

THE EFFECT OF VARYING PHASE VELOCITIES AND  
THE DIRECTION OF FLOW ON THE MASS TRANSFER  
COEFFICIENT IN A HORIZONTAL EXTRACTOR TUBE

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## I. INTRODUCTION

The application of extraction as a unit operation in chemical engineering is increasing in importance in many industries. By the use of extraction operation, it has been possible to recover many products and raw materials at a cost which has made the operation profitable. Extraction is replacing or supplementing the use of other operations such as drying, evaporation, and distillation.

Design engineers must know the overall mass transfer coefficients for a system before they can decide what size and type of equipment is necessary for a particular job. At the present time, the amount of tabulated data for various systems is very limited and incomplete, making it necessary for the design engineers to perform experiments for the systems which they plan to use.

The rate of extraction depends upon the mass transfer coefficient, which is also dependent upon the different materials used in the system, the concentration of the feed, the mass velocity, the temperature, the interfacial area, and the direction of flow. When two immiscible liquids are moving in opposite directions, the faster moving liquid carries along with it the interface and an adjacent portion of the slower moving liquid. The so-called countercurrent flow in

liquid-liquid extraction is, therefore, actually cocurrent or parallel flow at the interface. Previous research investigators at Virginia Polytechnic Institute have studied mass transfer between two liquid phases for the systems n-butanol-water and 1,1,2-trichloroethane-acetone-water. Further studies on these two systems would permit comparison and evaluation of the effect of direction of flow on the magnitude and variation of the mass transfer coefficients.

The purpose of this investigation was to determine the effect of variable phase velocities and the direction of flow on the overall mass transfer coefficient in a horizontal, unpacked extraction tube for the systems n-butanol-water and 1,1,2-trichloroethane-acetone-water.

## II. LITERATURE REVIEW

This section is a review of liquid-liquid extraction as it is known today. This review will cover definitions, uses, solvent systems, theory of mass transfer, physical characteristics of extraction systems, and methods of calculation of mass transfer.

### Definitions

For a clear presentation of the literature the definitions given in this section are those of Elgin<sup>(20,21)</sup>. The more commonly used symbols for liquid-liquid extraction are listed in Table I.

Solvent Extraction. Elgin defines solvent extraction as "an operation in which the separation of a mixture of different substances is accomplished by treatment with a selective solvent." It is necessary that one of the components of the mixture be partially soluble in the other components of the mixture so that at least two different phases are formed.

Solvent. The solvent is the liquid that is introduced to the feed mixture to remove one or more of the components of the mixture. The selection of the solvent depends upon the solubility of the different components of the feed in the solvent.

Solute. The solute is that component of the feed mixture that is most soluble in the solvent. It must be noted that the solvent is selected so that the desired portion of the feed will be dissolved in the solvent.

Feed. The feed is a mixture of two or more components, one of which is the solute or the substance to be collected by the solvent, the other being the carrier solvent.

Extract. The extract is that phase after the extraction operation which contains the solute, solvent, and possibly a small amount of the carrier solvent.

Raffinate. The raffinate is that phase after the extraction operation which contains the carrier solvent, and small amounts of the solute, and solvent, after a portion of the solvent has been removed.

Selectivity. The solvent is said to be selective for that component of the feed which is found in greater ratio to the other in the extract than in either the raffinate or the original feed.

Countercurrent Flow. Countercurrent flow is that mode of operation in which the feed and the solvent enter from opposite ends of the extraction equipment.

Cocurrent Flow. Cocurrent flow is that mode of operation of a liquid-liquid extractor in which the feed and the solvent enter from the same end of the extraction equipment.

### Industrial Uses of Solvent Extraction

Since 1939, liquid-liquid extraction has changed from a laboratory method of separation of mixtures into an industrial tool. The production of organic chemicals has created the need of a method of separation of azeotropic mixtures, materials that are heat sensitive, recovery of materials that are present only in small amounts, and in the separation of materials that have approximately the same boiling points.

Lubricating Oils. The petroleum industry was one of the first to recognize the value of liquid-liquid extraction as a refining operation. This method of refining of lubricating oils enabled the production of a more uniform grade of oils; also, it enabled the refining companies to recover many valuable chemicals from the oil stock that previously had been left in the oils. Some of the extraction processes that have been introduced into the petroleum industry are the Edleanu process<sup>(29)</sup> for the recovery of aromatic hydrocarbons from the kerosene cut in the refinery process, and

the propane process<sup>(30)</sup> for the recovery of paraffinic hydrocarbons from the oil stock, the propane being recovered easily by distillation due to its low boiling point.

Antibiotics. The pharmaceutical industry depends upon extraction techniques for the recovery and purification of antibiotics<sup>(53)</sup>. Penicillin is extracted from an acidified, filtered fermentation liquor, in which its original concentration is less than 0.02 pound per cubic foot, by amyl acetate. Deterioration losses are cut by rapid processing. Podbielniak centrifugal extractors have been widely applied for this operation.

Caustic Purification. Somewhat unusual among extraction processes is one that involves no organic chemicals. This process, by extraction with ammonia, removes traces of salt from caustic soda. At least four extraction units have been built, each having a capacity of over 100 tons per day. These units operate by having 50 per cent caustic flow countercurrent to ammonia of 70 to 90 per cent concentration; the system is under pressure to keep the ammonia liquefied. The salt concentration is reduced to 0.08 per cent as the ratio of ammonia is increased. It also extracts water to further raise the concentration of sodium hydroxide in the

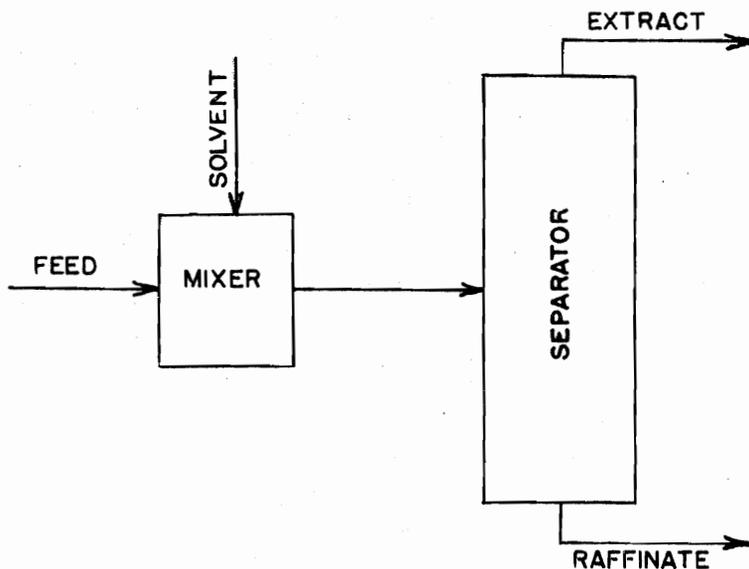
caustic. About 5 per cent of the sodium hydroxide is also extracted. The ammonia is recovered by distillation and scrubbing<sup>(53)</sup>.

### Solvent Extraction Operations

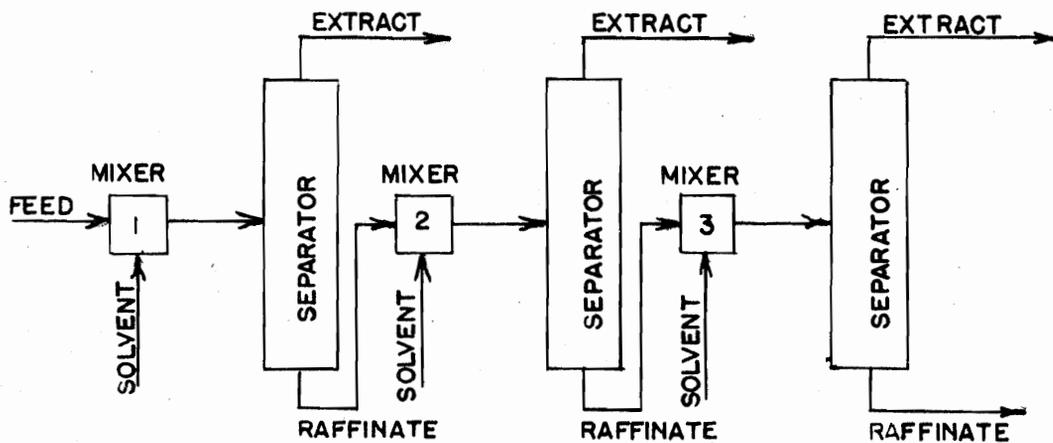
The different solvent extraction operations that are being used in industry will be explained in this section. It must be noted that these descriptions are for the basic operations since many industries use combinations of the operations that will be described.

Single Pass Extraction. The simplest method and that most common to laboratory scale is to bring the entire quantities of solvent and feed to be extracted together in one contact and then recover the product and solvent without further extraction, as shown in Figure 1. This method is the least effective and is rarely feasible on an industrial scale. As practiced, equilibrium is usually closely approached; hence the amount of solute extracted is fixed solely by the equilibrium relations and the quantity of solvent used. This method can be regarded as analogous to simple continuous or to flash distillation<sup>(24)</sup>.





SINGLE CONTACT EXTRACTION



SIMPLE MULTISTAGE CONTACT EXTRACTION

FIGURE 1. SINGLE CONTACT AND SIMPLE MULTISTAGE CONTACT EXTRACTION

ELGIN, J.C.: SOLVENT EXTRACTION, "CHEMICAL ENGINEERS' HANDBOOK," (J.H. PERRY, EDITOR), P.717, MCGRAW-HILL BOOK CO., INC., NEW YORK, NEW YORK, 1950, 3D ED.

Simple Multiple Contact. Simple multiple contact is sometimes called cocurrent multiple contact, as illustrated in Figure 1. Simple multiple contact differs from single contact because the solvent is divided into several small portions and introduced into the system at different points. The allotted portions of the solvent are removed from the system after each stage before the next portion of the solvent is introduced; the solvent being removed contains the solute that is being extracted from the feed. The percentage recovery of the extracted component is increased as the number of stages and the quantity of solvent are increased. If a sufficient number of stages and a sufficient amount of solvent are used, the raffinate can be stripped of the extracted component to a high degree, but with any specified amount of solvent a finite removal of the extract is approached as the number of stages approaches infinity. This method may be operated intermittently with a single mixer and settler unit or continuously with a series of such units<sup>(24)</sup>.

Countercurrent Multiple Contact. The countercurrent multiple contact method of operation employs the passage of fresh solvent and feed in opposite directions, each entering the system from different and opposite ends<sup>(24)</sup>. The extract

and raffinate layers pass continuously and countercurrently from stage to stage through the system. The finished extract and raffinate layers are withdrawn continuously from opposite ends of the system. Any number of stages may be employed, the usual number being from three to six. This method makes use of the fact that the weaker extract layer (solvent) which has been brought to equilibrium with a more dilute raffinate layer is still able to remove extract from a more concentrated raffinate layer or feed. The system may be composed of a series of mixers, each with its separate settler, or some form of continuous plate column may be used, as illustrated in Figure 2.

Countercurrent Extraction with Reflux. Elgin<sup>(26)</sup> states that unless the solvent in the feed is immiscible with the extracting solvent, neither true nor multistage continuous countercurrent extraction can completely separate the components of the feed in the extract and raffinate products. A theoretical limiting value of the extract product composition is attained which with infinite stages or tower height tends to approach equilibrium with the incoming feed.

Reflux may be supplied to either one or both the extract and raffinate ends of the extraction system in order to increase the degree of separation between the components of the

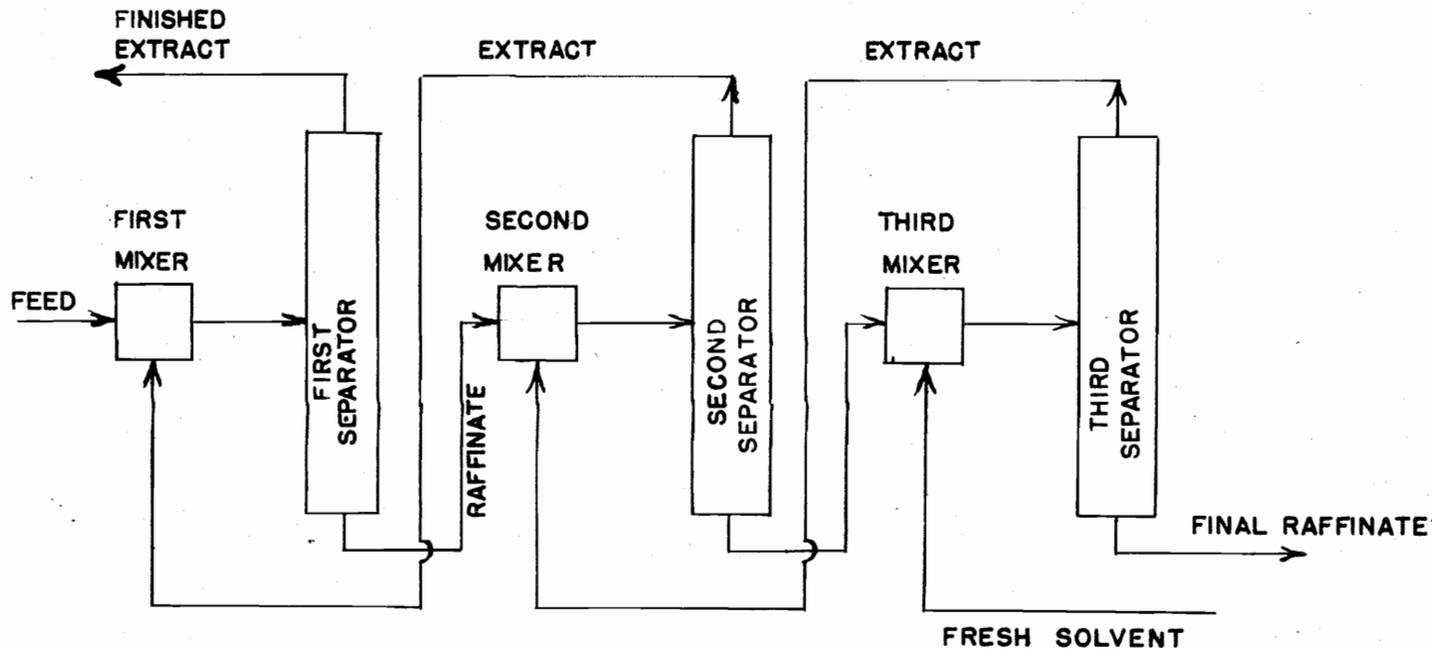


FIGURE 2. COUNTERCURRENT MULTISTAGE CONTACT EXTRACTION

ELGIN, J.C.: SOLVENT EXTRACTION, "CHEMICAL ENGINEERS' HANDBOOK," (J.H. PERRY, EDITOR), P. 717, MCGRAW-HILL BOOK CO., INC., NEW YORK, NEW YORK, 1950, 3D ED.

feed. Extract reflux is supplied by returning a portion of the extract layer from which the solvent has been wholly or partially removed. In general, with weaker feeds, extract reflux raises the concentration of the extract component in the extract layer above that corresponding to equilibrium with the feed while raffinate reflux improves its degree of stripping from the raffinate<sup>(26)</sup>.

The principles of reflux in extraction are analogous to that in distillation, however, it is subject to certain limitations not met in distillation. A flow diagram of counter-current extraction with reflux on both ends of the column is shown in Figure 4.

Simple Parallel Flow. Simple parallel flow in liquid-liquid extraction is mentioned only briefly by Treybal<sup>(46)</sup> and is defined as being a batch-wise extraction process, the raffinate from the preceding batch being used as the feed in the next batch operation. Parallel flow in extraction equipment such as the wetted-wall column and the spray column is possible. However, the use of countercurrent flow in these types of equipment is more practical when the design and operation of the equipment are considered.

Skrzes<sup>(41)</sup> found that the use of countercurrent flow in a horizontal tube extractor was in effect cocurrent or parallel

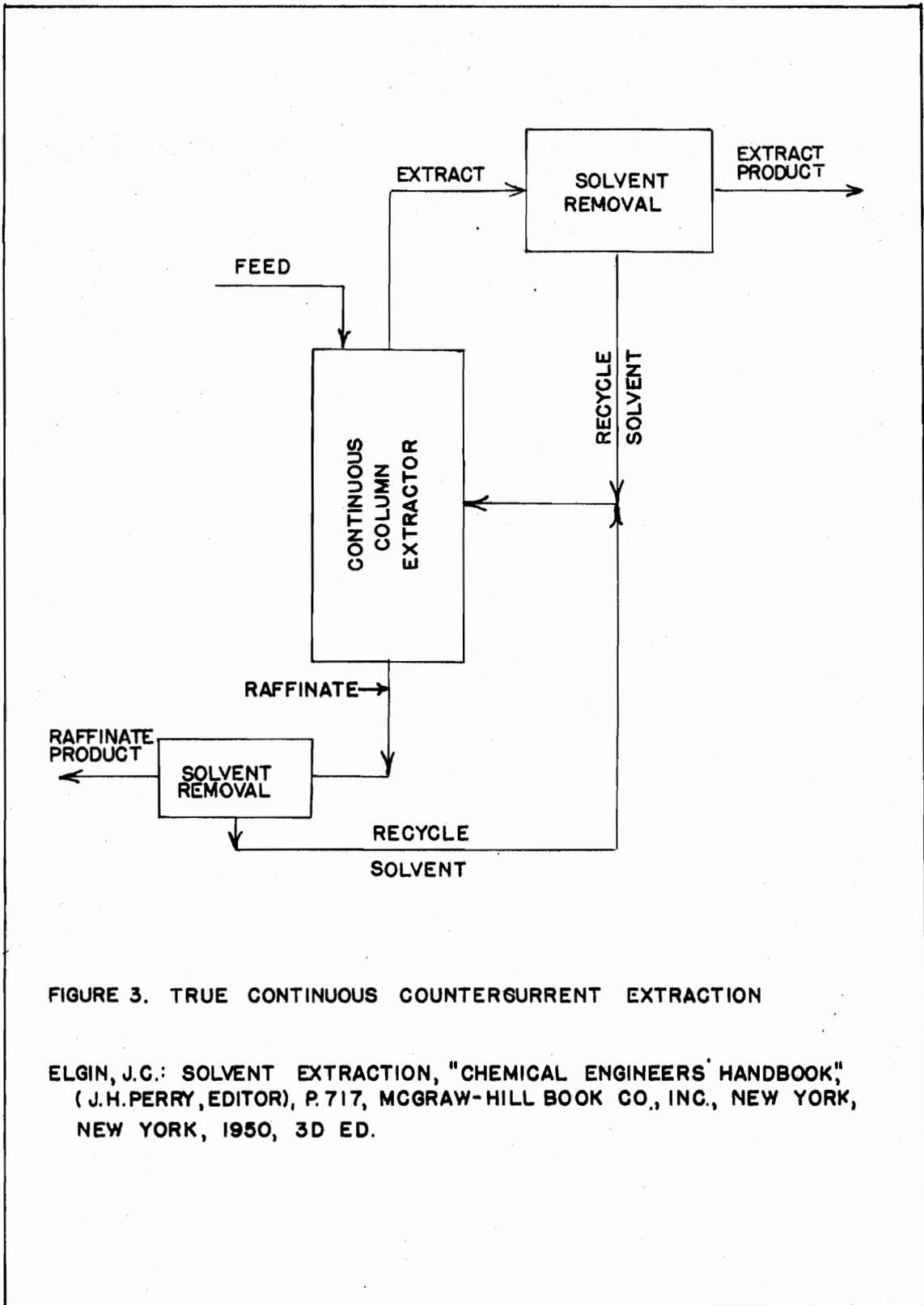


FIGURE 3. TRUE CONTINUOUS COUNTERCURRENT EXTRACTION

ELGIN, J.C.: SOLVENT EXTRACTION, "CHEMICAL ENGINEERS' HANDBOOK," (J.H.PERRY, EDITOR), P.717, MCGRAW-HILL BOOK CO., INC., NEW YORK, NEW YORK, 1950, 3D ED.

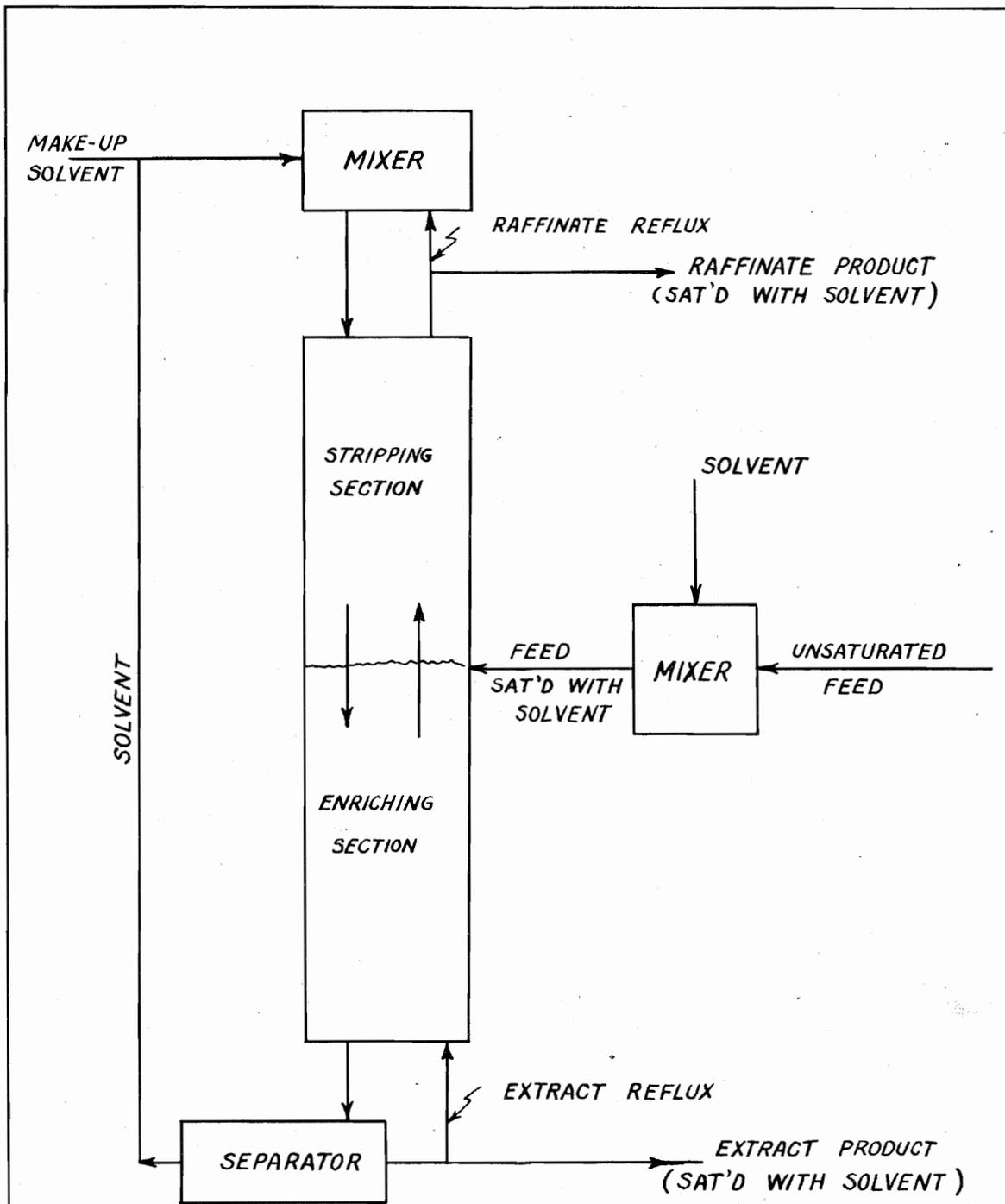


FIGURE 4, LIQUID-LIQUID EXTRACTOR WITH REFLUX

BULL, F.W. AND G.J. COLI: GRAPHICAL METHODS APPLIED TO EXTRACTION PROBLEMS, VA. POLY. INST., BLACKSBURG, VIRGINIA, ENG. EXPT. STA., BULLETIN NO. 72, 13 (1949)

flow at the interface, when the mass velocity of one phase was greater than the other. This effect was attributed to the friction between the two phases. The phase having the higher mass velocity would "drag along" the interface usually a portion of the other phase in the direction of the faster moving phase.

### Solvent Systems

The solvent systems that are used today are generally classed under one of four types of systems. This classification depends upon the properties of the system while in use in an extraction process. The four types of systems are the miscible, immiscible, chemical reacting, and precipitative<sup>(16,18)</sup>. The choice of the solvent in these systems depends upon the degree of extraction desired, the equipment used, and the equilibrium distribution for the systems.

Miscible System. The miscible system is defined as one in which the solute is soluble in both the carrier and the solvent, the concentration of the solute depending upon the selectivity of the solvents for the solute. In this type of system the solvent is immiscible with the carrier solvent.



Immiscible System. The immiscible system employs the use of two solvents, the first being used to extract the solute from the carrier solvent, the second being used to remove the first solvent that has been dissolved in the carrier solvent.

Chemical Reacting System. The chemical reacting system, as the name implies, is one in which a chemical reaction takes place. The reaction being between the solvent and the solute, the carrier solvent does not take part in the reaction.

Precipitative System. Under certain conditions of temperature and concentration, certain organic solvents will crystallize out of the system. If the solvent that contains the solute acts in this manner, it provides a method of separation for mixtures and solution.

### Industrial Extraction Equipment

The industrial extraction equipment that is most widely used will be discussed in this section. The several types of equipment that are under consideration are: spray towers, packed columns, wetted-wall columns, bubble-cap towers, centrifugal, and pulse columns. Recently, Morello and Poffenberger<sup>(28)</sup> have compiled and published a very complete bibliography on all types of extraction equipment.

Spray Towers. A spray column is a vertical cylinder through which one liquid flows as the continuous phase, with the second liquid dispersed in the first<sup>(47)</sup>. The direction of flow in the spray tower depends upon the densities of the liquid in contact. Spray columns are the simplest type of extraction equipment in construction and operation. They are noted for their low cost, high capacity, and ease of maintenance.

The efficiency of spray towers has been a subject of study. It has been found that 40 to 50 per cent of the extraction takes place at the point of dispersion of the discontinuous phase. Low efficiencies may result from coalescence of the droplets or recirculation in the tower, which reduces the counter-current action in the tower.

Packed Columns. Packing is frequently used to promote the contacting of the liquids flowing countercurrently through a column. Such columns are usually packed with standard materials, such as raschig rings, or berl saddles<sup>(48)</sup>. The packed column differs from the spray column only in the addition of the packing.

The packing in this type of column is often separated into several sections to allow the liquids to redistribute and to make the column easier to pack and unpack. The packing gives a greater area of contact between the phases, causing greater transfer between the phases. The packed column is objectionable in that solids are deposited on the packing and channeling occurs during operation of the column.

Wetted-wall Column. The wetted-wall column has not as yet been used extensively in industrial work<sup>(51)</sup>. This type of column is essentially the same as the spray and packed columns, the difference being that one phase is introduced so that it flows down the wall of the column while the other phase flows upward through the remaining portion of the column. This type of column has been used to determine the individual mass transfer coefficients in laboratory experiments.

Bubble-cap Towers. The bubble-cap tower is similar in construction to the fractional distillation tower. The difference in operation is that a lighter liquid phase is moving upward through the tower in the same path that a vapor would use in distillation<sup>(49)</sup>. The heavier phase is introduced at the top of the column and moves downward, countercurrent to the lighter phase. The bubble-cap towers have been found to be inefficient in use unless they are modified in design so that better dispersion is obtained in the discontinuous phase.

Pulsating Columns. The main objective of any of the previously mentioned extraction units is to obtain intimate contact between the phases in contact. Several designs have been proposed by Scheible<sup>(50)</sup>. One of the more recent designs<sup>(53)</sup> has been the forcing and drawing of phases through perforated plates by either having a system of oscillating plates or having a bellows on the column, which first pushes and then pulls the phases through the column.

The amount of solute transferred in the pulse type column has been found to be equivalent to ordinary towers three to five times as tall. It has also been found that the effectiveness of the pulse tower depends upon the solvents used. The forcing of the phases through the perforated

plates has a tendency to cause an emulsion which limits the effectiveness of the column.

Centrifugal Extractors. The two principal types of centrifugal extractors are the Podbielniak<sup>(52,53)</sup> and the Luwesta extractor<sup>(52,53)</sup>. Both of the extractors operate on the principle of the heavier liquid being forced to the outside of a container by centrifugal force while the lighter liquid is moved nearer the center of the container.

The Luwesta is essentially a cream separator while the Podbielniak extractor is packed with a long spiral, perforated ribbon. The heavier feed enters near the center of the container and the lighter fluids enter on the outer edge of the container. The centrifugal force causes countercurrent flow between the two liquids and the ribbon creates a shearing force on the interface thereby giving smaller droplets and a greater interfacial area.

The machines operate at a speed from 2000 to 5000 revolutions per minute, and have capacities from 2000 to 5000 gallons per hour. They have been adopted widely throughout the pharmaceutical industry, largely because of the low holdup and their versatility.

TABLE II

Performance of Commercial Extraction Equipment

Type	Liquid Capacity Both Streams	H.E.T.S.	Plate Efficiency
	cu ft/sq ft-hr	ft	%
Mixer- Settler	--	Approx. 1	75-100
Spray Column	50-250	10-20	--
Packed Column	20-150	5-20	--
Perforated Plate	10-200	1-20	30
Koch Column	10-200	2-7	20-75
Baffle Column	60-105	4-6	5-10
Bubble Cap Column	--	8-20	5-10
York-Scheibel Column	50-100	1-2	80-100
Podbielniak	20,000 gal/hr	3-20	--
Luwesta	3,800 gal/hr	3-4	--

Von Berg, R. L., and H. F. Wiegandt: Liquid-Liquid Extraction, Chem. Engr., 59, 6--., 189-200 (1952).

### Mechanisms of Mass Transfer

The liquid-liquid extraction process depends upon the ability of an immiscible solvent to be more selective for one component of the feed than the carrier solvent. The laws of distribution and diffusion equilibria are the prominent factors governing the transfer of components from the solution to the solvent.

Since this investigation deals with the overall mass transfer coefficient and its component parts, a discussion of the theory involving the design of the equipment will be omitted. The theory of design is available in the literature<sup>(20,42)</sup>.

Two-Film Theory of Mass Transfer. When dealing with problems involving mass transfer, it is usually assumed that the two-film theory applies<sup>(8,21)</sup>. This theory is based on the assumption that there is a stationary or slow moving film on the boundary surfaces of two moving fluids in contact. The application of this theory to liquid-liquid extraction requires the extension of the assumption to include two films instead of one film moving past a solid surface.

The two moving fluids in contact in a liquid-liquid extraction system are separated by an interface, on each side of which is the stationary or slow moving film. Monrad<sup>(28)</sup> points out that at low velocities the entire body of the fluid may be assumed to be the entire film. It has also been assumed that at the higher flow rates, the thickness of the film decreases until mixing occurs. For the extraction process to take place, the material must be transferred through the body of the fluid into the interface. The concept of "effective film thickness" is used to eliminate the effects of velocity and assumes the true diffusion through a stationary layer.

In extraction operations the effective film thickness must be considered for both sides of the interface. While considering the effective film thickness on each side of the interface, the concentration gradient from the body of the fluid to the interface must also be considered.

The resistances offered by each film near the interface are seldom equal. In the extraction process the concentration gradient across each film on the interface creates different driving forces for the transfer of material to the interface. The concentration difference and the film resistance causes either one or both films to



be considered as the controlling film in the diffusional process. For the production of a rapid transfer, it is necessary to reduce the effective film thickness, thereby reducing the resistance of the controlling film. The effective film thickness may be decreased by changing such operating variables as velocity, concentration difference, temperature, method of contacting, and solvent used.

Overall Mass Transfer Coefficient. The overall mass transfer coefficient is necessary for the intelligent design of any extraction system<sup>(21)</sup>. The overall mass transfer coefficient may be considered as the reciprocal of the resistances to mass transfer from the body of one phase to that of the other. If two films exist at the interface and the resistance of both are appreciable, the overall resistance is actually composed of two resistances in series, and is equal to the sum of the resistances of the individual films. The overall mass transfer coefficient may be expressed in terms of the individual film transfer coefficients as follows:

$$1/K_w = 1/k_w + H/k_g \quad (1)$$

where:

$K_w$  = overall mass transfer coefficient based on the water phase

$k_w$  = individual coefficient of water phase

$k_g$  = individual coefficient of the solvent phase

$H$  = distribution coefficient,  $\frac{\text{conc. of solute in feed}}{\text{conc. of solute in solvent}}$ .

Colburn<sup>(10)</sup>, Hunter and Nash<sup>(27)</sup>, Sherwood<sup>(33)</sup>, Appel and Elgin<sup>(18)</sup>, and Elgin and Browning<sup>(19)</sup>, have performed investigations on the overall mass transfer coefficient using countercurrent flow in spray and packed columns. Sherwood<sup>(37)</sup> used an equation for spray columns which is as follows:

$$K = \frac{L (C_2 - C_1)}{A (\Delta C)_{lm}} \quad (2)$$

where:

- K = overall mass transfer coefficient, lb-mols per hr-sq ft-unit  $\Delta C_{lm}$
- L = solvent rate, cu ft/hr
- $C_2$  = concentration of solute in leaving solvent, lb-mols/cu ft
- $C_1$  = concentration of solute in entering solvent, lb-mols/cu ft
- A = area of contact at any time, sq ft
- $\Delta C_{lm}$  = log mean driving force, based on  $C_1$  and  $C_2$  and the equilibrium concentration in solvent corresponding to the observed concentration in the aqueous phase at the bottom and top of the column.

Sherwood and his investigators carried out their extraction procedure with single drops of the dispersed phase. Their calculations were based on the assumption that the drops were spherical, so that it was possible to calculate the surface area of each drop. The area of contact was determined by finding the time of formation, the time required for a drop to rise to the top of the column, the number of drops introduced into the column per unit of time, and the diameter of each drop.

Elgin, et al<sup>(26)</sup>, developed another equation which is analogous to that used by Sherwood<sup>(33)</sup>. It is as follows:

$$K_w a = \frac{N/e}{V (\Delta C)_{lm}} \quad (3)$$

where:

- $K_w$  = average overall transfer coefficient based on the water phase, lb-mols per hr-sq ft-unit  $\Delta C_{lm}$
- $a$  = interfacial area per unit volume, sq ft/cu ft
- $V$  = effective volume of the column, cu ft
- $N$  = number of mols of solute transferred, lb-mols
- $e$  = elapsed time, hr
- $C_{lm}$  = log mean driving force, based on  $C_1$  and  $C_2$ , and the equilibrium concentration in the solvent corresponding to the observed concentration in the aqueous phase at the bottom and top of the column.

Sherwood and his investigators carried out their extraction procedure with single drops of the dispersed phase. Their calculations were based on the assumption that the drops were spherical, so that it was possible to calculate the

surface area of each drop. The area of contact was determined by finding the time of formation.

$$\frac{dN_e}{A d\theta} = k_w (C_w - C_w^*) = k_e (C_e^* - C_e) \quad (4)$$

or in the reverse direction,

$$\frac{dN_e}{A d\theta} = k_e (C_e - C_e^*) = k_w (C_w^* - C_w) \quad (5)$$

where:

- $N_e$  = mols of solute transferred, based on phase "e", lb-mols
- $A$  = total interfacial area of contact, sq ft
- $\theta$  = elapsed time, hr
- $k_w$  = individual extraction coefficient based on water phase, lb-mols per hr-sq ft-unit  $\Delta C$
- $k_e$  = individual extraction coefficient based on solvent phase, lb-mols per hr-sq ft-unit  $\Delta C$
- $C_e$  = concentration of solute in solvent phase, lb-mols per cu ft
- $C_w$  = concentration of solute in water phase, lb-mols per cu ft
- $C_w^*$  = concentration of solute in water phase, which would be in equilibrium with the concentration of solute in solvent phase, lb-mols per cu ft
  
- $C_w^* = C_e/H$

where:

- $H$  = distribution coefficient at equilibrium,  $C_e/C_w^*$
- $C_e^* = HC_w$

where:

- $C_e^*$  = concentration of solute in solvent phase which would be in equilibrium with the concentration of solute in the water phase, lb-mols per cu ft.

The significance of the terms and constants are entirely analogous to the case of a gas absorption process where the simple distribution law holds,

$$\frac{dN_e}{Ad\theta} = K_w (C_w - C_w^*) = K_e (C_e^* - C_e) \quad (6)$$

where:

- $K_w$  = overall, mass transfer coefficient based on the water phase, lb-mols per hr-sq ft-unit " $\Delta C$ "
- $K_e$  = overall, mass transfer coefficient based on the solvent phase, lb-mols per hr-sq ft-unit " $\Delta C$ ".

The magnitude of the distribution coefficient, " $H$ ", and the relative conditions of turbulence in the laminar films may be such that the rate of diffusion across one or the other may be sufficiently slow to control the rate of the entire process. In this case, the overall mass transfer coefficient is equal to the individual extraction coefficients.

There has been little experimental verification of these relations for mass transfer between two liquid phases but there is no reason to believe that these relations are not valid.

According to Elgin and Browning<sup>(19)</sup>, equation (6) may be integrated algebraically under conditions such that: (a) the simple distribution law holds, that is, " $H$ " is a constant;

(b) the mols of each solvent are constant through the extractor (complete immiscibility); and (c) the overall extraction coefficients, "K", is constant through the apparatus. If concentrations are expressed as mols of solute per mol of solute free solvent, in terms of phase "W", the result is:

$$\frac{N}{\theta} = W (X_1 - X_2) = K_w A \Delta X_{1m} \quad (7)$$

where:

- N = mols of solute transferred, lb-mol
- $\theta$  = elapsed time, hr
- $K_w$  = overall mass transfer coefficient, based on the water phase, lb-mols per hr-sq ft unit  $\Delta C$
- W = water flow rate, lb-mols per hr
- A = total interfacial area of contact, sq ft
- $X_1$  = mols of solute per mol of entering solute free solvent, lb-mols per lb-mol
- $X_2$  = mols of solute per mol of exit solute free solvent, lb-mols per lb-mol
- $\Delta X_{1m}$  = log mean driving force based on mols of solute per mol of solute free solvent.



Equation (7) is theoretically valid only where the mol ratio is directly proportional to the concentration which is the case where the mols of solute are small relative to those of the solvent. For practical purposes, except where the system departs widely from the simple distribution law, one can make use of the simple and convenient relation:

$$\frac{N}{\theta} = K_w A \Delta C_{lm} \quad (8)$$

where:

$$\Delta C_{lm} = \frac{(C_{w1} - C_{w1}^*) - (C_{w2} - C_{w2}^*)}{\ln \frac{(C_{w1} - C_{w1}^*)}{(C_{w2} - C_{w2}^*)}} \quad (9)$$

Equation (8) will give a close approximation of actual behavior unless the volume changes, the amount extracted and the concentrations involved are relatively large. All concentrations are expressed in pound-mols per cubic foot. The subscripts 1 and 2 refer to the different ends of the extraction equipment.

The Effect of Phase Velocities on the  
Overall Mass Transfer Coefficient

It has become evident through experimental work that the phase velocity affects the overall mass transfer coefficient in different types of extraction equipment. The results will be reviewed in this section.

Spray Columns. Elgin and Browning<sup>(19)</sup>, in some earlier investigations, determined the effect of phase velocities on the overall mass transfer coefficient for the system isopropyl ether, acetic acid, and water in a spray tower. The results obtained indicated that the overall mass transfer coefficient was dependent upon the phase velocities of both phases regardless of which was the continuous or discontinuous phase.

The overall transfer coefficient increased with the phase velocity of the ether phase while the water phase containing the acetic acid was held constant. The overall transfer coefficient also increased while the ether was held constant and the feed phase increased in velocity. The same effect was noted when the acetic acid was dissolved in the ether phase, used as the feed. Increases in the flow rate of either phase increased the overall mass transfer coefficient for the system.

Appel and Elgin<sup>(18)</sup> continued with an investigation of the effects of phase velocities using the system toluene, benzoic acid, and water, in a spray column. The results obtained from this investigation agreed with the previous results in that the overall mass transfer coefficient increased as either phase increased in velocity independent of which phase was the feed.

Packed Columns. Appel and Elgin<sup>(18)</sup> continued the investigation using a packed column. The system was the same as the one used in the spray column, toluene, benzoic acid, and water. In this investigation, the water was made the continuous phase and the toluene was the discontinuous phase. Varying the flow rate of the water did not appreciably affect the overall mass transfer coefficient while increases in the toluene phase flow rate increased the overall mass transfer coefficient. The use of a packed column with the system indicated that the phase velocity of the discontinuous phase, which was toluene, was the controlling factor for the overall mass transfer coefficient.

Sherwood, Evans, and Lengour<sup>(34)</sup> investigated the overall mass transfer coefficient with varying phase velocities for the system isobutyl ketone, acetic acid, and water. The results were based on the solvent phase which was isobutyl

ketone, and from the solubility relationship, they expected the overall mass transfer coefficient to vary with either phase velocity. The results obtained agreed with the results obtained by Appel and Elgin<sup>(18)</sup>, in that an increase in either phase velocity increased the overall mass transfer coefficient.

Comings and Briggs<sup>(10)</sup>, and Row, Koffalt, and Withrow<sup>(32)</sup>, did supporting work that agreed with the work done by the previous investigators. Logarithmic plots of "Ka" versus phase velocity showed that the slopes of the resulting straight lines in the different investigations to be practically equal.

Horizontal Tube Extractor. Bergelin, Lockhart, and Brown<sup>(1)</sup> used a horizontal extraction tube because the phase rates and the interfacial area could be determined accurately. Several investigators<sup>(10,21,32)</sup> had emphasized the fact that variations in the area had apparently hidden the variations in the overall mass transfer coefficient in spray and packed columns. The horizontal tube extractor with its known interfacial area, permitted a more basic study of the overall mass transfer coefficient, while varying the solvent and feed phase flow rate.

The results of Berglin, Lockhart, and Brown are significant in that the overall mass transfer coefficient was at a minimum when the phase velocities were equal. It was found by plotting the overall mass transfer coefficient versus the velocity that the overall mass transfer coefficient increased with an increase in velocity of one phase, and decreased to pass through a minimum while the velocity of the other phase increased<sup>(1)</sup>.

These results showing a decrease may be explained by the fact that the interface is moving when one phase of the system has a higher velocity than the other. The direction of movement of the interface is in the direction of the faster phase, thereby making the "plane of zero velocity" be in the body of the slower phase.

Skrzec<sup>(37)</sup> noted that small particles of dirt were carried in the direction of the faster phase, even in the body of the slower phase. It was also observed that when the phases were approximately equal in velocity that the particles at the interface did not move, or if so, very slowly. The results obtained by Skrzec indicated that the overall mass transfer coefficient reaches a minimum for equal phase rate in the horizontal tube countercurrent extractor.

### Studies with a Horizontal Tube Extractor

The investigations that have been performed in the Chemical Engineering Department at Virginia Polytechnic Institute, using countercurrent flow in a horizontal tube extractor are presented in this section.

#### Acetic Acid-Water-1,1,2-Trichloroethane. Oliver<sup>(31)</sup>

studied the effect of the mass velocity on the overall mass transfer coefficient, using the system acetic acid, water, and 1,1,2-trichloroethane. The results obtained from this investigation indicated that the overall mass transfer coefficient increased from 0.129 to 0.413 pound mol per hour per square foot per unit concentration difference, as the mass velocity of the water phase increased from 7900 to 17,900 pounds per hour per square foot. It was found, also, that the lowest value for the overall mass transfer coefficient was at equal phase velocities.

#### Acetone-Water-1,1,2-Trichloroethane. Bowman's<sup>(2)</sup> studies with the horizontal tube extractor consisted of varying the mass velocities of the feed and of the solvent to determine the effect on the overall mass transfer coefficient for the system water, acetone, and 1,1,2-trichloroethane. The results from Bowman's studies indicated that the overall mass transfer coefficient, based on the 1,1,2-trichloroethane phase, increased

almost linearly from 0.0290 to 0.0926 pound mol per hour per square foot per unit concentration difference, as the water phase mass velocity increased from 4140 to 20,050 pounds per hour per square foot. At the same time, the overall mass transfer coefficient based on the water phase, increased in the same manner through a range of values of 0.0450 to 0.171 pound mol per hour per square foot per unit concentration difference.

For five constant water phase mass velocities, the overall mass transfer coefficient based on the trichloroethane phase, decreased almost linearly through a range of values from 0.0926 to 0.0290 pound mols per hour per square foot per unit concentration difference as the trichloroethane phase mass velocity increased from 4340 to 21,970 pounds per hour per square foot. At the same time the overall mass transfer coefficient based on the water phase, decreased in the same manner through a range of values from 0.1371 to 0.0430 pound per hour per square foot per unit concentration difference.

n-Butanol-Water. Barger<sup>(11)</sup> investigated the effect of varying the phase velocities in countercurrent flow on the overall mass transfer coefficient for the system n-butanol and water. Attempts were also made to correlate the transfer coefficient with the physical properties of n-butanol and water.

The results obtained in this investigation agreed with the results of Bowman and Oliver in that the overall mass transfer coefficient increased with the increasing water flow rates. Variations of the butanol phase flow rate had no noticeable effect on the overall mass transfer coefficient for the transfer of butanol through the water film. The transfer coefficient for water through the butanol film increased linearly with increased flow rates of the butanol phase, from 0.43 to 3.5 moles per hour per square foot per unit concentration difference, for butanol phase rate of 2100 to 18,000 pounds per hour per square foot.

Correlations were used to determine the overall mass transfer coefficient for the n-butanol and water system based on the physical properties such as: water phase flow rate, n-butanol phase flow rate, and ratio of the phase rates. The limiting values for the physical properties were also determined<sup>(15)</sup>.

Correlation of Variables. Skrzec's<sup>(36)</sup> studies with the horizontal tube extractor consisted of an investigation of individual film transfer coefficients and the correlation of the results obtained with the physical properties of the systems. The systems studied by Skrzec were cyclohexanol and water, methyl ethyl ketone and water, n-butanol and water, furfural and water,



nitromethane and water, n-butanol-acetic acid and water, cyclohexanol-acetic acid and water, and methyl ethyl ketone-acetic acid and water. A schematic drawing of the equipment used in this investigation is presented in Figure 5, page 44.

The results obtained from the study of the individual transfer coefficients were similar to the results obtained by the previously mentioned investigators. It was found that the transfer coefficient for water through the solvent film increases gradually until a ratio of 1 to 1 is reached between the flow rates of the phases; at higher ratios of solvent to water rates the coefficient increases parabolically. With constant flow rates of solvent and increasing flow rates of the water phase, the transfer coefficient of water through the solvent film decreases hyperbolically. At the same time, the transfer coefficient for the transfer of solvent through the water film increases linearly with increasing water flow rate, reaching an upper value at a mass velocity of approximately 10,000 pounds per hour per square foot of water phase. While holding the water rate constant, it was found that increasing solvent rates had little effect on the transfer coefficient for transfer of solvent through the water film.

Skrzec, in his investigation, developed two equations by dimensional analysis for the determination of individual

film coefficients for two component systems based on the physical properties and the phase rates of the two phases. The equation for determining the individual transfer coefficient for water through the solvent film was correlated as follows:

$$\frac{k_s d}{D_w} = 0.00665 \left( \frac{d}{u_s D_w} \right)^{0.275} \left( \frac{u_s}{u_w} \right)^{0.44} \left( \frac{\sigma u_s}{\rho_s D_w} \right)^{0.63} \left( \frac{Re_s}{Re_w} \right)^{0.785} \quad (10)$$

where:

- $K_s$  = coefficient for water transfer through the solvent film, mols per hour per square foot per unit concentration difference, in mols per cubic foot
- $d$  = diameter of tube, or length of interface perpendicular to the direction of flow, feet
- $D_w$  = diffusion of water in solvent, square feet per hour
- $\sigma$  = interfacial tension, pounds per hour per hour
- $u_s$  = viscosity of solvent, pounds per hour per foot
- $u_w$  = viscosity of water, pounds per hour per foot
- $\rho_s$  = density of solvent, pounds per cubic foot
- $Re_s$  = Reynolds number of solvent phase, dimensionless
- $Re_w$  = Reynolds number of water phase, dimensionless.

The equation for determining the individual transfer coefficient for the solvent through the water film was correlated as follows:

$$\frac{k_w d}{D_s} = 0.000115 \left( \frac{d}{u_w D_s} \right)^{0.16} \left( \frac{u_s}{u_w} \right)^{0.44} \left( \frac{\sigma u_w}{\rho_w D_s} \right)^{0.64} \left( \frac{d G_w}{u_w} \right) \quad (11)$$

where:

- $k_w$  = coefficient for solvent transfer through the water film, moles per hour per square foot per unit concentration difference, in moles per cubic foot
- $d$  = diameter of tube, or length of interface, perpendicular to the direction of flow, feet
- $D_s$  = diffusivity of solvent in water, square feet per hour
- $\sigma$  = interfacial tension, pounds per hour per hour
- $u_s$  = viscosity of solvent, pounds per foot per hour
- $u_w$  = viscosity of water, pounds per foot per hour
- $\rho_w$  = density of water, pounds per cubic foot
- $G_w$  = mass velocity of water phase, pounds per hour per square foot.

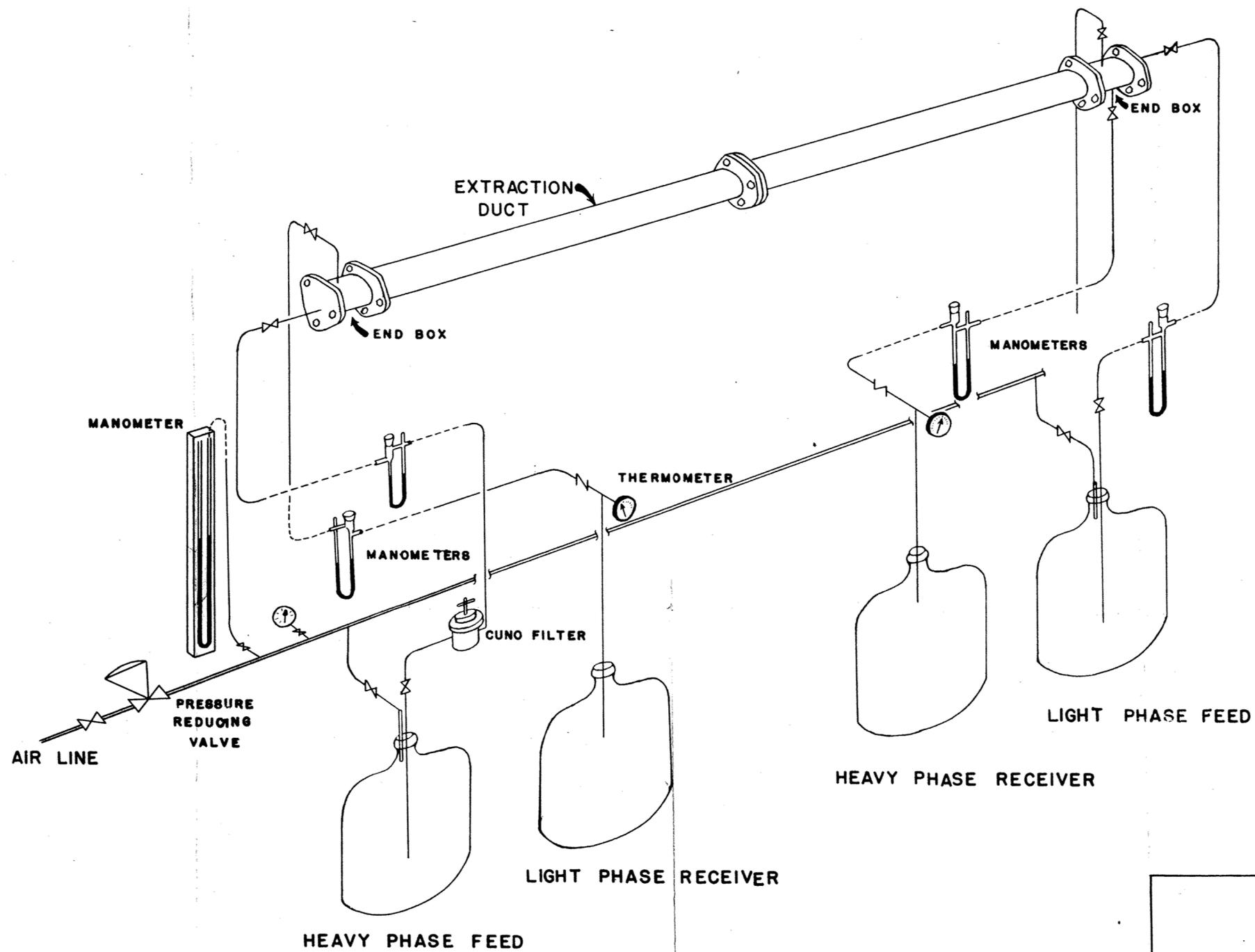


FIGURE 5  
 SCHEMATIC REPRESENTATION  
 OF HORIZONTAL, LIQUID-LIQUID  
 EXTRACTION APPARATUS

The equations for determining the individual transfer coefficient for the solvent and water films were found to be valid for the calculation of the systems used. These equations were also found to be valid for determining the individual transfer coefficients for the three component systems used.

Properties of n-Butanol, Acetone, 1,1,2-Trichloroethane  
and Water

A search of the literature was made to find the physical properties of n-butanol, acetone, 1,1,2-trichloroethane, and water. The values found in the literature for the molecular weight, boiling point, specific gravity, refractive index, viscosity, and solubilities for each of the chemicals, are presented in Tables III, IV, VII, and VIII.

The information found that dealt with the solubilities and refractive indices of mixtures of n-butanol and water, and 1,1,2-trichloroethane, acetone, and water, are shown in Tables V, VI, IX, X, and XI, and also in Figures 6, 7, 8, 10, and 12.

TABLE III

Physical Data for Normal Butyl Alcohol

1. Molecular weight . . . . .	74.1
2. Boiling point, °C . . . . .	117.0
3. Specific gravity at 25 °C . . . . .	0.810
4. Viscosity at 25 °C, centipoise . . . . .	0.894
5. Refractive index at 25 °C . . . . .	1.3990
6. Solubility of n-butanol in water, wt % . .	7.35
7. Solubility of water in n-butanol, wt % . .	20.27

International Critical Tables, Vol. III, pp. 112-13,  
McGraw-Hill Book Co., New York, N. Y., 1928.

TABLE IV

Physical Properties of Water

1. Molecular weight . . . . .	18.0
2. Boiling point, °C . . . . .	100.0
3. Specific gravity at 25 °C . . . . .	0.9970
4. Viscosity at 25 °C, centipoise . . . . .	0.8937
5. Refractive index at 25 °C . . . . .	1.3333

International Critical Tables, Vol. III, pp. 10-11,  
McGraw-Hill Book Co., New York, N. Y., 1928.

TABLE V

Solubility Data for n-Butanol and Water

Temperature °C	Weight Fraction n-Butanol	
	Butanol Phase wt %	Water Phase wt %
5	80.38	9.55
10	80.33	8.91
15	80.14	8.21
20	79.93	7.81
25	79.73	7.35
30	79.38	7.08
35	78.94	6.83
40	78.59	6.60
50	77.58	6.46
60	76.38	6.52
70	74.79	6.73
80	73.53	6.89
90	69.90	7.90
100	66.90	7.20
110	62.40	10.50
120	54.00	16.10

International Critical Tables, Vol. III,  
p.338, McGraw-Hill Book Co., Inc.,  
New York, N. Y., 1929.



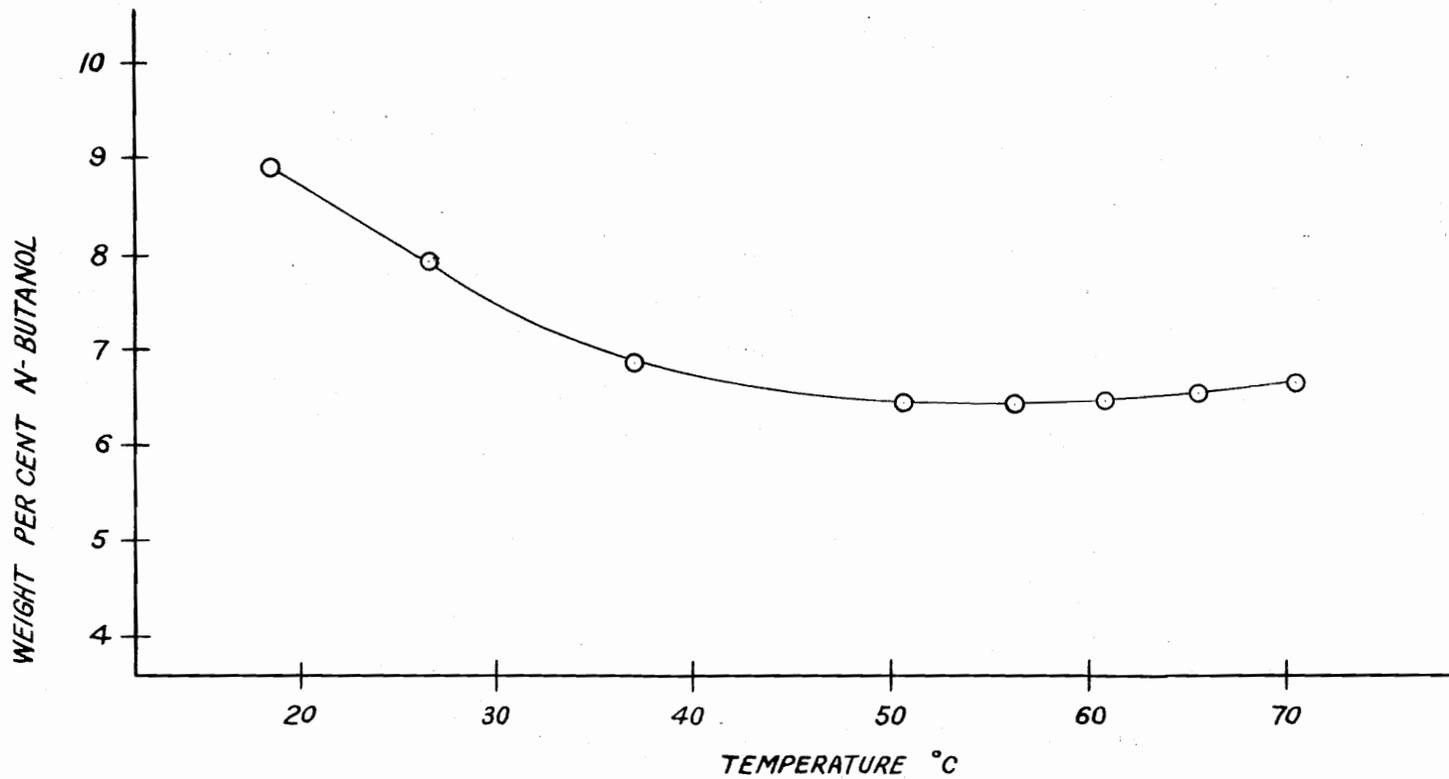


FIGURE 6, SOLUBILITY CURVE FOR N-BUTANOL IN WATER

"INTERNATIONAL CRITICAL TABLES", VOL. III, p 388, McGRAW-HILL BOOK CO., N.Y., N.Y. (1936)

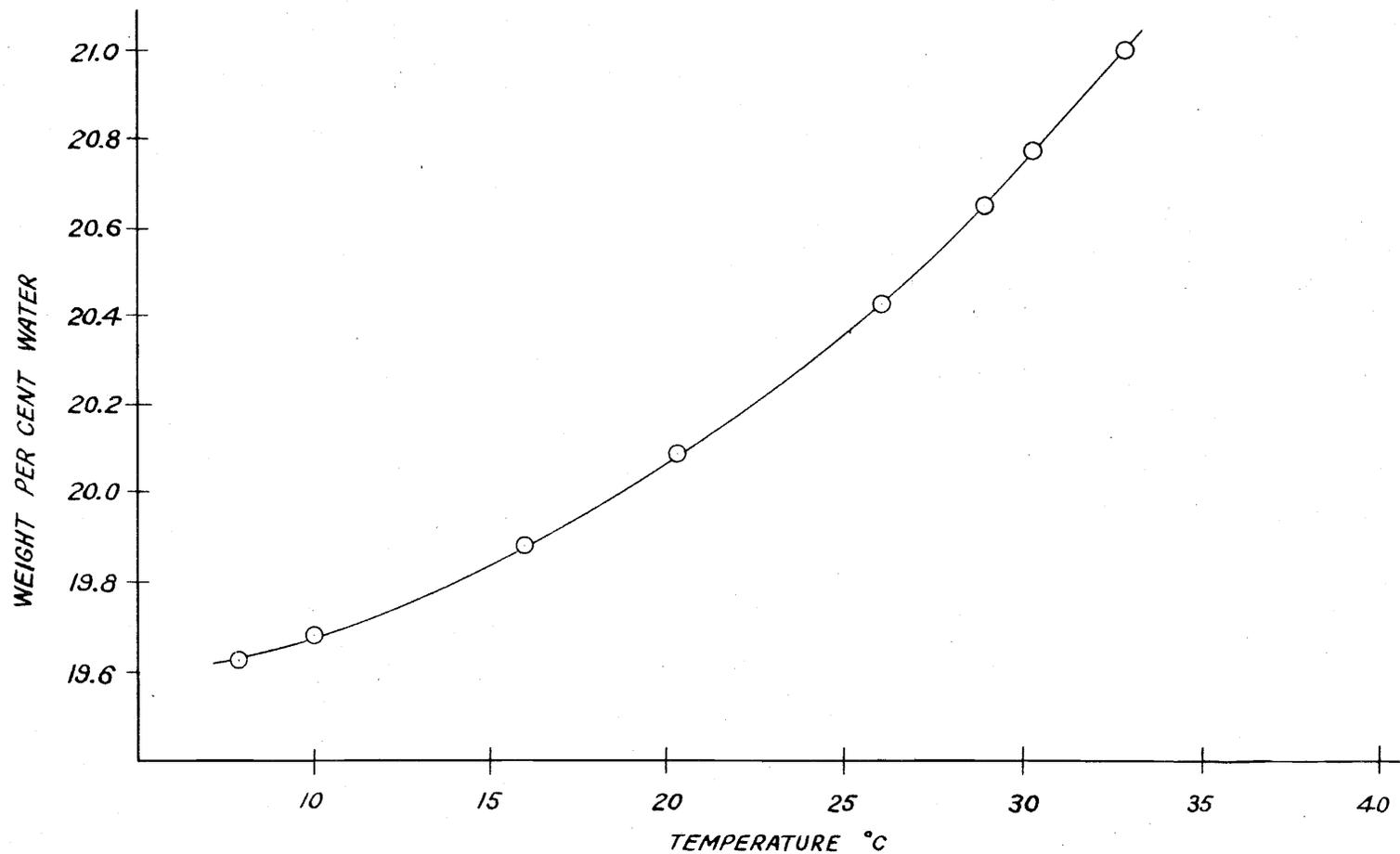


FIGURE 7. SOLUBILITY CURVE FOR WATER IN NORMAL BUTYL ALCOHOL

"INTERNATIONAL CRITICAL TABLES"; VOL. III, p 388, MCGRAW-HILL BOOK CO., N.Y., N.Y. (1936)

TABLE VI

Refractive Index Data for the Systemn-Butanol-Water at 25 °C

Sample No	Volume of Pure Components		Weight Fraction Pure Components		Refractive Index
	Water ml	Butanol ml	Water wt %	Butanol wt %	
1	50.00	0.00	100.00	0.00	1.3320
2	50.00	0.50	99.10	0.90	1.3329
3	50.00	1.00	98.40	1.60	1.3339
4	50.00	1.50	97.60	2.40	1.3347
5	50.00	2.00	96.90	3.10	1.3354
6	50.00	3.00	95.40	4.60	1.3369
7	50.00	4.00	94.00	6.00	1.3384
8	0.00	20.00	0.00	100.00	1.3969
9	0.15	20.00	0.92	99.08	1.3964
10	0.30	20.00	1.83	98.17	1.3962
11	0.45	20.00	2.72	97.28	1.3959
12	0.60	20.00	3.58	96.42	1.3957
13	0.90	20.00	5.28	94.72	1.3952
14	1.20	20.00	6.93	93.07	1.3943
15	1.80	20.00	9.95	90.05	1.3932
16	2.40	20.00	12.96	87.04	1.3920
17	3.00	20.00	15.69	84.31	1.3907
18	3.60	20.00	18.22	81.78	1.3892
19	3.87	20.00	19.50	81.50	1.3879

Eargle, J. C.: The Effect of Phase Velocity on the Mass Transfer Coefficients of Extraction and Correlation of Mass Transfer Coefficients Using the Systems n-Butanol-Water and n-Butanol-Acetic Acid-Water in a Horizontal Tube Extractor, p. 90. Unpublished M. Sc. Thesis, Library, Va. Poly. Inst., Blacksburg, Va., (1951).

TABLE VII

Physical Properties of 1,1,2-Trichloroethane

1. Molecular weight . . . . .	133.4
2. Boiling point, °C . . . . .	113.5
3. Specific gravity at 25 °C . . . . .	1.431
4. Viscosity at 25 °C, centipoise . . . . .	1.061
5. Refractive index at 25 °C . . . . .	1.4687
6. Solubility of 1,1,2-trichloroethane in water at 25 °C, wt % . . . . .	0.44
7. Solubility of water in 1,1,2-trichloro- ethane at 25 °C, wt % . . . . .	0.11

International Critical Tables, Vol. V,  
p. 216. McGraw-Hill Book Co., Inc.,  
New York, N. Y., 1928.

TABLE VIII

Physical Properties of Acetone

1. Molecular weight . . . . .	58.0
2. Boiling point, °C . . . . .	56.5
3. Specific gravity at 25 °C . . . . .	0.792
4. Viscosity at 25 °C, centipoise . . . . .	0.319
5. Refractive index at 25 °C . . . . .	1.3356

International Critical Tables, Vol. III,  
pp. 218-19. McGraw-Hill Book Co., Inc.,  
New York, N. Y., 1928.

TABLE IX

Refractive Indices of Acetone-Water Solutions

Concentration of Acetone	Refractive Index
wt %	$n_D^{25\text{ }^\circ\text{C}}$
0.00	1.3323
25.90	1.3501
45.50	1.3594
51.50	1.3614
61.70	1.3626
maximum - 73.50	1.3632
76.30	1.3632
87.96	1.3621
94.90	1.3596
100.00	1.3555

International Critical Tables, Vol. VII, p. 68.  
McGraw-Hill Book Co., Inc., New York, N. Y.,  
1928.

TABLE X

Properties of Acetone-1,1,2-Trichloroethane Solutions

Acetone wt %	Density gm/ml	Viscosity centipoises	Refractive Index $n_D^{25} \text{ }^\circ\text{C}$
0.0	1.4316	1.016	1.4687
10.5	1.3212	0.910	1.4498
20.1	1.2337	0.799	1.4343
30.1	1.1521	0.677	1.4210
40.6	1.0771	1.577	1.4079
49.3	--	--	1.3980
50.9	1.0140	0.509	--
60.6	0.9588	0.456	1.3871
69.1	0.9158	0.411	1.3796
80.0	0.8643	--	1.3698
89.1	0.8226	0.347	1.3630
100.0	0.7840	0.319	1.3556

Treybal, R. E., L. D. Weber, and J. F. Daley: The System Acetone-Water-1,1,2-Trichloroethane, Inc. Eng. Chem., 38, 817-21 (1946).

TABLE XI

Limiting Liquid Solubility Data for the System

Acetone-Water-1,1,2-Trichloroethane

1,1,2-Trichloroethane	Water	Acetone	Refractive Index
wt %	wt %	wt %	$n_D^{25} \text{ } ^\circ\text{C}$
99.89	0.11	0.00	1.4683
94.73	0.26	5.01	1.4588
90.11	0.36	9.53	1.5404
84.65	0.59	14.76	1.4416
79.58	0.76	19.66	1.4338
75.72	0.99	23.29	1.4285
70.36	1.43	28.21	1.4218
67.52	1.44	31.04	1.4178
64.17	1.87	33.96	1.4139
60.06	2.11	37.83	1.4088
54.88	2.98	42.14	1.4032
48.78	4.01	47.21	1.3972
43.88	5.00	51.12	1.3927
38.31	6.84	54.85	1.3888
31.67	9.78	58.55	1.3829
26.39	13.35	60.26	1.3792
24.04	15.37	60.59	--
23.20	16.63	60.17	1.3792
20.71	19.31	59.98	1.3758
15.39	26.38	58.33	1.3695
10.00	34.96	55.04	1.3672
9.63	35.38	54.99	1.3672
6.77	41.35	51.88	1.3652
4.35	48.47	47.18	1.3626
2.18	55.97	41.85	1.3601
1.72	61.11	37.17	1.3578
1.17	66.58	32.25	1.3543
1.02	71.80	27.18	1.3518
0.92	74.54	24.54	1.3501
0.78	80.40	18.82	1.3460
0.70	84.94	14.36	1.3430
0.65	87.63	11.72	1.3412
0.52	94.66	4.82	1.3362
0.44	99.56	0.00	1.3328

Treybal, R. E., L. D. Weber, and J. F. Daley:  
 The System Acetone-Water-1,1,2-Trichloroethane, Ind. Eng. Chem., 38, 817-21  
 (1946).



TABLE XII

Equilibrium Tie Line Concentrations for the System  
Acetone-Water-1,1,2-Trichloroethane

Water Phase			Trichloroethane Phase		
Trichloroethane	Water	Acetone	Trichloroethane	Water	Acetone
wt %	wt %	wt %	wt %	wt %	wt %
0.52	93.52	5.96	90.93	0.32	8.75
0.54	92.05	6.51	89.32	0.40	10.28
0.68	85.35	13.97	78.32	0.90	20.78
0.73	82.23	17.04	73.76	1.10	25.14
0.79	80.16	19.05	71.01	1.33	27.66
1.00	73.00	26.00	60.85	2.09	37.06
1.02	72.06	26.92	59.21	2.27	38.52
1.04	71.33	27.63	58.21	2.40	39.39
1.11	69.35	29.54	55.48	2.85	41.67
1.17	67.95	30.88	53.92	3.11	42.97
1.60	62.67	35.73	47.53	4.26	48.21
2.10	57.00	40.90	40.00	6.05	53.95
3.75	50.20	46.05	33.70	8.90	57.40
6.52	41.70	51.78	26.26	13.40	60.35

Treybal, R. E., L. D. Weber, and J. F. Daley: The System Acetone-Water-1,1,2-Trichloroethane, Ind. Eng. Chem., 38, 817-21 (1946).

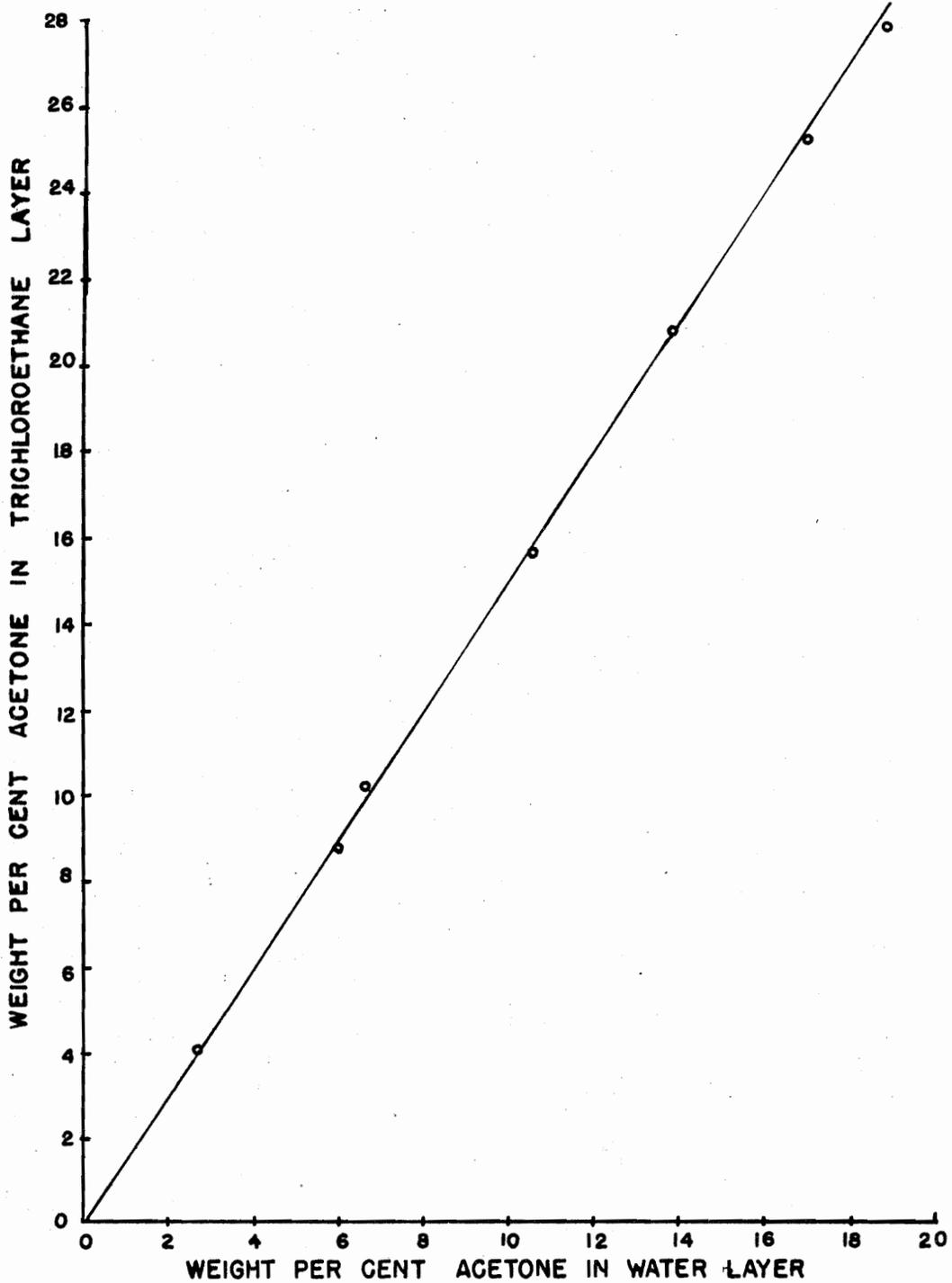


FIGURE 8, DISTRIBUTION OF ACETONE BETWEEN WATER AND 1,1,2-TRICHLOROETHANE AT 25 °C.

TREYBAL, R.E, L.D.WEBER, AND J.F.DALEY: THE SYSTEM ACETONE - WATER - 1,1,2-TRICHLOROETHANE, IND. ENG. CHEM., 38, 817-21, (1946).

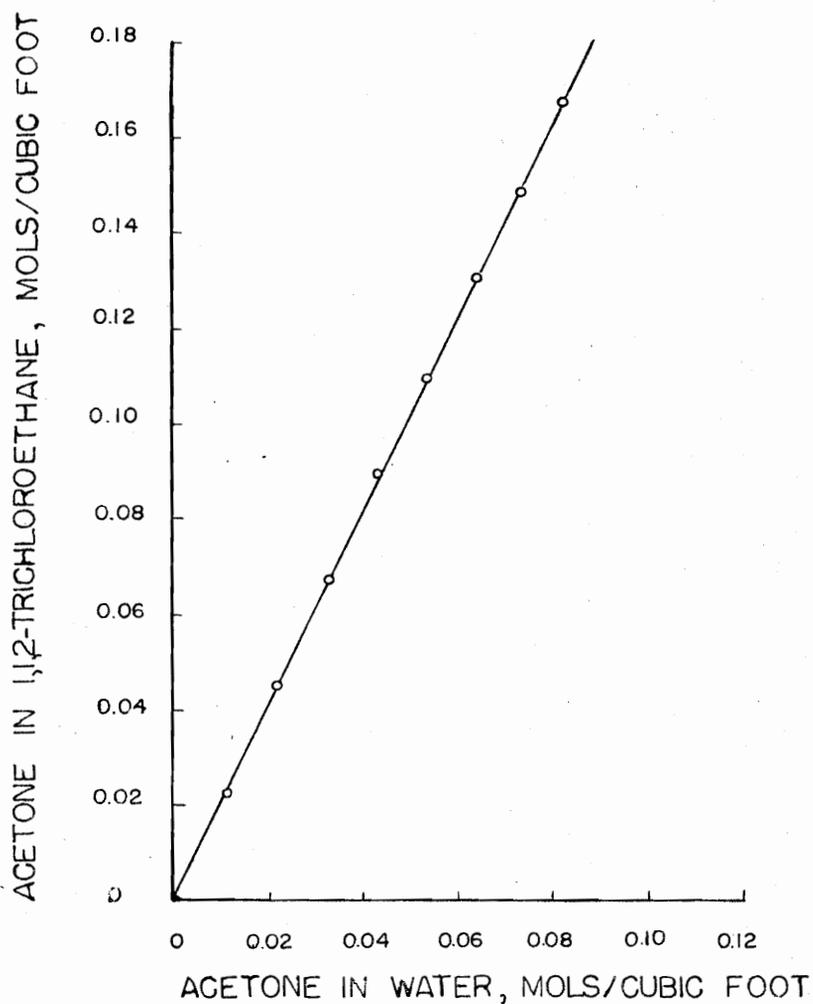


FIGURE 9, DISTRIBUTION OF ACETONE IN WATER AND 1,1,2-TRICHLOROETHANE AT 25 °C

TREYBAL, R. E., L. D. WEBER, AND J. F. DALEY: THE SYSTEM ACETONE-WATER-1,1,2-TRICHLOROETHANE, IND. ENG. CHEM., 38, 817-21, (1946).

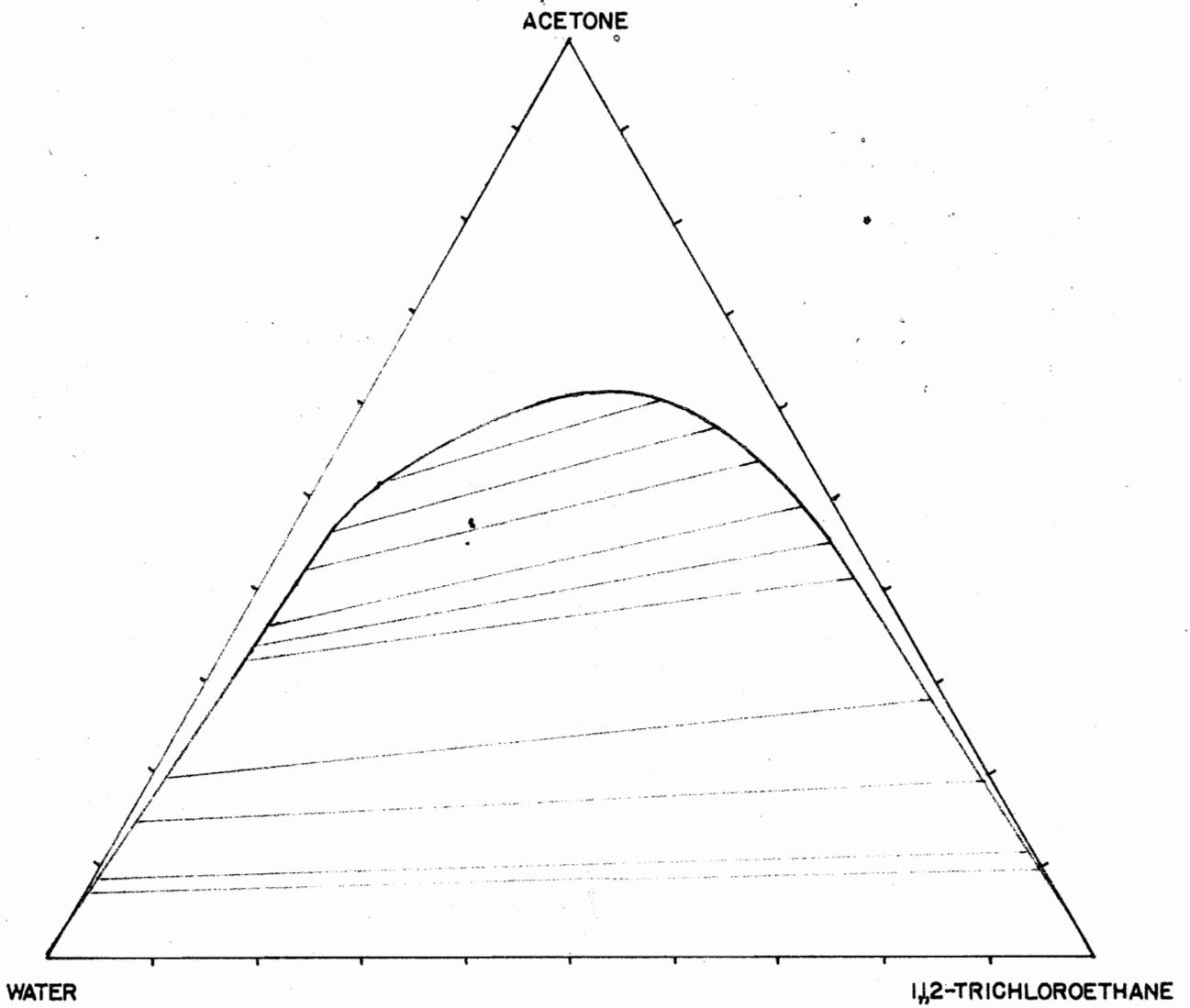


FIGURE 10. LIMITING, LIQUID-SOLUBILITY CURVE AND EQUILIBRIUM TIE LINES FOR THE SYSTEM ACETONE-WATER-1,1,2-TRICHLOROETHANE AT 25 °C.

TREYBAL, R.E., L.D. WEBER, AND J.F. DALEY: THE SYSTEM ACETONE-WATER-1,1,2-TRICHLOROETHANE, IND. ENG. CHEM., 38, 817-21 (1946).

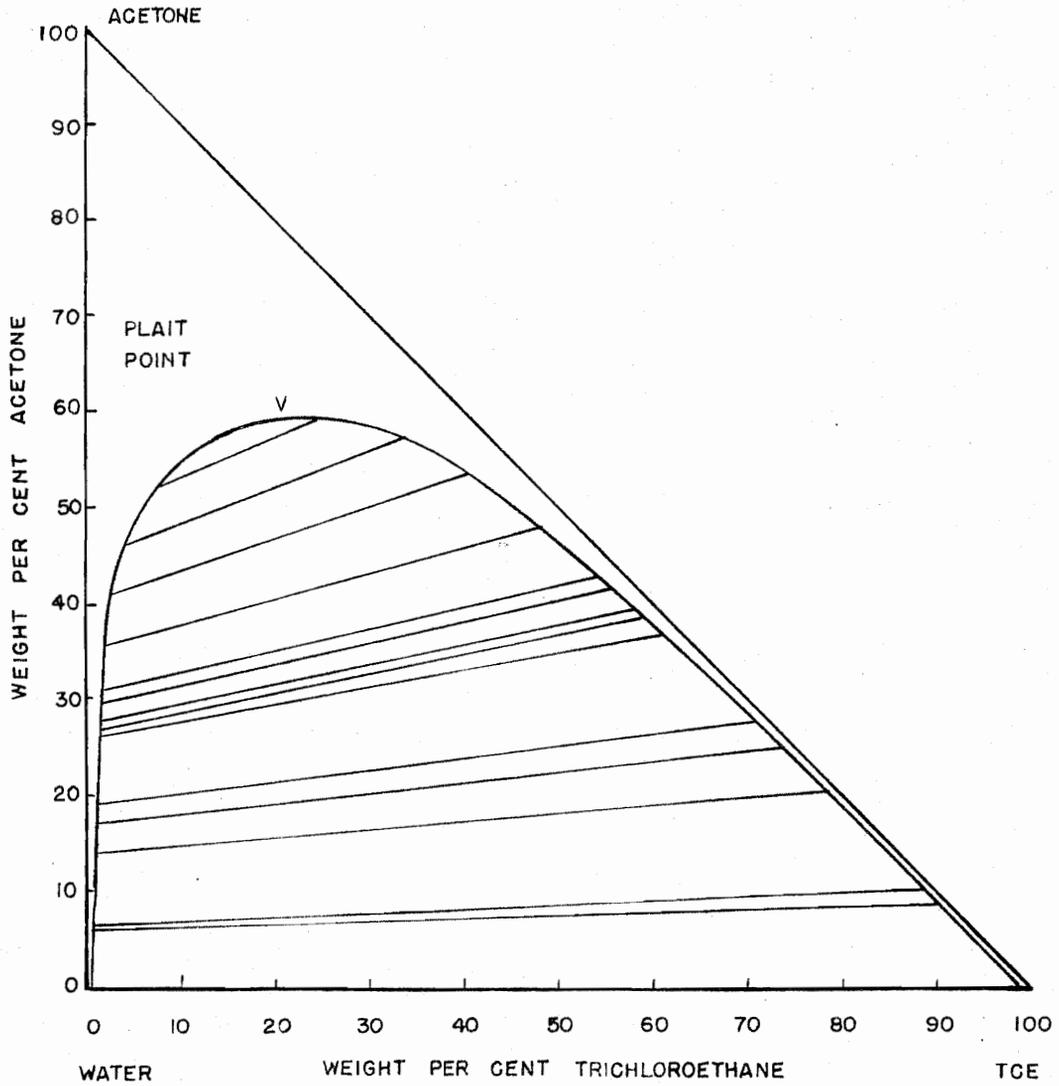
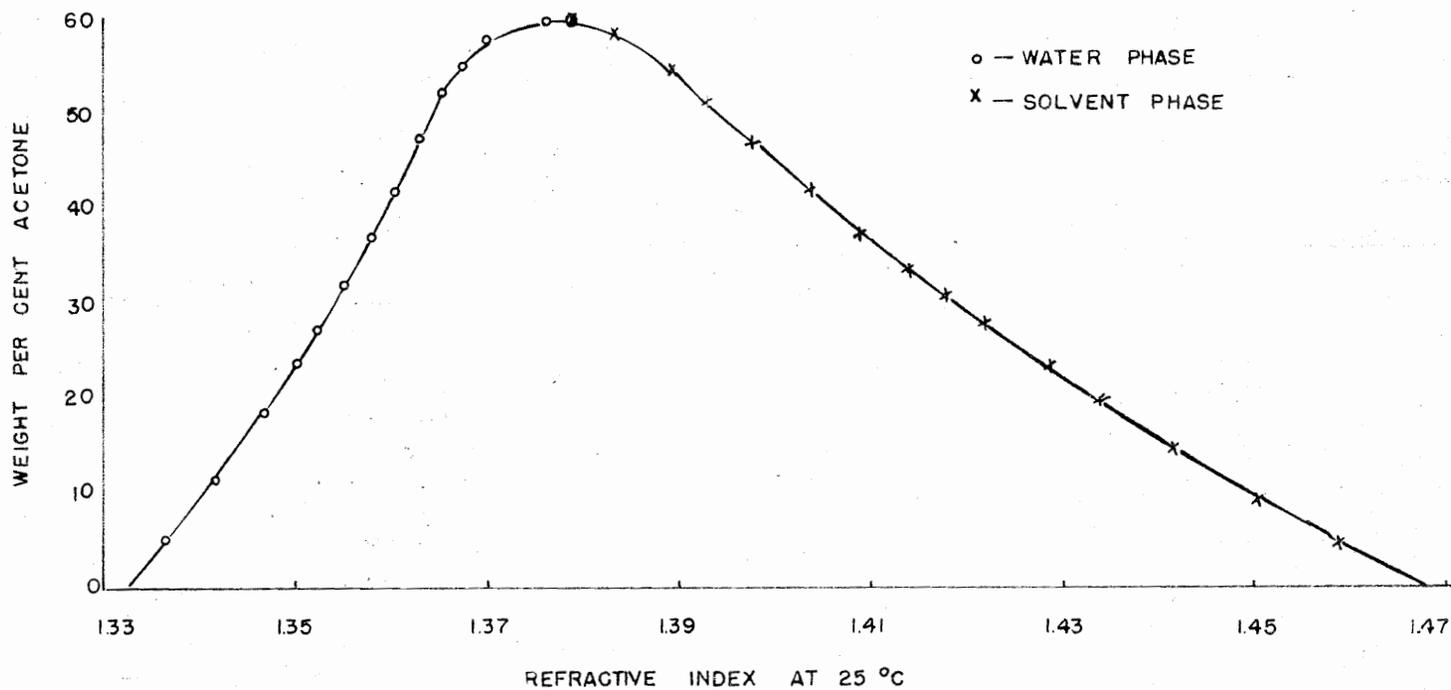


FIGURE II, LIMITING, LIQUID-SOLUBILITY CURVE AND EQUILIBRIUM TIE LINES FOR THE SYSTEM ACETONE-WATER-1,1,2-TRICHLOROETHANE AT 25 °C.

TREYBAL, R.E, L.D.WEBER, AND J.F.DALEY; THE SYSTEM ACETONE-WATER-1,1,2 TRICHLOROETHANE, IND. ENG. CHEM., 38, 817-21, (1946).



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FIGURE 12, REFRACTIVE INDEX CURVE FOR POINTS ON THE LIMITING LIQUID SOLUBILITY CURVE FOR THE SYSTEM ACETONE-WATER-1,1,2-TRICHLOROETHANE.

TREYBAL, R.E., L.D.WEBER, AND J.F.OAILEY: THE SYSTEM ACETONE-WATER-1,1,2-TRICHLOROETHANE, IND. ENG. CHEM. 38, 817-21 (1946).

### III. EXPERIMENTAL

The experimental section includes the purpose of the investigation, the plan of experimentation, the materials, a list of apparatus used in the experimentation, the equipment, the data and results obtained, and a sample of the calculations used in obtaining the results.

#### Purpose of the Investigation

The purpose of this investigation was to determine the effect of variable phase velocities and the direction of flow on the overall mass transfer coefficient in a horizontal, unpacked extraction tube for the systems n-butanol-water, and 1,1,2-trichloroethane-acetone-water.

#### Plan of Experimentation

The investigation was carried out according to the following plan:

Literature Review. A survey of the literature was conducted so that the general considerations of liquid-liquid extraction, definitions, applications, design of equipment, extraction systems, and theory, could be presented. A search

was also made for results obtained by previous investigators who used a horizontal tube extractor with countercurrent flow.

Construction of Equipment. The equipment used in this investigation was a horizontal tube extractor utilizing cocurrent flow. The extraction chamber consisted of two, four-foot, pyrex tubes mounted horizontally, with a calming section in one end of the tube. The feeds and solvents were forced from storage tanks by compressed air, through copper tubing, into the extraction chamber. The extracts and raffinates were removed from the extractor through copper tubing and collected in glass carboys. Orifice flow meters were installed in the feed, solvent, extract, and raffinate lines to aid in controlling the flow rate of the entering and leaving phases. The apparatus was constructed so that the flow rates of the phases could be varied and adequate samples could be taken from each phase.

Experimental Procedure. Four series of tests were made for each system used in the investigation. The flow rate of the water phase in the system n-butanol-water was held constant and the flow rate of the butanol phase varied from approximately 6000 to 25,000 pounds per hour per square foot. In each series of tests, the water phase flow rate was varied from approximately 4000 to 21,000 pounds per hour per square foot.



The four series of tests performed on the system 1,1,2-trichloroethane-acetone-water were done with the water rate being constant for each series, but varying from approximately 4000 to 22,000 pounds per hour per square foot, for the four series of tests. The trichloroethane phase flow rates varied from approximately 7000 to 31,000 pounds per hour per square foot for each series of tests.

The tests performed on the system 1,1,2-trichloroethane-acetone-water were done with the trichloroethane phase saturated with water, and the water phase saturated with trichloroethane. The acetone, which was the solute, was present in the trichloroethane phase to approximately five per cent by weight.

The extraction tests performed on both systems were done at approximately 25 °C. Also, the calibration curves for the analysis of samples using the dipping refractometer and the Abbe refractometer were determined for use at 25 °C.

Treatment of Data. The overall mass transfer coefficient based on the n-butanol phase and the water phase were calculated for each test in the series and plotted against the flow rate of each phase to show the effect of varying phase velocities. The ratio of the flow rate of the

n-butanol phase to that of the water phase was plotted against the transfer coefficient based on each phase.

The overall mass transfer coefficient based on the trichloroethane phase and on the water phase, were calculated for each test in the series and plotted against the flow rate of each phase to show the effect of varying phase velocities. The overall mass transfer coefficients based on the water phase and the trichloroethane phase were plotted against the ratios of the trichloroethane phase flow rates to the water phase flow rates.

Materials

The following materials were used in this investigation:

Acetone. N.F. G095J. Obtained from General Chemical Division, Allied Chemical and Dye Corp., New York, N. Y. Used as the solute.

Alcohol, Normal Butyl. Technical grade. Obtained from Commercial Solvents Corp., New York, N. Y. Used as a feed.

1,1,2-Trichloroethane. Technical grade. Obtained from Carbide and Carbon Chemicals Co., South Charleston, W. Va. Used as the carrier solvent.

Water, Distilled. Obtained from the Chemical Engineering Department, Virginia Polytechnic Institute, Blacksburg, Va. Used in the preparation of samples.

Water, Tap. Obtained from the water mains in Davidson Hall, Virginia Polytechnic Institute, Blacksburg, Va. Used as the extracting solvent.

TABLE XIII

Materials of Construction  
for Storage Tanks

## Material for Feed Tanks

Item	Quantity	Description	Supplier
Lumber	24 ft	2-inch x 2-inch softwood	New River Lumber Blacksburg, Va.
Steel cylinders	3 ea	9-gallon capacity, stainless steel, oval shaped	USA-Corp Chicago, Ill.
Metal strap	12 ft	$\frac{1}{2}$ -inch steel, $\frac{1}{32}$ -inch thick	USA-Corp Chicago, Ill.
Glass tube	5 ft	$\frac{3}{8}$ -inch o.d.	Fisher Scientific Silver Springs, Md.
Nuts and Bolts	4 ea	$\frac{1}{2}$ -inch dia. x $6\frac{1}{2}$ -inch, steel	Noland Co., Inc. Roanoke, Va.
Nuts and Bolts	12 ea	$\frac{1}{2}$ -inch dia. x $2\frac{1}{2}$ -inch, steel	Noland Co., Inc. Roanoke, Va.

## Material for Solvent (Water) Tank

Water tank	1 ea	30-gallon, cylindrical	Noland Co., Inc. Roanoke, Va.
Tank stand	1 ea	12-inch diameter, metal	Chem. Engr. Dept. V.P.I., Blacksburg, Va.
Thermometer	1 ea	$\frac{1}{2}$ -inch screw type, 6-inch stem, deal type weston, 10 to 300°C. range	Weston Inst. Co. Newark, N.J.
Sight gage valves	1 pr	$\frac{1}{2}$ -inch brass pipe fitting, angled with drain-cocks	Noland Co., Inc. Roanoke, Va.
Glass tube	1 ea	$\frac{5}{8}$ -inch o.d., x 44 inches	Fisher Scientific Silver Springs, Md.

## Pipe, Tubing, Valves, and Fittings for Tanks

Stopcocks	10 ea	$\frac{3}{8}$ -inch o.d., copper	Noland Co., Inc. Roanoke, Va.
Tees	8 ea	$\frac{3}{8}$ -inch o.d., copper	Noland Co., Inc. Roanoke, Va.
	3 ea	$\frac{1}{2}$ -inch standard galv. iron	Noland Co., Inc. Roanoke, Va.
	6 ea	$\frac{3}{8}$ -inch x $\frac{3}{8}$ -inch x $\frac{1}{4}$ -inch NPT, galv. iron	Noland Co., Inc. Roanoke, Va.
Adapters	11 ea	$\frac{3}{8}$ -inch NPT x $\frac{3}{8}$ -inch copper tube	Noland Co., Inc. Roanoke, Va.
Tubing	45 ea	$\frac{3}{8}$ -inch, o.d., copper	Noland Co., Inc. Roanoke, Va.
Gas jets	3 ea	$\frac{3}{8}$ -inch NPT, black iron	Noland Co., Inc. Roanoke, Va.
Nipples	6 ea	$\frac{1}{4}$ -inch x $1\frac{1}{2}$ -inch standard galv. iron, NPT	Noland Co., Inc. Roanoke, Va.
Reduc. bushing	6 ea	1 inch x $\frac{1}{2}$ -inch, std. galv.	Noland Co., Inc. Roanoke, Va.
Reduc. coupling	2 ea	$\frac{1}{2}$ -inch x $\frac{1}{4}$ -inch, standard galv. iron	Noland Co., Inc. Roanoke, Va.
Elbows	12 ea	$\frac{1}{2}$ -inch standard galv. iron	Noland Co., Inc. Roanoke, Va.
Unions	4 ea	$\frac{1}{2}$ -inch standard galv. iron	Noland Co., Inc. Roanoke, Va.
Valves	3 ea	$\frac{1}{2}$ -inch, bronze, globe type, plug seat, rising stem	Noland Co., Inc. Roanoke, Va.
	2 ea	$\frac{1}{2}$ -inch, bronze, gate type, rising stem	Noland Co., Inc. Roanoke, Va.
Pipe	21 ft	$\frac{1}{2}$ -inch galvanized iron	Noland Co., Inc. Roanoke, Va.

TABLE XIV

Materials of Construction for Horizontal Tube,  
Supports, Air Supply and Containers

## Material for Horizontal Tube

Item	Quantity	Description	Supplier
Glass pipe	2 ea.	1½-inch inside diameter 4 ft overall-flared ends	Corning Glass works Corning, N.Y.
Flanges	4 ea	3-hole aluminum 3/8-inch bolt bore	Corning Glass Works Corning, N.Y.
Gaskets	3 ea	Black rubber-1½-inch i.d. 1/8-inch thickness	Corning Glass Works Corning, N.Y.
Nuts and Bolts	15 ea	aluminum, hexagonal head 1/2-inch x 3/8-inch	Corning Glass Works Corning, N.Y.
Steel Plate	1 sq ft	carbon steel, 3/16-inch used in end flanges	Noland Co., Inc. Roanoke, Va.
Pipe	1 ft	Std.-1½-inch, schedule 40 Black iron	Noland Co., Inc. Roanoke, Va.
Copper screen	1 sq ft	16 mesh, light weight used for calming sections	Noland Co., Inc. Roanoke, Va.
Nipples	5 ea	6-inch x 1/2-inch, black iron, sched. 40	Noland Co., Inc. Roanoke, Va.
Valves	4 ea	1/2-inch bronze body, needle type globe, rising stem	Noland Co., Inc. Roanoke, Va.
Coek	1 ea	1/2-inch bronze body, plug type, screwed ends	Noland Co., Inc. Roanoke, Va.
Gaskets	2 sq ft	1/16-inch thick, soft cork	Noland Co., Inc. Roanoke, Va.

## Material for Tube Support

Plywood	1 ea	12 ft x 4 ft x 3/8-inch	New River Lumber Blacksburg, Va.
Clamps	4 ea	utility, rubber sleeved screw clamps	Fisher Scientific Silver Springs, Md.
Clamps	8 ea	screw type clamps	Fisher Scientific Silver Springs, Md.
Rods	4 ea	solid steel, 18-inch x ½-inch diameter	Noland Co., Inc. Roanoke, Va.
Lumber	30 ft	2-inch x 2-inch softwood	New River Lumber Blacksburg, Va.

## Air Supply Equipment

Pressure Regul.	1 ea	Comolow, brass body, plug and stem stainless steel, nylon diaph., for inlet pressures up to 150 psi	Comolow Corp. 2100 Arch St. Philad., Pa.
Air Compressor	1 ea	model 120-511, 300 r.p.m. Auto air compress	Borrowed from salt spray app. Ch.E. Dept.
Steel cylinder	1 ea	9-gallon capacity, stainless steel, oval shaped	USA-Corp Chicago, Ill.

## Containers

Carboys	3 ea	5-gallon capacity, glass	Fisher Scientific Silver Springs, Md.
Carboys	3 ea	12½-gallon capacity, glass	General Chem., Co. New York, N.Y.
Stoppers	3 ea	Number 11, black rubber	Fisher Scientific Silver Springs, Md.
Rod	1 ft	3/8-inch solid steel	Noland Co., Inc. Roanoke, Va.
Strap	2 ft	½-inch steel, 1/16-inch wall thickness	Noland Co., Inc. Roanoke, Va.

TABLE XV

Materials of Construction:Tubing, Pipe, Valves and Fittings

Item	Quantity	Description	Supplier
Black iron			
Nipples	10 ea	1/2-inch x 2-inch, standard	Noland Co., Inc. Roanoke, Va.
Nipples	4 ea	1/8-inch x 4-inch, std.	Noland Co., Inc. Roanoke, Va.
Tees	3 ea	1/2-inch x 1/2-inch x 1/2-inch	Noland Co., Inc. Roanoke, Va.
Reduc. coupling	4 ea	3/8-inch x 1/2-inch, std.	Noland Co., Inc. Roanoke, Va.
Reduc. bushing	6 ea	1/2-inch x 3/8-inch, std.	Noland Co., Inc. Roanoke, Va.
Reduc. bushing	8 ea	1/2-inch x 1/8-inch, std.	Noland Co., Inc. Roanoke, Va.
Valves	2 ea	1/2-inch, bronze, globe type, plug seat, rising stem	Noland Co., Inc. Roanoke, Va.
Valves	4 ea	1/2-inch, bronze, globe type, plug seat, rising stem	Noland Co., Inc. Roanoke, Va.
Pipe	20 ft	1/2-inch, standard sch. 40	Noland Co., Inc. Roanoke, Va.
Pipe	30 ft	1/2-inch, standard sch. 40	Noland Co., Inc. Roanoke, Va.
Copper			
Adapters	12 ea	1/2-inch NPT x 3/8-inch copper tube	Noland Co., Inc. Roanoke, Va.
Adapters	20 ea	1/2-inch NPT x 1/2-inch copper tube	Noland Co., Inc. Roanoke, Va.
Orifice clamps	4 ea	1/2-inch, standard sch. 40	Inland Supply Syracuse, N.Y.
Orifice plates	4 ea	3/32-inch orifice, copper	Ch.D. Dept. V.F.I., Blacksburg, Va.
Tubing	20 ft	1/2-inch o.d., copper	Noland Co., Inc. Roanoke, Va.
Tubing	70 ft	3/8-inch o.d., copper	Noland Co., Inc. Roanoke, Va.
Valves	14 ea	1/2-inch, bronze, needle type, rising stem	Noland Co., Inc. Roanoke, Va.
Valves	14 ea	3/8-inch, bronze, needle type, rising stem	Noland Co., Inc. Roanoke, Va.
Manometers	4 ea	Meriam, 20-inch scale 1/10-inch divisions	Meriam Inst. Co. Cleveland, Ohio
Thermometers	2 ea	1/2-inch, screw type 6-inch stem, dial type Weston, 50-300°F range	Western Inst. Co. Newark, N.J.
Mercury	25 lb	purified	Fisher Scientific Silver Springs, Md.

### Apparatus

The apparatus that was used in this investigation may be classified into three categories. These three categories of apparatus are: (1) apparatus used in the analytical work, (2) apparatus used in the solvent purification, and (3) apparatus used in the construction of the extraction tube and its accessory equipment.

The apparatus used in performing the analytical work was:

Balance. Analytical type, chain-o-matic, capacity 3000 gm, serial No RL8702. Manufactured by Christian Becker, Inc., New York, N. Y. Used in the preparation of known mixtures.

Balance. Triple beam type, capacity 21 kilograms, graduated in grams. Obtained from Fisher Scientific Company, Silver Spring, Md. Used to weigh exit streams from extraction tube.

Balance. Westphal type. Obtained from Fisher Scientific Company, Silver Spring, Md. Used to determine the specific gravity of mixtures.

Bath, Constant Temperature. Water, mechanically agitated, insulated, cooled by tap water circulated through a 20-foot coil of 3/8-inch copper tubing. The size of the bath was 30 by 41 by 14 inches. Obtained from the Chemical Engineering Department, Virginia Polytechnic Institute,

Blacksburg, Va. Used to maintain water circulating through the Abbe refractometer at a constant temperature.

Cylinders, Graduated. Pyrex, capacity 100 and 1000 ml at 20 °C. Obtained from Fisher Scientific Company, Silver Spring, Md. Used to measure solutions and to calibrate the manometers in the extraction equipment.

Motor. Electric 1/2-hp, 60 cy, 110 v, 15 amp, 1750 rpm, ac, No 516820. Manufactured by Robbins and Myers Company, Springfield, Ohio. Used to drive positive displacement pump in water circulating apparatus for refractometer.

Pipets. Glass, 1, 5, 10, and 25 ml at 20 °C. Obtained from Fisher Scientific Co., Silver Spring, Md. Used for measuring and transferring liquid materials.

Pump. Positive displacement, model PP-L, serial No 1767. Manufactured by Ace Equipment Co., Newark, N. J. Used to circulate water from constant temperature bath through the refractometer.

Refractometer. Spence-Abbe type, No 1777, range  $n_{25}^D$  1.30-1.70, accuracy two units in fourth decimal place. Manufactured by the American Optical Co., Buffalo, N. Y. Used to measure the refractive indices of mixtures of 1,1,2-trichloroethane-acetone-water.



Refractometer. Dipping type, No SB2163. Equipped with prisms "A" and "B", accuracy when converted to refractive indices is three units in fifth decimal place. Manufactured by Bausch and Lomb Optical Co., Rochester, N. Y. Used in the analysis of n-butanol-water mixtures, and 1,1,2-trichloroethane-acetone-water mixtures.

Stirrer. "Lightnin" mixer, 110 v, 60 cy, single phase, ac, model "L". Manufactured by Mixing Equipment Co., Inc., Rochester, N. Y. Used to agitate water in the constant temperature bath.

The apparatus used to purify the solvents consisted of the following:

Adapter. Pyrex glass, No 5058, joint No 24/40 and 10/30 at top for thermometer. Obtained from Ace Glass Co., Vineland, N. J. Used to mount thermometer in distillation column.

Column, Distillation. Vigreux pyrex glass, length 24 inches, catalog No 6925, joint No 24/40. Obtained from Ace Glass Co., Vineland, N. J. Used to rectify vapors in solvent purification.

Condenser. Graham type, jacket 2000 mm long, catalog No 5950, joint No 24/40. Obtained from Ace Glass Co., Vineland, N. J. Used to condense vapors of distilling column.

Flask, Distilling. Pyrex glass, round bottom, three neck, with no 24/40 joint, capacity 5 liters, catalog No 6945. Obtained from Ace Glass Co., Vineland, N. J. Used as still pot for distillation column.

Flask. Pyrex glass, 150, 250, and 2000 ml. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to hold purified solvents.

Mantle, Heating. Electric heating, 110 v, 5 amp, ac, size 5l. Made by Glas-Col Apparatus Co., Terre Haute, Ind. Used to heat still pot.

Stoppers. Rubber, size 12. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to stopper flask and still pot.

Thermometer. Ground glass fitting, joint No 10/30, 3-inch stem, range 0 to 250 °C, 1 °C divisions. Obtained from Fisher Scientific Co., Silver Spring, Md. Used to measure the temperature of solvent vapors in the distillation column.

The materials used in the construction of the extraction tube and the accessory equipment, such as the feed tanks, the solvent tanks, and the air supply equipment, are found in Tables XIII, XIV, and XV.

### Method of Procedure

The procedure used for the construction and the experimental work in this investigation will be considered under three headings: (1) the design and construction, (2) the operation of the extraction tube, and (3) the analytical work involved in the analysis of the samples obtained from the extraction operation.

Design and Construction. The horizontal extraction tube used by Bowman<sup>(3)</sup> and Skrzec<sup>(36)</sup>, shown in Figure 5, page 44, was redesigned as shown in Figure 13, page 77. The principal design change in the horizontal tube extractor involved the direction of flow of the entering phases. The original extraction tube utilized countercurrent flow; this was changed in this investigation to parallel or cocurrent flow by changing the hook-up of the feed and solvent lines entering the tube. A new end plate was constructed for the right end of the tube; also, a small plate was added to separate the entering phases and to reduce mixing while the phases were in the end box portion of the tube.

All rubber tubing previously used as feed and solvent lines was replaced with copper tubing. Orifice flow meters were inserted into the entering and leaving lines.

Operating Procedure. The operating procedure was divided into four stages which were: (1) filling of storage tanks,

(2) adjusting flow rates, (3) sampling, and (4) concluding the test. The reference letters used in the procedure refer to Figure 13, page 77. The systems used in this investigation were n-butanol-water, and 1,1,2-trichloroethane-acetone-water. The operating procedure was the same for both systems.

Storage Tanks. The feed tanks F, were filled with n-butanol for the system n-butanol-water, and 1,1,2-trichloroethane containing 5 weight per cent acetone and saturated with water for the system 1,1,2-trichloroethane-acetone-water. These tanks were filled by connecting a rubber hose from valve V-20 at the top of the tank to a water aspirator. The feed was sucked into the tank through the hose on valve V-20 when the valves V-25 and V-30 were opened and the valves V-24, V-23, and V-27 were closed. After the feed was in the tank, the valves V-25, V-20, and V-30, were closed and valve V-17 was opened.

The solvent storage tank G, was filled with tap water for the system n-butanol-water, and with tap water saturated with 1,1,2-trichloroethane for the system 1,1,2-trichloroethane-acetone-water. The solvent storage tank was filled by opening valve V-16 which was connected to the water main by rubber tubing. While the solvent tank was being filled, valves V-15 and V-33 were closed. After the solvent tank

was filled, valve V-16 was closed and V-15 was opened so that compressed air could be admitted to the tank.

The compressed air was admitted to the system by opening valve V-34, and adjusting the air pressure to 14 pounds per square inch, gage by the constant pressure valve E. The compressed air was admitted to the feed tank F, by opening valve V-20. The compressed air was admitted to the solvent tank by opening valve V-15 and closing valve V-16.

The initial step in the actual operation of the extraction tube was the filling of the extraction tube with the system being used and the leveling of the interface. The tube was filled by opening valve V-14 and allowing the heavier phase to fill the bottom half of the tube. The top half of the tube was then filled with the lighter phase by opening valve V-13. During the filling operation, the entering end of the extraction tube was lowered so that the air could escape through the light phase exit line. When the tube was filled, the by-pass valves V-2, V-5, V-8, and V-11 for the manometers were closed.

The extraction tests were started at this point in the procedure. The valves V-13 and V-14 on the entering

feed and solvent lines were adjusted until the manometers indicated the desired flow rates. The valves V-35 and V-36 were then adjusted until the exit extract and raffinate manometers indicated the exit flow rates that would keep the interface in the center of the extraction tube. The exit flow rates were obtained by adjustment and measurement, the manometers being used only to indicate changes in the desired flow rates.

The time allowed for the system to reach equilibrium was determined by measuring the volume of flow from the apparatus. A volume change of three times the volume of the extraction tube was found to be sufficient for equilibrium to be reached. Each test was of sufficient time to allow 12 volume changes in the tube. Samples were collected of the extract and raffinate after every three volume changes during a test. Samples of the feed and solvent were collected at the start and finish of the test. The exact length of time for each test was determined by use of a timer. The weights of the extract and raffinate collected during the test were also found.

After determining the weights of extract and raffinate, and timing the test, the test was concluded by closing valves V-13, V-14, V-36, and V-37. If another test was to

be performed, the two phases were left in the extraction tube while the feed and solvent tanks were refilled. If no other tests were to be performed, the tube was drained and the phases were collected separately. In all tests the extract (water phase) was weighed and poured down the drain; the raffinate, after being weighed, was saved for re-use.

Analysis of Samples. The samples obtained during a test were analyzed by use of a dipping refractometer at 25 °C, for the system n-butanol-water. The samples obtained for the system 1,1,2-trichloroethane-acetone-water were divided, the trichloroethane phase being analyzed by determining the refractive indices at 25 °C by use of an Abbe refractometer, the water phase samples being analyzed by use of a dipping refractometer at 25 °C. The instruments used in the analyses had been previously calibrated by using known samples of the system used in the extraction test.

Data Recorded. The data recorded during the extraction tests performed on the systems 1,1,2-trichloroethane-acetone-water, and n-butanol-water were: (1) time of test, minutes, (2) temperature of the entering streams, °F, (3) weight of extract and raffinate, grams, (4) entering manometer readings, inches of mercury, (5) air pressure, pounds per square inch, and (6) refractive index or dipping refractometer scale readings for all samples collected during the tests.

### Data and Results

The calibration data, experimental data, and the results obtained during this investigation are presented in the following section.

Calibration Data. The calibration curves for the dipping refractometer and the system n-butanol-water are shown in Figures 14 and 15, pages 84 and 86. The calibration curve for the water phase of the system 1,1,2-trichloroethane-acetone-water for the dipping refractometer are shown in Figure 16, page 88. The calibration curve for the trichloroethane phase of the system 1,1,2-trichloroethane-acetone-water when used in the Abbe refractometer are shown in Figure 17, page 90.

System n-Butanol-Water. The data and results obtained in the determination of the overall mass transfer coefficient for the system n-butanol-water at 25 °C, in a horizontal tube extractor using cocurrent flow and varying mass velocities are given in Table XXI, page 93. The curves obtained when the overall mass transfer coefficient based on the water and the n-butanol phase were plotted against the varying mass velocities of the water phase at three constant n-butanol mass velocities are shown in Figure 19, page 94. The curves obtained when the overall mass transfer coefficients based on the water and the n-butanol phases were plotted against the varying mass velocities of the n-butanol



phase and three constant water phase flow rates are also shown in Figure 19, page 94. The curves obtained by plotting the overall mass transfer coefficient based on the water and the n-butanol phases against the ratio of the n-butanol phase to the water phase are shown in Figures 20 and 21, pages 96 and 97.

System 1,1,2-Trichloroethane-Acetone-Water. The data and results obtained in the determination of the overall mass transfer coefficient for the system 1,1,2-trichloroethane-acetone-water at 25 °C, in a horizontal tube extractor using cocurrent flow and varying mass velocities are given in Table XXIV, page 100. The curves obtained when the overall mass transfer coefficients based on the trichloroethane and the water phases were plotted against varying mass velocities of the water phase at four constant trichloroethane mass velocities are shown in Figure 22, page 99. The curves obtained when the overall mass transfer coefficient based on the trichloroethane and the water phases were plotted against varying mass velocities of the trichloroethane phase at three constant water phase mass velocities are also shown in Figure 22, page 99. The curves obtained by plotting the overall mass transfer coefficient against the ratio of the trichloroethane phase flow rates to the water phase flow rates are shown in Figures 23 and 24, pages 101 and 102.

TABLE XVI

Dipping Refractometer Calibration Data for Prism "A"  
and Mixtures of n-Butanol in Water at 25 °C

Water	n-Butanol	Scale Reading
wt %	wt %	
100.00	0.00	13.20
98.29	1.71	16.85
97.01	2.99	19.57
95.48	4.52	24.51
94.58	5.42	26.30

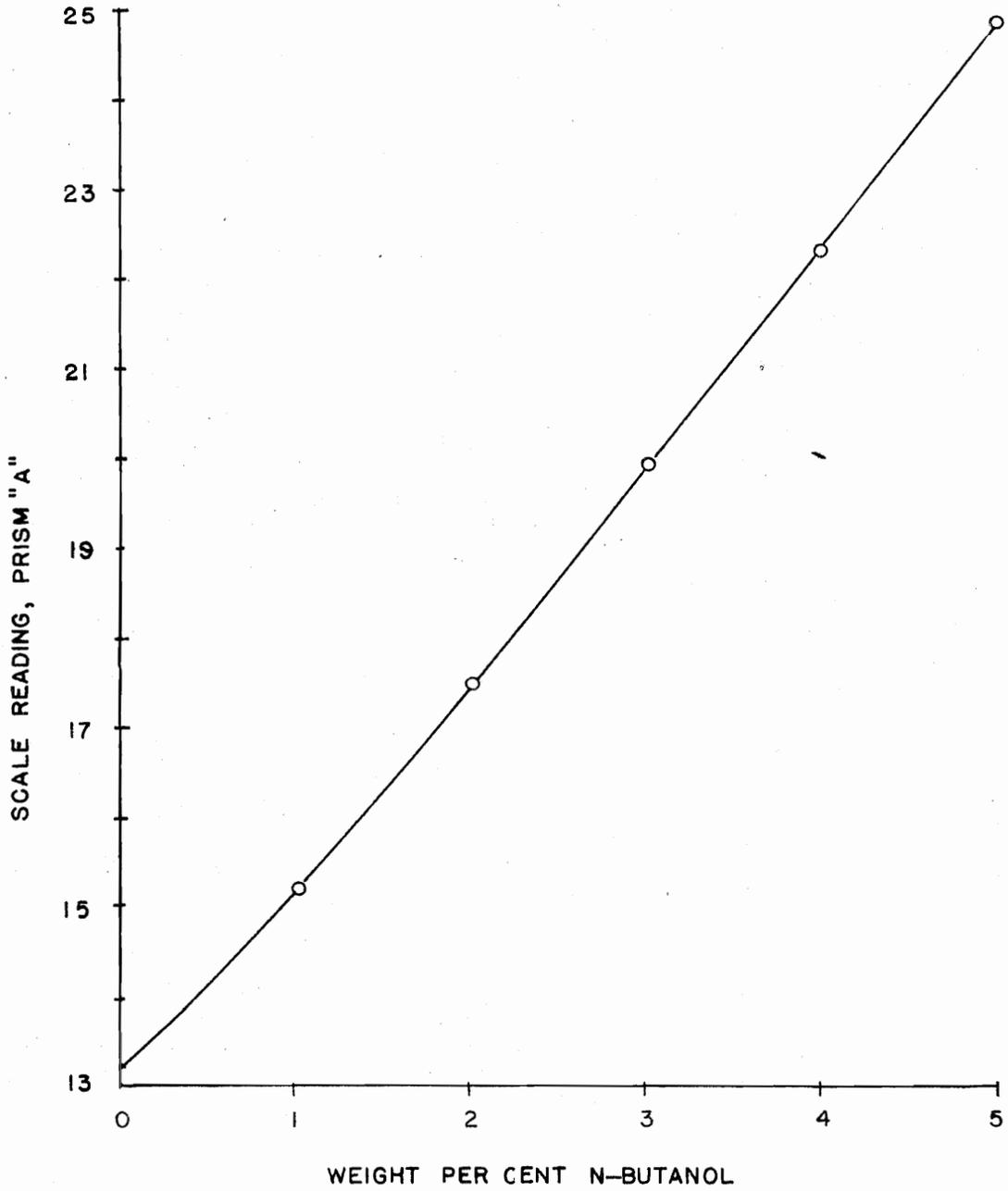


FIGURE 14. DIPPING REFRACTOMETER CALIBRATION CURVE FOR SOLUTIONS OF N BUTANOL IN WATER AT 25 °C.

TABLE XVII

Dipping Refractometer Calibration Data for Prism "B"  
and Mixtures of Water in n-Butanol at 25 °C

n-Butanol wt %	Water wt %	Scale Reading
100.00	0.00	77.43
94.98	5.02	73.20
90.98	9.02	70.65
84.01	15.99	63.31
79.82	21.18	55.53

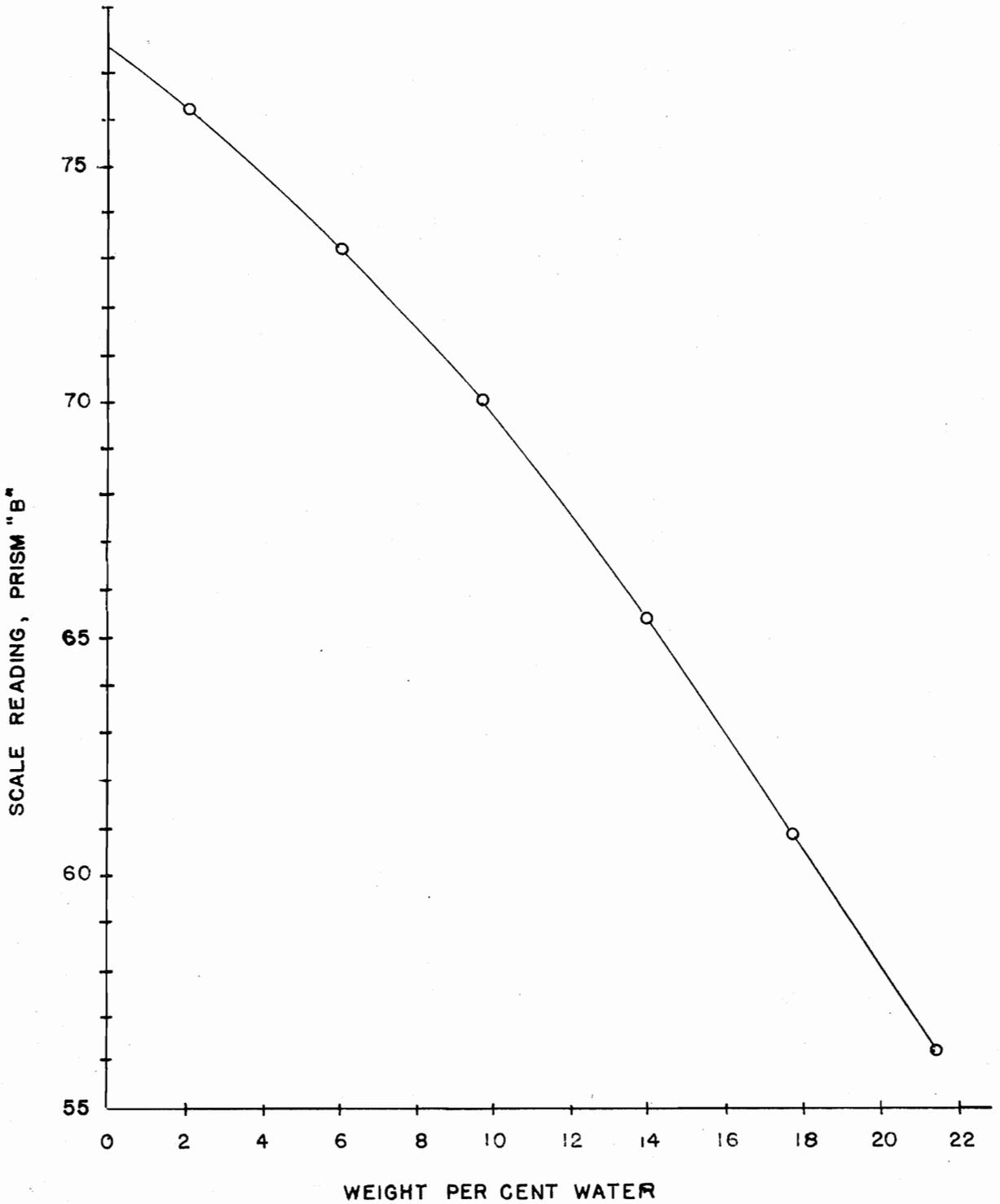


FIGURE 15, DIPPING REFRACTOMETER CALIBRATION CURVE FOR SOLUTIONS OF WATER IN N-BUTANOL AT 25 °C.

TABLE XVIII

Dipping Refractometer Calibration Data for Prism "A"  
and Mixtures of Acetone in Water Saturated with  
1,1,2-Trichloroethane at 25 °C

Water wt %	Acetone wt %	Scale Reading
100.00	0.00	14.14
99.01	0.99	15.46
98.20	1.80	16.92
97.10	2.90	19.28
96.14	3.86	21.03
95.14	4.86	23.05

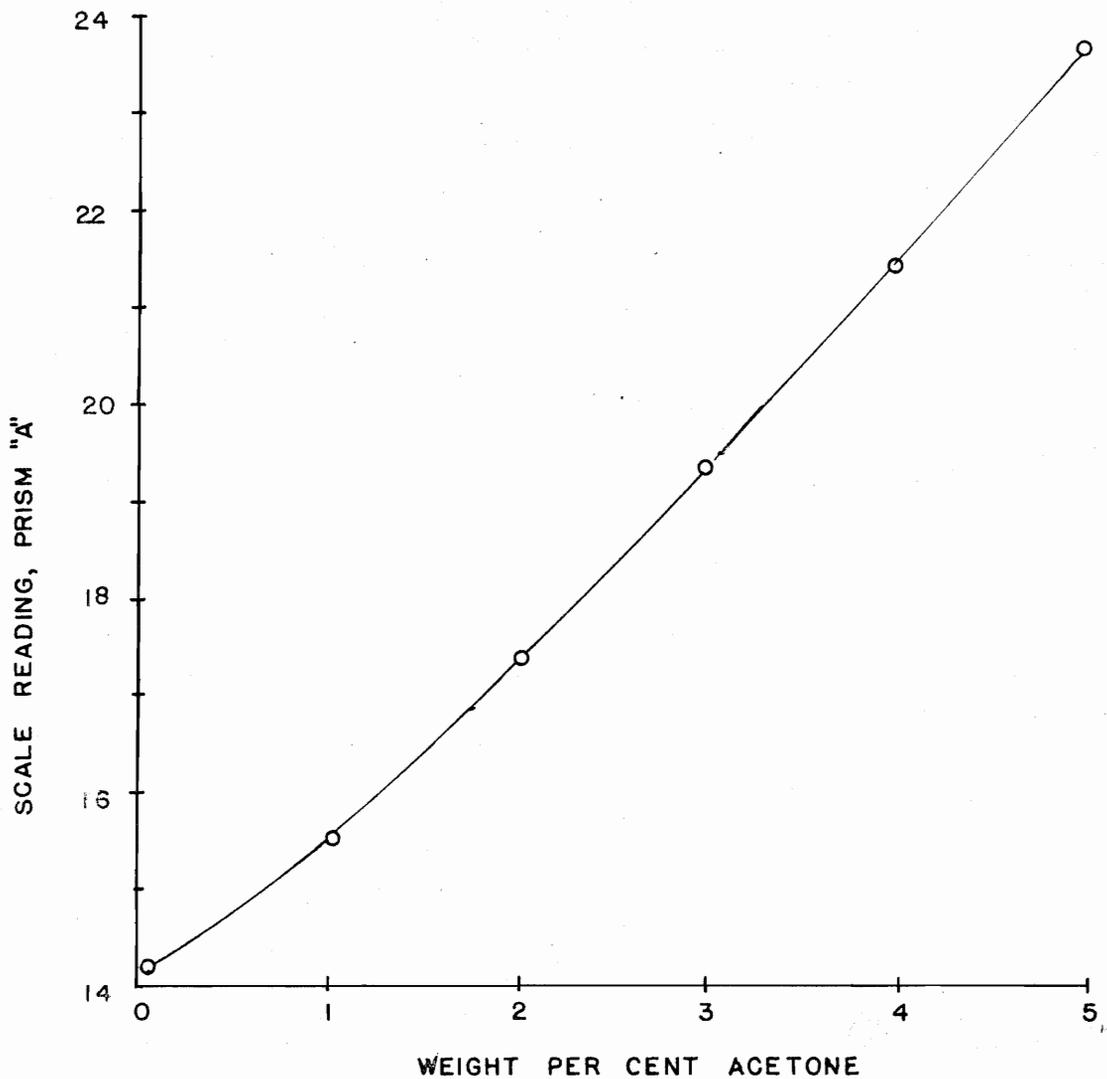


FIGURE 16. DIPPING REFRACTOMETER SCALE READING FOR WATER SATURATED WITH 1,1,2-TRICHLOROETHANE AND ACETONE AT 25 °C.

TABLE XIX

Calibration Data for the Abbe Refractometer for Use  
with Mixtures of Acetone in 1,1,2-Trichloroethane  
Saturated with Water at 25 °C

1,1,2-Trichloroethane	Acetone	Refractive Index
wt %	wt %	$n_D^{25\text{ °C}}$
100.00	0.00	1.4676
99.23	0.77	1.4662
97.71	2.29	1.4636
97.25	2.75	1.4629
95.42	4.58	1.4596
93.23	6.77	1.4555



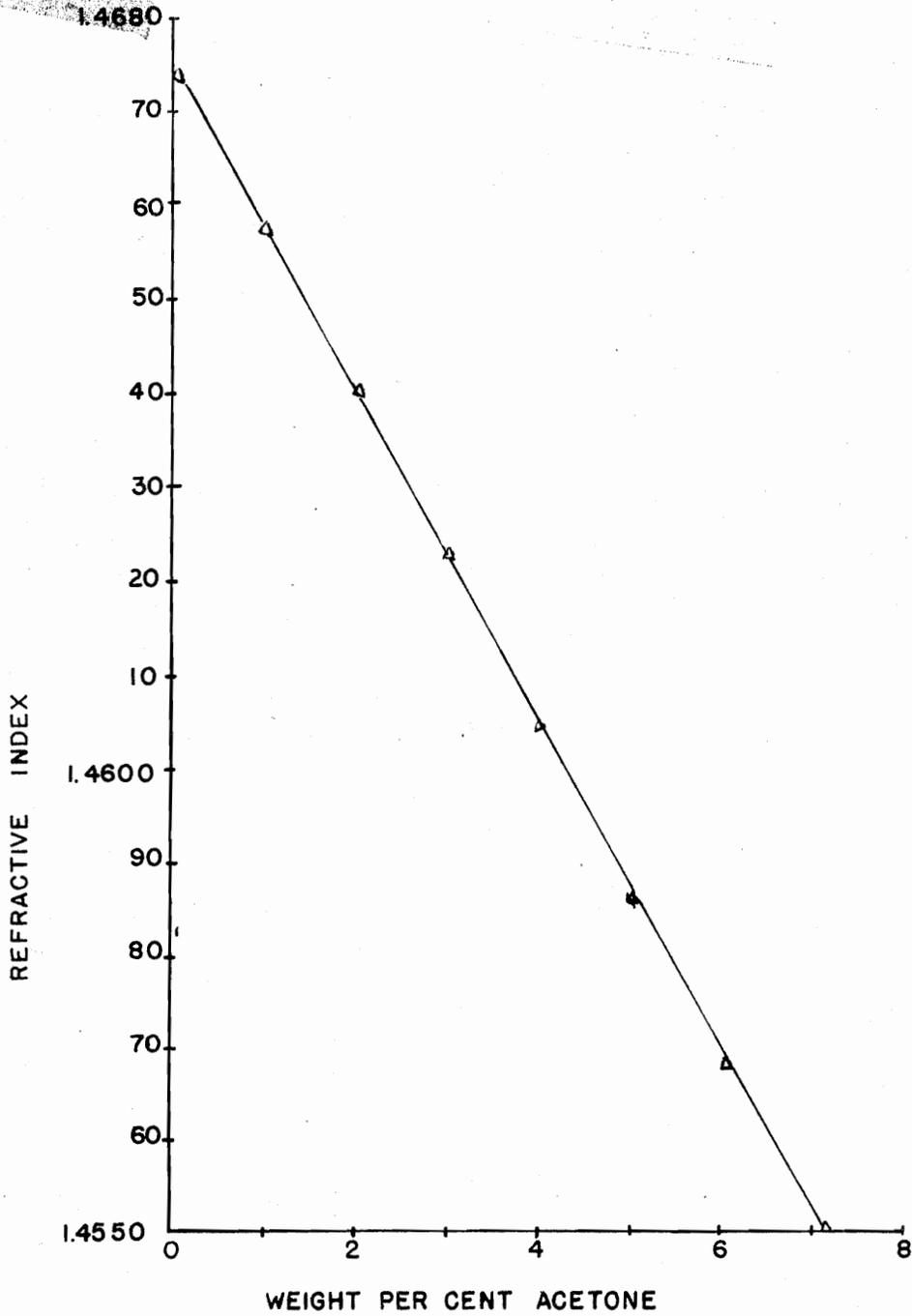


FIGURE 17. REFRACTIVE INDEX OF 1,1,2-TRICHLOROETHANE SATURATED WITH WATER AND ACETONE SOLUTIONS. AT 25 °C.

TABLE XX

Specific Gravity Determinations for Mixtures  
of n-Butanol and Water at 25 °C

Test No.	n-Butanol wt %	Water wt %	Specific Gravity
1	100.00	0.00	0.8100
2	94.98	5.02	0.8210
3	90.98	9.02	0.8253
4	84.01	15.99	0.8398
5	79.20	21.80	0.8430

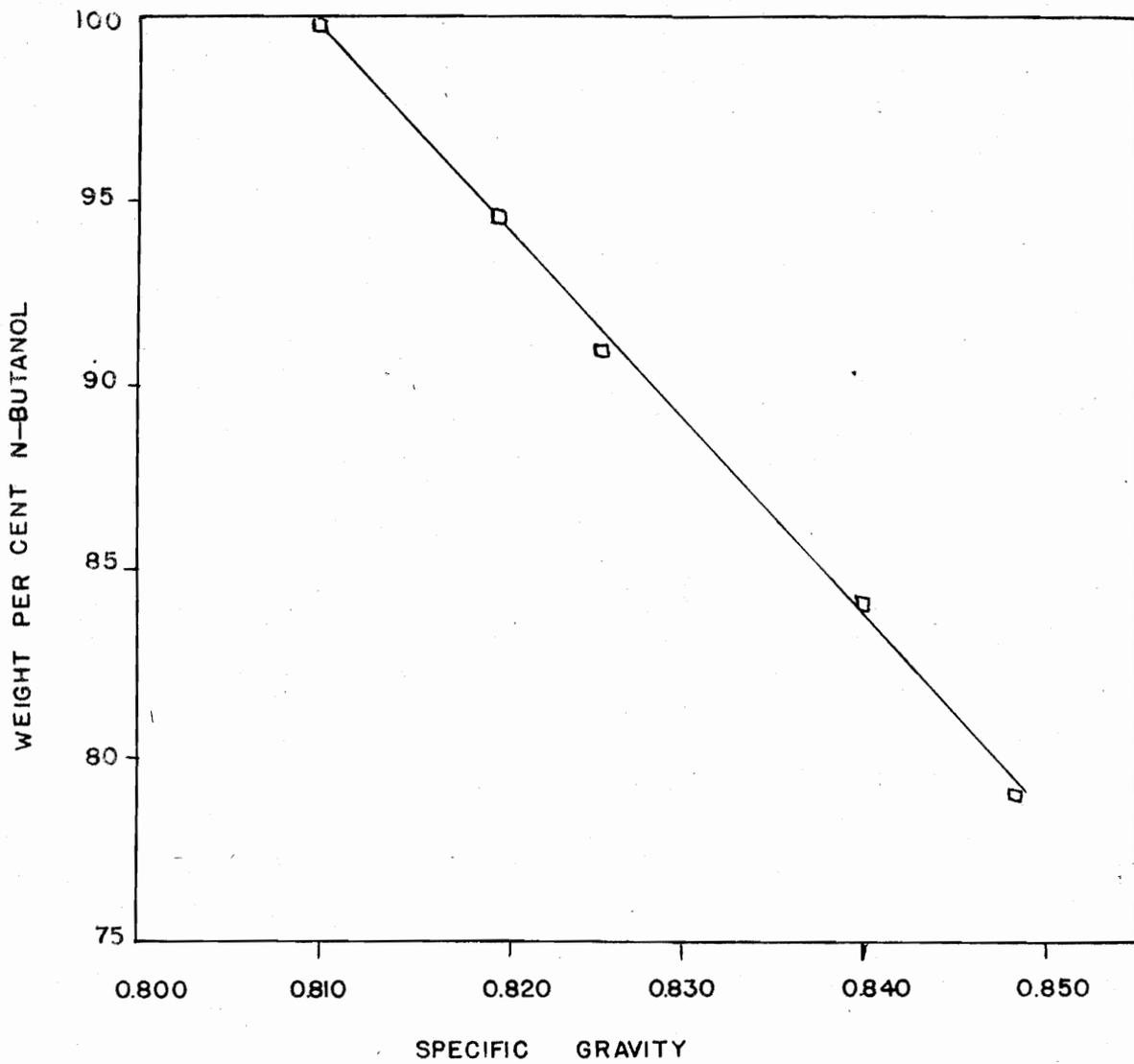


FIGURE 18. SPECIFIC GRAVITY OF N-BUTANOL AND WATER SOLUTIONS AT 25 °C.



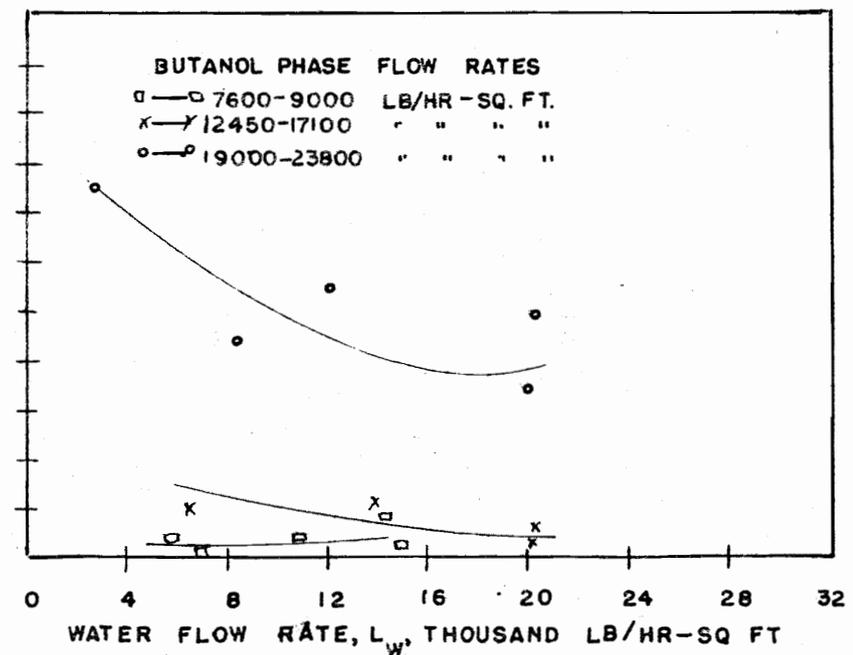
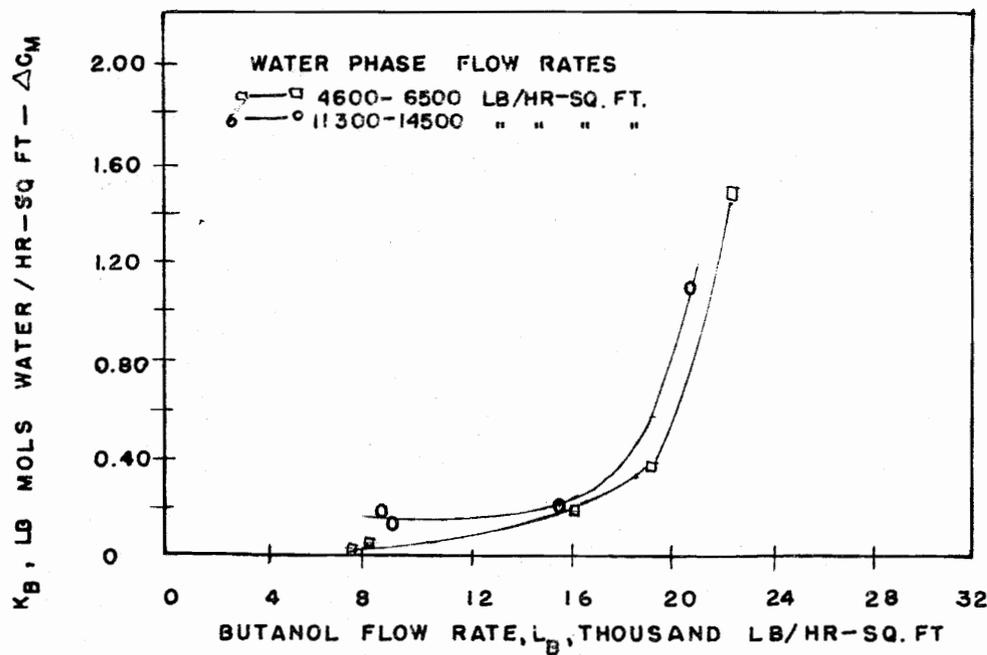
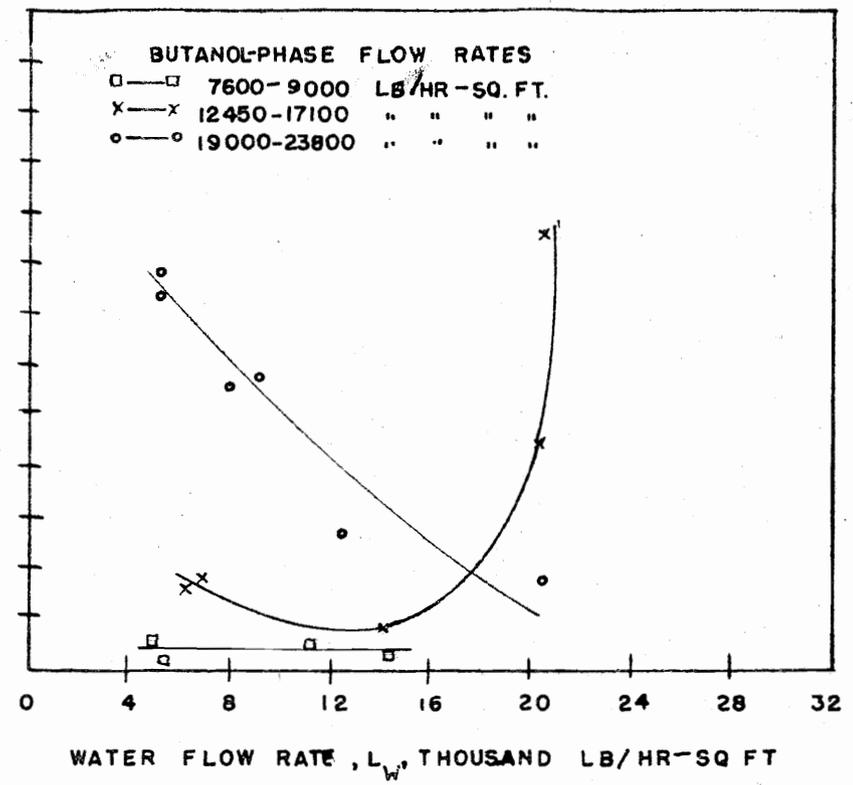
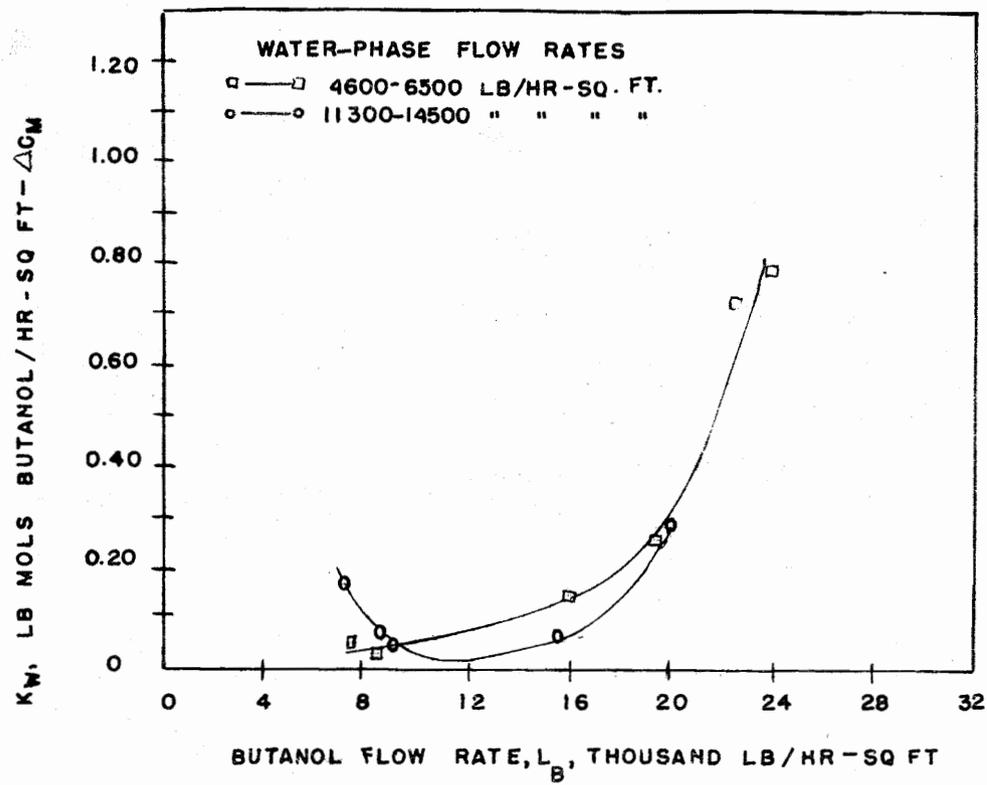


FIGURE 19, OVERALL TRANSFER COEFFICIENTS, BASED ON THE WATER FILM AND THE BUTANOL FILM, AS FUNCTIONS OF THE BUTANOL AND THE WATER PHASE FLOW RATES FOR THE SYSTEM N BUTANOL AND WATER, AT 25 °C.

TABLE XXII

Data for the Correlation of the Overall Mass Transfer  
Coefficients and the Ratio of the Phase Velocities for  
the System n-Butanol-Water at 25 °C

Test No.	Overall Mass Transfer Coefficients		Ratio of Phase Flow Rates
	$K_W$	$K_B$	$L_B/L_W$
	$\frac{\text{mols water}}{\text{hr-sq ft-}^\circ\text{C}_m}$	$\frac{\text{mols butanol}}{\text{hr-sq ft-}^\circ\text{C}_m}$	
8	0.0245	0.0428	1.36
9	0.0379	0.4544	1.70
10	0.1707	0.0041	2.40
11	0.5571	0.8397	2.33
12	0.2620	0.2785	2.96
13	0.1975	0.0709	0.54
14	0.0636	0.1017	0.79
15	0.0606	0.2378	1.11
16	0.2896	1.1282	1.64
17	0.5831	2.7405	2.01
18	0.5819	5.0056	1.90
19	0.0346	0.1892	0.61
20	0.8689	0.0057	0.61
21	0.4609	0.9187	0.81
22	0.6146	1.0008	1.01
23	0.1888	0.5566	1.13
24	0.0520	0.0153	1.30
26	0.1421	0.2261	2.47
27	0.7457	1.5692	4.81
28	0.7935	2.5765	5.16

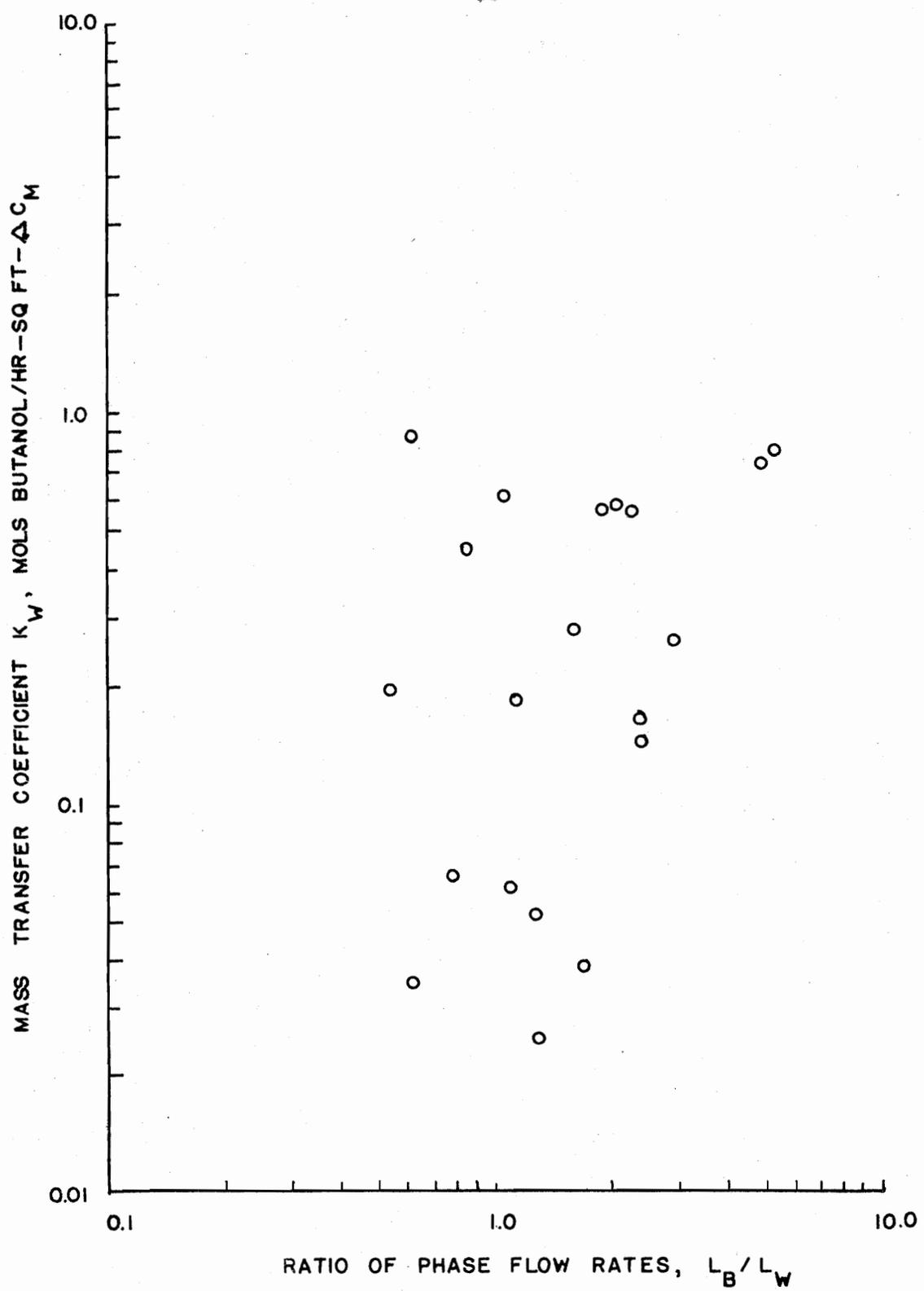


FIGURE 20, VARIATIONS OF THE MASS TRANSFER COEFFICIENT FOR BUTANOL INTO WATER WITH THE RATIO OF PHASE FLOW RATES FOR THE SYSTEM N-BUTANOL-WATER AT 25 °C.

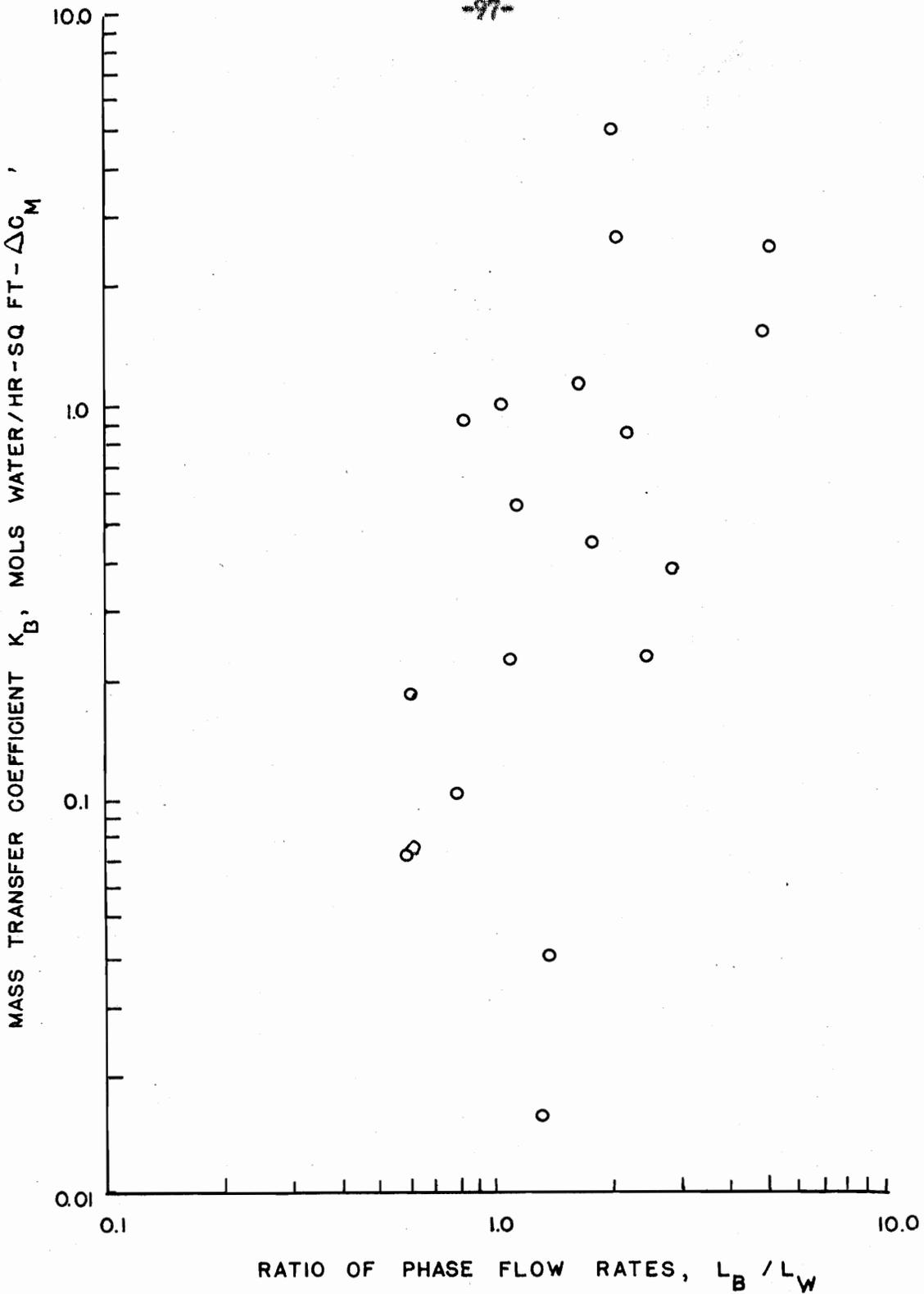


FIGURE 21 VARIATIONS OF THE MASS TRANSFER COEFFICIENT FOR WATER INTO BUTANOL WITH THE RATIO OF THE PHASE FLOW RATES FOR THE SYSTEM N-BUTANOL-WATER AT 25 °C.



TABLE XXIII

## Extraction Data and Results for the System

## 1,1,2-Trichloroethane-Acetone-Water

Area of interface = 1.00 sq ft

Cross-sectional area of each phase = 0.00612 sq ft

Test No	Time of Test min	Stream	Temp °F	Total Weight of Exit Streams gm	Exit Phase Flow Rates		Dipping Refractometer Scale Reading of Exit Water Phase	Refractive Index of the Exit T.C.E. Phase $n_D^{25^\circ C}$	Acetone in Exit Phases			Dipping Refractometer Scale Reading of Entering Water Phase	Refractive Index of the Entering T.C.E. Phase $n_D^{25^\circ C}$	Acetone in Entering Phases			Logarithmic Mean Concentration Difference		Mass Transfer Coefficient	
					lb/hr	lb/hr-sq ft			wt %	lb/hr	mol/hr			wt %	lb/hr	mol/hr	$\frac{\Delta C_{TCE}}{\ln \frac{C_{TCE} - C_{TCE}^*}{C_{TCE} - C_{TCE}^*}}$ cu ft	$\frac{\Delta C_{1,1,2}}{\ln \frac{C_{1,1,2} - C_{1,1,2}^*}{C_{1,1,2} - C_{1,1,2}^*}}$ cu ft	$\frac{K_{TCE}}{hr-sq ft \Delta C_{TCE}}$ mol	$\frac{K_{1,1,2}}{hr-sq ft \Delta C_{1,1,2}}$ mol
30	5	T.C.E. Water	74				14.83	1.4600	4.25			14.14	1.4592	4.70						
	10	T.C.E. Water	72				14.91	1.4600	4.25					0.0						
	15	T.C.E. Water		11,725 3,180	103.28 28.02	16,865 4,575	14.90	1.4599	4.30 0.58	4.399 0.156	0.0757 0.0027	14.14	1.4592	4.70 0.0	4.555 0.0	0.0784 0.0	0.0736	0.0423	0.0364	0.0633
31	4	T.C.E. Water	74				14.85	1.4600	4.25			14.14	1.4595	4.53						
	8	T.C.E. Water	72				14.88	1.4600	4.25					0.0						
	12	T.C.E. Water		9,825 2,770	108.20 30.50	17,669 4,981	14.90	1.4600	4.25 0.58	4.598 0.171	0.0792 0.0029	14.14	1.4596	4.48 0.0	4.769 0.0	0.0821 0.0	0.0633	0.0434	0.0464	0.0678
32	4	T.C.E. Water	75				14.57	1.4600	4.25			14.14	1.4599	4.30						
	8	T.C.E. Water	72				14.58	1.4602	4.15					0.0						
	12	T.C.E. Water		14,635 5,210	161.50 57.50	26,387 9,394	14.54	1.4603	4.20 0.30	6.783 0.184	0.1167 0.0032	14.14	1.4600	4.25 0.0	6.967 0.0	0.1199 0.0	0.0652	0.0443	0.0484	0.0713
33	3	T.C.E. Water	74				14.67	1.4599	4.30			14.14	1.4597	4.42						
	6	T.C.E. Water	72				14.79	1.4601	4.20					0.0						
	9	T.C.E. Water		12,725 3,680	187.31 54.01	30,605 8,825	14.75	1.4603	4.25 0.47	7.961 0.248	0.1370 0.0049	14.14	1.4599	4.30 0.0	8.209 0.0	0.1413 0.0	0.0591	0.0434	0.0720	0.0985
34	4	T.C.E. Water	74				14.41	1.4597	4.42			14.14	1.4590	4.80						
	8	T.C.E. Water	72				14.40	1.4598	4.21					0.0						
	12	T.C.E. Water		5,115 5,450	56.45 60.00	9,223 9,803	14.47	1.4598	4.36 0.26	2.473 0.132	0.0425 0.0023	14.14	1.4591	4.75 0.0	2.605 0.0	0.0448 0.0	0.0815	0.0440	0.0279	0.0516
35	3	T.C.E. Water	74				14.63	1.4593	4.64			14.14	1.4591	4.75						
	6	T.C.E. Water	72				14.57	1.4594	4.37					0.0						
	9	T.C.E. Water		6,325 3,920	92.77 57.47	15,157 9,390	14.56	1.4596	4.48 0.33	4.239 0.195	0.0729 0.0034	14.14	1.4591	4.75 0.0	4.434 0.0	0.0763 0.0	0.0734	0.0522	0.0457	0.0642
36	3	T.C.E. Water	74				14.60	1.4596	4.48			14.14	1.4592	4.70						
	6	T.C.E. Water	72				14.54	1.4594	4.35					0.0						
	9	T.C.E. Water		11,555 6,850	169.49 100.56	27,692 16,430	14.51	1.4596	4.53 0.34	7.677 0.332	0.1420 0.0057	14.14	1.4591	4.75 0.0	8.009 0.0	0.1377 0.0	0.0713	0.0435	0.0801	0.1350
37	2	T.C.E. Water	74				14.75	1.4596	4.48			14.14	1.4590	4.80						
	4	T.C.E. Water	74				14.70	1.4597	4.47					0.0						
	5.5	T.C.E. Water		7,925 4,315	190.36 103.74	31,102 16,950	14.72	1.4597	4.42 0.44	8.452 0.477	0.1455 0.0082	14.14	1.4591	4.75 0.0	8.929 0.0	0.1537 0.0	0.0707	0.0464	0.1160	0.1770
38	2	T.C.E. Water	74				14.80	1.4601	4.20			14.14	1.4600	4.25						
	4	T.C.E. Water	74				14.82	1.4600	4.50					0.0						
	6	T.C.E. Water		7,960 2,500	175.30 55.30	28,642 9,035	14.84	1.4601	4.25 0.51	7.397 0.282	0.1273 0.0049	14.14	1.4599	4.30 0.0	7.679 0.0	0.1322 0.0	0.0563	0.0409	0.0863	0.1190
39	2	T.C.E. Water	74				14.50	1.4595	4.53			14.14	1.4591	4.75						
	4	T.C.E. Water	74				14.51	1.4598	4.24					0.0						
	7	T.C.E. Water		3,175 5,840	59.91 110.31	9,788 18,023	14.55	1.4597	4.42 0.16	2.654 0.309	0.0467 0.0053	14.14	1.4593	4.64 0.0	2.963 0.0	0.0510 0.0	0.0668	0.0469	0.0791	0.1135
40	2	T.C.E. Water	74				14.65	1.4599	4.30			14.14	1.4593	4.64						
	4	T.C.E. Water	74				14.62	1.4596	4.39					0.0						
	6	T.C.E. Water		5,405 4,615	119.00 101.70	19,443 16,616	14.59	1.4598	4.48 0.37	5.212 0.366	0.0897 0.0063	14.14	1.4592	4.70 0.0	5.578 0.0	0.0960 0.0	0.0652	0.0434	0.0966	0.1455
41	2	T.C.E. Water	74				14.41	1.4602	4.15			14.14	1.4599	4.30						
	4	T.C.E. Water	74				14.65	1.4603	4.40					0.0						
	6	T.C.E. Water		6,460 11,415	142.50 251.80	23,283 41,141	14.66	1.4603	4.09 0.41	5.825 1.032	0.1013 0.0177	14.14	1.4599	4.30 0.0	6.917 0.0	0.1191 0.0	0.0613	0.0430	0.2890	0.4130
42	2	T.C.E. Water	74				14.43	1.4602	4.15			14.14	1.4600	4.25						
	4	T.C.E. Water	74				14.60	1.4603	4.35					0.0						
	6	T.C.E. Water		7,165 5,140	157.80 113.20	25,782 18,492	14.58	1.4604	4.04 0.54	6.454 0.385	0.1111 0.0066	14.14	1.4601	4.20 0.0	6.839 0.0	0.1177 0.0	0.0641	0.0435	0.1031	0.1521
43	2	T.C.E. Water	74				14.56	1.4602	4.15			14.14	1.4601	4.20						
	4	T.C.E. Water	74				14.60	1.4601	4.32					0.0						
	6	T.C.E. Water		9,015 4,645	199.00 102.30	32,514 16,714	14.62	1.4603	4.20 0.26	8.258 0.347	0.1421 0.0060	14.14	1.4601	4.20 0.0	8.605 0.0	0.1481 0.0	0.0515	0.0422	0.1160	0.1415
44	3	T.C.E. Water	74				14.66	1.4603	4.09			14.14	1.4594	4.58						
	6	T.C.E. Water	74				14.60	1.4603	4.41					0.0						
	9	T.C.E. Water		3,180 8,190	46.68 120.21	7,627 19,641	14.59	1.4603	4.09 0.34	1.909 0.444	0.0328 0.0076	14.14	1.4596	4.47 0.0	2.353 0.0	0.0404 0.0	0.0618	0.0430	0.1235	0.1775
45	3	T.C.E. Water	74				14.45	1.4595	4.30			14.14	1.4600	4.25						
	6	T.C.E. Water	74				14.46	1.4600	4.32					0.0						
	9	T.C.E. Water		6,825 8,030	100.23 117.94	16,376 19,270	14.55	1.4601	4.25 0.31	4.239 0.365	0.0730 0.0063	14.14	1.4599	4.30 0.0	4.604 0.0	0.0792 0.0	0.0669	0.0453	0.0940	0.1385
46	2	T.C.E. Water	74				14.63	1.4600	4.25			14.14	1.4598	4.36						
	4	T.C.E. Water	74				14.65	1.4601	4.37					0.0						
	6	T.C.E. Water		6,270 5,855	138.50 129.20	22,629 21,109	14.60	1.4601	4.20 0.35	5.845 0.478	0.1006 0.0082	14.14	1.4599	4.30 0.0	6.323 0.0	0.1088 0.0	0.0644	0.0450	0.1280	0.1825
47	2	T.C.E. Water	74				14.75	1.4598	6.77			14.14	1.4590	7.04						
	4	T.C.E. Water	74				14.63	1.4595	4.47					0.0						
	6	T.C.E. Water		7,920 5,610	174.90 123.60	28,576 20,395	14.70	1.4593	6.79 0.43	11.875 0.531	0.2045 0.0091	14.14	1.4591	7.00 0.0	12.406 0.0	0.2136 0.0	0.0108	0.0725	0.2450	0.1257
48	2	T.C.E. Water	74				14.75	1.4593	6.77			14.14	1.4591	7.00						
	4	T.C.E. Water	74				14.91	1.4595	6.69					0.0						
	6	T.C.E. Water		8,860 5,380	195.50 118.60	31,942 19,378	14.87	1.4597	6.72 0.63	13.137 0.906	0.2261 0.0138	14.14	1.4590	7.04 0.0	13.944 0.0	0.2399 0.0	0.0104	0.0707	1.3350	0.1965

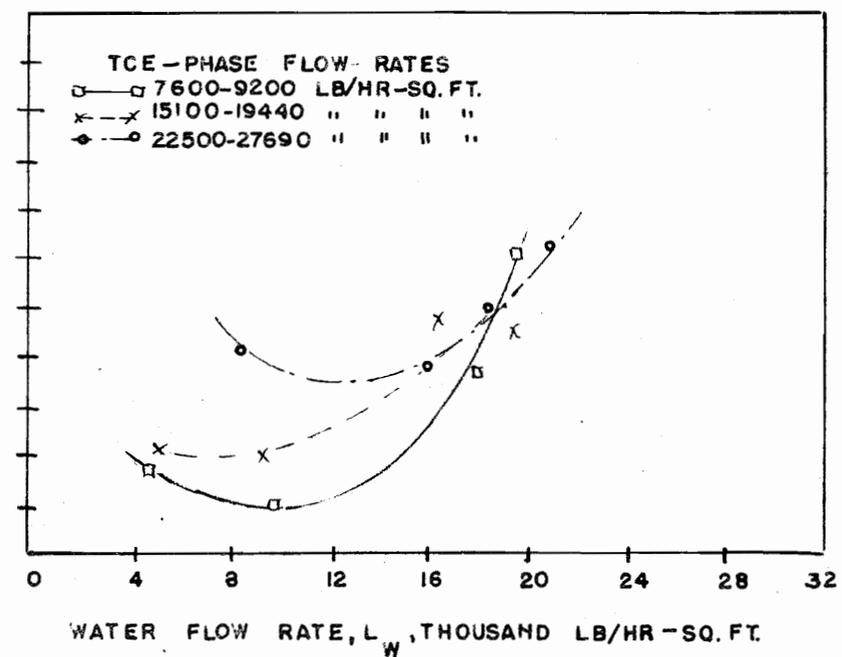
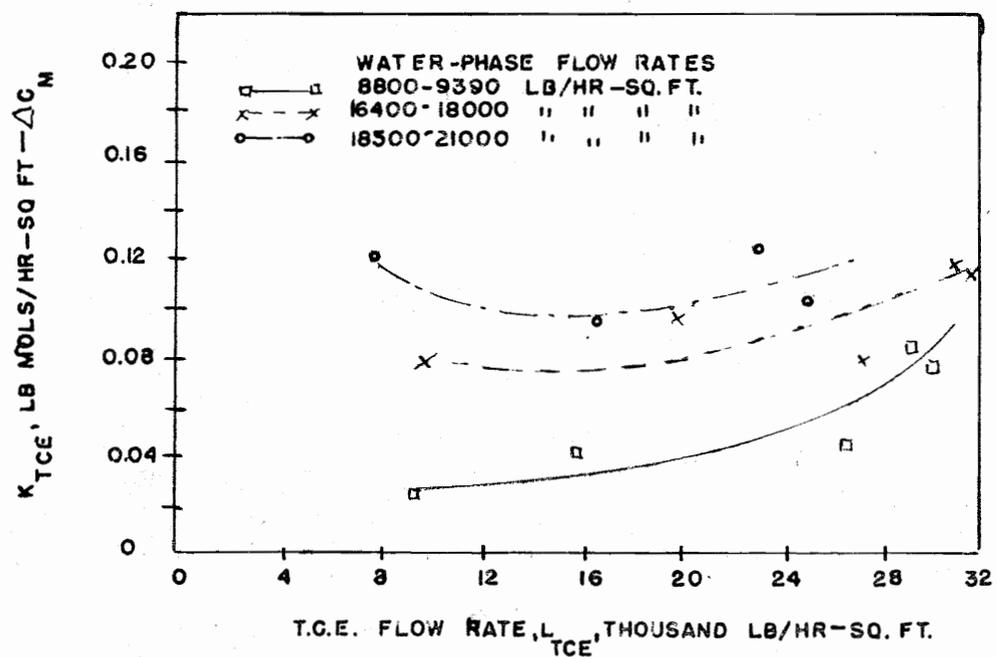
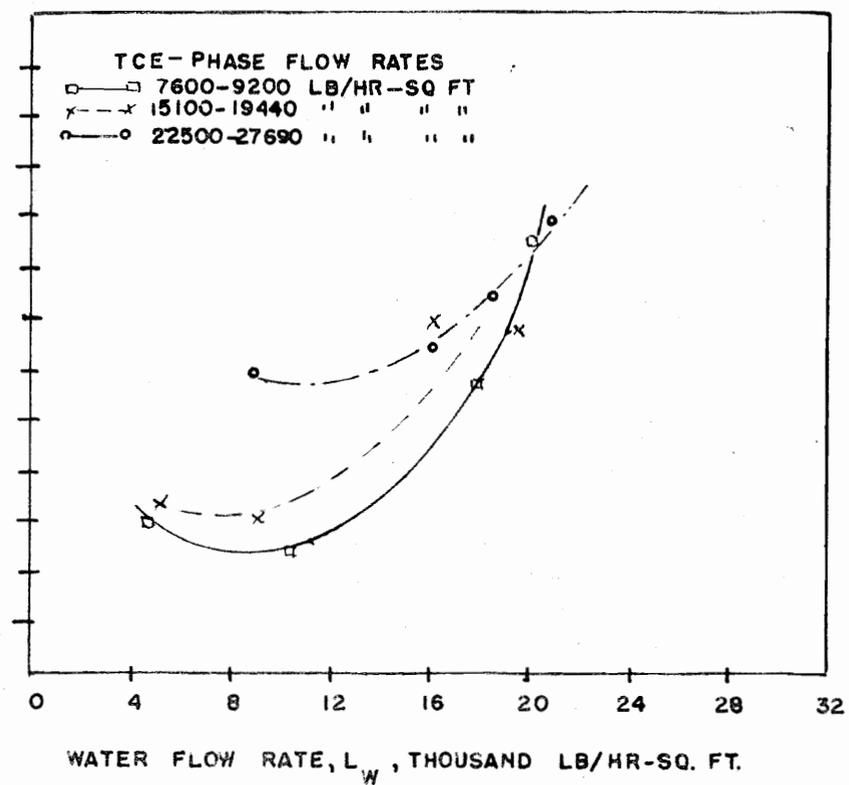
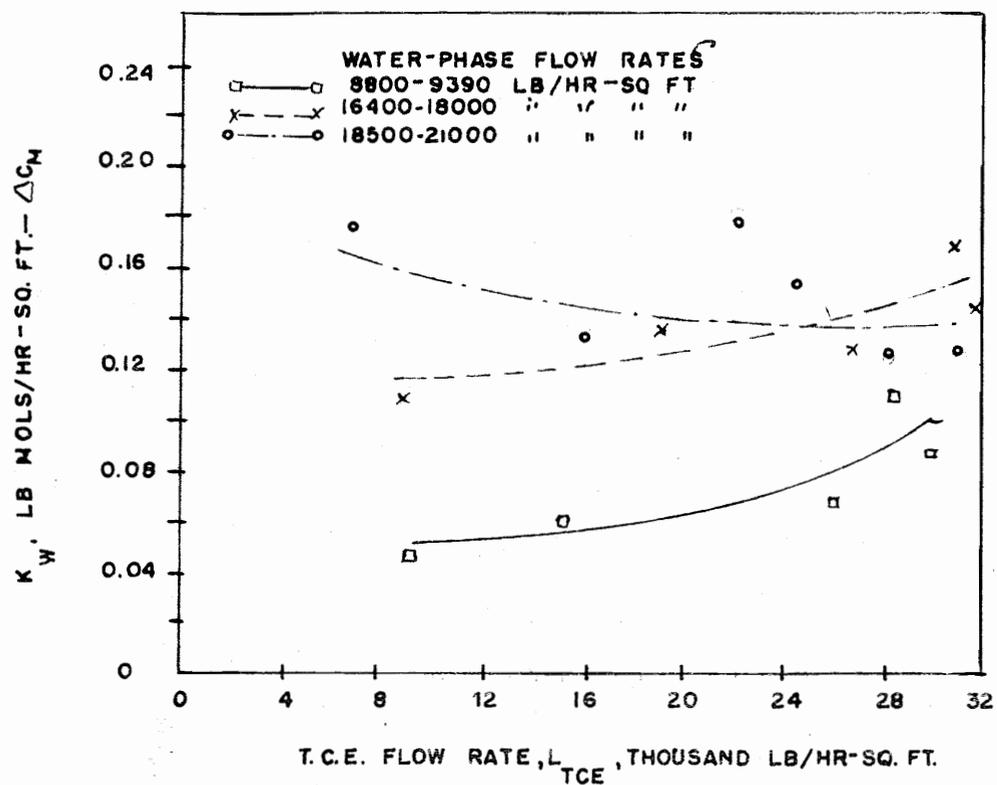


FIGURE 22. OVERALL TRANSFER COEFFICIENTS, BASED ON THE WATER FILM AND THE TRICHLOROETHANE FILM AS FUNCTIONS OF THE TRICHLOROETHANE AND THE WATER PHASE FLOW RATES FOR THE SYSTEM 1,1,2-TRICHLOROETHANE-ACETONE-WATER AT 25 °C.

TABLE XXIV

Data for the Correlation of the Overall Mass Transfer  
Coefficients and the Ratio of the Phase Velocities for  
the System 1,1,2-Trichloroethane-Acetone-Water at 25 °C

Test No.	Overall Mass Transfer Coefficients		Ratio of Phase Flow Rates
	$K_{TCE}$	$K_{H_2O}$	$L_{TCE}/L_{H_2O}$
	$\frac{\text{mols acetone}}{\text{hr-sq ft-}\Delta C_{1n}}$	$\frac{\text{mols acetone}}{\text{hr-sq ft-}\Delta C_{1n}}$	
30	0.0364	0.0633	3.68
31	0.0464	0.0678	3.54
32	0.0484	0.0713	2.80
33	0.0720	0.0983	3.46
34	0.0279	0.0516	0.94
35	0.0457	0.0642	1.61
36	0.0801	0.1350	1.68
37	0.1160	0.1770	1.83
38	0.0863	0.1190	3.16
39	0.0791	0.1135	0.54
40	0.0966	0.1455	1.17
41	0.2890	0.4130	0.56
42	0.1031	0.1521	1.39
43	0.1160	0.1415	1.94
44	0.1235	0.1775	0.38
45	0.0940	0.1385	0.84
46	0.1280	0.1825	1.07
47	0.8450	0.1275	1.41
48	1.3350	0.1965	1.64

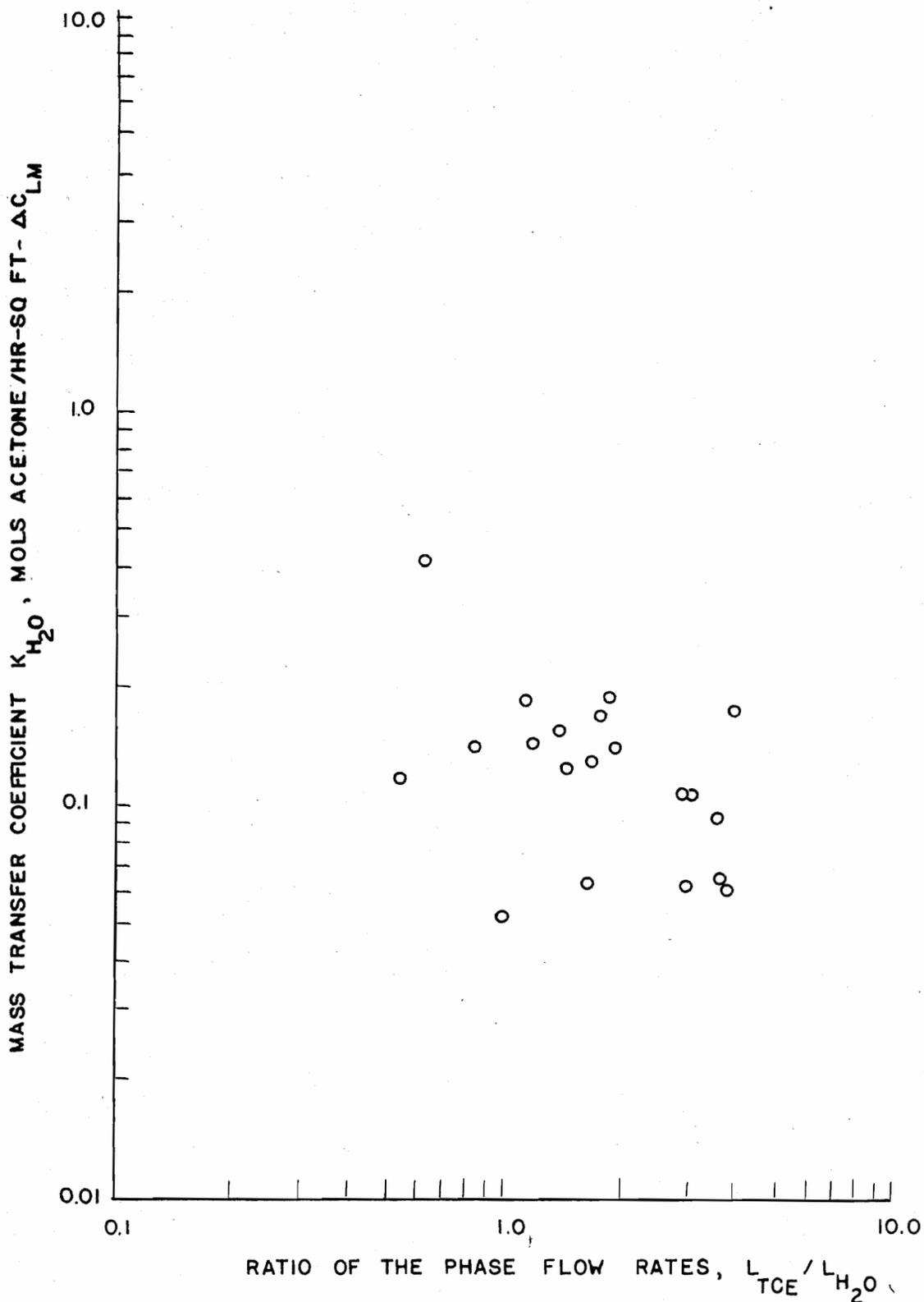


FIGURE 23. VARIATIONS OF THE MASS TRANSFER COEFFICIENT BASED ON THE WATER PHASE FOR THE TRANSFER OF ACETONE FROM 1,1,2-TRICHLOROETHANE INTO WATER AT 25 °C

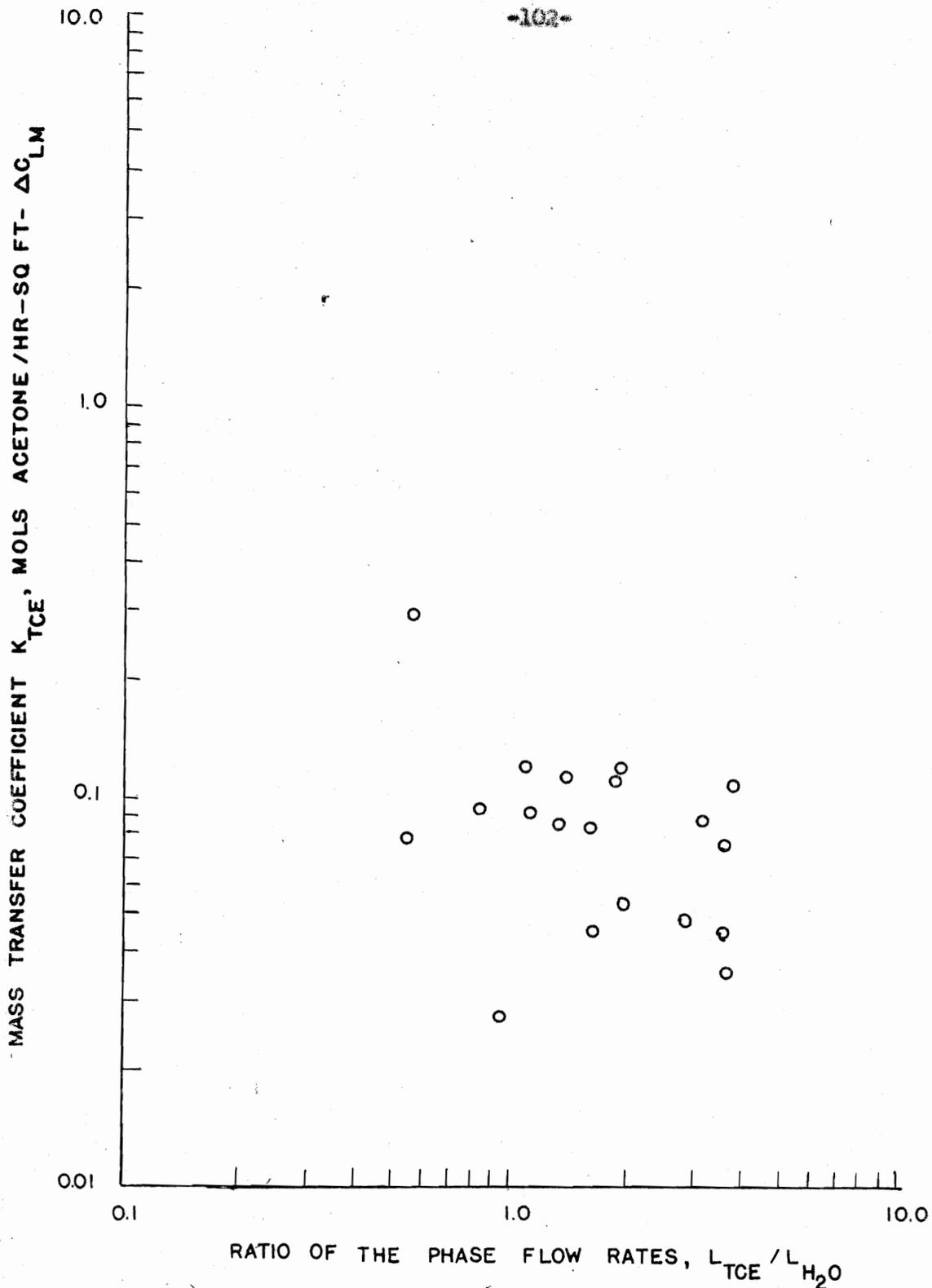


FIGURE 24, VARIATIONS OF THE MASS TRANSFER COEFFICIENT BASED ON THE TRICHLOROETHANE PHASE FOR THE TRANSFER OF ACETONE FROM 1,1,2-TRICHLOROETHANE INTO WATER AT 25 °C

Sample Calculations for the System n-Butanol-Water

The calculations for obtaining the overall mass transfer coefficient for the system n-butanol-water are given in the following paragraphs. The data used are from Test 8, Table XXI, page 93.

Determination of Exit Phase Flow Rates. The determination of the exit phase flow rates for the water and the butanol phase was accomplished in the following manner, for the n-butanol phase flow rate.

$$L_B = \frac{W_B}{454 \times A_S \times \theta} \quad (12)$$

where:

$L_B$  = butanol phase flow rate, lb/hr-sq ft

$W_B$  = weight of butanol phase leaving the extraction tube, gm

$A_S$  = cross-sectional area of the butanol phase, sq ft

$\theta$  = time of test, hr

$$L_B = \frac{6,960}{454 \times 0.00612 \times 0.30} = 8,360 \text{ lb/hr-sq ft.}$$

For the water phase flow rate,

$$L_W = \frac{W_W}{454 \times A_S \times \theta} \quad (13)$$

where:

$L_W$  = water phase flow rate, lb/hr-sq ft

$W_W$  = weight of the water phase leaving the extraction  
tube, gm

$A_S$  = cross-sectional area of the water phase, sq ft

$\theta$  = time of the test, hr

$$L_W = \frac{5,120}{454 \times 0.00612 \times 0.30} = 6,130 \text{ lb/hr-sq ft.}$$

Exit Flow Rate in Mols per Hour. The exit flow rates of the butanol and water streams were obtained in the following manner. For the butanol stream,

$$G_{BBE} = \frac{W_B \times C_{BBE}}{454 \times \theta \times M_B} \quad (14)$$

where:

$G_{BBE}$  = exit flow rate of butanol stream, lb mols/hr

$W_B$  = weight of exit butanol stream, gm

$C_{BBE}$  = weight fraction butanol in the exit butanol stream, wt %

$\theta$  = time of test, hr

$M_B$  = molecular weight of n-butanol

$$G_{BBE} = \frac{6,960 \times 0.842}{454 \times 0.30 \times 74} = 0.5801 \text{ lb mols/hr.}$$



The weight of water in the exit butanol stream was determined by the equation,

$$G_{WBE} = \frac{W_B \times C_{WBE}}{454 \times \theta \times M_W} \quad (15)$$

where:

$G_{WBE}$  = exit flow rate of water in the butanol stream,  
lb mols/hr

$W_B$  = weight of butanol phase, gm

$C_{WBE}$  = weight fraction of water in the exit butanol  
stream, wt %

$\theta$  = time of test, hr

$M_W$  = molecular weight of water

$$G_{WBE} = \frac{6,960 \times 0.1580}{454 \times 0.30 \times 18} = 0.4482 \text{ lb mols/hr.}$$

For the water stream, the weight of butanol in the stream was found by the following equation,

$$G_{BWE} = \frac{W_W \times C_{BWE}}{454 \times \theta \times M_B} \quad (16)$$

where:

$G_{BWE}$  = exit flow rate of butanol in the water stream,  
lb mols/hr

$W_W$  = weight of the exit water stream, gm

$C_{BWE}$  = weight fraction of butanol in the exit water  
stream, wt %

$\theta$  = time of test, hr

$M_B$  = molecular weight of n-butanol

$$G_{BWE} = \frac{5,120 \times 0.0020}{454 \times 0.30 \times 74} = 0.00148 \text{ lb mols/hr.}$$

The water flow rate for water in the exit water phase was found by the following equation:

$$G_{WWE} = \frac{W_W \times C_{WWE}}{454 \times \theta \times M_W} \quad (17)$$

where:

$G_{WWE}$  = exit flow rate of water in the water stream,  
lb moles/hr

$W_W$  = weight of exit water phase, gm

$C_{WWE}$  = weight fraction of water in the exit water  
stream, wt %

$\theta$  = time of test, hr

$M_W$  = molecular weight of water

$$G_{WWE} = \frac{6,130 \times 0.997}{454 \times 0.30 \times 18} = 2.0774 \text{ lb moles/hr.}$$

Entering Phase Flow Rates. Calculations for determining the phase flow rates for the water and the butanol stream entering were calculated as follows.

The weight of butanol in the entering butanol stream was determined by the following equation,

$$G_{BBI} = G_{BBE} + G_{BWE} \quad (18)$$

where:

$G_{BBI}$  = butanol in the butanol stream entering,  
lb mols/hr

$G_{BBE}$  = butanol in the exit butanol stream,  
lb mols/hr

$G_{BWE}$  = butanol in the exit water stream, lb mols/hr

$$G_{BBI} = 0.5801 + 0.00148 = 0.58158 \text{ lb mols/hr.}$$

The weight of water in the entering butanol stream was determined by the following equation:

$$G_{\text{WBI}} = \frac{\frac{G_{\text{BBI}} \times M_{\text{B}}}{C_{\text{BBI}}} - G_{\text{BBI}} \times M_{\text{B}}}{M_{\text{W}}} \quad (19)$$

where:

- $G_{\text{WBI}}$  = water in the entering butanol stream, lb mols/hr
- $G_{\text{BBI}}$  = butanol in the entering butanol stream, lb mols/hr
- $M_{\text{B}}$  = molecular weight of n-butanol
- $M_{\text{W}}$  = molecular weight of water
- $C_{\text{BBI}}$  = weight fraction of butanol in the entering butanol stream, wt %

$$G_{\text{WBI}} = \frac{\frac{0.58158 \times 74.12}{0.8440} - 0.58158 \times 74.12}{18}$$

$$= 0.4425 \text{ lb mols/hr.}$$

The entering water stream did not contain any butanol. The phase flow rate for the water entering was determined by the following equation,

$$G_{WVI} = G_{WVE} + G_{WBE} - G_{WBI} \quad (20)$$

where:

$G_{WVI}$  = water in the entering water stream, lb mols/hr

$G_{WVE}$  = water in the exit water stream, lb mols/hr

$G_{WBE}$  = water in the exit butanol stream, lb mols/hr

$G_{WBI}$  = water in the entering butanol stream, lb  
mols/hr

$$\begin{aligned} G_{WVI} &= 2.0774 + 0.4432 - 0.4425 \\ &= 2.0831 \text{ lb mols/hr.} \end{aligned}$$

Material Transferred. The amount of water transferred to the butanol stream and the amount of butanol transferred to the water stream, was determined in the following manner. The water transferred to the butanol stream was calculated by the following equation,

$$(N/\theta)_w = G_{WBE} - G_{WBI} \quad (21)$$

where:

$(N/\theta)_w$  = water transferred to the butanol stream,  
lb mols/hr

$G_{WBE}$  = water in the exit butanol stream, lb mols/hr

$G_{WBI}$  = water in the entering butanol stream, lb  
mols/hr

$$(N/\theta)_w = 0.4482 - 0.4425 = 0.0057 \text{ lb mols/hr.}$$

The butanol transferred to the water stream is equal to the amount of butanol leaving the water stream since the entering water contained no butanol.

$$(N/\theta)_b = G_{BWE} = 0.00148 \text{ lb mols/hr.}$$

Arithmetic Mean Concentration Difference. The arithmetic mean concentration difference for the butanol in the water stream was found by use of the following equation.

$$\Delta C_m = \frac{\Delta C_1 - \Delta C_2}{2} \quad (22)$$

where:

$C_1$  = difference between the amount of butanol in water and the amount of butanol required for saturation under the same conditions at the entering end of the tube, weight per cent

$C_2$  = difference between the amount of butanol in the water and the amount of butanol required for the saturation under the same conditions at the leaving end of the tube, weight per cent

$C_m$  = arithmetic mean concentration difference for butanol in the water stream, weight per cent.



The calculations used in determining the value of  $\Delta C_1$  is shown as follows. The same procedure was also used in determining the value of  $\Delta C_2$ .

$$\Delta C_1 = C^* - C_1 \quad (23)$$

where:

$\Delta C_1$  = the difference in the amount of butanol in the water and the amount of butanol required for saturation under the same conditions at the entering end of the tube, wt %

$C^*$  = concentration of butanol in water at saturated conditions, wt % (page 48)

$C_1$  = concentration of butanol in water at entering end of tube, wt %

$$C_1 = 7.35 - 0 = 7.35 \text{ weight per cent.}$$

The same method was used in determining the value for  $\Delta C_2$ , the difference being that the concentration of butanol in the water at the exit end of the tube was used in the calculations,

$$\begin{aligned}\Delta C_2 &= C^* - C_2 && (24) \\ &= 7.35 - 0.28 \\ &= 7.07 \text{ weight per cent}\end{aligned}$$

The arithmetic mean concentration difference was then calculated by the equation previously given.

$$\Delta C_m = \frac{7.35 + 7.07}{2} = 7.21 \text{ weight per cent.} \quad (25)$$

The arithmetic mean concentration difference was then changed to the units of mols per cubic foot for use in the equation for determining the overall mass transfer coefficient based on the water film, by the following equation,

$$\Delta C_{mb} = \Delta C_m \times w / M_B \quad (26)$$

where:

$\Delta C_{mb}$  = arithmetic mean concentration difference for butanol in the water stream, lb mols/cu ft

$\Delta C_m$  = arithmetic mean concentration difference for butanol in water, wt %

$w$  = the average density of the water stream, lb/cu ft

$M_B$  = molecular weight of butanol

$$\begin{aligned} C_{mb} &= 0.0721 \times 62.20 / 74.12 \\ &= 0.06049 \text{ lb mols/cu ft.} \end{aligned}$$

The determination of the arithmetic mean concentration difference for the water in the butanol stream was carried out in a similar manner as that for butanol in the water stream. The value for the saturation concentration of butanol in water was found in Table V, page 48. The equations used to determine the value of the arithmetic mean concentration difference for water in butanol are as follows:

$$\Delta C_1 = 20.27 - 15.60 = 4.67 \text{ weight per cent}$$

$$\Delta C_2 = 20.27 - 15.80 = 4.47 \text{ weight per cent}$$

$$\Delta C_m = \frac{4.47 + 4.67}{2} = 4.57 \text{ weight per cent.}$$

The arithmetic mean concentration difference in weight per cent was then converted to pound mols per cubic foot by the following equation,

$$\Delta C_{MW} = \Delta C_M \times \rho / M_W \quad (27)$$

where:

$\Delta C_{MW}$  = arithmetic mean concentration difference for water in the butanol stream, lb mols/cu ft

$\Delta C_M$  = arithmetic mean concentration difference for water in the butanol stream, wt %

$\rho$  = average density of the butanol stream, lb/cu ft

$M_W$  = molecular weight of water

$$\begin{aligned} C_{MW} &= 0.0457 \times 52.41/18 \\ &= 0.1331 \text{ lb mols/cu ft.} \end{aligned}$$

Calculation of the Overall Mass Transfer Coefficient. The overall mass transfer coefficient based on the water film was determined by use of the following equation:

$$K_w = \frac{(N/\theta)_B}{A \times \Delta C_{mb}} \quad (28)$$

where:

$K_w$  = overall mass transfer coefficient based on the water film, mols butanol/hr-sq ft-concentration difference

$A$  = area of the interface between the two phases, sq ft

$\Delta C_{mb}$  = the arithmetic mean concentration difference for butanol in the water phase, lb mols butanol/cu ft

$(N/\theta)_B$  = rate of transfer of butanol to water, lb mols/hr

$$K_w = \frac{0.00148}{1.0 \times 0.06049}$$

= 0.0245 mols butanol/hr-sq ft-concentration difference.

The overall mass transfer coefficient based on the butanol film was determined by use of the following equation:

$$K_b = \frac{(N/\theta)_w}{A \times C_{mw}} \quad (29)$$

where:

$K_b$  = overall mass transfer coefficient based on the butanol film, lb mols water/hr-sq ft concentration difference

$(N/\theta)_w$  = rate of transfer of water into the butanol stream, mol water/hr

$A$  = area of the interface between the two phases, sq ft

$C_{mw}$  = the arithmetic mean concentration difference for water in the butanol phase, lb mols water/cu ft

$$K_b = \frac{0.0057}{1.0 \times 0.1331}$$

= 0.0428 lb mols water/hr-sq ft-concentration difference.

Sample Calculations for the System

1,1,2-Trichloroethane-Acetone-Water

The calculations for obtaining the overall mass transfer coefficient for the transfer of acetone from 1,1,2-trichloroethane into water are presented in the following section. The data used in the calculations are from Test 30, Table XXIII, page 98.

Phase Flow Rates. The flow rate for the trichloroethane was determined by use of the following equation:

$$L_{TCE} = \frac{W_{TCE}}{454 \times A_s \times \theta} \quad (30)$$

where:

$L_{TCE}$  = flow rate for trichloroethane phases

$W_{TCE}$  = weight of the trichloroethane phase, gm

$A_s$  = cross-sectional area of the trichloroethane phase, sq ft

$\theta$  = time of test, hr

$$\begin{aligned} L_{TCE} &= \frac{11,725}{454 \times 0.00612 \times 0.25} \\ &= 16,863 \text{ lb/hr-sq ft.} \end{aligned}$$



The water phase flow rate was determined by use of the following equation,

$$L_{H_2O} = \frac{W_{H_2O}}{454 \times A_s \times \theta} \quad (31)$$

where:

$L_{H_2O}$  = flow rate for the water phase, lb/hr-sq ft

$W_{H_2O}$  = weight of the water phase, gm

$A_s$  = cross-sectional area of the water phase,  
sq ft

$\theta$  = time of test, hr

$$L_{H_2O} = \frac{3,170}{454 \times 0.00612 \times 0.25}$$

$$= 4,578 \text{ lb/hr-sq ft.}$$

Acetone in Exit Streams. The weight of acetone in the trichloroethane stream was found by using the following equation:

$$G_{ATCE} = \frac{W_{TCE} \times C_{ATCE}}{454 \times \theta \times M_A} \quad (32)$$

where:

$G_{ATCE}$  = acetone in the exit trichloroethane stream,  
lb mols/hr

$W_{TCE}$  = weight of the trichloroethane stream, gm

$C_{ATCE}$  = concentration of acetone in the exit  
trichloroethane stream, wt %

$\theta$  = time of test, hr

$M_A$  = molecular weight of acetone

$$\begin{aligned} G_{ATCE} &= \frac{11,725 \times 0.0427}{454 \times 0.25 \times 58.08} \\ &= 0.0757 \text{ lb mols/hr.} \end{aligned}$$

The weight of the acetone in the water stream was found by using the following equation:

$$G_{\text{AH}_2\text{O}} = \frac{W_{\text{H}_2\text{O}} \times C_{\text{AH}_2\text{O}}}{454 \times \theta \times M_A} \quad (33)$$

where:

$G_{\text{AH}_2\text{O}}$  = acetone in the exit water stream, lb mols/hr

$W_{\text{H}_2\text{O}}$  = weight of the water stream, gm

$C_{\text{AH}_2\text{O}}$  = concentration of acetone in the water stream,  
wt %

$\theta$  = time of test, hr

$M_A$  = molecular weight of acetone

$$\begin{aligned} G_{\text{AH}_2\text{O}} &= \frac{3,180 \times 0.0058}{454 \times 0.25 \times 58.08} \\ &= 0.0027 \text{ lb mols/hr.} \end{aligned}$$

Acetone in the Entering Phases. There was no acetone in the entering water phase; however, the amount of acetone in the entering trichloroethane phase was determined by the following equation,

$$G_{AITCE} = G_{ATCE} + G_{AH_2O} \quad (34)$$

where:

$G_{AITCE}$  = acetone in the entering trichloroethane phase,  
lb mols/hr

$G_{ATCE}$  = acetone in the exit trichloroethane phase,  
lb mols/hr

$G_{AH_2O}$  = acetone in the exit water phase, lb mols/hr

$$\begin{aligned} G_{AITCE} &= 0.0757 + 0.0027 \\ &= 0.0784 \text{ lb mols/hr.} \end{aligned}$$

Acetone Transferred. The acetone transferred from the trichloroethane phase into the water phase was the amount of acetone in the exit water phase. Therefore, the mols of solute transferred equals the mols of acetone in the exit water stream.

Concentration of Acetone in the Streams. The concentration of the acetone in the four streams were converted from weight per cent to pound mols per cubic foot by using the following relationship. For acetone in the exit water stream:

$$C_{\text{AH}_2\text{O}} = \frac{C_{\text{ATCE}} \times w}{58.08} \quad (35)$$

where:

$C_{\text{AH}_2\text{O}}$  = concentration of acetone in the exit water stream, lb mols/cu ft

$C_{\text{ATCE}}$  = concentration of acetone in the exit water stream, wt %

$w$  = average density of the exit water stream, lb/cu ft

$$\begin{aligned} C_{\text{AH}_2\text{O}} &= \frac{0.0058 \times 62.25}{58.08} \\ &= 0.00599 \text{ lb mols/cu ft.} \end{aligned}$$

Logarithmic Mean Concentration Difference. The logarithmic mean concentration differences based on the water and the trichloroethane phases were calculated by using the following equations:

$$\Delta C_{\ln H_2O} = \frac{(C_{AIH_2O}^* - C_{AIH_2O}) - (C_{AH_2O}^* - C_{AH_2O})}{\frac{\ln (C_{AIH_2O}^* - C_{AIH_2O})}{(C_{AH_2O}^* - C_{AH_2O})}} \quad (36)$$

$$= \frac{(C_{ATCE/H} - C_{AIH_2O}) - (C_{AITCE/H} - C_{AH_2O})}{\frac{\ln (C_{ATCE/H} - C_{AIH_2O})}{(C_{AITCE/H} - C_{AH_2O})}} \quad (37)$$

$$\Delta C_{\ln TCE} = \frac{(C_{AITCE} - C_{AITCE}^*) - (C_{ATCE} - C_{ATCE}^*)}{\frac{\ln (C_{AITCE} - C_{AITCE}^*)}{(C_{ATCE} - C_{ATCE}^*)}} \quad (38)$$

$$= \frac{(C_{AITCE} - HC_{AH_2O}) - (C_{ATCE} - HC_{AIH_2O})}{\frac{\ln (C_{AITCE} - HC_{AH_2O})}{(C_{ATCE} - HC_{AIH_2O})}} \quad (39)$$

where:

$C_{AIH_2O}^*$  = concentration of acetone in the entering water phase that would be in equilibrium with the concentration of acetone in the entering trichloroethane phase, lb mols/cu ft

- $C_{AIH_2O}$  = concentration of acetone in the entering water phase, lb mols/cu ft
- $C_{AH_2O}$  = concentration of acetone in the leaving water phase that would be in equilibrium with the concentration of acetone in the leaving trichloroethane phase, lb mols/cu ft
- $C_{AITCE}$  = concentration of acetone in the entering trichloroethane phase, lb mols/cu ft
- $C_{ATCE}$  = concentration of acetone in the exit trichloroethane phase, lb mols/cu ft
- $C_{AITCE}^*$  = concentration of acetone in the entering trichloroethane phase that would be in equilibrium with the acetone in the entering water phase, lb mols/hr
- $C_{ATCE}^*$  = concentration of acetone in the exit trichloroethane phase that would be in equilibrium with the concentration of acetone in the exit water phase, lb mols/cu ft
- $C_{H_2O}$  = concentration of acetone in the exit water phase, lb mols/cu ft
- $H$  = distribution coefficient of acetone in water and trichloroethane =  $\frac{\text{conc. acetone in trichloroethane}}{\text{conc. acetone in water}}$
- $\Delta C_{lnH_2O}$  =  $\frac{(0.0647/1.48 - 0) - (0.0714/1.48 - 0.00599)}{2.3 \log \frac{(0.0657/1.48)}{(0.0714/1.48 - 0.00599)}}$
- = 0.0423 lb mols/cu ft.
- $\Delta C_{lnTCE}$  =  $\frac{(0.0714 - 1.48 \times 0.00599) - (0.0647 - 1.48 \times 0)}{2.3 \log \frac{(0.0714 - 1.48 \times 0.00599)}{(0.0647 - 1.48 \times 0)}}$
- = 0.0736 lb mols/cu ft.

Overall Mass Transfer Coefficients. The overall mass transfer coefficients based on the water and on the trichloroethane phases were calculated by using the following equations:

$$K_{H_2O} = \frac{N/\theta}{A \times \Delta C_{ln H_2O}} \quad (40)$$

$$K_{TCE} = \frac{N/\theta}{A \times \Delta C_{ln TCE}} \quad (41)$$

where:

$K_{H_2O}$  = overall mass transfer coefficient based on the water phase, lb mols/hr-sq ft-concentration difference

$K_{TCE}$  = overall mass transfer coefficient based on the trichloroethane phase, lb mols/hr-sq ft-concentration difference

$N$  = mols of acetone transferred, lb mols

$\theta$  = time of test, hr

$\Delta C_{ln H_2O}$  = log mean concentration difference based on the water phase

$\Delta C_{ln TCE}$  = log mean concentration difference based on the trichloroethane phase

$$K_{H_2O} = \frac{0.0027}{1.0 \times 0.0423} = 0.0633 \text{ lb mols/hr-sq ft-concentration difference}$$

$$K_{TCE} = \frac{0.0027}{1.0 \times 0.0736} = 0.0364 \text{ lb mols/hr-sq ft-concentration difference.}$$



#### IV. DISCUSSION

This section contains a discussion of the experimental procedure and the results obtained. The recommendations for future work and the limitations imposed on this particular investigation are also presented in this section.

##### Discussion of Experimental Procedure

The discussion of certain points on the equipment, operating procedure, and analytical procedures follows.

Design of Equipment. The basic equipment such as the extraction tube, storage tanks, and air supply equipment, was the same equipment used by Bowman<sup>(3)</sup>. The changes in the equipment that were made for this investigation involved the replacement of all rubber tubing and iron pipes by 3/8-inch copper tubing. This change was made to reduce the corrosion problem met by the previous investigators. The substitution of copper tubing permitted raising the maximum phase flow rate from 22,000 pounds per hour per square foot to 40,000 pounds per hour per square foot. The change was recommended by previous investigators using the horizontal tube extractor.

A second important change in the equipment was arranging the copper tubing so that cocurrent flow was used in the

extraction tube. This change was also recommended by previous investigators<sup>(11,36)</sup> to eliminate the distortion of the interface between the two phases, and to study the effect of the faster moving phase which carried with it a portion of the slower moving phase.

Manometers connected across 3/32-inch orifices were used in both entering and leaving lines to aid in the control of the phase flow rates. The manometers were used only as indicators of the flow rates and not as means for determining the final average flow rate. However, the calibration data obtained to check the accuracy of the manometers indicated that they were sufficiently accurate to be used as close approximations of the flow rates for the water phase.

Bronze needle valves adapted for copper tubing were used to replace the pinch clamps that had previously been used on the equipment. It was found that pinch clamps were not sensitive enough to allow the regulation of the phase flow rates as desired.

A calming section was provided at the entering end of the extraction tube. This was accomplished by adding a 1/16-inch steel plate between the entering streams of the extractor.

Operating Procedure. The length of time between samples was determined by calculating the time required for three complete volume changes in the extraction tube at a particular flow rate. The preliminary work with the extraction tube indicated that the systems used in this investigation reached steady state conditions before two volume changes had been completed. The total length of time per test was the time required for 12 complete volume changes of the extraction tube.

During the performance of the extraction test, the interface between the two phases was observed to bow upward near the center of the tube when the heavier phase exceeded phase flow rate of 19,000 pounds per hour per square foot, and the light phase flow rate was less than 10,000 pounds per hour per square foot. This bowing effect usually occurred when the phase flow rate differential was between 9000 and 10,000 pounds per hour per square foot. With the interface at the center of the tube, bowing of the interface decreased the interfacial area between the two phases. However, it is believed that the change in the interfacial area did not affect the maximum deviation of the results.

I was also noted that for tests performed at approximately equal flow rates exhibited streamline flow when observed

perpendicular to the interface. The wave fronts were observed by watching bits of insoluble material and bubbles of air that had collected at the interface between the two phases.

The most probable source of errors in this investigation was the mixing of the two entering phases as they passed the tip of the calming plate in the horizontal tube extractor. When one phase velocity exceeded the other by more than two to one, and when the slower phase was above 9000 pounds per hour per square foot, wave motion was also observed at the interface. This action changed the area of contact between the two phases. The system n-butanol-water was affected more than the system 1,1,2-trichloroethane-acetone-water since the butanol and the water tended to form a cloudy emulsion which stayed at the interface.

A factor that could also affect the data obtained was the temperature of the feed. The temperature of the water in the storage tank could be controlled by turning steam into the tank. However, it was impractical to heat the butanol tanks by this method since no water could be added to the butanol, and no provision was made for indirect heating.

Method of Analysis. The method of analysis used in this investigation was determination of the refractive indices of the phases of the systems. The dipping refractometer was calibrated for use with known solutions of n-butanol and water; also for solutions of acetone and water saturated with trichloroethane. The analysis of the trichloroethane phase was accomplished by calibrating an Abbe refractometer with known solutions of acetone in trichloroethane saturated with water.

All samples were analyzed at 25 °C, the temperature being controlled to within 0.1 °C. Controlling the temperature of the water bath while using the dipping refractometer was somewhat difficult. The insulation had to be left off of the glass walls of the constant temperature bath so that the light could pass through the walls of the bath to enter the refractometer.

The use of water saturated with trichloroethane as the solvent, and trichloroethane saturated with water as the carrier solvent, was recommended by Bowman<sup>(7)</sup>. The use of the saturated solvent and carrier solvent prevented the transfer of one into the other during the extraction operation. It was found experimentally in this investigation that the assumption that water and trichloroethane were completely immiscible caused an error in the determination of the weight per cent acetone transferred

to water of between 15 and 20 per cent, thereby making the results obtained by Bowman questionable.

### Discussion of Results

Discussion of the results of this study will include comments on the accuracy of data and results, and on the data obtained for the system n-butanol-water; and for the system 1,1,2-trichloroethane-acetone-water.

Accuracy of Data and Results. The accuracy of the data and results obtained in this investigation depends mainly upon the method of analysis of the samples obtained. The weighing of the exit phases from the extraction tube was accurate to within 0.5 per cent of the total weight.

The dipping refractometer, when used with the system n-butanol-water, was calibrated with known samples prepared by using an analytical balance. The scale reading of the dipping refractometer, when used with the butanol and water, was reproducible to within  $\pm 0.02$  scale divisions. This accuracy enabled the determination of the butanol in the water to within  $\pm 0.02$  weight per cent of the total butanol present. The amount of water in butanol was also determined to within  $\pm 0.02$  weight per cent of the total water present.

The calculations used to obtain the overall, mass-transfer coefficient were carried out to five significant figures on a calculator and then rounded off to four figures after the calculations were completed. From the method of analysis and the calculations, it is believed that the accuracy obtained for the overall, mass-transfer coefficients in the system n-butanol-water is  $\pm 0.0003$  in the calculated results.

The accuracy obtained for the overall, mass-transfer coefficient for the system 1,1,2-trichloroethane-acetone-water was limited by the accuracy of the Abbe refractometer. The use of the Abbe refractometer permitted the determination of the acetone in the trichloroethane saturated with water to within  $\pm 0.32$  weight per cent. Since all of the tests were performed with the trichloroethane phase containing at least 4.2 weight per cent acetone, the maximum deviation of the final results would not exceed 10 per cent of the calculated values of the overall, mass-transfer coefficient. The 10 per cent maximum deviation for the calculated values of the overall, mass-transfer coefficient would then be  $\pm 0.01$  units.

In the calculation of the concentration differences, the distribution coefficient of acetone between water and trichloroethane at equilibrium appears. The equations which were used to determine the concentration differences were based on the

assumption that this distribution coefficient was a constant over the range of concentrations being used. To prove the validity of this assumption, a series of distribution coefficients were calculated from about two to twenty per cent acetone as was shown in Table XII, page 57. When these points were plotted in Figure 8, page 58, a straight line resulted, proving that the assumption of constant distribution coefficient was correct.

The System n-Butanol-Water. The overall, mass-transfer coefficients based on both the butanol and the water films decreased as the variable-phase flow rate approached the velocity of the constant phase, and increased as the variable-phase flow rate exceeded the velocity of the constant-phase flow rate. This may be explained by the degree of turbulence or mixing at the interface between the two phases. The greater the velocity gradient across the interface, the greater the tendency of the faster moving phase to pull along a portion of the slower phase and cause turbulence at the interface. However, as the two phases approach equal velocities, the velocity gradient across the interface decreases, and the rolling motion or turbulence at the interface also decreases.



The graphs, Figure 19, page 94, showing the variation of the overall, mass-transfer coefficients based on the water film, with varying, water-phase flow rate, at a butanol-phase flow rate of 19,000 to 23,800 pounds per hour per square foot, does not appear to conform to the general decrease and increase of the transfer coefficient as the varying, water-phase flow rate approaches and exceeds the constant, butanol-phase flow rate. This discrepancy is caused by the limit of the variable, water-phase flow rate. The maximum value of the water-phase flow rate was 20,000 pounds per hour per square foot, which equals the value of the constant, butanol-phase flow rate. At this point on the curve, the value of the overall, mass-transfer coefficient was at a minimum, thereby showing only one half of the curve possible with this type of plot.

At the lower limits of the varying butanol and water-phase velocities some curves, on the other plots in Figure 19, page 94, also showed only one half of the curve which would have been possible if still lower phase flow rates had been used in the investigation. Phase flow rates below 4000 pounds per hour per square foot were not studied since most commercial application seldom uses flow rates lower than this.

A series of tests were performed at water-phase flow rates of 19,000 to 23,800 pounds per hour per square foot and with butanol-phase flow rates from 7000 to 28,000 pounds per hour per square foot. These points, when plotted on Figure 19, page 94, gave no indication of being related to the curves already obtained, nor did they appear to be related individually. A check of the observations made during the performance of the tests at this high phase flow rate, showed that turbulence was present during the test, so these points were not used.

Attempts made to correlate the ratios of the phase flow rates and the overall, mass-transfer coefficients were unsuccessful. The plots showing the ratios of the phase flow rates versus the overall, mass-transfer coefficients in Figures 19 and 20, pages 94 and 96, indicate that a straight line could be drawn through the points; however, the points did not fall very near the lines. For this reason lines were not drawn on the figures.

The observations made during the performance of the experimental work indicate that the ratio of the phase flow rates affects the magnitude of the transfer coefficient more than the individual phase flow rates. The explanation for

this phenomenon is that at low flow rates of from 4000 to 6000 pounds per hour per square foot, the degree of turbulence at the interface is controlled by the velocity gradient across the interface.

The values for the overall, mass-transfer coefficients based on the butanol and the water films, obtained in this investigation using cocurrent flow, were of about the same magnitude as those obtained by Eargle<sup>(11)</sup> when using counter-current flow. The other operating variables being held approximately equal, the range of values for the overall, mass-transfer coefficients based on the water film in Eargle's work was from 0.05 to 1.0 mol per hour per square foot per unit concentration difference. This corresponds to the range of values for the same coefficient obtained in this investigation. The only significant difference in the results was the shape of the curves when the overall, mass-transfer coefficients were plotted against varying water and butanol-phase velocities. This difference in shape is believed to have been caused by the accuracy of the method of analysis.

When the calibration curves for the manometers were determined before the actual experimental work was started, it was found that the manometers were accurate to within

± 200 pounds per hour per square foot. However, since the same source of air was used for pressure on both storage tanks, a change in one phase flow rate affected the other phase flow rate, making it very difficult to reproduce the flow rates closer than those reported.

The System 1,1,2-Trichloroethane-Acetone-Water. The overall coefficients for transfer of acetone from 1,1,2-trichloroethane to water, based on both the water and the trichloroethane phases, at constant water-phase flow rates and varying trichloroethane flow rates developed the same type of curves as encountered with similar plots for the system n-butanol-water. However, the degree of accuracy of the curves shown in Figure 22, page 99, was not the same, since the points on the curves were not as evenly distributed, as those in Figure 19, page 94. The overall, mass-transfer coefficient based on both phases again decreased as the variable trichloroethane-phase flow rate approached the constant water-phase flow rate, and increased as the variable trichloroethane-phase flow rate exceeded the constant water-phase flow rate. It is again apparent that the degree of turbulence at the interface affected the values for the overall, mass-transfer coefficients.

The curves on Figure 22, page 99, plots of the overall, mass-transfer coefficients at constant trichloroethane-phase flow rates versus varying water-phase flow rates, do not conform to the general pattern of a decrease and then an increase of the transfer coefficients as the variable water-phase velocity approaches and then exceeds the constant trichloroethane-phase velocity. The increase of the overall, mass-transfer coefficients from 0.04 to 0.20 pound per hour per square foot per unit concentration difference as the water-phase velocity increases from 4,000 to 20,000 pounds per hour per square foot, indicates that the water film is the controlling film for the system. The changing of the trichloroethane-phase flow rate of 7600 to 9200, to 22,500 to 27,690 pounds per hour per square foot increased the overall, mass-transfer coefficients only 0.04 pound mol per hour per square foot per unit concentration difference. This gives additional proof that the water film is the controlling film.

The points on Figures 23 and 24, pages 101 and 102, showing the ratios of the phase flow rates plotted against the overall, mass-transfer coefficients, are grouped together instead of being strung out as with the system n-butanol-water.

The attempts to correlate the ratios of the phase flow rates and the overall, mass-transfer coefficients were unsuccessful since it was found that the values of the transfer coefficients depended upon the phase velocity almost entirely. It seems that the turbulence at the interface is dependent upon the water-phase flow rate which also governs the values of the overall, mass-transfer coefficient.

The direction of flow does not affect the value of the overall, mass-transfer coefficient, as the results obtained by Bowman<sup>(2)</sup>, using countercurrent flow, are practically identical with those obtained in this investigation. It seems that this is evidence that cocurrent flow exists when countercurrent flow is used, as proposed by Skrzec<sup>(36)</sup>.

### Recommendations

During this investigation for determining the overall, mass-transfer coefficients for the systems n-butanol-water and 1,1,2-trichloroethane-acetone-water, certain observations and difficulties presented themselves and are the basis for the recommendations given in this section.

Constant Temperature Bath. A glass aquarium was used as the constant temperature bath for the dipping refractometer. It is suggested that either a constant temperature bath be purchased, or one made, that will hold the temperature at  $25 \pm 0.1$  °C for several hours at a time. A bath that will hold the sample temperature constant is necessary for use with the dipping refractometer since small temperature changes will cause a large error in the refractometer reading.

Air Bleed Line. During the extraction test performed on the system n-butanol and water, it was found that when the feed tanks were filled with butanol, air entered the line connecting the feed tanks and the extraction tube. When the extraction equipment was started into operation after the feed tanks had been filled, the air in the connecting lines formed a suspension when the butanol entered the extraction tube. The addition of

an air bleed line for each of the entering streams would allow the air to be vented before the test was started.

Temperature of Feed and Solvent. During the extraction test, the temperature of the water could be controlled by allowing steam to enter the storage tank. The temperature of the feed was approximately the temperature of the room since there were no provisions for controlling the temperature. It is recommended that the feed tanks be heated by the wraparound type of electrical heater and then be insulated to help in holding the feed at the desired temperature. Insulation would also help in holding the temperature of the solvent at the desired level during the extraction test.

Temperature of the Extraction Test. During the survey of the literature for this investigation, it was found that the temperature affected the systems tested. It is recommended that the effects of temperature be studied for the systems n-butanol-water and 1,1,2-trichloroethane-acetone-water. Treybal<sup>(43)</sup> states that for most systems there is a critical temperature of solution where the interface between two phases disappears and complete mixing is possible. It is also suggested that attempts be made to design a laboratory extraction unit in which mixing is done above and separation below the critical temperature.



Concentration of Water in Butanol. During this investigation it was found that if the butanol entering the extraction tube contained more than 15 weight per cent water, an emulsion was sometimes formed while moving the butanol through the connecting line to the extraction tube. It is recommended that future tests with butanol and water that the weight per cent of water in the butanol be limited to a maximum of 10 weight per cent.

Ratio of the Phase Flow Rates. Since the attempts to correlate the overall, mass-transfer coefficients and the ratios of the phase flow rates were unsuccessful, it is recommended that a study be made to determine a correlation between the transfer coefficients and the ratio of the phase velocities. It is also suggested that maximum and minimum values for the ratios of the phase velocities that cause change in the interfacial area, be determined.

### Limitations

The limitations of the variables under which the extraction tests were performed on the systems n-butanol-water and 1,1,2-trichloroethane-acetone-water, are divided into three parts: the extraction equipment, the system n-butanol-water, and the system 1,1,2-trichloroethane-acetone-water.

Extraction Equipment. The apparatus used throughout this investigation was a horizontal tube extractor, utilizing continuous cocurrent flow. The extraction tube was made of two, four-foot sections of pyrex tubing, joined by metal flanges. The inside diameter of the extraction tube was one and one-half inches, making the interfacial area one square foot when the interface was at the center of the tube.

n-Butanol-water. The phase flow rates used in the extraction test performed on the system n-butanol-water had maximum values of 25,500 pounds per hour per square foot for butanol, and 20,700 pounds per hour per square foot for water. The minimum phase flow rates for the system were 7600 pounds per hour per square foot for butanol, and 4610 pounds per hour per square foot for water.

The concentration of water in the entering butanol phase varied from 10.6 to 17.8 weight per cent during the performance

of the tests. The temperature of both the entering butanol and water streams varied from 20 to 25 °C during the extraction test. The samples obtained from the extraction tests were analyzed at 25 °C by means of a dipping refractometer.

1,1,2-Trichloroethane-Acetone-Water. The phase flow rates used in the extraction tests performed on the system 1,1,2-trichloroethane-acetone-water had maximum values of 41,100 pounds per hour per square foot for the trichloroethane phase, and 23,100 pounds per hour per square foot for the water phase. The minimum values of the phase flow rates for the same system were 7627 pounds per hour per square foot for the trichloroethane phase, and 4574 pounds per hour per square foot for the water phase. The temperature of the extraction test varied between 22 and 25 °C.

The concentration of the acetone in the entering trichloroethane phase varied between 4.0 and 6.5 weight per cent during the investigation. The acetone content of the extraction samples was determined at 25 °C. The samples of the water phase were analyzed by means of a dipping refractometer. The trichloroethane-phase samples were analyzed by means of an Abbe refractometer.

## V. CONCLUSIONS

The conclusions drawn from the results obtained during this investigation are divided into two groups: one being for the system n-butanol-water, the other being for the system 1,1,2-trichloroethane-acetone-water.

### Conclusions for the System n-Butanol-Water

An evaluation of the results obtained from a study of the transfer of n-butanol and of water in a horizontal extraction tube with an inside diameter of one and one-half inches, and 96 inches long, when the butanol-phase flow rate was varied from 7600 to 25,000 pounds per hour per square foot and the water-phase flow rate was varied from 4160 to 20,700 pounds per hour per square foot at a temperature of 25 °C, led to the following conclusions:

1. The transfer of butanol through the water film decreases as the velocity of the butanol phase approaches the velocity of the water phase, the minimum being reached when the velocities are equal, and then increases from 0.01 to 0.85 pound mols per hour per square foot per unit concentration difference, as the butanol-phase flow rate exceeds the water-phase flow rate.

2. The transfer of water through the butanol film decreases as the velocity of the butanol phase approaches the velocity of the water phase, the minimum being reached at the points of equal phase velocities and then it increases from 0.02 to 1.5 pound mols per hour per square foot per unit concentration difference as the butanol-phase flow rate increases to 22,000 pounds per hour per square foot.

3. The transfer of butanol through the water film decreased from 0.8 to 0.1 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate increased from 4000 to 20,000 pounds per hour per square foot at a butanol flow rate of 19,000 to 23,800 pounds per hour per square foot.

4. The transfer of butanol through the water film decreased from 0.2 to 0.1 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate varied from 7000 to 14,000 pounds per hour per square foot, then increased to 0.9 pound mols per hour per unit concentration difference as the water rate further increased to 20,000 pounds per hour per square foot at a butanol-phase flow rate of 12,450 to 17,100 pounds per hour per square foot.

5. At a butanol-phase flow rate of 7600 to 9000 pounds per hour per square foot, the rate of butanol transferred remained unaffected by the water-phase flow rate (4000 to 15,000 pounds per hour per square foot) at 0.05 pound mols butanol per hour per square foot per unit concentration difference.

6. The transfer of water through the butanol film, at a butanol-phase flow rate of 19,000 to 23,800 pounds per hour per square foot, decreased from 1.5 to 0.80 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate increased from 4000 to 20,000 pounds per hour per square foot; but at a butanol-phase flow rate of 7600 to 17,000, the transfer coefficient remained practically unaffected by increasing the water-phase flow rate over the same range.

7. The use of cocurrent flow in the horizontal tube extractor did not change the range of values of the overall, mass-transfer coefficient based on the water film as compared with countercurrent flow in the same type of equipment and with similar operating conditions.

Conclusions for the System 1,1,2-Trichloroethane-  
Acetone-Water

An evaluation of the results obtained from a study of the transfer of acetone from 1,1,2-trichloroethane into water in a horizontal tube extractor with an inside diameter of one and one-half inches, and 96 inches long, when the trichloroethane-phase flow rate varied from 7620 to 41,100 pounds per hour per square foot, and the water-phase flow rate varied from 4575 to 23,000 pounds per hour per square foot, and the concentration of the acetone in the trichloroethane varied from 4.0 to 6.5 weight per cent, at 25 °C, led to the following conclusions:

1. The overall, mass-transfer coefficient based on the water phase, at three groups of water-phase flow rates of 8800 to 9390, 16,400 to 18,000, and 18,500 to 21,000 pounds per hour per square foot, varied from 0.05 to 0.09, 0.12 to 0.5, and from 0.17 to 0.13 pound moles of acetone per hour per square foot per unit concentration difference as the flow rate of the trichloroethane phase increased from 8000 to 32,000 pounds per hour per square foot.

2. The overall, mass-transfer coefficient based on the water phase, at three groups of trichloroethane-phase flow rates of 7600 to 9200, 15,100 to 19,400, and 22,500 to 27,690

pounds per hour per square foot varied from 0.6, 0.65, and 1.2 to 1.65 pound mols of acetone per hour per square foot per unit concentration difference as the water-phase flow rate increased from 4000 to 22,000 pounds per hour per square foot.

3. The overall, mass-transfer coefficient based on the trichloroethane phase, at three groups of water-phase flow rates of 8800 to 9390, 16,400 to 18,000, and 18,500 to 21,100 pounds per hour per square foot varied from 0.03 to 0.08, 0.08 to 0.11, and from 0.10 to 0.12 pound mols per hour per square foot per unit concentration difference as the trichloroethane-phase flow rate increased from 8000 to 31,000 pounds per hour per square foot.

4. The overall, mass-transfer coefficient based on the trichloroethane phase, at three groups of trichloroethane-phase flow rates of 7600 to 9200, 15,100 to 19,400, and 22,500 to 27,690 pounds per hour per square foot, varied from 0.04 to 0.14, 0.04 to 0.12, and from 0.08 to 0.13 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate increased from 4000 to 22,000 pounds per hour per square foot.



5. The use of phase flow rates that are in the turbulent range are impractical in a horizontal tube extractor, since turbulence changes the interfacial area.

6. Since the results obtained in this investigation agree with the results obtained using countercurrent flow, it indicates that the theory that parallel flow is present even when countercurrent flow is used, is probably correct.

## VI. SUMMARY

The purpose of this investigation was to study the effect of the direction of flow with varying phase velocities on the overall, mass-transfer coefficient in a horizontal, unpacked, extraction tube, for the system n-butanol-water, and for the system 1,1,2-trichloroethane-acetone-water. The equipment used for both systems was a horizontal tube contactor, constructed of pyrex glass tubing 96 inches long, with an inside diameter of one and one-half inches. This summary is divided into two parts, one being for the system n-butanol-water, and the other being for the system 1,1,2-trichloroethane-acetone-water.

### Summary for the System n-Butanol-Water in Cocurrent Flow

The butanol-phase flow rates varied from 7600 to 25,000 pounds per hour per square foot, and the water-phase flow rates varied from 4160 to 20,700 pounds per hour per square foot.

The transfer of butanol through the water film decreases as the velocity of the butanol phase approaches the velocity of the water phase, the minimum being reached when the velocities are equal, and then increases from 0.01 to 0.85

pound mols per hour per square foot per unit concentration difference, as the butanol-phase flow rate exceeds the water-phase flow rate.

The transfer of water through the butanol film decreases as the velocity of the butanol phase approaches the velocity of the water phase, the minimum being reached at the points of equal phase velocities and then it increases from 0.02 to 1.5 pound mols per hour per square foot per unit concentration difference as the butanol-phase flow rate increases to 22,000 pounds per hour per square foot.

The transfer of butanol through the water film decreased from 0.8 to 0.1 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate increased from 4000 to 20,000 pounds per hour per square foot at a butanol flow rate of 19,000 to 23,800 pounds per hour per square foot.

The transfer of butanol through the water film decreased from 0.2 to 0.1 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate varied from 7000 to 14,000 pounds per hour per square foot, then increased to 0.9 pound mols per hour per unit concentration difference as the water rate further increased to 20,000 pounds per hour per square foot at a butanol-phase flow rate of 12,450 to 17,100 pounds per hour per square foot.

At a butanol-phase flow rate of 7600 to 9000 pounds per hour per square foot, the rate of butanol transferred remained unaffected by the water-phase flow rate (4000 to 15,000 pounds per hour per square foot) at 0.05 pound mols butanol per hour per square foot per unit concentration difference.

The transfer of water through the butanol film, at a butanol-phase flow rate of 19,000 to 23,800 pounds per hour per square foot, decreased from 1.5 to 0.80 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate increased from 4000 to 20,000 pounds per hour per square foot; but at a butanol-phase flow rate of 7600 to 17,000, the transfer coefficient remained practically unaffected by increasing the water-phase flow rate over the same range.

The use of cocurrent flow in the horizontal tube extractor did not change the range of values of the overall, mass-transfer coefficient based on the water film as compared with countercurrent flow in the same type of equipment and with similar operating conditions.

Summary for the System 1,1,2-Trichloroethane-  
Acetone-water in Cocurrent Flow

The flow rates for the system 1,1,2-trichloroethane-acetone-water varied from 4757 to 23,100 pounds per hour per square foot for the water phase, and from 7627 to 41,100 pounds per hour per square foot for the trichloroethane phase. The acetone was present in the entering trichloroethane phase and varied from 4.0 to 6.5 weight per cent.

The overall, mass-transfer coefficient based on the water phase, at three groups of water-phase flow rates of 8800 to 9390, 16,400 to 18,000, and 18,500 to 21,000 pounds per hour per square foot, varied from 0.05 to 0.09, 0.12 to 0.5, and from 0.17 to 0.13 pound mols of acetone per hour per square foot per unit concentration difference as the flow rate of the trichloroethane phase increased from 8000 to 32,000 pounds per hour per square foot.

The overall, mass transfer coefficient based on the water phase, at three groups of trichloroethane-phase flow rates of 7600 to 9200, 15,100 to 19,400, and 22,500 to 27,690 pounds per hour per square foot varied from 0.6 to 0.65, and 1.2 to 1.65 pound mols of acetone per hour per square foot per unit concentration difference as the water-phase flow

rate increased from 4,000 to 22,000 pounds per hour per square foot.

The overall, mass-transfer coefficient based on the trichloroethane phase, at three groups of water-phase flow rates of 8,800 to 9,390, 16,400 to 18,000, and 18,500 to 21,100 pounds per hour per square foot varied from 0.03 to 0.08, 0.08 to 0.11, and from 0.10 to 0.12 pound mols per hour per square foot per unit concentration difference as the trichloroethane-phase flow rate increased from 8,000 to 31,000 pounds per hour per square foot.

The overall, mass-transfer coefficient based on the trichloroethane phase, at three groups of trichloroethane-phase flow rates of 7,600 to 9,200, 15,100 to 19,400, and 22,500 to 27,690 pounds per hour per square foot, varied from 0.04 to 0.14, 0.04 to 0.12, and from 0.08 to 0.13 pound mols per hour per square foot per unit concentration difference as the water-phase flow rate increased from 4,000 to 22,000 pounds per hour per square foot.

The use of phase flow rates that are in the turbulent range are impractical in a horizontal tube extractor, since turbulence changes the interfacial area.

Since the results obtained in this investigation agree with the results obtained using countercurrent flow, it indicates that the theory that parallel flow is present even when countercurrent flow is used, is probably correct.

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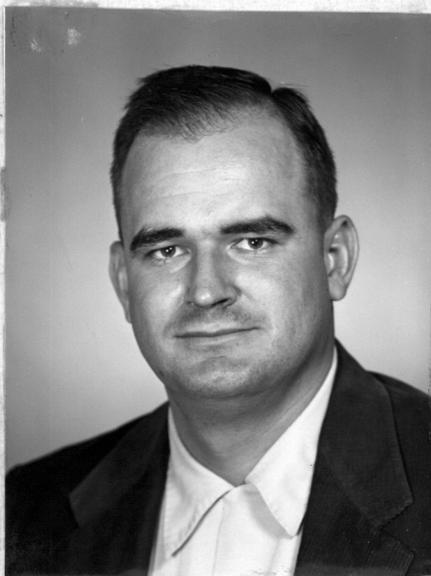
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IX. VITA



Jack Langdon Bowling was born in Bluefield, West Virginia, on March 8, 1929. He lived in Bluefield until 1932, when he moved to Mullens, West Virginia. He attended grammar school and high school in Mullens, completing high school in 1946. He enrolled at Virginia Polytechnic Institute in the fall of 1946 and attended school for two years. He enrolled again at Virginia Polytechnic Institute in 1949 and received the B. Sc. degree in chemical engineering in 1952.

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