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UNIVERSITY OF LOUISVILLE

A STUDY OF DIFFUSION IN DISTILLATION
//

A Thesis

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

MASTER OF CHEMICAL ENGINEERING

Department of Chemical Engineering

Joseph G. Duffey
///

June, 1949

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A STUDY OF DIFFUSION IN DISTILLATION

Joseph G. Duffey

Approved by the Examining Committee

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June, 1949

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ABSTRACT

A study of the mass transfer phenomena in fractional distillation was made. Small scale single slot, single cap apparatus was used to obtain the necessary experimental data.

The mass transfer coefficient $K_G A$ was calculated using several combinations of liquid and vapor compositions in the methanol - water system under static liquid conditions. The correlation showed that $K_G A$ held about constant over the entire range of concentrations.

The problem of bubble area was studied and reasonable estimations of area, bubble frequency, and time of rise were made. This information is to be incorporated into the calculation of the mass transfer coefficient for the continuous procedure.

The apparatus used in the static liquid experiments was modified to permit a continuous procedure with liquid flowing across the plate. The steady state liquid and vapor compositions were determined and Murphree efficiencies were calculated. A study of possible methods of calculating the mass transfer coefficient K_G from the available data was made.

INTRODUCTION

Distillation is one of the most important and widely used unit operations in the field of Chemical Engineering. There are many methods of effecting a separation by distillation but normally the process is divided into two large categories: the continuous and the batch operations. These may be subdivided into the nature of the apparatus, but in both instances the industrial designers have turned to a plate column as a very useful design. The calculations for the design of necessary equipment involves a determination of the necessary separation trays, or plates as they are called. Although procedures for the calculation of the theoretical number of stages is available, translation of this figure into actual practice requires an estimation of plate efficiency.

Various factors that enter into the efficiencies of plates have been studied by many investigators with conflicting results. Nichols (1) summarized the information given in the literature and suggested an experimental unit in which some of the factors could be more accurately studied, and McKay (2) designed and constructed an experimental unit for this work. The latter also obtained information on some of the hydraulic factors affecting plate efficiency.

The purpose of this investigation is to continue the basic research and study the influence of mass transfer rates on these efficiencies.

HISTORICAL

The work of Sorel (3) at the end of the nineteenth century provided the first mathematical approach to a design of plate distillation columns. Previous to that time distillation was practiced, but with little scientific approach. Sorel used equilibrium relationships between liquid and vapor to obtain a number of plates theoretically necessary for a given separation. Since that time, much work has been done in an effort to determine the factors that fix the ratio of the calculated number of theoretical plates to the actually necessary number, a ratio commonly called overall plate efficiency.

Studies of hydraulic action, including effects of slot height, slot width, submergence, liquid path, baffling, the radius of action of bubbles from the slot, turbulence, bubble size, surface area, time of contact, etc., have been made by many investigators including Davies (4), Good (5), Williams (6), Carey (7), Geddes (8), McKay (2), and Davidson (9), and a number of empirical relationships have been established. The investigators have established beyond any reasonable doubt that the hydraulic relationships in themselves, although important, are not necessarily paramount. They have indicated that even though similar hydraulic conditions exist, plate efficiency may vary over a wide range.

Factors of vapor and liquid contact, as entrainment, frothing, vapor rates, plate spacing, and physical properties of the system have been studied by Kirschbaum (10), Peavy

and Baker (11), O'Connell (12), (14), Colburn (13), and Stigger (15), to mention a few. The relationships developed by these investigators indicates that enrichment is based upon mass transfer characteristics which in turn are dependent upon mutual diffusion rates as well as other physical conditions.

The conditions affecting mass transfer forms the primary objective for this investigation. Mass transfer is dependent on area, thickness of stagnant layers, total pressure, relative pressure differences, time, and a coefficient dependent upon the materials and the interaction, which is called the mass transfer coefficient.

In distillation work, the net condensation of the less volatile component from the vapor within the interface, and the net evaporation of the more volatile component within the interface takes place almost instantaneously.

The liquid and gas films present resistances to the transfer of material, hence the necessity for the driving force which is a direct function of the concentration differences across the films.

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

Murphree (23) has defined a point efficiency as the ratio of the change in gas composition on the plate to the change in composition corresponding to attainment of equilibrium.

This ratio may be expressed as:

$$E_m = \frac{y_2 - y_1}{y^* - y_1} \quad (1)$$

where E_m is the Murphree point efficiency
 y_1 is the partial pressure of entering vapor
 y_2 is the partial pressure of exit vapor
 y^* is the partial pressure of gas in equilibrium with the liquid.

In the usual plate construction it is common for the liquid to flow from one side to the other before overflowing to the next plate below. The result is something in between a true countercurrent action and the assumed single contact, and it is possible for the average composition of the gas leaving the plate to be less rich than would correspond to equilibrium with the liquid leaving the plate at one side. Therefore it is possible, and not uncommon, for either the Murphree or the overall plate efficiencies to be greater than 100%.

Geddes (8) presented a method using the film concept to determine plate efficiency and arrived at equation (2).

$$\ln (1 - E_m) = - \frac{3kt}{a} \quad (2)$$

where E_m is the local Murphree efficiency
 k is the overall transfer coefficient, (lb. mols)
 t is the time for a gas bubble to rise through a fluid, (seconds)
 a is the bubble radius, (feet)

Surowiec and Furnas (16) derived an equation analogous to Nusselt's work and adapted to mass transfer:

$$k_L = \frac{B'}{M} \left(\frac{D_L \rho_L^{\frac{5}{3}} g^{\frac{1}{3}} \Gamma^{\frac{1}{3}}}{3^{\frac{1}{3}} H^{\frac{1}{3}} \mu_L^{\frac{1}{3}}} \right)^{\frac{2}{3}} \quad (3)$$

where B' is a constant

g is acceleration due to gravity, ft./hr.²

Γ is peripheral liquid rate, lb./hr.)(ft.) of wetted perimeter

D_L is diffusivity in sq. ft./hr.

μ_L is viscosity of liquid, lb./hr.)(ft.)

H is column height, feet

M is molecular weight of more volatile component

From equation (2) they determined that the resistance was largely in the gas phase, using data obtained from a tower without packing.

Walter and Sherwood (17) indicated a relation between the transfer coefficient and the ratio of the slot velocity to its area. Their relation is represented by equation (4).

$$\ln(1 - E_m) = \frac{K_g A_{s1} h P}{V} \quad (4)$$

where E_m is the Murphree efficiency (point vapor)

K_g transfer coefficient, lb. mols/hr.)(atm.)
(sq. in. slot area)(in. liquid depth)

A_{s1} is total slot area/plate, (sq. in.)

h is effective liquid depth, in.

P is total pressure, atm.

V is gas rate, lb. mols/hr.

For equalmolar counter diffusion of two vapors, as

is approximated in rectification, Sherwood (22) presents the equation:

$$N_A = \frac{k}{x} \left(\frac{P_{A1} - P_{A2}}{P} \right) \quad (5)$$

where N_A is the rate of diffusion of component A,
g. mols/(sec.)(sq. cm.)
 k is a constant
 x is the distance in the direction of diffusion,
cm.
 P_{A1} is entering partial pressure, atm.
 P_{A2} is exit partial pressure, atm.
 P is total pressure, atm.

Litwin (18) and Taylor and Furnas (19) observed a material increase in the mass transfer coefficient when an increased surface area is exposed to the liquid.

The lack of agreement among investigators and the absence of reliable data on many of the factors involved in mass transfer have led to this research project and it is hoped that this research will add some clarification to the problem.

THEORETICAL

BUBBLE CHARACTERISTICS

Mass transfer in fractional distillation is dependent on the area of contact, or more specifically the area of the bubbles of vapor as they pass through the liquid.

For a long time the bubbles were assumed to be spheres. Geddes (8) in his calculation of plate efficiencies, assumes the bubbles to be spheres of radius

$$a = 0.78 \left(\frac{\sigma (h+w)}{\rho_L - \rho_V} \right)^{\frac{1}{3}} \quad (6)$$

where a is bubble radius, feet
 σ is surface tension, pounds per foot
 ρ_L is liquid density, pounds per cubic foot
 ρ_V is vapor density, pounds per cubic foot
 w is slot width, feet
 h is slot opening, feet

Photographs included in this thesis establish the fact that bubbles are neither spheres nor any regular geometric figure. In fact they are so irregular that it would be difficult to establish an equivalent sphere.

Davidson (9) recognized that the bubbles were not spheres and measured the volume of the bubbles. From this he calculated the radius of a sphere of equal volume as

$$a = \sqrt[3]{\frac{3v}{4\pi}} = 0.620 v^{\frac{1}{3}} \quad (7)$$

where a is the bubble radius, cm.
 v is the bubble volume, cc.

Inasmuch as it is almost impossible to measure the volume of a single bubble at even low vapor rates, the only way of getting an average volume is to measure the total volume and attempt to count the number of bubbles formed per unit time. Dividing the two values would give an average volume.

To use this method an accurate procedure for measuring the bubble frequency must be devised. Eversole, Wagner, and Stackhouse (20), working with capillaries, found the bubble frequency practically constant at about 53 bubbles per second. Davidson (21) attempted to correlate his data for circular orifices with the constant frequency equation

$$n = 5.29 \frac{q^{.047}}{r^{.477}} \quad (8)$$

where n is the bubble frequency, bubbles/sec.

q is the volumetric gas flow rate, cc/sec.

r is the orifice radius, cm.

Stroboscopic light has been used to measure frequency but in this case the possibilities of harmonics exist.

If the bubbles do attain a constant frequency as predicted by the literature, any increase in vapor flow would increase the volume of the bubbles rather than increasing the number of bubbles. Therefore as the vapor flow is increased, the surface area of the bubbles increase and this directly affects the mass transfer coefficient.

Geddes (8) assumes the time of contact to be

$$t = \frac{(\text{mean slot submergence})}{4 a^{0.37}} \quad (9)$$

where a is the radius of a sphere as calculated by equation (6).

From the above discussion it is evident that the area of contact must be approximated and that no accurate method of determining this area has been devised.

DETERMINATION OF MASS TRANSFER COEFFICIENTS BY THE STATIC SOLUTION METHOD

At any time after the start of an experiment a bubbling system may be considered as a series of bubbles existing in more or less vertical position between the orifice in the bubble cap and the surface of the liquid, i.e.

If these bubbles could be examined as to composition, they would have partial pressure of absorbable material, a , varying from p_a (entering) to p_a (leaving). The liquid around the bubbles would have been partially affected by the absorbing action and would therefore be somewhat higher in concentration

of "a" than the bulk of the absorbent, but for each bubble this quantity would be small, and would furthermore be controlled to a large extent by the kg/kl ratio. Since the distillation would be at the boiling point, kl is probably of relatively little significance, and since the amount of "a" in a single bubble is small, for calculation purposes the liquid concentration is therefore considered to be constant at the average bulk value.

With the assumed average bulk liquid concentration, an equilibrium $p_a(\text{liq.})$ could be evaluated from known data. This procedure gives a Δp for material transfer from gas to liquid of $(p_a \text{ entering} - p_a \text{ liq.})$ at the bubble cap orifice to $(p_a \text{ leaving} - p_a \text{ liq.})$ at the surface.

Basically the amount of material transferred over any period of time may be written as

$$w = K_g A (\Delta p_{\text{mean}}) \Theta \quad (10)$$

where w is moles of transferred absorbable material

$K_g A$ is mass transfer coefficient in mols/(min.)
 $(\Delta P_{\text{atmos.}})$

Θ is time of gas transfer to solution which is equivalent to a total contact time of the gas on a continuous vertical bubble basis.

Sherwood (22) presents an equation similar to this, equation (4), and also the type is found in other absorption calculations with the use of an overall mean ΔP .

Perry (23) presents the following equations:

$$\Delta p_{om} = \frac{(p_1 - p_1^*) - (p_2 - p_2^*)}{\ln \frac{(p_1 - p_1^*)}{(p_2 - p_2^*)}} \quad (11)$$

where Δp_{om} is overall log mean Δp
 p is partial pressure
 p^* is partial pressure of gas which would be in equilibrium with liquid

$$\int_0^{N_a} dN_a = V_1 \int_{Y_2}^{Y_1} dY = V_1 (Y_1 - Y_2) = K_g A p_{om} s \int_0^H dH \quad (12)$$

where N_a is number of moles of component "a"
 V_1 is lb.-mols of dry inert gas passing through system
 Y is composition of gas phase
 K_g is overall absorption coefficient, lb.-mols/(sq. ft.)(hr.)(atm.)
 A is active surface area, sq. ft.
 Δp_{om} is overall log mean Δp
 s is cross-sectional area of tower, sq. ft.
 H is height of tower, ft.

In all instances, p_{mean} is taken as a log mean driving force, and so, comparably, in this case it was also assumed to be acceptable.

$$\frac{dW}{d\theta} = K_g A (\Delta p_{lm}) \quad (13)$$

but from any starting zero concentration,

$$W = (\text{mols solvent})c \quad (14)$$

where c is concentration in mols of absorbed material/mol solvent.

$$\frac{V_w}{V_\theta} = (\text{mols solvent}) \frac{V_c}{V_\theta} \quad (15)$$

and substituting: $\frac{V_c}{V_\theta} (\text{mols solvent}) = K_g A (\Delta p_{lm})$. (16)

If the liquid concentration is obtained as a function of time of run, $\frac{V_c}{V_\theta}$ can be evaluated graphically, mols of solvent can be controlled, Δp_{lm} can be evaluated from the previous analyses, and $K_g A$ can be calculated. It is to be noted that this is a combination or overall constant and should be affected by many variables.

The classical analysis, as obtained from the literature, assumes that a single bubble is followed in its travel through a solution and transfer coefficients are evaluated from a unit area basis by calculation to an overall basis. The procedure indicated by the foregoing analysis is a reverse mechanism attempted because of the inadequacy of area values and contact times in bubble cap contact. The classical theory also assumes both constant vapor volume and contact area. Neither appear to be admissable in this problem.

DETERMINATION OF MASS TRANSFER COEFFICIENTS BY THE CONTINUOUS PROCEDURE METHOD

If vapor of constant composition is passed into a single slot bubble cap on a plate across which liquid of known composition is flowing, and an approximate area of contact is assumed, a mass transfer coefficient may be calculated.

A gas of known volume enters the solution as a bubble which grows on the slot, disengages, and passes up

through the fluid. The area of contact varies from zero to some unknown value just as before the bubble emerges from the slot, the contact area may be said to be zero, although there is some contact between vapor and liquid inside the cap. As the bubble is formed at the slot, it assumes a shape resembling a semi-ellipsoid although deviations from this solid are very pronounced at the top of the bubble. At a second position, midway between the cap and liquid surface, the bubble is mushroom shaped and has a volume about the same as at the slot. As an approximation, the bubble at this second position could be said to resemble an oblate spheroid. A third distinct bubble position is near the surface in a two inch liquid submergence but the bubble has about the same size and shape as at the second bubble position.

If the bubbles are assumed to have the shapes indicated, their areas may be calculated from known formulae. It seems reasonable to use the area at the second bubble position, assumed to be an oblate spheroid, as an average area of contact.

The time of contact at the gas - liquid interface is unknown, but estimates of this item may be made. The shape of the bubbles have an effect on the time of rise such that the rise of the bubble near the slot is faster than at any other position. However through experimental observations, an approximate value of time of rise may be determined.

The composition of the entering vapor may be expressed as p_{A1} and p_{B1} , both known, or as comparable y_{A1}

and y_{B1} . The composition of the exit vapor may be approximated by a condensation procedure and may be expressed as p_{A2} and p_{B2} , or as y_{A2} and y_{B2} .

The composition of liquid at initial contact is unknown, although the composition of the entering and exit liquids are known but due to the size of the system and the rate of vapor flow, these values are relatively close together. Thus either an average or final liquid composition could be used for reference.

After a short period of time, the exit liquid and vapor compositions hold about constant. If "w" mols of liquid per minute are being passed through the system:

$$w (x_{A1} - x_{A2}) = N_A \quad (17)$$

where w is liquid flow, mols/min.

x_{A1} is entering liquid composition, mol fraction

x_{A2} is exit liquid composition, mol fraction

N_A is no. mols of component A diffused/min.

The number of mols diffused as found by equation (17), is the total number of mols of A diffused per unit time into all of the bubbles rising through the liquid in the same period of time. Therefore care must be taken in specifying an area through which the mass transfer has taken place.

Keeping in mind the method used to obtain liquid and vapor compositions, area of contact, and mols diffused, an approximate equation of the form of equation (10) may be used:

$$N_A = K_g A (\Delta P_{\text{mean}}) \quad (18)$$

where N_A is no. of mols of component A diffused/min.

K_g is mass transfer coefficient, mols/(min.)
(atm.)(sq. ft.)

A is area of contact, sq. ft.

ΔP_{mean} is log mean ΔP of vapor compositions

The use of the log mean, while not exact, probably provides the best approximation to the actual conditions. For a more exact estimation of the mass transfer coefficient, an improved form of equation (18) must be developed. However since such an equation is not available at the present time, the experimental data may be correlated using Murphree efficiencies as calculated from equation (1). With the large variety of data available, the trends in the point efficiency should be evident.

EXPERIMENTAL

The apparatus used in performing the experiments of this investigation may be divided into three parts:

1. Apparatus for static liquid experiments.
2. Apparatus for continuous procedure.
3. Apparatus for determining bubble characteristics.

STATIC LIQUID EXPERIMENTS

In the static liquid experiments a wide - mouth reagent bottle of a capacity of about 2.5 liters was used. Inside this bottle was placed a single slot bubble cap constructed similar to the one used by Davidson (9). A piece of $2\frac{1}{2}$ inch diameter copper tubing was closed at one end by a rubber stopper which contained an opening for the vapor tube. The slot was two inches long and $\frac{1}{8}$ inch wide. The vapor passed through the slot and out another copper tube to the condenser. The liquid inside the bottle was set at a two inch submergence of the slot. In the top of the bottle was a stopper through which the entering and exit vapors passed in copper tubes, and an opening was provided for sampling. This stopper was tight enough so that no liquid or vapor could leak in or out of the bottle.

The bottle containing the bubble cap was submerged completely in a constant temperature water bath. This bath was a copper tank, 3 ft. in diameter and 3 ft. high, open at the top and equipped with a window so that the bubble cap could be easily viewed. Steam was passed through copper coils to

control the temperature to 0.5°C . An electric stirring device was installed to assist in maintaining the constant water temperature. Through use of this constant temperature bath, the solution in the bottle was held at its boiling point or slightly below it to prevent any spontaneous bubble formation.

In all of this work, it was important to know the composition of the entering vapor. Several methods for producing the vapor were tried, all working on the principle that if liquid of known composition is placed on a hot surface and let vaporize instantly without any build-up of liquid, the composition of the resulting vapor will be identical with the composition of the original liquid.

A cylindrical iron vessel, $3\frac{3}{4}$ in. diameter, 6 in. high, and $1/8$ in. thick, was found to be the best generator. The vessel was heated with a Meker burner and small pieces of copper tubing were placed in the bottom to give a greater heating surface. Two openings were provided in the metal top: one for the incoming liquid from a 250 ml. burette, and the other for the emerging vapor. Since it was not possible to see if any liquid was forming on the inside of the container, the only method of checking the liquid formation was by direct analysis of the liquid and vapor. Various mixtures of methanol and water were admitted, and in each instance the resulting vapor had the same composition and weight as the original liquid, within normal experimental accuracy.

The vapor emerging from the cap was passed through a copper tube to a glass coil condenser where the distillate was collected.

The method of operation of the static liquid apparatus was as follows. The constant temperature bath was filled to a level such that the glass bottle containing the bubble cap, which contained a definite amount of liquid, was completely submerged. The bath was then heated to the boiling point of the solution on the plate and held at that temperature. During the heating process, the electric stirring device was in operation and provided good agitation. After the bath was at the proper temperature, the vapor generator was heated with a Meker burner and a 250 ml. burette was filled. A stopwatch was started and the liquid in the burette was admitted at a definite rate. Every few minutes, samples of the liquid in the bottle and condensed vapor from the bottle were taken. These samples consisted of only a few drops so that the total volume of liquid and vapor was not changed appreciably. The run was continued for 25 minutes, after which the samples were analyzed with an Abbe refractometer. A plot of composition vs. refractive index provided the information necessary to graph the rate of change of liquid and vapor compositions with time.

The system methanol - water was investigated at several liquid and vapor compositions and rates, as this system included the central portion of the viscosity - volatility product range.

The data obtained in this section of the work are included completely in the appendix of the thesis, but all summary data are included in Table I and illustrated, together with the calculated K_GA values, in Figures 1 to 14. The totality of K_GA values are replotted in Figure 15 for observation of interrelationships.

The values of the mass transfer coefficient K_GA correlated into an approximately constant value regardless of liquid or vapor compositions in the methanol - water system. An average of the coefficients on a time basis showed that the K_GA was slightly higher at the initial part of a run and then worked into a nearly straight line function after 10 minutes of a run. This is reasonable since the compositions did change more rapidly at the start.

It may be noticed from the composite K_GA values that some scattered points are present. These may be attributed to the following factors:

1. Normal experimental errors.
2. A small error in taking the slope of the liquid line changes K_GA considerably.
3. A small error in liquid composition makes an appreciable error in p_{a1} since the slope of the equilibrium curve is great for low liquid concentrations.
4. Any error in p_{a1} makes a large difference in the denominator of the log term since the quantity $(p_{ag} - p_{a1})$ is usually a small number in rich vapor runs. This, of course, changes K_GA .

TABLE I. SUMMARY DATA OF METHANOL - WATER MASS TRANSFER
EXPERIMENTS

Run	Time	Mols Solvent	$P_{ag'}$	P_{ag}	P_{al}	$\frac{J_c}{J_B}$	KgA
1	5	27.5	.067	.20	.03	.05	15.9
	10	26.9	.118	.20	.085	.05	20.5
	15	26.0	.142	.20	.10	.05	19.5
	20	25.6	.1535	.20	.115	.05	21.7
	25	25.2	.162	.20	.125	.05	23.4
5,6,7	5	20.7	.27	.80	.21	.4	35.8
	10	19.4	.373	.80	.33	.4	43.1
	15	18.3	.428	.80	.405	.3	42.2
	20	17.6	.46	.80	.445	.157	25.7
	25	16.95	.475	.80	.470	.11	24.3
8,9	5	6.72	.028	0	.788	-2	18.7
	10	7.56	.053	0	.760	-2	20.65
	15	8.65	.075	0	.731	-2	24.6
	20	10.1	.083	0	.69	-1.6	24.7
	25	11.2	.10	0	.65	-1.25	23.2
	30	12.7	.113	0	.59	-1.0	24.2
10,11 12	5	55.5	.048	.20	.035	.02	18.7
	10	55.5	.078	.20	.20	.02	18.7
	15	55.6	.103	.20	.07	.02	15.7
	20	55.6	.113	.20	.08	.02	16.5
	25	55.7	.128	.20	.09	.02	16.6

TABLE I. SUMMARY DATA OF METHANOL - WATER MASS TRANSFER
EXPERIMENTS (CONT.)

Run	Time	Mols Solvent	P_{ag}	P_{ag}	P_{al}	$\frac{P_{al}}{P_{ag}}$	KgA
13,14	5	40.3	.519	0	.562	-0.3	59.9
	10	43.0	.500	0	.530	-0.2	49.4
	15	44.8	.498	0	.514	-0.1	31.2
	20	46.9	.498	0	.490	-0.1	38.7
	25	49.2	.498	0	.462	-0.1	25.0
15,16	5	55.0	.055	.40	.025	.056	22.5
	10	54.2	.136	.40	.055	.056	16.7
	15	53.2	.162	.40	.095	.056	19.0
	20	52.3	.184	.40	.125	.056	20.9
	25	51.2	.208	.40	.15	.056	21.8
17,18 19	5	32.4	.740	0	.692	-0.3	35.0
	10	34.5	.698	0	.674	-0.2	33.2
	15	36.0	.674	0	.660	-0.15	30.8
	20	37.4	.660	0	.647	-0.1	22.1
	25	38.7	.637	0	.635	-0.07	24.4
20,21	5	54.0	.130	.60	.095	.11	33.6
	10	52.9	.188	.60	.144	.10	30.0
	15	51.5	.227	.60	.180	.08	24.3
	20	50.8	.256	.60	.220	.07	24.4
	25	50.0	.280	.60	.245	.06	21.7

TABLE I. SUMMARY DATA OF METHANOL - WATER MASS TRANSFER
EXPERIMENTS (CONT.)

Run	Time	Mols Solvent	P_{ag}	P_{ag}	P_{al}	$\frac{v \cdot c}{v \cdot \theta}$	KgA
22,23, 24	5	20.0	.820	0	.785	-0.5	38.0
	10	21.4	.800	0	.775	-0.25	22.9
	15	23.7	.793	0	.759	-0.25	23.0
	20	25.5	.789	0	.749	-0.25	23.6
	25	27.2	.789	0	.738	-0.25	23.0
25,26, 27	5 - 25	Equilibrium					
29,30	5	31.4	.670	.80	.660	0.04	25.4
	10 - 25	Equilibrium					
31,32	5 - 25	Equilibrium					
33,34, 35	5	10.7	.906	.20	.878	1.7	20.5
	10	11.5	.900	.20	.870	1.7	22.5
	15	12.2	.892	.20	.866	1.7	23.7
	20	13.7	.881	.20	.852	1.7	26.9
	25	16.4	.870	.20	.830	1.7	32.8
36,37	5	37.9	.605	.80	.588	.06	29.5
	10 - 25	Equilibrium

FIGURE 1 METHANOL-WATER MASS TRANSFER EXPERIMENT

FIGURE 3 METHANOL - WATER MASS TRANSFER EXPERIMENTS

FIGURE 5 METHANOL - WATER MASS TRANSFER EXPERIMENTS

FIGURE 8 METHANOL - WATER MASS TRANSFER EXPERIMENTS

FIGURE 12 METHANOL - WATER MASS TRANSFER EXPERIMENTS

FIGURE 13 METHANOL - WATER MASS TRANSFER EXPERIMENTS

FIGURE 14 METHANOL - WATER MASS TRANSFER EXPERIMENTS

FIGURE 15 COMPOSITE K_{ga} VALUES, ETHANOL - WATER MASS TRANSFER EXPERIMENTS

5. Most deviation occurs during the first part of the run when the slopes of the curves are greatest. An error in composition during the first few minutes of the run could change the slope of the curve appreciably and therefore change K_gA . Mixing here is probably a factor as K_1 is actually not infinitely large.

6. Since only a few drops constitute a sample, the probability of getting a non-representative sample of liquid or vapor is great.

In an attempt to eliminate some of the above errors, at least two runs were made under each condition and the best curve was drawn in. The values as given should be fairly accurate although a few scattering points are noted.

Some of the runs reached equilibrium so rapidly that only one or two, and in some cases, no K_gA values could be computed.

Considering all of the above factors, the experimental evidence points to the fact that the mass transfer coefficient K_gA tends toward a constant value and that the concentration of liquid or vapor does not affect this value appreciably. The method of calculation of this coefficient, since it was on an overall basis, incorporated many variables. Apparently the effects of these variables cancel out and the coefficient holds approximately constant.

The apparatus for the continuous procedure consisted of the same constant temperature bath, vapor generator, bottle containing cap, bubble cap, and condenser as in the static

liquid experiments. The vapor flow was identical to the flow in the previous apparatus but it was necessary to provide liquid flow across the plate for these experiments.

A large glass container with a low outlet was placed above the constant temperature bath as a reservoir for the liquid. This was connected to the experimental plate by a coiled copper tube and the flow of liquid was by gravity. The liquid inlet was below the surface of the liquid on the plate. To remove liquid from the plate, a syphon system was constructed. This consisted of a copper tube running from below the liquid surface on the plate to outside the constant temperature bath. At the end of this copper tube was a length of rubber tubing with a screw clamp to control the flow. The level of liquid in the bottle was held constant and the exit flow was held constant, thus the rate of flow across the plate was known and able to be controlled.

The method of operation for the continuous procedure was the same as for the static liquid process except that the flow of liquid had to be controlled. Samples were taken every few minutes and analyzed by a refractometer.

The system methanol - water was investigated extensively at several liquid and vapor rates and compositions as methanol - water covers the central portion of the viscosity - volatility product range. Isolated tests in the systems carbon tetrachloride - benzene and acetone - water were made to complete the range of viscosity - volatility product, carbon tetrachloride - benzene being low and acetone - water being high.

Controlled experiments on the small scale apparatus provided information which should be reliable within normal experimental error. The apparatus was so designed that it would represent one section of one plate of a normal industrial fractionating column. One slot in the cap was used to enable observation of the individual bubbles in which the diffusional process takes place. A normal sized cap was used and the slot dimensions were comparable to those used in industry. The flow of liquid past the slot was in the usual fashion. Vapor came into the cap in the conventional manner and was removed from the system through a condenser. For a study of plate efficiency it seemed expedient that the basic parts of the process be investigated, thus the present design of equipment evolved.

In all three systems the steady state compositions were determined and are recorded in Table II. A variety of conditions was chosen so that the investigation would cover the mass transfer rate changes. All of the steady state compositions seem reasonable, although where compositions are close together the experimental error may be significant. When the proper mathematical relationship is developed, this data should indicate the trend of the mass transfer coefficient K_g .

The steady state compositions were used to determine the Murphree point efficiency and the results showed that the efficiencies for all three systems were in the same order of magnitude. However definite trends were noticed.

TABLE II. SUMMARY DATA OF CONTINUOUS PROCEDURE MASS TRANSFER
EXPERIMENTS

20 mol % CH_3OH vapor

0 mol % CH_3OH liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x %	y %	Equilibrium		E_m %
				x %	y %	
120	10	0.3	4.5	0.3	2.3	87.5
120	20	0.5	8.9	0.5	4.0	69.5
300	10	0.2	2.6	0.2	1.5	94.0

40 mol % CH_3OH vapor

0 mol % CH_3OH liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x %	y %	Equilibrium		E_m %
				x %	y %	
120	10	0.7	8.0	0.7	5.9	93.8
120	20	0.9	11.6	0.9	7.0	86.0
300	10	0.3	5.8	0.3	2.3	90.7

0 mol % CH_3OH vapor

40 mol % CH_3OH liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x %	y %	Equilibrium		E_m %
				x %	y %	
120	10	38.9	68.0	38.9	72.2	94.4
120	20	38.1	65.5	38.1	71.8	91.2
300	10	39.2	68.5	39.2	72.4	94.6

TABLE II. SUMMARY DATA OF CONTINUOUS PROCEDURE MASS TRANSFER
EXPERIMENTS (CONT.)

0 mol % CH_3OH vapor

20 mol % CH_3OH liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x	y	Equilibrium		E_m %
		%	%	x %	y %	
120	10	16.6	50.0	16.6	54.6	91.5
120	20	15.5	47.5	15.5	52.0	91.3
300	10	18.5	51.0	18.5	56.0	91.0

20 mol % CH_3OH vapor

40 mol % CH_3OH liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x	y	Equilibrium		E_m %
		%	%	x %	y %	
120	10	40.0	71.8	40.0	72.9	98.1
120	20	39.6	70.5	39.6	72.6	96.0
300	10	40.0	72.6	40.5	72.9	98.2

20 mol % CCl_4 vapor

80 mol % CCl_4 liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x	y	Equilibrium		E_m %
		%	%	x %	y %	
120	10	77.0	76.1	77.0	77.8	97.0
120	20	72.5	71.1	72.5	73.6	95.2
300	10	78.0	77.1	78.0	78.7	97.4

TABLE II. SUMMARY DATA OF CONTINUOUS PROCEDURE MASS TRANSFER
EXPERIMENTS (CONT.)

40 mol % CCl_4 vapor

80 mol % CCl_4 liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x %	y %	Equilibrium		E_m %
				x %	y %	
120	10	77.6	76.7	77.6	78.4	95.5
120	20	73.5	72.1	73.5	74.5	93.0
300	10	78.5	77.5	78.5	79.2	95.6

10 mol % CH_3COCH_3 vapor

7 mol % CH_3COCH_3 liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x %	y %	Equilibrium		E_m %
				x %	y %	
120	10	6.1	62.7	6.1	63.5	98.4
120	20	5.1	58.5	5.1	59.0	99.0
300	10	6.3	63.5	6.3	64.0	99.1

40 mol % CH_3COCH_3 vapor

7 mol % CH_3COCH_3 liquid

Liquid Rate cc/min.	Vapor Rate cc liq./min.	x %	y %	Equilibrium		E_m %
				x %	y %	
120	10	6.8	63.5	6.8	65.6	91.8
120	20	6.0	61.2	6.0	63.0	92.1
300	10	6.9	65.0	6.9	66.0	96.1

The efficiency seemed to be lower at higher vapor rates. This is to be expected since at higher rates the bubble volume increases but the area of contact does not increase proportionately. In industrial practice economic factors must be considered but it must be remembered that the lower the vapor rate is, the higher will be the efficiency.

Increasing the vapor composition increased the efficiency for a given liquid composition in the methanol - water system. This is reasonable because more of the material in which one is interested is being admitted to the system and the total change in composition is less.

An increase in liquid composition increased the efficiency for a constant vapor composition. A richer liquid would be expected to provide a more efficient change in vapor composition.

Increasing the liquid rate did not change the efficiency appreciably. Considering the rates used in these experiments, not much of a change would be expected. However if the liquid rate were increased 100 times, there could conceivably be a change in the efficiency.

The efficiencies for all three systems ran about the same but the carbon tetrachloride - benzene and acetone - water systems ran slightly higher than the methanol - water system. Where the efficiencies are close to 100%, the experimental error in determining compositions may be significant.

The Murphree point efficiencies as calculated from the data seem reasonable and it is believed that a proper mathematical relationship will give reliable information on the behavior of the mass transfer coefficient.

BUBBLE CHARACTERISTICS

Several pieces of apparatus were used in determining the characteristics of the bubbles such as size, shape, and frequency.

Light for observation of the bubbles was obtained from a commercial Strobotac, which is an instrument used to produce light of variable frequency, called stroboscopic light. A large glass cylinder, sealed at the bottom, was helpful in studying the bubbles at high submergence rates under various vapor rates. Submergence up to 20 inches could be used in this cylinder.

By using the Strobotac and outside calipers, the size of the bubbles could be approximated. This was found to be the best way of obtaining the dimensions of the bubbles.

Many observations of bubbles, both with stroboscopic light and in daylight, establish the fact that bubbles rising through a solution are not spherical, rather they are flat and mushroom shaped. The bubbles change shape as they emerge from the slot and proceed up through the liquid. However, the volumes of the bubbles at various levels appear to be about constant for a given vapor rate.

Since a sphere has the minimum area per unit volume, all calculations based on spheres are in error in the area

term toward the low area assumption. The mass transfer coefficient is a direct function of the area of contact, hence the difference between the area of a sphere and the actual area of the bubbles may make an appreciable difference in the calculation of the coefficient.

Stroboscopic observations indicate that the best geometric figure to use as the average volume of the bubbles is an oblate spheroid. In a two inch depth of liquid, bubbles appear to have three distinct positions under stroboscopic light: one bubble at the slot, another at a medium depth, and a third near the surface of the liquid. The bubble at the medium depth, called the second bubble position, was estimated to be about an average size bubble. If this bubble is assumed to be an oblate spheroid, its dimensions being known by direct measurement with calipers and a stroboscope, conventional formulae may be used to calculate the area and volume of the bubbles. This method of estimating the area of a bubble is not entirely accurate but is the best approximation to the actual area that can be made at this time.

In order to establish more accurately the volume of a bubble, putty models were made using the measured dimensions of the bubbles and their shapes. To get the volumes, the models were placed in graduates containing water and the volume was found by observing the change in liquid level.

High speed photographs were taken to establish definitely the shapes of the bubbles. The shutter speed was fast enough so that the bubbles could be seen individually. Figures 16 - 23 show the bubbles at various vapor rates in the

Fig. 16

Photograph of Bubbles

Vapor Rate = 0.5 cfm
Submergence = 6.25 in.

Fig. 17

Photograph of Bubbles

Vapor Rate = 0.5 cfm
Submergence = 6.25 in.

Fig. 18

Photograph of Bubbles

Vapor Rate = 0.5 cfm
Submergence = 6.25 in.

Fig. 19

Photograph of Bubbles

Vapor Rate = 0.5 cfm
Submergence = 6.25 in.

Fig. 20

Photograph of Bubbles

Vapor Rate = 0.5 cfm
 Submergence = 6.25 in.
 Side View

Fig. 21

Photograph of Bubbles

Vapor Rate = 0.5 cfm
 Submergence = 6.25 in.
 Side View

Fig. 22

Photograph of Bubbles

Vapor Rate = 0.05 cfm

Submergence = 7 in.

Fig. 23

Photograph of Bubbles

Vapor Rate = 0.05 cfm

Submergence = 10 in.

Circular orifice

air - water system. Also the photographs show that the shape of the orifice has no effect on the shape of the resulting bubbles. The extremes of a circular horizontal orifice and a rectangular vertical orifice were used to establish this fact.

The Strobotac was tried as a means of measuring the frequency of the bubbles but it was difficult to get consistent readings due to the irregularity of bubble formation and the irregular sizes and shapes of the bubbles. However an approximate value of frequency was obtained although the possibility of harmonics was present.

Another method of obtaining the bubble frequency was tried using the stroboscopic principle, although this time relating it to sound. An electronic instrument called a Strobeconn was used. This instrument had twelve stroboscopic tubes upon which the frequency of any note in the range of the piano could be read. It is used to tune musical instruments in bands and orchestras.

Since the bubbles do make a sound when they emerge from the slot, it was postulated that perhaps the frequency with which the bubbles formed would correspond to some note on the musical scale. Knowing the frequency, or harmonic, of the musical note, the bubble frequency would be determined.

The microphone of the instrument was held close to the liquid and it was found that a reading was available on the Strobeconn. In any measurement of sound, the harmonics of the fundamental tone are also present. Thus the observed reading may have been a harmonic of the actual bubble fre-

quency. However the Stroboconn did give an indication of the approximate bubble frequency.

In measuring the frequency of bubble formation using stroboscopic methods, the presence of harmonics made the actual frequency uncertain. The frequency appeared to be about constant at approximately 20 bubbles/sec., but this value could be 40 bubbles/sec. when harmonics are considered. The constant frequency observation is consistent with predictions in the literature. Both the Stroboscope and the Stroboconn gave readings in the same order of magnitude. It is believed that stroboscopic apparatus is one of the best means of studying bubble rates and shape.

Calculation of the frequency of the bubbles from a knowledge of the total amount of entering vapor and an approximate volume of a single bubble, gave a frequency varying from 20 to 40 bubbles/sec. which agreed with a harmonic of the stroboscopic measurements. From experimental observations the range of bubble frequency may be set as 20 to 40 bubbles per second, but more refined measurements must be made before the exact frequency may be determined.

The Stroboconn also gave good information as to whether the frequency of bubbles reaches a constant value or not. The vapor flow was started and a scope on the instrument indicated a definite note. As the flow was increased, the note showing on the scope did not change for normal vapor rates. This indicated that the frequency of bubble formation was holding constant even though the rate of flow was changing.

The time necessary for a bubble to pass through a two inch depth of liquid under normal vapor rates is so minute as to be difficult to estimate. However an extrapolation procedure gave some idea of the order of magnitude of the time. Figure 24 shows the time of rise from the point at which the bubble breaks from the slot to the point at which it meets the surface of the liquid. This is not the total time of contact since the bubble was exposed to the liquid during the time it was growing on the slot and during the time it was disintegrating at the surface of the liquid. The experiments therefore measured the time for the bubble to pass through the second and third positions. To correct for the first position time of contact, assuming that the time is about the same at each position, it would be necessary to add one half of the measured time (0.05 sec.) to the time required to pass through the final two stages to get the total time of contact. Thus the experimental value for the total time of contact in a 2 inch submergence is 0.15 sec. This is admittedly a rough estimate of the time of contact, but is considered to be a fair approximation since only visual measurements were made.

It must be remembered when evaluating the correlations of this investigation, that whenever the area term appears, an approximation has been made. Extensive work was done to attain the best approximation so that while the values may not be entirely accurate, they should not be too much in error.

FIGURE 24 DETERMINATION OF TIME OF RISE OF BUBBLES

SUMMARY AND CONCLUSIONS

Experimentation with a variety of concentrations of liquid and vapor in the methanol - water system using a static procedure produced values of the mass transfer coefficient K_gA that were almost constant at around 23 mols/(min.)(atm.). While some scattered points were noted, the correlation seemed to be within the limits of error inherent in the method of experimentation.

The use of a continuous procedure provided data from which the mass transfer coefficient K_g may be calculated. Inasmuch as the proper relationship for calculating this coefficient is not immediately available, the data were used to calculate Murphree point efficiencies for the various conditions. The results showed the efficiency to increase under higher vapor compositions and higher liquid compositions. The efficiency was lower at higher vapor rates and the efficiency did not change appreciably under an increased liquid rate.

Bubbles of vapor passing through a liquid are not spherical but are flat and mushroom shaped. The best approximation to an average area would be to assume the bubble to be an oblate spheroid and calculate the area. The time of rise was estimated by an extrapolation procedure and the frequency of bubble formation was found by use of stroboscopic instruments. Any calculation of this nature involving the area of contact must be evaluated with the knowledge that the area has been estimated and not rigidly determined.

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APPENDIX

LIST OF SYMBOLS

A	Area of contact
E_m	Murphree point efficiency
K_g	Mass transfer coefficient, gas film, mols/(min.)(atm.)
K_l	Mass Transfer coefficient, liquid film
c	Concentration, mols absorbed material/mol solvent
p	Partial pressure, atm.
w	Transferred absorbable material, mols
x	Mol fraction in liquid
y	Mol fraction in vapor

Subscripts

A	Component A
B	Component B
g	Vapor entering
g'	Vapor leaving
1	Entering
2	Exit

Greek

Δ	Change in
$\ominus$	Time, minutes

SAMPLE CALCULATIONS

STATIC LIQUID EXPERIMENTS

Run 1 at $\theta = 25$ minutes

Wt. of solution in flask @ 25 minutes = 470 g.

$$\frac{\frac{x}{32}}{\frac{x}{32} + \frac{470 - x}{18}} = 0.0185$$

$$x = 15.7 \text{ g CH}_3\text{OH}$$

$$470 - 15.7 = 454.3 \text{ g solvent}$$

$$\frac{454.3}{18} = 25.2 \text{ mols solvent}$$

$$\text{Slope of liquid line} = \frac{\frac{d c}{d \theta}}{\frac{d c}{d \theta}} = .05$$

$$p_{ag'} = .162$$

$$p_{ag} = .20$$

$$p_{al} = .125$$

$$\frac{\frac{d c}{d \theta} (\text{mols solvent})}{\frac{d c}{d \theta}} = \frac{K_G A (p_{ag'} - p_{al}) - (p_{ag} - p_{al})}{\ln \frac{(p_{ag'} - p_{al})}{(p_{ag} - p_{al})}}$$

$$.05(25.2) = K_G A \frac{(.20 - .125) - (.162 - .125)}{\ln \frac{(.20 - .125)}{(.162 - .125)}}$$

$$K_G A = 23.4$$

SAMPLE CALCULATIONS (CONT.)

CONTINUOUS PROCEDURE EXPERIMENTS

Calculation of Murphree Efficiency

Methanol - Water system: 0 mol % CH_3OH vapor40 mol % CH_3OH liquid

vapor rate = 20 cc/min.

Liquid rate = 120 cc/min.

$$E_m = \frac{y_2 - y_1}{y^* - y_1}$$

$$E_m = \frac{65.5 - 0}{71.8 - 0} = 91.2\%$$

Table No. III

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 5055 Run No. 1
 Solution wt., final, gms. = 4700 Initial Concentrations:
 Vapor rate, gms./run = 237 Vapor, mol % methanol = 20
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 0
 Vapor condensate, gms./run = 250 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3367	20.....	13.8..
..2.....	".....	V.....	1.3295	0.....	0....
..3.....	6.....	L.....	1.3331	8.1.....	5.0..
..4.....	".....	V.....	1.3302	1.0.....	0.5..
..5.....	10.....	L.....	1.3344	11.8.....	7.5..
..6.....	".....	V.....	1.3298	0.....	0....
..7.....	15.....	L.....	1.3351	13.9.....	9.0..
..8.....	".....	V.....	1.3302	1.0.....	0.5..
..9.....	20.....	L.....	1.3356	15.5.....	10.4..
..10.....	".....	V.....	1.3304	1.4.....	0.75..
..11.....	25.....	L.....	1.3358	16.2.....	10.9..
..12.....	".....	V.....	1.3306	1.9.....	1.2..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. ~~IV~~

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 400 Run No. 5
 Solution wt., final, gms. = 328.7 Initial Concentrations:
 Vapor rate, gms./run = 207 Vapor, mol % methanol = 80
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 0
 Vapor condensate, gms./run = 293 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3310	81.5	260..
..2.....	".....	V.....	1.3298	0	0....
..3.....	6.....	L.....	1.3311	3.1	1.8..
..4.....	".....	V.....	1.3370	21.0	14.8..
..5.....	10.....	L.....	1.3382	28.8	22.7..
..6.....	".....	V.....	1.3318	4.8	3.0..
..7.....	15.....	L.....	1.3386	41.0	37.5..
..8.....	".....	V.....	1.3328	7.4	4.5..
..9.....	20.....	L.....	1.3387	40.0	37.0..
..10.....	".....	V.....	1.3351	13.9	9.1..
..11.....	25.....	L.....	1.3384	42.8	40.0..
..12.....	".....	V.....	1.3344	11.8	7.5..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. V

Static Mass Transfer Experiments
with Methanol-Water System

Solution wt., initial, gms. = <u>400</u>	Run No. <u>6</u>
Solution wt., final, gms. = <u>386</u>	Initial Concentrations:
Vapor rate, gms./run = <u>207</u>	Vapor, mol % methanol = <u>80</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>0</u>
Vapor condensate, gms./run = <u>240</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L....	1.3310	81.5	26.0
..2.....	".....	V....	1.3298	0	0
..3.....	6.....	L....	1.3370	21.0	14.8
..4.....	".....	V....	1.3310	2.8	1.7
..5.....	10.....	L....	1.3381	28.0	21.8
..6.....	".....	V....	1.3325	6.6	4.0
..7.....	15.....	L....	1.3384	43.7	43.0
..8.....	".....	V....	1.3330	7.9	4.8
..9.....	20.....	L....	1.3381	45.4	46.0
..10.....	".....	V....	1.3340	10.7	6.7
..11.....	25.....	L....	1.3378	47.7	50.0
..12.....	".....	V....	1.3348	13.0	8.5

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. VI

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = <u>400</u>	Run No. <u>7</u>
Solution wt., final, gms. = <u>383</u>	Initial Concentrations:
Vapor rate, gms./run = <u>207</u>	Vapor, mol % methanol = <u>80</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>0</u>
Vapor condensate, gms./run = <u>240</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3309	82.0	275.
..2.....	".....	V.....	1.3298	0.0	0...
..3.....	6.....	L.....	1.3367	19.6	13.2.
..4.....	".....	V.....	1.3307	2.1	1.3..
..5.....	10.....	L.....	1.3381	28.0	21.9.
..6.....	".....	V.....	1.3319	5.0	3.0..
..7.....	15.....	L.....	1.3385	42.0	40.0.
..8.....	".....	V.....	1.3329	7.6	4.7..
..9.....	20.....	L.....	1.3382	44.5	44.0.
..10.....	".....	V.....	1.3344	11.8	7.5..
..11.....	25.....	L.....	1.3378	47.7	48.0.
..12.....	".....	V.....	1.3348	13.0	8.5..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. VII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 350 Run No. 8
 Solution wt., final, gms. = 318 Initial Concentrations:
 Vapor rate, gms./run = 150 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 5 Liquid, mol % methanol = 60
 Vapor condensate, gms./run = 205 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3298	0.0	0..
..2.....	".....	V.....	1.3360	60.0	8.3..
..3.....	6.....	L.....	1.3222	5.7	3.5..
..4.....	".....	V.....	1.3371	52.6	6.4..
..5.....	10.....	L.....	1.3322	5.7	3.5..
..6.....	".....	V.....	1.3379	47.0	5.0..
..7.....	15.....	L.....	1.3328	7.4	4.5..
..8.....	".....	V.....	1.3384	42.8	4.0..
..9.....	20.....	L.....	1.3339	8.9	5.5..
..10.....	".....	V.....	1.3376	24.5	17.5..
..11.....	25.....	L.....	1.3343	11.5	7.3..
..12.....	".....	V.....	1.3368	20.0	14.0..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. VIII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 350 Run No. 9
 Solution wt., final, gms. = 339 Initial Concentrations:
 Vapor rate, gms./run = 150 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 5 Liquid, mol % methanol = 60
 Vapor condensate, gms./run = 185 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3298	0.0	0....
..2.....	".....	V.....	1.3360	60.0	83....
..3.....	6.....	L.....	1.3323	6.0	3.7...
..4.....	".....	V.....	1.3373	51.3	5.8....
..5.....	10.....	L.....	1.3310	2.8	1.7....
..6.....	".....	V.....	1.3378	47.7	50.2...
..7.....	15.....	L.....	1.3327	7.0	4.4....
..8.....	".....	V.....	1.3386	40.9	32....
..9.....	20.....	L.....	1.3339	10.4	6.4....
..10.....	".....	V.....	1.3381	28.0	20....
..11.....	25.....	L.....	1.3343	11.5	7.3....
..12.....	".....	V.....	1.3374	23.4	17....

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. IX

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 10
 Solution wt., final, gms. = 980 Initial Concentrations:
 Vapor rate, gms./run = 237 Vapor, mol % methanol = 20
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 0
 Vapor condensate, gms./run = 225 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3304	1.4	0.75
..2.....	".....	V.....	1.3309	2.5	1.6
..3.....	6.....	L.....	1.3305	1.6	1.0
..4.....	".....	V.....	1.3321	5.5	3.4
..5.....	10.....	L.....	1.3307	2.1	1.3
..6.....	".....	V.....	1.3333	8.6	5.3
..7.....	15.....	L.....	1.3305	1.6	1.0
..8.....	".....	V.....	1.3340	10.7	6.6
..9.....	20.....	L.....	1.3307	2.1	1.3
..10.....	".....	V.....	1.3344	11.8	7.5
..11.....	25.....	L.....	1.3309	2.6	1.7
..12.....	".....	V.....	1.3346	12.4	8.0

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. X

Static Mass Transfer Experiments
with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>11</u>
Solution wt., final, gms. = <u>1006</u>	Initial Concentrations:
Vapor rate, gms./run = <u>237</u>	Vapor, mol % methanol = <u>20</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>0</u>
Vapor condensate, gms./run = <u>225</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3299	0.3	0.2..
..2.....	".....	V.....	1.3312	3.3	2.0..
..3.....	6.....	L.....	1.3300	0.5	0.3..
..4.....	".....	V.....	1.3319	5.0	3.1..
..5.....	10.....	L.....	1.3301	0.7	0.4..
..6.....	".....	V.....	1.3328	7.4	4.5-
..7.....	15.....	L.....	1.3301	0.7	0.4..
..8.....	".....	V.....	1.3338	10.0	6.2..
..9.....	20.....	L.....	1.3302	1.0	0.6..
..10.....	".....	V.....	1.3344	11.8	7.5..
..11.....	25.....	L.....	1.3303	1.2	0.7..
..12.....	".....	V.....	1.3347	12.7	8.3..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XI

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1110 Run No. 12
 Solution wt., final, gms. = 1010 Initial Concentrations:
 Vapor rate, gms./run = 237 Vapor, mol % methanol = 20
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 0
 Vapor condensate, gms./run = 225 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3299	0.3	0.2
..2.....	".....	V.....	1.3310	2.8	1.7
..3.....	6.....	L.....	1.3300	0.5	0.3
..4.....	".....	V.....	1.3318	4.8	3.0
..5.....	10.....	L.....	1.3301	0.7	0.4
..6.....	".....	V.....	1.3327	7.0	4.3
..7.....	15.....	L.....	1.3302	1.0	0.6
..8.....	".....	V.....	1.3339	10.4	6.4
..9.....	20.....	L.....	1.3303	1.2	0.7
..10.....	".....	V.....	1.3347	12.7	8.3
..11.....	25.....	L.....	1.3303	1.2	0.7
..12.....	".....	V.....	1.3348	12.9	8.4

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 43
 Solution wt., final, gms. = 1105 Initial Concentrations:
 Vapor rate, gms./run = 250 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 20
 Vapor condensate, gms./run = 130 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3368	20.0	13.8
..2.....	".....	V.....	1.3334	69.0	124
..3.....	6.....	L.....	1.3364	18.4	12.6
..4.....	".....	V.....	1.3360	55.5	69
..5.....	10.....	L.....	1.3358	16.1	10.8
..6.....	".....	V.....	1.3368	49.8	55
..7.....	15.....	L.....	1.3355	15.2	10.0
..8.....	".....	V.....	1.3368	49.8	55
..9.....	20.....	L.....	1.3350	13.6	9.0
..10.....	".....	V.....	1.3368	49.8	55
..11.....	25.....	L.....	1.3345	12.1	7.8
..12.....	".....	V.....	1.3368	49.8	55

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XIII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 14
 Solution wt., final, gms. = 1092 Initial Concentrations:
 Vapor rate, gms./run = 250 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 20
 Vapor condensate, gms./run = 140 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3367	19.6	13.5
..2.....	".....	V.....	1.3364	52.6	62.
..3.....	6.....	L.....	1.3361	17.2	11.7
..4.....	".....	V.....	1.3361	54.8	68.
..5.....	10.....	L.....	1.3357	16.1	10.8
..6.....	".....	V.....	1.3367	50.5	57.
..7.....	15.....	L.....	1.3354	14.8	9.8
..8.....	".....	V.....	1.3368	49.8	55
..9.....	20.....	L.....	1.3350	13.6	9.0
..10.....	".....	V.....	1.3368	49.8	55-
..11.....	25.....	L.....	1.3344	11.8	7.5
..12.....	".....	V.....	1.3368	49.8	55-

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XIV

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 15
 Solution wt., final, gms. = 970 Initial Concentrations:
 Vapor rate, gms./run = 227 Vapor, mol % methanol = 40
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 0
 Vapor condensate, gms./run = 248 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3299	0.3	0.2..
..2.....	".....	V.....	1.3321	5.5	3.4..
..3.....	6.....	L.....	1.3300	0.5	0.3..
..4.....	".....	V.....	1.3337	9.8	6.0..
..5.....	10.....	L.....	1.3301	0.7	0.4..
..6.....	".....	V.....	1.3350	13.6	9.0..
..7.....	15.....	L.....	1.3303	1.2	0.7..
..8.....	".....	V.....	1.3362	17.6	12.0..
..9.....	20.....	L.....	1.3305	1.7	1.0..
..10.....	".....	V.....	1.3364	18.4	12.6..
..11.....	25.....	L.....	1.3308	2.3	1.4..
..12.....	".....	V.....	1.3370	21.1	14.8..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XV

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 16
 Solution wt., final, gms. = 950 Initial Concentrations:
 Vapor rate, gms./run = 227 Vapor, mol % methanol = 40
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 0
 Vapor condensate, gms./run = 270 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3299	0.3	0.4...
..2.....	".....	V.....	1.3321	5.5	3.4...
..3.....	6.....	L.....	1.3300	0.5	0.3...
..4.....	".....	V.....	1.3336	9.5	5.8...
..5.....	10.....	L.....	1.3301	0.7	0.4...
..6.....	".....	V.....	1.3350	13.6	9.0...
..7.....	15.....	L.....	1.3304	1.4	0.8...
..8.....	".....	V.....	1.3354	14.8	9.9...
..9.....	20.....	L.....	1.3306	1.8	1.2...
..10.....	".....	V.....	1.3364	18.4	12.6...
..11.....	25.....	L.....	1.3308	2.3	1.4...
..12.....	".....	V.....	1.3369	20.6	14.3...

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XVI

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 17
 Solution wt., final, gms. = 1152 Initial Concentrations:
 Vapor rate, gms./run = 250 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 40
 Vapor condensate, gms./run = 80 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3384	36.0	31.8
..2.....	".....	V.....	1.3308	78.9	20.5
..3.....	6.....	L.....	1.3383	33.0	27.8
..4.....	".....	V.....	1.3319	74.1	16.0
..5.....	10.....	L.....	1.3382	31.4	25.5
..6.....	".....	V.....	1.3365	51.9	6.0
..7.....	15.....	L.....	1.3381	30.0	24.0
..8.....	".....	V.....	1.3334	69.9	12.9
..9.....	20.....	L.....	1.3380	28.9	22.7
..10.....	".....	V.....	1.3322	75.4	17.1
..11.....	25.....	L.....	1.3379	28.0	21.8
..12.....	".....	V.....	1.3342	66.1	11.0

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XVII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 18
 Solution wt., final, gms. = 1141 Initial Concentrations:
 Vapor rate, gms./run = 250 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 40
 Vapor condensate, gms./run = 80 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3384	36.0	31.8
..2.....	".....	V.....	1.3315	78.4	20.0
..3.....	6.....	L.....	1.3382	31.4	25.7
..4.....	".....	V.....	1.3328	72.6	14.8
..5.....	10.....	L.....	1.3382	31.4	25.7
..6.....	".....	V.....	1.3334	68.9	12.9
..7.....	15.....	L.....	1.3381	30.0	24.0
..8.....	".....	V.....	1.3341	66.5	11.0
..9.....	20.....	L.....	1.3378	27.0	20.8
..10.....	".....	V.....	1.3350	62.0	9.1
..11.....	25.....	L.....	1.3376	25.4	19.2
..12.....	".....	V.....	1.3315	78.4	20.0

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XVIII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. =	<u>1000</u>	Run No. <u>19</u>
Solution wt., final, gms. =	<u>1131</u>	Initial Concentrations:
Vapor rate, gms./run =	<u>250</u>	Vapor, mol % methanol = <u>0</u>
Vapor rate, cc liq./min. =	<u>10</u>	Liquid, mol % methanol = <u>40</u>
Vapor condensate, gms./run =	<u>100</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3383	33.0	27.8
..2.....	".....	V.....	1.3328	72.6	148
..3.....	6.....	L.....	1.3381	30.0	24.0
..4.....	".....	V.....	1.3332	71.0	136
..5.....	10.....	L.....	1.3380	28.9	22.7
..6.....	".....	V.....	1.3337	68.3	120
..7.....	15.....	L.....	1.3380	28.9	22.7
..8.....	".....	V.....	1.3338	67.7	116
..9.....	20.....	L.....	1.3380	28.9	22.7
..10.....	".....	V.....	1.3341	66.5	110
..11.....	25.....	L.....	1.3379	28.0	21.8
..12.....	".....	V.....	1.3368	49.8	55

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XIX

Static Mass Transfer Experiments
with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>20</u>
Solution wt., final, gms. = <u>970</u>	Initial Concentrations:
Vapor rate, gms./run = <u>216</u>	Vapor, mol % methanol = <u>60</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>0</u>
Vapor condensate, gms./run = <u>250</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3299	0.3	0.2
..2.....	".....	V.....	1.3342	11.2	7.0
..3.....	6.....	L.....	1.3303	1.2	0.7
..4.....	".....	V.....	1.3351	13.9	9.2
..5.....	10.....	L.....	1.3307	2.1	1.3
..6.....	".....	V.....	1.3365	18.8	12.9
..7.....	15.....	L.....	1.3311	3.0	1.8
..8.....	".....	V.....	1.3368	20.0	13.8
..9.....	20.....	L.....	1.3314	3.8	2.4
..10.....	".....	V.....	1.3377	26.1	20.0
..11.....	25.....	L.....	1.3316	4.3	2.7
..12.....	".....	V.....	1.3379	28.0	21.8

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XX

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 21
 Solution wt., final, gms. = 965 Initial Concentrations:
 Vapor rate, gms./run = 216 Vapor, mol % methanol = 60
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 0
 Vapor condensate, gms./run = 253 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3300	0.5	0.3
..2.....	".....	V.....	1.3336	9.5	5.8
..3.....	6.....	L.....	1.3303	1.2	0.7
..4.....	".....	V.....	1.3351	13.9	9.2
..5.....	10.....	L.....	1.3307	2.1	1.3
..6.....	".....	V.....	1.3365	18.8	12.9
..7.....	15.....	L.....	1.3310	2.8	1.7
..8.....	".....	V.....	1.3372	22.7	16.5
..9.....	20.....	L.....	1.3313	3.6	2.3
..10.....	".....	V.....	1.3375	24.6	18.5
..11.....	25.....	L.....	1.3316	4.3	2.7
..12.....	".....	V.....	1.3379	28.0	21.8

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXI

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 22
 Solution wt., final, gms. = 1135 Initial Concentrations:
 Vapor rate, gms./run = 250 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 60
 Vapor condensate, gms./run = 100 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3367	51.5	59.0.
..2.....	".....	V.....	1.3299	82.1	275
..3.....	6.....	L.....	1.3370	49.4	55.0.
..4.....	".....	V.....	1.3306	79.6	215
..5.....	10.....	L.....	1.3375	45.7	46.5
..6.....	".....	V.....	1.3308	78.9	20.5
..7.....	15.....	L.....	1.3378	43.4	40.2
..8.....	".....	V.....	1.3316	75.4	171
..9.....	20.....	L.....	1.3379	42.5	40.0
..10.....	".....	V.....	1.3314	76.3	180
..11.....	25.....	L.....	1.3380	41.7	39.0
..12.....	".....	V.....	1.3312	77.1	188

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 23
 Solution wt., final, gms. = 1142 Initial Concentrations:
 Vapor rate, gms./run = 250 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 60
 Vapor condensate, gms./run = 94 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3361	55.2	69.0
..2.....	".....	V.....	1.3300	81.8	26.5
..3.....	6.....	L.....	1.3370	49.4	55.0
..4.....	".....	V.....	1.3309	78.4	20.0
..5.....	10.....	L.....	1.3373	47.3	50.0
..6.....	".....	V.....	1.3318	74.6	16.3
..7.....	15.....	L.....	1.3378	43.4	40.2
..8.....	".....	V.....	1.3313	76.7	18.2
..9.....	20.....	L.....	1.3381	41.0	38.0
..10.....	".....	V.....	1.3316	75.4	17.1
..11.....	25.....	L.....	1.3380	41.7	39.0
..12.....	".....	V.....	1.3314	76.3	18.0

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXIII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 24
 Solution wt., final, gms. = 1120 Initial Concentrations:
 Vapor rate, gms./run = 250 Vapor, mol % methanol = 0
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 60
 Vapor condensate, gms./run = 110 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3360	55.8	70.0
..2.....	".....	V.....	1.3305	80.0	22.0
..3.....	6.....	L.....	1.3368	50.8	63.0
..4.....	".....	V.....	1.3308	78.9	20.5
..5.....	10.....	L.....	1.3370	49.4	55.0
..6.....	".....	V.....	1.3311	77.5	19.1
..7.....	15.....	L.....	1.3371	48.6	53.0
..8.....	".....	V.....	1.3312	77.1	18.8
..9.....	20.....	L.....	1.3375	45.7	46.5
..10.....	".....	V.....	1.3313	76.7	18.2
..11.....	25.....	L.....	1.3380	41.7	39.0
..12.....	".....	V.....	1.3314	76.3	18.0

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXIV

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 25
 Solution wt., final, gms. = 965 Initial Concentrations:
 Vapor rate, gms./run = 216 Vapor, mol % methanol = 60
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 20
 Vapor condensate, gms./run = 230 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3368	20.0	13.9..
..2.....	".....	V.....	1.3356	57.9	77.0..
..3.....	6.....	L.....	1.3368	20.0	13.9..
..4.....	".....	V.....	1.3356	57.9	77.0..
..5.....	10.....	L.....	1.3368	20.0	13.9..
..6.....	".....	V.....	1.3358	56.8	73.0..
..7.....	15.....	L.....	1.3369	20.8	14.6..
..8.....	".....	V.....	1.3357	57.4	75.0..
..9.....	20.....	L.....	1.3370	21.4	15.2..
..10.....	".....	V.....	1.3356	57.9	77.0..
..11.....	25.....	L.....	1.3369	20.8	14.6..
..12.....	".....	V.....	1.3357	57.4	75.0..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXV

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 26
 Solution wt., final, gms. = 965 Initial Concentrations:
 Vapor rate, gms./run = 216 Vapor, mol % methanol = 60
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 20
 Vapor condensate, gms./run = 212 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3368	20.0	13.9..
..2.....	".....	V.....	1.3357	57.4	75.0..
..3.....	6.....	L.....	1.3369	20.8	14.6..
..4.....	".....	V.....	1.3354	58.8	79.0..
..5.....	10.....	L.....	1.3369	20.8	14.6..
..6.....	".....	V.....	1.3356	57.9	77.0..
..7.....	15.....	L.....	1.3369	20.8	14.6..
..8.....	".....	V.....	1.3358	56.4	72.0..
..9.....	20.....	L.....	1.3369	20.8	14.6..
..10.....	".....	V.....	1.3362	54.6	67.0..
..11.....	25.....	L.....	1.3370	21.4	15.21
..12.....	".....	V.....	1.3358	56.8	73.0..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXVI

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>27</u>
Solution wt., final, gms. = <u>970</u>	Initial Concentrations:
Vapor rate, gms./run = <u>216</u>	Vapor, mol % methanol = <u>60</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>20</u>
Vapor condensate, gms./run = <u>214</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3368	20.0	13.9..
..2.....	".....	V.....	1.3360	55.7	69.0..
..3.....	6.....	L.....	1.3368	20.0	13.9..
..4.....	".....	V.....	1.3363	54.0	66.0..
..5.....	10.....	L.....	1.3368	20.0	13.9..
..6.....	".....	V.....	1.3358	56.8	73.0..
..7.....	15.....	L.....	1.3368	20.0	13.9..
..8.....	".....	V.....	1.3358	56.8	73.0..
..9.....	20.....	L.....	1.3368	20.0	13.9..
..10.....	".....	V.....	1.3360	55.7	69.0..
..11.....	25.....	L.....	1.3368	20.0	13.9..
..12.....	".....	V.....	1.3358	56.8	73.0..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXVII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>29</u>
Solution wt., final, gms. = <u>1005</u>	Initial Concentrations:
Vapor rate, gms./run = <u>207</u>	Vapor, mol % methanol = <u>80</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>30</u>
Vapor condensate, gms./run = <u>180</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3381	30.0	23.9.
..2.....	".....	V.....	1.3330	69.4	12.7.
..3.....	6.....	L.....	1.3381	30.0	23.9.
..4.....	".....	V.....	1.3336	66.9	11.2.
..5.....	10.....	L.....	1.3382	31.2	25.5.
..6.....	".....	V.....	1.3338	66.1	10.9.
..7.....	15.....	L.....	1.3383	32.9	27.7.
..8.....	".....	V.....	1.3333	68.2	12.0.
..9.....	20.....	L.....	1.3383	32.9	27.7.
..10.....	".....	V.....	1.3338	66.1	10.9.
..11.....	25.....	L.....	1.3384	35.0	30.5.
..12.....	".....	V.....	1.3337	66.5	11.1.

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXVIII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>30</u>
Solution wt., final, gms. = <u>975</u>	Initial Concentrations:
Vapor rate, gms./run = <u>207</u>	Vapor, mol % methanol = <u>80</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>30</u>
Vapor condensate, gms./run = <u>212</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3381	30.0	23.9
..2.....	".....	V.....	1.3334	67.7	116
..3.....	6.....	L.....	1.3381	30.0	23.9
..4.....	".....	V.....	1.3336	66.9	112
..5.....	10.....	L.....	1.3382	31.2	25.5
..6.....	".....	V.....	1.3337	66.5	111
..7.....	15.....	L.....	1.3381	30.0	23.9
..8.....	".....	V.....	1.3338	66.1	109
..9.....	20.....	L.....	1.3381	30.0	23.9
..10.....	".....	V.....	1.3337	66.5	111
..11.....	25.....	L.....	1.3382	31.2	25.5
..12.....	".....	V.....	1.3338	66.1	109

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXIX

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 31
 Solution wt., final, gms. = 1020 Initial Concentrations:
 Vapor rate, gms./run = 216 Vapor, mol % methanol = 60
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 30
 Vapor condensate, gms./run = 171 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3381	30.0	23.9
..2.....	".....	V.....	1.3342	64.4	10.1
..3.....	6.....	L.....	1.3381	30.0	23.9
..4.....	".....	V.....	1.3338	66.1	10.9
..5.....	10.....	L.....	1.3381	30.0	23.9
..6.....	".....	V.....	1.3336	66.9	11.2
..7.....	15.....	L.....	1.3381	30.0	23.9
..8.....	".....	V.....	1.3341	64.8	10.3
..9.....	20.....	L.....	1.3380	28.9	22.7
..10.....	".....	V.....	1.3336	66.9	11.2
..11.....	25.....	L.....	1.3381	30.0	23.9
..12.....	".....	V.....	1.3336	66.9	11.2

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. ~~XXX~~

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>321</u>
Solution wt., final, gms. = <u>495</u>	Initial Concentrations:
Vapor rate, gms./run = <u>216</u>	Vapor, mol % methanol = <u>60</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>30</u>
Vapor condensate, gms./run = <u>170</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3381	30.0	23.9..
..2.....	".....	V.....	1.3337	66.5	11.1...
..3.....	6.....	L.....	1.3381	30.0	23.9..
..4.....	".....	V.....	1.3336	66.9	11.2..
..5.....	10.....	L.....	1.3381	30.0	23.9..
..6.....	".....	V.....	1.3335	67.3	11.5...
..7.....	15.....	L.....	1.3381	28.9	22.7..
..8.....	".....	V.....	1.3336	66.9	11.2..
..9.....	20.....	L.....	1.3381	30.0	23.9..
..10.....	".....	V.....	1.3336	66.9	11.2..
..11.....	25.....	L.....	1.3381	30.0	23.9..
..12.....	".....	V.....	1.3336	66.9	11.2..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXXI

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 33
 Solution wt., final, gms. = 1100 Initial Concentrations:
 Vapor rate, gms./run = 237 Vapor, mol % methanol = 20
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 80
 Vapor condensate, gms./run = 100 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3329	69.9	129..
..2.....	".....	V.....	1.3275	90.6	536..
..3.....	6.....	L.....	1.3334	67.8	117..
..4.....	".....	V.....	1.3269	92.6	695..
..5.....	10.....	L.....	1.3334	67.8	117..
..6.....	".....	V.....	1.3274	91.0	562..
..7.....	15.....	L.....	1.3334	67.8	117..
..8.....	".....	V.....	1.3279	89.2	480..
..9.....	20.....	L.....	1.3346	62.7	94..
..10.....	".....	V.....	1.3276	90.3	518..
..11.....	25.....	L.....	1.3353	59.4	82..
..12.....	".....	V.....	1.3277	89.9	494..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXXII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 34
 Solution wt., final, gms. = 1080 Initial Concentrations:
 Vapor rate, gms./run = 237 Vapor, mol % methanol = 20
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 80
 Vapor condensate, gms./run = 105 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3333	68.2	120..
..2.....	".....	V.....	1.3274	91.0	562..
..3.....	6.....	L.....	1.3324	72.0	144..
..4.....	".....	V.....	1.3274	91.0	562..
..5.....	10.....	L.....	1.3329	69.9	129..
..6.....	".....	V.....	1.3279	89.2	480..
..7.....	15.....	L.....	1.3334	67.8	117..
..8.....	".....	V.....	1.3284	87.4	425..
..9.....	20.....	L.....	1.3346	62.7	94..
..10.....	".....	V.....	1.3382	88.1	445..
..11.....	25.....	L.....	1.3352	60.0	83..
..12.....	".....	V.....	1.3287	86.4	395..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXXIII

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = 1000 Run No. 35
 Solution wt., final, gms. = 1030 Initial Concentrations:
 Vapor rate, gms./run = 237 Vapor, mol % methanol = 20
 Vapor rate, cc liq./min. = 10 Liquid, mol % methanol = 80
 Vapor condensate, gms./run = 165 Submergence, in. = 2

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3322	72.8	149..
..2.....	".....	V.....	1.3271	92.0	640..
..3.....	6.....	L.....	1.3328	70.4	132..
..4.....	".....	V.....	1.3271	92.0	640..
..5.....	10.....	L.....	1.3325	71.6	140..
..6.....	".....	V.....	1.3278	89.5	474..
..7.....	15.....	L.....	1.3335	67.3	115..
..8.....	".....	V.....	1.3278	89.5	474..
..9.....	20.....	L.....	1.3341	64.8	103..
..10.....	".....	V.....	1.3279	89.2	458..
..11.....	25.....	L.....	1.3348	61.8	90..
..12.....	".....	V.....	1.3280	88.8	440..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXXIV

Static Mass Transfer Experiments

with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>36</u>
Solution wt., final, gms. = <u>1020</u>	Initial Concentrations:
Vapor rate, gms./run = <u>207</u>	Vapor, mol % methanol = <u>20</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>20</u>
Vapor condensate, gms./run = <u>165</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3368	20.0	13.8...
..2.....	".....	V.....	1.3328	70.3	13.1...
..3.....	6.....	L.....	1.3370	21.4	14.4...
..4.....	".....	V.....	1.3352	59.9	8.3...
..5.....	10.....	L.....	1.3370	21.4	14.4...
..6.....	".....	V.....	1.3356	57.8	7.6...
..7.....	15.....	L.....	1.3371	22.0	14.7...
..8.....	".....	V.....	1.3357	57.4	7.5...
..9.....	20.....	L.....	1.3372	22.7	15.1...
..10.....	".....	V.....	1.3351	60.4	8.5...
..11.....	25.....	L.....	1.3372	22.7	15.1...
..12.....	".....	V.....	1.3352	59.9	8.3...

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXXV

Static Mass Transfer Experiments
with Methanol-Water System

Solution wt., initial, gms. = <u>1000</u>	Run No. <u>37</u>
Solution wt., final, gms. = <u>1015</u>	Initial Concentrations:
Vapor rate, gms./run = <u>207</u>	Vapor, mol % methanol = <u>80</u>
Vapor rate, cc liq./min. = <u>10</u>	Liquid, mol % methanol = <u>20</u>
Vapor condensate, gms./run = <u>165</u>	Submergence, in. = <u>2</u>

Sample	Time	Positions*	n ²⁵	Comp., mol% Methanol	Molality
..1.....	3.....	L.....	1.3368	20.0	13.8..
..2.....	".....	V.....	1.3350	60.9	8.7..
..3.....	6.....	L.....	1.3369	20.7	14.1..
..4.....	".....	V.....	1.3351	60.4	8.5..
..5.....	10.....	L.....	1.3371	22.0	14.7..
..6.....	".....	V.....	1.3357	57.4	7.5..
..7.....	15.....	L.....	1.3371	22.0	14.7..
..8.....	".....	V.....	1.3352	59.9	8.3..
..9.....	20.....	L.....	1.3372	22.7	15.1..
..10.....	".....	V.....	1.3352	59.9	8.3..
..11.....	25.....	L.....	1.3372	22.7	15.1..
..12.....	".....	V.....	1.3352	59.9	8.3..

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

Table No. XXXVI

Mass Transfer Experiments
by Continuous Procedure

System: CH₃OH - H₂O Run No. 38

Entering Vapor composition, mol % CH₃OH = 20

Entering Liquid composition, mol % CH₃OH = 0

Flow rate of liquid as cc/min. = 120

Flow rate of vapor as cc of liquid/min. = 10

Slot opening, in. = $\frac{1}{2}$ Submergence, in. = 2

Sample	Time	Position*	n ²⁵	Mol % <u>CH₃OH</u>
..1.	3.	L.	1.3299	0.3
..2.	"	V.	1.3309	2.6
..3.	6.	L.	1.3299	0.3
..4.	"	V.	1.3309	2.6
..5.	10.	L.	1.3299	0.3
..6.	"	V.	1.3309	2.6
..7.	15.	L.	1.3299	0.3
..8.	"	V.	1.3309	2.6
..9.	20.	L.	1.3299	0.3
..10.	"	V.	1.3308	2.4
..11.	25.	L.	1.3298	0.0
..12.	"	V.	1.3309	2.6

* Position indicates point of sample removal:

L = Pot Liquid

V = Condensed Vapor

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VITA

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