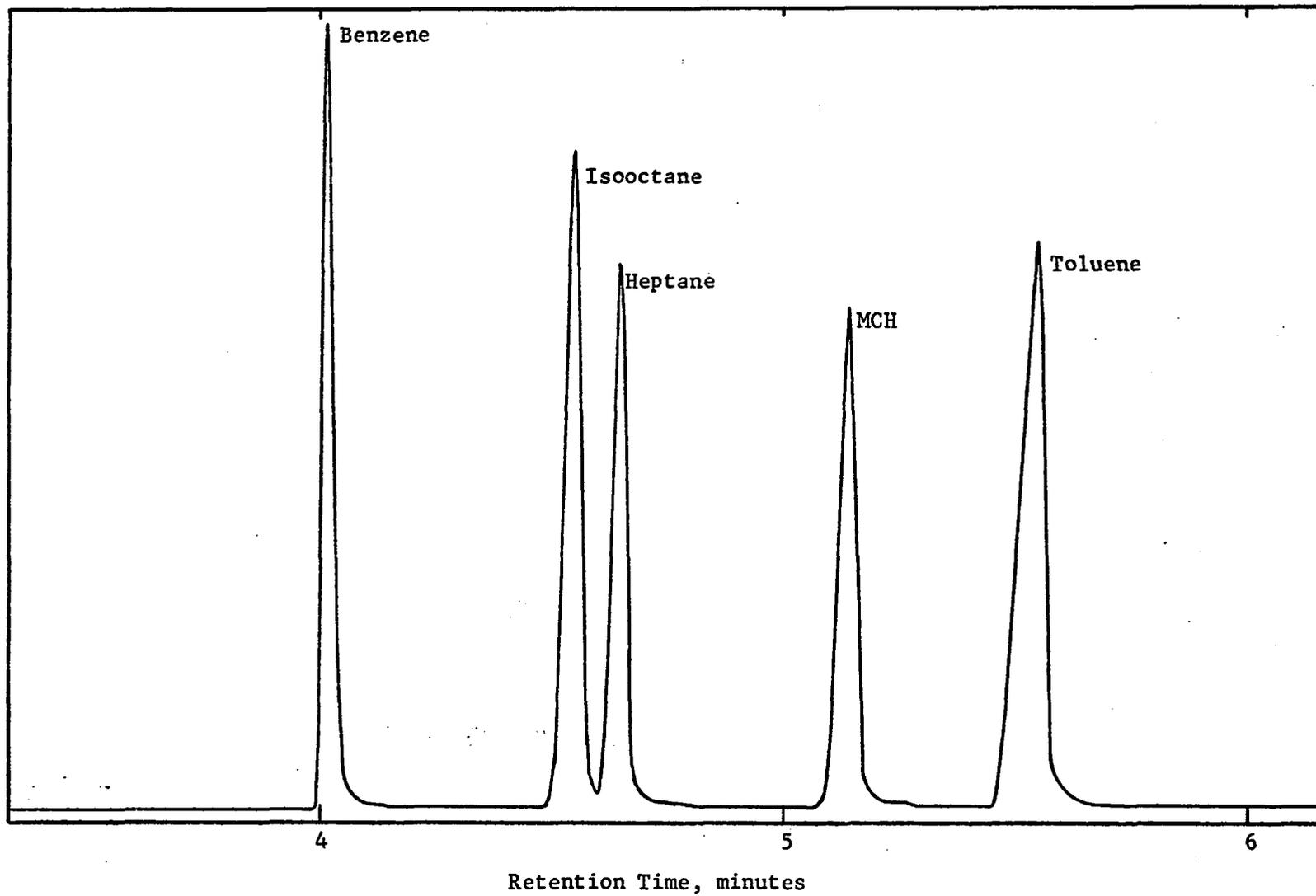


Figure 7. Sample Gas-Liquid Chromatograph Record

each temperature was measured four times per 16-point print cycle. Iron-constantan thermocouples were used and the recorder range was 0°C to 150°C.

Procedure

The operating procedure consisted of three parts: start-up, continuous operation, and shut-down. Prior to any set of experimental runs it was necessary to calculate the desired operating conditions. These calculations for the fraction of bottoms liquid which was to be returned to the reboiler (V/L at the column base) were necessary so that the experimental runs would fall in the region under study, fifty to eighty percent of flooding.

Start-Up Procedure

The column was operated at total reflux to allow all parts of the system to come to the desired temperatures. An initial charge of material was introduced to bring all liquid levels to the desired operating levels. Cooling water flow to the overhead condenser was started and steam was fed to the reboiler jacket. Valves were adjusted so that all condensed vapors were returned to the top of the packed section and all liquid removed from the bottom chamber was fed to the reboiler. The system was operated in this manner for three to four hours.

Procedure for Continuous Operation

Flow from the feed system was initiated. Steam was supplied to the preheater to bring the temperature of the feed close to its

bubble point. The electric heater was then used for fine adjustment of the temperature. The reflux line was closed and overhead product flow to the receiver began. The desired V/L ratio (precalculated) was set in the reboiler system by adjusting the stream splitter until the proper rates were indicated. The system was then maintained under these conditions for the duration of the run.

The first set of samples was taken when the temperature recorder indicated that all temperatures were constant. Two additional sets of samples were then drawn at 15 minute intervals. Analyses performed on several different runs early in the experimental program showed that if thermal steady state was maintained, the composition of the samples did not change more than one or two-tenths of a percent. Therefore, three equivolume blends of the three samples taken were made and analyzed to give the compositions of the streams.

The flow rates of the overhead and bottoms product were measured several times during the course of a run by weighing a sample collected over a given interval of time. The sum of these two rates was compared with the feed rate as a further check for steady state operation.

During the course of a day's operation, experimental runs were made at three different V/L ratios. The receiver drums which were collecting the combined overhead and bottoms products were transported to the upper level to become additional feed. They were carried up on an elevator and were connected to the feed system as the feed drums were emptied. The continuous operation of the column took an average of four to five hours to complete.

Shut-Down Procedure

The heat sources were turned off at the conclusion of the day's final run. All lines were drained of organic material and the cooling water was shut off. The next morning after the equipment had cooled an aspirator bottle was used to remove material which had drained from the packing and material which had collected in the low spots in the associated piping. When the system was completely cleared of organic, a different binary pair could be fed to initiate a new set of runs.

Two different procedures could be used when all runs on a given packing-column combination were complete. The dry packing could be removed with an industrial vacuum cleaner and the column repacked with a different packing or the operation could be switched to a different sized column. Changing columns necessitated removing blinds from flanges on the new column and placing corresponding blinds in connections to the old column. In either case it was necessary to spend a day preparing for a new set of runs.

Experimental Data Obtained

The data obtained from each experimental run is contained in Appendix B. This data can be categorized in the following groups: rate data, temperature data, pressure data, composition data, and fixed data. The fixed data consists of the column size, packing type and size, and the organic pair processed. The rate data is made up of the flow rates of the feed, overhead product, and the bottoms product as well as the V/L ratio from the reboiler system rotameters. The chromatographic analyses of the process streams comprise the

composition data. The ambient pressure and the pressure in the lower chamber were recorded. The temperatures of all process streams were obtained from the multipoint temperature recorder chart.

Experimental Problems

Several different aspects of the design of the equipment caused experimental problems. Some of these problems necessitated immediate solutions before experimental data could be taken. Others invalidated a portion of the initial data taken by causing operation in a region other than that under study. And finally, a few problems were endured but should be corrected before any additional work is carried out.

Feed Temperature

A feed which was heated to near its bubble point was used to allow the packed section to operate with a V/L ratio that was fairly constant. Initial runs were made with feed which was either not heated or was heated to a temperature approximately 20F° under its bubble point. In either case a majority of the vapor produced by the reboiler was condensed in elevating the feed to its bubble point. When the temperature of the feed was raised within 5F° of the bubble point this sensible heat effect was minimized and the vapor rate was constant in the packed section.

Overhead Condensation

Analysis of the results from the initial runs made with feed at the proper temperature indicated that the overhead product contained a higher concentration of the volatile component than possible from

theoretical considerations. This fact indicated the existence of a partial enriching stage in addition to the desired stripping stages. The only explanation for this type of behavior was condensation due to heat losses in the overhead system. The condensate was contacting the vapor from the stripper and was raising the vapor concentration by acting as reflux.

Fiberglass insulation was wrapped around the overhead system but did not eliminate the noted effect. The addition of electric heating tapes allowed energy to be added to the pipe walls to account for the losses. During operation with the overhead heaters, the vapor leaving the stripper was slightly superheated due to contact with the hot pipe.

This problem could be completely eliminated by placing the overhead condenser at an elevation below the top of the columns. Any material which condensed in the piping in this configuration would then drain to the condenser and leave the system rather than returning to the column as reflux.

Redistributors

The initial experimental plan called for redistributors to be placed in the packing to redirect liquid from the walls back into the packing. Flat ring or orifice plate redistributors were placed in the packing based on a reproducible relationship which involved the column diameter, D , and the packing size, d_p . The rings were made from one-eighth inch aluminum sheet and had a width equal to the particle size.

They were separated in the bed at a distance (in inches) equal to one and a half times the ratio, D/d_p .

No problems were encountered with the redistributors placed in the packing until the first runs were made with a packing having a low packing factor. A low packing factor is characteristic of the larger packings which require wide redistributors. To achieve eighty percent flooding in a tower packed with a packing having a low packing factor it is necessary to operate at a high V/L ratio. The reduction in effective diameter caused by the redistributors had the effect of causing the vapor velocity to become several hundred percent of flooding at the ring. This effect occurred primarily at the top ring. The feed liquid entered the upper chamber but could not overcome the high vapor rate at the ring and therefore could not flow down the column. Since more feed was entering the chamber the only exit route was through the piping to the condenser. No bottoms product was available under these conditions because no liquid was replenishing the reboiler system.

Two solutions to this problem were available, either to modify the redistributor or to remove them completely. A reduction of the width of the redistributor eliminated the major reason for placing them in the packing, which led to the decision to remove them. Further inquiry (19) indicated that industrial distillation research showed that proper distribution of both phases being fed to the packing eliminates the need for redistribution.

Uncalculatable Data

Several sets of runs with the heptane-methylcyclohexane binary pair produced no usable results. The stream compositions obtained indicated that the temperature-composition data for the system was highly inaccurate. The runs did show that the stripping section was performing correctly but no mass transfer efficiency information could be calculated. The toluene impurity which led to doubts about considering the system a binary pair, led to the decision to eliminate the runs with this material.

Continuing Problems

A few problems continually caused difficulties throughout the experimental program. The first of these problems was excessive drum handling. The use of a feed tank and a feed pump and a method for transferring the recombined products to this tank would eliminate this problem. These changes would allow the inventory of flammable material to be reduced and thereby make the entire system safer.

The operation of the preheater is by nature a very sensitive operation to control. The addition of slightly more heat than necessary causes the feed to vaporize, greatly expanding the feed volume. This expansion further slows down the flow rate, thus contributing to the problem. Pumped feed would keep the rate constant by insuring that the feed pressure remained fairly high. A reduction in preheater area would also help keep the preheater under control.

The columns were mounted in a triangular configuration so that a simple tee-shaped manifold could be used at top and bottom. This arrangement has no degrees of freedom so it was necessary to loosen several fittings to change gaskets or to place blinds when switching columns. A linear manifold would allow for the columns to be connected individually so that one column could be operated while another was being repacked.

Two minor problems were also annoying. Rubber was used for the gaskets and for tubing connections to the sight glass. The hot aromatics caused the rubber to rapidly lose its resilience. Condensation in the manometer leads led to an organic layer on top of the manometer fluid.

Summary of Experimental Program

A total of 181 runs were made. The first ten runs involved the feed temperature and the overhead condensation problems. Runs 11 to 74 were made with redistributors placed in the packing. Of these runs, twelve were made with the heptane-MCH mixture. The 107 runs remaining comprised the experimental runs which provided data for the study of factors influencing the scale-up of packed distillation columns.

CHAPTER 4

EXPERIMENTAL RESULTS

The experimental data taken during the course of the experimental program was used to determine the mass transfer efficiency of the packed section under specific operating conditions. In this chapter the data from an experimental run will be processed to demonstrate the calculations performed. The results from the set of 107 experimental runs are presented in tabular form and trends displayed are discussed.

Experimental Data

The data presented is for Run 170, a typical run with the benzene-toluene mixture. The six inch column ($D=6$) was packed with 3/4 inch Berl saddles ($P=170$). The flow rates of the various streams in lb/min were: Feed= $L_0=5.87$, Overhead Product= $V_1=3.32$, and the Bottoms Product= $B=2.55$. A V/L ratio of 0.58 was measured at the reboiler. The results of the GLC analyses gave the composition of the streams in mole fraction benzene: $x_F=0.536$, $y_{OH}=0.699$, and $x_B=y_B=0.345$. The pressure drop across the packed section was 9.2 inches of water. The ambient pressure was 701 mm Hg abs. The temperatures of the streams were: $T_F=86^\circ\text{C}$, $T_{OH}=90^\circ\text{C}$, $T_{BL}=96^\circ\text{C}$, and $T_{BV}=105^\circ\text{C}$.

Preliminary Calculations

Preliminary calculations must be performed to convert the experimental data to forms which are more useful for further calculations.

Temperature Conversions

The temperatures were converted to degrees Rankine and compared with the temperature-composition diagram to verify that the thermal behavior was as expected. The feed temperature, 647°R, was 4 R° below the bubble point of the mixture. The overhead temperature, 654°R, was the same as the expected value. The bottom liquid temperature, 665°R, was 4 R° above the expected and the bottom vapor temperature, 681°R, was 9 R° above the expected. These higher temperatures were due to an elevated pressure and superheating of the bottom vapor as previously mentioned in Chapter 3.

Pressure Conversion

The pressure drop was converted to millimeters of Hg and added to the ambient pressure to obtain the bottoms pressure, 718 mm Hg abs.

Weight Fractions

Several of the calculations to be performed require that the stream compositions be known in weight fraction, c , therefore a conversion from mole fraction must be made. The values are:

$$c_F = \frac{xM_a}{xM_a + (1-x)M_b} = \frac{.536(78)}{.536(78) + .464(92)} = 0.495$$

$$c_{OH} = 0.663 \text{ and } c_B = 0.309$$

Overall Material Balance

The overall material balance was checked to see if steady state operation was achieved. The feed rate was 5.87 lb/min. The output, $B+V_1$, was the sum of 2.55 and 3.32 or 5.87 lb/min.

Component Material Balance

The component material balance was performed to check the consistency of the analyses.

$$\text{Input} = Fc_F = 2.91 \text{ lb benzene/min}$$

$$\text{Output} = Bc_B + Vc_{OH} = 2.99 \text{ lb benzene/min}$$

The values indicate a three percent error in the component material balance.

Flooding Calculations

The flooding calculation is performed at both the top and bottom of the packed section and the values are averaged to give a mean degree of flooding for the packed section.

Physical Properties

The density of the liquid phase is obtained by assuming an ideal solution of the pure components at their bubble points.

$$\frac{1}{\rho_L} = \frac{c_F}{\rho_{La}} + \frac{(1-c_F)}{\rho_{Lb}} = \frac{0.495}{50.62} + \frac{0.505}{48.92} = 0.0201$$

$$\rho_L = 49.8 \text{ lb/ft}^3$$

$$\psi = \rho_{H_2O}/\rho_L = 1.25$$

The density of the vapor phase is obtained from the ideal gas law.

$$\rho_V = \frac{\pi \bar{M}}{RT} = \frac{(701)(82.2)}{(555)(654)} = 0.159 \text{ lb/ft}^3$$

The viscosity of the liquid is obtained from a linear extrapolation between the pure component bubble point viscosities as all values were approximately the same.

$$\mu_L = .536(.317) + .464(.248) = 0.285 \text{ cp}$$

The pure component physical properties were obtained from the American Petroleum Institute Project Number 44 (31).

Percent Flooding

The physical properties are substituted into the variables of the generalized pressure drop correlation (see Appendix D). The two variables are:

$$X = (L/V_F)(\rho_V/\rho_L)^{1/2} = 0.0276/V_F$$

$$Y = \frac{(V_F/S)^2 P \psi \mu_L^{0.2}}{g_c \rho_V \rho_L} = 0.649 V_F^2$$

The above expressions in conjunction with Figure D-1 lead to the flooding vapor rate, $V_F = 5.49 \text{ lb/min}$. The degree of flooding is then calculated, $V/V_F = 60.4\%$. A similar calculation using the values obtained for the bottom of the packed section yields a value of 61.8% flooding. The values at both ends of the packed section were averaged to give the mean degree of vapor flooding ($F = 61.1$).

Height Equivalent to a Theoretical Plate

The Ponchon-Savarit method of solution of Equations 10, 11, and 12 was performed and is presented in Figure 8. The graphical solution shows two full theoretical plates and a partial plate. When Equation 13 is used one obtains 2.21 theoretical plates for Run 170. The number of theoretical plates is then divided into the total packed height of 5 feet and a height equivalent to a theoretical plate of 2.26 feet is obtained.

Height of an Overall Gas Phase Transfer Unit

A plot of the operating points from Figure 8 ($x_0, y_1; x_1, y_2; x_2, y_3; \text{ and } x_B, y_B$) showed that the operating equation can be represented by a straight line ($y = mx + n$). It is also possible to represent the equilibrium relationship by a series of straight lines ($y^* = ax + b$). The integration of Equation 3a can be performed analytically because of the linearities.

$$\text{NOG} = \int_{y_B}^{y_{\text{OH}}} \frac{dy}{(y^*-y)} = \left(\frac{m}{m-a} \right) \ln \left[\frac{(bm-an)-(m-a)y_{\text{OH}}}{(bm-an)-(m-a)y_B} \right]$$

For the data of Run 170 three linear segments were required to represent the equilibrium line and the solution became:

$$\text{NOG} = \int_{.345}^{.445} \frac{dy}{(y^*-y)} + \int_{.445}^{.629} \frac{dy}{(y^*-y)} + \int_{.629}^{.699} \frac{dy}{(y^*-y)}$$

$$\text{NOG} = 0.51 + 1.47 + 1.13 = 3.11$$

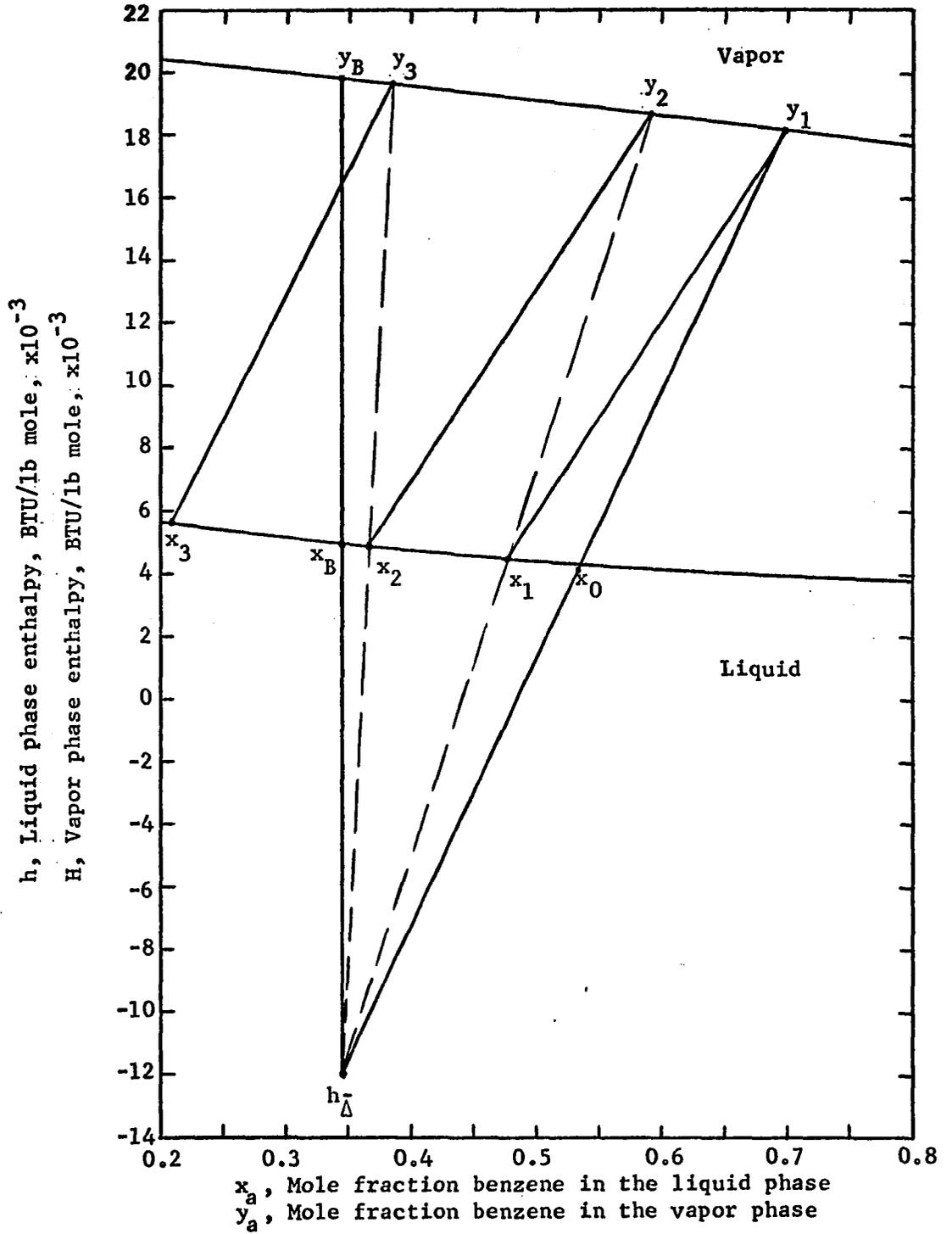


Figure 8. Ponchon-Savarit Solution for Run 170

The value of NOG divided into five feet gave the height of an overall gas phase transfer unit of 1.61 feet.

Summary of Experimental Results

The solution for the number of theoretical plates for each run was performed graphically using the enthalpy-composition diagrams presented in Appendix C. The degree of flooding, component material balance, and number of transfer units were calculated with the aid of the departmental PDP-9 computer using programs developed by the author. Appendix B presents all calculated and experimental data obtained from the laboratory runs. Table 1 presents the calculated results showing the mass transfer efficiency, HETP and HOG, and the operational parameters: column diameter, D; packing factor, P; and the degree of flooding, F.

Discussion of Experimental Results

The literature presents several figures in which the mass transfer efficiency of an enriching section is displayed as a function of the flow rates in the section. In nearly all cases the curves plotted represent the behavior at a constant reflux ratio with liquid rate a constant fraction of the amount of vapor rising in the section. Enough data concerning a curve of this type was presented in Eckert and Walter (8) to enable conversion of the flow conditions to percent of flooding which allowed their Figure 10 to be replotted as Figure 9. Figure 9, which follows the table, shows that in the region of commercial interest, fifty to eighty percent of flooding, the

Table 1. Summary of Results Calculated from Experimental Data

(a) Identifications			(b)	(c)	(d)	(e)	
(1) Packing	(2) Column, ID	(3) Mixture	Run Number	F, Mean % Flooding	HETP (ft)	HOG (ft)	
1/4 inch Intalox Saddles (P=725)	2"	I	104	69.8	5.00	2.18	
			105	61.7	5.00	2.42	
			106	50.3	5.32	2.25	
		II	107	66.7	3.52	1.43	
			108	77.8	5.00	2.15	
	4"	I	109	67.1	3.30	1.69	
			152	51.0	3.86	1.61	
			153	64.7	3.26	1.53	
		II	154	79.4	3.39	1.68	
			155	79.2	3.88	2.23	
	1/2 inch Intalox Saddles (P=200)	2"	I	156	66.2	5.00	2.24
				157	49.9	5.40	2.30
				119	55.6	2.92	1.77
			II	120	66.4	2.15	1.55
				121	77.2	1.25	1.14
4"		I	116	52.9	3.04	1.83	
			117	67.9	2.12	1.39	
			118	82.6	1.36	1.08	
		II	137	51.2	2.71	1.68	
			138	65.0	1.84	1.38	
6"		I	139	78.3	1.09	1.00	
			134	81.8	0.85	0.73	
			135	68.0	1.42	1.01	
		II	136	53.6	2.60	1.51	
			158	82.2	0.98	0.91	
3/4 inch Intalox Saddles (P=145)	4"	I	159	66.4	2.28	1.72	
			160	52.3	3.13	1.87	
			161	52.1	3.92	2.18	
		II	162	67.1	1.85	1.35	
			163	88.0	0.72	0.63	
	4"	I	75	48.3	1.39	0.99	
			76	58.4	1.52	1.22	
			77	66.7	0.99	0.87	
		II	78	51.8	1.87	1.15	
			79	62.6	1.49	1.07	
			80	73.4	0.75	0.69	

Table 1, Continued

	(a)		(b)	(c)	(d)	(e)
(1)	(2)	(3)				
3/4 inch Intalox Saddles	6"	I	81	55.3	2.62	1.86
			82	62.2	1.71	1.32
		II	83	73.2	0.77	0.78
			84	66.4	1.48	1.22
			85	54.4	2.75	1.90
1 inch Intalox Saddles (P=98)	6"	I	86	44.1	4.23	3.10
			87	53.6	2.28	2.26
			88	60.0	1.78	1.75
		II	89	60.4	1.36	1.18
			90	52.1	2.12	1.33
			91	44.0	2.50	1.67
1/2 inch Berl Saddles (P=240)	2"	I	122	69.7	2.55	1.64
			123	60.7	3.23	2.09
			124	48.5	4.70	2.60
		II	125	74.7	2.13	1.48
			126	62.2	2.87	1.78
	4"	I	127	51.1	3.71	2.04
			149	49.3	2.35	1.33
			150	62.6	2.03	1.40
		II	151	77.5	1.10	0.82
			146	57.1	2.50	1.46
	6"	I	147	69.4	2.05	1.34
			148	83.0	1.32	0.98
			179	79.1	1.46	1.22
		II	180	61.3	2.81	1.77
			181	46.6	4.62	2.53
3/4 inch Berl Saddles (P=170)	4"	I	176	68.5	2.40	1.67
			177	79.6	1.44	1.07
			178	50.8	3.09	1.89
		II	140	48.0	1.74	0.90
			141	57.5	1.45	0.91
	6"	I	142	68.4	1.29	1.03
			143	59.3	2.15	1.45
			144	70.5	1.51	1.15
		II	145	80.6	0.86	0.76
			170	61.1	2.26	1.61
II	171	76.2	1.45	1.41		
	172	48.6	4.15	2.36		

Table 1, Continued

	(a)		(b)	(c)	(d)	(e)
(1)	(2)	(3)				
3/4 inch	6"	II	173	48.5	3.57	2.21
Berl			174	80.2	0.86	0.81
Saddles			175	63.9	1.93	1.35
1/2 inch	2"	I	110	63.0	5.00	2.32
Raschig			111	74.7	4.22	2.17
Rings			112	81.8	3.88	2.23
(P=580)		II	113	54.1	5.40	2.20
			114	72.7	4.20	2.23
			115	81.5	4.13	2.42
	4"	I	128	82.3	3.26	1.68
			129	66.8	5.00	2.21
			130	51.8	5.20	2.11
		II	131	80.8	3.01	1.70
			132	70.4	3.50	1.86
			133	57.9	5.00	2.08
	6"	I	167	79.5	4.10	2.16
			168	69.2	3.87	1.93
			169	53.5	2.90	1.13
		II	164	48.0	5.49	2.77
			165	70.2	4.60	2.60
			166	80.4	3.75	2.46
3/4 inch	4"	I	98	82.0	1.46	0.98
Raschig			99	70.4	2.56	1.56
Rings			100	59.4	3.00	1.75
(P=255)		II	101	60.6	2.83	1.74
			102	71.8	2.32	1.52
			103	82.7	1.77	1.26
	6"	I	92	51.0	2.76	1.48
			93	62.0	2.66	1.64
			94	73.0	2.08	1.50
		II	95	77.3	2.04	1.43
			96	65.9	3.07	2.07
			97	54.2	4.22	2.56

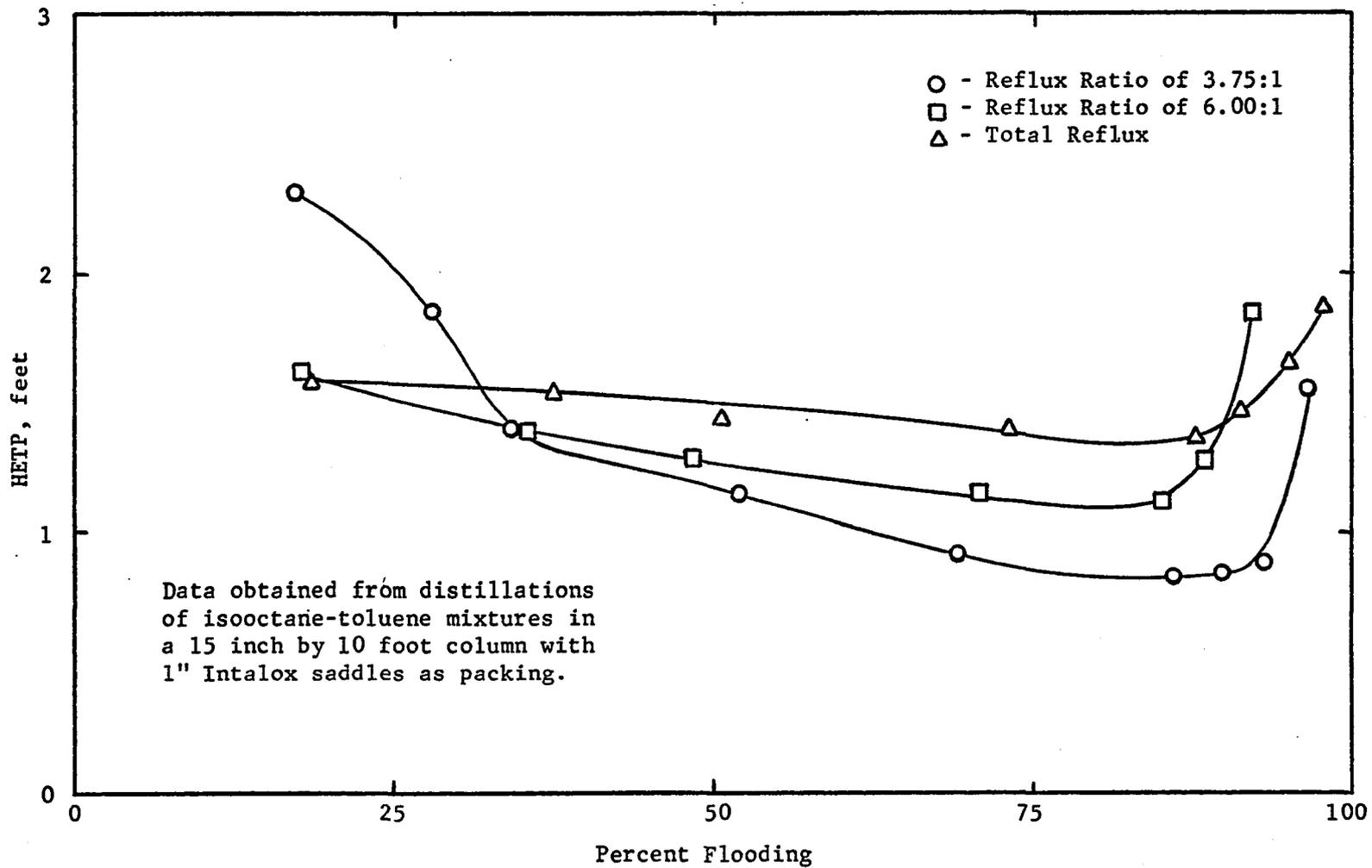
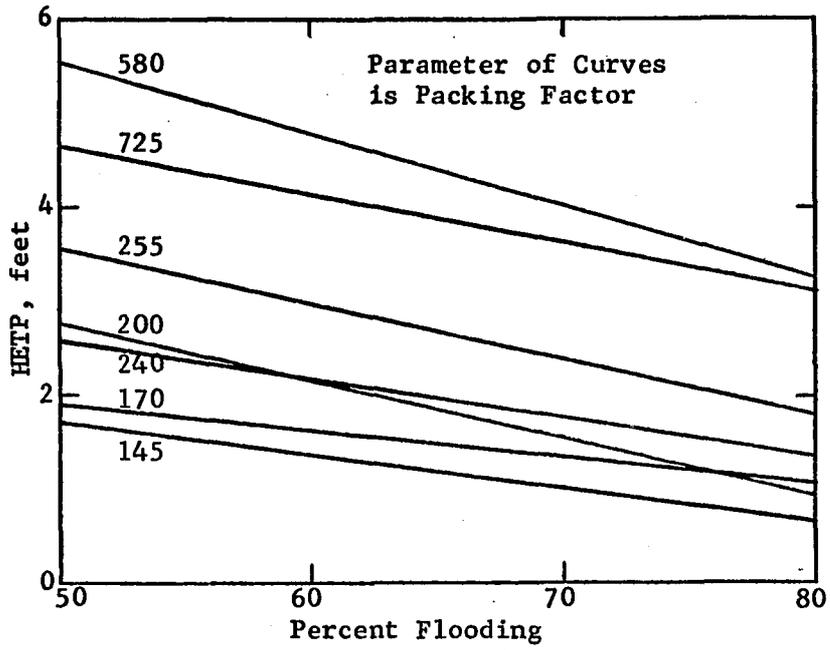


Figure 9. The Effect of Flow Conditions on Mass Transfer Efficiency.
Based on Figure 10 of Eckert and Walter (8).

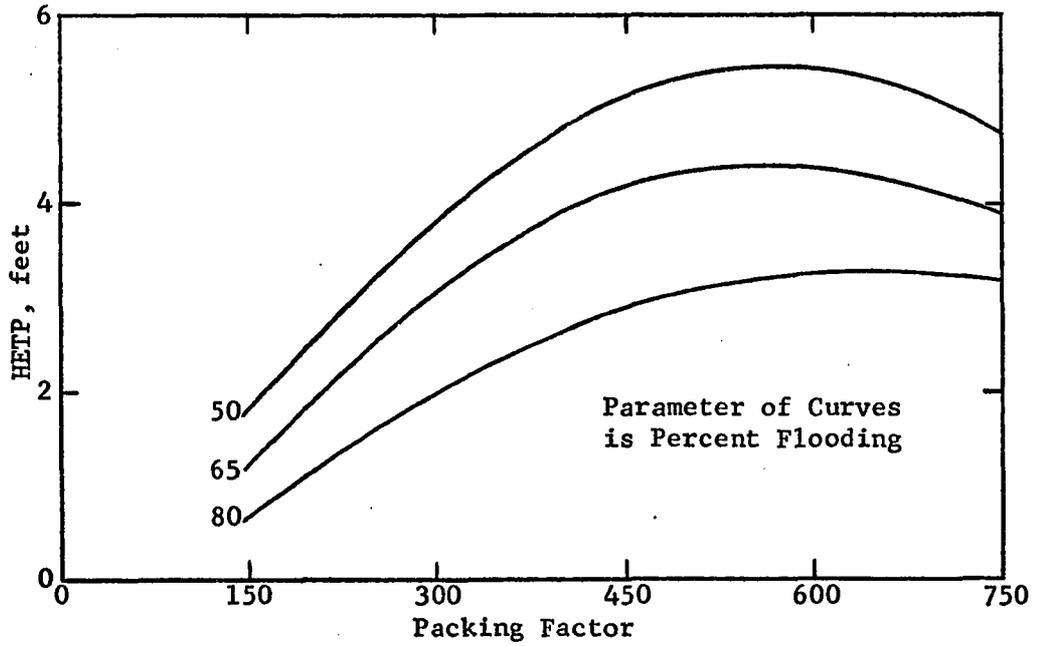
curves appear quite linear with each having a negative slope. There is no reason to expect that the general behavior indicated in Figure 9 for the enriching section should be dissimilar for the stripping section.

Preliminary examination of the experimental data showed a great deal of scatter and thus it was difficult to note any trends. The experimental points taken during operation of a single diameter column with a given packing, regardless of chemical mixture, were fit with a straight line relating mass transfer efficiency and degree of flooding to enable further examination of the data. Of the seventeen fits, only three did not display the negative slope expected on the basis of the curves in the literature. Further scrutiny of these three sets of data indicated that four experimental points (Runs 86, 107, 109, and 169) were apparently inconsistent with the other data of the sets. When these points were eliminated, new fits for the three sets displayed negative slopes similar to those obtained for the other fourteen sets. Figure 10a presents the straight line fits for the data obtained with the four inch column.

The straight line fits above were used for information concerning the mass transfer efficiency as a function of the other two macro-scale parameters: column diameter and packing factor. No consistent relationship was noted when the mass transfer efficiency was plotted versus column diameter. However, when the efficiency was plotted versus the packing factor, Figure 10b resulted. This figure indicates that mass transfer efficiency decreases with increasing



(a)



(b)

Figure 10. Trends Displayed by the Smoothed Four Inch Column Data.

(a) Mass Transfer Efficiency versus Degree of Flooding.

(b) Mass Transfer Efficiency versus Packing Factor.

packing factor, that is, HETP increases as packing factor does. This observation agrees with the recommendation of Fair (12) who states that a packing with a low packing factor is preferable for packed distillation service.

The scatter in the experimental data suggested examination of the process variables and their influence on the calculations made. The composition and flow rate of the process streams were found to be the variables which had the greatest effect on the calculations. An error of one percent in the measurement of the flow rates leads to an error of four percent in the degree of flooding. The compositions have minimal influence on the physical properties of both phases and thus minimal effect on the flooding calculation. Determination of the degree of flooding using the generalized pressure drop correlation is by nature a rather imprecise calculation because of scatter in the data used to determine the empirical packing factor. Problems associated with this correlation will be discussed in Chapter 6.

The compositions of the process streams are the variables which control the determination of the mass transfer efficiency. The flow rates of the streams do not enter into the mass transfer efficiency calculations. Examination of the component material balances presented in Appendix B shows that errors up to five percent are common. These errors generally indicate that more material was leaving the packed section than was entering. The GLC calibration curves in the region of interest are steep so that a one percent change in peak height fraction may result in as much as a two percent change in mole

fraction. Peak height fraction is the height of the chromatograph peak of the most volatile component divided by the sum of the heights of the peaks for both components. The maximum error in the measurement of the height of the peaks resulted in an error of approximately 0.003 in the peak height fraction. Based on this error, the conclusion may be drawn that the compositions may be in error by as much as one-half a mole percent. The magnitude of the error tends to increase as the concentration increases, leading to the conclusion that the chance for error is greater in the overhead and feed streams than in the bottoms stream. Since most of the volatile material was in the overhead and feed streams, errors in the component material balance were to be expected.

The effect of errors in concentration was studied by determining the mass transfer efficiency for a hypothetical section using typical concentrations. Each of the concentrations was varied by one mole percent in both the positive and negative direction. The variations were made both individually and in combination to determine the extremes of mass transfer efficiency possible. The maximum change yielding an increase in efficiency was noted when the overhead composition was raised and the other compositions lowered. The increase in efficiency was found to be 25% for HETP and 32% for HOG. The maximum decrease in efficiency was found when the changes made in composition above were reversed. The decreases were 11% and 27% for HETP and HOG respectively. This suggests that correlations with HETP as the dependent variable should be somewhat better than HOG

correlations. Since the expected concentration errors were about half of the values used in the above study, it may be assumed that the experimental values for HETP and HOG are good to $\pm 15\%$.

It should be stressed that all the experimental results in Table 1 were used in the development of an empirical equation as discussed in the next chapter. Even though some of the experimental data points appeared to be inconsistent with others, no definite reasons for their exclusion seemed to exist. The questionable points would not have as large an influence on the result when 107 points were being correlated as when six points were being used to determine the equation of a straight line.

CHAPTER 5

CORRELATION OF EXPERIMENTAL DATA

The results calculated from the data of one hundred and seven experimental runs have been presented in Chapter 4. This information consists of the mass transfer efficiency, both HOG and HETP, in terms of the correlating parameters: column diameter, D ; packing factor, P ; and the degree of flooding, F . Chapter 2 has presented the technique of multiple linear regression (MLR) which will be used to evaluate the coefficients of relationships tested as correlating equations. Since the F -test of Equation 40 may be obtained from the standard deviation, s , and the correlation coefficient, r , and a tabular value of significance is required for comparison, only the values of s and r will be presented in this chapter as an indication of the degree of fit. The relations used as correlating equations will be presented in three groups: ratio equations, polynomial equations, and equations that relate HETP and HOG.

Fitting Procedure

Equations of a polynomial form can be fit directly using an MLR program supplied by the University Computer Center (10). The ratio form equations required special programming since each experimental run was used as the basis for the other 106 runs. The interaction of the data resulted in 5671 different points on a ratio basis and the

available polynomial program was incapable of generating these points prior to implementation of the MLR routine.

The procedure for determination of the coefficients was identical regardless of the form used. Three fits were made on different bases for each equation form. These bases were: the full data set, and the data for each chemical mixture independently. The values of the coefficients of an equation with the general linear form

$$Y = c_0 + c_1X_1 + c_2X_2 + c_3X_3 + \dots \quad (44)$$

were obtained from each of the three fits and compared. The value of each coefficient obtained from the full data set was generally close to the mean value from the individual fits based on the data from each chemical mixture. One of the coefficients was selected as the best coefficient for the initial trial; this selection was made on the basis of the three values having the same order of magnitude. The term composed of the selected coefficient and its associated variable was then removed from the regression by repeating the individual fits for the modified equation

$$Y - c_2X_2 = c_0 + c_1X_1 + c_3X_3 + \dots \quad (45)$$

The use of this technique allowed the coefficients to be refined one at a time. The final values of the coefficients were generally similar to the coefficients of the initial fit with the full data set.

The empirical equations obtained using the above procedure were then used to calculate predicted values of the mass transfer

efficiency for each data point. The residual between the calculated and experimental values for the points was then used to calculate the value of the standard deviation and the correlation coefficient for use in rating the equation forms selected. The data for experimental Run 91 was used as the reference for the ratio equations. It was selected because it had a low residual in most of the fits tried.

The data with HETP as the dependent variable was used when many equation forms were being tested. The equation form which was selected as the best form for the HETP data was then used to fit the HOG data. When the empirical determination of the relationship between HOG and HETP had been made, the equations which represented the best fit of each height alone were used to calculate the predicted value of the corresponding mass transfer efficiency on the other basis.

Summary of Correlation Equations

Table 2 summarizes all the equation forms used in the determination of an empirical equation to fit the experimental data. The equations of each group are presented in order of increasing quality based on the values of the statistical tests. Examination of the calculated mass transfer efficiencies presented in Table 1 shows that HETP is greater than HOG, a fact which is also evident in the average values, 2.77 feet for HETP and 1.64 feet for HOG. This difference results from the two methods of viewing the mass transfer efficiency and because of these differences the standard deviations of HETP and HOG may not be directly compared. The polynomial equations had coefficients which were dependent on chemical mixture, identified by either

Table 2. Summary of Correlation Equations

<u>Ratio Equations</u>		
1. $HETP = H_R [0.85e^{-RD} - 2.48e^{-RP} + 6.64e^{-RF}]$	$s = 1.83$	$r = 0.810$
2. $HETP = 1.06 H_R \cdot \exp[0.05(1-RD) - 0.27(1-RP) + 1.30(1-RF)]$	$s = 1.82$	$r = 0.813$
3. $HETP = 0.95 H_R (RD)^{-0.02} (RP)^{0.68} (RF)^{-1.07 \cdot RD}$	$s = 1.71$	$r = 0.838$
4. $HETP = H_R + 0.21e^{-RD} - 7.92e^{-RP} + 10.15e^{-RF}$	$s = 1.62$	$r = 0.854$
5. $HETP = H_R + 0.39e^{RD} + 0.002e^{RP} - 0.226e^{RF}$	$s = 1.06$	$r = 0.941$
6. $HETP = H_R (RD)^{-0.01} (RP)^{0.71} e^{1.68(1-RF)}$	$s = 1.03$	$r = 0.944$
7. $HETP = H_R \{-0.08[1 - e^{(1-RD)}] + 2.04[1 - e^{(1-RP)}] - 1.19[1 - e^{(1-RF)}]\}$	$s = 0.97$	$r = 0.950$
8. $HETP = H_R (RP)^{0.49 \cdot RD} (RF)^{-1.29 \cdot RD}$	$s = 0.95$	$r = 0.953$
9. $HETP = H_R - 0.64[1 - e^{(1-RD)}] + 1.84[1 - e^{(1-RP)}] - 4.09[1 - e^{(1-RF)}]$	$s = 0.93$	$r = 0.954$

Table 2, Continued

10.	$HETP = 0.94 H_R(RD)^{-0.015} (RP)^{0.713} (RF)^{-1.72}$	$s = 0.88$	$r = 0.959$
11.	$HOG = H_R(RD)^{-0.022} (RP)^{0.365} (RF)^{-1.15}$	$s = 0.45$	$r = 0.967$

Polynomial Equations

12.	$HETP = c_0(N) [1.0 - 0.12D + 0.014D^2 + 0.16(P/100) - 0.012(P/100)^2 - 1.83(F/100) + 0.88(F/100)^2]$ $c_0(I) = 10.88 \quad c_0(II) = 7.26$	$s = 0.93$	$r = 0.955$
13.	$HETP = c_0(N) - 0.001D + 1.43(P/100) - 0.101(P/100)^2 - 15.9(F/100) + 7.54(F/100)^2$ $c_0(I) = 7.28 \quad c_0(II) = 6.60$	$s = 0.76$	$r = 0.970$
14.	$HETP = c_0(N) + 1.35(P/100) - 0.099(P/100)^2 - 15.8(F/100) + 7.46(F/100)^2$ $c_0(I) = 7.01 \quad c_0(II) = 6.79$	$s = 0.62$	$r = 0.980$
15.	$HETP = c_0(N) + c_1(N) [(P/100) - 0.073(P/100)^2] - 15.8(F/100) + 7.46(F/100)^2$ $c_0(I) = 7.01 \quad c_0(II) = 6.79$ $c_1(I) = 1.27 \quad c_1(II) = 1.42$	$s = 0.59$	$r = 0.982$
16.	$HETP = c_0(N) + 1.28(P/100) - 0.13(P/100)^2 - 17.9(F/100) + 7.38(F/100)^2 + 0.518(P/100)(F/100)$ $c_0(I) = 7.79 \quad c_0(II) = 7.89$	$s = 0.57$	$r = 0.983$

Table 2, Continued

17.	$HOG = [c_0(N) + 2.5(P/100) - 0.225(P/100)^2 - 39.6(F/100) + 24.7(F/100)^2]^{1/2}$		
	$c_0(I) = 15.85$	$c_0(II) = 13.20$	$s = 0.64$ $r = 0.931$
18.	$HOG = c_0(N) + 0.541(P/100) - 0.0434(P/100)^2 - 7.17(F/100) + 3.58(F/100)^2$		
	$c_0(I) = 3.70$	$c_0(II) = 3.64$	$s = 0.40$ $r = 0.973$

Interrelationships

19.	$HOG = 7.19 - [44.89 - 5(HETP)]^{1/2}$		
			$s = 0.46$ $r = 0.966$
20.	$HOG = 0.885 (HETP)^{0.626}$		
			$s = 0.40$ $r = 0.974$
21.	$HOG = 1.027 (HETP)^{1/2}$		
			$s = 0.39$ $r = 0.975$
22.	$HETP = 0.948 (HOG)^2$		
			$s = 0.75$ $r = 0.971$

a parenthetical I or II. The dimensionless ratios of the independent parameters are indicated by R followed by the identification letter of the parameter. The mass transfer efficiency of the reference run is referred to as H_R .

Discussion of Correlation Equations

The equations based on dimensionless ratios of the independent variables were the first group of equation types used in correlating the experimental data. The author felt that equations of this type would be the easiest to use as a scale-up design equation. Equation 41, the simplest ratio equation,

$$\frac{HETP}{HETP_R} = c_0 (RD)^{c_1} (RP)^{c_2} (RF)^{c_3} \quad (41)$$

was selected for the initial correlation attempts. These first attempts were made using graphical techniques but the large number of interacted data points as well as normal experimental scatter made any graphical method impractical and the technique of multiple linear regression became the prime correlating tool.

Several different ratio equation forms were used but the simplest and first used form presented above was found to be the best equation of this type for fitting the experimental data. Equations 10 and 11 of Table 2 present this form of ratio equation for both concepts of mass transfer efficiency. One observation which may be made is the lack of importance of the column diameter ratio due to the magnitude of its exponent.

The initial polynomial equation that was used in the attempt to fit the experimental data was second order in each of the independent variables. Equations of higher order were considered too involved for use as a general scale-up equation. Equation 12 of Table 2 presents the coefficients of the second order polynomial equation of the form

$$\text{HETP} = c_0 + c_1 D + c_2 D^2 + c_3 P + c_4 P^2 + c_5 F + c_6 F^2 \quad (46)$$

Examination of the coefficients indicated the expected parabolic behavior with respect to each of the independent variables. Comparison with Figure 10 is possible if two of the variables are held constant and the other is allowed to traverse the desired region. Degree of flooding versus the mass transfer efficiency has a slight concavity similar to the curves in Figure 9 rather than the assumed linear behavior displayed in Figure 10a. Packing factor versus mass transfer efficiency has the same general shape as Figure 10b but the vertex of the parabola occurs at a higher value of the packing factor. The behavior of the terms involving column diameter is notable because the vertex is in the experimental region where the curvature is slight. However, when the diameter is increased to values larger than the experimental values, the curvature of the parabola is such that poor mass transfer efficiency, i.e., large values of HETP and HOG, is predicted. It should be stressed that it is very dangerous to extrapolate to a region which is outside that used for the empirical determination of the coefficients.

The column diameter term that was second order was removed from the proposed equation so that the severe curvature would cease to be a problem. The linear diameter term of the modified polynomial (Table 2, Equation 13) had a coefficient of -0.001 . The magnitude of this coefficient (slope) is so small that column diameter effects are minimal, an observation consistent with that observed in the ratio equations.

A series of fits were made with the diameter term eliminated. This series included equations with one and two adjustable parameters and with a cross-product term. Although some improvement was noted as the equation form became more complex, the form with one adjustable parameter (Equation 14 of Table 2) seems to be the most useful scale-up equation because of its simplicity. The adjustable parameter may be thought of as a parameter which represents the chemical mixture. The value of this term would be obtained as a portion of the pilot plant study. The second order polynomial form that was obtained for HETP was used to fit $(HOG)^2$, a relationship suggested by an empirical fit of the interaction of the two efficiency concepts, but a better fit was obtained when the term was not squared (Equation 18 of Table 2) and the polynomial equations for both types of mass transfer efficiency had the same form.

The best equations representing the relationship between HOG and HETP are the final equations of Table 2. These equations allow the mass transfer efficiency on one basis to be transformed and are useful if data from several sources are being compared. Since any

given pilot plant study would be made on a common basis, the need for equations of this type are minimal.

Predicted curves of mass transfer efficiency versus packing factor based on Equations 10 and 14 of Table 2 are compared in Figure 11 with the smoothed experimental curve previously presented in Figure 10b. The curves, based on runs performed with the four inch column, are for 65 percent of vapor flooding. The polynomial equation gives a curve which has the same general shape as the smoothed data and gives reasonable predicted values over the entire range. The curve based on the ratio equation using the reference point indicated predicts the lower portion of the experimental region in a manner similar to that of the polynomial. However, values that are much greater than experimental are predicted for large packing factors. It should be pointed out that visual comparison of these multi-dimensional equations is very difficult and an attempt to superimpose the response surfaces predicted by each of the equations would be impossible.

The statistical tests performed to characterize the equation forms for selection of the best fit may be used to obtain an indication of the meaning of a prediction made with the equation. One method of doing this is to assume that each predicted point is in error by some constant fraction, e . The sum of the squares of the residuals then becomes

$$\Sigma \delta^2 = \Sigma (eY)^2 = e^2 \Sigma Y^2 \quad (47)$$

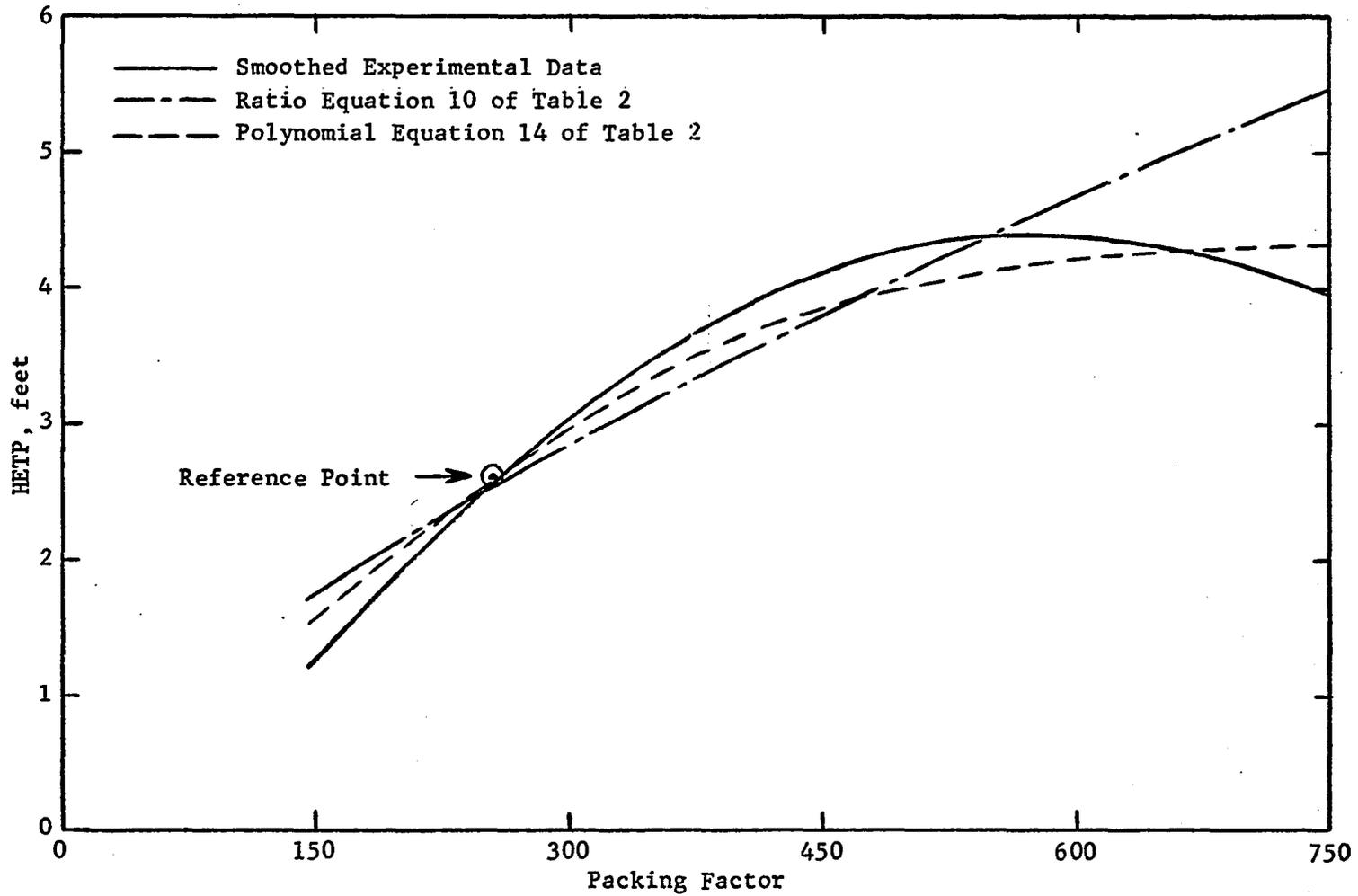


Figure 11. Comparison of Empirical Fits and Smoothed Experimental Data at 65 Percent of Vapor Flooding in the Four Inch Column

This expression may then be combined with the definition of the correlation coefficient presented in Equation 39 to give

$$r^2 = 1 - (\Sigma \delta^2 / \Sigma Y^2) = 1 - e^2 \quad (48)$$

The error fraction expressed as percentage may then be calculated when the correlation coefficient is available. Equation 14 of Table 2 for HETP has a value of 0.980 for the correlation coefficient which yields an average percent error of $\pm 20\%$. The average percent error for HOG is $\pm 23\%$. These error values are reasonable when one considers that the experimental values used to develop the correlation equations were estimated to be $\pm 15\%$.

The application of the results of the correlation study will be discussed in the next chapter. The strengths and weaknesses of the correlations as an aid to scale-up design will be noted. Also suggestions for further work that will improve the general applicability of the correlations will be made.

CHAPTER 6

DISCUSSION AND RECOMMENDATIONS

This research was carried out to study the effect of variation of three macro-scale design parameters on the mass transfer efficiency of a packed column operated as the stripping section of a continuous rectification column. The three design parameters studied were the column diameter, the material used to pack the column, and the counter-current vapor-liquid flow conditions in the column section. Each of these parameters was varied to cover the operating region common to most pilot plant studies. The mass transfer efficiency, calculated as both HETP and HOG, was determined for each of the one hundred and seven runs comprising the experimental program.

The values of the mass transfer efficiency and the design parameters for the experimental runs were used to obtain empirical correlations which represented the behavior of packed stripping sections in the region of study. The mathematical technique of multiple linear regression was the tool used to correlate the experimental data. Two equation families, based on different ideas, supplied the specific equation forms used to correlate the experimental data. An equation form based on dimensionless ratios of the design parameters assumes that one specific run is typical and may be used as a reference for all subsequent runs. A polynomial equation form fits the data by

adjusting the values of the coefficients. If the relationships between the individual correlating parameters and the mass transfer efficiency are assumed to be unaffected by chemical mixture, a general polynomial equation with one chemical mixture dependent coefficient is obtained. The experimental data was fit to several specific equation forms of each type and the best ratio and polynomial equation forms were selected. The dimensionless ratio equations which best fit the experimental data are

$$\text{HETP} = 0.94 (\text{HETP})_R (\text{RD})^{-0.015} (\text{RP})^{0.713} (\text{RF})^{-1.72} \quad (49)$$

and

$$\text{HOG} = (\text{HOG})_R (\text{RD})^{-0.022} (\text{RP})^{0.365} (\text{RF})^{-1.15} \quad (50)$$

The best polynomial equations are

$$\text{HETP} = c_0(N) + 1.35(P/100) - 0.099(P/100)^2 - 15.8(F/100) + 7.46(F/100)^2 \quad (51)$$

and

$$\text{HOG} = c_0(N) + 0.541(P/100) - 0.043(P/100)^2 - 7.17(F/100) + 3.58(F/100)^2 \quad (52)$$

where $c_0(N)$ is the chemical mixture dependent coefficient. These equations represent the effect the parameters of this study have on the mass transfer efficiency of packed stripping sections.

Discussion

Pilot plant data is used as the reference run in the case of the dimensionless ratio equation and for determination of the mixture dependent coefficient in the case of the polynomial equation. Once

the mixture dependent coefficient has been determined, no further pilot plant data is required when the polynomial equation is being used, but when the ratio equation is being used, the reference run must be used in all calculations. Therefore, the polynomial equations are much easier to use. The ratio equations do not fit the experimental data as well as the polynomial equations. For these reasons, the polynomial equations, Equations 51 and 52, should be considered the prime result of the current work.

The influence of the design parameters on the mass transfer efficiency of packed stripping sections in the experimental region may be found in the polynomial equations. Column diameter ($2'' < D < 6''$) has no effect on the mass transfer efficiency. The mass transfer efficiency decreases (HETP or HOG increases) as the packing factor ($98 < P < 750$) increases. The mass transfer efficiency increases as the degree of vapor flooding ($44\% < F < 88\%$) increases. These effects were determined for the region studied and the use of Equations 51 and 52 outside this region converts these equations to scale-up relationships.

The use of empirical equations as an aid to scale-up design requires that predictions of mass transfer efficiency in commercial-sized columns be reliable. Therefore, the validity of the equations in the commercial region must be verified. To obtain confirmation, one must have actual data concerning the same distillation performed in both pilot plant and commercial columns. A prediction of the mass transfer efficiency of the commercial column is made based on the information obtained with the pilot plant column. This prediction is

then compared with the actual value to evaluate the empirical equation for scale-up use. Unless data from both columns is available, the use of an empirical equation as a scale-up equation cannot be confirmed.

No data for the operation of commercial-sized stripping sections, characterized by a vapor rate less than the liquid rate, was found in the open literature for either the benzene-toluene mixture or the isooctane-toluene mixture so that comparisons could not be made with the pilot plant data of this study. Eckert (6) did present stripping section data for mixed methyl chlorides from a carbon tetrachloride plant and for high pressure (150 to 300 psia) columns operated in a natural gas plant. The reported data was for the operation of commercial columns, but no direct check of the validity of the scale-up equations could be made with this data either, as no pilot plant data was available for small columns performing these separations. However, some qualitative comparisons are possible if Eckert's data is assumed representative of normal behavior in commercial columns. The column diameters, 18 to 48 inches, were much greater than those of the current study. The degree of flooding, 40 to 70%, had approximately the same range as that covered in this study. The packing factors ranged from 20 to 98 whereas the latter value was the smallest used in the current study. The mass transfer efficiencies, reported as HETP, ranged between 1.5 and 3.3 feet, values which were of the same order of magnitude as those experimentally determined in this study. The operating region represented by the data of Eckert may be compared with the curves of Figure 11. This comparison

indicates that the resulting values of HETP based on the polynomial equations of this study would all be less than 1.5 feet. Since diameter effects have been excluded and the columns were operated in the same flooding region, extrapolation of the efficiency-packing factor relationship is a possible reason for the discrepancies. Further examination of the literature data indicated that the liquid rates were from two to ten times the liquid rate of the current study. Obviously the packing factor-liquid rate interactions have a great effect on the mass transfer efficiency of packed stripping sections.

The liquid rate macro-scale parameter which was held constant in the current work appears to be very important based on the above observations. Since the packing void space in which the vapor and liquid are contacted is constant for a given packing, an increase in the liquid flow rate reduces the space available for vapor flow. This reduction increases the resistance to vapor flow and thus the pressure drop. If the resistance is increased sufficiently, the column is said to be flooded, resulting in a large pressure drop and pulsating flow. A constant degree of flooding may be maintained in the section if an increase in liquid rate is followed by a decrease in the vapor rate. The vapor to liquid ratio in conjunction with the liquid rate therefore determine the degree of flooding in the packed section. It must be pointed out that in a stripping section the V/L ratio can never be greater than one because the reboiler may vaporize only all the liquid flowing down the section. Conversely, the V/L ratio in the enriching section can never be less than one because the maximum liquid rate is obtained when all the vapor is condensed and returned to the section.

A series of calculations were made to gain insight into the effect of variable liquid rate on the flooding behavior of a packed stripping section. The liquid viscosity and the densities of the phases were assumed to be the same as those reported in the example in Chapter 4. The V/L ratio required to cause the packed section to flood was calculated at three liquid rates for each of three values of the packing factor. The results of these calculations, shown below, include the 0.5 lb/sec-ft² liquid rate of the current work as well as two higher values.

<u>Packing Factor</u>	<u>V/L @ 100% Vapor Flooding</u>		
	<u>L = 0.5</u>	<u>L = 1.0</u>	<u>L = 2.0</u>
20	3.448	1.500	0.681
100	1.333	0.588	0.225
200	0.840	0.360	0.125

Remembering that a V/L ratio of one is the maximum possible in a stripping section, it is obvious that in some instances it is impossible to flood the packed section. The observation made previously, at constant packing factor an increase in the liquid rate requires a decrease in the V/L ratio to maintain the same degree of flooding, is confirmed. Another observation is that if the column is repacked with a material having a decreased packing factor, an increase in the V/L ratio is required to keep the degree of flooding unchanged, assuming constant liquid rate. The effect of simultaneous decrease in packing factor and increase in liquid rate, again at a constant degree of flooding, is to reduce the necessary V/L ratio. In addition, a tendency for higher efficiencies as the V/L ratio was increased was observed

in the analysis of the experimental data. Therefore, the reduction in V/L ratio, occurring when the simultaneous changes were made, results in a lower mass transfer efficiency. The data of Eckert (6) at lower packing factors and higher liquid rates, demonstrates behavior consistent with the above observations, i.e., lower efficiencies than those expected on the basis of the current work.

The correlation developed in the current work may be used as a scale-up design equation if a restriction is placed on the manner in which it is applied. Realizing that the liquid rate has considerable effect on the operation of the section, one must use pilot plant data obtained at the same liquid rate as the rate used in the design of the commercial column. The pilot plant column must be operated at several liquid rates, with the data obtained at each rate used to determine the mixture dependent coefficient at that rate. The use of the pilot plant data in this manner assumes that the effects noted in this study are similar if not identical to the effects that will be observed at higher liquid rates and therefore, only the chemical mixture dependent coefficient is affected by liquid rate. This assumption will need to be investigated in further work. Other recommendations for further work, enabling a more general scale-up design equation to be obtained, are presented in the following sections.

Recommendations for Further Work

The operation of the experimental apparatus to investigate the effect of liquid rate and to include some additional packing materials in the correlation will not require any modification of the apparatus

except those changes suggested in Chapter 3 to improve the operational procedure. Extensive modification will be necessary to study the effect of bed depth and to determine if similar effects are observed when the packed sections are operated as enriching sections. Modification of the capacity design methods require no experimental equipment.

Additional Packings

Three additional packings should be studied: Pall rings made of metal in 5/8 inch ($P=70$) and 1 inch ($P=48$) sizes, and 5/8 inch ceramic Raschig rings ($P=380$). Runs performed on columns packed with these materials, six-inch for the 1 inch packing and four-inch for the others, will contribute two things to the correlation. The runs with $P = 48$ and 70 will extend the relationship between mass transfer efficiency and packing factor into the commercial region. Also, the runs with $P = 380$ will give some data points in the region $255 < P < 580$ which was not investigated in this study. It should be pointed out that packings with factors greater than 275 are of little commercial interest but pilot plant columns are likely to be packed with these packings.

Liquid Rate Study

A study of the effect of liquid rate on the mass transfer efficiency of a packed stripping section should be performed. The four-inch column should be used for a majority of this study because of the flow rates involved. The amount of vapor produced by the reboiler limits this study. Based on a minimum value for the

falling-film heat transfer coefficient, ten pounds of hydrocarbon per minute may be vaporized. Considering the interaction between the liquid rate, the degree of flooding, and the V/L ratio it should be possible to operate the section with a liquid rate of 5.0 lb/sec-ft^2 , some ten times the rate in the current work. The low rate of this study was used so that the duration of the run, limited by the amount of material in the feed drums, was sufficient to attain steady state operation. To attain the higher rates suggested, elimination of drum handling, accomplished with the use of a feed pump and a closed, recirculating system as suggested in Chapter 3, is required.

When the series of runs is completed, new empirical correlations incorporating the liquid rate parameter would be developed. These equations would be obtained using MLR to evaluate the coefficients and would include both data from the current work and the new data. Runs with only one chemical mixture should be sufficient.

Effect of Bed Depth

The equipment would have to be modified so that packed sections of different heights could be investigated. The major modification would be made in the overhead system so that the vapors from the section would not have to pass through a great deal of piping to reach the condenser. It was mentioned in Chapter 3 that placing the condenser at a lower elevation would enable the majority of the overhead piping to drain to the condenser and thus eliminate the possibility of internal reflux. Another modification would be required so that the liquid from the feed system could be fed to the upper chamber, regardless of

elevation. The author suggests that packed sections up to ten feet in length should be studied.

Silvey and Keller (33) suggest that the mass transfer efficiency dependence on bed depth reported by several authors, as mentioned in Chapter 2, is caused by improper correction for end effects. They feel that characterization of the efficiency based on end samples is dangerous and that bed samples should be drawn to correctly determine the operating line. The fact that several samples are analyzed also increases the reliability of the mass transfer efficiency evaluation. No end corrections were made in the current study but it does seem reasonable to include provision for the acquisition of bed samples as a portion of the study of the depth effects.

Enriching Section Verification

The empirical correlation obtained after the results of the suggested studies are incorporated with the results of this study will include the effect of the liquid rate and bed depth parameters in addition to the effects of the parameters of this study. This comprehensive equation will represent the mass transfer behavior of a stripping section over a wide range of operating conditions. A series of runs should be made on packed sections operating as enriching sections covering the same experimental region. The same general type of behavior should be obtained but some coefficients of the empirical equation will change. The flow conditions are such that vapor rate becomes the rate of importance in the enriching section and for that reason liquid flooding is considered rather than vapor flooding. The

number of experimental runs would be considerably less because of the experience gained from the stripping section studies.

To operate the packed columns as enriching sections, modifications of the experimental apparatus are required. The liquid feed would feed directly to the reboiler where it would be totally flashed. The vapor rising through the column would pass to the overhead condenser for total condensation. The liquid from the condenser would go to a reflux splitting device where a constant fraction would be returned to the top of the packed section. The liquid would pass down the column and exit, remaining at its bubble point throughout. A large cooler would be required to enable the hot liquid bottoms to be handled easily. Operation of a packed section as an enricher is much easier than operation as a stripper. As a result of this fact, nearly all published data is for this mode of operation. Therefore, a great deal of data obtained with commercial columns already exists for comparison with the data which would be obtained with the pilot plant scale columns in this laboratory. Successful completion of this proposed study would result in a thorough understanding of the scale-up behavior of packed distillation columns.

Generalized Pressure Drop Correlation

The major weakness of the current study is the use of the GPDC to characterize the flooding behavior of the packed section. The degree of flooding calculated from this correlation depends on the value of the packing factor. This packing factor is empirically determined as the value which gives the best fit over a wide range of

vapor-liquid flow conditions. Therefore, the operating parameters included in the empirical correlation, developed to fit the experimental data of this study, are interrelated and the degree of flooding cannot be determined unless the packing factor is known. The value of the packing factor in the experimental region is not necessarily the best value for that region and consequently there is a chance for the GPDC to predict erroneous behavior as reported by Clay et al. (3). This introduces uncertainty in both correlating parameters and reduces the reliability of the empirical correlations which predict the scale-up behavior.

The major problem with the GPDC is that both terms of the correlation contain information concerning both phases. The flooding behavior of the section may not be easily discussed for this reason and involved studies such as that relating maximum possible flooding as a function of both liquid rate and packing factor are required. I therefore recommend a fundamental investigation of two-phase, counter-current flow in porous media be undertaken. The goal of this study would be a correlation in which the properties of both phases are separated. It may be possible to assume that the packed bed may be represented as a series of parallel capillaries with liquid flow down the walls and vapor flow up the center. The number and size of these capillaries would then take into account differences between packings. The data of Eckert, Foote, and Walter (9) is available to evaluate empirical packing constants appearing in the new correlation. There is no way to completely separate the type of packing from the degree of

flooding but a correlation of this type would be considerably easier to apply for design purposes.

The new flooding correlation would then be used in conjunction with the experimental data of both the current and future studies to calculate new values for the degree of flooding. The values based on the revised correlation could then be recorrelated to obtain scale-up design equations for both the stripping and enriching sections. These new equations would then supplant the equations based on the GPDC.

Equations 51 and 52, the polynomial equations obtained in this study, indicate the effect that packing factor and degree of flooding will have on the mass transfer efficiency of a packed stripping section. The effect of variation of column diameter was found to be minimal in the region covered in this investigation. The equations may be used as scale-up design equations if the pilot plant experimental data is taken at a liquid rate identical to that expected in the commercial column. The completion of the recommended studies will enable the results obtained in this work to be extended to packed distillation columns in general. All possible variations of the design parameters would be included in the comprehensive empirical correlation and restrictions to its use would not exist.

CHAPTER 7

CONCLUSIONS

The current work has investigated the mass transfer efficiency of packed stripping sections. The efficiency, measured as both height equivalent to a theoretical plate and height of an overall gas phase transfer unit, was studied by varying column diameter, packing material, and degree of vapor flooding in order to obtain empirical relationships which represented the two concepts of mass transfer efficiency in terms of these parameters. The conclusions which follow result from this investigation.

1. Vapor flooding must be considered in a packed section when the liquid rate is relatively constant and changes are made in the vapor rate such as occurs in a stripping section. Conversely, liquid flooding must be considered when the liquid rate is varied and the vapor rate remains steady as in an enriching section.
2. The previously reported correlations are quite complicated, often involving variables which are not readily available. Many of these correlations are also limited in scope. For these reasons, the previous correlations are unacceptable for use as scale-up design equations.

3. New temperature-composition data for the chemical mixture of heptane and methylcyclohexane must be obtained, due to the inconsistencies in the literature data, before meaningful results may be expected from runs with this mixture. In addition, the heptane-MCH feedstock used in this study should be fractionated to remove the toluene impurity.
4. The overhead condenser should be moved so that the pipes, which carry the vapor from the upper chamber, descend to the condenser in order to prevent internal reflux.
5. If proper distribution of both phases fed to the packed section is provided, the redistributors, which cause flooding, are unnecessary.
6. A feed tank and feed pump should be added and the system repiped so that recirculation of the combined products to provide fresh feed occurs, enabling the apparatus to be operated with increased safety and at higher liquid rates.
7. The manifolded systems around the upper and lower chambers should be eliminated and provision made for each column to be individually mounted.
8. Errors in the component material balance were caused by errors in measurement of the flow rate and composition of the process streams.
9. The calculated values of the mass transfer efficiency may be as much as fifteen percent high or low due to possible errors in the concentration of the process streams.

10. The best form of an equation, based on dimensionless ratios of the correlating parameters, for fitting the experimental data is

$$\text{HETP} = c_0 (\text{HETP})_R (D/D_R)^{c_1} (P/P_R)^{c_2} (F/F_R)^{c_3}$$

where HETP may be replaced by HOG and R denotes a reference value.

11. The best form of a polynomial equation to use to fit the experimental data is

$$\text{HETP} = c_0 + c_1 P + c_2 P^2 + c_3 F + c_4 F^2.$$

12. The best equation forms of both types have the required simplicity to be useful as scale-up design equations.
13. The empirical relationship between the two forms of mass transfer efficiency was determined to be

$$\text{HOG} = 1.027 \sqrt{\text{HETP}}.$$

14. Empirical correlations using the best equations of polynomial form fit the experimental data better than the best equations based on dimensionless ratios.
15. Column diameter has minimal effect on the mass transfer efficiency of packed stripping sections.
16. The mass transfer efficiency of packed stripping sections in the normal operating region increases as the degree of flooding increases.
17. The mass transfer efficiency of packed stripping sections increases as packings with increased vapor-liquid flow capacity (lower packing factors) are used.

18. The average error in the prediction of HETP with the polynomial equation was $\pm 20\%$ (for HOG $\pm 23\%$) only slightly greater than the expected accuracy of the experimental values.
19. The use of the empirical correlations as scale-up design equations cannot be confirmed due to the lack of commercial data corresponding to the data of the current work.
20. The polynomial equations may be used as scale-up design equations if the chemical mixture dependent coefficient is determined in the pilot plant studies at the same liquid rate as the rate desired in the commercial column.
21. Runs must be made with $5/8$ inch ceramic Raschig rings and with $5/8$ and 1 inch metal Pall rings to extend the range of the correlation.
22. A study of the effect of variation of liquid rate on the mass transfer efficiency of packed stripping sections must be performed to eliminate the restriction placed on the use of the correlation of this work.
23. A study must be made to determine the effect of the height of the packed stripping section on the section's mass transfer efficiency.
24. The comprehensive stripping section correlation, with the above two parameters added to the three parameters of this study, must be evaluated to see if similar effects are observed in a packed enriching section.

25. A new flooding correlation, involving separate terms for the behavior of the vapor and liquid phases, must be developed to replace the generalized pressure drop correlation.

APPENDIX A

NOMENCLATURE

Roman Letters

A_p	Surface area of one piece of packing
a	Interfacial area per cubic foot of packed volume
a_e	Effective interfacial area
a_v	Specific surface area per cubic foot of packed volume
B	Bottoms product rate, moles/time
C_L	Liquid heat capacity
c	Empirical constant with subscripted index or weight fraction
D	Column diameter
d_p	Packing size
\mathcal{D}	Mass diffusivity
e	Constant error fraction
F	Percent flooding
f	Value of the F-test
g	Acceleration of gravity
g_c	Newton's gravitational constant
HETP	Height equivalent to a theoretical plate
HG	Height of a gas film transfer unit
HL	Height of a liquid film transfer unit
HOG	Height of an overall gas phase transfer unit
HOL	Height of an overall liquid phase transfer unit

H	Vapor phase enthalpy
h	Liquid phase enthalpy
ΔH_{SOL}	Integral heat of solution
K	Mass transfer coefficient, lb mole/hr-ft ² -mole fraction
k	Film mass transfer coefficient, lb mole/hr-ft ² -mole fraction
k'	Mass transfer coefficient of Morris, lb/hr-ft ² -(lb/ft ³)
L	Liquid rate, moles/time
L_H	Liquid hold-up, volume of liquid/volume of packing
L_w^*	Column wetting rate, star indicates standard column value
M	Molecular weight
m	Slope of the equilibrium line
N_a	Mass transfer rate of the most volatile component
NOG	Number of overall gas phase transfer units
NOL	Number of overall liquid phase transfer units
n_o	Number of experimental observations
n_k	Number of independent variables
P	Packing factor of the GPDC
P'	Packing coefficient of Morris
q_B	Heat supplied to the reboiler
RD	Dimensionless diameter ratio, D/D_R
RF	Dimensionless flooding ratio, F/F_R
RP	Dimensionless packing factor ratio, P/P_R
r	Correlation coefficient
S	Cross-sectional area of the column
Sc	Schmidt number, $\mu/\rho D$
s	Standard deviation

T	Absolute temperature
V	Vapor rate, moles/time
V_F	Vapor rate at flooding
v_r	Gas velocity relative to the liquid surface
X	Independent variable in an empirical equation
x	Mole fraction of the most volatile material in the liquid phase
Y	Dependent variable in an empirical equation
y	Mole fraction of the most volatile material in the vapor phase
Z	Total packed height
z'	Packed height between redistributors

Greek Letters

α	Relative volatility
ϵ	Porosity
δ	Residual
λ	Latent heat of vaporization
μ	Viscosity
π	System pressure
ρ	Density
σ	Surface tension
ψ	Reciprocal specific gravity

Subscripts

a	Most volatile component of the binary pair
B	Bottom of the column
b	Least volatile component of the binary pair
C	Driving force in pounds per cubic foot concentration

F	Feed
f	Film value of Morris
G	Gas phase value
i	Value at the interface
J	General theoretical plate
L	Liquid phase value
lm	Logarithmic mean value
N	Value at the base of the packed section
OH	Overhead
p	Partial pressure driving force
R	Reference value or room temperature and pressure (1 atm-20°C)
RG	Vapor phase at RTP
RS	Property of the most volatile component at RTP
V	Vapor phase value
x	Driving force with the liquid concentration in mole fraction
y	Driving force with the vapor concentration in mole fraction
0,1,2	Plate number or constant index
$\bar{\Delta}$	Hypothetical difference stream

Superscripts

*	Equilibrium value
-	Average value

Symbols

m/o	Mole percent
w/o	Weight percent

APPENDIX B

EXPERIMENTAL DATA

Notes on the Following Tables

Certain abbreviations were used in the following tables to make the column headings more compact. These are as follows: OH indicates the overhead product stream, Btm denotes the bottoms product stream, the temperature of the liquid and vapor at the base of the packing are indicated by BLIQ and BVAP respectively, the composition of the various streams are in mole fraction abbreviated as Mole Frac., and V/L is the split ratio read off of the rotameters in the reboiler system. The binary mixtures are identified by Roman numerals as mentioned in Chapter 3.

Table B-1. Fixed Operating Data for Mixtures I and II

(a) Packing Material	(b) Column Diameter	(c) Chemical Mixture	(d) Redistribution	(e) Run Numbers	
1/4 inch Intalox Saddles	2"	I	Yes	38, 39, 40, 47, 48, 49	
			No	104, 105, 106	
	4"	II	Yes	41, 42, 43, 50, 51, 52	
			No	107, 108, 109	
		I	Yes	53, 54, 55	
			No	152, 153, 154	
		II	Yes	56, 57, 58	
			No	155, 156, 157	
1/2 inch Intalox Saddles	2"	I	No	119, 120, 121	
		II	No	116, 117, 118	
	4"	I	No	137, 138, 139	
		II	No	134, 135, 136	
		6"	I	No	158, 159, 160
			II	No	161, 162, 163
3/4 inch Intalox Saddles	4"	I	No	75, 76, 77	
		II	No	78, 79, 80	
	6"	I	Yes	66, 67, 68	
			No	81, 82	
		II	Yes	69, 70, 71	
			No	83, 84, 85	
1 inch Intalox Saddles	6"	I	Yes	59, 60, 61, 62	
			No	86, 87, 88	
		II	Yes	63, 64, 65	
			No	89, 90, 91	

Table B-1, Continued

(a)	(b)	(c)	(d)	(e)
1/2 inch Berl Saddles	2"	I	No	122, 123, 124
		II	No	125, 126, 127
	4"	I	No	149, 150, 151
		II	No	146, 147, 148
	6"	I	No	179, 180, 181
		II	No	176, 177, 178
3/4 inch Berl Saddles	4"	I	No	140, 141, 142
		II	No	143, 144, 145
	6"	I	No	170, 171, 172
		II	No	173, 174, 175
1/2 inch Raschig Rings	2"	I	Yes	29, 30, 31
			No	110, 111, 112
		II	Yes	32, 33, 34
			No	113, 114, 115
	4"	I	Yes	1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13
			No	128, 129, 130
		II	Yes	14, 15, 16
			No	131, 132, 133
	6"	I	Yes	20, 21, 22
			No	167, 168, 169
		II	Yes	23, 24, 25
			No	164, 165, 166
3/4 inch Raschig Rings	4"	I	No	98, 99, 100
		II	No	101, 102, 103
	6"	I	No	92, 93, 94
		II	No	95, 96, 97

Table B-2. Operating Data for Benzene and Toluene Runs

(a) Run Number	(b) Flow Rate, lb/sec-ft ²			(c) V/L @ Base	(d) Mole Frac. Benzene			(e) Temperature, °F				(f) Ambient Pressure (mm Hg)	(g) Pressure Drop (" H ₂ O)
	(1) Feed	(2) OH	(3) Btm		(1) Feed	(2) OH	(3) Btm	(1) Feed	(2) OH	(3) BLIQ	(4) BVAP		
1	0.494	0.096	0.415	0.237	0.500	0.747	0.435	172	190	196	232	700	3.1
2	.503	.132	.360	.312	.617	.800	.548	171	187	194	228	700	5.8
3	.503	.009	.496	.098	.607	.886	.571	167	181	190	214	700	0.5
4	.498	.087	.417	.241	.571	.805	.530	186	189	194	228	700	2.0
5	.505	.157	.368	.368	.588	.781	.497	189	190	198	228	700	4.7
6	.505	.068	.475	.167	.596	.809	.560	186	189	194	220	700	1.2
7	.491	.043	.470	.134	.542	.797	.465	185	189	189	196	700	2.5
8	.491	.103	.383	.234	.541	.727	.487	185	191	191	207	700	3.1
9	.491	.134	.364	.312	.530	.726	.463	186	191	193	213	700	4.5
10	.509	.066	.430	.169	.546	.732	.500	187	207	192	199	703	0.9
11	.503	.144	.364	.328	.528	.719	.462	183	194	193	214	707	4.7
12	.503	.096	.427	.242	.533	.730	.500	183	194	191	208	706	2.2
13	.503	.053	.464	.165	.540	.740	.516	186	194	189	199	706	0.7
20	.495	.167	.325	.353	.530	.722	.462	181	192	194	207	700	5.8
21	.500	.081	.420	.145	.535	.717	.510	187	192	192	204	700	1.2
22	.495	.120	.325	.250	.530	.723	.494	184	192	193	205	700	3.3
29	.572	.093	.479	.240	.520	.662	.503	186	196	192	205	702	2.2
30	.572	.093	.414	.300	.517	.593	.488	186	200	193	205	701	19.1
31	.586	.029	.558	.161	.516	.694	.517	184	194	192	205	700	-10.0
38	.550	.050	.500	.194	.555	.739	.509	185	191	192	205	697	1.5
39	.550	.079	.478	.222	.538	.746	.505	188	190	192	205	697	3.4
40	.543	.086	.414	.283	.531	.718	.503	186	192	192	204	695	25.0
47	.572	.064	.508	.196	.541	.745	.513	185	191	192	204	699	2.1
48	.578	.078	.500	.222	.542	.750	.512	185	191	192	204	699	3.9
49	.572	.114	.472	.260	.543	.699	.497	185	191	193	205	698	18.6
53	.500	.055	.418	.163	.547	.745	.519	187	191	191	204	702	1.3
54	.497	.100	.360	.251	.546	.730	.501	188	192	192	205	701	5.2

Table B-2, Continued

(a)	(b)			(c)	(d)			(e)				(f)	(g)
	(1)	(2)	(3)		(1)	(2)	(3)	(1)	(2)	(3)	(4)		
55	0.500	0.124	0.372	0.305	0.540	0.721	0.480	188	192	193	206	701	9.0
59	.497	.082	.415	.155	.544	.739	.494	187	191	193	205	701	-1.4
60	.496	.120	.376	.259	.526	.725	.470	187	192	194	206	700	-0.6
61	.497	.167	.330	.369	.533	.729	.446	187	192	195	207	700	4.4
62	.497	.236	.261	.440	.526	.693	.431	188	194	196	208	700	4.3
66	.497	.199	.298	.415	.551	.722	.428	188	192	196	208	696	-2.0
67	.497	.352	.145	.650	.531	.640	.232	189	197	208	217	696	9.9
68	.497	.455	.042	.800	.544	.593	.036	190	200	222	224	696	7.0
75	.500	.234	.266	.454	.536	.733	.367	186	192	202	213	699	2.0
76	.500	.292	.208	.586	.533	.699	.232	186	194	208	217	699	3.5
77	.496	.356	.140	.711	.544	.711	.112	185	193	217	222	699	5.6
81	.498	.254	.244	.540	.539	.688	.350	183	194	201	212	700	5.2
82	.497	.330	.167	.650	.544	.705	.260	184	193	206	216	700	2.5
86	.497	.255	.242	.525	.556	.663	.417	185	196	197	209	700	1.7
87	.495	.385	.110	.735	.522	.607	.217	184	199	209	217	699	2.1
88	.498	.406	.092	.803	.537	.621	.152	187	198	214	220	698	0.2
92	.497	.159	.338	.330	.541	.732	.455	188	192	195	207	698	0.4
93	.493	.216	.277	.440	.537	.714	.403	185	193	198	209	698	0.2
94	.494	.274	.220	.550	.525	.698	.326	185	194	202	213	698	0.1
98	.500	.285	.215	.610	.535	.727	.307	186	192	203	214	700	0.2
99	.500	.240	.260	.509	.532	.711	.396	186	193	198	210	700	0.6
100	.500	.194	.306	.407	.530	.708	.428	187	193	196	208	699	0.6
104	.495	.103	.392	.250	.522	.703	.487	188	193	193	205	700	2.2
105	.508	.079	.429	.205	.533	.701	.487	188	193	193	205	700	0.1
106	.500	.050	.450	.161	.533	.710	.502	188	193	192	204	700	-0.1
110	.500	.114	.386	.241	.531	.702	.482	189	193	193	205	700	0.5
111	.500	.150	.350	.310	.534	.705	.473	189	193	194	206	700	0.9
112	.500	.179	.321	.357	.531	.699	.461	189	193	195	207	699	1.3
119	.500	.214	.286	.440	.530	.702	.408	189	193	197	209	699	1.5
120	.500	.278	.222	.561	.536	.699	.328	189	194	202	213	699	3.0
121	.500	.336	.164	.682	.531	.687	.162	189	194	214	221	699	1.0

Table B-2, Continued

(a)	(b)			(c)	(d)			(e)				(f)	(g)
	(1)	(2)	(3)		(1)	(2)	(3)	(1)	(2)	(3)	(4)		
121	0.500	0.336	0.164	0.682	0.531	0.687	0.162	189	194	214	221	699	1.0
122	.500	.272	.228	.530	.529	.695	.355	189	194	200	212	699	2.9
123	.500	.214	.286	.439	.532	.688	.404	189	194	198	209	699	1.6
124	.500	.150	.350	.320	.532	.685	.456	189	194	195	207	699	0.6
128	.522	.164	.358	.340	.530	.717	.462	187	193	194	207	700	0.8
129	.485	.127	.358	.279	.530	.705	.482	188	193	193	206	700	1.1
130	.481	.091	.390	.184	.530	.712	.502	187	193	193	205	699	0.4
137	.490	.206	.284	.403	.532	.705	.398	188	193	198	210	700	1.8
138	.494	.206	.284	.561	.537	.700	.287	188	194	205	215	700	0.5
139	.494	.345	.149	.702	.529	.687	.114	188	194	217	222	699	2.0
140	.504	.215	.289	.395	.530	.733	.395	188	192	198	210	698	0.4
141	.506	.276	.230	.514	.530	.727	.317	189	192	203	213	698	0.2
142	.493	.313	.180	.650	.535	.709	.206	189	193	210	218	696	1.1
149	.517	.174	.343	.302	.542	.731	.430	188	192	196	208	701	0.1
150	.498	.251	.247	.464	.533	.712	.359	188	193	200	211	701	1.2
151	.504	.304	.200	.604	.536	.730	.238	188	192	208	217	700	2.2
152	.513	.064	.449	.140	.530	.726	.506	188	192	192	204	701	0.1
153	.498	.102	.396	.212	.529	.725	.496	188	192	193	205	701	0.2
154	.491	.147	.344	.296	.530	.720	.483	188	192	193	206	701	0.4
158	.498	.363	.135	.735	.532	.686	.077	187	194	219	223	702	0.3
159	.500	.283	.217	.562	.532	.682	.315	187	195	203	213	701	0.1
160	.498	.209	.289	.405	.542	.713	.442	189	193	195	208	700	0.1
167	.495	.179	.316	.345	.532	.703	.467	187	193	194	206	698	2.6
168	.499	.139	.360	.277	.534	.713	.478	188	193	193	206	698	3.6
169	.503	.095	.408	.177	.535	.738	.497	187	191	193	205	697	1.6
170	.488	.276	.212	.577	.536	.699	.345	187	194	201	212	701	9.2
171	.503	.363	.140	.732	.525	.643	.137	188	197	215	221	701	8.4
172	.509	.217	.392	.400	.547	.704	.468	188	193	194	206	701	10.6
179	.501	.320	.181	.630	.535	.702	.226	186	193	209	217	698	3.5
180	.499	.234	.265	.447	.529	.700	.399	186	193	198	210	698	4.7
181	.495	.154	.342	.298	.538	.695	.470	188	194	194	206	697	4.0

Table B-3. Operating Data for Isooctane and Toluene Runs

(a) Run Number	(b) Flow Rate, lb/sec-ft ²			(c) V/L @ Base	(d) Mole Frac. Isooctane			(e) Temperature, °F				(f) Ambient Pressure (mm Hg)	(g) Pressure Drop (" H ₂ O)
	(1) Feed	(2) OH	(3) Btm		(1) Feed	(2) OH	(3) Btm	(1) Feed	(2) OH	(3) BLIQ	(4) BVAP		
14	0.500	0.066	0.436	0.188	0.340	0.435	0.312	206	215	215	218	703	0.8
15	.500	.104	.398	.250	.340	.442	.317	206	214	215	218	703	1.8
16	.500	.141	.368	.312	.340	.430	.308	206	215	215	218	703	3.8
23	.493	.075	.418	.140	.357	.447	.332	203	215	215	217	702	1.2
24	.497	.110	.387	.220	.344	.442	.321	203	215	215	218	702	2.6
25	.493	.147	.346	.308	.338	.438	.300	203	215	215	218	702	5.7
32	.551	.021	.493	.190	.357	.485	.360	203	214	215	217	700	0.4
33	.543	.057	.486	.228	.366	.459	.353	203	215	215	218	699	1.2
34	.551	.072	.479	.275	.366	.455	.345	205	215	215	218	699	11.0
41	.551	.029	.522	.186	.368	.476	.359	203	215	215	218	697	2.5
42	.551	.050	.508	.216	.366	.462	.358	203	215	215	218	697	3.0
43	.537	.064	.465	.250	.366	.452	.353	203	215	215	218	696	22.5
50	.558	.043	.515	.190	.367	.470	.367	203	215	215	217	698	1.6
51	.558	.050	.508	.216	.367	.457	.359	203	215	215	217	698	2.6
52	.558	.072	.486	.246	.367	.457	.358	203	215	215	217	698	5.0
56	.500	.047	.460	.134	.365	.458	.352	208	215	215	218	700	2.3
57	.500	.081	.430	.193	.362	.448	.347	208	215	215	218	700	3.4
58	.500	.110	.389	.258	.368	.452	.347	208	215	215	218	700	5.7
63	.497	.068	.429	.153	.360	.447	.358	207	215	215	218	699	1.0
64	.497	.123	.374	.253	.365	.438	.340	208	216	216	218	698	5.2
65	.497	.173	.324	.363	.365	.443	.320	207	215	216	219	698	1.0
69	.498	.271	.232	.487	.367	.437	.280	206	216	217	220	703	0.7
70	.498	.335	.164	.607	.360	.430	.241	206	216	218	221	703	0.4
71	.497	.406	.091	.763	.362	.420	.130	206	216	221	223	701	0.5
78	.500	.256	.244	.471	.355	.446	.276	205	216	217	220	697	2.0
79	.500	.323	.177	.621	.365	.447	.222	205	216	217	221	697	3.6
80	.500	.398	.102	.783	.347	.428	.080	205	216	223	224	697	6.4

Table B-3, Continued

(a)	(b)			(c)	(d)			(e)				(f)	(g)
	(1)	(2)	(3)		(1)	(2)	(3)	(1)	(2)	(3)	(4)		
83	0.495	0.437	0.058	0.855	0.361	0.413	0.046	204	216	224	225	703	7.0
84	.497	.366	.131	.700	.362	.429	.178	204	216	220	222	702	1.0
85	.495	.263	.232	.510	.360	.433	.283	202	216	217	220	701	2.7
89	.495	.377	.118	.762	.359	.426	.163	201	216	220	222	699	6.2
90	.496	.305	.191	.620	.359	.437	.237	202	216	218	221	698	3.2
91	.497	.228	.269	.487	.359	.438	.283	202	216	217	220	698	2.8
95	.495	.282	.213	.560	.365	.445	.268	203	216	217	220	701	0.4
96	.498	.228	.270	.440	.365	.438	.303	203	216	216	219	701	0.7
97	.497	.178	.319	.323	.369	.440	.331	203	216	216	219	700	0.2
101	.500	.185	.315	.385	.367	.452	.322	203	215	216	219	698	0.7
102	.500	.234	.266	.490	.368	.452	.300	203	215	217	219	698	1.5
103	.500	.285	.215	.592	.361	.445	.250	203	216	218	221	698	0.7
107	.515	.057	.458	.216	.353	.450	.344	208	215	215	218	698	1.8
108	.515	.086	.429	.258	.354	.440	.338	208	216	216	218	697	3.0
109	.522	.093	.429	.275	.353	.445	.332	208	216	216	219	697	1.6
113	.500	.057	.443	.195	.353	.440	.345	208	216	215	218	697	0.2
114	.500	.121	.379	.273	.352	.435	.329	209	216	216	219	696	0.4
115	.507	.157	.350	.318	.356	.431	.320	209	216	216	219	696	0.8
116	.494	.172	.322	.387	.357	.438	.308	209	216	216	219	696	0.8
117	.500	.257	.243	.534	.353	.438	.274	209	216	217	220	696	1.7
118	.500	.350	.150	.698	.351	.435	.202	209	216	219	222	695	1.0
125	.500	.279	.221	.545	.359	.438	.265	209	216	217	220	698	3.0
126	.493	.217	.286	.432	.353	.435	.300	209	216	217	219	698	1.1
127	.507	.143	.364	.310	.353	.437	.323	209	216	216	219	698	0.2
131	.496	.156	.340	.326	.351	.440	.315	207	216	216	219	698	2.4
132	.490	.128	.362	.266	.355	.441	.325	207	216	216	219	698	1.3
133	.490	.087	.403	.198	.360	.447	.346	207	215	215	218	698	0.8
134	.493	.382	.111	.752	.361	.442	.111	206	216	222	224	700	2.7
135	.506	.273	.213	.548	.359	.448	.247	206	215	218	221	700	0.3
136	.506	.206	.300	.378	.358	.445	.302	206	216	216	219	700	1.2
143	.517	.276	.241	.487	.360	.442	.273	207	216	217	220	697	0.8

Table B-3, Continued

(a)	(b)			(c)	(d)			(e)				(f)	(g)
	(1)	(2)	(3)		(1)	(2)	(3)	(1)	(2)	(3)	(4)		
144	0.510	0.323	0.187	0.627	0.358	0.440	0.223	207	216	218	221	696	0.3
145	.498	.394	.104	.785	.356	.435	.100	207	216	222	224	696	3.1
146	.492	.208	.284	.378	.359	.446	.302	207	215	216	219	703	0.2
147	.502	.264	.238	.498	.358	.442	.270	207	216	217	220	703	0.7
148	.500	.321	.179	.638	.357	.444	.212	207	216	219	221	703	1.1
155	.502	.121	.381	.263	.362	.444	.336	206	215	216	218	701	0.4
156	.498	.091	.407	.200	.362	.445	.344	207	215	215	218	701	0.2
157	.493	.053	.440	.133	.358	.444	.350	207	215	215	218	701	0.1
161	.495	.178	.316	.374	.357	.435	.316	206	216	216	219	701	0.1
162	.497	.270	.227	.540	.355	.437	.257	206	216	218	220	701	0.2
163	.498	.390	.108	.783	.357	.440	.072	205	216	223	224	700	0.3
164	.483	.068	.415	.145	.358	.435	.341	205	216	216	218	701	0.2
165	.498	.145	.353	.246	.361	.435	.330	206	216	216	219	700	0.2
166	.485	.167	.318	.331	.358	.429	.313	206	216	216	219	700	0.3
173	.511	.206	.305	.360	.357	.432	.308	205	216	216	219	699	2.4
174	.508	.386	.122	.755	.360	.430	.082	205	216	223	224	699	-2.0
175	.509	.286	.223	.550	.358	.440	.261	205	216	218	220	698	0.6
176	.498	.248	.250	.487	.361	.437	.274	206	216	217	220	699	2.1
177	.492	.313	.184	.614	.359	.444	.226	206	216	218	221	699	6.3
178	.499	.151	.348	.312	.355	.440	.320	206	216	216	219	699	4.6

Table B-4. Calculated Data from Runs with Benzene and Toluene

(a) Run Number	(b) % Flooding		(c) Material Balance, lb Benzene/min		(d) Number of	
	(1) Top	(2) Base	(1) Input	(2) Output	(1) Theoretical Plates	(2) Transfer Units
1 to 9	No usable information could be calculated from the operating data for these runs.					
10	44.3	52.3	1.37	1.30	1.39	2.92
11	70.9	82.9	1.30	1.33	1.62	3.22
12	55.3	71.0	1.31	1.39	1.47	3.44
13	38.7	55.5	1.38	1.37	1.50	3.96
20	78.2	81.6	2.91	3.04	1.57	3.34
21	49.6	45.5	2.98	3.03	0.93	2.38
22	63.0	64.5	2.91	3.03	1.31	3.06
29	57.0	79.3	0.38	0.39	0.70	1.25
30	57.1	84.1	0.38	0.33	0.46	0.61
31	29.6	65.8	0.39	0.40	0.78	1.86
38	44.4	76.6	0.40	0.38	1.25	2.90
39	55.8	82.8	0.38	0.39	1.71	5.31
40	61.4	89.1	0.37	0.35	1.00	2.61
47	50.9	78.2	0.40	0.40	1.52	5.50
48	57.4	86.3	0.40	0.41	1.73	4.84
49	72.4	92.7	0.40	0.41	0.92	1.80
53	45.0	57.5	1.34	1.31	1.48	3.92
54	64.5	71.9	1.33	1.29	1.26	2.79
55	73.8	90.7	1.32	1.37	1.34	2.72
59	20.6	19.5	3.01	2.96	1.58	3.68
60	26.0	27.6	2.89	2.94	1.70	3.79
61	32.2	35.7	2.94	3.01	1.86	3.98
62	41.0	36.0	2.90	3.09	1.44	2.41
66	44.3	46.2	3.05	3.04	1.76	2.94
67	67.9	51.9	2.93	2.90	2.37	2.62
68	83.8	30.5	3.01	3.05	4.84	3.82

Table B-4, Continued

(a)	(b)		(c)		(d)	
	(1)	(2)	(1)	(2)	(1)	(2)
75	49.7	46.9	1.31	1.33	3.59	5.06
76	58.7	58.0	1.30	1.25	3.28	4.09
77	68.6	64.7	1.32	1.35	5.05	5.72
81	52.7	57.8	3.00	2.91	1.91	2.69
82	64.5	60.0	3.01	3.12	2.92	3.79
86	43.4	44.9	3.08	3.02	1.17	1.61
87	59.9	47.1	2.87	2.88	2.19	2.21
88	62.7	57.3	2.97	2.99	2.81	2.86
92	50.2	51.8	2.99	3.01	1.81	3.38
93	62.1	62.0	2.95	2.98	1.88	3.05
94	74.0	72.1	2.88	2.95	2.40	3.34
98	76.3	87.8	1.31	1.36	3.42	5.08
99	67.0	73.7	1.30	1.35	1.95	3.20
100	57.7	61.0	1.30	1.32	1.66	2.86
104	64.3	75.4	0.33	0.34	1.00	2.29
105	53.8	70.0	0.35	0.34	1.00	2.06
106	41.6	59.1	0.34	0.34	0.94	2.22
110	62.0	64.0	0.34	0.35	1.00	2.15
111	73.4	76.0	0.35	0.35	1.18	2.31
112	81.3	82.2	0.34	0.35	1.29	2.24
119	55.1	56.1	0.34	0.35	1.71	2.82
120	66.0	66.8	0.35	0.35	2.33	3.22
121	76.9	77.6	0.34	0.34	4.00	4.39
122	71.2	68.1	0.34	0.35	1.96	3.06
123	60.3	61.3	0.34	0.34	1.55	2.39
124	47.5	49.6	0.34	0.34	1.06	1.92
128	78.7	85.7	1.35	1.39	1.54	2.97
129	64.7	68.9	1.26	1.29	1.00	2.27
130	52.4	51.2	1.25	1.27	0.96	2.37
137	52.8	49.6	1.28	1.27	1.84	2.98

Table B-4, Continued

(a)	(b)		(c)		(d)	
	(1)	(2)	(1)	(2)	(1)	(2)
138	68.1	61.8	1.30	1.30	2.72	3.62
139	78.4	78.2	1.28	1.27	4.59	5.02
140	50.8	45.2	1.31	1.34	2.87	5.54
141	61.0	54.1	1.31	1.35	3.46	5.50
142	67.0	69.8	1.29	1.29	3.86	4.87
150	67.2	57.9	1.30	1.32	2.47	3.58
151	78.0	77.0	1.32	1.34	4.56	6.13
152	48.9	53.0	1.33	1.35	1.30	3.11
153	63.8	65.5	1.29	1.33	1.53	3.26
154	80.1	78.6	1.27	1.33	1.47	2.97
158	81.7	82.8	2.94	2.95	5.11	5.51
159	67.3	65.5	2.95	2.93	2.19	2.90
160	53.8	50.8	3.00	3.11	1.60	2.68
167	82.2	76.8	2.93	3.06	1.22	2.31
168	69.6	68.7	2.96	3.03	1.29	2.59
169	55.3	51.8	2.99	3.05	1.73	4.43
170	60.4	61.8	2.90	2.99	2.21	3.11
171	75.4	77.1	2.93	2.84	3.45	1.45
172	51.2	46.1	3.10	3.24	1.21	2.13
179	81.1	77.1	2.98	3.00	3.42	4.10
180	64.0	58.6	2.93	3.02	1.78	2.83
181	47.9	45.2	2.97	2.99	1.08	1.98
149	52.4	46.1	1.37	1.35	2.13	3.77

Table B-5. Calculated Data from Runs with Isooctane and Toluene

(a) Run Number	(b) % Flooding		(c) Material Balance, lb Isooctane/min		(d) Number of	
	(1) Top	(2) Base	(1) Input	(2) Output	(1) Theoretical Plates	(2) Transfer Units
14	46.9	62.1	1.03	1.00	1.75	3.91
15	61.3	73.1	1.03	1.04	1.92	4.60
16	74.3	84.4	1.03	1.05	1.58	3.12
23	50.4	47.5	2.42	2.37	1.42	3.13
24	63.7	63.1	2.36	2.37	1.68	5.22
25	76.1	78.0	2.30	2.31	2.00	9.11
32	24.7	71.8	0.31	0.30	-- *	1.17
33	43.8	83.4	0.32	0.32	1.88	3.47
34	52.3	96.1	0.32	0.32	1.24	2.98
41	35.2	83.3	0.32	0.32	-- *	2.20
42	48.4	91.9	0.32	0.33	1.23	4.44
43	53.4	94.9	0.31	0.31	1.00	2.45
50	40.9	83.5	0.33	0.33	-- *	2.90
51	47.5	92.4	0.33	0.34	1.00	2.79
52	58.7	99.7	0.40	0.41	1.00	2.82
56	43.4	56.7	1.10	1.11	1.63	2.46
57	59.6	70.5	1.09	1.11	1.10	3.45
58	71.4	82.6	1.11	1.11	1.20	2.46
63	19.7	21.7	2.46	2.73	0.86	2.24
64	28.2	28.7	2.49	2.48	0.97	1.83
65	35.0	36.7	2.49	2.47	1.54	2.48
69	58.0	48.3	2.50	2.51	1.90	2.73
70	67.9	52.0	2.46	2.51	2.44	3.23
71	79.2	55.7	2.47	2.48	3.90	4.40
78	55.7	47.8	1.07	1.09	2.68	4.33
79	66.2	58.9	1.10	1.10	3.36	4.65
80	77.8	62.6	1.05	1.06	6.64	7.23
83	83.8	62.6	2.49	2.49	6.52	6.42

Table B-5, Continued

(a)	(b)		(c)		(d)	
	(1)	(2)	(1)	(2)	(1)	(2)
84	72.7	60.0	2.47	2.45	3.37	4.08
85	56.7	52.0	2.45	2.45	1.82	2.64
89	61.3	59.4	2.44	2.45	3.68	4.23
90	52.2	52.0	2.44	2.44	2.84	3.75
91	42.1	46.0	2.45	2.41	2.00	3.00
95	79.2	75.5	2.48	2.49	2.45	3.50
96	68.2	63.7	2.50	2.49	1.62	2.41
97	57.5	51.0	2.51	2.52	1.19	1.95
101	59.0	62.1	1.11	1.11	1.77	2.88
102	69.3	74.3	1.11	1.12	2.15	3.28
103	79.8	85.6	1.09	1.08	2.82	3.97
107	49.9	83.4	0.29	0.29	1.42	3.49
108	63.8	91.8	0.29	0.29	1.00	2.32
109	64.7	96.6	0.29	0.29	1.52	2.96
113	42.4	65.9	0.28	0.28	0.92	2.28
114	69.0	76.5	0.28	0.28	1.19	2.24
115	80.8	82.2	0.29	0.29	1.21	2.07
116	49.7	56.2	0.28	0.28	1.64	2.73
117	66.3	69.4	0.28	0.29	2.36	3.60
118	83.1	82.2	0.28	0.29	3.67	4.64
125	75.8	73.6	0.29	0.29	2.35	3.38
126	60.7	63.8	0.28	0.28	1.74	2.81
127	48.8	53.4	0.28	0.29	1.35	2.46
131	79.4	82.2	1.06	1.06	1.66	2.95
132	69.8	71.0	1.05	1.06	1.43	2.69
133	55.0	60.7	1.07	1.08	1.00	2.40
134	88.1	75.5	1.07	1.08	5.90	6.82
135	72.1	63.9	1.10	1.10	3.52	4.94
136	56.2	50.8	1.10	1.10	1.92	3.30
143	64.2	54.3	1.13	1.13	2.33	3.45

Table B-5, Continued

(a)	(b)		(c)		(d)	
	(1)	(2)	(1)	(2)	(1)	(2)
144	71.9	69.1	1.10	1.10	3.30	4.36
145	83.8	77.5	1.07	1.09	5.82	6.60
146	61.4	52.9	1.07	1.08	2.00	3.42
147	73.1	65.8	1.09	1.09	2.44	3.72
148	84.4	81.7	1.08	1.08	3.78	5.07
155	75.8	82.6	1.10	1.10	1.29	2.24
156	63.5	69.0	1.09	1.09	1.00	2.23
157	46.0	53.9	1.07	1.07	0.92	2.17
161	51.0	53.2	2.43	2.44	1.28	2.29
162	67.8	66.5	2.43	2.41	2.70	3.69
163	90.0	86.1	2.44	2.43	6.92	7.91
164	47.4	48.6	2.38	2.35	0.91	1.80
165	76.1	64.2	2.47	2.46	1.09	1.92
166	82.3	78.5	2.38	2.35	1.33	2.03
173	52.2	44.9	2.51	2.51	1.40	2.67
174	82.8	77.6	2.51	2.39	5.79	6.20
175	65.6	62.2	2.50	2.51	2.60	3.70
176	70.0	67.0	2.47	2.42	2.08	2.99
177	83.0	76.2	2.45	2.46	3.47	4.68
178	50.1	51.5	2.43	2.44	1.62	2.65

* It was not possible to calculate the number of theoretical plates for these runs.

Table B-6. Data from Runs with Heptane and Methylcyclohexane

Run Number	Identifications		Flow Rate, lb/sec-ft ²			Mole Frac. Heptane			Temperature °F			
	Packing	Column	Feed	OH	Btm	Feed	OH	Btm	Feed	OH	BLIQ	BVAP
17	1/2 inch	4"	0.503	0.038	0.460	0.507	0.524	0.480	198	206	206	206
18	Raschig		.503	.083	.436	.491	.517	.488	198	206	206	206
19	Rings		.503	.138	.360	.500	.520	.498	202	206	206	206
26		6"	.498	.065	.433	.500	.517	.487	200	206	206	206
27			.498	.109	.389	.506	.518	.503	203	206	206	206
28			.498	.141	.357	.501	.517	.488	199	206	206	206
35		2"	.514	.086	.457	.495	.505	.478	204	206	206	206
36			.521	.079	.450	.500	.528	.485	203	206	206	206
37			.514	.114	.365	.501	.511	.496	202	206	206	206
44	1/4 inch	2"	.514	.064	.486	.506	.533	.506	205	206	206	206
45	Intalox		.514	.079	.450	.506	.527	.503	205	206	206	206
46	Saddles		.514	.114	.400	.506	.525	.503	205	206	206	206

Run Number	V/L @ Base	Ambient Pressure (mm Hg)	Pressure Drop (" H ₂ O)	% Flooding		Material Balance, lb Heptane/min	
				Top	Base	Input	Output
17	0.135	706	0.6	38.2	56.7	1.35	1.28
18	.215	706	1.4	60.4	77.9	1.31	1.36
19	.281	706	2.8	82.3	83.0	1.34	1.34
26	.118	701	1.0	52.6	48.5	3.00	2.95
27	.202	701	2.2	71.0	66.3	3.04	3.04
28	.300	702	5.3	83.3	88.0	3.01	2.98
35	.190	699	1.2	61.8	74.2	0.36	0.37
36	.255	699	12.0	60.1	93.5	0.36	0.36
37	.295	699	9.0	73.1	88.2	0.36	0.33
44	.196	700	0.9	58.0	90.3	0.37	0.40
45	.226	700	2.2	66.3	94.0	0.37	0.38
46	.276	699	13.9	82.7	100.8	0.37	0.37

APPENDIX C

ENTHALPY-COMPOSITION DIAGRAMS

The enthalpy-composition diagrams used in the calculation of the number of theoretical plates by the Ponchon-Savarit method were obtained by substituting appropriate values in Equations 11 and 12.

$$h = [xC_{La}M_a + (1-x)C_{Lb}M_b](T_L - T_R) + \Delta H_{SOL} \quad (11)$$

$$H = y[C_{La}M_a(T_V - T_R) + \lambda_a M_a] + (1-y)[C_{Lb}M_b(T_V - T_R) + \lambda_b M_b] \quad (12)$$

The data used in calculating the phase enthalpies from these equations is discussed below.

Temperature-Composition Data

The temperature-composition data which has been presented graphically in Chapter 3 was reduced from 760 mm Hg to 700 mm Hg using the method of Othmer and Gilmont (26). This method plots the log of the system pressure versus the vapor phase concentration with liquid phase concentration as a parameter for a family of curves. The corrections obtained by this method resulted in a decrease in phase temperature of 5F° which was relatively constant.

Heat Capacity Data

The heat capacity of the pure liquid components as a function of temperature were obtained from Perry's Handbook (27) and API Project

Number 44 (31). In the calculation of a phase enthalpy at a particular temperature, the mean value of the heat capacity between the phase temperature and the reference temperature was used. Therefore, a slight effect of temperature is noticed in the heat capacities. No data could be found relating liquid heat capacity as a function of liquid phase concentration. This data would have had the effect of adding another degree of non-ideality to the liquid phase enthalpy calculation.

Latent Heat of Vaporization Data

The latent heat of vaporization as a function of temperature for each pure component was obtained from API Project Number 44 (31). The value of λ at the temperature of the vapor phase (dew point) was then used to calculate the vapor phase enthalpy.

Integral Heat of Solution Data

The integral heat of solution as a function of composition was available only for the benzene-toluene mixture. The data was found in the International Critical Tables (40). An extensive literature search found that very little integral heat of solution data for any hydrocarbon pairs existed in any reference but the above and that none was available for the other mixtures in this study. The data for Mixture I at 16°C is as follows:

<u>Mole Fraction Benzene</u>	<u>ΔH_{SOL}, BTU/lb mole</u>
0.0	0
0.1	-144
0.2	-260
0.3	-356
0.4	-394
0.5	-401
0.6	-396
0.7	-334
0.8	-223
0.9	-113
1.0	0

Presentation of Enthalpy-Composition Data

The values of the phase enthalpies calculated from Equations 11 and 12 are presented in the tables and figures which follow. The information is presented first in tabular form and then graphically for each chemical mixture.

Table C-1. Calculated Enthalpy-Composition Data for Benzene and Toluene.
The system pressure is 700 mm Hg abs.

Mole Fraction Benzene in the Phase	Bubble Point °F	Liquid Phase Enthalpy BTU/lb mole	Dew Point °F	Vapor Phase Enthalpy BTU/lb mole
0.0	225.8	6,820	225.8	21,250
0.1	217.6	6,340	-	-
0.2	210.4	5,650	218.2	20,410
0.3	203.8	5,510	-	-
0.4	198.0	4,780	209.5	19,550
0.5	192.7	4,470	-	-
0.6	187.7	4,200	199.5	18,760
0.7	183.0	3,960	-	-
0.8	178.7	3,860	187.2	17,910
0.9	174.7	3,740	-	-
1.0	171.1	3,650	171.1	16,950

The reference temperature at which the enthalpies of the pure liquids are equal to zero is 16°C.

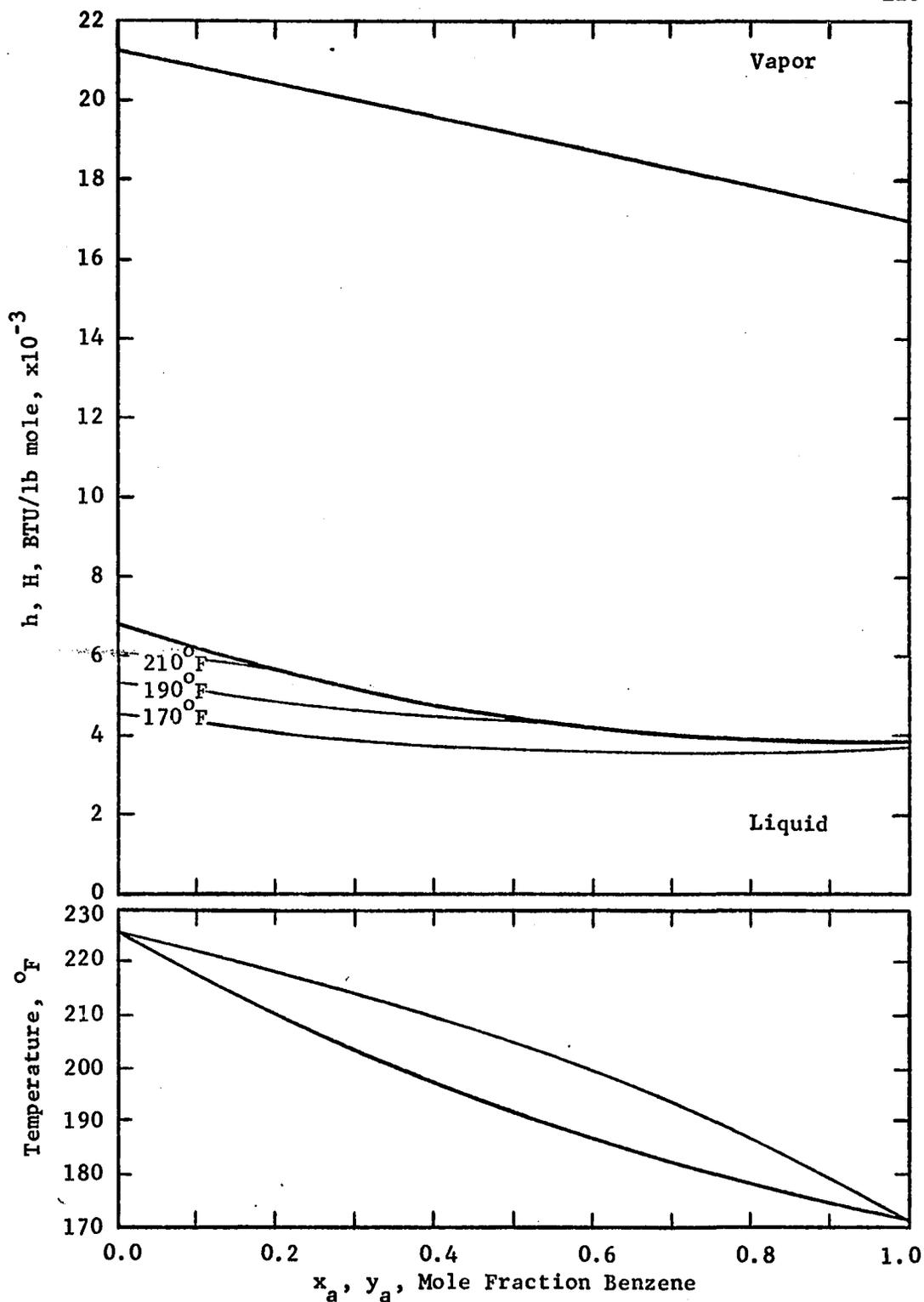


Figure C-1. Enthalpy-Composition Diagram for Benzene and Toluene at 700 mm Hg abs. $T_R = 16^\circ\text{C}$.

Table C-2. Calculated Enthalpy-Composition Data for Isooctane and Toluene.
The system pressure is 700 mm Hg abs.

Mole Fraction Isooctane in the Phase	Bubble Point °F	Liquid Phase Enthalpy BTU/lb mole	Dew Point °F	Vapor Phase Enthalpy BTU/lb mole
0.0	225.8	5,320	225.8	19,880
0.1	223.4	5,530	222.3	19,930
0.2	219.2	5,650	221.6	20,140
0.3	216.5	5,820	-	-
0.4	214.3	6,000	216.6	20,280
0.5	212.3	6,180	-	-
0.6	210.7	6,370	211.8	20,420
0.7	209.2	6,550	-	-
0.8	207.8	6,720	208.3	20,560
0.9	206.6	6,910	-	-
1.0	205.4	7,110	205.4	20,700

The reference temperature at which the enthalpies of the pure liquids are equal to zero is 100°F.

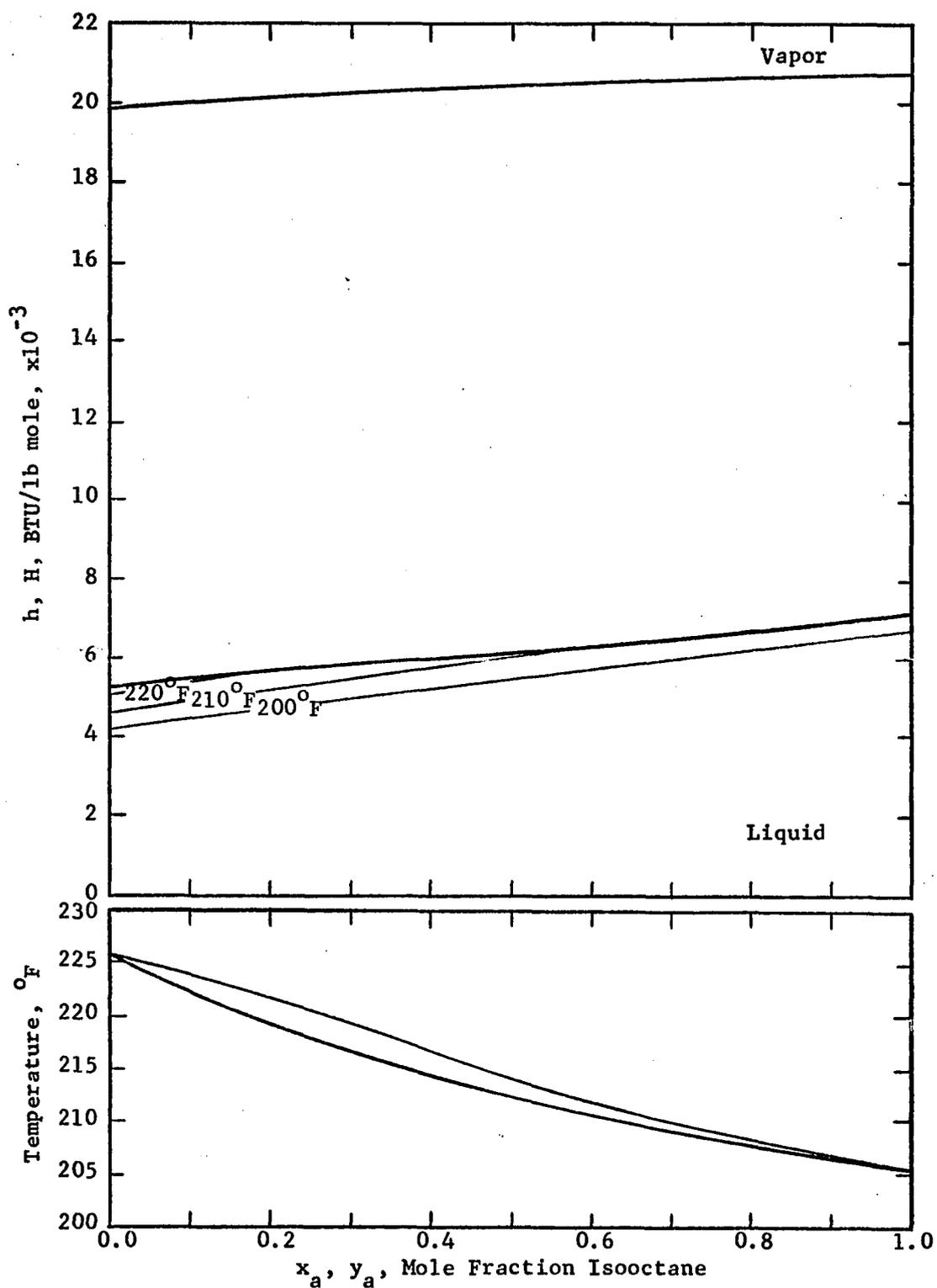


Figure C-2. Enthalpy-Composition Diagram for Isooctane and Toluene at 700 mm Hg abs. $T_R = 100^\circ\text{F}$.

Table C-3. Calculated Enthalpy-Composition Data for Heptane and Methylcyclohexane.
The system pressure is 700 mm Hg abs.

Mole Fraction Heptane in the Phase	Bubble Point °F	Liquid Phase Enthalpy BTU/lb mole	Dew Point °F	Vapor Phase Enthalpy BTU/lb mole
0.0	208.2	5,260	208.2	19,780
0.2	207.2	5,880	207.3	19,860
0.4	206.4	5,990	206.5	19,930
0.6	205.5	6,080	205.6	20,030
0.8	204.7	6,220	204.8	20,120
1.0	203.9	6,340	203.9	20,160

The reference temperature at which the enthalpies of the pure liquids are equal to zero is 100°F.

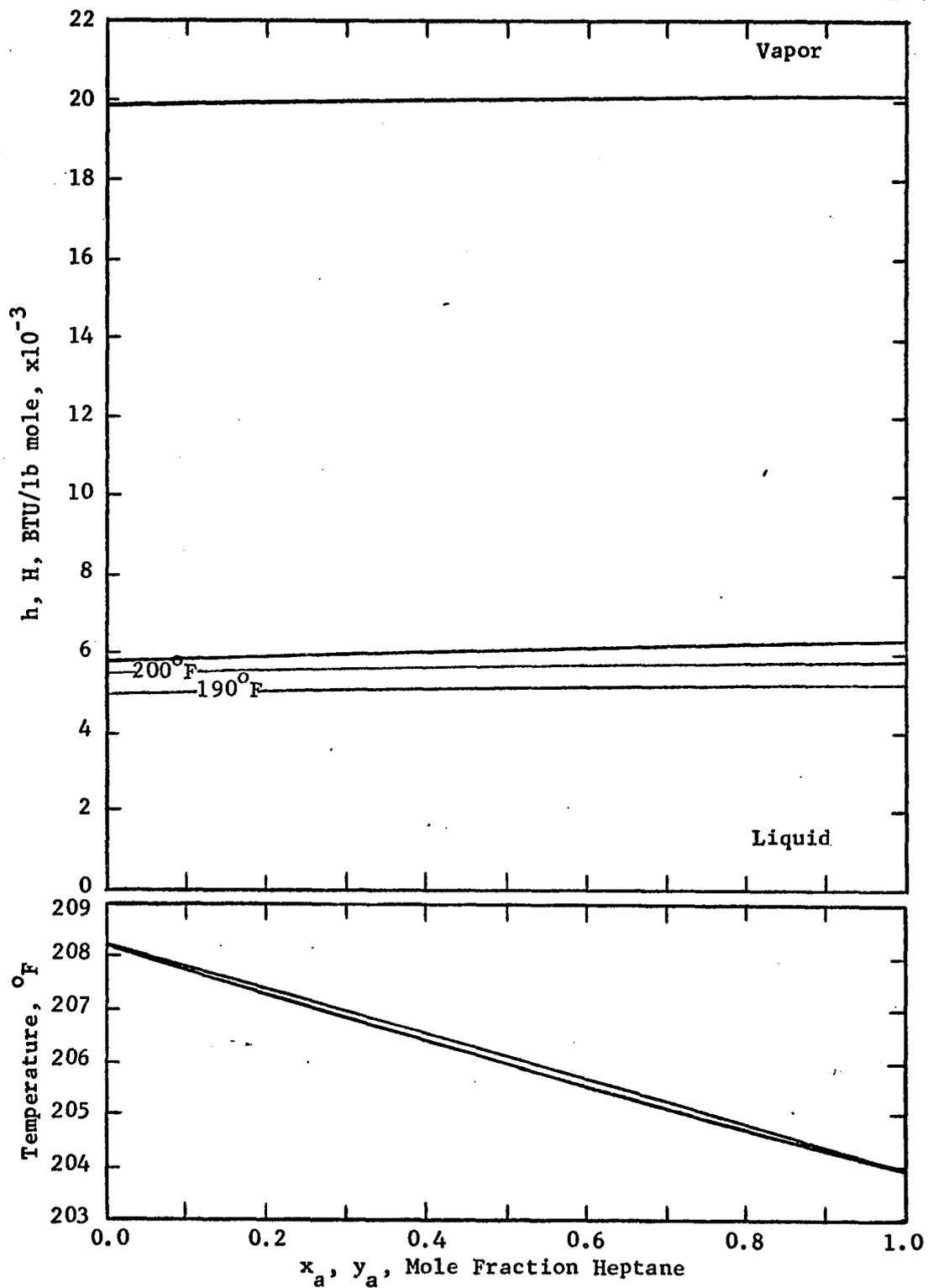


Figure C-3. Enthalpy-Composition Diagram for Heptane and MCH at 700 mm Hg abs. $T_R = 100^\circ\text{F}$.

APPENDIX D

GENERALIZED PRESSURE DROP CORRELATION

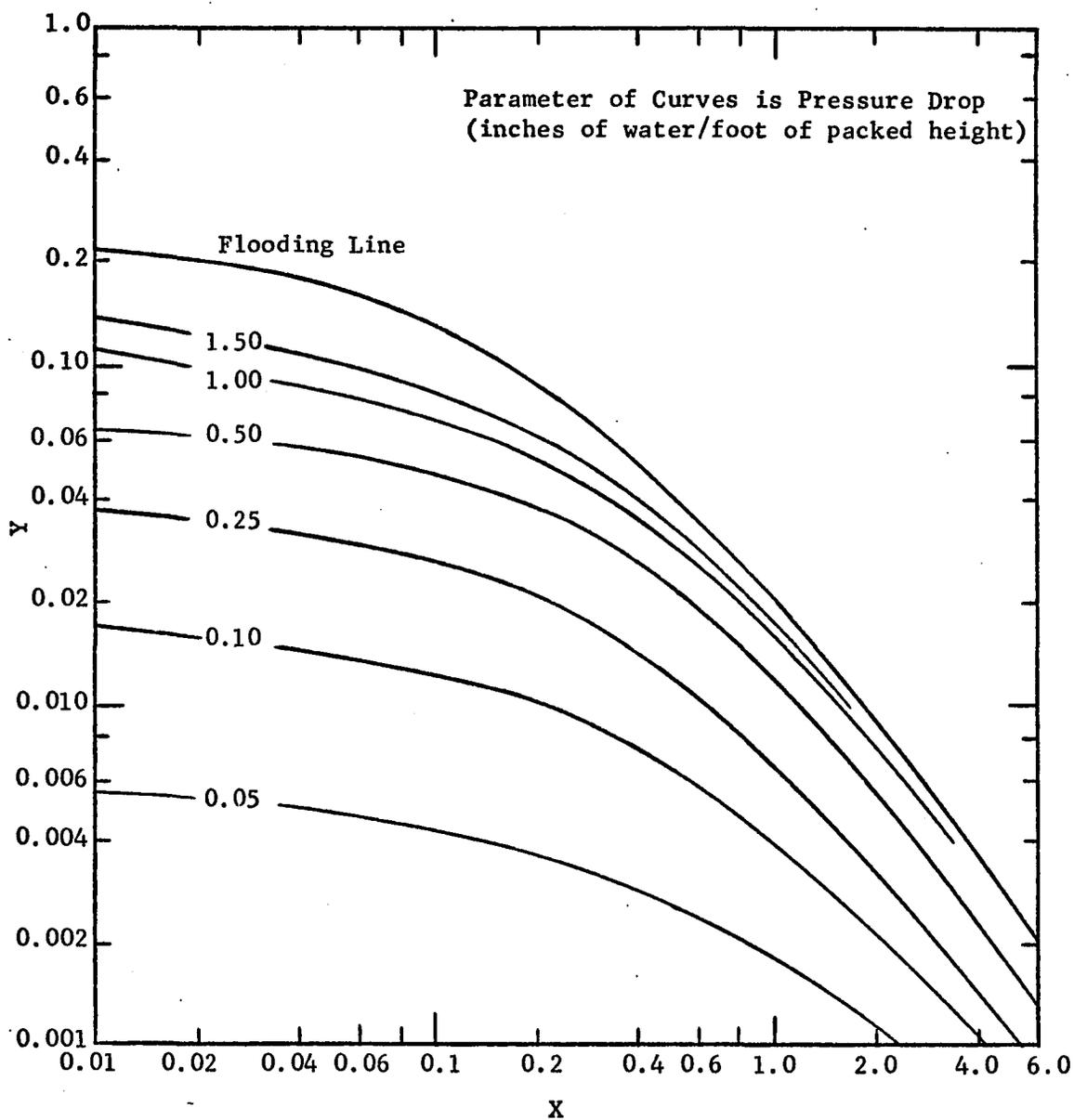
The data of Eckert et al. (9) is presented in a figure which has been redrawn as Figure D-1. The curve labeled 'flooding line' was fit and the following polynomial equation was obtained:

$$Y = 3.188(10^{-3}) + \frac{2.257(10^{-2})}{X} - \frac{1.179(10^{-3})}{X^2} + \frac{2.451(10^{-5})}{X^3}$$

When the physical properties of the two fluid phases are known, it is possible to calculate the vapor flooding rate for a given liquid rate. It should be noted that mass flow rates are used rather than molar flow rates as indicated in the Nomenclature.

The empirically determined packing factors for the packings used in this study are presented in the following table and are obtained from the above source.

<u>Packing</u>	<u>Packing Factor</u>
Intalox Saddles	
1/4 inch	725
1/2 inch	200
3/4 inch	145
1 inch	98
Berl Saddles	
1/2 inch	240
3/4 inch	170
Raschig Rings	
1/2 inch	580
3/4 inch	255



$$X = \frac{L}{V} \left(\frac{\rho_V}{\rho_L} \right)^{\frac{1}{2}}$$

$$Y = \frac{(V/S)^2 P_{\psi L}^{0.2}}{g_c \rho_V \rho_L}$$

Figure D-1. Generalized Pressure Drop Correlation

LIST OF REFERENCES CITED

1. Bromley, E. C., and D. Quiggle, Ind. Eng. Chem., 25, 1137 (1933).
2. Chilton, T. H., and A. P. Colburn, Ind. Eng. Chem., 27, 255 (1935).
3. Clay, H. A., J. W. Clark, and B. L. Munro, Chem. Eng. Progr., 62, No. 1, 51 (1966).
4. Cornell, D., W. G. Knapp, H. J. Close, and J. R. Fair, Chem. Eng. Progr., 56, Part I-No. 7, 68 (1960), Part II-No. 8, 49 (1960).
5. Eckert, J. S., Chem. Eng. Progr., 57, No. 9, 54 (1961).
6. Eckert, J. S., Chem. Eng. Progr., 59, No. 5, 76 (1963).
7. Eckert, J. S., Chem. Eng. Progr., 66, No. 3, 39 (1970).
8. Eckert, J. S., and L. F. Walter, Hydrocarbon Proc., 43, No. 2, 107 (1964).
9. Eckert, J. S., E. H. Foote, and L. F. Walter, Chem. Eng. Progr., 62, No. 1, 59 (1966).
10. Efrogymson, M. A., "Mathematical Methods for Digital Computers," John Wiley and Sons, Inc., New York (1960).
11. Ellis, S. R. M., Chem. Eng. News, 31, 4613 (1953).
12. Fair, J. R., Chemical Engineering, 72, No. 14, 107 (1965).
13. Fair, J. R., Chem. Eng. Progr., 66, No. 3, 45 (1970).
14. Hands, C. H. G., and F. R. Whitt, J. of App. Chem., 1, 135 (1951).
15. Hughes, H. E., and J. O. Maloney, Chem. Eng. Progr., 48, No. 2, 195 (1952).
16. Jordan, D. G., "Chemical Process Development," Volume II, Interscience Publishers, New York (1968).
17. Leva, Max, "Tower Packing and Packed Tower Design," The U. S. Stoneware Company, Akron (1953).
18. Lobo, W. E., E. L. Friend, F. Hashmall, and F. A. Zenz, Trans. of A. I. Ch. E., 41, 693 (1945).

19. Lockhart, F. J., Professor, Dept. of Chem. Eng., University of Southern California, personal communication, (1970).
20. Morris, G. A., "Proceedings of the International Symposium on Distillation, 1959," London, 146 (1960).
21. Morris, G. A., and J. Jackson, "Absorption Towers," Butterworths Scientific Publications, London (1953).
22. Murch, D. P., Ind. Eng. Chem., 45, 2616 (1953).
23. Neville, A. M., and J. B. Kennedy, "Basic Statistical Methods for Engineers and Scientists," International Textbook Company, Scranton (1968).
24. Novinski, N. L., "Molar Response Factors by Gas-Liquid Chromatography," Chemical Engineering 299 Report, The University of Arizona, Tucson (1969).
25. Otake, T., and K. Okada, Chem. Eng. (Japan), 22, 176 (1953).
26. Othmer, D. F., and R. Gilmont, Ind. Eng. Chem., 36, 858 (1944).
27. Perry, J. H., ed., "Chemical Engineers' Handbook," Third Edition, McGraw-Hill Book Company, New York (1950).
28. Peters, W. A., Jr., Ind. Eng. Chem., 14, 476 (1922).
29. Porter, K. E., and J. J. Templeman, Trans. Inst. Chem. Eng. (London), 46, T86 (1968).
30. Prah1, W. H., Chemical Engineering, 76, No. 17, 89 (1969).
31. Rossini, F. D., ed., "Selected Values of Physical and Thermodynamic Properties of Hydrocarbons and Related Compounds," American Petroleum Institute Research Project Number 44, Carnegie Institute of Technology, Pittsburg (1953).
32. Sherwood, T. K., G. H. Shipley, and F. A. L. Holloway, Ind. Eng. Chem., 30, 765 (1938).
33. Silvey, F. C., and G. J. Keller, Chem. Eng. Progr., 62, No. 1, 68 (1966).
34. Simpson, K. O., Graduate Student, Dept. of Chem. Eng., University of Arizona, personal communication, (1970).
35. Thornton, J. D., and F. H. Garner, J. of App. Chem., Supplement Number 1, S75 (1951).
36. Todd, Floyd, Ind. Eng. Chem., 32, 287 (1940).

37. Treybal, R. E., "Mass Transfer Operations," Second Edition, McGraw-Hill Book Company, New York (1967).
38. Van Winkle, M., "Distillation," McGraw-Hill Book Company, New York (1967).
39. Vivian, J. E., P. L. T. Brian, and V. J. Krukonis, A. I. Ch. E. J., 13, 174 (1967).
40. Washburn, E. W., ed., "International Critical Tables of Numerical Data, Physics, Chemistry and Technology," McGraw-Hill Book Company, New York, Volume 5, 159 (1929).
41. Yoshida, F., and T. Koyanagi, A. I. Ch. E. J., 8, 309 (1962).