

# Measurement of Mass Transfer Coefficients in Liquid-Liquid Mixing

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Most of the work reported in the literature on liquid-liquid mass transfer is for steady state conditions in extraction columns and spray towers (1, 3, 5, 10, 12, 14, 17, 20). The reports have correlated the volumetric mass transfer coefficients with the flow rates of the continuous and dispersed phases. These results are useful for specifying the values of liquid flow rates for column operation, but more information is needed for complete elucidation of the basic mechanisms of liquid-liquid mass transfer in such systems. Of particular importance are data which will relate size of equipment to the rate of mass transfer, and no data on continuous flow liquid-liquid mass transfer systems are available for equipment of different size to provide such data.

The use of continuous flow equipment allows the experimenter to reach a steady state and to make sufficient measurements to determine accurate material balances and rate coefficients. However in two-phase liquid-liquid mass transfer operations the equipment size itself is a very significant variable and interacts not only with fluid motion conditions but also with the physical properties of the fluids. Accordingly data are required for engineering design on a large scale. To evaluate the effect of size large scale continuous types of experiments would be highly desirable, but the amount of material to be handled and the size of auxiliary equipment necessary makes the cost of such studies much too high for practical consideration. Therefore a batch type of experiment is desired which will minimize the bulk of the equipment and material to be handled and still provide for sufficient accuracy so that the data may be translated to the performance of a continuous flow operation. Then large size batch experiments can be made at far less cost than for large scale continuous operations and data obtained to elucidate the effects of size for continuous flow systems.

Single-drop studies (4, 7, 18) have been used to develop both theoretical and empirical models to describe the relationship of mass transfer rates to the variables such as the fluid flow conditions, the physical properties of the liquids, etc. Such models describe the experimental results to a fair degree of accuracy, but their main weakness is that they are for single drops. When masses of drops are present, there is considerable coalescence and breakup of them causing the formation of new surface area as well as redistribution of the solute in the dispersed phase, hence the significant difference in overall rate transfer data between single and multiple drops in a turbulent field.

In studies made with masses of drops the physical properties of the liquids and the flow motion of the system should be changed as independent variables because there is interaction between fluid motion and physical properties (16). Few such studies (6, 9, 11) have heretofore been made, perhaps because of the large number of interrelated variables which make an independent study of the variables difficult.

Lewis (6) worked with an apparatus that gave constant interfacial area between two liquid phases and studied the relationship of the mass transfer rates to the variables. His setup consisted of having two liquids as separate layers with each phase mixed by a separate rotating impeller. At the surface of contact of the two liquids there was an annular gasket that helped maintain a constant interfacial area of contact. Lewis correlated his data in the usual conventional Colburn *J*-factor method. Mayers (9) improved the Lewis correlation by studying more systems in the same type of apparatus. Both Mayers' and Lewis's studies brings out clearly the large number of variables that control the rates of mass transfer as well as the strong interdependency of the variables. They correlated their mass transfer rates to the Reynolds numbers within the two phases, the viscosities of the two phases, and the Schmidt numbers. Their correlation is the first of its kind, where the liquid-liquid mass transfer rates were correlated with the basic dimensionless groups that represent the fluid flow regime. But these studies were for separate layers of the two liquids and as such do not really represent the flow conditions that exist in a dispersion.

Nagata (11) studied the rates of mass transfer for liquid-liquid extraction in mixing tanks. He used chemical reaction to measure the mass transfer rates and worked with a 0.65% dispersion of the organic phase, with small density differences between the organic drops and the continuous aqueous phase. His conclusions were that the rates of mass transfer are proportional to the interfacial area. The present work used an 8.2% dispersion of organic phase in a mixing tank and shows a different result at this ratio of phases. Fluid flow conditions were varied by varying the size and speed of the impeller and the physical properties of the liquids.

The limited objectives of this work were to select a chemical system, an analytical method, an experimental method, and a method to analyze results that would allow for rapid measurement of mass transfer rates between two liquid phases in turbulent flow condition in a mixing tank and to conduct experiments to elucidate the effectiveness and the magnitude of some variables that influence the mass transfer rates in liquid-liquid systems. A later report will extend the work to cover changes in equipment size and to correlate more of the significant variables.

## EQUIPMENT

A single 6-in. diameter, flat-bottomed glass cylinder was used as a mixing tank, and total liquid depth was 6 in. It was fitted with four stainless steel baffles equally spaced on the interior periphery of the tank. The baffles extended the full length of the tank (9 in.) and were one tenth the diameter of the tank in width. Stainless steel six flat-blade turbine impellers used were dimensionally similar to those recommended by Rushton (15). The diameters of the impellers were 2 and 3 in. and were driven by a motor whose speed was varied with a standard autotransformer and measured with a mechanical tachometer. The motor drive and impeller were connected by  $\frac{3}{8}$  in. bakelite shaft. Bakelite was chosen to prevent troubles due to grounding of the conductivity measuring unit.

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## Chemical System

**Solvents.** Water and mixtures of water and corn syrup were used as the continuous phase. Xylene, paraffin oil, and mixtures of xylene and paraffin oil were used as the dispersed phase. The choice of the continuous phase was made because the solutes used were either mineral or organic acids and would ionize in the aqueous phase; their concentrations could be easily measured by chemical and physical methods. Various mixtures of both water phase and organic phase were used to obtain the effect of a wide range of physical properties of the phases on the mass transfer rate of a solute.

## Solute

Batch types of experiments were used because of low cost and ease of operation. It was necessary therefore that the solute have low rates of transfer so that concentration changes of solute with time could be measured accurately. Common solutes such as acetic acid and benzoic acid were studied. These solutes gave very high rates of transfer and in some cases almost instantaneous equilibration, which made accurate concentration measurements very difficult to obtain. Such high rates are due to large interfacial areas of a dispersion in a mixing tank and the high driving forces for mass transfer resulting from the high solute solubility in either or both of the two phases.

The interfacial area was decreased to a certain extent by using a relatively small ratio of organic to water phase. An 8.2% dispersion was chosen to achieve relatively small transfer areas. This concentration of dispersed phase did however provide enough drops so that coalescence and redispersion were taking place (8) and should play a part in the transfer mechanism.

Lower rates of transfer may also be obtained by having low driving forces. For example if the solute is being transferred from continuous to the dispersed phase, it is desirable that the solute have low concentration in the continuous phase and a high equilibrium solubility in the dispersed phase. The low concentration gives low driving forces, and high equilibrium solubility allows transfer of measurable amounts of solute. Acetic acid and benzoic acid may be transferred from a low concentration in continuous phase, but their solubilities in the dispersed phase do not allow easily measurable changes in concentration. Octanoic acid was selected as the solute; it has low solubility in the continuous phase, it gives lower driving forces, and it has a solubility in the dispersed phase which allows easily measurable amounts of transfer.

## Analytical Setup

Electrical conductivity of the continuous phase was the method selected for analysis of solute concentration, and then changes in concentration in the dispersed phase were calculated by material balance. Several methods of analysis were considered before the electrical conductivity measurements were selected. Trial runs were made with chemical analysis wherein a sample was drawn out through a screened glass tube and then analyzed by titration, but this was not continued because of two types of errors inherent in this method. The time necessary for sampling was about 5 sec., and since most of the mass transfer took place in about 100 sec., the errors due to lapse of time during sampling could be appreciable. When a sample was drawn out, the dispersed phase was filtered through a screen, and such a separation caused the drops of the dispersed phase to deform and probably resulted in rapid surface changes which would affect the transfer rate during sampling time.

The conductivity measurements made in situ were felt to be more accurate because they were instantaneous and could be recorded rapidly. The conductivity equipment consisted of a conductivity cell made by sealing two platinum wires in a 8 mm. soft glass tube 18 in. in length. The exposed ends of the wires served as electrodes, and the ends within the glass tube were welded to copper wires for connection to the measuring unit. The electrodes were 10 mm. long and were separated by a distance of 1.5 mm. The free end of the electrodes were joined with a bead of soft glass for better structural stability. The output of this unit was recorded continuously on a high-speed ( $\frac{1}{2}$  sec. full scale pen travel) potentiometer.

The presence of the organic dispersed phase caused two types of difficulties in the conductivity measurements. First

the dispersed-phase drops tend to stick to the electrodes and thus cause a decrease in the electrode area. This is minimized by cleaning of the electrodes with water, acetone, and de-ionized water before each experiment, and also by placing the electrodes in the direct discharge flow from the impeller. This flow prevented the drops from sticking to the electrodes by sweeping them off. Second the presence of the organic drops in the continuous phase passing between the electrodes caused decrease in the specific conductance readings and was found to be dependent on chemical and physical properties of the solvents. Therefore calibrations were made for each pair of solvents that were used, and these calibrations were found to be independent of concentration of octanoic acid and the speed and size of the impeller.

The calibration of the conductivity recording apparatus was made in two parts. First the relation of the concentration of octanoic acid to the instrument reading was obtained by adding measured increments of standardized acid solution to a known volume of the continuous phase material and measuring the solution conductivity after each addition; no dispersed phase was present. Then secondly conductivity readings for a dispersed system at equilibrium were made, after which the dispersion was allowed to settle and another conductivity determined for the clear continuous phase. A plot of conductivity readings without the organic phase and the conductivity readings with the organic phase was made for each pair of solvents used and served as the calibrations for the system.

## EXPERIMENTAL METHOD

The method consists of adding a small increment of the continuous phase to a steady state drop size aqueous-organic dispersion. The solute present in the incremental addition was found to distribute itself very rapidly in the continuous phase, after which most of the transfer took place to the dispersed phase. The changes in the concentration of acid in the continuous phase were measured and recorded continuously from just before the incremental addition. The equation derived to represent the concentration change, on the basis of material balance and rate, is

$$\text{Log} \frac{(C_c - C_c^*)}{(C_{ci} - C_c^*)} = \frac{K_o A C_{co}}{2.3 V_c (C_{co} - C_c^*)} (\theta - \theta_i) \quad (1)$$

and can be evaluated from the data taken as just described.

The advantages of this over several other experimental methods tried out are as follows. Since equilibrium drop size is achieved before addition of the increment, there should result the least disturbance of the fluid flow regime and interfacial area when an increment of acid is added. This facilitates accurate measurement of transfer rates. The concentration changes are highly reproducible with this rapid addition of the solute. The rapid incremental addition distributes itself within 1 to 2 sec. for the 6-in. tank (and within 2 to 3 sec. for tanks three times the size used). The calculation of the mass transfer coefficient consists of taking the slopes of log concentration vs. time curves, and the simplicity of such calculations leads to good accuracy.

## DETAILS OF THE EXPERIMENTAL METHOD AND SAMPLE CALCULATION

The following are the details of run number 12 which is used to illustrate the procedure. 2,000-ml. aqueous phase (demineralized down to 0.1 p.p.m. corresponding to a 1,000,000-ohm resistance) was placed in the tank; 200 ml. of xylene were then added. The mixer was started, and then the conductivity cell was placed in the liquid so that the direct discharge flow from the impeller passed between the electrodes of the cell. When the drops of xylene reached equilibrium size, a volume of 250 ml. of a solution of octanoic acid (0.0041 g. moles/liter) in de-

TABLE 1. EXPERIMENTAL DETAILS OF RUN NO. 12

Continuous phase:	5 centipoise corn syrup-water mixture
Dispersed phase:	xylene
Tank size:	6 in.
Impeller size:	3 in.
Rev./min.:	327

$(\theta - \theta_i)$ sec.	$C_c \times 10^4$ moles/liter	$\frac{(C_c - C_c^*)}{(C_{ci} - C_c^*)}$
0	2.75	1.00
10	2.13	0.69
20	1.72	0.48
30	1.45	0.34
40	1.23	0.23
60	1.03	0.13
80	0.94	0.08
120	0.83	0.03
	0.78	0.00

mineralized aqueous phase was poured rapidly from a beaker into the dispersion. The conductivity was measured and recorded from the moment the solution was added until no further significant changes in conductivity were observed. Mixing was continued for thirty more minutes, and conductivity was measured again and found to have decreased only slightly; this value was used for the equilibrium concentration.

From the equation for the concentration-time relation [Equation (1)] it is seen that a plot of  $\log (C_c - C_c^*) / (C_{ci} - C_c^*)$  vs.  $(\theta - \theta_i)$  should give a straight line whose slope is proportional to the mass transfer coefficient  $K_oA$ . Table 1 lists the concentration-time data for this run. Column 1 shows the values of time  $(\theta - \theta_i)$  where  $\theta_i$  is the time at which the disturbing effect due to the instantaneous addition of the continuous phase increment has ceased and smooth and steady mass transfer from the continuous phase to the dispersed drops has started.  $C_{ci}$  corresponds to the continuous phase concentration corresponding to this time. Column 2 shows the corresponding values of concentration corresponding to the time in column 1, and column 3 lists the values of  $(C_c - C_c^*) / (C_{ci} - C_c^*)$ .

Figure 1 is a plot of  $\log (C_c - C_c^*) / (C_{ci} - C_c^*)$  vs.  $(\theta - \theta_i)$ . The data justify drawing a straight line. The slope of this line is equal to

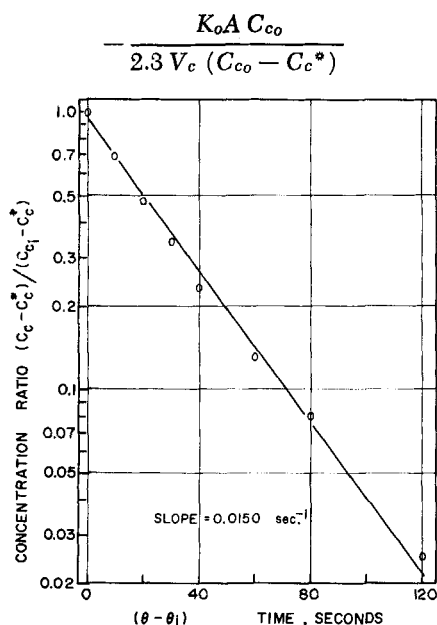


Fig. 1. Run 12 concentration ratio as a function of time.

From the experimental plot (Figure 1) the value of the slope is determined to be  $0.0150 \text{ sec}^{-1}$ .

The values of other quantities in the equation are  $V_c = 2,000 \text{ ml}$ ,  $V_d = 200 \text{ ml}$ ,  $v = 250 \text{ ml}$ ,  $N = 327 \text{ rev./min.}$ ,  $C_o = 4.16 \times 10^{-3} \text{ g. moles/liter}$ ,  $C_{c0} = \frac{250 \times 4.16 \times 10^{-3}}{2,250}$

$= 4.61 \times 10^{-4} \text{ g. moles/liter}$ , and  $C_c^* = 0.78 \text{ g. moles/liter}$

From these values the  $K_oA$  value was calculated as

$$\text{Slope} = 0.0150 = \frac{K_oA (4.16 \times 10^{-4})}{2.3 \times 2,250 (4.16 - 0.78) 10^{-4}}$$

$$K_oA = 75.0 \text{ cc./sec.}$$

## RESULTS

The results were of two types. First the batch method with incremental additions of octanoic acid to the continuous phase turned out to be an acceptable and useful technique, suitable for use in larger sized systems. Second information on the effect of fluid physical properties and mixing conditions was obtained. This second part as such is not a complete study but is the basis of further work dealing with the effect of these and other variables to be used for scale-up relationships.

In batch types of experiments it is difficult to measure concentration changes accurately with time. Relatively slow rates of transfer were achieved with octanoic acid as the solute. After various solutes were investigated, octanoic acid was selected as the solute because it has low solubility in the demineralized aqueous phase resulting in low driving forces, and it has high solubility in the organic phase so that relatively large and measurable quantities of solute could be transferred. The presence of octanoic acid had no significant effect on the interfacial tension between the phases; the maximum variation of interfacial tension between the two phases was a 2.5% increase when saturated with the acid. A continuous in situ conductivity measurement of the continuous aqueous phase was chosen as the analytical method as against chemical or other physical methods of analysis.

The physical properties of the solvents and solute, the physical setup of the mixing tank, and the operating variables such as the speed and size of the mixing impeller, all affected the interfacial area (13) and mass transfer rate. Only the viscosities of the two solvents were changed to study the effect of physical properties. When the viscosities were changed, the density of the solvents and the interfacial tension changed only slightly (less than 2% each).

The speed of the impeller was studied as a variable, but the size of the impeller was not. The three independent variables were the viscosities of the continuous and dispersed phases and the speed of the impeller. Experiments to study these variables were grouped into two sets.

In one set of experiments the dispersed-phase viscosity was held constant at 0.8 centipoise, and the continuous-phase viscosity and speed were varied. Three levels of continuous phase viscosity (0.8, 10, and 20 centipoise) were used. For each of these the speed was varied from 300 to 700 rev./min. Figure 2 is a logarithmic plot showing the effect of speed on the transfer coefficient for various values of continuous-phase viscosities. The slope of the lines is 2.1, showing that the coefficient varies as the 2.1 as power of speed, and this effect is independent of the dispersed-phase viscosity. Figure 3 is a logarithmic cross plot of Figure 2 at 500 rev./min. The slope of the line shows that the transfer coefficient varies as the  $-0.60$  power of continuous-phase viscosity.

The second set of experiments was carried out holding continuous-phase viscosity (0.8 centipoise) constant and varying the dispersed-phase viscosity and the speed. Four

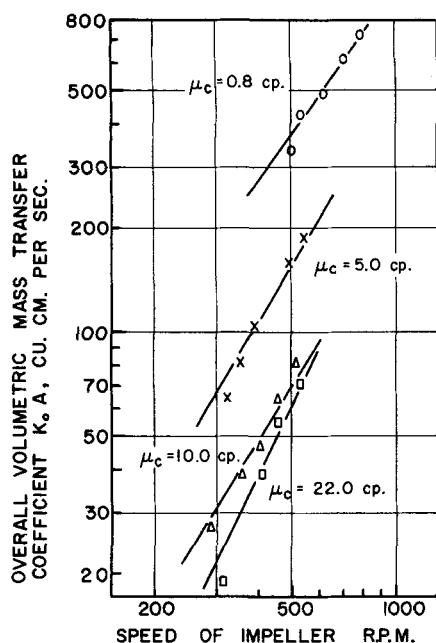


Fig. 2. Effect of speed and continuous-phase viscosity on mass transfer coefficient.

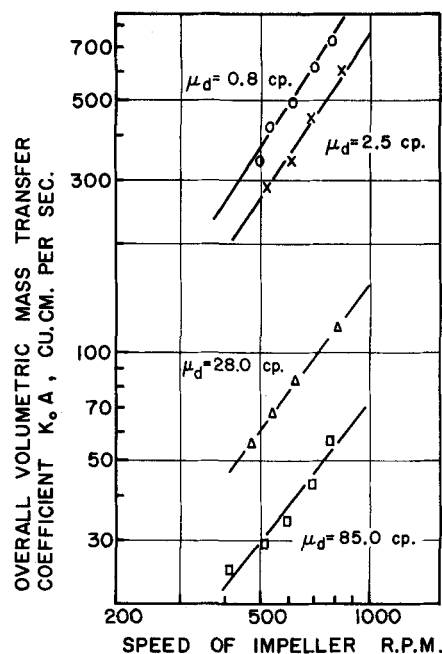


Fig. 4. Effect of speed and dispersed-phase viscosity on mass transfer coefficient.

levels of dispersed-phase viscosity (0.8, 2.5, 28.0 and 85.0 centipoise) were used, and for each of these viscosities speed was varied from 500 to 800 rev./min. Figure 4 shows the effect of speed on the coefficient with dispersed-phase viscosity as the parameter. The coefficient varies as the 2.1 power of speed and seems to be independent of dispersed-phase viscosity. Figure 5 is a cross plot of Figure 4 at 500 rev./min. and shows the effect of dispersed phase viscosity on the coefficient; the coefficient varies as the  $-0.55$  power of the dispersed-phase viscosity.

In both types of experiments it should be noted that the coefficients were high, running above 300 cc./sec. for waterlike liquids.

## DISCUSSION OF RESULTS

Figures 2 and 4 show effect of speed on the transfer coefficient, and since the slope of the lines is 2.1, the volumetric mass transfer coefficient  $K_o A$  varies approximately as the square of the speed regardless of the dynamic viscosity of the phases. In a mixing tank the energy input is proportional to cube of the speed ( $N$ ) and to the fifth power of impeller diameter ( $D$ ). This energy is utilized as ( $ND^3$ ) for the mass flow and ( $N^2 D^2$ ) for the turbulence generated by the impeller (16). Therefore the variation of the coefficient as  $N^{2.1}$  indicated that mostly the effect is due to the turbulence generated in the impeller discharge

stream. The variation of  $K_o A$  with speed may be due to the variation of  $K_o$  with speed, or the variation of area  $A$  with speed, or both. Rodger (13) and deVity (2) in their studies of interfacial areas have shown that area  $A$  varies as the 1.4 power of speed. Shinnar and Church (19) applying concepts of local isotropy for interfacial area also show that area should vary as the 1.4 power of speed. Since the volumetric mass transfer coefficient  $K_o A$  is the product of the surface coefficient times the area, and the area appears to vary with the 1.4 power of speed, it is evident that  $K_o$  is also a function of speed since the product  $K_o A$  varies with the 2.1 power of speed. Apparently both the coefficient  $K_o$  and the area  $A$  are affected similarly but not equally by impeller speed, and hence by the power imposed by the impeller.

Nagata (11) in his studies has shown that the variation of  $K_o A$  with speed corresponds closely to the variation of area with speed. His studies were for 0.65% dispersion in a small size tank (10-cm. diameter) and for liquids of low density differences; his area measurements were made on a withdrawn sample and not in situ. The difference in the exponents may be due to low percentage of dispersion used, to the size of the tank, or to the method of area measurement.

Comparing the value of mass transfer coefficient in

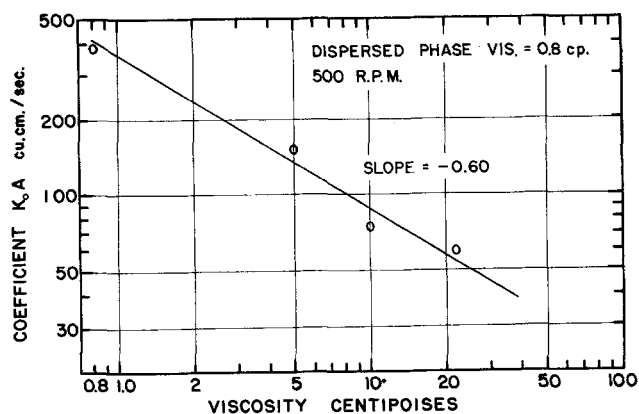


Fig. 3. Effect of continuous-phase viscosity on mass transfer coefficient.

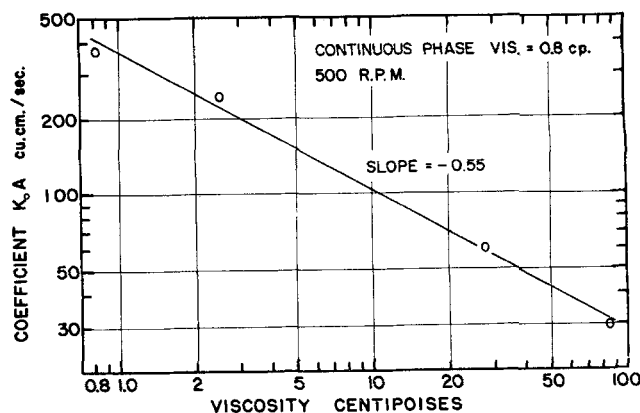


Fig. 5. Effect of dispersed-phase viscosity on mass transfer coefficient.

mixing tanks with the other types of liquid-liquid contacting equipment, for packed columns, Sherwood et al. (17) report  $K_o a$  values of about  $15 \text{ hr.}^{-1}$  for the most optimum design and operation; for spray columns Johnson and Bliss (5) report  $K_o a$  values of about  $50 \text{ hr.}^{-1}$  for their best value. As against these the value of  $K_o a$  in this study was about  $630 \text{ hr.}^{-1}$  for corresponding liquid viscosities. The much higher values for the mixing tank are in part due to much larger interfacial areas per unit volume ( $A = a/V$ ) and in part to higher coefficients per unit area.

Figure 3 shows the effect of continuous-phase viscosity on mass transfer coefficients at 500 rev./min. The volumetric coefficient  $K_o A$  varies as the  $-0.60$  power of viscosity. Rodger (13) and de Vitry (2) have shown that viscosity has very little effect in the interfacial areas. This indicates that the coefficient  $K_o$  also varies as the  $-0.60$  power of the continuous-phase viscosity. Since diffusivity is inversely proportional to the viscosity, it implies that the coefficient  $K_o$ , or the rate of transfer, varies as the  $+0.60$  power of diffusivity. If the actual transfer mechanism can be represented by the two-film theory, then the transfer rate should be directly proportional to diffusivity. In accordance with the penetration theory the rate will be proportional to the square root (or  $0.5$  power) of diffusivity. The data appear to be closer to the square root variation which may mean that the transfer mechanism is by way of the penetration model.

Figure 5 shows the effect of dispersed-phase viscosity on the transfer coefficient. The coefficient varies as the  $-0.55$  power of viscosity (or  $+0.55$  power of diffusivity). This corresponds almost exactly to the square root and implies that surface renewal is the controlling mechanism in the dispersed phase. It is to be noted that the coefficient  $K_o$  is a measure of the mass transfer rate, and in accordance with the film theory it is equal to the ratio of diffusivity to fictitious film thickness. The surface renewal of the dispersed phase occurs owing to the coalescence and dispersion of the drops in the mixing tank as well as by diffusion. Thus the rates of mass transfer in liquid-liquid extraction seem to depend on surface renewal by diffusion and turbulence in the continuous phase and by the surface renewal by diffusion and coalescence and breakup in the dispersed phase.

## CONCLUSIONS

A batch experimental method wherein an instantaneous addition of an increment of the continuous phase is made has been used successfully to measure the liquid-liquid mass transfer coefficients for transfer to masses of drops, where the drops constituted about 10% of the total batch. The method has good reproducibility and should lend itself to large size equipment study and hence to relatively inexpensive experimentation to achieve scale-up data for the effects of all significant physical and operating variables.

Concentrations of solute in the continuous phase were measured by conductivity after the effects of the presence of a dispersed phase were accounted for. Octanoic acid has a slow measurable rate of mass transfer and was found to be the most suitable solute of the various electrolytes considered.

Viscosities of both phases, and the impeller speed, were found to be significant independent variables. The volumetric mass transfer coefficient decreases when the viscosity of either phase is increased, and the coefficient increases approximately as the square of the impeller speed.

The transfer rates are very high for the tank with a turbine type of mixing impeller compared with packed and spray towers. The rates appear to depend primarily

on the turbulence in the continuous phase and the surface renewal of the dispersed phase.

## ACKNOWLEDGMENT

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## NOTATION

$a$	= interfacial area per unit volume, sq. cm./cc.
$A$	= total interfacial area, sq. cm.
$C$	= concentration of solute, g. moles/liter
$V$	= volume, ml.
$v$	= volume of continuous phase addition containing solute, ml.
$K_o A$	= overall volumetric mass transfer coefficient, cc./sec.
$K_o a$	= overall coefficient, sec. <sup>-1</sup> (or hr. <sup>-1</sup> )
$N$	= speed, rev./min.
$D$	= impeller diameter
$\theta$	= time, sec.

## Subscripts

$o$	= continuous phase
$d$	= dispersed phase

## Sub Subscripts

$i$	= value of concentration or time, at a chosen initial value for mass transfer calculations
$o$	= value of concentration at the instance the mass transfer starts

## Superscripts

$*$	= equilibrium value
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