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# THE INFLUENCE OF AGITATION ON A CONTINUOUS COUNTER-CURRENT LIQUID-LIQUID EXTRACTION COLUMN

BY

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SUBMITTED TO THE FACULTY OF
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## ABSTRACT

This work presents operating data and correlations of agitated extraction equipment studied under continuous counter-current flow.

The experimental work was carried out in an agitated column 36 inches in length with an internal diameter of 2 inches using the system toluene, benzoic acid and water. The working section of the column was 24 inches. The design of the column used for the experimental work is similar to that proposed in a patent by van Dijck (3). The column is similar only to the extent that the patent covers numerous conditions of vertical agitation.

The distribution data and the equilibrium solubility curve were determined by Appel and Elgin (1).

The experimental results obtained indicate that an agitated counter-current extraction column possesses a number of advantages over its stationary counterpart. Agitation increases the efficiency of a liquid-liquid extraction column and by proper control of agitation the extraction coefficient can be made nearly independent of the dispersed phase flow rate.

The results obtained also made possible the derivation of an emperical equation correlating H.T.U., agitation and thruput for the system benzoic acid, toluene and water.

$$(H.T.U.) = \frac{33.4}{T} (2.73 - \mathrm{Log}_{10} R.P.M.)$$

# INTRODUCTION

Simple liquid extraction is a process wherein a solute, either liquid or dissolved solid, is transferred from one solution to another by counter-current contacting of the two liquid phases. Equipment for carrying out the process of liquid-liquid extraction can be conveniently divided; according to Morello and Poffenberger (2); into two basic types. The factor which determines into which class a piece of equipment belongs is the method of separation of the immiscible phases (gravity or centrifugal). In general mixer settler combinations and counter-current columns are the most common forms of equipment employing gravity separation. Gravity separated extractors may be subclassified as to whether the contact between phases is thru extended films or droplets. In general contacting is done with one phase continuous and the other distributed to give the necessary large interfacial area. Most types of counter-current columns suffer from the disadvantage that the only force available for the maintenance of dispersion of the dispersed phase is that which is rived from the interfacial tension of the two liquids. This force is insufficient to produce any further breakup of the dispersed phase and there is no mechanism for overcoming the effects of drop coalescence. This results in a decrease of the surface contact between the two liquid phases. A counter-current column when used for extraction is therefore a much less efficient device than when used for contacting a liquid with a vapor or a gas.

Another type of extraction unit which falls into the gravity class is the mixer settler extractor combination. Although mixer settler extractors provide sufficient force for agitation and dispersion, they generally involve batch operation and where the extraction requires a large number of equilibrium stages it is often costly and awkward to process materials in this type of equipment.

A prime example of the centrifugal type of liquid-liquid extraction unit is the Podbielniak Extractor. In this type contacting and separating of two liquid phases, either of which may contain suspended solids, is effected continuously and counter-currently in the contact elements of a spinning rotor.

One of the first reported designs which combined a form of mechanical agitation with the counter-current action of the extraction column was that of van Dijck (3). The proposed apparatus was an unpacked column fitted with close fitting perforated plates attached together by means of chains with a large counterweight suspended from the bottom. The column was so constructed that the plates could be moved up and down with respect to the column and its contents by means of an external reciprocating mechanism. However no experimental results have been published on the operation of this type of column either by van Dijck or by later investigators.

In more recent years other designs for combining the counter-current action of extraction column with the power-

driven mechanical agitator have appeared (4) (5) (6) (23). Most of these new designs for extraction columns consist of a number of mixers arranged vertically in a column with packed or settling sections between each zone and although many of these devices are quite complicated in a mechanical sense their reported efficiences are high.

A still more recent design is that of Feick and Anderson (7) who devised a packed column and supplied agitation to the liquids by causing them to be moved up and down by means of an external flexible diaphragm. Feick and Anderson measured the performance of the column with and without agitation and determined by calculation of extraction coefficients, H.T.U. values and number of equilibrium contacts that agitation improved the performance of their column severalfold. These investigators determined that by the proper control of agitation the extraction coefficient could be made almost independent of the rate of flow of the liquid phases. They also found that agitation lowered the flooding point of the column to some extent and that packing with a high void fraction was desirable.

From the foregoing considerations an extraction column was proposed which would combine the advantages of countercurrent flow with mechanical agitation; while at the same time would eliminate the complicated vertical type of agitator and settling sections of the Scheibel (6) type of column and would also eliminate entirely the need for packing as in the Feick and Anderson (7) column. In order to

accomplish this a column was designed which was made up of a series of semi-circular plates spaced at regular intervals on two vertical shafts which could be raised and lowered in-order to supply the desired agitation.

## THEORY

Liquid-liquid extraction, one of the diffusional operations, is the mass transfer of a solute; either dissolved solid or liquid; from one liquid phase to another. There are two methods by which mass transfer may be accomplished, molecular diffusion and eddy diffusion. Molecular diffusion is the mechanism of transfer of a substance either thru a fluid which is motionless or, if the fluid is in laminar flow, in a direction perpendicular to the velocity of the fluid. Eddy diffusion is the transfer of matter thru a fluid flowing in turbulent flow. In turbulent flow eddies exist within the body of the fluid which carry with them any dissolved solid in bulk. Since the velocities of the eddies are great in comparison to the velocities of molecular diffusion, it is to be expected that eddy diffusion, or the transfer of the solute under such conditions, is much the more rapid process.

The phenomenon of molecular diffusion has been studied from many points of view, frequently conflicting. Fick (9) applied the well known Fourier equation for rate of heat flow to the problem of diffusion. The mechanism of the two processes is not identical, since in the penetration of a liquid by a diffusing solute there will necessarily be displacement of the liquid and consequent volume changes arising for which the Fourier equation does not account. As an approximation, however, the Fick concept is very useful particularly since the Fourier equation has been integrated for many situations which

parallel problems in diffusion.

Maxwell (10) and later Stefan (11) considered the simultaneous movement of both components of the solution through which the solute is diffusing and conceived the approach that the resistance to diffusion of a substance through a solution is proportional to the relative velocities of the molecules involved, the distance through which diffusion occurs and the concentrations of the molecules involved.

Arnold's (12) kinetic theory and Eyring's (13) absolute rate theory are two later emperical estimations which were formulated to predict molecular diffusivity with some success.

The complete diffusion process ordinarily occurs through successive regions of a fluid in laminar and turbulent flow. In the region between those strictly in laminar and turbulent flow, the nature of the change from laminar flow to turbulence is obviously of importance. The Prandtl-Taylor concept of a strictly laminar film, with a well defined boundry separating it from the turbulent region has been shown to be incorrect and there exists instead a gradual change from one condition to another.

The first complete picture of the mass transfer process was introduced by Lewis and Whitman (14) who applied the above concepts to a typical transfer of a solute from one liquid to another nonconsolute liquid in contact with it, under steady state. These investigators assumed both liquids

to be in motion in a general direction parallel to the interface between them and postulated there will exist concentration gradients in both phases which act as driving forces for the transfer and that the resistance to diffusion can be represented by an effective film thickness for each phase. They also postulated that at the interface itself equilibrium would be established. Because of the difficulty of finding the exact concentrations at the interface, the mass-transfer coefficients $k_E$ and $k_R$; defined by $\theta N = k_R dS(c_R - c_{Ri}) = k_E dS(c_{Ei} - c_E)$ are usually not known. However, by the introduction of an overall mass-transfer coefficient $K_E$ or $K_R$ and the definition of a concentration $C_E^* = mc_R$ or $C_E = mC_R^*$ the complete mass-transfer operation may be represented by $dN = K_R dS \Delta C_{OR}$ or $dN = K_E dS \Delta C_{OE}$.

Chilton and Colburn (20) (21) showed that the efficiency of a continuous, counter-current liquid-liquid extraction process is most simply expressed in terms of the concentration change to be effected in either liquid stream and the driving force. This quantity they defined as "Number of Transfer Units" (N), where for diffusion of one component $N = \int_{x_2}^{x_1} \frac{(1-x)f\,dx}{(1-x)(x^*-x)}$. They also defined the height of a transfer unit H.T.U. as the effective height of the column H divided by the number of transfer units $N_{OT} = \frac{H}{(HTU)_{OT}}$. This quantity they found to be more reproducible than that of the individual coefficients which vary rapidly with flow rates.

Treybal (15) derived for design purposes the total ex-

pression for amount of solute transferred from the raffinate phase to the extract phase.

$$N_{tOR} = \frac{1}{1-\frac{mE}{R}} \ln\left[\left(1-\frac{mE}{R}\right)\left(\frac{x_{E1}-mx_{R2}}{x_{E2}-mx_{R2}}\right)+\frac{mE}{R}\right] + \frac{1}{2}\ln\frac{1-x_{E2}}{1-x_{E1}} \quad (1)$$

In order to properly use this equation calculations should be based on the phase that offers the greater resistance to mass-transfer; m large, the raffinate phase; m small, the extract phase.

Perry (16) and Treybal (18) have summarized the effect of various variables, packing characteristics, flow rates, agitation and column end design film resistance on the efficiency of counter-current continuously operated liquid-liquid extraction columns.

Laddha and Smith (17) after a study of the mass-transfer resistances in liquid-liquid extraction in packed and unpacked columns concluded that changes in interfacial area with flow rates and physical properties have an important influence on any correlation of H.T.U.. Appel and Elgin (1) presented a correlation of the mass-transfer coefficient with drop size velocity and column holdup of the dispersed phase.

The general principles of rates of mass-transfer of the diffusional operations as absorption and distillation were applied to liquid-liquid extraction by Hunter and Walsh (18) and Elgin and Browning (19). Chilton and Colburn (20), Colburn (21) and others have shown in detail the similarity of rate of mass-transfer equations for these chemical engineering operations.

Elgin and Browning (19) have shown for cases where the system does not depart considerably from the simple distribution law, and where large volume changes, amount of solute extracted and concentrations involved are not large the rate of mass-transfer for liquid-liquid extraction may be written:

$$\frac{N}{\theta} = K_{\omega a} V \left[ \frac{(C^*_{\omega_1} - C_{\omega_2}) - (C^*_{\omega_2} - C_{\omega_2})}{2.3 \operatorname{Log} \frac{(C^*_{\omega_1} - C_{\omega_1})}{(C^*_{\omega_2} - C_{\omega_2})}} \right] \qquad (2)$$

$$\frac{N}{\theta} = K_{\omega a} V \left[ \frac{\Delta C_1 - \Delta C_2}{2.3 \operatorname{Log} \frac{\Delta C_1}{\Delta C_2}} \right] = K_{\omega a} V (\Delta C)_{\text{log mean}} \qquad (3)$$

$$K_{\omega a} \frac{N/\theta}{V(\Delta C)_{\text{log mean}}} \qquad (4)$$

Chilton and Colburn (20) introduced the term H.T.U. (height of a transfer unit) in distillation and absorption computations and showed the relation of the H.T.U. values to the well known mass-transfer coefficients and also pointed out some advantages of the use of H.T.U. over the latter coefficients. Their equations were;-

For Absorption $\quad \text{H.T.U.} = \frac{H}{n} = \frac{H}{\int_{p_1}^{p_2} \frac{dp}{\Delta p} \left( \frac{P_g f}{P_g} \right)} = \frac{G}{K_a P_{gf} M_m} \qquad (5)$

For Packed Column Distillation $\quad \text{H.T.U.} = \frac{H}{n} = \frac{H}{\int_{y_1}^{y_2} \frac{dy}{\Delta y}} = \frac{G}{K_a \pi M_m} \qquad (6)$

Chilton and Colburn (20) also pointed out that the same methods could be applied to counter-current liquid-liquid extraction operations.

Elgin and Browning (10) employing a procedure analogous

to that of Chilton and Colburn for distillation and absorption obtained the following expressions (6) and (7) for liquid-liquid extraction.

$$(H.T.U.)_{ow} = \frac{H}{n} = \frac{H}{\int_{C_{w_1}}^{C_{w_2}} \frac{dC}{\Delta C}} = \frac{V'_w}{K_{wa}S} \qquad (6)$$

For the case of dilute solutions and assuming the simple distribution law to hold equation (6) was simplified to:

$$(H.T.U.)_{ow} = \frac{H}{n} = \frac{H}{\frac{C_{w_2} - C_{w_1}}{(\Delta C)_{log\ mean}}} \quad \frac{V'_w}{K_{wa}} \qquad (7)$$

Colburn pointed out that heat transfer, dehumidification, absorption, distillation and extraction operations in general involve two film resistances and derived the relation of overall H.T.U. to single film values of H.T.U.. His equations are: For absorption (diffusion in one direction)

$$(H.T.U.)_{OG} = (H.T.U.)_G + (H.T.U.)_L \left(\frac{mGm}{Lm}\right) \frac{(1-x)_f}{(1-y)_f} \qquad (8)$$

For distillation (equi-molar counter diffusion)

$$(H.T.U.)_{OG} = (H.T.U.)_G + (H.T.U.)_L \left(\frac{mGm}{Lm}\right)$$

Colburn showed that the above methods developed for absorption and distillation also applied to extraction:

$$(H.T.U.)_{ow} = (H.T.U.)_w + (H.T.U.)_t \ \frac{V_w}{V_t} \ \frac{dc_w}{dc_t}$$

The purpose of this work was to determine the effect of agitation, if any, upon the extraction efficiency of a counter current liquid-liquid extraction column using the system benzoic acid, toluene and water and to compare the efficiency

of this type of column with other agitated columns. An attempt was also made to obtain a correlation of H.T.U., agitator speed and total thruput for the above system.

## NOMENCLATURE

| | |
|---|---|
| $a$ | - Area of interphase contact |
| $c$ | - Concentration |
| $d$ | - Differential operator |
| $G$ | - Mass velocity of gas or vapor |
| $H$ | - Effective height of column |
| HTU | - Height of a transfer unit |
| $k$ | - Mass transfer film coefficient |
| $K$ | - Overall mass transfer coefficient |
| $L_m$ | - Molar liquid velocity |
| $M_m$ | - Average molecular weight of vapor stream |
| $m$ | - Slope of equilibrium curve $dy^*/dx$ |
| $N/\theta$ | - Amount of solute transfered per unit time |
| $n$ | - Number of transfer units |
| $p$ | - Partial pressure of diffusing component |
| $p^*$ | - Equilibrium pressure of diffusing component (partial) |
| $\Delta p$ | - $p - p^*$ |
| $p_f$ | - Partial pressure of inert component $(\Pi - p)$ |
| $p_{gf}$ | - Logarithmic mean of $(\Pi - p)$ and $(\Pi - p^*)$ |
| $S$ | - Cross-section of column |
| $V$ | - Effective volume of column |
| $V_w$ | - Superficial velocity, water phase |
| $V_w'$ | - Rate of flow of water phase |
| $V_t$ | - Superficial velocity, toluene phase |
| $x$ | - Mol fraction of diffusing component in liquid |
| $y$ | - Mol fraction of diffusing component in gas $p/\Pi$ |
| $y^*$ | - Equilibrium mol fraction of diffusing component out of liquid $p^*/\Pi$ |
| $x^*$ | - Equilibrium value of $x$ corresponding to $y$ |
| $(1-x)_f$ | - Log mean of $(1-x)$ and $(1-x^*)$ |
| $(1-y)_f$ | - Log mean of $(1-y)$ and $(1-y^*)$ |
| $\Delta$ | - Difference between value and equilibrium value |
| $\Delta y$ | - $y - y$ |
| $\Pi$ | - Total pressure |
| $\theta$ | - Time |

## SUBSCRIPTS

| | |
|---|---|
| E | - Extract phase |
| I | - Concentrated end of column |
| O | - Overall |
| R | - Raffinate phase |
| t | - Toluene |
| w | - Water |
| Z | - Dilute end of column |

## EXPERIMENTAL PROCEDURE

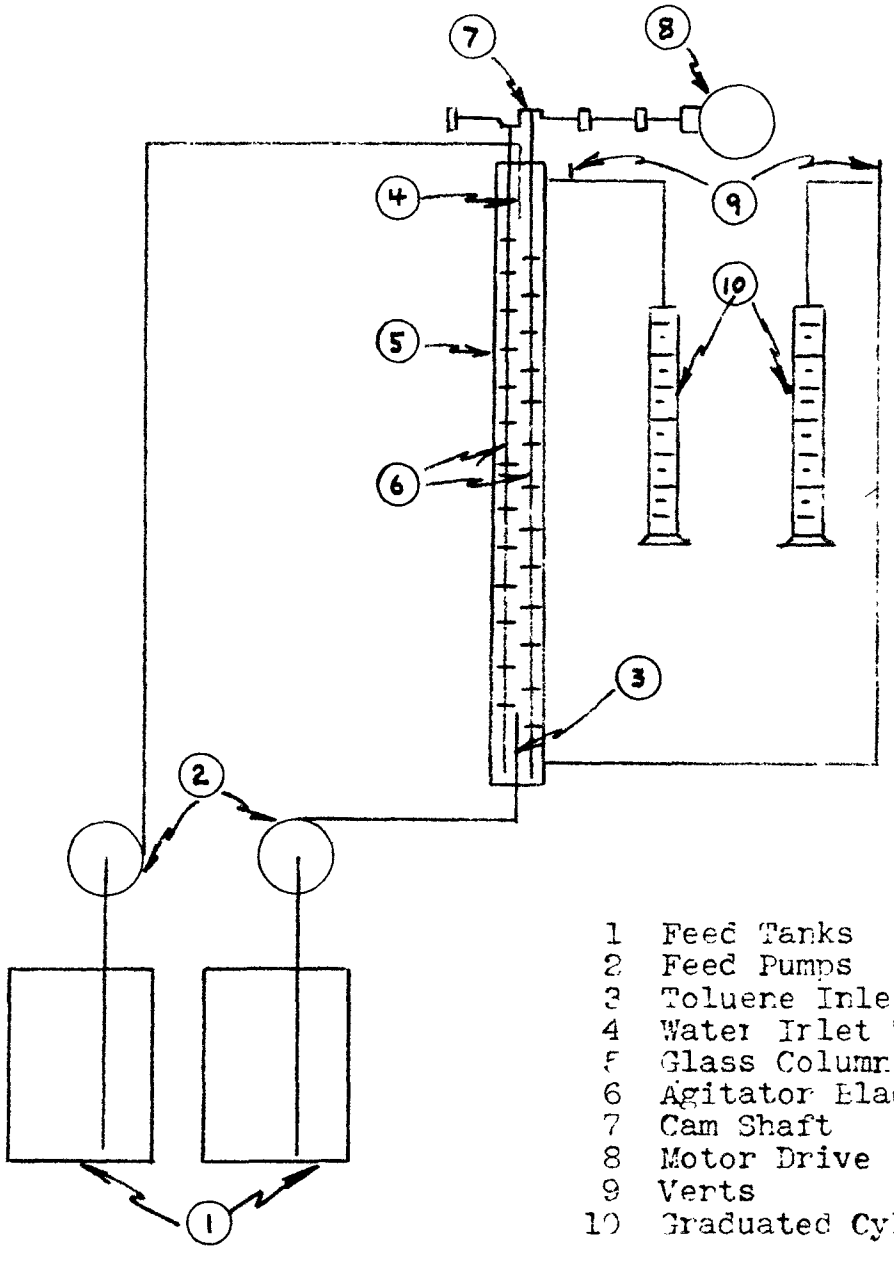
The experimental apparatus was constructed as shown diagrammatically in Figure 1. Two five gallon glass cartoys were used for feed reservoirs. Solvent and aqueous feed were pumped into the column thru two extremely accurate proportionating pumps (Research Appliance Co., Pittsburg, Pa. Model No. 1000). Agitation was supplied by a Sunbeam Mixmaster motor coupled to the agitators thru a brass cam shaft and supported by three rooler bearings. The cam shaft was constructed so that the vertical motion of the agitator blades was exactly one-half of an inch.

The column was constructed of borosilicate glass pipe 8 inches inside diameter and an overall length of 36 inches. The solvent inlet was a 5 mm stainless steel tube; while the aqueous feed line, raffinate and extract takeoff were 6 mm glass tubing. The agitator assembly was constructed entirely of stainless steel. Twenty-five semi-circular stainless steel plates 1/16 of an inch in thickness were supported on each of two stainless steel shafts 1/8 of an inch in diameter. The plates were supported and separated by stainless steel tubular spacers. The agitator shafts were supported by stainless steel tubing 4 inches in length and 3/16 of an inch inside diameter. The agitator supports were held in place with a stainless steel frame and rubber stoppers. The working section of the column was 24 inches in length.

The water forming the continuous phase was brought into

Figure 1

Schematic Diagram of Apparatus




1 Feed Tanks
2 Feed Pumps
3 Toluene Inlet Tube
4 Water Inlet Tube
5 Glass Column - 2 in. I.D.
6 Agitator Blades and Shaft
7 Cam Shaft
8 Motor Drive
9 Vents
10 Graduated Cylinders

the column at the top while the toluene containing the benzoic acid was introduced at the bottom of the column to form the discontinuous phase. The two fluids were fed from the reservoirs through the regulating pumps into the column. The level of the interface at the top of the column was adjusted by changing the height of the raffinate take-off line.

The column was agitated by means of a reciprocating movement of the agitator shafts which were attached to the eccentric drive. The eccentric was driven by a variable speed motor coupled to a rheostat by means of which the speed could be controlled accurately. The column throughput was measured by collecting extract and raffinate in graduated glass cylinders.

The equilibrium data used for benzoic acid were those of Appel and Elgin (1) supplemented by data of Feick and Anderson (7). The data of Row, Koffolt and Withrow (8) although covering a wider range of temperatures were found by other investigators to be inaccurate.

In making a run the feed tanks were filled and the temperatures recorded. The temperatures of the solutions varied between 19.5 C and 21.0 C throughout the work. The column was filled with water and the two pumps were set at the desired flow rates. The agitator was started and set for the desired speed and the operation was continued until

at least 3 liters of each liquid had passed thru the column. Samples of the effluent streams were taken for analysis and titrated. The column operation was continued until an additional one-half liter of each liquid had passed through and samples of the effluent streams were again removed for analysis. The samples were analyzed by titration with standard sodium hydroxide solution using thymol blue as the end-point indicator. Ethyl alcohol was added to the toluene samples in order to render them miscible with the titrating liquid. The column was considered to be in steady state when the rate of change of concentration was zero. Material balances were made on all runs and the data were rejected if the error exceeded $\pm 1\%$.

During the initial stages of the experimental work flow rates of 50 ml. per minute were chosen for both the dispersed and continuous phases, while the agitator speed was varied. When it became apparent that total column through-put was an important variable; various other flowrates were checked at the same agitator speeds. After each run was made the pumps, process lines and column were flushed thoroughly with water and allowed to drain.

The extraction coefficients (8) were found from the equation:-

$$K_a = \frac{N}{\theta V \Delta C_{log\ mean}} \qquad (8)$$

For the special case where the incoming water phase contains

no solute $\Delta C_{lm}$ is given by:-

$$\Delta C_{lm} = \frac{C^*_{w_1} - (C^*_{w_2} - C_{w_2})}{\ln\left[\frac{C^*_{w_1}}{C^*_{w_2} - C_{w_2}}\right]} \qquad (9)$$

The height of a transfer unit H.T.U. was found from the equation:-

$$H.T.U. = L/K_a \qquad (10)$$

The number of equilibrium stages was found by the usual graphical procedures used in calculating theoretical plates in distillation or absorption work (22).

## NOMENCLATURE

| | |
|---|---|
| a | - Area of interphase contact, square feet per cubic foot |
| C | - Solute concentration, pound moles per cubic foot |
| C* | - Equilibrium solute concentration, pound moles per cubic foot |
| $\Delta C_{lm}$ | - Logarithmic-mean interphase concentration difference, pound moles per cubic foot |
| HTU | - Height of a transfer unit, feet |
| Ka | - Over-all coefficient per unit volume, pound moles per (hour)(square foot)(unit $\Delta C$) |
| ln | - Natural logarithm |
| L | - Liquid flow rate, cubic feet per hour per square foot |
| N | - Amount of solute transferred between phases, moles |
| V | - Volume of column, cubic feet |
| $\theta$ | - Time, hours |

## SUBSCRIPTS

| | |
|---|---|
| w | - Water phase |
| t | - Toluene phase |
| 1 | - Inlet |
| 2 | - Outlet |

## DISCUSSION

The present investigation shows that agitation has a considerable affect on the efficiency of a continuous counter current liquid-liquid extraction column. The operation of the column is shown in a quantative way in Figures 2 through 8. These pictures were taken with a Zeiss Contessa camera using a Tessar f 2.8 lens. Figure 2 shows the experimental equipment in operation, while Figure 3 shows the appearance of a section of the column when in operation without agitation. Figure 4 shows the affect of very mild agitation (50 rpm). The toluene phase no longer adheres to the bottom of the agitator blades and the globules of toluene can be seen suspended in the aqueous phase. Figures 5 through 8 show the increasingly fine dispersion obtained as the speed is increased from 150 to 500 rpm. It was impossible to increase the speed of agitation above 500 rpm with the system used in this work as the materials in the column emulsified above this speed.

A complete tabulation of all runs made is shown in Table 1. The first four runs were made with no agitation and the agitator blades in the open position. Runs 1 and 2 were made with the dispersed phase entering directly through the open inlet tube, while runs 3 and 4 were made with the solvent entering through a teflon spray-head containing eight 0.0156 inch diameter holes spaced at 45 degrees on a ¾ inch anular circumference. The spray-head was eliminated

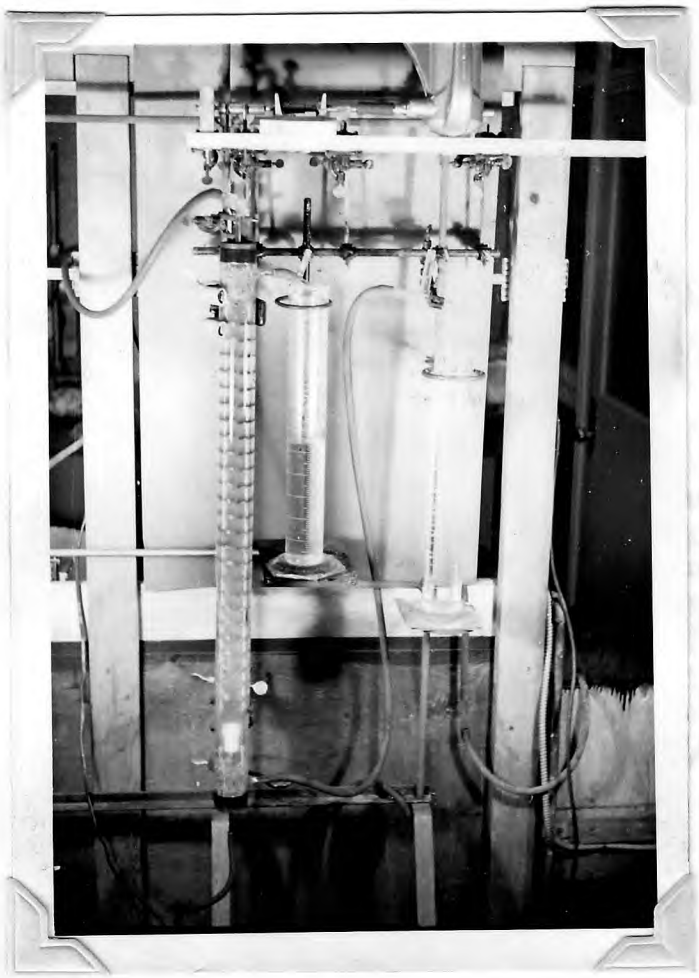


**Figure 2**
**Column in Operation**

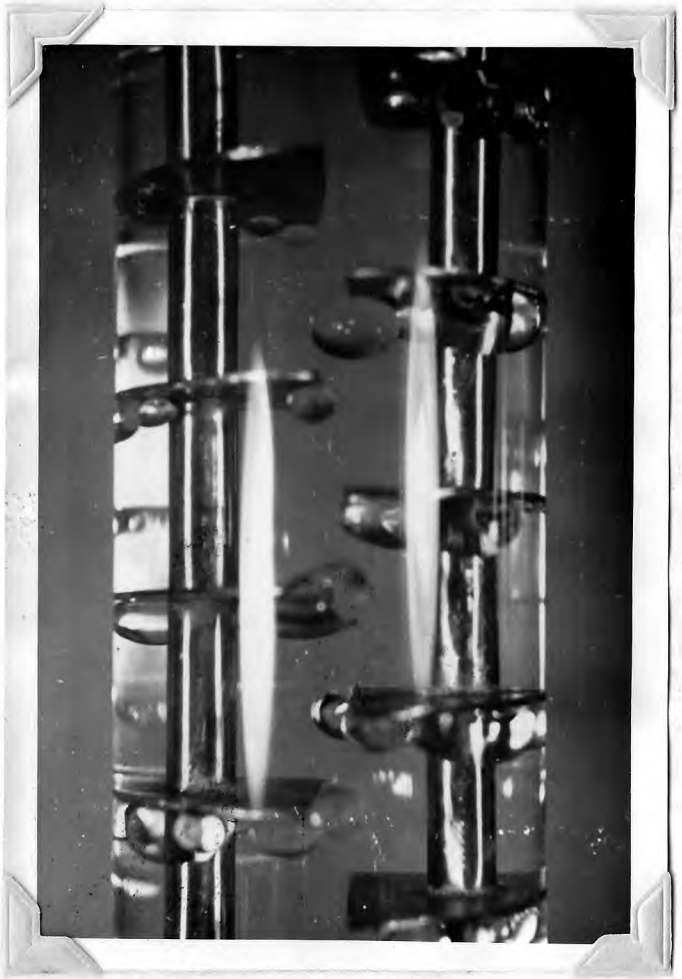


Figure 3  0 rpm

Figure 4  50 rpm

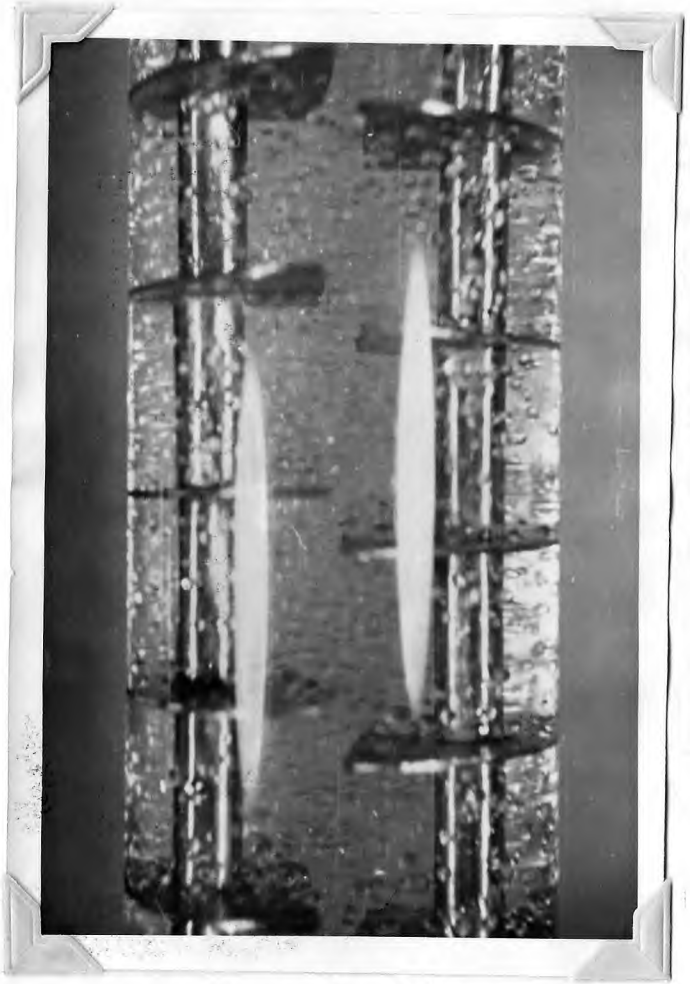


Figure 5 150 rpm

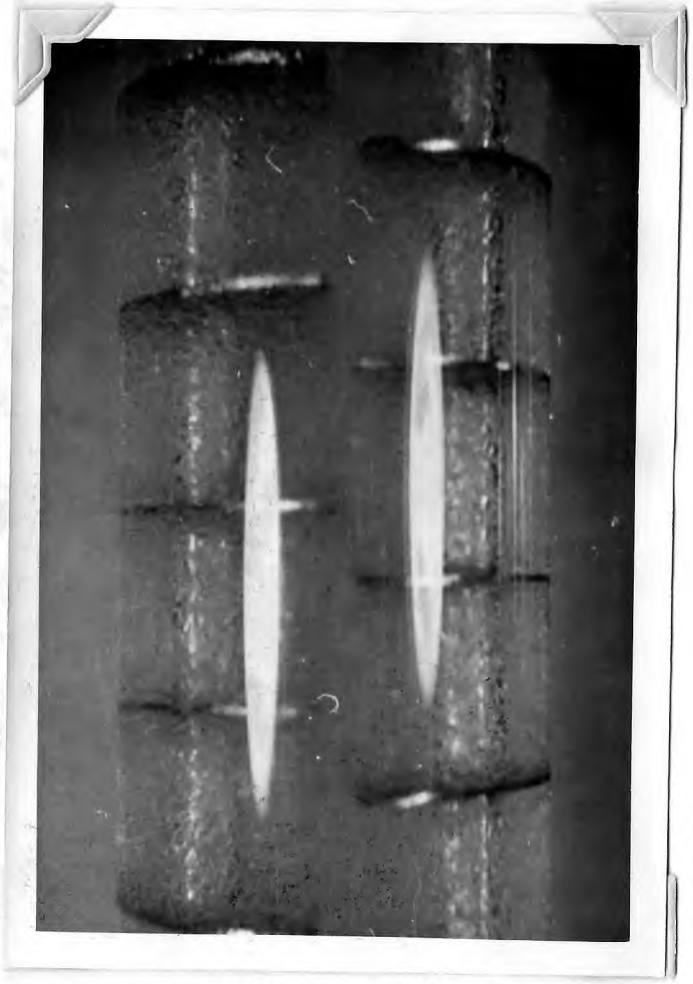


Figure 6 250 rpm

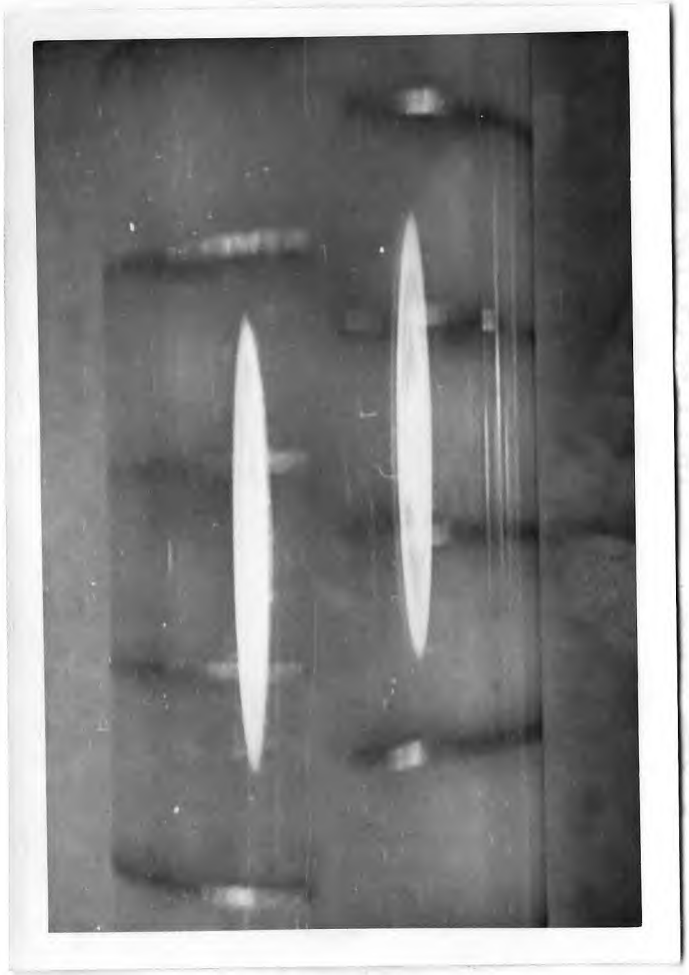


Figure 7 350 rpm

Figure 8 500 rpm

## TABLE 1

PERFORMANCE OF COLUMN WHEN EXTRACTING BENZOIC ACID FROM TOLUENE WITH WATER
DISPERSED PHASE - TOLUENE  COLUMN HEIGHT 24 INCHES

| Run No | Acid Concentration lb moles/cu ft | | | | Flow Rate cu ft/hr/sq ft | | Nozzle | Agitator Speed rpm | Extraction Coefficient K | Height of transfer Unit ft |
|---|---|---|---|---|---|---|---|---|---|---|
| | Toluene In | Water In | Toluene Out | Water Out | Toluene | Water | | | | |
| 1 | 0.01020 | 0.0 | 0.0100 | 0.000223 | 4.77 | 4.66 | - | 0 | 0.82 | 5.68 |
| 2 | 0.01020 | 0.0 | 0.00995 | 0.000235 | 4.80 | 4.86 | - | 0 | 0.85 | 5.51 |
| 3 | 0.01020 | 0.0 | 0.00967 | 0.000620 | 4.57 | 4.28 | S - H | 0 | 3.53 | 1.21 |
| 4 | 0.01020 | 0.0 | 0.00966 | 0.000570 | 4.61 | 4.46 | S - H | 0 | 3.04 | 1.52 |
| 5 | 0.01020 | 0.0 | 0.00943 | 0.000820 | 4.95 | 4.27 | - | 250 | 3.38 | 1.26 |
| 6 | 0.01020 | 0.0 | 0.00955 | 0.000771 | 4.90 | 4.86 | - | 250 | 3.72 | 1.24 |
| 7 | 0.01020 | 0.0 | 0.00918 | 0.001017 | 4.77 | 4.77 | - | 350 | 6.98 | 0.68 |
| 8 | 0.01020 | 0.0 | 0.00911 | 0.001040 | 4.86 | 4.86 | - | 350 | 7.23 | 0.65 |
| 9 | 0.01024 | 0.0 | 0.00976 | 0.000510 | 4.77 | 4.72 | - | 150 | 2.53 | 1.86 |
| 10 | 0.01024 | 0.0 | 0.00980 | 0.000498 | 4.81 | 4.86 | - | 150 | 2.43 | 1.94 |
| 11 | 0.01024 | 0.0 | 0.00990 | 0.000367 | 4.77 | 4.77 | - | 50 | 1.51 | 3.12 |
| 12 | 0.01024 | 0.0 | 0.00993 | 0.000360 | 4.81 | 4.86 | - | 50 | 1.48 | 3.19 |
| 13 | 0.01024 | 0.0 | 0.00907 | 0.001120 | 4.77 | 4.71 | - | 500 | 7.94 | 0.60 |
| 14 | 0.01024 | 0.0 | 0.00905 | 0.001142 | 4.81 | 4.86 | - | 500 | 7.78 | 0.61 |
| 15 | 0.01024 | 0.0 | 0.00916 | 0.000722 | 4.81 | 6.82 | - | 350 | 11.65 | 0.59 |
| 16 | 0.01024 | 0.0 | 0.00888 | 0.000980 | 3.40 | 4.86 | - | 350 | 6.40 | 0.76 |
| 17 | 0.01024 | 0.0 | 0.00840 | 0.000867 | 2.43 | 4.86 | - | 350 | 5.70 | 0.85 |
| 18 | 0.01024 | 0.0 | 0.00754 | 0.000800 | 1.46 | 4.86 | - | 350 | 5.11 | 0.95 |
| 19 | 0.01024 | 0.0 | 0.00933 | 0.000293 | 1.46 | 4.86 | - | 150 | 1.69 | 2.88 |
| 20 | 0.01024 | 0.0 | 0.00862 | 0.000510 | 1.46 | 4.86 | - | 250 | 2.84 | 1.73 |
| 21 | 0.01024 | 0.0 | 0.00795 | 0.000920 | 1.46 | 4.86 | - | 500 | 5.79 | 0.84 |
| 22 | 0.01024 | 0.0 | 0.00953 | 0.000463 | 3.40 | 4.86 | - | 150 | 2.31 | 2.10 |
| 23 | 0.01024 | 0.0 | 0.00937 | 0.000576 | 3.40 | 4.86 | - | 250 | 3.47 | 1.40 |
| 24 | 0.01024 | 0.0 | 0.00879 | 0.000980 | 3.40 | 4.86 | - | 500 | 7.47 | 0.65 |
| 25 | 0.01024 | 0.0 | 0.01015 | 0.000565 | 4.86 | 1.46 | - | 250 | 0.88 | 1.66 |
| 26 | 0.01024 | 0.0 | 0.00940 | 0.000236 | 1.46 | 4.86 | - | 50 | 0.95 | 5.90 |
| 27 | 0.01024 | 0.0 | 0.00877 | 0.000953 | 3.40 | 4.86 | - | 400 | 6.44 | 0.75 |
| 28 | 0.01024 | 0.0 | 0.00788 | 0.000576 | 1.94 | 7.77 | - | 250 | 6.56 | 1.18 |

S - H  Spray Head

because of its large affect on column efficiency. The experimental results showed that for equivalent rates of flow the spray-head, with no agitation, gave column efficiencies equal to those obtained when the column was being agitated at a speed of 250 rpm. The extraction coefficients for Runs 1 and 2 averaged 0.84 pound moles per (hour)(square foot)(unit $\Delta C$) while the H.T.U. values averaged 5.5 feet

Experimental Runs 5 through 14, made at increasing rates of agitation with the rate of flow of both phases held constant, show that under various conditions of agitation the extraction coefficients are increased four to ten fold and the values for H.T.U. are decreased from 5.5 feet at zero agitation to 0.6 feet at 600 rpm.

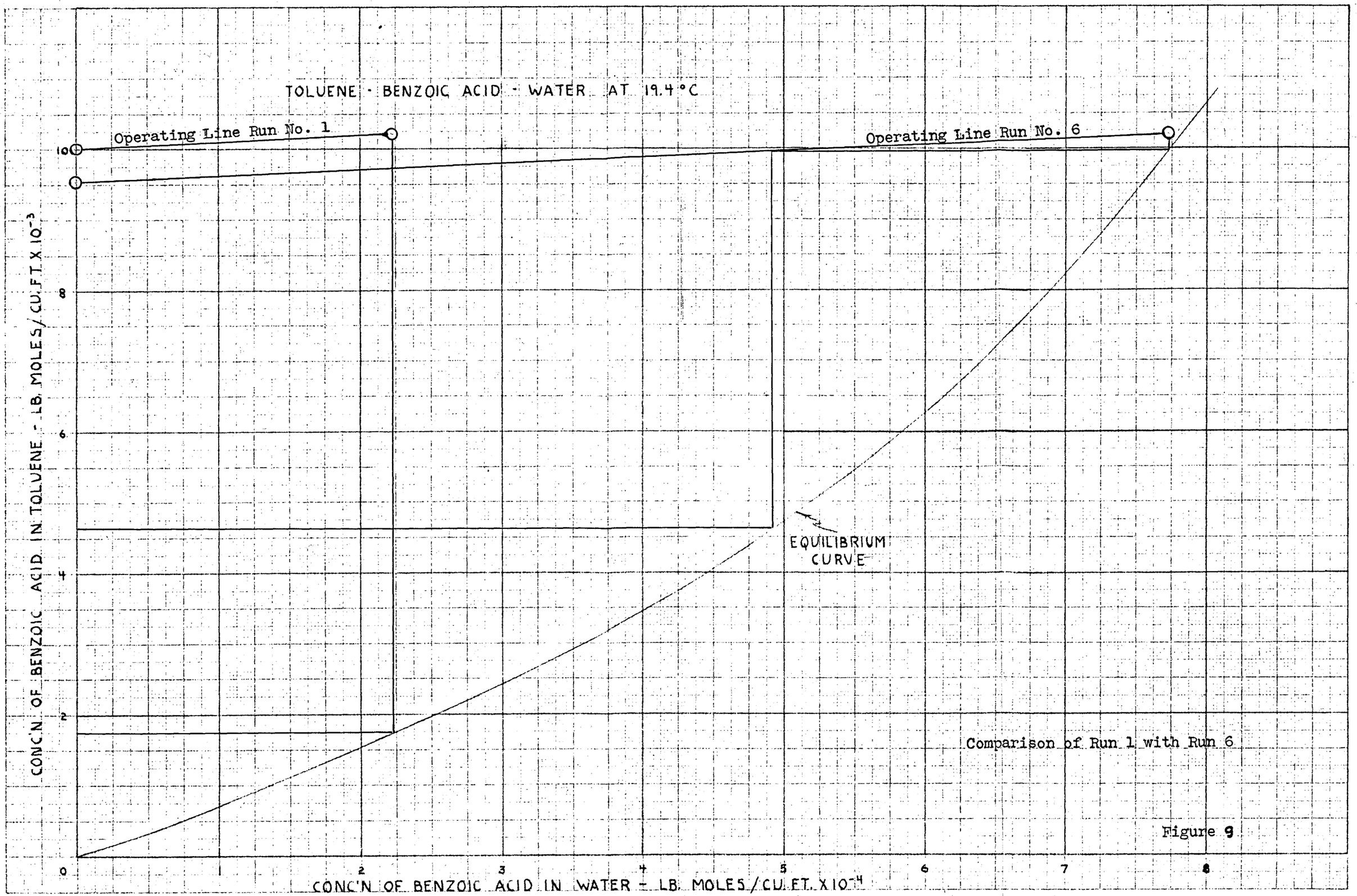
The increase in efficiency of the column under agitation is shown in Figure 9 in which Run 1 is compared to Run 6 (agitator speed 250 rpm). In the agitated run the water leaving the column is very nearly in equilibrium with the entering toluene phase. The number of equilibrium stages as measured by "stepping off" the curve is slightly more than one and one-half. The unagitated run as seen from Figure 9 departs widely from equilibrium at all points in the column. The number of equilibrium stages is somewhat difficult to estimate, but is certainly less than one-tenth.

In Runs 1 through 14 the flow rate of both phases fed to the column was kept equal. This method of operation produces a close approach to equilibrium or "pinch" at the

bottom of the column while the top of the column departs quite widely from equilibrium. Run 6 Figure 9 shows this effect quite well. Because of the "pinch" at the top of the column, it is difficult to estimate the number of equilibrium stages by the stepping off procedure. However, a practical idea of the improvement resulting from agitation may be gained by comparing the concentration of the acid in the effluent toluene streams.

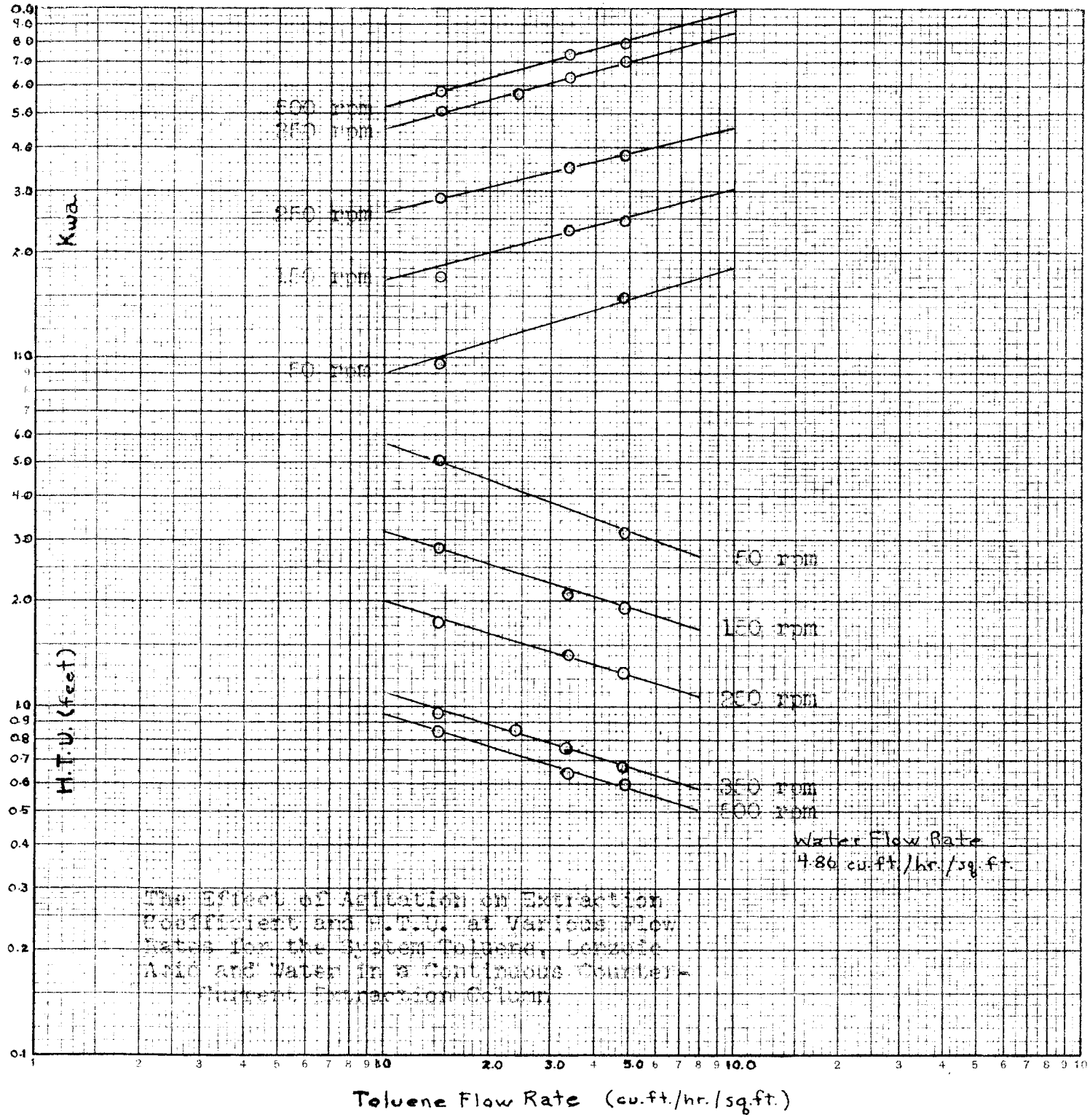
In order to obtain a correlation between the rate of flow of the dispersed phase and the extraction coefficient, a series of runs were made (Runs 15 through 28) in which the water feed rate was held constant and the toluene feed rate and the agitator speed were varied. Figure 10 is a correlation of both $K_{wa}$ and H.T.U. against flow rate of the dispersed phase. Examination of the data of this figure indicates that under proper conditions of agitation the extraction coefficient can be maintained at a high value with little regard to the rate of flow of the dispersed phase.

In considering the causes for the increased extraction coefficient under agitation, it is convenient to regard this quantity as the product of K, the overall coefficient per unit area, and of a, the area of interphase contact. It is evident that an increase in either one of these quantities will result in an increase in the value of the extraction coefficient $K_a$. An increase in K might result from a decrease in thickness of the stagnant film or either the




Comparison of Run 1 with Run 6

Figure 9




The Effect of Agitation on Extraction Coefficient and H.T.U. at Various Flow Rates for the System Toluene, Benzoic Acid and Water in a Continuous Counter-Current Extraction Column

Figure 1

water or the toluene side of the interface or both. An increase in the quantity a could be due not only to the finer subdivision of the dispersed phase, but also to the fact that the total holdup of the dispersed phase is increased because of the slower rate of rise of the finer droplets. In view of the fact that the experimental evidence of this work shows an increase in the total holdup of the dispersed phase in addition to the increasingly finer dispersion obtained with greater agitation the chief effect of the agitation is on (a) the area of contact between the phases. However, the possibility of a minor effect of agitation on K cannot be completely ruled out.

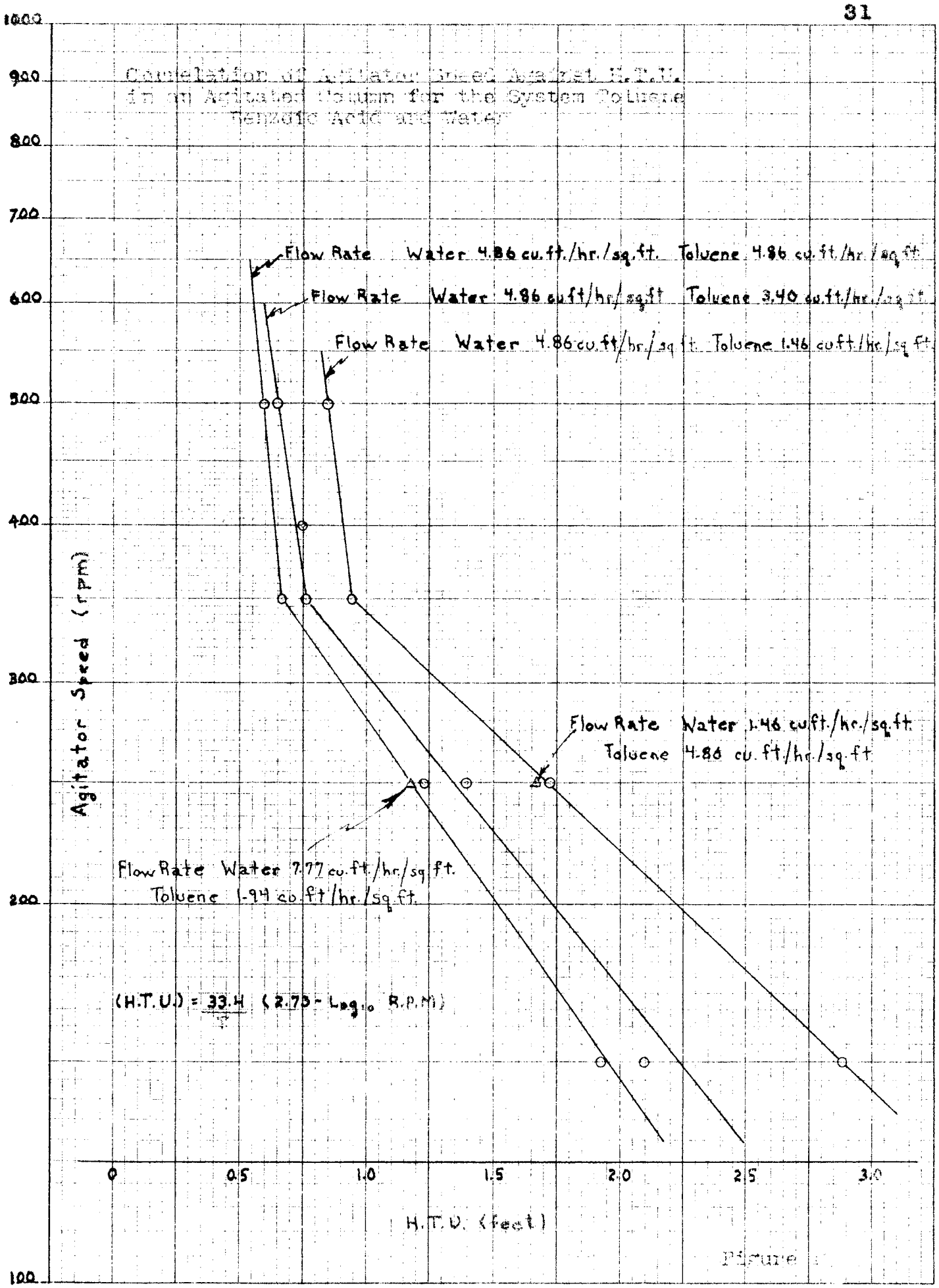
Feick and Anderson (7) correlated data collected from the literature with data from their own work and found that the values of $K_{wa}$ for their agitated runs exceeded those for packed and spray columns at corresponding toluene rates. It is unfortunate that these investigators varied both flow rate and agitator speed indiscriminately and were not able to make a complete correlation of their own work against that which they collected from the literature.

Examination of some of the published work (8) in unagitated columns where experimental runs were made with the more conventional packing ($\frac{1}{2}$-inch and $\frac{1}{4}$-inch Raschig rings) showed efficiencies based on H.T.U. averaging about 3 feet. During the course of this work other investigators from this laboratory obtained data on this same system using a 1.0 inch Scheibel agitated column. Using the same thruput

as in Runs 1 through 14 of this work and an agitator speed of 2060 rpm, these investigators obtained values of $K_{wa}$ and H.T.U. of 2.08 pound moles per (hour)(square foot)(unit $\Delta C$) and 2.16 feet respectively.

Another method of correlating the data of Table 1 is shown in Figure 11 where H.T.U. is plotted against agitator speed. This plot shows three curves each representing a different total thruput through the column. Figure 11 also shows two individual points, Run 28 and Run 28, which were made at the same thruput as the curves they correspond to, but, with different toluene and water rates.

Examination of Figure 11 shows a break (change of slope) in each of the three curves corresponding to a value of 380 rpm for agitator speed. Observations made during the experimental work showed an increase in holdup of the toluene phase with an increase in agitator speed. Table 2 shows toluene holdup for various agitator speeds. During the course of the experimental work several unsuccessful runs were made at agitator speeds of 800 rpm. These runs were thought to be primarily unsuccessful because of emulsification of the two phases, however, it was found that at this speed the column became flooded. Examination of the apparatus showed that the toluene had become the continuous phase and the water the dispersed phase. The break in the curves is therefore thought to be the point at which stage inversion occurs in the column with this particular system. A further examination of Figure 11 shows that for thruputs plotted, any increase in agitation


Correlation of Agitator Speed Against H.T.U.
in an Agitated Column for the System Toluene
Benzoic Acid and Water
Flow Rate Water 4.86 cu.ft./hr./sq.ft. Toluene 4.86 cu.ft./hr./sq.ft.
Flow Rate Water 4.86 cu.ft/hr/sq.ft Toluene 3.40 cu.ft/hr./sq.ft.
Flow Rate Water 4.86 cu.ft/hr./sq.ft. Toluene 1.46 cu.ft./hr./sq.ft.
Flow Rate Water 1.46 cu.ft./hr./sq.ft.
Toluene 4.86 cu.ft./hr./sq.ft.
Flow Rate Water 7.77 cu.ft./hr./sq.ft.
Toluene 1.94 cu.ft./hr./sq.ft.
(H.T.U.) = 33.4 / T (2.75 - Log10 R.P.M.)
Agitator Speed (rpm)
H.T.U. (feet)
1000
900
800
700
600
500
400
300
200
100
0
0.5
1.0
1.5
2.0
2.5
3.0

Figure

TABLE 2

| Agitator Speed rpm | Column Holdup cubic feet |
|---|---|
| 50 | 0.0009 |
| 150 | 0.0023 |
| 250 | 0.0056 |
| 350 | 0.0114 |
| 500 | 0.0191 |

Volume of working section of column = 0.0437 cubic feet

above 350 rpm produces only an extremely small change in the value of H.T.U.

As was previously stated Run 25 and Run 28 were made in order to check the effect of varying the ratio of the rate of flow of both phases while at the same time maintaining the same total thruput through the column. Run 25 (toluene rate 4.86 cubic feet per hour per square foot - water rate 1.46 cubic feet per hour per square foot) corresponds to Run 20 where the flow ratio is reversed. Run 28 (toluene rate 1.94 cubic feet per hour per square foot - water rate 7.77 cubic feet per hour per square foot) corresponds to Run 6 (toluene rate 4.86 cubic feet per hour per square foot - water rate 4.86 cubic feet per hour per square foot). Examination of Figure 11 shows that Run 25 lies extremely close to Run 20 while Run 28 lies in the same relative position to Run 6.

From the above data it was determined that total column thruput was the influencing factor in determining H.T.U. values for any given agitator speed and the following analysis was made on the lower portion of the three curves of Figure 11 in order to obtain an equation which would satisfy the experimental data. The lower portions of the three curves of Figure 11 (below 350 rpm) fit the equation $\log y = Sx + I$, where $y$ = agitator speed, $S$ = slope of curve, $x$ = H.T.U. and $I$ = intercept value. A plot of S versus thruput (T) for the curves gives $T/S$ = Constant = 33.4. Extrapolation of the

three curves to the y axis shows that the three curves pass through the point x = 0 y = 537. This point corresponds to an intercept value of $\log_{10}$ of 537 = 2.73. Substitution of these values in the original equation gives:-

$$\log_{10} rpm = 2.73 - \frac{T}{33.4} (H.T.U.)$$

Rearrangement of the equation gives:-

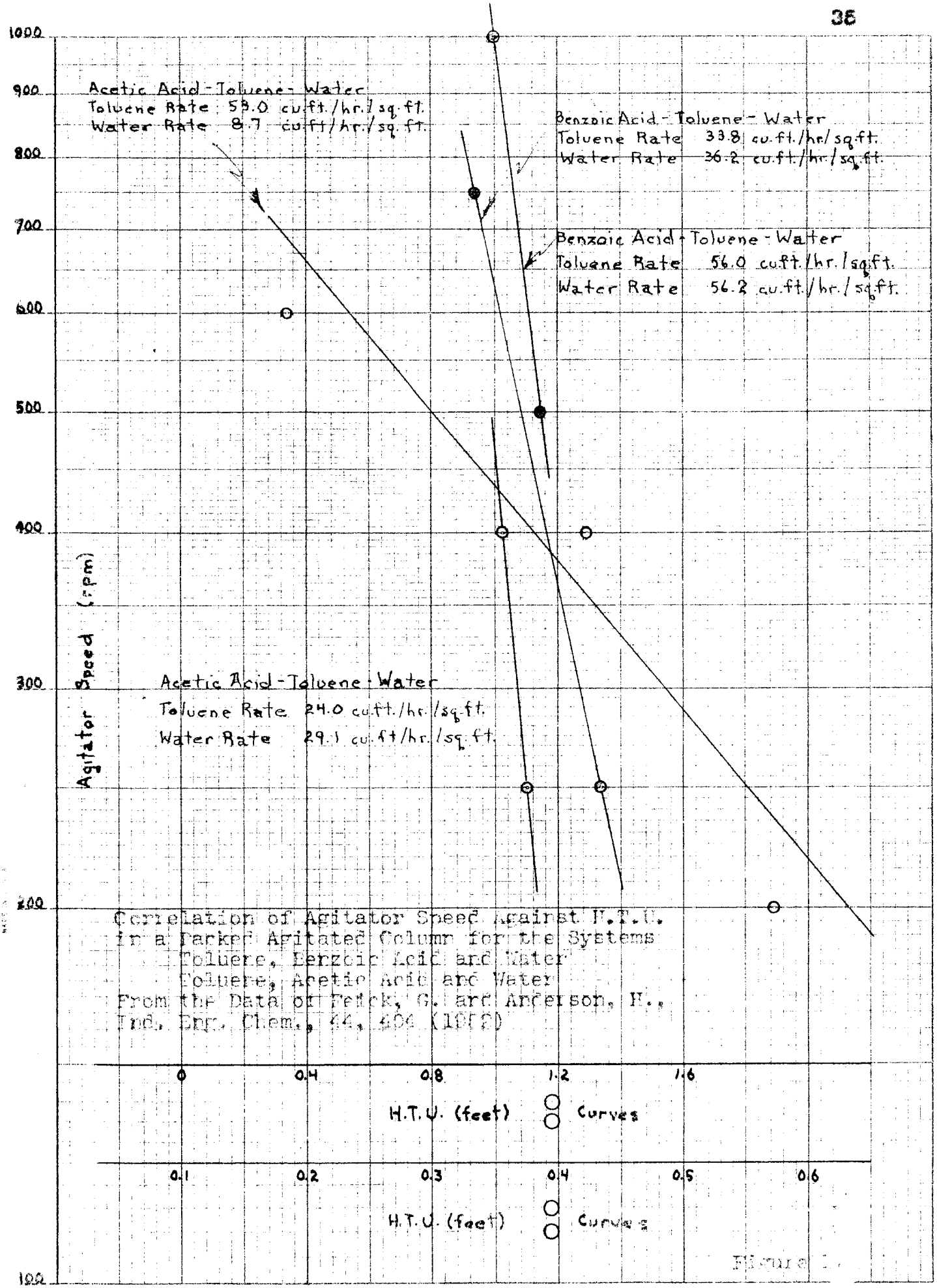
$$H.T.U. = \frac{33.4}{T} (2.73 - \log_{10} rpm)$$

In order to check the accuracy of the derived equation, several points on the curves were calculated mathematically using the same values for flow rate and agitator speed as were used in the experimental work. These are listed below;-

| Run No. | Experimental Value H.T.U. | Calculated Value H.T.U. |
|---|---|---|
| 12 | 1.94 | 1.91 |
| 16 | 0.76 | 0.77 |
| 18 | 0.95 | 0.98 |
| 19 | 2.88 | 2.88 |

As was previously stated Feick and Anderson (7) made no attempt to obtain a correlation of agitator speed, total thruput and H.T.U. from their experimental data. However, this investigator after a careful check of their paper attempted to correlate some of the results in the same manner as was done with the data of this work in Figure 11.

Figure 12 is a plot of the experimental data of Feick and Anderson (ibid), however, before any comparison is made it should be stated that there is not sufficient data avail-




Correlation of Agitator Speed Against H.T.U. in a Parker Agitated Column for the Systems Toluene, Benzoic Acid and Water Toluene, Acetic Acid and Water From the Data of Petrick, G. and Anderson, H., Ind. Eng. Chem., 44, 404 (1952)

Figure 1.

able in their work for a complete analysis. The main point of similarity between the two sets of data is in the type and slope of curve obtained in the region above 250 rpm. The three curves designated by ● ● and ● are believed to be portions of curves similar to those of Figure 11 of this work, however, all of the points on these curves are believed to be above a point which would correspond to the break points of the curves of Figure 11. It is unfortunate that no data is available in Feick and Anderson's work at lower agitator speeds for the thruputs plotted in Figure 11.

In order to compare the results from this column with those from other types of extraction columns it is best to use results with the same chemical system, overall acid concentration, thruput and column diameter. Data are available for the same chemical system, but these are not exactly comparable data for the remainder of the above mentioned conditions. Some of the highest efficiencies reported by other investigators using the system toluene, benzoic acid and water are those reported by Feick and Anderson (ibid) in their packed agitated column. A comparison of H.T.U. values obtained in this work with those reported by these investigators shows that the increase in efficiency of both columns is of the same order of magnitude. The present investigation shows that the agitated extraction column investigated here has an extremely high efficiency when compared with the efficiencies of conventional packed and

spray columns. For example, the H.T.U. of this column is of the order of one-fourth to one-fifth of the values for packed columns under similar conditions of operation. The thruput of the packed column is somewhat greater. However, packed columns are usually not operated close to the flooding point since the maximum efficiency occurs at a lower thruput. Since no packing or settling zones are used in this column it is easier to keep clean and can better handle materials containing suspended solids. Lastly the column is an extremely flexible piece of equipment with regard to variable thruput and agitator speed.

## SUMMARY

A substantial increase in extraction efficiency is obtained thru the use of agitation in counter current liquid-liquid extraction columns. The large extraction coefficients and low values of H.T.U. obtained under agitation indicate that the size of the agitated column may be much less than that of the standard design spray or packed column. Since by proper control of agitation, the extraction coefficient can be made nearly independent of the dispersed phase flow rate, the agitated column is a more flexible piece of equipment than its stationary counterpart and will be better able to handle processing fluctuations with a minimum of change in product quality or recovery. For the system used in this work (benzoic acid-toluene-water) the Height of a Transfer Unit can be found mathematically from the equation:-

$$H.T.U. = \frac{33.4}{T} (2.73 - \log_{10} R.P.M.)$$

## LITERATURE CITED


(1) Appel, F.J. and Elgin, J.C., Ind. Eng. Chem., 29, 451 (1937)

(2) Morello, V.S., and Poffenberger, N., Ind. Eng. Chem., 42, 1021 (1950)

(3) van Dijck, N.J.D., U.S. Patent 2,011,186 (1935)

(4) Othmer, D.F., U.S. Patent 2,000,806 (1935)

(5) Mc Connell, E.E., U.S. Patent 2,091,645 (1937)

(6) Scheibel, E.G., U.S. Patent 2,493,261 (1950)

(7) Feick, G. and Anderson, H.M., Ind. Eng. Chem., 44, 404 (1952)

(8) Row, S.B., Koffolt, J.H. and Withrow, J.R., Trans. Am. Inst. Chem. Engrs., 37, 559 (1941)

(9) Fick, A., Ann. Physick., 94, 59 (1855)

(10) Maxwell, J.C., Phil. Trans. Royal Soc., 157, 49 (1866)

(11) Stefan, J., Wien. Silzungsber, 63, 63 (1871)

(12) Arnold, J.H., J. Am. Chem. Soc., 52, 3937 (1930)

(13) Eyring, H., J. Chem. Phys., 4, 283 (1936)

(14) Lewis, W.K. and Whitman, W.G., Ind. Eng. Chem., 16, 1215 (1924)

(15) Treybal, R.E., Liquid Extraction, Mc Graw - Hill Book Company, Inc., New York 1951

(16) Perry, H.H. Ed., Chemical Engineers Handbook, 3rd Ed., Mc Graw - Hill Book Company, Inc., New York 1950

(17) Laddha, G.S., and Smith, J.M., Chem. Eng. Prog., 46, 195 (1950)

(18) Hunter, T.C. and Walsh, A.W., J. Soc. Chem. Ind., 51 285 T (1932)

(19) Elgin, J.C. and Browning, F.M., Trans. Am. Inst. Chem. Engrs., 31, 639 (1935); 32, 105 (1936)

(20) Chilton, T.H. and Colburn, A.P., Ind. Eng. Chem., 27, 255-60, 904 (1935)

(21) Colburn, A.P., Trans. Am. Inst. Chem. Engrs., 35, 211 (1939)

(22) Sherwood, T.K., Absorption and Extraction, 1st Ed., Mc Graw - Hill Book Company, Inc., New York 1937

(23) Oldshue, J.Y. and Rushton, J.H., Chem. Eng. Prog., 48, 297 (1952)

## APPENDIX

Equilibrium Data for the System
Toluene-Benzoic Acid-Water at 19.4°C

| Concentration Of Benzoic Acid in Toluene pound moles/cubic foot | Concentration Of Benzoic Acid in water pound moles/cubic foot |
|---|---|
| 0.00042 | 0.000063 |
| 0.00131 | 0.000172 |
| 0.00211 | 0.000268 |
| 0.00273 | 0.000332 |
| 0.00395 | 0.000442 |
| 0.00458 | 0.000490 |
| 0.00590 | 0.000578 |
| 0.00662 | 0.000621 |
| 0.00770 | 0.000677 |
| 0.00928 | 0.000745 |
| 0.01040 | 0.000789 |