THE UNIVERSITY OF MICHIGAN

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MASS TRANSFER BETWEEN ISOBUTANOL AND WATER IN CONCURRENT FLOW THROUGH A PACKED COLUMN

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NOMENCLATURE

a - interfacial area for mass transfer, area per unit column volume

$C_{\alpha\beta}$ - molal concentration of component α in the β phase, moles per unit volume

$C^{*}_{\alpha\beta}$ - molal concentration of α in the saturated β phase, moles per unit volume

$C_{\alpha\beta_i}$ - molal concentration of α at the interface of the β phase, moles per unit volume

H - total height of packing, unit length

$k_{\alpha\beta}$ - local transfer coefficient for the component α in the β phase, unit length per time

$k'_{\alpha\beta}$ - local transfer coefficient for the component α in the β phase, reciprocal time

$\bar{k}'_{\alpha\beta}$ - mean transfer coefficient for the component α in the β phase, reciprocal time

L - moles of water-rich phase flowing, moles per time

L_o - inlet moles of water-rich phase, moles per time

L' - mass of water-rich phase flowing, mass per time

N_α - number of moles of component α transferring per unit time, per unit area

S - cross-section of empty column, area

V - moles of isobutanol-rich phase flowing, moles per time

V_o - inlet flow of the isobutanol-rich phase, moles per time

V' - mass of isobutanol-rich phase flowing, mass per time

V_R - unit volume of empty column

x_α - mole fraction of component α in the L phase

X_α - mass fraction of component α in the L phase

y_α - mole fraction of component α in the V phase

Y_α - mass fraction of component α in the V phase

Z - internal height in the column, length

Subscripts

A - denotes isobutanol component

f - denotes a final condition

L - denotes the water-rich phase

o - denotes an initial condition

V - denotes the isobutanol-rich phase

W - denotes water component

I. INTRODUCTION

Industrial liquid-liquid extraction is ordinarily carried out in countercurrent packed or spray columns or in equipment of the mixer-settler type. Countercurrent columns allow continuous contacting of the liquid phases but the throughput of this type of column is seriously limited by the low flow rates obtainable using fluid density as the driving force. Mixer-settler equipment is bulky and expensive and is inefficient unless care is taken in the mixing and settling.

In this study, mass transfer between two liquid phases is investigated with the two phases flowing concurrently through a packed bed. The packed bed serves the purpose of dispersing the two phases, thus creating a very large interfacial area for mass transfer. Since the flow rates of the two phases are restricted only by the allowable pressure drop across the bed, large mass rates per unit cross sectional area can be handled. The phases leaving the column can be expected to approach equilibrium compositions as the length of the column is increased, i.e., the process becomes equivalent to a single equilibrium stage. A multistage operation can be simulated by a number of concurrent units operated in series, with the flow of extract and raffinate being countercurrent from unit to unit.

Most commercial applications of liquid extraction involve two relatively immiscible solvents and a distributed solute. The mass transfer process then involves three components, two transferring in one direction and the third in the opposite direction. Colburn and Welsh[1] suggested for fundamental investigations of mass transfer between liquid phases that

the solute be eliminated and transfer limited to two relatively immiscible solvents into one another. When the system is thus restricted, evaluation of the individual phase mass transfer coefficients is possible.

This scheme has been used in numerous studies of mass transfer coefficients in countercurrent packed and spray columns and other special types of contacting equipment. Investigations of two component liquid systems in countercurrent packed columns include that of Colburn and Welsh with isobutanol-water; Laddha and Smith[5] with isobutyraldehyde-water and 3 pentanol-water: Gayler and Pratt[2] with ethyl-acetate-water; Smith and Beckman[8] with methyl isobutyl carbinol-water and methyl ethyl ketone-water. The isobutanol-water system was utilized by Ruby and Elgin[7] and Heertjes, Holve and Talsma[4] in investigating transfer in countercurrent spray columns and by Gordon and Sherwood[3] and Lewis[6] in special transfer cells. No mention has been found in the literature of any investigations of mass transfer, in liquid systems, in a packed bed with concurrent flow, with either two or three components.

The isobutanol-water system was chosen for this investigation because of the prior work with other types of flow and because of experimental and analytical convenience. Transfer was investigated as a function of bed height and individual phase flow rates, in upward flow through a column packed with glass beads. Only one column diameter ($\frac{1}{2}$ inch) and one bead size (3 mm) were utilized.

II. RATIONALIZATION FOR THE CORRELATION OF DATA

The steady state transfer of a component α between two liquid phases is generally taken to be proportional to a transfer coefficient times the concentration difference of α between the bulk and the interface of either of the phases,

$$N_\alpha = k_{\alpha V}(C_{\alpha V} - C_{\alpha V_i}) = k_{\alpha L}(C_{\alpha L_i} - C_{\alpha L}) \qquad (1)$$

where N_α = the rate of transfer of α, moles per unit time per unit area for transfer,

$C_{\alpha L}$, $C_{\alpha V}$ = the bulk concentration of α in the L and V phases, moles per unit volume.

$C_{\alpha L_i}$, $C_{\alpha V_i}$ = the concentration of α at the interface of the L and V phases, moles per unit volume,

$k_{\alpha L}$, $k_{\alpha V}$ = the individual phase mass transfer coefficients of the component α in the L and V phases, unit length per unit time.

Interfacial concentrations are difficult if not impossible to measure. Therefore, the interface is assumed to be in static equilibrium,

$$C_{\alpha V_i} = mC_{\alpha L_i} \qquad (2)$$

where m is usually some function of temperature, pressure and concentration. If the system under consideration is only two component, Equation (2) is completely determined at any given temperature and pressure. These concentrations will be those of the mutually saturated phases, $C^*_{\alpha V}$ and $C^*_{\alpha L}$.

For the system isobutanol-water, the equation for transfer, Equation (1), can be written (where L becomes the water-rich phase and V the isobutanol-rich phase) for water transferring,

$$N_W = k_{WV}(C^*_{WV}-C_{WV}) = -k_{WL}(C^*_{WL}-C_{WL}) \qquad (3)$$

and for isobutanol transferring,

$$-N_A = k_{AV}(C^*_{AV}-C_{AV}) = -k_{AL}(C^*_{AL}-C_{AL}) \qquad (4)$$

The rate of transfer of the component α can be equated to the rate of longitudinal transport of component α in the two phases,

$$N_\alpha = \frac{d(Lx_\alpha)}{adV_R} = \frac{-d(Vy_\alpha)}{adV_R} \qquad (5)$$

where L , V = the number of moles flowing in the L and V phases per unit time,

x_α, y_α = the mole fraction of α in the L and V phases,

a = the interfacial area for mass transfer per unit column volume,

V_R = unit volume of the column without packing.

For example, the rate of transfer of water into the isobutanol-rich phase can be written,

$$\frac{d(Vy_W)}{aSdZ} = k_{WV}(C^*_{WV}-C_{WV}) \qquad (6)$$

where V = the number of moles flowing in the isobutanol-rich phase per unit time,

SdZ = the unit volume of the column,

S = the cross section of the empty column,

Z = the unit height of column,

C^*_{WV} = the static equilibrium concentration of water in isobutanol.

The interfacial area for mass transfer between the liquid phases in a packed column is extremely difficult to measure directly or even to estimate. However, it can be grouped and measured in combination with the phase transfer coefficient as $(k_{\alpha\beta}a)$, i.e., as a volumetric coefficient $k'_{\alpha\beta}$. The final form of the transfer equation to be used to represent the data is then, for example, for water transferring,

$$\frac{d(Vy_w)}{SdZ} = k'_{WV}(C^*_{WV}-C_{WV}) \quad . \qquad (7)$$

From an overall and a component material balance around the column, an expression for L or V can be derived. Since

$$dL = -dV$$

or

$$(Vy_\alpha)_o - Vy_\alpha = Lx_\alpha - (Lx_\alpha)_o$$

and

$$L + V = L_o + V_o$$

there results

$$V = \frac{(Vy_\alpha)_o + (Lx_\alpha)_o - x_\alpha(L_o + V_o)}{(y_\alpha - x_\alpha)} \quad . \qquad (8)$$

From Equation (8), it is seen that once the x_α and y_α are known as a function of height in the column, Equation (7) is completely determined, and the phase transfer coefficients $k'_{\alpha\beta}$ can be computed as a function of height.

III. EXPERIMENTAL PROGRAM

A. Introduction

As implied in the preceding section, the experimental problem is to determine phase concentrations as a function of height in the packed column. Internal sampling or analysis of the phases as they rise in the column would be the preferred solution of the problem, but were ruled out as experimentally unfeasible. Instead, the outlet phase streams of a series of columns of different packed heights were sampled and analysed. Sampling the outlet streams is in itself a difficult problem due to the necessity of separating them. The separation was accomplished with a receiver-settler placed on the top of the column. This posed the problem of estimating the amount of transfer which takes place in the outlet tube and settler.

The experimental work then took on two aspects: (1) Determining the receiver outlet concentrations with a series of columns of different packed heights as a function of the individual phase flow rates, and (2) estimating the mass transfer which takes place in the outlet tube and receiver as a function of the phase flow rates and the receiver outlet concentrations. This latter transfer will be called the receiver "end effect."

What follows treats in detail the first aspect, determining receiver outlet concentrations for the various column lengths. The end effect is treated separately in a subsequent section.

B. Experimental Equipment

The equipment used to carry out the determination of the receiver outlet phase concentrations of the various column lengths is shown in a schematic diagram in Figure 1, the main fixtures of which are (1) the feed reservoirs, (2) the proportioning pumps for the feed streams, (3) the column inlet packing gland, (4) the glass column with packing, (5) the receiver-settler.

The feed reservoirs were four foot long sections of 100 mm diameter Pyrex glass tubing, dished at the bottom and fitted with a glass ball joint. A side arm riser of 10 mm diameter glass tubing was attached to this to facilitate reading the liquid level in the larger tubes. Behind the side arm riser was placed a meter stick, and flow rates were determined by reading the height of the liquid level in the riser before the pumps were turned on and after they were turned off.

The pumps used to move the isobutanol and water to the column were Milton Roy proportioning pumps of approximately 5.5 gals/hr capacity. They were of stainless steel construction. A brass ball and spring check valve was fitted into the pump outlets to neutralize the head of liquid in the reservoirs so that the feed came through the pump only on the forward stroke of the plunger. The feed pulses were then successfully dampened by placing 1000 ml Pyrex round-bottom flasks in the outlet lines as partially air-filled surge vessels.

The column inlet packing gland was turned out of aluminum bar stock. As indicated in Figure 2, the alcohol and water feed lines led

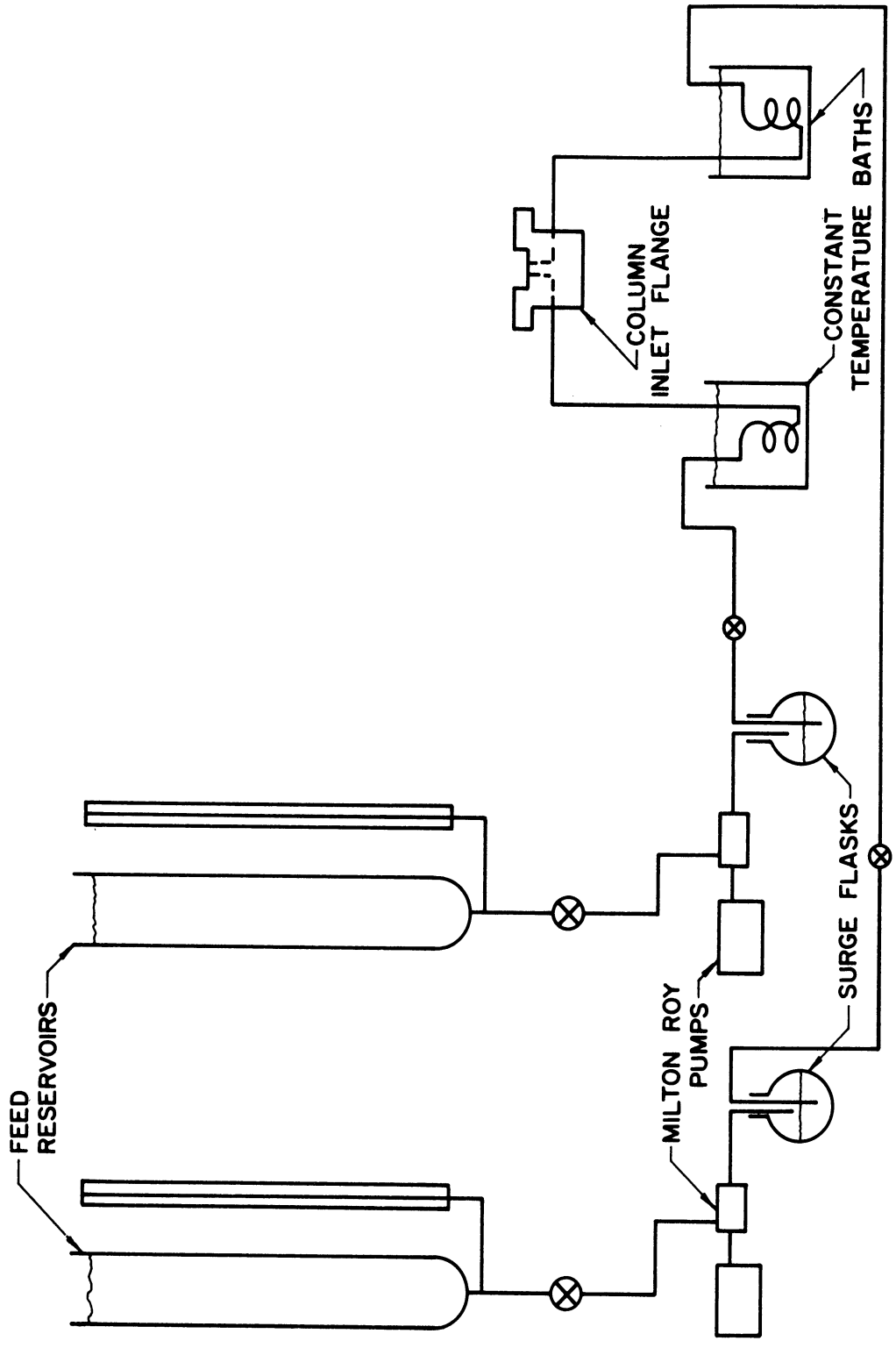


Figure 1. Schematic Flow Diagram of Equipment.

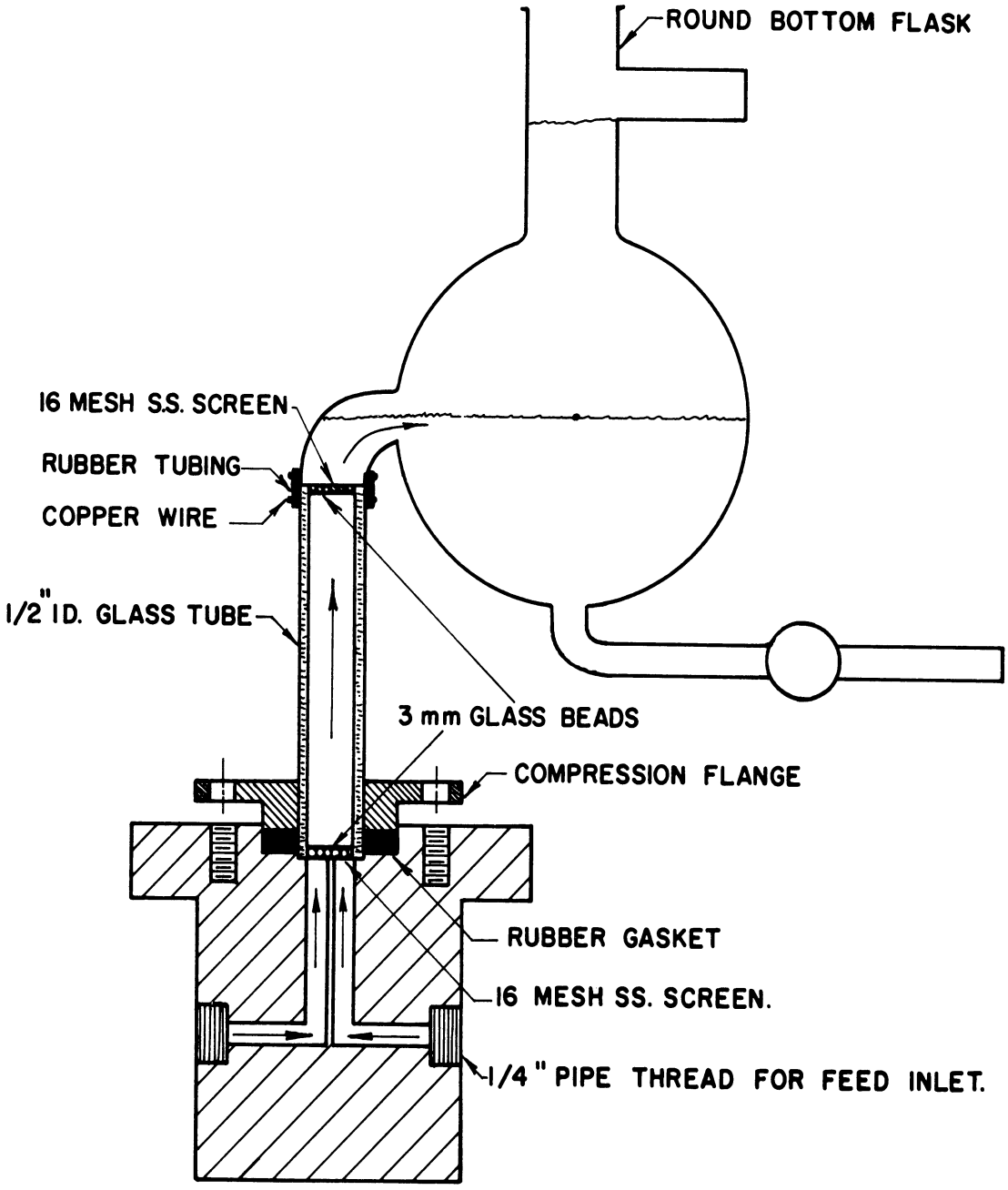


Figure 2. Column Inlet Flange and Column and Receiver Assembly.

separately to either side of the base of the flange, went as far into the base as the middle and then, still separately, made a right angle turn to go up through the remainder of the flange to the bottom of the column. The flange and column connection was made pressure tight by a bolted compression fitting with a hard rubber gasket 2" O.D x 3/4" I.D. x 1/4".

The column was 1/2" I.D. Pyrex gauge glass cut to the desired height. To keep the packing in place while handling the column, the bottom of the column was closed with a piece of 16 gauge stainless steel screen, the screen being held to the glass by a black, de Kotinsky-type cement which was insoluble in isobutanol. The packing was made up of fairly uniform 3 mm glass spheres.

Of the various receivers that were tried, a round bottom flask with a short hooked side arm at its equator, as shown in Figure 2, was found to be the most satisfactory. Two different sizes, a 200 ml and a 500 ml, were used. The level of the phases in the receiver were controlled manually by adjusting the flow of the water phase through a stopcock in an outlet at the base of the flask. The alcohol phase then overflowed through an outlet in the neck of the flask. The water and alcohol phase samples were taken off in 1 oz. screw cap bottles at these two points.

The inlet feed streams were kept at a fairly constant temperature by using a crude coil-in-box type constant temperature bath on each feed line. The temperature of the bath was maintained manually by adjusting the proportions of hot and cold tap water which made up the cooling water of the bath.

Copper tubing, 1/4" diameter, was used throughout the equipment for the flow lines. All the valves in the equipment were either brass Hoke toggle valves or brass Hoke needle valves. The receiver was attached to the column by a piece of rubber tubing, tightened down to the column and receiver side arm ends by copper wire.

C. Experimental Conditions

A column diameter of 1/2" was chosen so that fairly high mass velocities could be attained without using unreasonable amounts of isobutanol. Commercial grade isobutanol was used as received and was obtained from the Carbon and Carbide Chemicals Company. Its index of refraction was 1.39382, which agrees substantially with values published in other studies. Distilled water was used for the water phase. It was necessary to acidify the water to 0.0001 N HCl in order to avoid incurring an emulsion. The phases separated very quickly when the water was acidified.

Bed heights of 1", 2", 4" and 6" were used. A temperature of 25°C $\pm$ 0.5°C was maintained throughout the work. The inlet streams were always the pure components.

The flow rates of the inlet phases were determined by convenient percentages of the total plunger stroke of the proportioning pumps. The water rate was varied in five steps from approximately 3,700 to 20,000 gms/hr, and the isobutanol rate also in five steps from approximately 2,200 to 16,600 gms/hr. The highest flow rates corresponded to about 5/6 of the total stroke and the lowest about 1/10. The flow rates corresponded to 6,000 to 32,000 lbs/(hr)(ft)^2 in the water phase and 3,500 to 27,000 lbs/(hr)(ft)^2 in the isobutanol phase.

D. Experimental Procedure

The four columns, with wire screens cemented in place at the bottom, were filled with glass beads until there was no looseness apparent when the tubes were shaken. After a column was filled with beads, a stainless steel screen was placed on the top and held in place by a piece of rubber tubing, the same rubber tubing which served to connect the column with the receiver side arm. A column was made up only once and was stored in distilled water when not in use. This was an attempt to avoid variations in the packing arrangement of each column. The porosity of the columns ranged from 0.36 for the 1" column to 0.34 for the 6" column.

The receiver was placed on the top of the column and was adjusted to a position held during all the runs by means of three marks on the equator of the flask which indicated when the receiver was in a level position. In this way, the phases always entered the receiver in the same way. The interface between the separated phases was adjusted to the marks on the flask equator and was held there by adjusting the flow of the water phase leaving the receiver. This eliminated variations in phase residence time and interfacial area.

For a given bed height, the experimental data were obtained in the following manner. With one of the receivers in place, a water flow rate was fixed, and receiver outlet phase samples were taken for a series of isobutanol flow rates, with each isobutanol rate being a separate run. The water flow rate was then changed and receiver outlet phase samples were taken varying the alcohol rates again. This was continued for a

number of water rates. The second receiver was then put in place and the procedure repeated with appropriate flow rates. In general, the larger receiver was used to handle the higher flow rates and the smaller receiver the lower flow rates. Some flow rates were established in both receivers for purposes of comparison.

The pump stroke settings were not finely adjusted after the initial coarse setting. Each flow rate corresponded to a certain pump stroke length. Since the flow rates could be reproduced within 5% with the coarse setting, no further adjustment was made. The data obtained were ultimately adjusted graphically to nominal flow rates (those which minimized the adjustment). The system of nominal flow rates used to obtain the variable column length data is indicated in Table I.

TABLE I

SCHEME OF NOMINAL FLOW RATE VARIATION-APPROXIMATE FLOW RATES IN lbs/(hr)(ft)^2

Large Receiver		Small Receiver	
Water	Isobutanol	Water	Isobutanol
12,000	13,000	18,000	6,800
	19,000		13,000
	27,000		3,500
18,000	27,000	12,000	3,500
	19,000		6,800
	13,000		13,000
25,000	13,000	6,000	13,000
	6,800		19,000
	19,000		6,800
	27,000		3,500
32,000	27,000		
	19,000		
	13,000		
	6,800		

Sampling from the two phases took place at their outlets in the neck and bottom of the receiver and consisted of filling one 1 oz. screw cap bottle with the water phase and another with the alcohol phase.

When the receiver and column were in place, the inlet line constant temperature baths at the correct temperature and the pumps set for the desired water and isobutanol rates, the liquid levels of the feed reservoirs were read, and the pumps were started. After the phases filled the receiver, the water phase level was set at the marks at the receiver equator by adjusting the flow through the stopcock in the water outlet line. After this level was steady, the system was allowed to come to steady state before any samples were taken. This waiting time corresponded to the approximate time it would take the phases to replace themselves 7 to 10 times in the receiver. This time was estimated by taking a series of samples from the large receiver when both the water and isobutanol flow rates were fairly low. It turned out that after a replacement of about 4 times, there was no change in the outlet phase compositions. The replacement time of 7-10 was used, however, as a conservative estimate.

After the samples were taken, the pumps were turned off and the liquid levels of the feed reservoirs again read. The exact flow rates were obtained from calibration curves of height of liquid in the reservoir vs. weight, the difference in the two readings being the weight of the liquid pumped. The time of the run was determined with a stop watch.

The phase samples were analysed for composition by measuring the refractive index with a Bausch and Lomb Precision Refractometer. The refractive index of a sample could be determined easily within $\pm$ 0.00005

units. Calibration curves for phase composition in weight percent of water vs. index of refraction are presented in Figures 23 and 24 in Appendix E. The addition of the small amount of HCl to the distilled water did not change the index of refraction readings for a given composition nor the values of the equilibrium compositions.

IV. RESULTS OF THE VARIABLE COLUMN LENGTH EXPERIMENT

The original data obtained with the four column heights 1", 2", 4" and 6", with the inlet feeds being pure components, are tabulated in Appendix A. The data consist of inlet phase flow rates and indices of refraction of the receiver outlet phases (water-rich and isobutanol-rich). The flow rates obtained by the coarse pump adjustment were usually very close to the nominal rates tabulated in Table I. Small deviations were handled by the following procedure.

A plot of outlet phase index of refraction vs. alcohol inlet rate at the fixed inlet water rate was first made. This plot established the dependence of the index of refraction of the outlet phase on inlet alcohol rate (at the constant inlet water rate) and was used to adjust the index of refraction of the outlet phase (water-rich or isobutanol-rich) to what it would have been if the inlet alcohol rate had been the nominal rate included in Table I. This procedure, which is illustrated graphically in Figure 3, retained the identity of the data points which cross-plotting would not do.

When the adjustment of the receiver outlet indices of refraction was carried out for the alcohol rate, the adjusted points were plotted against water rate at the nominal alcohol rates. Examples of these plots are shown as Figures 4, 5 and 6. Outlet water phase refractive indices from a 4" and a 6" column are shown in Figures 4 and 5 respectively. Outlet isobutanol phase refractive indices are shown for a 2" and a 6" column

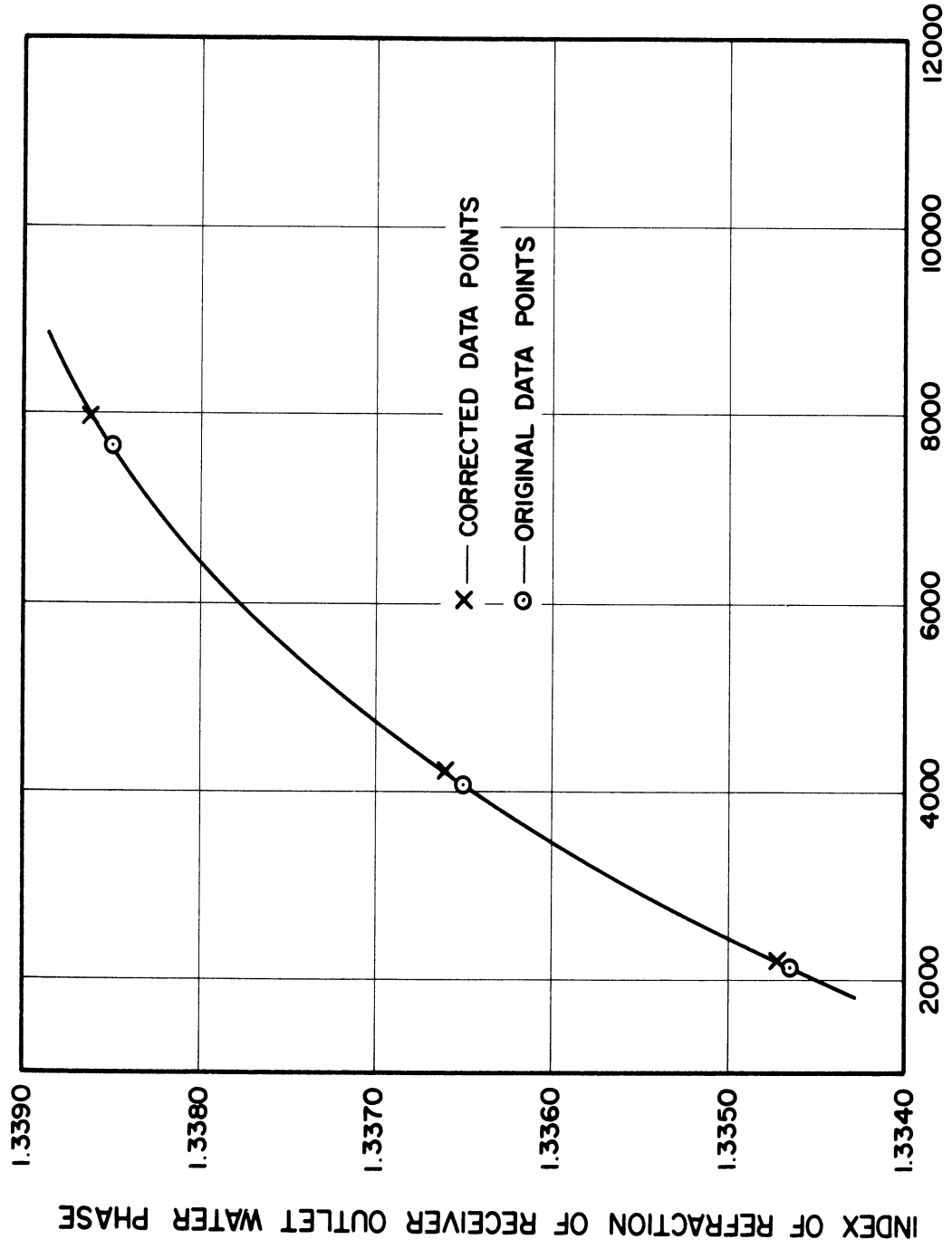


Figure 3. Procedure for Correcting Data to Nominal Flow Rates.

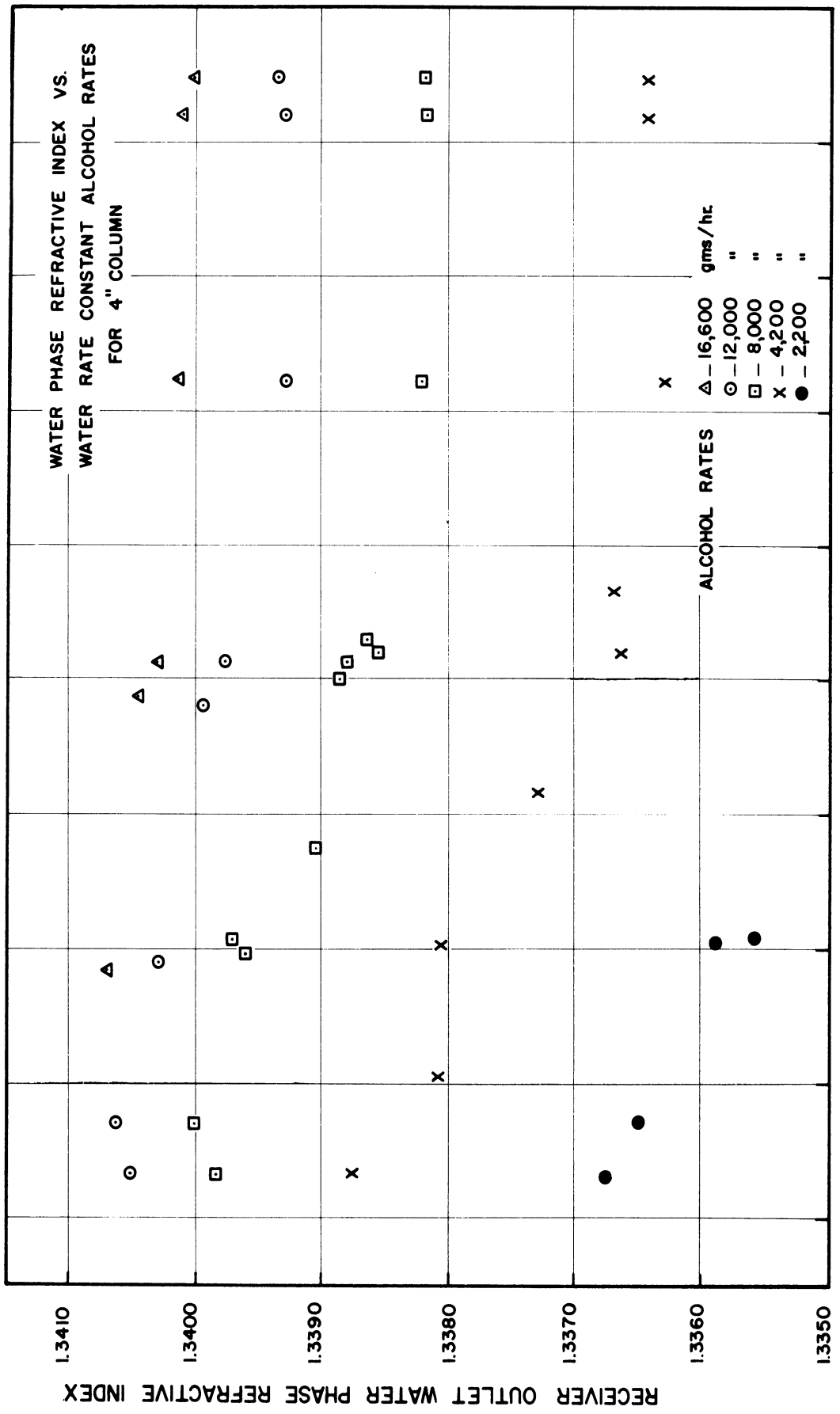


Figure 4. Water Phase Data Corrected to Nominal Alcohol Rates, 4-Inch Column.

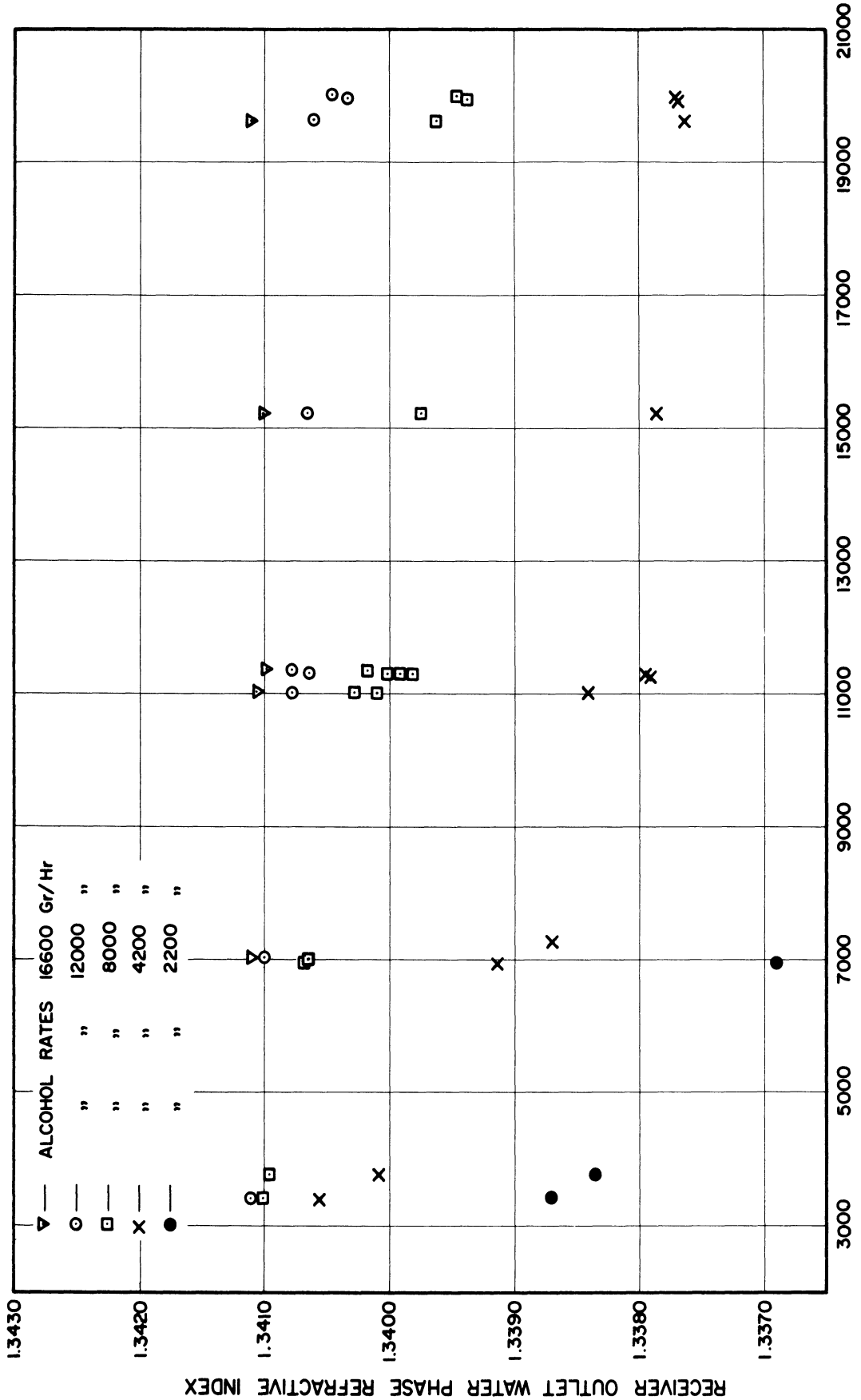


Figure 5. Water Phase Data Corrected to Nominal Alcohol Rates, 6-Inch Column.

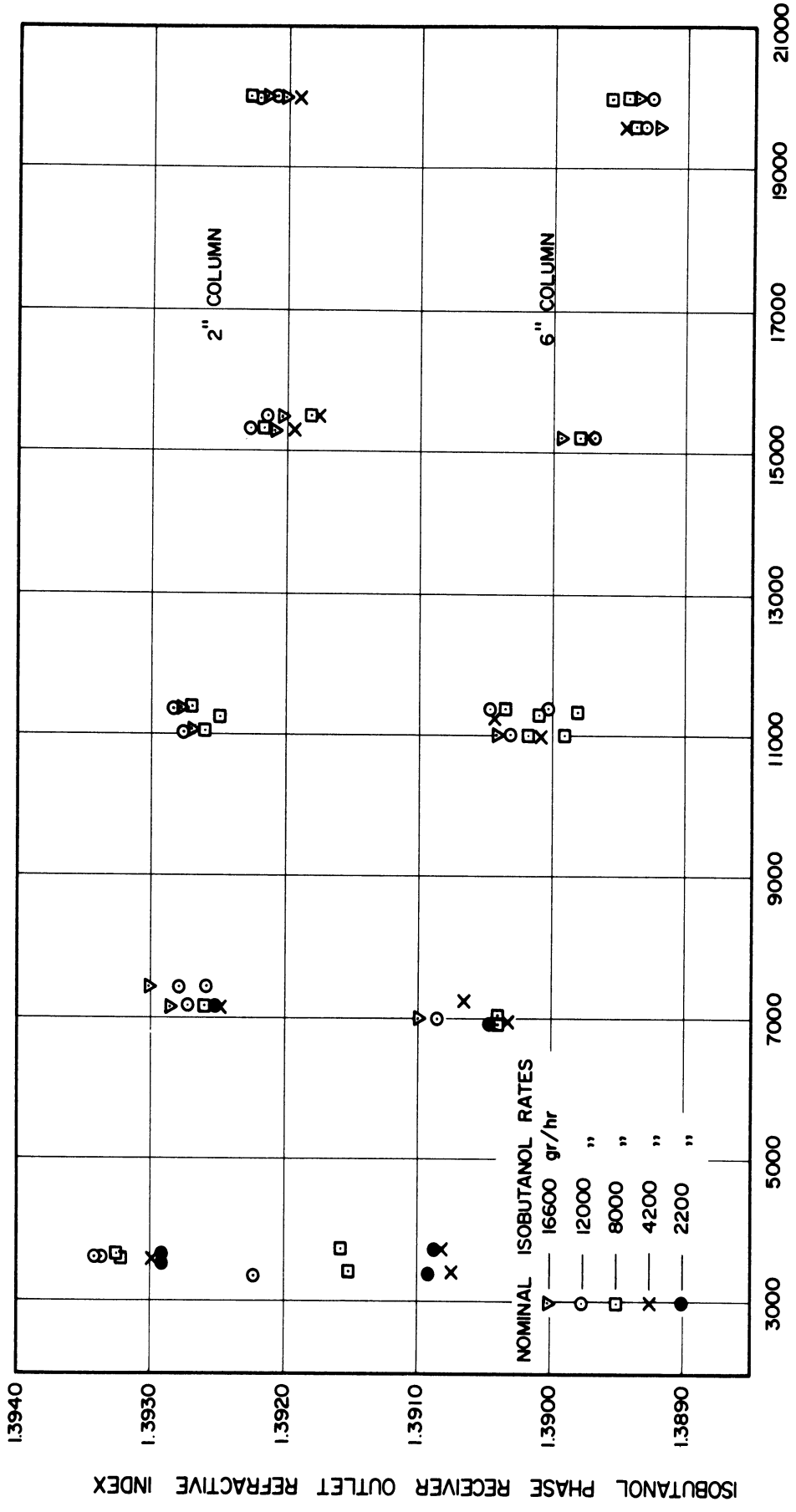


Figure 6. Alcohol Phase Data Corrected to Nominal Alcohol Rates, 2-Inch and 6-Inch Columns.

in Figure 6. The points on these plots were then adjusted to the nominal water rates. These doubly adjusted data points are tabulated in Appendix B for the various column lengths used, with the weight fractions corresponding to the index of refraction readings added.

Figures 4 and 5 show that the outlet indices of refraction of the water phase are sharply separated as functions of the inlet flow rates. However, Figure 6 shows that those of the alcohol phase do not vary nearly as much with flow rate. At a given bed height and at a constant water rate, the degree of saturation of the isobutanol phase is practically insensitive to the alcohol rate.

An idea of the reproducibility of the data is also given by Figures 4, 5 and 6. All duplicate data points (except those from different receivers during the same column run) were obtained in completely independent runs, that is, on different days, with a re-setting up of the column and receiver. It would appear then, that the data are fairly well reproducible.

V. THE CONTRIBUTION OF THE RECEIVER TO TOTAL TRANSFER

The results of the experiment with the various column lengths show the variation in mass transfer that takes place in the system consisting of the packed bed plus the receiver. Pure components were fed at various rates in this experiment. The portion of the total transfer that takes place in the receiver is difficult to assess directly and will be discussed at some length in this section. The conclusion arrived at, however, is simply that the contribution of the receiver to the total transfer is not significant, and hence, the variable column length data may be treated as if the receiver outlet phase compositions were column outlet phase compositions.

Qualitative support for the reasonableness of this conclusion can be drawn from the low mass transfer coefficients that are obtained in countercurrent spray and packed column experiments relative to those obtained at the higher rates of flow possible in concurrent flow through a packed bed. An additional factor in the concurrent case is that the two phases leave the column and enter the receiver at nearly equal velocities, and that the dispersed isobutanol phase is coalescing rapidly.

The quantitative support gathered in this section for the assumption of negligible receiver transfer relative to column transfer can be summarized in the following three points, which will be discussed at the end of the section.

1. No receiver transfer could be detected in experiments in which the transfer in a column plus receiver was measured as a function of inlet flow rates and inlet phase compositions.

2. The receiver outlet indices of refraction appear to extrapolate to the pure component index of refraction at zero bed height.

3. Outlet phase compositions were found to be independent of the size of receiver in experiments in which the flow rates for the two receivers overlapped.

A. Estimating the Receiver Effect Experimentally--The Method

The end effect of the receiver could be determined directly if it were possible to reproduce the physical condition of the dispersed phases entering the receiver side arm from the column without incurring any mass transfer in the process. It then would be a simple matter to vary the composition of the inlet streams to the receiver and produce plots of the outlet phase composition as a function of inlet phase composition. These plots would then define the transfer occurring in the receiver.

Since it is impossible to disperse the phases without incurring some unknown amount of mass transfer, it was decided to determine the effect of inlet phase composition and flow rates on the receiver outlet phase compositions of a 1" column. A 1/2" I.D. column with one inch of packing was chosen for these experiments because this height of packing was about the minimum which would create a dispersion, at all flow rates, of the phases.

Now, if it is assumed that the phase transfer coefficients, k'_{WV} and k'_{AL}, are independent of height in the column, i.e., the phases are uniformly dispersed along the length of the column, then this 1" column

plus the receiver in place on top of it, can be equated to the last inch of packing plus the receiver of the 2", 4" and 6" columns. Then, in effect, by subtracting the transfer which takes place in the 1" column plus receiver from the total transfer of the 2", 4" and 6" columns, it is possible to estimate the compositions of the phases entering the last inch of these latter columns.

In other words, with the results of this experiment with the 1" column, an attempt is made to estimate graphically the phase compositions entering the last inch of packing of the 2", 4" and 6" columns that produced the receiver outlet phase compositions measured in the variable column length experiment. These phase compositions are then estimates of the internal column compositions. For a given inlet flow rate condition, the internal phase compositions can be plotted against height of column, since their values are known at 0", 1", 3" and 5" of column packing. A superposition of these plots on similar plots of receiver outlet compositions at 1", 2", "4 and 6" of column will yield the receiver effect, i.e., the difference between the curves.

1. Experimental

The equipment used to study the effect of inlet phase compositions and flow rates on the receiver outlet phase compositions was the same as that described previously for the variable column length experiment. It was decided that three compositions of each phase would be run making a total of nine combinations of inlet phase compositions. These compositions were roughly 0, 40 and 80 percent of the saturation compositions of the phases. About 15 gallons of a phase were needed to run through a complete

set of flow rates. The 45 gallons needed for the three runs using a particular inlet phase composition were mixed at one time in a 55 gallon, 316 stainless steel drum and stored for future use in 5 gallon carboys. The feed reservoirs were filled directly from the carboys, using compressed air, and thus the feed system was completely closed to air, eliminating possible concentration changes of the feed during the long period needed to run all the required flow rates. The nominal flow rates at which these experiments were run were those tabulated in Table I. The experimental procedure used was the same as that used in the variable column length experiments.

2. Experimental Results

The original data obtained in this experiment are tabulated in Appendix C. They are based on the feed calibration obtained for pure components and must be multiplied by the density ratio corresponding to the various inlet phase compositions in order to obtain true weights of solution flowing in gms. per hour of the solution in question. These data were then treated in exactly the same manner as in the variable column length experiment. The refractive indices were first plotted against isobutanol rate at constant water rates and then replotted against water rate choosing refractive indices at constant alcohol rates as in Table I. This was done for each combination of water phase and isobutanol phase inlet compositions. Examples of the indices of refraction of the receiver outlet phases corrected to the nominal isobutanol rates are shown as Figures 7 and 8. In Figure 7, the indices of refraction of the receiver outlet water phase are shown plotted against the inlet water rate, the inlet

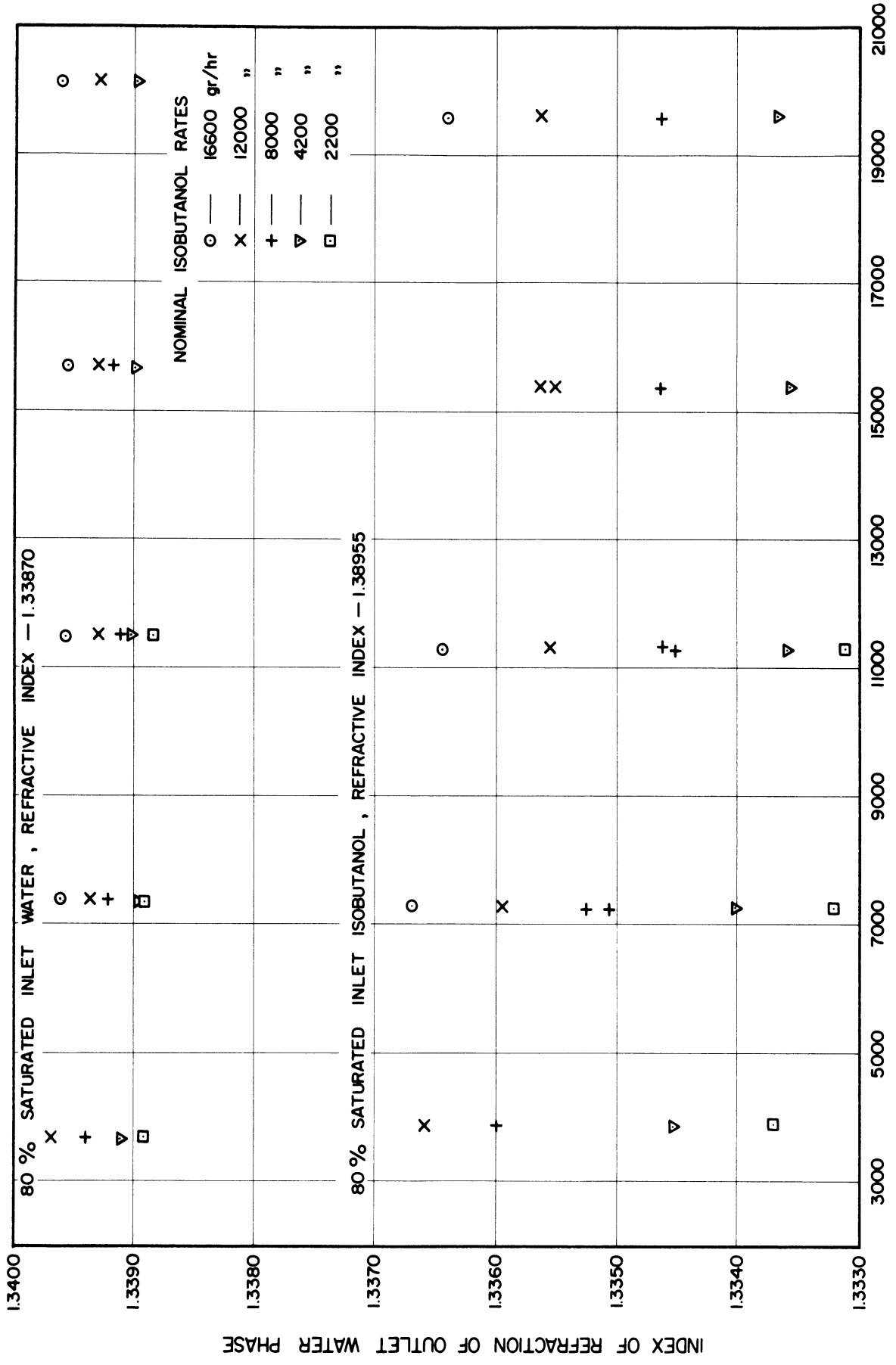


Figure 7. Water Phase Data, 1-Inch column, Corrected to Nominal Isobutanol Rates.

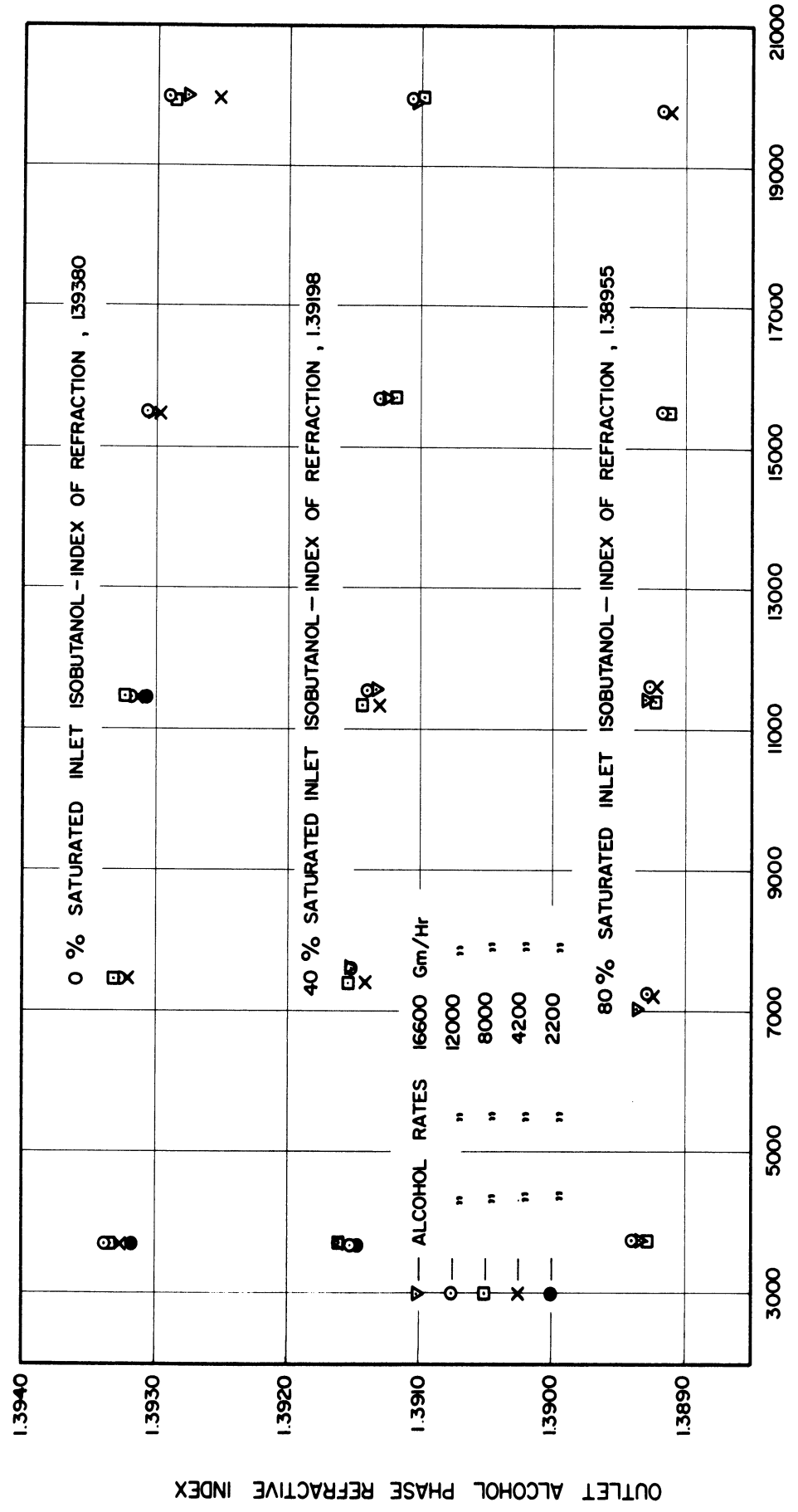


Figure 8. Isobutanol Phase Data, 1-Inch Column, Corrected to Nominal Isobutanol Rates.

isobutanol phase being approximately 80% saturated with water. The top set of points are for a water inlet saturation of approximately 80% and the lower set for a water inlet of 0% saturation.

In Figure 8, the outlet isobutanol phase refractive indices are plotted against the water rate, with the inlet water phase being 40% saturated with isobutanol. Three inlet isobutanol phase compositions, 0%, 40% and 80% saturated with water, are shown. The secondary parameter in these two plots is the nominal inlet flow rate of the isobutanol phase. It is interesting to note that the receiver outlet composition (refractive index) of the isobutanol phase is quite insensitive to the alcohol rate, and to some degree of the water rate.

The plots in Figures 7 and 8 (outlet refractive index vs. actual water rate, parameters-inlet phase saturation, actual alcohol rate) are based on the inlet rates of the phases of various percentages of saturation. These plots, therefore, cannot be used directly to construct column inlet vs. receiver outlet compositions for comparison with receiver outlet compositions from the variable column length experiment. These latter data are plotted on a basis of pure inlet components. Therefore, to effect the comparison, the column inlet vs. receiver outlet composition plots must also be constructed on a basis of pure inlet components, i.e., if a pure water phase started out at L_o' gms/hr and a pure alcohol phase started out at V_o' gms/hr, what would their respective flow rates L' and V' be at a point in the column where their respective saturations were, say, 40 and 80 percent? These are found by a component material balance,

$$L_o' = L'X_W + V'Y_W$$

$$V_o' = L'X_A + V'Y_A$$

where X_α, Y_α are mass fractions of component α in the L and V streams,

L', V' are actual mass rates at the point in the column being considered,

L'_o, V'_o are the mass rates of pure components that are equivalent to L' and V' when their compositions reach X_α and Y_α, and are nominal rates found in Table I.

Indices of refraction of the receiver outlet phase can then be found for the L' and V' 's computed as above on plots such as Figures 7 and 8. These indices of refraction constitute an estimation of the outlet phase refractive index, when the inlet phases are of a certain percentage of saturation and have total component flow rates L'_o and V'_o. Examples of these plots are shown as Figures 9-12. These plots are the goal of the experiment.

Aside from their intended use in the receiver end effect problem, these plots also have the additional property of purporting to show the effect of the concentration of one phase on the amount of mass transfer that takes place into the other phase. It is seen from the data that the composition of the water phase apparently has very little influence on the transfer of water into the isobutanol phase. On the other hand, the more water present in the isobutanol phase, the more isobutanol transferred to the water phase. This is especially true at higher flow rates. However, the region of respective saturation of the phases in Figures 9-12, where this effect is most pronounced, never have to be considered when the receiver effect is estimated. In order for the effect of saturation

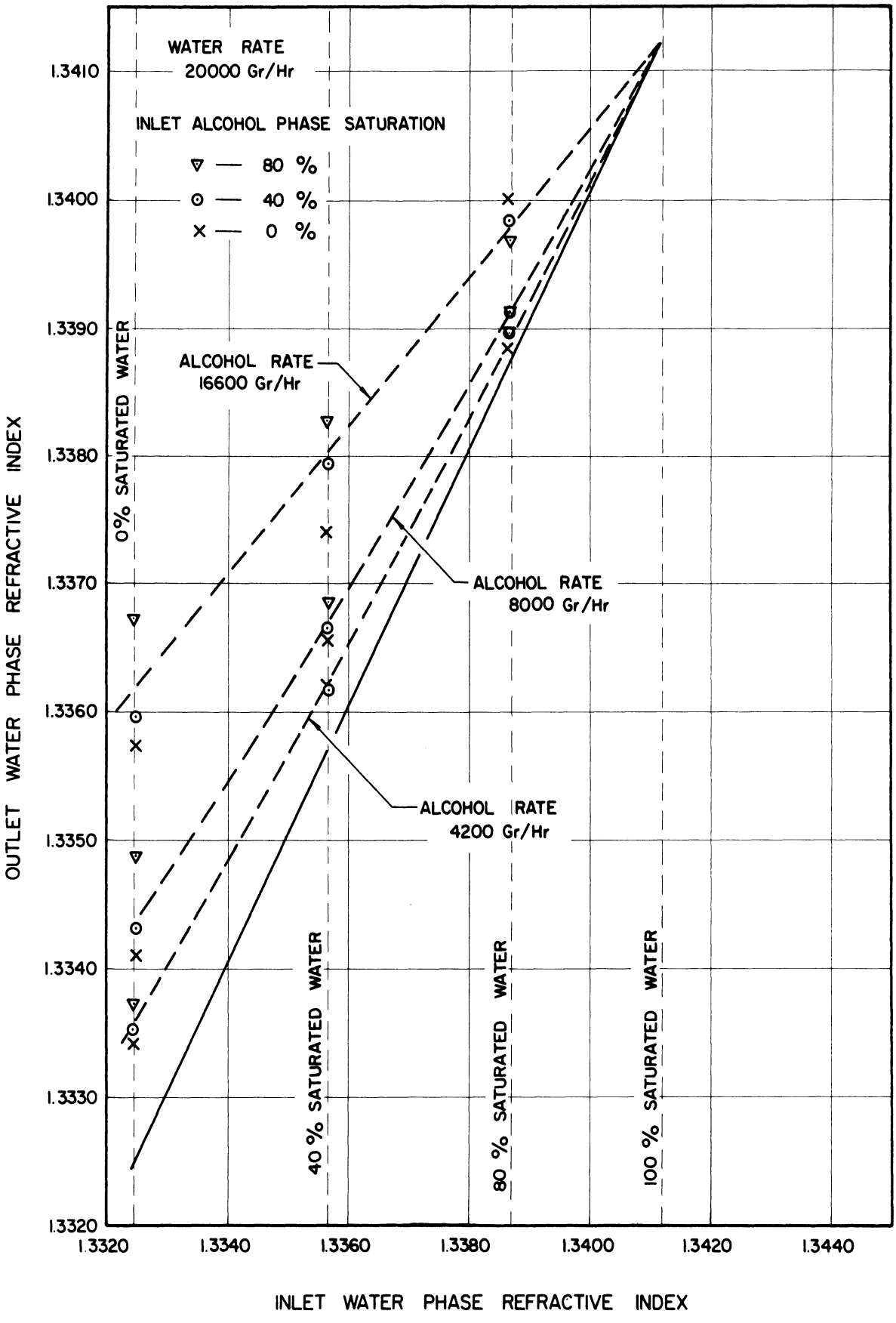


Figure 9. Inlet vs. Outlet Water Phase Refractive Indices, 1-Inch Column.

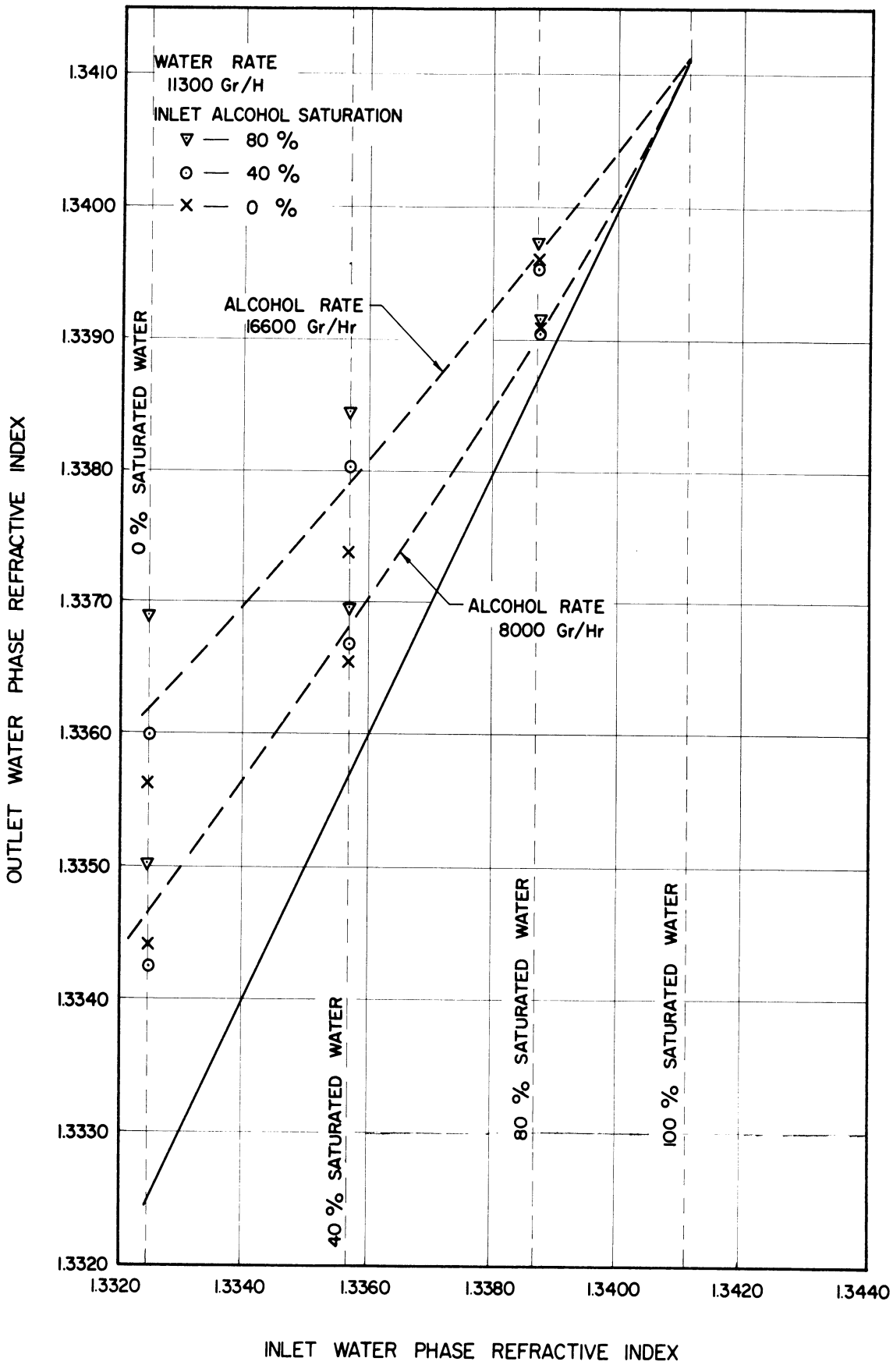


Figure 10. Inlet vs. Outlet Water Phase Refractive Indices, 1-Inch Column.

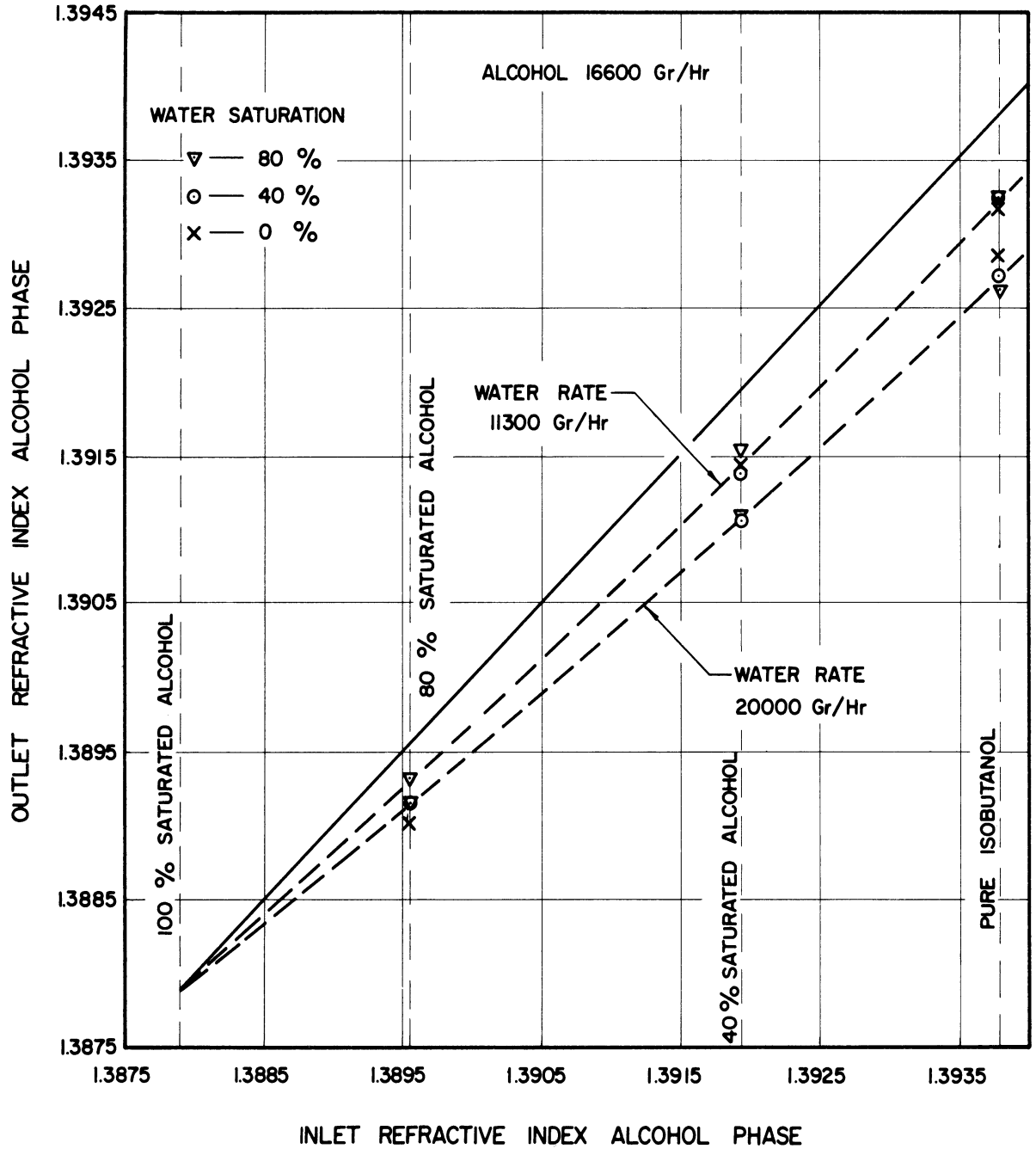


Figure 11. Inlet vs. Outlet Isobutanol Phase Refractive Indices, 1-Inch Column.

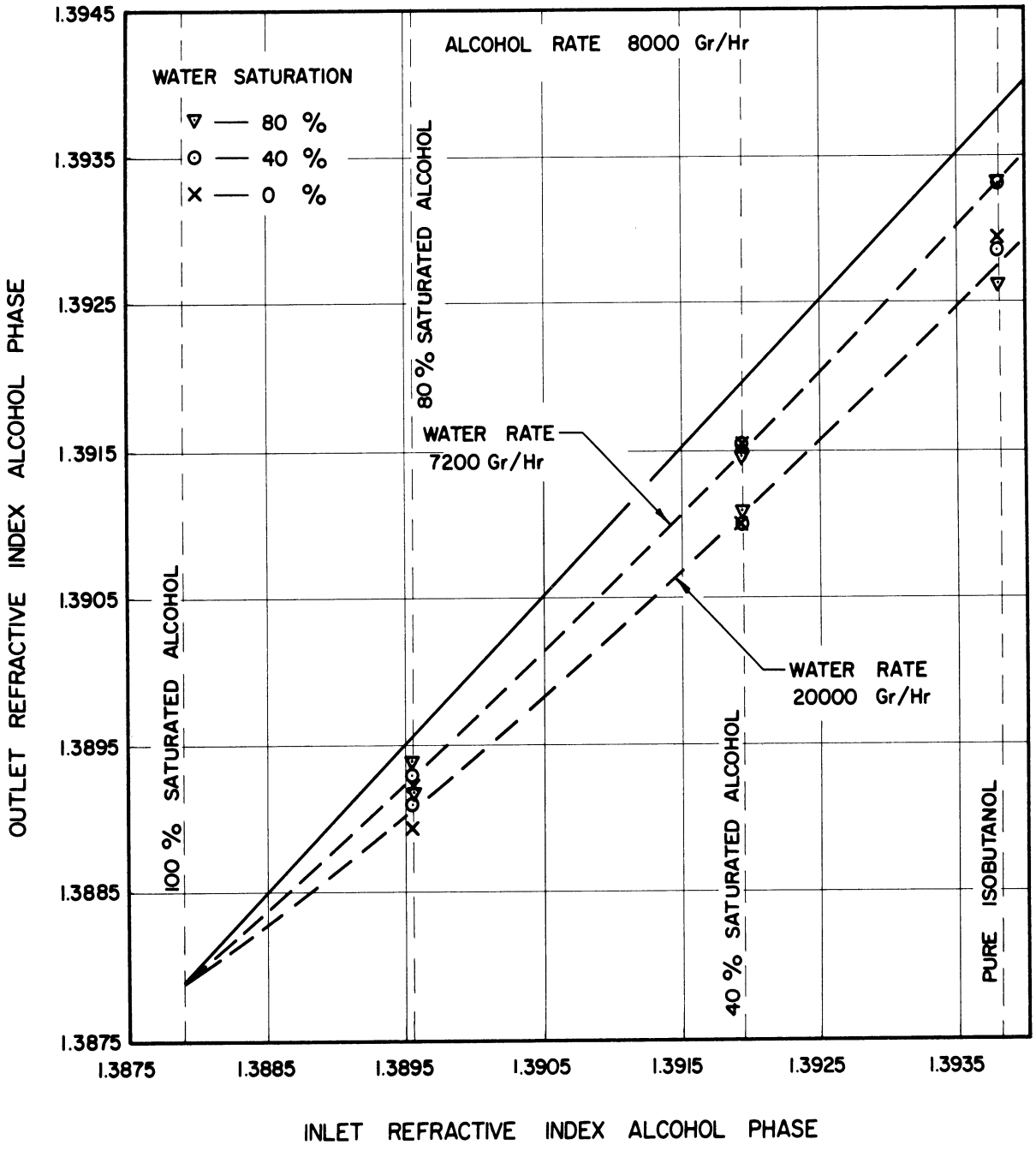


Figure 12. Inlet vs. Outlet Isobutanol Phase Refractive Indices, 1-Inch Column.

to be significant (considering the scatter in the variable column length experiment) the alcohol phase must be highly saturated with water while the water phase must be relatively pure. Since the variable column length data has been taken with the inlet phases being pure components, this situation never exists in the column. As a matter of fact, in the variable column length experiment, the water phase becomes saturated far more quickly than the isobutanol phase, therefore keeping the concentration effect of the isobutanol phase not particulary significant (see Figure 9).

3. Procedure for Estimation of Internal Column Phase Indices of Refraction

From plots such as those shown in Figures 9-12 it is possible, under the assumption that the phase transfer coefficients k'_{AL} and k'_{WV} are independent of height (uniform dispersion with height), to estimate how much mass transfer took place in the receiver and last inch of packing of the 2", 4" and 6" columns, thereby estimating internal column compositions at 0", 1", 3" and 5" of bed height. For example, consider the water phase under the column flow conditions of 20,000 gms/hr pure inlet water and 16,600 gms/hr pure inlet isobutanol. At a bed height of 6", the receiver outlet water phase had a refractive index of 1.3411 (cf. Appendix B). Now, taking this as the refractive index of the water phase at the receiver outlet, as in Figure 9, the refractive index of the inlet water phase to a 1" column plus receiver is estimated to be 1.3409. This is then assumed to be the refractive index of the water phase leaving the fifth inch of packing in the 6" bed. The procedure is repeated for the

4" column and a refractive index of the water phase is estimated for the third inch of packing and again for the 2" column. It is to be remembered that the level of saturation of the alcohol phase should be taken into account when estimating these water phase refractive indices. A summary of the values obtained for this example is given in Table II below and plotted as the 16,600 gms/hr alcohol rate parameter of Figure 13. Where duplicate data points were available for the various column lengths, the arithmetic average was taken.

TABLE II

SUMMARY--ESTIMATION OF INTERNAL COLUMN REFRACTIVE INDICES OF THE WATER PHASE--INLET FEED RATES: WATER - 20,000 gms/hr, ISOBUTANOL - 16,600 gms/hr.

total height of bed	receiver outlet ref. index	height of est.	est. internal ref. index
6"	1.34109	5"	1.3409
4"	1.34007	3"	1.3391
2"	1.33793	1"	1.3358
1"	1.33595	0"	1.33245

The plots shown in Figures 13-16 are representative of this procedure for both the water phase (Figures 13, 14) and the isobutanol phase (Figures 15, 16). These plots include both the original refractive indices of the receiver outlet with the various columns (the dots) and the estimated internal refractive indices (the x's). If there is a significant receiver effect, a curve through the internal refractive indices should lie

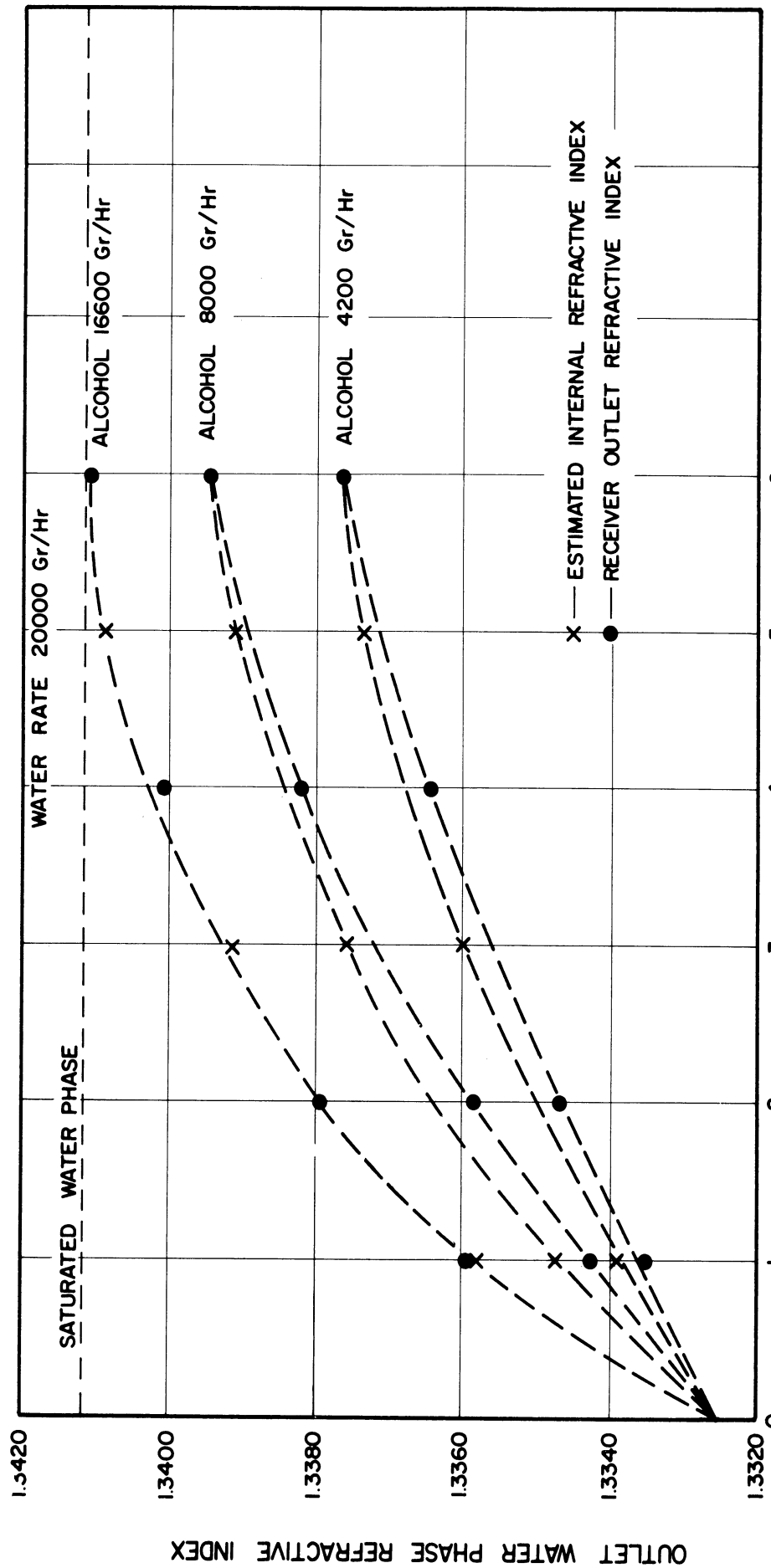


Figure 13. Receiver Effect Estimation Curves, Water Phase.

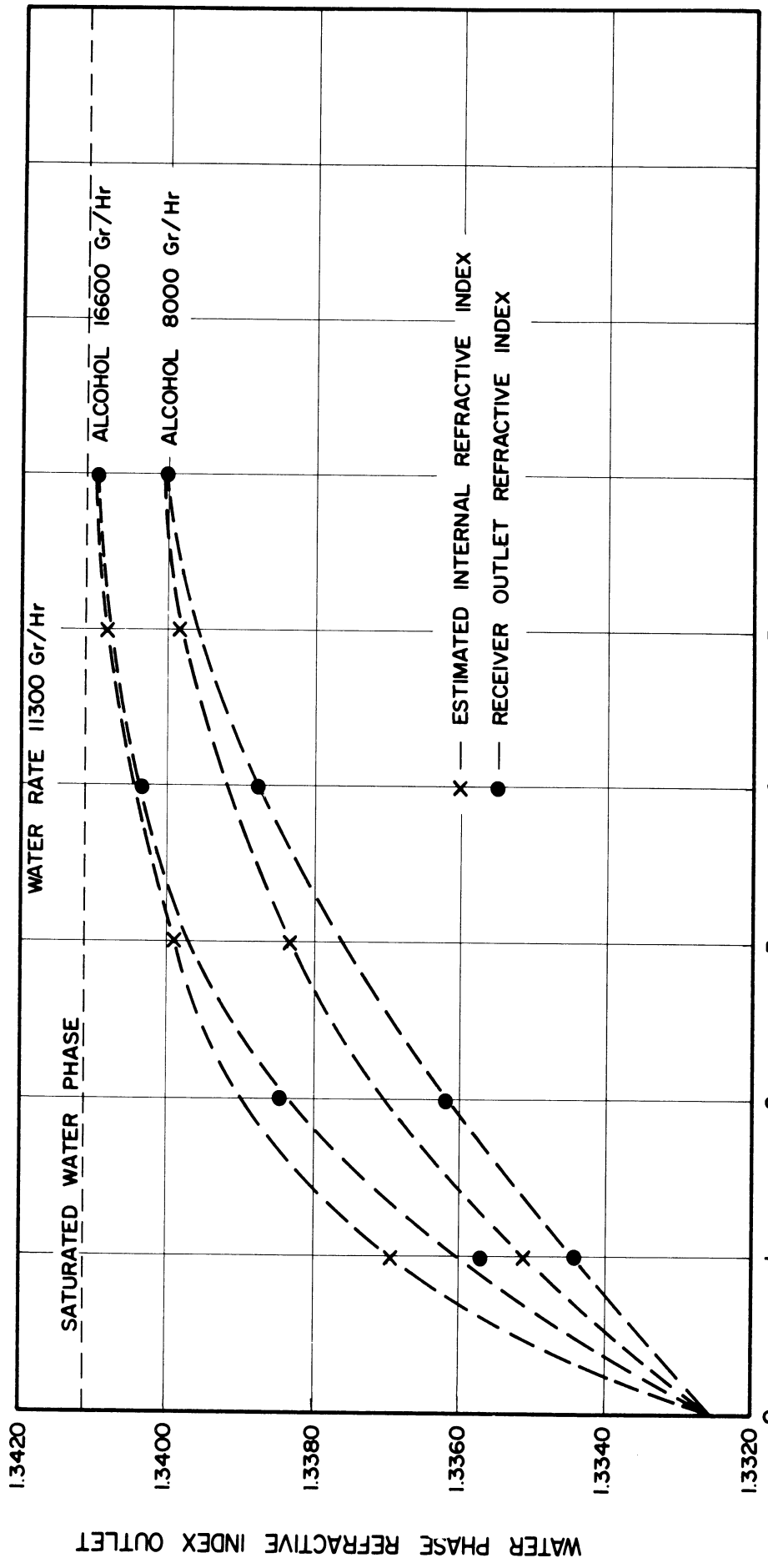


Figure 14. Receiver Effect Estimation Curves, Water Phase.

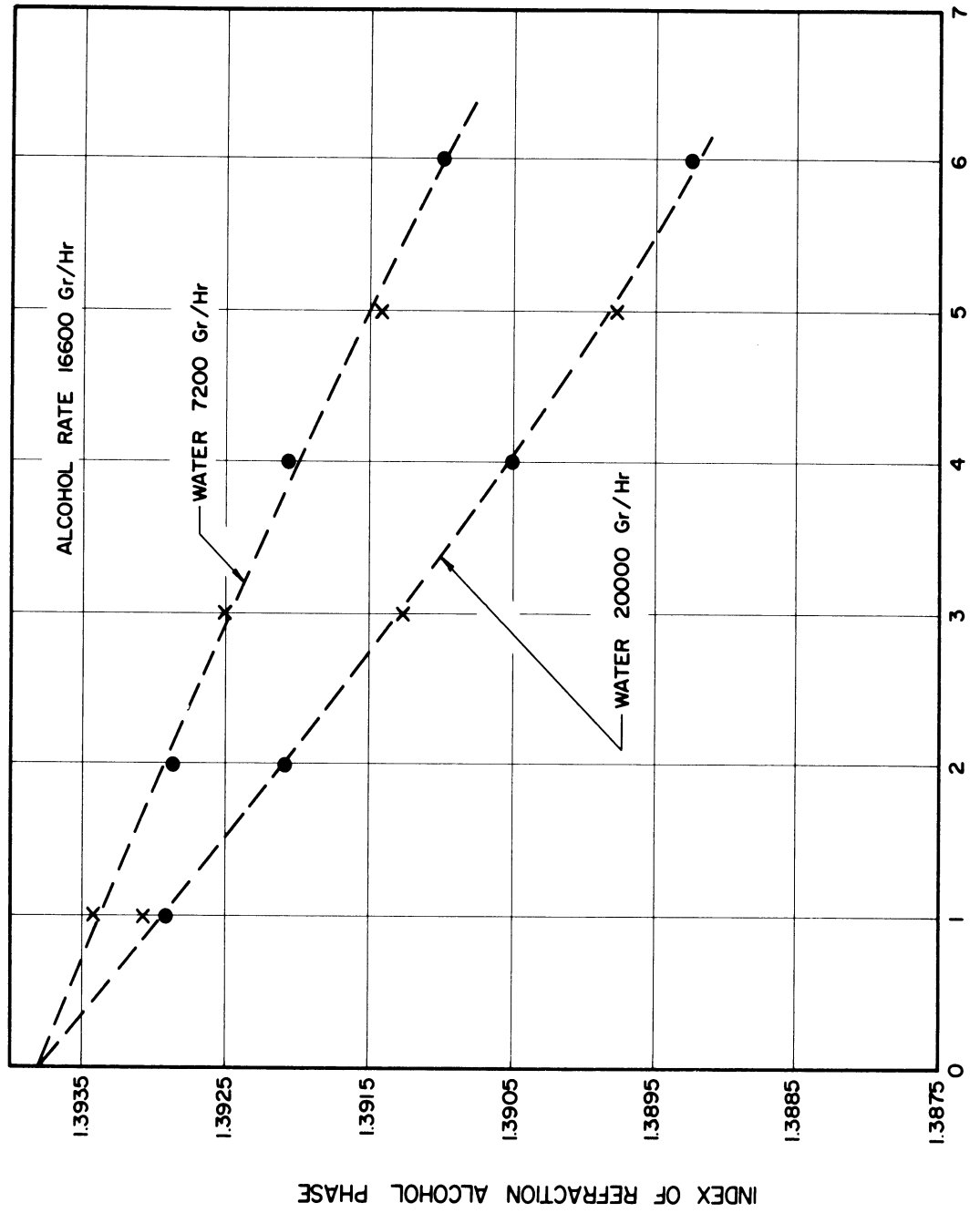


Figure 15. Receiver Effect Estimation Curves, Isobutanol Phase.

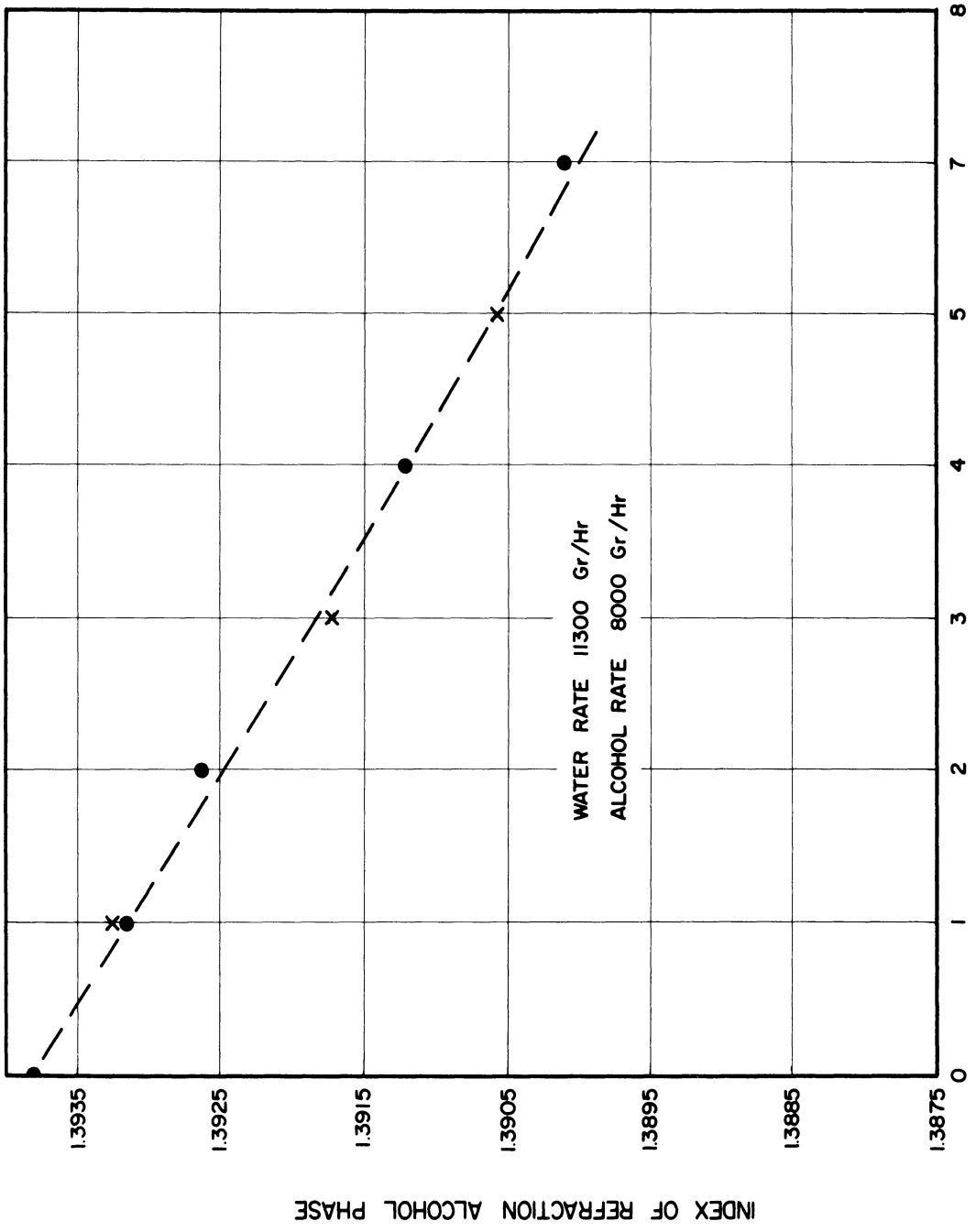


Figure 16. Receiver Effect Estimation Curves, Isobutanol Phase.

nearer the less saturated end of the refractive index scale than a curve through the receiver outlet points, i.e., a positive amount of transfer should be taking place in the receiver. Therefore, Figures 13-16 yield an estimate of the transfer which occurred in the receiver with total bed heights of 1", 2", 4" and 6".

B. Discussion

The three points mentioned in the introduction to this section as summarizing the support for the assumption of negligible receiver transfer will now be discussed in detail, taking into consideration the relevance for the first point of the experimental work discussed above.

1. By considering Figures 13 and 14 for the water phase, it is apparent that, with the exception of the highest flow rates run, 20,000 gms/hr water and 16,600 gms/hr isobutanol, the procedure described in Section A, above, yields an estimate of a negative amount of transfer taking place in the receiver for the 2", 4" and 6" columns. This is indicated by the curves through the internal refractive indices (the x's) lying above those drawn through the receiver outlet indices or, toward more saturated water solutions. Those of the highest flow rates are essentially coincident, indicating an estimate of zero transfer in the receiver at this condition.

In the plots for the isobutanol phase, Figures 15 and 16, the curves are essentially coincident regardless of flow rate. These curves bear out the assertion that the receiver effect is not large enough to constitute a significant proportion of the total mass transfer incurred in the column packing and the receiver.

Now, the method of estimation is based on the assumption of uniform dispersion along the column length, making the first inch physically equivalent to any other. This condition should be most closely approached at the highest flow rates run, i.e., at 20,000 gms/hr water and 16,600 gms/hr alcohol. Therefore, at these rates, the method of estimation should come closest to estimating the true receiver effect. Moreover, the receiver effect at these rates should be greatest, due to the turbulence in the receiver side arm. However, it is at these rates that the receiver effect is estimated to be zero. At lower flow rates, the first inch is probably not equivalent to later ones and the receiver effect correspondingly is estimated to be negative. Since the turbulence of the phases entering the receiver at these lower rates is much reduced, the receiver effect is probably reduced also. And since the transfer in the receiver can not be detected when it should be greatest, it seems safe to conclude that it is negligible at all conditions.

At all flow rates, the internal refractive indices of the alcohol phase appear to coincide with those of the receiver outlet, thus showing that for the alcohol phase also, the receiver effect can not be detected. In both the experiment of this section and that of varying the column length, the outlet phase compositions of the alcohol phase were relatively insensitive to flow rates. Because of this, the same reasoning applied to the water phase can be applied to the alcohol phase.

In other words, the experiment varying the inlet phase compositions shows that the effect of the receiver on outlet phase compositions

is small enough so that the experiment can detect a difference in transfer taking place in the first inch of packing of a column as compared with the last inch. Therefore, unless the first inch of packing is completely ineffective in causing mass transfer, the receiver effect cannot constitute any significant proportion of the total transfer.

2. Further indirect evidence can be cited to support this conclusion. As Figures 13-16 shows the receiver outlet refractive indices, in every case, can be extrapolated without difficulty to the refractive index of the pure component (water or isobutanol) and never to that of finite saturation. Though this extrapolation cannot be used in any quantitative sense, it indicates qualitatively that the receiver outlet phases of the one inch column, where the driving force for mass transfer is greatest, does not contain a very significant contribution from the receiver. Since the effect does not appear significant for the one inch column, it follows that for the remaining columns, where it should be diminishing, it will also remain small.

3. The third point in support of negligible receiver effect is that receiver size did not appear to affect receiver outlet phase concentrations. The outlet phase compositions vary continuously over the entire range of flow rates and coincide in the region where the receivers overlap, as seen in Figures 3-5. Since the phases certainly separate under different conditions and have different residence times in the two receivers, the effect on the outlet compositions should be different. If this effect were large, then the difference should be detected. Since it is not, the effect must not be significant.

There is no absolute estimate of the receiver effect in any of the forgoing points supporting the assertion that it is negligible. It is nevertheless clear, however, that all evidence points to the conclusion that there will be little difference in choosing to call the receiver outlet compositions equivalent to the corresponding column outlet compositions. With this assumption, the data from the variable column length experiment can be treated as internal phase compositions, from which the individual phase transfer coefficients k'_{AL} and k'_{WV} can be derived. This is the subject of the following section.

VI. THE INDIVIDUAL PHASE TRANSFER COEFFICIENTS

The individual phase transfer coefficients can be calculated from the data obtained in the variable column length experiment (and tabulated in Appendix B) provided that the following two assumptions are made. (1) The transfer in the receiver is negligible, and (2) the internal compositions at any height are the same as the outlet compositions from a packed column of the same height.

The data obtained are incremental in nature and the transfer coefficients cannot be obtained directly from the differential model, Equation (5). There are several ways of processing the data in order to obtain phase transfer coefficients $k'_{\alpha\beta}$. On the one hand, if the phase transfer coefficient is considered independent of height in the column, Z, then Equation (5) can be integrated explicitly. For example, for water transferring into the isobutanol-rich phase, there results,

$$\int_{(Vy_W)_o}^{(Vy_W)_f} \frac{d(Vy_W)}{(C^*_{WV} - C_{WV})} = \bar{k}'_{WV}\, SH \qquad (9)$$

where H is the total length of the column,

S is the empty cross-section of the column,

Vy_W is the moles of water flowing in the isobutanol-rich phase at any point in the column,

$\bar{k}'_{WV}$ is the mean transfer coefficient for the column, $(hr.)^{-1}$.

The left hand integral can be evaluated graphically using the values of Vy_W and $(C^*_{WV} - C_{WV})$ calculated from the data. This procedure gives an

average phase coefficient for the entire length of the column.

On the other hand, a more satisfactory procedure, in that it indicates the variation and consistency of the coefficients with height, is to derive differential rates from the incremental data.

The calculation of phase coefficients from Equation (5) can proceed in two ways.

1. Substituting differences for the differentials in this equation, the incremental rates of mass transfer, $\Delta(Vy_W)/\Delta Z$, can be smoothed graphically with respect to Z on an equal area plot ($\Delta(Vy_W)/\Delta Z$ vs. Z). The smoothed (differential) rates of mass transfer are then plotted against the driving force. From Equation (5) it can be seen that the slope of a curve through these points is the transfer coefficient at the driving force where the slope is measured. A straight line through the origin gives a mean coefficient for the entire column.

2. A far more critical approach involves the calculation of incremental coefficients, $k'_{\alpha\beta}$, from Equation (10) below, for example, for the coefficient for water transferring into the isobutanol-rich phase,

$$\frac{\Delta(Vy_W)}{\Delta Z} = k'_{WV}\, S(C^*_{WV} - C_{WV})_{mean} \qquad (10)$$

This procedure depends on the arbitrary choice of a mean value of the concentration difference across the increment of column length and is successful only if the variation in this quantity is moderate. Therefore, in so far as the choice of a mean ΔC is arbitrary, this procedure yields a completely unbiased estimate of the coefficients $k'_{\alpha\beta}$ over the increment of column. For convenience in later discussion, the resulting coefficients will be referred to as integral, equal area and incremental.

VII. DISCUSSION OF RESULTS

The data from the variable column length experiment were treated both by the equal area and the incremental procedures. A few selected conditions were treated by the integral method. Sample calculations for all three procedures may be found in Appendix D.

It was found that the number of increments of column length used were not sufficient to enable accurate graphical differentiation of the data. This was especially true of the data for water transferring into the alcohol phase. In this case, some of the coefficients derived by the equal area procedure were quite inconsistent by comparison with those obtained by the incremental or integral methods. For the case of isobutanol transferring into the water-rich phase, the situation was better, and the equal area method usually gave coefficients in agreement with coefficients calculated by the other methods. Since the incremental coefficients were more consistent as well as independent of any data-smoothing bias, they were utilized in subsequent correlations. They are tabulated in their entirety in Tables V, VI and VII, Appendix D. The incremental coefficients for isobutanol transferring into the water-rich phase, k'_{AW}, are tabulated in Table V. Those for water transferring into the isobutanol-rich phase, k'_{WV}, in Table VI. The sample calculations and these tables are expressed in the mixed units obtained in the experiment.

A summary of these coefficients, in units of $lbs/(hr)(ft)^2$, is presented in Table VII. The coefficients in this table were obtained by taking an arithmetic average of the incremental coefficients with respect to height.

The incremental coefficients for the water phase are seen in Table V to be essentially constant with respect to column height, although the coefficient for the first inch is almost always a little lower than those for the succeeding increments. This is further illustrated in Figure 17 for a few selected inlet flow conditions. The low first inch coefficients would be expected due to insufficient mixing and are in agreement with the results of the end effect experiment, where it was found that a one inch column accounted for too little transfer in the last inch of the 2", 4" and 6" columns.

The alcohol phase coefficients, at least at the higher flow rates, seem to increase with increasing bed height. This increase is usually about 70% of the coefficient of the first inch, which is strikingly greater than the increase of the coefficients in the water phase. At lower flow rates, the incremental coefficients, though somewhat erratic, are more nearly invariant with bed height. This is illustrated in Figure 18, where the coefficients are plotted for two water rates, each with a high and a low inlet isobutanol rate.

The behavior of the incremental phase coefficients taken together is curious. For one coefficient to increase with increasing bed height while the other coefficient remains relatively constant, indicates that the effect is not just one of increasing the interfacial area for transfer. Since the effect cannot be attributed with certainty to composition effects or to height, use of the incremental coefficients in other situations is subject to considerable uncertainty. Hence, average coefficients $\bar{k}'_{\alpha\beta}$ were computed and are those given in Table VII. These

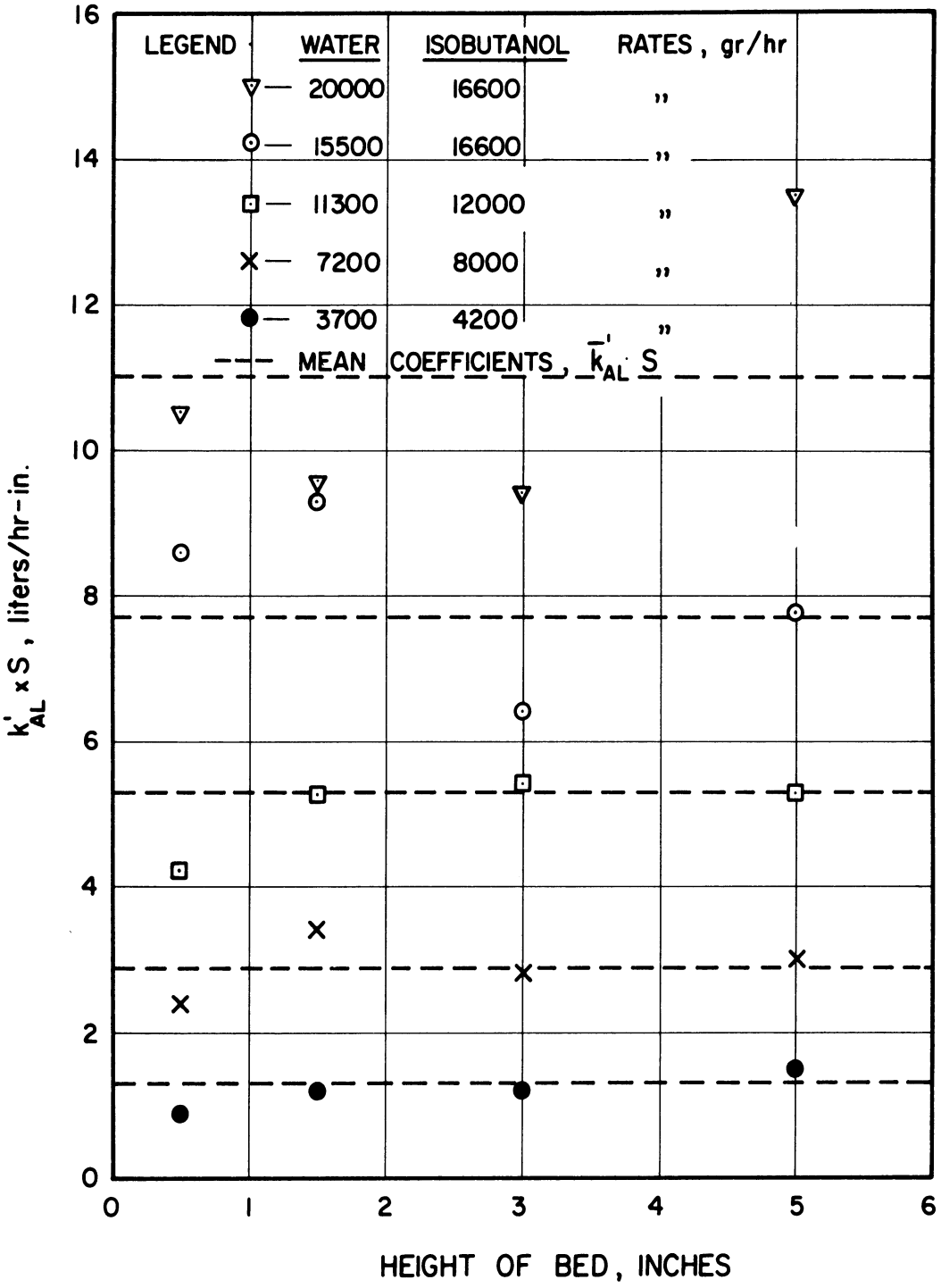


Figure 17. Incremental Water Phase Transfer Coefficients.

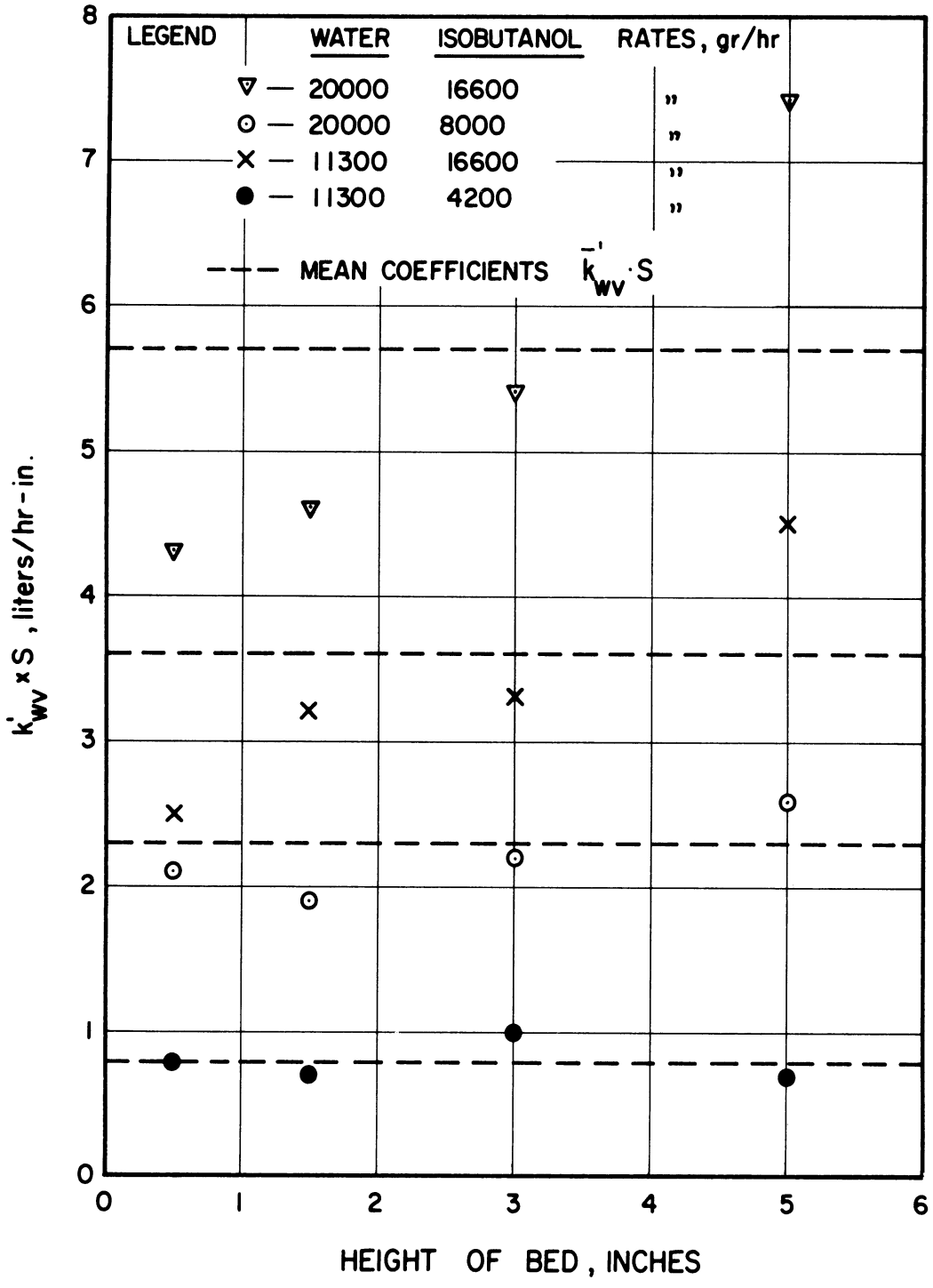


Figure 18. Incremental Isobutanol Phase Transfer Coefficients.

values are shown plotted in Figures 19 and 20. The water phase coefficients are shown in Figure 19 and those of the alcohol phase are shown in Figure 20. It is seen that their behavior is very regular, and that the average coefficients are functions of the flow rates of both phases.

Using a bivariate, least squares regression procedure with an equation of the type $y = Ax_1^n x_2^m$, (after taking logarithms) adequate representations for the dependence of the phase coefficients on the phase flow rates were obtained. For the water phase coefficient the equation is

$$\bar{k}'_{AL} = 0.00069(L_o)^{.71} (V_o)^{.78} \qquad (11)$$

and for the alcohol phase, the equation is

$$\bar{k}'_{WV} = 0.00056(L_o)^{.46} (V_o)^{.98} \qquad (12)$$

where $\bar{k}'_{\alpha\beta}$ has units of $(hr)^{-1}$ and L_o and V_o have units of $lbs/hr.ft^2$. The standard errors of the powers are 0.05 and 0.04 for Equation (11) and 0.06 and 0.05 for Equation (12). The coefficients of determination of the regression equations are 0.97 and 0.97 respectively, which are very respectable values for mass transfer data.

From Equation (11), it is seen that the water phase coefficient is influenced equally by the water and isobutanol flow rates and that the effect diminishes with increasing flow rate. From Equation (12), the isobutanol phase coefficient is much less affected by the water rate than the isobutanol rate, the coefficient increasing very nearly with the first power of the alcohol rate while increasing only with the square root of the water rate. Data for countercurrent flow follow much the same behavior.

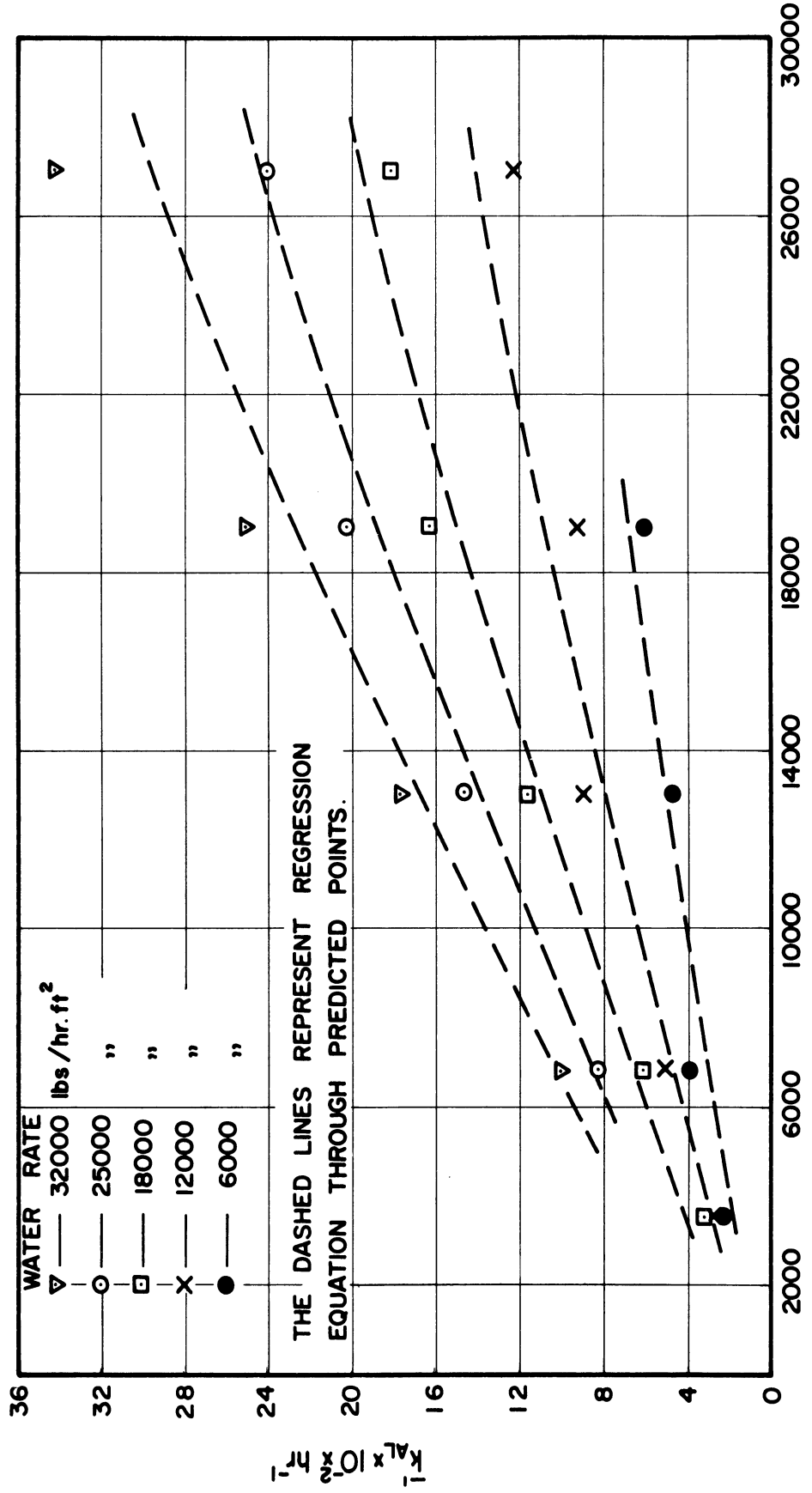


Figure 19. Averaged Water Phase Transfer Coefficients.

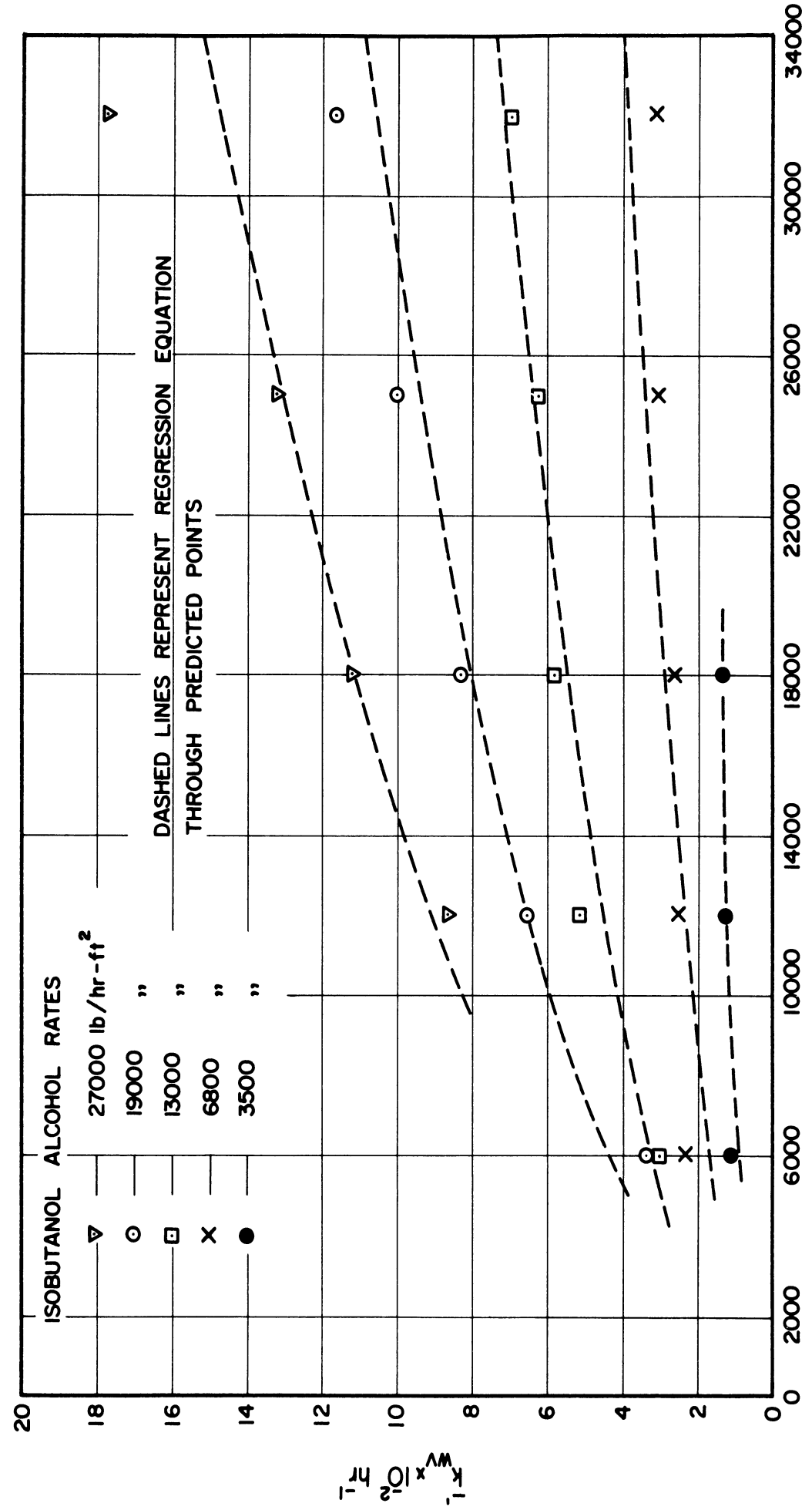


Figure 20. Averaged Isobutanol Phase Transfer Coefficients.

A comparison of the numerical values of the phase transfer coefficients obtained in concurrent flow with those obtained by Colburn and Welsh in countercurrent flow immediately indicate the possible advantages of concurrent flow. In countercurrent flow, the flow rates were restricted to 500-1,350 lbs/hr.ft^2 for the continuous water phase and to 250-2,090 lbs/hr.ft^2 for the discontinuous isobutanol phase. The transfer coefficients obtained, $\overline{k}'_{\alpha\beta}$, ranged from approximately 3 to 30 (hr)$^{-1}$ for the water phase and from 6 to 70 (hr)$^{-1}$ for the isobutanol phase.

For the range of flow rates studied in concurrent flow (6,000 to 32,000 lbs/hr.ft^2 for the water phase and 3,500 to 27,000 lbs/hr.ft^2 for the isobutanol phase) the phase coefficients ranged from 230 to 3,400 (hr)$^{-1}$ for the water phase and from 110 to 1770 (hr)$^{-1}$ for the isobutanol phase. The flow rates are much higher in the concurrent work than were allowable in the countercurrent work and the phase coefficients for the concurrent case are correspondingly much larger. From Equations (11) and (12) and from the plots of the coefficients, Figures 19 and 20, it would appear that the coefficients will continue to increase with increasing flow rate. Also, if Equations (11) and (12) are used to predict the concurrent transfer coefficients for flow rates used in the countercurrent work, the values are of comparable magnitude. For example, for a water rate of 1,350 lbs/hr.ft^2 and an isobutanol rate of 2,090 lbs/hr.ft^2, the transfer coefficients are 40 (hr)$^{-1}$ for the water phase and 35 (hr)$^{-1}$ for the isobutanol phase, as compared with coefficients of 30 and 70 (hr)$^{-1}$ respectively obtained in the countercurrent work. Therefore, the concurrent column, operating at very low phase flow rates with poor dispersion

still gives approximately the same coefficients as a countercurrent column operating at optimum flow rates. At higher flow rates the concurrent coefficients are much greater while the countercurrent column becomes inoperative due to flooding.

It is possible to calculate numbers for the transfer coefficients for water in the water-rich phase and for isobutanol in the isobutanol-rich phase. It is, however, not apparent that these coefficients have any utility, especially as they behave ambiguously as the bulk of one phase approaches equilibrium, while the other phase differs substantially from equilibrium, with the rate of transfer across the phase interface remaining finite. This anomalous behavior is apparent from an inspection of Equation (3), for taken literally, it shows that as the water phase becomes saturated, the rate of transfer of water, N_W, should become very small if the transfer coefficient k_{WL} remains relatively constant. However, it is known that water will transfer into the isobutanol-rich phase until this phase becomes saturated, regardless of the concentration driving force in the water-rich phase. Therefore, in this situation, the coefficient for water is the water-rich phase must increase appreciably. For example, for inlet flow rates of 20,000 gms/hr water and 16,600 gms/hr isobutanol, the incremental coefficients of water in the water-rich phase, $k'_{WL} * S$, and k'_{WL} for the column increments are:

	0-1 in	1-2 in	2-4 in	4-6 in	
$k'_{WL} * S$	6.7	10.0	16.3	54.5	liters/hr,in
k'_{WL}	2,070	3,090	5,040	16,800	hr^{-1} .

VIII. CONCLUSIONS

Several conclusions may be stated:

(1) Concurrent flow through a packed bed affords an effective means for obtaining high mass transfer coefficients in liquid systems. The coefficients for both phases increase as the flow rates of the phases are increased. The average column phase transfer coefficients can be expressed as a power function of both phase flow rates, $\bar{k}'_{\alpha\beta} = AL_o^n V_o^m$.

(2) The incremental phase transfer coefficients for the water phase are essentially independent of height in the column, excluding the first inch of bed, where the coefficient was generally a little lower than for succeeding increments.

(3) The incremental coefficients of the isobutanol phase appear to increase somewhat with bed height at higher flow rates, while at lower flow rates they can be considered constant. The increase is usually about 70% over the value of the coefficient of the first inch.

APPENDIX A

ORIGINAL VARIABLE COLUMN LENGTH DATA

APPENDIX A

TABLE A-1

ORIGINAL DATA FOR 1 INCH COLUMN

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	20,025	1.33608	16,400	1.39289	L
2	19,950	1.33578	16,530	1.39295	L
3	20,025	1.33485	11,870	1.39305	L
4	19,950	1.33489	11,700	1.39298	L
5	20,025	1.33428	7,830	1.39292	L
6	19,950	1.33415	7,710	1.39293	L
7	20,025	1.33354	4,125	1.39280	L
8	19,950	1.33349	4,100	1.39293	L
9	15,600	1.33614	16,590	1.39307	L
10	15,600	1.33472	12,030	1.39309	L
11	15,600	1.33417	7,920	1.39292	L
12	15,600	1.33332	4,225	1.39293	L
13	11,300	1.33595	16,475	1.39323	L
14	11,350	1.33535	16,550	1.39332	L
15	11,350	1.33495	12,025	1.39331	L
16	11,300	1.33504	11,900	1.39326	L
17	11,300	1.33503	11,950	1.39322	L
18	11,300	1.33440	7,875	1.39319	L
19	11,300	1.33433	7,850	1.39325	L
20	11,350	1.33453	8,050	1.39316	L
21	11,300	1.33440	8,000	1.39310	L
22	11,300	1.33444	7,800	1.39311	L
23	11,350	1.33329	4,125	1.39316	S
24	11,350	1.33323	2,140	1.39280	S
25	7,200	1.33578	16,560	1.39341	L
26	7,200	1.33498	11,480	1.39341	L
27	7,200	1.33472	7,860	1.39328	L
28	7,200	1.33466	7,900	1.39316	L
29	7,520	1.33491	7,880	1.39331	L
30	7,175	1.33349	4,000	1.39320	S
31	7,175	1.33323	2,240	1.39310	S
32	3,675	1.33569	11,975	1.39344	S
33	3,675	1.33492	8,060	1.39340	S
34	3,675	1.33423	4,360	1.39326	S
35	3,675	1.33375	2,230	1.39310	S

TABLE A-2

ORIGINAL DATA FOR 2 INCH COLUMN

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	20,000	1.33807	16,680	1.39216	L
2	19,980	1.33780	16,620	1.39202	L
3	19,980	1.33685	11,920	1.39220	L
4	20,000	1.33705	11,870	1.39217	L
5	20,000	1.33585	8,190	1.39229	L
6	19,980	1.33589	7,800	1.39202	L
7	19,980	1.33471	4,250	1.39189	L
8	20,000	1.33465	4,100	1.39213	L
9	15,300	1.33852	16,700	1.39210	L
10	15,300	1.33743	12,030	1.39226	L
11	15,300	1.33614	7,950	1.39213	L
12	15,300	1.33479	4,225	1.39194	L
13	15,500	1.33484	4,180	1.39174	L
14	11,075	1.33877	16,625	1.39256	L
15	11,075	1.33850	16,600	1.39268	L
16	11,375	1.33817	16,550	1.39277	L
17	11,375	1.33717	12,000	1.39280	L
18	11,075	1.33742	11,975	1.39259	L
19	11,075	1.33746	12,075	1.39274	L
20	11,075	1.33633	7,925	1.39259	L
21	11,375	1.33624	7,875	1.39268	L
22	11,075	1.33608	7,950	1.39256	L
23	11,300	1.33611	8,075	1.39268	L
24	11,190	1.33601	8,070	1.39280	S
25	11,300	1.33620	7,950	1.39246	S
26	11,350	1.33608	7,960	1.39271	S
27	11,350	1.33446	4,200	1.39250	S
28	11,190	1.33453	4,275	1.39276	S
29	11,190	1.33381	2,200	1.39251	S
30	11,350	1.33341	2,275	1.39298	S
31	7,200	1.33887	16,575	1.39286	L
32	7,430	1.33897	16,625	1.39301	L
33	7,430	1.33827	12,060	1.39280	L
34	7,200	1.33829	12,160	1.39273	L
35	7,200	1.33720	8,060	1.39262	L

TABLE A-2 CONT'D

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
36	7,430	1.33727	8,040	1.39259	L
37	7,200	1.33711	8,100	1.39262	S
38	7,200	1.33533	4,125	1.39247	S
39	7,200	1.33401	2,180	1.39249	S
40	3,650	1.33795	12,100	1.39341	S
41	3,620	1.33817	11,960	1.39337	S
42	3,650	1.33704	8,020	1.39324	S
43	3,620	1.33743	7,940	1.39322	S
44	3,620	1.33634	4,220	1.39297	S
45	3,650	1.33608	4,400	1.39299	S
46	3,650	1.33472	2,165	1.39292	S
47	3,620	1.33472	2,280	1.39290	S

TABLE A-3

ORIGINAL DATA FOR 4 INCH COLUMN

Sample	Water Phase		Isobutanol Phase		
	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	19,500	1.34013	16,600	1.39057	L
2	19,960	1.34001	16,530	1.39038	L
3	19,960	1.33935	11,800	1.39039	L
4	19,300	1.33929	11,900	1.39077	L
5	19,350	1.33813	7,860	1.39068	L
6	19.960	1.33813	7,830	1.39050	L
7	19,960	1.33638	4,075	1.39017	L
8	19,500	1.33636	4,075	1.39042	L
9	15,480	1.34015	16,620	1.39098	L
10	15,480	1.33929	12,000	1.39117	L
11	15,450	1.33624	4,050	1.39098	L
12	15,390	1.33816	7,710	1.39110	L
13	10,725	1.34045	16,200	1.39153	L
14	11,250	1.34029	16,550	1.39159	L
15	11,250	1.33974	11,900	1.39129	L
16	10,600	1.33994	11,925	1.39141	L
17	11,000	1.33883	7,850	1.39129	L
18	11,250	1.33882	7,950	1.39105	L
19	11,600	1.33855	7,770	1.39135	S
20	11,400	1.33849	7,680	1.39115	S
21	11,400	1.33650	4,075	1.39108	S
22	12,325	1.33662	4,075	1.39113	S
23	11,400	1.33466	2,150	1.39145	S
24	11,100	1.33467	2,050	1.39147	S
25	6,680	1.34070	16,660	1.39207	L
26	6,800	1.34029	11,960	1.39198	L
27	6,940	1.33955	7,780	1.39177	L
28	7,100	1.33971	7,950	1.39095	S
29	8,500	1.33893	7,850	1.39159	S
30	9,350	1.33720	4,075	1.39129	S
31	7,100	1.33810	4,275	1.39077	S
32	7,100	1.33585	2,140	1.39086	S
33	8,880	1.33488	2,185	1.39129	S
34	4,400	1.34061	12,000	1.39237	S
35	3,640	1.34051	12,240	1.39280	S
36	3,640	1.33981	7,920	1.39238	S
37	4,400	1.33994	7,620	1.39210	S
38	3,640	1.33871	4,140	1.39180	S
39	5,180	1.33798	4,060	1.39189	S
40	3,640	1.33665	2,080	1.39171	S
41	4,460	1.33633	2,060	1.39189	S

TABLE A-4

ORIGINAL DATA 6 INCH COLUMN

Sample	Water Phase		Isobutanol Phase		
	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	19,600	1.34109	16,700	1.38922	L
2	19,600	1.34058	11,870	1.38932	L
3	19,960	1.34038	12,200	1.38941	L
4	20,000	1.34038	11,730	1.38925	L
5	19,600	1.33961	7,920	1.38935	L
6	19,960	1.33932	7,800	1.38956	L
7	20,000	1.33938	7,800	1.38932	L
8	19,960	1.33762	4,100	1.38940	L
9	20,000	1.33766	4,150	1.38928	L
10	19,600	1.33752	4,025	1.38946	L
11	15,200	1.34099	16,500	1.38992	L
12	15,200	1.34064	11,820	1.38970	L
13	15,200	1.33955	7,440	1.38977	L
14	15,200	1.33768	3,950	1.38973	L
15	11,000	1.34102	16,400	1.39038	L
16	11,325	1.34095	16,575	1.39041	L
17	11,000	1.34072	11,550	1.39028	L
18	11,325	1.34077	12,000	1.39005	L
19	11,325	1.34063	12,325	1.39045	L
20	11,000	1.34000	7,450	1.39016	L
21	11,325	1.34013	7,700	1.38973	L
22	11,325	1.33984	7,575	1.39038	L
23	11,000	1.34019	7,560	1.38989	S
24	11,300	1.33987	7,900	1.39008	L
25	11,300	1.33975	7,900	1.39025	S
26	11,000	1.33834	4,050	1.39007	S
27	11,250	1.33791	4,200	1.39042	S
28	11,300	1.33794	4,225	1.39020	S
29	11,300	1.33556	2,150	1.39026	S
30	11,000	1.33586	1,960	1.39016	S
31	7,000	1.34109	16,460	1.39099	L
32	7,000	1.34099	11,800	1.39086	L
33	7,000	1.34061	7,640	1.39039	L
34	6,950	1.34058	7,575	1.39038	S
35	6,950	1.33897	3,975	1.39031	S

TABLE A-4 CONT'D

Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
36	7,250	1.33865	4,150	1.39065	S
37	6,950	1.33643	1,920	1.39047	S
38	3,400	1.34109	11,800	1.39220	S
39	3,400	1.34096	7,500	1.39141	S
40	3,740	1.34096	8,080	1.39156	S
41	3,740	1.34006	4,180	1.39080	S
42	3,400	1.34038	3,800	1.39068	S
43	3,400	1.33821	1.960	1.39091	S
44	3,740	1.33804	2,000	1.39084	S

APPENDIX B

VARIABLE COLUMN LENGTH DATA CORRECTED TO NOMINAL FLOW RATES

APPENDIX B

TABLE B-1

FLOW RATE CORRECTED DATA FOR 1 INCH COLUMN

	Water Phase			Isobutanol Phase		
No.	L_o gms/hr	Index of Refraction	Wt. Fr. H_2O	V_o gms/hr	Index of Refraction	Wt. Fr. H_2O
1	20,000	1.33608	.9650	16,600	1.39289	.0335
2	20,000	1.33580	.9678	16,000	1.39295	.0315
3	20,000	1.33488	.9760	12,000	1.39305	.0280
4	20,000	1.33490	.9758	12,000	1.39298	.0300
5	20,000	1.33425	.9818	8,000	1.39292	.0330
6	20,000	1.33420	.9824	8,000	1.39293	.0330
7	20,000	1.33352	.9890	4,200	1.39280	.0370
8	20,000	1.33350	.9893	4,200	1.39290	.0335
9	15,500	1.33610	.9648	16,600	1.39307	.0275
10	15,500	1.33472	.9775	12,000	1.39309	.0275
11	15,500	1.33420	.9824	8,000	1.39292	.0330
12	15,500	1.33332	.9910	4,200	1.39293	.0330
13	11,300	1.33595	.9663	16,600	1.39323	.0215
14	11,300	1.33535	.9717	16,600	1.39332	.0180
15	11,300	1.33495	.9753	12,000	1.39331	.0180
16	11,300	1.33504	.9745	12,000	1.39326	.0205
17	11,300	1.33503	.9747	12,000	1.39322	.0220
18	11,300	1.33440	.9805	8,000	1.39319	.0230
19	11,300	1.33433	.9810	8,000	1.39325	.0205
20	11,300	1.33450	.9795	8,000	1.39316	.0240
21	11,300	1.33440	.9805	8,000	1.39310	.0260
22	11,300	1.33444	.9800	8,000	1.39311	.0260
23	11,300	1.33335	.9907	4,200	1.39316	.0240
24	11,300	1.33325	.9917	2,200	1.39280	.0370
25	7,200	1.33578	.9680	16,600	1.39341	.0145
26	7,200	1.33505	.9745	12,000	1.39341	.0145
27	7,200	1.33475	.9772	8,000	1.39328	.0195
28	7,200	1.33470	.9776	8,000	1.39316	.0240
29	7,200	1.33490	.9758	8,000	1.39331	.0180
30	7,200	1.33352	.9890	4,200	1.39320	.0225
31	7,200	1.33320	.9922	2,200	1.39310	.0260
32	3,700	1.33569	.9687	12,000	1.39344	.0140
33	3,700	1.33490	.9758	8,000	1.39340	.0150
34	3,700	1.33415	.9828	4,200	1.39326	.0200
35	3,700	1.33370	.9873	2,200	1.39310	.0260

TABLE B-2

FLOW RATE CORRECTED DATA FOR 2 INCH COLUMN

	Water Phase			Isobutanol Phase		
No.	L_o gms/hr	Index of Refraction	Wt. Fr. H_2O	V_o gms/hr	Index of Refraction	Wt. Fr. H_2O
1	20,000	1.33805	.9467	16,600	1.39216	.0585
2	20,000	1.33780	.9490	16,600	1.39202	.0625
3	20,000	1.33690	.9577	12,000	1.39220	.0570
4	20,000	1.33710	.9557	12,000	1.39217	.0580
5	20,000	1.33580	.9677	8,000	1.39229	.0545
6	20,000	1.33593	.9665	8,000	1.39202	.0625
7	20,000	1.33470	.9777	4,200	1.39189	.0665
8	20,000	1.33468	.9778	4,200	1.39213	.0595
9	15,500	1.33848	.9427	16,600	1.39210	.0605
10	15,500	1.33740	.9529	12,000	1.39226	.0555
11	15,500	1.33614	.9645	8,000	1.39213	.0595
12	15,500	1.33479	.9768	4,200	1.39194	.0650
13	15,500	1.33484	.9765	4,200	1.39174	.0710
14	11,300	1.33870	.9405	16,600	1.39256	.0455
15	11,300	1.33845	.9429	16,600	1.39268	.0415
16	11,300	1.33825	.9448	16,600	1.39277	.0380
17	11,300	1.33720	.9548	12,000	1.39280	.0370
18	11,300	1.33738	.9530	12,000	1.39259	.0450
19	11,300	1.33740	.9528	12,000	1.39274	.0390
20	11,300	1.33630	.9630	8,000	1.39259	.0450
21	11,300	1.33630	.9630	8,000	1.39268	.0415
22	11,300	1.33610	.9650	8,000	1.39256	.0455
23	11,300	1.33610	.9650	8,000	1.39268	.0415
24	11,300	1.33610	.9650	8,000	1.39280	.0370
25	11,300	1.33620	.9638	8,000	1.39246	.0490
26	11,300	1.33612	.9646	8,000	1.39271	.0405
27	11,300	1.33446	.9798	4,200	1.39250	.0475
28	11,300	1.33453	.9793	4,200	1.39276	.0385
29	11,300	1.33381	.9867	2,200	1.39251	.0470
30	11,300	1.33340	.9903	2,200	1.39298	.0305
31	7,200	1.33887	.7389	16,600	1.39286	.0350
32	7,200	1.33900	.9377	16,600	1.39301	.0295
33	7,200	1.33825	.9448	12,000	1.39280	.0370
34	7,200	1.33825	.9448	12,000	1.39273	.0400
35	7,200	1.33720	.9548	8,000	1.39262	.0435
36	7,200	1.33727	.9540	8,000	1.39259	.0450

TABLE B-2 CONT'D

	Water Phase			Isobutanol Phase		
No.	L_o gms/hr	Index of Refraction	Wt. Fr. H_2O	V_o gms/hr	Index of Refraction	Wt. Fr. H_2O
37	7,200	1.33710	.9557	8,000	1.39262	.0435
38	7,200	1.33535	.9718	4,200	1.39247	.0485
39	7,200	1.33405	.9838	2,200	1.39249	.0480
40	3,700	1.33794	.9477	12,000	1.39341	.0150
41	3,700	1.33820	.9453	12,000	1.39337	.0160
42	3,700	1.33705	.9562	8,000	1.39324	.0210
43	3,700	1.33745	.9525	8,000	1.39322	.0215
44	3,700	1.33630	.9631	4,200	1.39297	.0310
45	3,700	1.33600	.9657	4,200	1.39299	.0300
46	3,700	1.33475	.9771	2,200	1.39292	.0330
47	3,700	1.33470	.9776	2,200	1.39290	.0335

TABLE B-3

FLOW RATE CORRECTED DATA FOR 4 INCH COLUMN

	Water Phase			Isobutanol Phase		
No.	L_o gms/hr	Index of Refraction	Wt. Fr. H_2O	V_o gms/hr	Index of Refraction	Wt. Fr. H_2O
1	20,000	1.34010	.9273	16,600	1.39057	.1020
2	20,000	1.34003	.9280	16,600	1.39038	.1065
3	20,000	1.33940	.9340	12,000	1.39039	.1065
4	20,000	1.33930	.9348	12,000	1.39077	.0970
5	20,000	1.33820	.9453	8,000	1.39068	.0990
6	20,000	1.33820	.9453	8,000	1.39050	.1038
7	20,000	1.33643	.9620	4,200	1.39017	.1120
8	20,000	1.33643	.9620	4,200	1.39040	.1060
9	15,500	1.34015	.9268	16,600	1.39098	.0915
10	15,500	1.33929	.9350	12,000	1.39117	.0865
11	15,500	1.33825	.9448	8,000	1.39110	.0885
12	15,500	1.33635	.9628	4,200	1.39098	.0915
13	11,300	1.34040	.9245	16,600	1.39153	.0765
14	11,300	1.34030	.9253	16,600	1.39159	.0750
15	11,300	1.33977	.9304	12,000	1.39129	.0830
16	11,300	1.33990	.9292	12,000	1.39141	.0800
17	11,300	1.33885	.9391	8,000	1.39129	.0830
18	11,300	1.33882	.9395	8,000	1.39105	.1038
19	11,300	1.33870	.9405	8,000	1.39135	.0818
20	11,300	1.33860	.9415	8,000	1.39115	.0870
21	11,300	1.33665	.9600	4,200	1.39108	.0890
22	11,300	1.33685	.9580	4,200	1.39120	.0860
23	11,300	1.33470	.9776	2,200	1.39145	.0790
24	11,300	1.33475	.9771	2,200	1.39147	.0785
25	7,200	1.34065	.9220	16,600	1.39207	.0610
26	7,200	1.34025	.9258	12,000	1.39198	.0640
27	7,200	1.33960	.9320	8,000	1.39177	.0700
28	7,200	1.33970	.9310	8,000	1.39095	.0922
29	7,200	1.33930	.9348	8,000	1.39170	.0720
30	7,200	1.33775	.9495	4,200	1.39150	.0775
31	7,200	1.33800	.9472	4,200	1.39077	.0970
32	7,200	1.33585	.9674	2,200	1.39086	.0945
33	7,200	1.33560	.9695	2,200	1.39145	.0790
34	3,700	1.34065	.9220	12,000	1.39250	.0475
35	3,700	1.34047	.9238	12,000	1.39280	.0370
36	3,700	1.33983	.9298	8,000	1.39238	.0515
37	3,700	1.34005	.9278	8,000	1.39220	.0572
38	3,700	1.33875	.9400	4,200	1.39180	.0690
39	3,700	1.33845	.9428	4,200	1.39200	.0635
40	3,700	1.33675	.9590	2,200	1.39171	.0715
41	3,700	1.33675	.9590	2,200	1.39200	.0635

TABLE B-4

FLOW RATE CORRECTED DATA FOR 6 INCH COLUMN

No.	Water Phase			Isobutanol Phase		
	L_o gms/hr	Index of Refraction	Wt. Fr. H_2O	V_o gms/hr	Index of Refraction	Wt. Fr. H_2O
1	20,000	1.34109	.9180	16,600	1.38922	.1355
2	20,000	1.34058	.9227	12,000	1.38932	.1330
3	20,000	1.34038	.9246	12,000	1.38941	.1308
4	20,000	1.34045	.9240	12,000	1.38925	.1348
5	20,000	1.33960	.9320	8,000	1.38935	.1320
6	20,000	1.33937	.9342	8,000	1.38956	.1270
7	20,000	1.33945	.9335	8,000	1.38928	.1340
8	20,000	1.33768	.9502	4,200	1.38940	.1310
9	20,000	1.33768	.9502	4,200	1.38928	.1340
10	20,000	1.33765	.9505	4,200	1.38946	.1295
11	15,500	1.34099	.9188	16,600	1.38992	.1180
12	15,500	1.34064	.9221	12,000	1.38970	.1235
13	15,500	1.33975	.9306	8,000	1.38977	.1220
14	15,500	1.33785	.9485	4,200	1.38973	.1230
15	11,300	1.34102	.9185	16,600	1.39038	.1065
16	11,300	1.34095	.9192	16,600	1.39041	.1060
17	11,300	1.34075	.9212	12,000	1.39028	.1095
18	11,300	1.34077	.9210	12,000	1.39005	.1150
19	11,300	1.34063	.9222	12,000	1.39045	.1050
20	11,300	1.34010	.9273	8,000	1.39016	.1120
21	11,300	1.34018	.9265	8,000	1.38973	.1230
22	11,300	1.34000	.9283	8,000	1.39038	.1065
23	11,300	1.34028	.9255	8,000	1.38989	.1190
24	11,300	1.33990	.9292	8,000	1.39009	.1140
25	11,300	1.33980	.9300	8,000	1.39025	.1100
26	11,300	1.33835	.9438	4,200	1.39007	.1140
27	11,300	1.33791	.9480	4,200	1.39042	.1055
28	11,300	1.33795	.9476	4,200	1.39020	.1110
29	11,300	1.33570	.9685	2,200	1.39026	.1095
30	11,300	1.33605	.9653	2,200	1.39016	.1120
31	7,200	1.34110	.9177	16,600	1.39099	.0910
32	7,200	1.34100	.9188	12,000	1.39086	.0945
33	7,200	1.34065	.9220	8,000	1.39039	.1065
34	7,200	1.34068	.9218	8,000	1.39038	.1065
35	7,200	1.33905	.9373	4,200	1.39031	.1085

TABLE B-4 CONT'D

No.	Water Phase			Isobutanol Phase		
	L_o gms/hr	Index of Refraction	Wt. Fr. H_2O	V_o gms/hr	Index of Refraction	Wt. Fr. H_2O
36	7,200	1.33868	.9407	4,200	1.39065	.1000
37	7,200	1.33680	.9585	2,200	1.39047	.1045
38	3,700	1.34110	.9177	12,000	1.39220	.0570
39	3,700	1.34095	.9192	8,000	1.39141	.0800
40	3,700	1.34095	.9192	8,000	1.39156	.0760
41	3,700	1.34010	.9273	4,200	1.39080	.0960
42	3,700	1.34040	.9244	4,200	1.39068	.0990
43	3,700	1.33855	.9420	2,200	1.39090	.0935
44	3,700	1.33835	.9438	2,200	1.39084	.0950

APPENDIX C

ORIGINAL RECEIVER EFFECT EXPERIMENTAL DATA

APPENDIX C

TABLE C-1

ORIGINAL DATA: 1 INCH COLUMN, PURE WATER - 40% SATURATED ISOBUTANOL INLET
INLET REFRACTIVE INDICES: WATER - 1.33245, ISOBUTANOL - 1.39195

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	7,100	1.33394	4,060	1.39141	L
2	7,100	1.33456	7,660	1.39148	L
3	7,100	1.33572	11,880	1.39147	L
4	11,150	1.33504	11,880	1.39147	L
5	11,150	1.33582	16,625	1.39141	L
6	11,150	1.33407	8,000	1.39146	L
7	15,200	1.33443	8,000	1.39177	L
8	15,200	1.33342	4,175	1.39123	L
9	15,200	1.33530	12,000	1.39117	L
10	15,200	1.33613	16,550	1.39117	L
11	7,220	1.33614	16,550	1.39150	L
12	19,600	1.33588	16,550	1.39104	L
13	19,600	1.33508	11,870	1.39111	L
14	19,600	1.33420	7,800	1.39106	L
15	19,600	1.33362	4,200	1.39087	L
16	11,350	1.33375	4,200	1.39120	S
17	11,350	1.33417	7,920	1.39135	S
18	11,350	1.33303	2,130	1.39126	S
19	7,350	1.33332	2,130	1.39126	S
20	7,350	1.33383	4,175	1.39129	S
21	7,350	1.33456	8,100	1.39144	S
22	3,800	1.33537	8,100	1.39153	S
23	3,800	1.33604	12,000	1.39159	S
24	3,800	1.33404	4,000	1.39150	S
25	3,800	1.33381	2,120	1.39132	S

TABLE C-2

ORIGINAL DATA: 1 INCH COLUMN, PURE WATER - 80% SATURATED ISOBUTANOL INLET
INLET REFRACTIVE INDICES: WATER - 1.33245, ISOBUTANOL - 1.38955

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	7,300	1.33517	7,460	1.38919	L
2	7,300	1.33595	11,980	1.38917	L
3	7,300	1.33669	16,600	1.38922	L
4	11,325	1.33646	16,600	1.38916	L
5	11,325	1.33556	11,960	1.38913	L
6	11,325	1.33463	8,050	1.38904	L
7	15,400	1.33466	8,050	1.38892	L
8	15,400	1.33356	4,225	1.38883	L
9	15,400	1.33562	12,000	1.38890	L
10	15,400	1.33552	12,000	1.38904	L
11	19,600	1.33640	16,530	1.38895	L
12	19,600	1.33562	11,930	1.38895	L
13	19,600	1.33459	7,860	1.38889	L
14	19,600	1.33368	4,250	1.38886	L
15	11,300	1.33359	4,250	1.38897	S
16	11,300	1.33456	8,140	1.38907	S
17	11,300	1.33315	2,340	1.38890	S
18	7,250	1.33322	2,240	1.38899	S
19	7,250	1.33404	4,300	1.38901	S
20	7,250	1.33507	4,300	1.38901	S
21	3,900	1.33598	7,980	1.38913	S
22	3,900	1.33659	12,000	1.38925	S
23	3,900	1.33453	4,175	1.38913	S
24	3,900	1.33374	2,260	1.38911	S

TABLE C-3

ORIGINAL DATA: 1 INCH COLUMN, 40% SATURATED WATER-PURE ISOBUTANOL INLET
INLET REFRACTIVE INDICES: WATER - 1.33568, ISOBUTANOL - 1.39380

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	7,420	1.33675	7,560	1.39325	L
2	7,420	1.33720	11,900	1.39331	L
3	7,420	1.33762	16,700	1.39331	L
4	11,450	1.33771	16,700	1.39316	L
5	11,450	1.33701	11,650	1.39319	L
6	11,450	1.33659	7,950	1.39316	L
7	15,500	1.33659	7,950	1.39301	L
8	15,500	1.33621	4,175	1.39295	L
9	15,500	1.33698	12,030	1.39307	L
10	15,500	1.33749	16,500	1.39301	L
11	19,975	1.33727	16,500	1.39277	L
12	19,975	1.33711	11,880	1.39289	L
13	19,975	1.33662	7,920	1.39287	L
14	19,975	1.33627	4,225	1.39253	L
15	11,450	1.33614	4,225	1.39316	S
16	11,450	1.33646	7,920	1.39322	S
17	11,450	1.33601	2,200	1.39307	S
18	7,450	1.33604	2,200	1.39316	S
19	7,450	1.33633	4,300	1.39319	S
20	7,450	1.33659	7,900	1.39331	S
21	3,700	1.33711	7,900	1.39331	S
22	3,700	1.33759	11,975	1.39337	S
23	3,700	1.33669	4,175	1.39325	S
24	3,700	1.33636	2,220	1.39316	S

TABLE C-4

ORIGINAL DATA: 1 INCH COLUMN, 40% SATURATED WATER - 40% SATURATED ISOBUTANOL INLET. INLET REFRACTIVE INDICES: WATER - 1.33568, ISOBUTANOL - 1.39195

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	7,550	1.33706	7,500	1.39150	L
2	7,550	1.33750	12,000	1.39153	L
3	7,550	1.33813	16,600	1.39156	L
4	11,550	1.33797	16,600	1.39132	L
5	11,550	1.33720	11,900	1.39138	L
6	11,550	1.33662	7,900	1.39138	L
7	15,700	1.33675	7,900	1.39120	L
8	15,700	1.33617	4,175	1.39120	L
9	15,700	1.33717	12,000	1.39132	L
10	15,700	1.33788	16,600	1.39123	L
11	19,950	1.33791	16,600	1.39105	L
12	19,950	1.33724	12,000	1.39108	L
13	19,950	1.33688	7,920	1.39099	L
14	19,950	1.33617	4,200	1.39102	L
15	11,350	1.33617	4,200	1.39129	S
16	11,350	1.33677	7,830	1.39144	S
17	11,350	1.33591	2,300	1.39135	S
18	7,400	1.33607	2,300	1.39141	S
19	7,400	1.33641	4,200	1.39141	S
20	7,400	1.33691	7,880	1.39150	S
21	3,700	1.33756	7,880	1.39151	S
22	3,700	1.33807	11,975	1.39153	S
23	3,700	1.33677	4,175	1.39159	S
24	3,700	1.33633	2,260	1.39147	S

TABLE C-5

ORIGINAL DATA: 1 INCH COLUMN, 40% SATURATED WATER - 80% SATURATED ISOBUTANOL INLET. INLET REFRACTIVE INDICES: WATER - 1.33568, ISOBUTANOL - 1.38955

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	6,900	1.33730	7,860	1.38934	L
2	6,900	1.33771	11,980	1.38931	L
3	6,900	1.33829	16,750	1.38934	L
4	11,400	1.33823	16,750	1.38921	L
5	11,400	1.33740	11,925	1.38927	L
6	11,400	1.33678	8,000	1.38928	L
7	15,500	1.33678	8,000	1.38913	L
8	15,500	1.33627	4,325	1.38913	L
9	15,500	1.33737	12,000	1.38917	L
10	15,500	1.33810	16,620	1.38916	L
11	19,800	1.33775	12,800	1.38907	L
12	19,800	1.33665	8,040	1.38916	L
13	19,800	1.33624	4,200	1.38910	L
14	11,600	1.33624	4,200	1.38919	S
15	11,600	1.33672	7,890	1.38925	S
16	11,600	1.33588	2,240	1.38922	S
17	7,250	1.33604	2,240	1.38923	S
18	7,250	1.33646	4,300	1.38922	S
19	7,250	1.33724	7,950	1.38922	S
20	3,725	1.33788	7,950	1.38925	S
21	3,725	1.33788	11,975	1.38939	S
22	3,725	1.33688	4,175	1.38930	S
23	3,725	1.33627	2,220	1.38932	S

TABLE C-6

ORIGINAL DATA: 1 INCH COLUMN, 80% SATURATED WATER-PURE ISOBUTANOL INLET
INLET REFRACTIVE INDICES: WATER - 1.33870, ISOBUTANOL - 1.39380

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	7,075	1.33922	7,980	1.39331	L
2	7,075	1.33955	11,920	1.39325	L
3	7,075	1.33981	16,550	1.39331	L
4	11,400	1.33961	16,550	1.39322	L
5	11,400	1.33935	11,925	1.39317	L
6	11,400	1.33913	8,000	1.39319	L
7	15,700	1.33915	8,000	1.39295	L
8	15,700	1.33897	4,200	1.39280	L
9	15,700	1.33934	11,900	1.39301	L
10	15,700	1.33971	16,600	1.39301	L
11	20,250	1.33994	16,600	1.39268	L
12	20.250	1.33955	12,000	1.39265	L
13	20,250	1.33925	8,220	1.39262	L
14	20.250	1.33874	4,230	1.39274	L
15	11,500	1.33903	4,230	1.39295	S
16	11,500	1.33903	8,070	1.39322	S
17	11,500	1.33890	2,180	1.39280	S
18	7,300	1.33893	2,180	1.39307	S
19	7,300	1.33908	4,275	1.39298	S
20	7,300	1.33919	7,960	1.39331	S
21	3,550	1.33935	7,960	1.39331	S
22	3,550	1.33958	12,000	1.39337	S
23	3,550	1.33916	4,200	1.39325	S
24	3,550	1.33903	2,200	1.39316	S

TABLE C-7

ORIGINAL DATA: 1 INCH COLUMN, 80% SATURATED WATER - 40% SATURATED ISOBUTANOL INLET. INLET REFRACTIVE INDICES: WATER - 1.33870, ISOBUTANOL - 1.39195

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	7,360	1.33932	7,540	1.39142	L
2	7,360	1.33942	12,000	1.39154	L
3	7,360	1.33948	16,600	1.39165	L
4	11,500	1.33948	16,600	1.39153	L
5	11,500	1.33930	11,900	1.39153	L
6	11,500	1.33904	7,925	1.39153	L
7	15,800	1.33906	7,925	1.39141	L
8	15,800	1.33897	4,125	1.39111	L
9	15,800	1.33947	11,880	1.39117	L
10	15,800	1.33968	16,620	1.39132	L
11	20,200	1.33987	16,620	1.39108	L
12	20,200	1.33948	11,900	1.39111	L
13	20,200	1.33925	8,010	1.39105	L
14	20,200	1.33900	4,175	1.39086	L
15	11,500	1.33900	4,175	1.39117	S
16	11,500	1.33915	7,890	1.39132	S
17	11,500	1.33884	2,270	1.39117	S
18	7,340	1.33887	2,270	1.39123	S
19	7,340	1.33903	4,225	1.39129	S
20	7,340	1.33917	7,870	1.39147	S
21	3,650	1.33945	7,870	1.39153	S
22	3,650	1.33961	12,025	1.39162	S
23	3,650	1.33917	4,100	1.39141	S
24	3,650	1.33897	2,160	1.39147	S

TABLE C-8

ORIGINAL DATA: 1 INCH COLUMN, 80% SATURATED WATER - 80% SATURATED ISOBUTANOL INLET. INLET REFRACTIVE INDICES: WATER - 1.33870, ISOBUTANOL - 1.38955

	Water Phase		Isobutanol Phase		
Sample	Flow Rate gms/hr	Index of Refraction	Flow Rate gms/hr	Index of Refraction	Receiver Size
1	7,400	1.33922	7,520	1.38931	L
2	7,400	1.33935	11,900	1.38935	L
3	7,400	1.33961	16,600	1.38937	L
4	11,500	1.33955	16,600	1.38933	L
5	11,500	1.33928	11,950	1.38933	L
6	11,500	1.33913	7,900	1.38923	L
7	15,700	1.33916	7,900	1.38907	L
8	15,700	1.33897	4,225	1.38899	L
9	15,700	1.33929	11,940	1.38916	L
10	15,700	1.33955	16,600	1.38917	L
11	20,125	1.33961	16,600	1.38916	L
12	20,125	1.33929	12,030	1.38913	L
13	20,125	1.33897	7,770	1.38922	L
14	20,125	1.33896	4,275	1.38898	L
15	11,500	1.33903	4,275	1.38904	S
16	11,500	1.33909	7,920	1.38922	S
17	11,500	1.33884	2,210	1.38904	S
18	7,325	1.33893	2,210	1.38907	S
19	7,325	1.33890	4,225	1.38919	S
20	7,325	1.33921	7,880	1.38929	S
21	3,700	1.33938	7,880	1.38922	S
22	3,700	1.33968	11,925	1.38937	S
23	3,700	1.33909	4,200	1.38925	S
24	3,700	1.33894	2,280	1.38916	S

APPENDIX D

CALCULATION OF PHASE TRANSFER COEFFICIENTS

APPENDIX D

CALCULATION OF PHASE TRANSFER COEFFICIENTS

Regardless of the procedure used to compute the phase transfer coefficients k'_{AL} and k'_{WV}, it is necessary to calculate, from the phase compositions tabulated in Appendix B, the following information for each phase, at each bed height and for each combination of flow rates (cf. Table I). For the water-rich phase;

(1) the number of moles of isobutanol flowing - Lx_A

(2) the molal concentration of isobutanol - C_{AL}

(3) the concentration driving force for the transfer of isobutanol $(C^*_{AL} - C_{AL})$.

For the isobutanol-rich phase;

(1) the number of moles of water flowing - Vy_W

(2) the molal concentration of water - C_{WV}

(3) the driving force for the transfer of water into the isobutanol $(C^*_{WV} - C_{WV})$.

The tabulated mass fractions were first converted to mole fractions by the relation,

$$x_W = X_W/(0.75676X_W + 0.24324)$$

$$y_W = Y_W/(0.75676Y_W + 0.24324)$$

where x_W, y_W = the mole fraction of water in the water-rich and isobutanol-rich phases respectively and X_W, Y_W = the mass fraction of water in the water-rich and isobutanol-rich phases respectively. Of course, x_A and y_A are found by $(1.0 - x_W)$ and $(1.0 - y_W)$.

The moles of a component, say water flowing in the alcohol phase, was computed using Equation (8) and multiplying by y_W, or

$$V = \frac{L_o - x_W(L_o + V_o)}{(y_W - x_W)}$$

and the moles of water equals Vy_W. The molal concentrations were computed using the relations

$$C_{AL} = x_A/(0.0925\ x_A + 0.018\ x_W)$$

and

$$C_{WV} = y_W/(0.0925\ y_A + 0.018\ y_W) \quad .$$

The molal concentrations for the saturated phases were computed to be

$$C^*_{AL} = 1.10 \text{ gm moles/liter}$$

and

$$C^*_{WV} = 7.68 \text{ gm moles/liter.}$$

The values of Lx_A, Vy_W, C_{AL}, C_{WV}, $(C^*_{AL} - C_{AL})$ and $(C^*_{WV} - C_{WV})$ were computed for all the data points tabulated in Appendix B on the IBM 704. The execution time was approximately one minute. If there were duplicate values of Lx_A, Vy_W, ΔC_{AL} and ΔC_{WV} at any flow condition, the arithmetic mean was used in subsequent computation.

Two sets of sample calculations follow which are intended to exhibit some of the properties of the data. The first will concern computing transfer coefficients for the water phase and the second the alcohol phase.

The water phase transfer coefficient k'_{AL} for the flow condition of 11,300 gms/hr water and 12,000 gms/hr isobutanol will be computed by the several methods discussed in Section VI, (1) by drawing an equal area through the differenced values of Lx_A, (2) by averaging the k'_{AL}'s computed

for the increments of the column and (3) by an integral average. The numbers pertinent to the calculations are given in Table III.

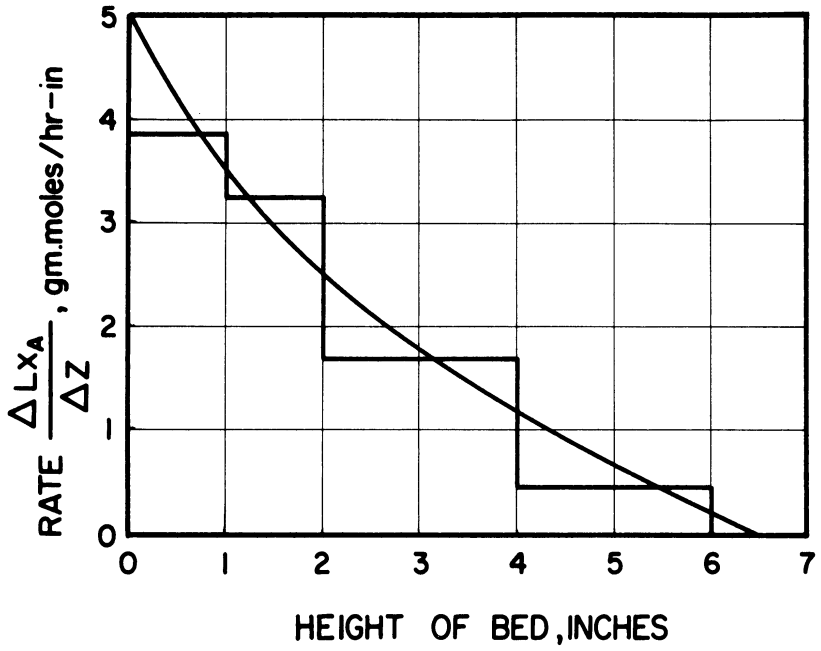
TABLE III

CALCULATION OF WATER PHASE TRANSFER COEFFICIENTS
INLET FLOW RATES: WATER - 11,300 gms/hr
ISOBUTANOL - 12,000 gms/hr

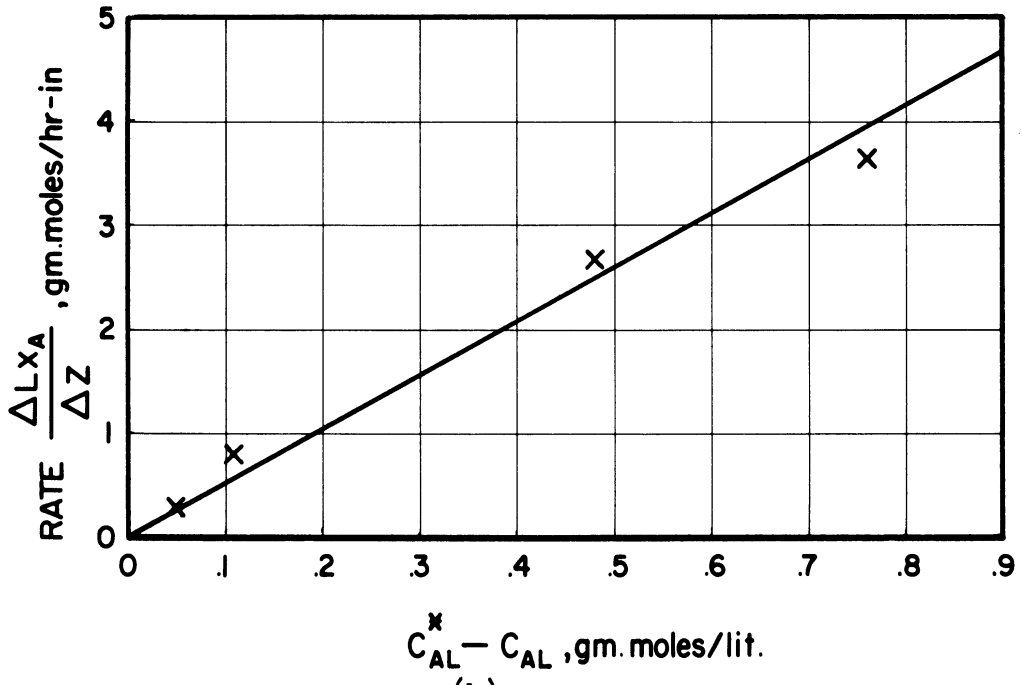
Z (in.)	Lx_A gm.mol/hr	$\Delta(Lx_A)$	$\Delta(Lx_A)/\Delta Z$ gm.mol/hr.in	ΔC_{AL} gm.mol/lit.	$(\Delta C_{AL})_m$	$k'_{AL} * S$ lit/hr.in	$(\Delta C_{AL})^{-1}$
0	0			1.10			.91
		3.86	3.86		.93	4.15	
1	3.86			.76			1.32
		3.26	3.26		.62	5.26	
2	7.12			.48			2.08
		3.40	1.70		.296	5.74	
4	10.52			.111			9.0
		.90	.45		.085	5.29	
6	11.41			.058			11.8

$\bar{k}'_{AL} * S = 5.25$ $\qquad$ $\bar{k}'_{AL} = 1{,}620\ (hr)^{-1}$

For method (1), the values of $\Delta(Lx_A)/\Delta Z$ are plotted as in Figure 21a against the height of bed. An equal area curve is drawn through the rate rectangles. This ostensibly gives the rate of transfer of isobutanol into the water phase as a smooth function of height of packing. Now, since the driving force, $(C^*_{AL} - C_{AL})$, is known at bed heights of 0", 2", 4" and 6", a plot such as Figure 21b can be drawn (the rate vs. driving force). The slope of the line through the points of this plot yields the quantity $\bar{k}'_{AL} * S$ in the units liters/hr.in. assuming, of course, that the $\bar{k}'_{AL}$ is a constant. From Figure 21b, it is seen that this is not entirely true for the equal area



(a)



(b)

Figure 21. Equal Area Calculation of Water Phase Transfer Coefficients.

curve as drawn in Figure 21a, since the points are somewhat scattered. However, to a degree of approximation, a straight line is sufficient. A least squares line through the points and through the origin, yields a slope of 5.25.

For method (2), the incremental coefficients, a mean driving force over the increment is needed, and this was taken as the arithmetic mean of the initial and final ΔC_{AL} of the increment in question. This corresponds to the column $(\Delta C_{AL})_m$ in Table III. The rate of transfer in that increment, $\Delta(Lx_A)/\Delta Z$, is then divided by this mean driving force, yielding an average $k'_{AL} * S$ for the increment of the bed in question. The values are then averaged once more, yielding a mean $\bar{k}'_{AL} * S$ for the entire composite column. In this case, it turns out that this mean is 5.25, the same value as that obtained in method (1). Method (2) has the advantage of presenting the incremental coefficients of the column, employing no data smoothing at any point in the treatment of the column data. From Table III, and also shown in Figure 17, the column of $k'_{AL} * S$ values shows that the water phase coefficient is indeed quite constant throughout the composite column.

For method (3), computing the integral average coefficient, a plot of $1/(C^*_{AL} - C_{AL})$ vs. Lx_A is constructed and integrated graphically. This procedure gives a value of 5.8 to $\bar{k}'_{AL} * S$.

The water phase data was generally very well-behaved and the difference in the methods of computing an average column coefficient was not significant. However, the incremental coefficients of method (2) gave a much better indication of the stability of the original data and of

any effect of the height of bed on the coefficients. This is brought out more clearly in the treatment of the alcohol phase data, which was far more erratic than the water phase data, and on inspection of the incremental coefficients of method (2) showed some tendency to vary with bed height. Very often method (1) failed even to approximate a transfer coefficient which would yield the correct amount of total transfer. This was probably due in part to the tendency of the isobutanol phase coefficient to increase with increasing bed height, but mostly to the impossibility of drawing a correct equal area curve through the too few rate rectangles that could be constructed from the data. As an illustration of this, a sample calculation for the alcohol phase transfer coefficient is presented for the flow condition of 11,300 gms/hr. water and 8,000 gms/hr. isobutanol. The required numbers are given in Table IV.

TABLE IV

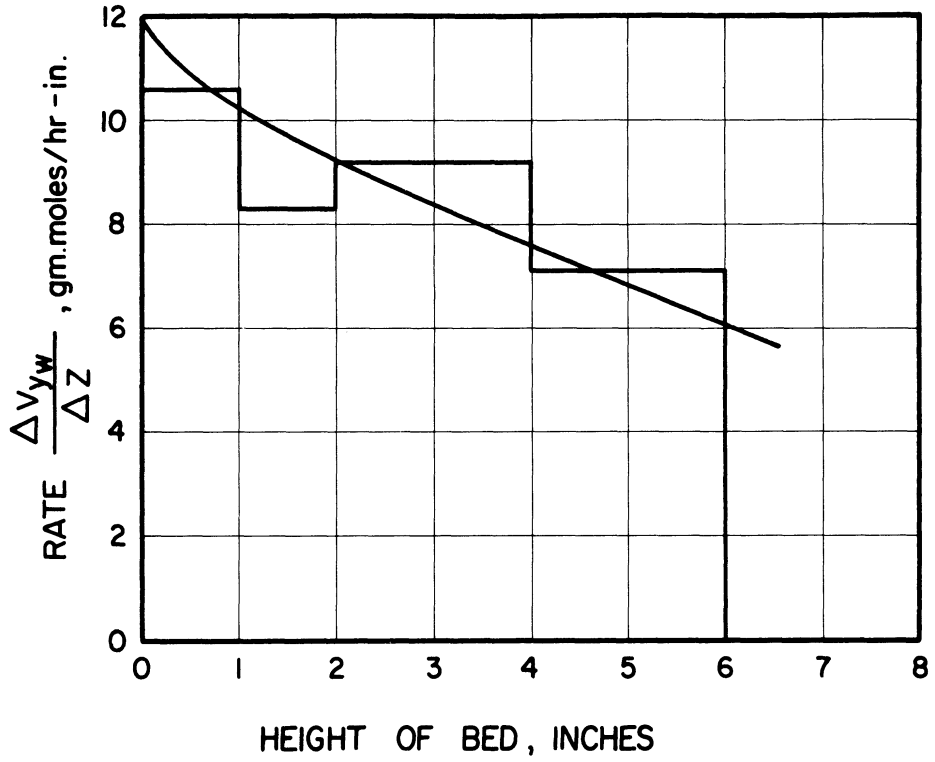
CALCULATION OF ISOBUTANOL PHASE TRANSFER COEFFICIENTS
INLET FLOW RATES: WATER - 11,300 gms/hr
ISOBUTANOL - 8,000 gms/hr.

Z (in.)	Vy_W gm.mol/hr.	$\Delta(Vy_W)$	$\Delta(Vy_W)/\Delta Z$ gm.mol/hr.in	ΔC_{WV} gm.mol/lit.	$(\Delta C_{WV})_m$	$k'_{WV} * S$ lit/hr.in	$(\Delta C_{WV})^{-1}$
0	0			7.68			.13
		10.58	10.58		7.14	1.48	
1	10.58			6.61			.151
		8.32	8.32		6.19	1.34	
2	18.90			5.76			.174
		18.39	9.20		4.82	1.91	
4	37.29			3.89			.258
		14.18	7.09		3.02	2.35	
6	51.47			2.16			.463

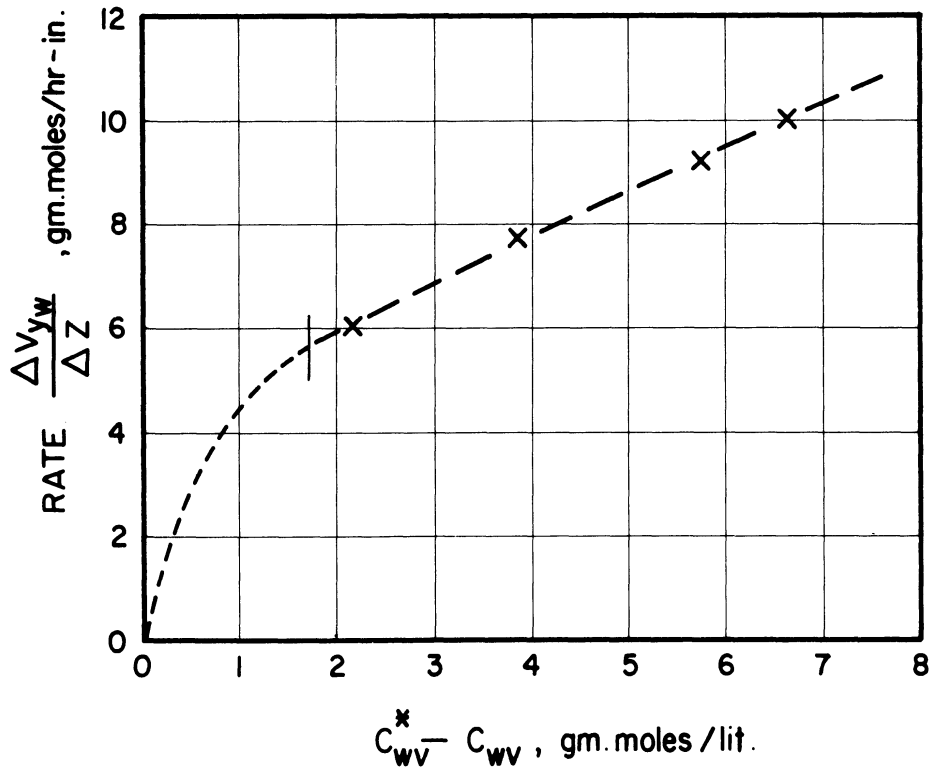
$\bar{k}'_{WV} * S = 1.89$ $\qquad$ $\bar{k}'_{WV} = 580\ (hr)^{-1}$

The corresponding plots of rate vs. bed height and the "equal area" smoothed rate vs. driving force are shown in Figures 22a and 22b. Drawing an equal area curve through the rate rectangles of Figure 22a, points on the rate vs. driving force plot, Figure 22b, are obtained which do approximate a straight line. This straight line, however, does not tend to the origin and, if only the straight portion is considered, the slope yields a $\bar{k}'_{WV} * S$ of 0.9, while an averaging of the incremental coefficients yields a value of 1.89. The integral averaging method yields a value of 1.91. Clearly, an ordinary smoothing of the rate rectangles, in this case, by an equal area curve, does not yield transfer coefficients consistent with the amount of mass transfer taking place. This was true for a great many of the calculations concerning the isobutanol phase coefficients.

Due to the above situation, only the results of calculating the incremental coefficients for the variable column length data are presented. The incremental transfer coefficients for isobutanol transferring into water are tabulated in Table V, and those for water transferring into isobutanol are tabulated in Table VI.



(a)



(b)

Figure 22. Equal Area Calculation of an Isobutanol Transfer Coefficient.

TABLE V

INCREMENTAL WATER PHASE TRANSFER COEFFICIENTS

Inlet Flow Condition-gms/hr		k'_{AL} * S for Bed Increment $\frac{\text{liters}}{\text{hr.in.}}$				$\bar{k}'_{AL}$ * S	$\bar{k}'_{AL}$ $-hr^{-1}$
water	isobutanol	0-1 in.	1-2 in.	2-4 in.	4-6 in.		
20,000	16,600	10.5	9.5	9.4	13.5	11.0	3,400
	12,000	7.0	8.2	8.0	8.5	8.0	2,480
	8,000	4.9	4.8	6.4	5.7	5.7	1,750
	4,200	2.9	3.6	3.1	3.3	3.2	990
15,500	16,600	8.6	9.3	6.4	7.8	7.7	2,390
	12,000	5.0	8.2	5.2	7.9	6.6	2,030
	8,000	3.8	5.1	4.3	5.5	4.7	1,460
	4,200	1.8	3.4	2.2	3.1	2.6	810
11,300	16,600	5.3	7.8	5.1	-	5.8	1,810
	12,000	4.2	5.3	5.4	5.3	5.3	1,620
	8,000	3.1	3.4	3.9	4.0	3.7	1,160
	4,200	1.4	2.2	2.1	2.1	2.0	620
	2,200	1.2	.5	1.0	1.1	1.0	310
7,200	16,600	3.5	5.9	3.2	-	4.0	1,230
	12,000	2.7	5.1	3.5	1.6	3.0	920
	8,000	2.4	3.4	2.8	3.0	2.9	890
	4,200	1.0	2.0	2.0	1.4	1.6	500
	2,200	.7	.9	.9	.9	.9	270
3,700	12,000	1.7	2.2	2.0	-	1.9	600
	8,000	1.3	1.7	1.7	1.3	1.5	460
	4,200	.9	1.2	1.2	1.5	1.3	390
	2,200	.6	.6	.7	.9	.7	230

TABLE VI

INCREMENTAL ISOBUTANOL PHASE TRANSFER COEFFICIENTS

Inlet Flow Condition-gms/hr		k'_{WV} * S for Bed Increment $\frac{liters}{hr.in.}$				$\bar{k}'_{WV}$ * S	$\bar{k}^{+}_{WV}$ $-hr^{-1}$
water	isobutanol	0-1 in.	1-2 in.	2-4 in.	4-6 in.	average	
20,000	16,600	4.3	4.6	5.4	7.4	5.7	1,770
	12,000	2.7	3.2	3.6	4.7	3.8	1,170
	8,000	2.1	1.9	2.2	2.6	2.3	700
	4,200	1.2	1.1	1.2	0.8	1.0	320
15,500	16,600	3.6	5.4	3.6	4.8	4.3	1,320
	12,000	2.6	3.2	2.4	4.6	3.3	1,010
	8,000	2.1	2.1	1.4	2.6	2.0	630
	4,200	1.1	1.5	0.6	1.1	1.0	300
11,300	16,600	2.5	3.2	3.3	4.5	3.6	1,110
	12,000	1.9	2.1	2.9	3.2	2.7	830
	8,000	1.5	1.3	1.9	2.4	1.9	580
	4,200	.8	.7	1.0	.7	0.8	260
	2,200	.7	.0	.5	.5	0.4	130
7,200	16,600	1.8	2.5	2.5	3.7	2.8	860
	12,000	1.3	2.5	1.7	2.8	2.1	655
	8,000	1.3	1.7	1.2	2.4	1.7	520
	4,200	.7	1.0	.7	.9	.8	250
	2,200	.4	.4	.4	.4	.4	130
3,700	12,000	1.3	.1	1.5	1.0	1.0	325
	8,000	.9	.4	1.3	1.1	1.0	310
	4,200	.7	.4	.8	1.0	.8	235
	2,200	.4	.1	.4	.4	.4	110

TABLE VII

SUMMARY OF INDIVIDUAL PHASE TRANSFER COEFFICIENTS

Inlet Flow Condition lbs/hr.ft^2		Water Phase Coefficient	Isobutanol Phase Coefficient
Water	Isobutanol	k'_{AL} hr^{-1}	k'_{WV} hr^{-1}
32,000	27,000	3,400	1,770
	19,000	2,480	1,170
	13,000	1,750	700
	6,800	990	320
25,000	27,000	2,390	1,320
	19,000	2,030	1,010
	13,000	1,460	630
	6,800	810	310
18,000	27,000	1,810	1,110
	19,000	1,620	830
	13,000	1,160	580
	6,800	620	260
	3,500	310	130
12,000	27,000	1,230	860
	19,000	920	655
	13,000	890	520
	6,800	500	250
	3,500	270	130
6,000	19,000	600	325
	13,000	460	310
	6,800	390	235
	3,500	230	110

APPENDIX E

REFRACTIVE INDEX CALIBRATION WATER AND ISOBUTANOL

APPENDIX E

REFRACTIVE INDEX CALIBRATION WATER AND ISOBUTANOL

The index of refraction - weight fraction calibration was determined by taking the refractive index of a sample of predetermined composition. The data obtained checked with that of Colburn and Welsh.[1] Commercial grade isobutanol and distilled water acidified to 0.0001 N HCl were used. The data are presented in Table VIII and are plotted in Figures 23 and 24.

TABLE VIII

INDEX OF REFRACTION - WEIGHT FRACTION CALIBRATION
OF ISOBUTANOL - WATER AT 25°C.

Sample No.	Index of Refraction	Weight Fraction Water
1	1.33245	1.0
2	1.33323	.9922
3	1.33485	.9761
4	1.33640	.9623
5	1.33791	.9482
6	1.33947	.9335
7	1.34077	.9210
8	1.34118	.9170-sat. water
9	1.38795	.1670-sat. isobutanol
10	1.38849	.1536
11	1.38958	.1266
12	1.39062	.1009
13	1.39180	.0692
14	1.39283	.0363
15	1.39347	.0124
16	1.39382	0.0

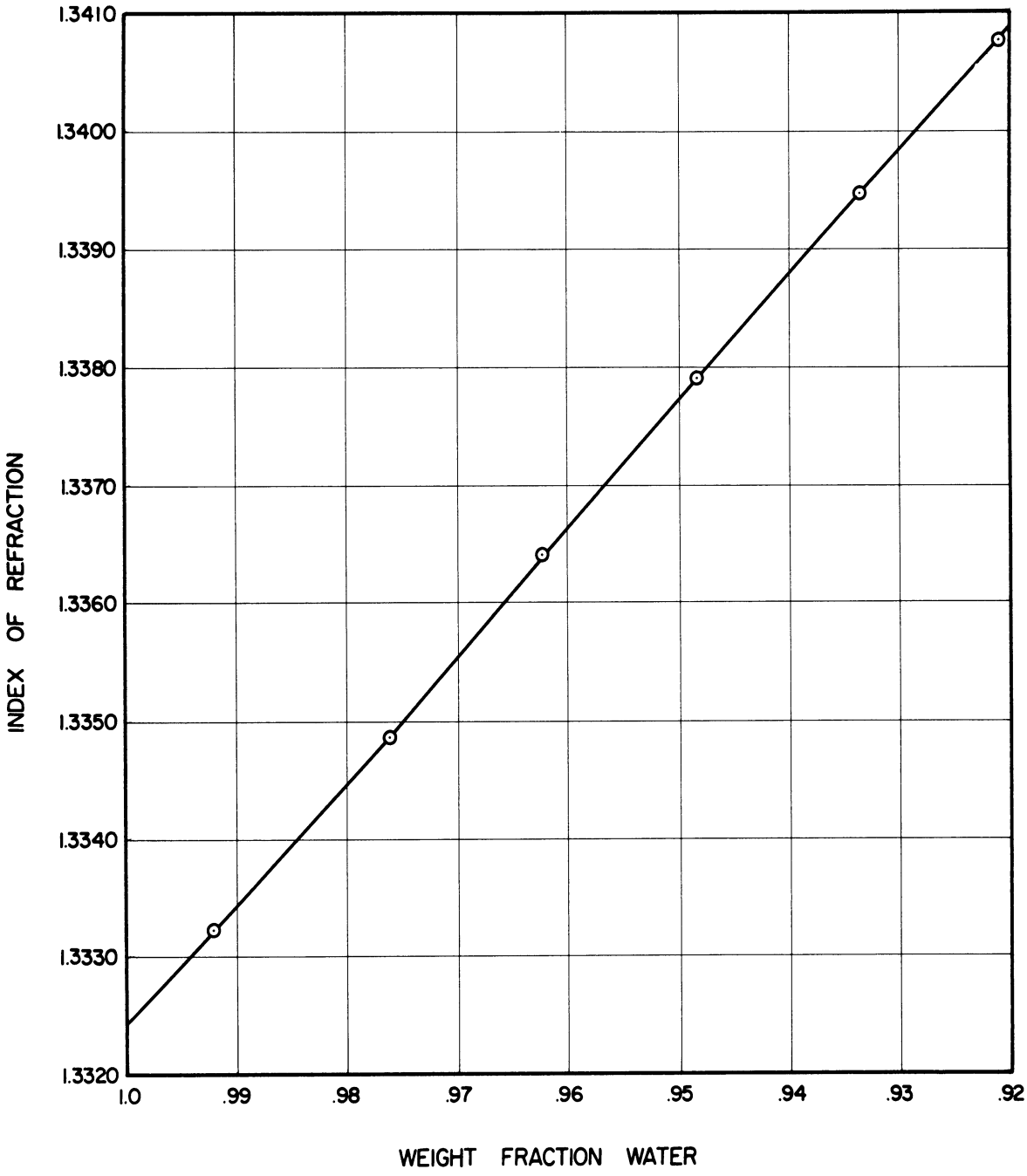


Figure 23. Index of Refraction Calibration for Water Phase.

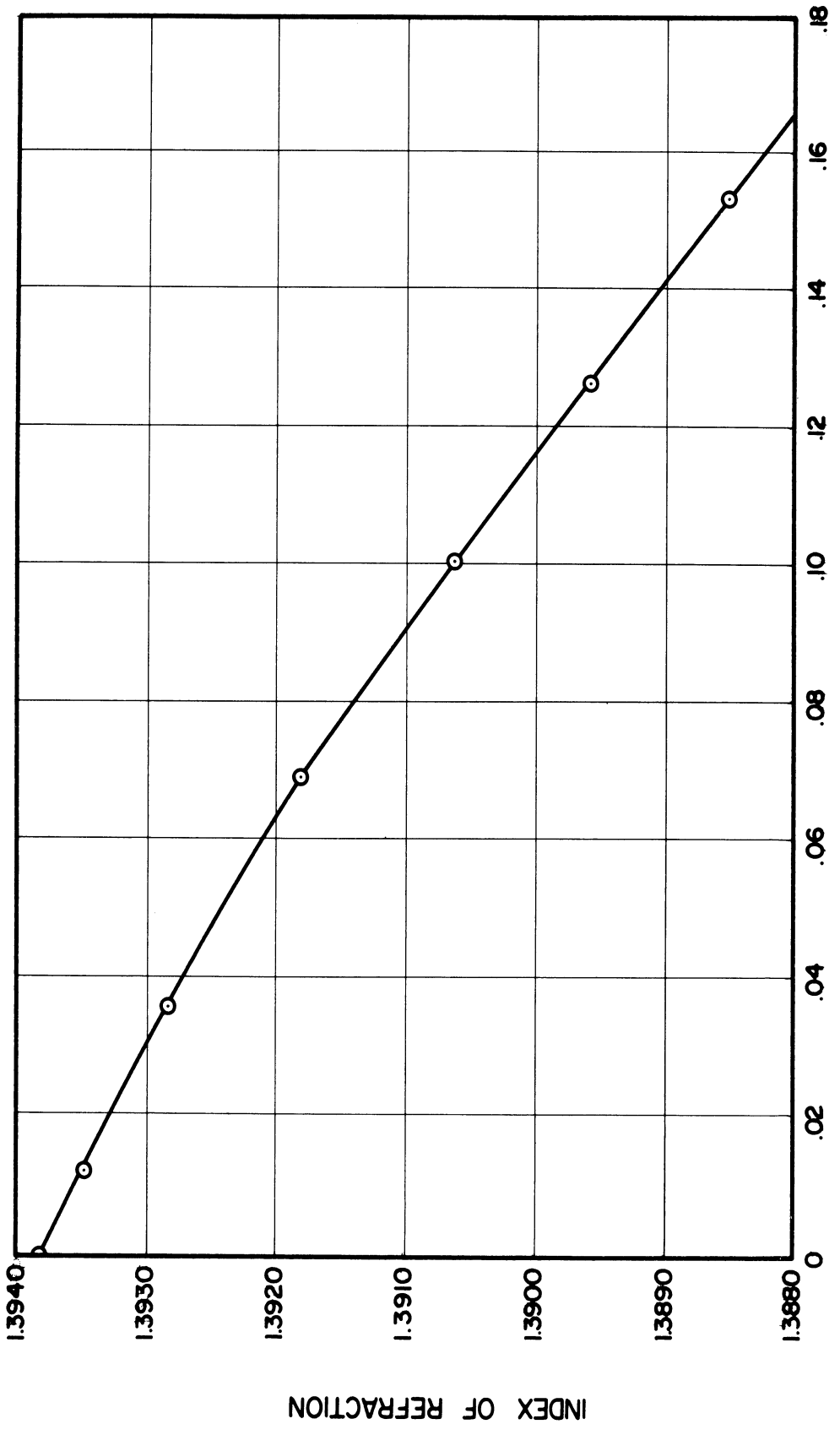


Figure 24. Index of Refraction Calibration for Isobutanol Phase.

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