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AN ORIFICE PLATE PULSE COLUMN FOR LIQUID-LIQUID EXTRACTION

By

David C. O'Brien

Submitted in Partial Fulfillment of the
Requirements for the Degree of Master
of Science from the Massachusetts
Institute of Technology
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ABSTRACT

A study was made of the performance of an orifice plate pulse column which is essentially a spray column containing internal constrictions in the form of orifices. The chemical system studied was acetic acid-water-hexone, and the variables considered were pulse frequency, throughput, phase continuity, and direction of solute transfer. For comparison purposes, several runs were made with a conventional spray column.

Curves are presented showing the effect of the column variables on the column efficiency (HTU). The trends observed are explained as being due principally to the competing effects of the area for mass transfer and back-mixing. Photographs of a typical column section show qualitatively the effect of the important column variables.

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I. SUMMARY

During the past ten years liquid-liquid extraction has become one of the most important unit operations in the field of chemical engineering. Along with the development of new chemical systems, extraction devices also have been improved. The pulse column is an outstanding example of an improvement of vertical extraction columns. Investigations have shown that the addition of a low frequency pulse to a sieve plate column will increase the extraction efficiency while maintaining a high column throughput.

Previous investigators, interested in utilizing the advantages of the high throughputs afforded by spray towers, studied the effect of pulsing on spray tower operation. These investigators concluded that the effect of pulsing a spray tower was slight and that improvement could be obtained only by incorporating internal constrictions into the spray tower design. It was suggested that regularly spaced orifices would provide the necessary constrictions and yet would allow throughputs higher than those attainable in sieve plate pulse columns. Also, there might be cases where an orifice plate pulse column would have decided advantages over a sieve plate pulse column, such as for extraction from slurries.

The orifice plate pulse column used in this investigation consisted of individual sections of Pyrex pipe, 4 inches long with an internal diameter of 1-1/2 inches, fitted together with flanges and gaskets. The gaskets served as orifices and constricted the area for flow to 25 per cent of the column cross-sectional area. The purpose of this investigation was twofold:

- 1) To determine the effect of pulse frequency on extraction efficiency and discontinuous phase holdup over a range of throughputs, and
- 2) To determine the effect of pulse frequency on the maximum throughput attainable in an orifice plate pulse column.

The chemical system employed for this investigation was acetic acid, water and methyl-isobutyl ketone. Most of the data were taken with transfer of acetic acid into the organic phase (termed extraction) with the aqueous

phase dispersed. Aqueous feed concentration for the extraction runs was maintained approximately constant and equal to 14 wt. per cent acetic acid. Some data were also obtained with the organic phase discontinuous, again for extraction runs. Other runs were made on the regeneration of organic solvent (termed stripping) with the aqueous phase dispersed. Five extraction runs were made with a spray tower (a single straight section substituted for the orifice sections) with the ketone as the dispersed phase.

For transfer of acetic acid into the organic phase, solute-free aqueous flow rates ranged from 75 to 400 grams per minute. The ratio of organic flow to aqueous flow was held approximately constant at 1.3 gms. organic/gm. aqueous (on a solute-free basis). Pulse height was also held constant throughout the investigation at a value of 1/2 inch. Frequencies were varied from 0 to 200 cycles per minute, with the exception of one spray tower run which was pulsed at 300 cycles per minute. Photographs were taken of orifice plate pulse column operation to show changes in drop size, holdup and dispersion.

Extraction efficiencies were expressed in terms of overall HTU's, calculated from log mean driving forces. HTU values for extraction runs varied from 35 to 20 inches at low throughputs and from 30 to 13 inches at high throughputs. The lowest HTU values were obtained at high frequencies. One spray tower run at intermediate flow rates and 300 cycles per minute resulted in an HTU of 11 inches. The flooding range curve exhibited a maximum at 100 cycles per minute. Combined flow rates at flooding varied from 550 gm./min. (solute-free basis) at 0 and 180 cycles per minute to 1200 gm./min. at the maximum.

On the basis of the results of this investigation it was concluded that: (1) pulsing tended to increase holdup and improve extraction efficiency, particularly at high frequencies and at high throughputs, (2) pulsing tended to create smaller drops and increase back-mixing, particularly at low throughputs, (3) an increase in throughput tended to increase holdup and improve extraction efficiency, particularly at high frequencies, and, (4) an increase in throughput tended to increase drop size and decrease back-mixing.

It was further concluded that (5) dispersion of the organic phase in preference to the aqueous phase, when solute transfer is into the organic phase, resulted in more holdup and increased transfer area. These factors increased the extraction efficiency at high flow rates and low frequencies. In addition, (6) pulsing in an orifice plate pulse column tended to increase maximum throughput by pumping action and to decrease maximum throughput by increasing holdup.

In view of the effects of pulsing in an orifice plate pulse column and the possibility that the potential of the spray tower as an extraction device may be increased by pulsing, it is recommended that: (1) a more detailed study be made on an orifice plate pulse column to determine the effect of frequency on average drop size, back-mixing and coalescence, perhaps with the aid of motion pictures and dyes, (2) studies be made to determine the effect of pulse height, cam shape and orifice size, shape and spacing on HTU, the factors influencing HTU, and flooding, and (3) studies also be made to determine the effect of pulsing on spray tower operation and extraction efficiency, with particular emphasis on the mechanism of dispersion and the effect of varied inlet design.

II. INTRODUCTION

Continuous countercurrent liquid-liquid extraction is one of the most fertile areas for chemical engineering research today. Although horizontal contactors and mixer-settler techniques are being developed, a major portion of solvent extraction is done in vertical columns (4). The latter's importance is evidenced by increased usage as well as the growing efforts of investigators to improve spray and packed tower operation.

Improvements may be classified in three categories: greater extraction efficiency (as measured by HTU, HETS, or per cent recovery), higher throughput and increased versatility. Considerable attention has been devoted to the development of better packing and the addition of mechanical agitators to columns in order to increase the available area for mass transfer. Although packing and other internal additions improve the extraction efficiency, they frequently have detrimental effects on throughput and versatility. Recent developments, however, have shown that applying a pulse to a packed column, or a column fitted with sieve plates, increases the extraction efficiency while maintaining a high column throughput (2, 4, 11).

Some of the preliminary sieve plate pulse column work was performed at the M.I.T. Engineering Practice School at Oak Ridge (1, 2, 6, 8, 10). In conjunction with this work it was suggested that high frequency pulsing of a spray column might favorably affect its operation as a contacting device. In November, 1952, two M.I.T. students, R.M. Lurie and R.G. Shaver, attempted to determine the effect of high frequency pulsing (7-12 cycles per second) on a spray column (7). Most of their work was done on a spray column constructed from glass sections which were joined at three points with flanges and gaskets. Through an oversight, the gaskets at these points constricted the cross-sectional area for flow to 63% of the column cross-section. It was found that pulsing this column improved extraction efficiency, lowering HTU by as much as a factor of three. Later, upon discovery of the constrictions it was realized that the results had been biased. The constricting gaskets were removed and replaced with gaskets of the proper diameter. The investigators observed

little improvement in dispersion and extraction efficiency relative to spray tower operation when this second column was pulsed. It therefore was concluded that pulsing would not improve spray column operation unless some constriction of flow was present.

The first column which Lurie and Shaver had constructed was basically a crude orifice plate pulse column. The orifice plate pulse column differs only slightly from the sieve plate pulse column in that the many small holes of the sieve plate are replaced by one large orifice. At the outset, such a column would appear to have several inherent advantages. Extraction efficiency would be improved in comparison to the spray tower, as observed by Lurie and Shaver (7). It also seems that the substitution of an orifice would increase the maximum throughput over that of a sieve plate pulse column under similar operating conditions. Furthermore, a single large hole would probably allow extraction from slurries, an operation in which plugging would pose a serious problem, especially in packed or sieve plate pulse columns. Construction and maintenance costs would probably be less than for a sieve plate pulse column.

In September, 1953, Myers, et. al. (9) constructed an orifice plate pulse column which successfully extracted material from a slurry. The report stated that a similar attempt by other investigators using mixer-settler equipment resulted in failure. In May, 1954, Buchanan, et. al. (3) constructed a similar column which achieved extraction efficiencies comparable to those of a sieve plate pulse column operating with the same system under the same flow conditions. The extraction efficiencies of the orifice plate pulse column were obtained at higher frequencies, but the maximum throughput was observed to be greater than for a sieve plate pulse column operating with the same pulse.

Important operating variables of an orifice plate pulse column are pulse height, pulse frequency, wave shape, and flow rates. Design variables include orifice size, column diameter, plate spacing and the nature of the chemical system. Data from Myers, et. al. (9) were on a complex chemical system and offered little aid in determining the effects of the above variables on extraction efficiency and maximum throughput.

Data from the latter project were not available at the start of this investigation.

Acetic acid, water, and methyl-isobutyl ketone (hexone), a chemical system used by many previous researchers, was employed in this investigation (1, 2, 5, 6, 7, 8, 11). This work constitutes a preliminary examination of pulse column variables. Its primary purpose was two-fold:

- 1) To study the effect of pulse on extraction efficiencies over a range of column throughputs.
- 2) To study the effect of pulse on the maximum throughput and holdup of the discontinuous phase.

Experimental work was conducted from March through July, 1954, at the Massachusetts Institute of Technology Engineering Practice School at Oak Ridge, Tennessee.

III. PROCEDURE

The orifice plate pulse column used in this investigation was 1-1/2 inches in diameter and contained 19 orifices spaced 4 inches apart. The orifice diameter was 3/4 inches, thus reducing the area for flow to 25 per cent of its normal value. The important auxiliary equipment consisted of a pulsing unit, Saran tubing, valves and several storage tanks. Three of these tanks were used as feed pumps by utilizing air as a displacement medium (See Appendix, A. Details of Apparatus). Figure 1 is a schematic flow diagram of the system and Figure 2 is a photograph of the equipment. Figure 3 is a closeup of the pulsing apparatus. All runs except five were made with the orifice plate pulse column. These five runs were made with a spray tower of the same dimensions (See Figure 4).

The major portion of the data were obtained for the transfer of acetic acid from water to hexone (methyl-isobutyl ketone). These runs were called extractions runs and the runs with reversed transfer were called stripping runs. For extraction runs, the aqueous feed concentration was held roughly constant at 14 per cent acetic acid by weight; the organic feed contained relatively little acid, usually 0.3 per cent or less.

At the start of a run, the pulsing unit was turned on and was set at the desired frequency. Frequencies ranged from 0 to 200 cycles per minute. Pulse amplitude in the column was 1/2 inches and was the same throughout the investigation. The pulse pattern was essentially a sine wave.

After adjusting the air pressure on the displacement pumps, flow of the continuous phase (usually organic) was then fixed at the operating level, and the interface was placed midway between the continuous phase inlet and the discontinuous phase outlet. The interface was controlled by varying the aqueous phase outflow with an overflow gravity leg. Flow of the discontinuous phase was then started and the interface was re-adjusted until the operation was stable.

Flows were controlled with needle valves and were set with the aid of rotameters. The majority of flow measurements were made on the outlet

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ORIFICE PLATE PULSE COLUMN AND AUXILIARIES

DATE	DRAWN BY	FILE NO.	FIG.
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CLOSEUP OF
PULSING APPARATUS

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SPRAY TOWER

DATE	DRAWN BY	FILE NO.	FIG.
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streams with a graduated cylinder and a stopwatch. Aqueous flows ranged from 75 - 400 gm. solute free solvent/min. The phase flow ratio was kept relatively constant at 1.3 gm. of solute free organic per gm. solute free aqueous.

The length of a run varied with the throughput and was roughly equal to the time required for six or seven changes of the continuous phase. Two samples, spaced thirty minutes apart, of both outlet streams were taken after the column had reached steady state. After the last samples were taken, flows were stopped and the two phases were allowed to separate. The interface was then returned to the operating position, and the overflow was collected in a cylinder. The overflow was equal to the discontinuous phase holdup.

The acid-rich hexone from extraction runs was stored and used as feed for stripping runs. Acid-depleted raffinate from extraction runs was discarded. The aqueous feed for stripping runs was tap water. Stripped hexone and the acid-rich aqueous product were reused as makeup for extraction run feeds.

Samples from all streams were analyzed for acid content by titration with sodium hydroxide to a phenolphthalein end point. The specific gravities of the samples were determined with hydrometers (See Appendix, B. Chemical System and Analytical Methods). Efficiencies were calculated from the concentrations in the form of HTU_{oa} (Height of a Transfer Unit, Overall, based on the aqueous phase). The effective height for extraction was assumed to be 85 inches.

Flooding was investigated at several frequencies for both aqueous and organic phases discontinuous. Approximately 15 minutes was allowed to determine if the column was flooded at any particular throughput. Throughputs were varied from low to high at discrete intervals. Aqueous discontinuous flooding occurred when the aqueous phase could not pass entirely through the first orifice and thus was partially pushed out the top of the column. Organic discontinuous flooding was defined as the throughput at which coalescence of the drops occurred tending to make the discontinuous phase continuous.

IV. RESULTS

The results of this investigation are presented in Figures 5 - 17 and Tables I and II. All results presented in the figures are for extraction runs. Figures 5, 6, 7, 10, and 11 respectively correspond to solute free aqueous flows of 75, 160, 240, 320, and 400 gm./min. and are plots showing HTU_{OO} , HTU_{Oa} and per cent discontinuous phase holdup over a frequency range of 0 - 180 cycles/min. These plots also show individual spray tower runs as well as runs which compare aqueous discontinuous runs with organic discontinuous runs.

Figures 8 and 9 are photographic studies of runs presented in Figure 7. Figures 12, 13, and 14 are photographs illustrating the effect on HTU of throughput and the reversal of phase continuity. Figure 15 is a plot of the flooding range of the orifice plate pulse column as a function of frequency, and Figure 16 is a photograph of organic discontinuous phase flooding. Figure 17 pictorially illustrates the five spray tower runs.

Table I lists the important values for the preliminary extraction runs at 30 cycles/min. A similar listing for the stripping runs is given in Table II.

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EFFECT OF PULSE ON
HTU AND HOLDUP

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EFFECT OF PULSE ON
 HTU AND HOLDUP

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ORIFICE PLATE PULSE COLUMN

SPRAY TOWER

△ ORGANIC DISCONTINUOUS

□ ORGANIC DISCONTINUOUS

○ AQUEOUS DISCONTINUOUS

SOLUTE FREE AQUEOUS FLOW - 240 GM./MIN. RANGE (233-252)

SOLUTE FREE ORGANIC FLOW - 320 GM./MIN. RANGE (302-331)

1/2" PULSE HEIGHT

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EFFECT OF PULSE ON
HTU AND HOLDUP

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EFFECT OF PULSE ON ORIFICE PLATE PULSE COLUMN OPERATION

UPSTROKE
 AQUEOUS FLOW - 240 GM./MIN.
 TOTAL FLOW - 560 GM./MIN.
 FREQ. - 45 RPM., RUN 13
 VOLUME HOLDUP - (N.D.)
 HTU_{00} - 28.7" HTU_{0a} - 33.7"

DOWNSTROKE
 AQUEOUS FLOW - 240 GM./MIN.
 TOTAL FLOW - 560 GM./MIN.
 FREQ. - 45 RPM., RUN 13
 VOLUME HOLDUP - (N.D.)
 HTU_{00} - 28.7" HTU_{0a} - 33.7"

AQUEOUS FLOW - 240 GM./MIN.
 TOTAL FLOW - 560 GM./MIN.
 FREQ. - 75 RPM., RUN 25
 VOLUME HOLDUP - 5.6%
 HTU_{00} - 22.5" HTU_{0a} - 27."

AQUEOUS FLOW - 240 GM./MIN.
 TOTAL FLOW - 560 GM./MIN.
 FREQ. - 105 RPM., RUN 37
 VOLUME HOLDUP - 7.2%
 HTU_{00} - 18.2" HTU_{0a} - 20.3"

NOT TO SCALE
 AQUEOUS FLOW - 240 GM./MIN.
 TOTAL FLOW - 560 GM./MIN.
 FREQ. - 120 RPM., RUN 43
 VOLUME HOLDUP - 7.4%
 HTU_{00} - 16.9" HTU_{0a} - 19.4"

AQUEOUS FLOW - 240 GM./MIN.
 TOTAL FLOW - 560 GM./MIN.
 FREQ. - 150 RPM., RUN 49
 VOLUME HOLDUP - 14.5%
 HTU_{00} - 14.2" HTU_{0a} - 16.1"

AQUEOUS DISCONTINUOUS
 1/2" PULSE HEIGHT
 FLOWS ON SOLUTE FREE BASIS

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ORIFICE PLATE PULSE
 COLUMN STUDIES

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EFFECT OF PULSE ON ORIFICE PLATE PULSE COLUMN OPERATION

DOWNSTROKE
AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 45 RPM., RUN 67
VOLUME HOLDUP - 12.0 %
HTU₀₀ - 16.0" HTU_{0a} - 17.8"

UPSTROKE
AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 45 RPM., RUN 67
VOLUME HOLDUP - 12.0 %
HTU₀₀ - 16.0" HTU_{0a} - 17.8"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 75 RPM., RUN 68
VOLUME HOLDUP - 13.9 %
HTU₀₀ - 13.0" HTU_{0a} - 14.2"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 105 RPM., RUN 69
VOLUME HOLDUP - 17.0 %
HTU₀₀ - 14.6" HTU_{0a} - 15.8"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 120 RPM., RUN 64
VOLUME HOLDUP - 22.3 %
HTU₀₀ - 13.5" HTU_{0a} - 16.2"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 150 RPM., RUN 70
VOLUME HOLDUP - 30.1 %
HTU₀₀ - 14.1" HTU_{0a} - 15.3"

HEXONE DISCONTINUOUS
1/2" PULSE HEIGHT
FLOWS ON SOLUTE FREE BASIS

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ORIFICE PLATE PULSE
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EFFECT OF PULSE ON
HTU AND HOLDUP

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EFFECT OF PULSE ON
HTU AND HOLDUP

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EFFECT OF PHASE CONTINUITY ON ORIFICE PLATE PULSE COLUMN OPERATION

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ.-105 RPM., RUN 37
VOLUME HOLDUP- 7.2%
HTU_{oo}-18.2" HTU_{oa}-20.3"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ.-120 RPM., RUN 43
VOLUME HOLDUP- 7.4%
HTU_{oo}-16.9" HTU_{oa}-19.4"

AQUEOUS FLOW- 240 GM./MIN.
TOTAL FLOW- 560 GM./MIN.
FREQ.-150 RPM., RUN 49
VOLUME HOLDUP - 14.5%
HTU_{oo}-14.2" HTU_{oa}-16.1"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW-560 GM./MIN.
FREQ.-105 RPM., RUN 69
VOLUME HOLDUP - 17.0%
HTU_{oo}-14.6" HTU_{oa}-15.8"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW-560 GM./MIN.
FREQ.-120 RPM., RUN 64
VOLUME HOLDUP - 22.3%
HTU_{oo}-13.5" HTU_{oa}-16.2"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ.-150 RPM., RUN 70
VOLUME HOLDUP - 30.1%
HTU_{oo}-14.1" HTU_{oa}-15.3"

1/2" PULSE HEIGHT
FLOWS ON SOLUTE FREE BASIS

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ORIFICE PLATE PULSE
COLUMN STUDIES

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EFFECT OF THROUGHPUT ON ORIFICE PLATE PULSE COLUMN OPERATION

AQUEOUS FLOW - 75 GM./MIN.
TOTAL FLOW - 180 GM./MIN.
FREQ. - 120 RPM., RUN 41
VOLUME HOLDUP - 3.7%
HTU_{oo} - 23.9" HTU_{oa} - 26.7"

AQUEOUS FLOW - 160 GM./MIN.
TOTAL FLOW - 375 GM./MIN.
FREQ. - 120 RPM., RUN 42
VOLUME HOLDUP - 6.2%
HTU_{oo} - 17.7" HTU_{oa} - 20.5"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 120 RPM., RUN 43
VOLUME HOLDUP - 7.4%
HTU_{oo} - 16.9" HTU_{oa} - 19.4"

AQUEOUS FLOW - 320 GM./MIN.
TOTAL FLOW - 740 GM./MIN.
FREQ. - 120 RPM., RUN 44
VOLUME HOLDUP - 8.8%
HTU_{oo} - 16.8" HTU_{oa} - 18.4"

AQUEOUS FLOW - 400 GM./MIN.
TOTAL FLOW - 940 GM./MIN.
FREQ. - 120 RPM., RUN 45
VOLUME HOLDUP - (N.D.)
HTU_{oo} - 17.7" HTU_{oa} - 19.1"

AQUEOUS DISCONTINUOUS
1/2" PULSE HEIGHT
FLOWS ON SOLUTE FREE BASIS

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
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CARBIDE AND CARBON CHEMICALS COMPANY
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ORIFICE PLATE PULSE COLUMN STUDIES

DATE 8-9-'54	DRAWN BY D.C.O'B.	FILE NO. EPS-K209	FIG. 13
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EFFECT OF THROUGHPUT ON ORIFICE PLATE PULSE COLUMN OPERATION

AQUEOUS FLOW - 75 GM./MIN.
TOTAL FLOW - 180 GM./MIN.
FREQ. - 120 RPM., RUN 62
VOLUME HOLDUP - 7.9 %
HTU₀₀ - 28.3" HTU_{0a} - 31.8"

AQUEOUS FLOW - 160 GM./MIN.
TOTAL FLOW - 375 GM./MIN.
FREQ. - 120 RPM., RUN 63
VOLUME HOLDUP - 13.7 %
HTU₀₀ - 17.4" HTU_{0a} - 20.3"

AQUEOUS FLOW - 240 GM./MIN.
TOTAL FLOW - 560 GM./MIN.
FREQ. - 120 RPM., RUN 64
VOLUME HOLDUP - 22.3 %
HTU₀₀ - 13.5" HTU_{0a} - 16.2"

AQUEOUS FLOW - 320 GM./MIN.
TOTAL FLOW - 740 GM./MIN.
FREQ. - 120 RPM., RUN 65
VOLUME HOLDUP - 29.4 %
HTU₀₀ - 13.8" HTU_{0a} - 15.2"

AQUEOUS FLOW - ~400 GM./MIN.
TOTAL FLOW - ~940 GM./MIN.
FREQ. - 120 RPM., NO RUN

HEXONE DISCONTINUOUS
1/2" PULSE HEIGHT
FLOWS ON SOLUTE FREE BASIS

MASSACHUSETTS INSTITUTE OF TECHNOLOGY ENGINEERING PRACTICE SCHOOL CARBIDE AND CARBON CHEMICALS COMPANY <small>A DIVISION OF UNION CARBIDE AND CARBON CORPORATION</small>			
ORIFICE PLATE PULSE COLUMN STUDIES			
DATE 8-9-'54	DRAWN BY D.C.O'B.	FILE NO. EPS-K209	FIG. 14

ORGANIC DISCONTINUOUS

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FLOODING IN AN ORIFICE PLATE PULSE COLUMN

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EFFECT OF THROUGHPUT & PULSE ON SPRAY TOWER OPERATION

AQUEOUS FLOW - 75 GM./MIN.
TOTAL FLOW - 180 GM./MIN.
NO PULSE - RUN 75
VOLUME HOLDUP - 1.8 %
HTU₀₀ - 35.2" HTU_{0a} - 39.9"

AQUEOUS FLOW - 150 GM./MIN.
TOTAL FLOW - 360 GM./MIN.
NO PULSE - RUN 76
VOLUME HOLDUP - 5.9 %
HTU₀₀ - 17.9" HTU_{0a} - 19.5"

AQUEOUS FLOW - 225 GM./MIN.
TOTAL FLOW - 545 GM./MIN.
NO PULSE - RUN 77
VOLUME HOLDUP - 9.1 %
HTU₀₀ - 13.3" HTU_{0a} - 14.4"

AQUEOUS FLOW - 225 GM./MIN.
TOTAL FLOW - 545 GM./MIN.
FREQ. - 200 RPM. - RUN 78
VOLUME HOLDUP - 10.8 %
HTU₀₀ - 13.4" HTU_{0a} - 14.2"

AQUEOUS FLOW - 225 GM./MIN.
TOTAL FLOW - 545 GM./MIN.
FREQ. - 300 RPM. - RUN 79
VOLUME HOLDUP - 14.9 %
HTU₀₀ - 11.4" HTU_{0a} - 12.0"

AQUEOUS FLOW - 225 GM./MIN.
TOTAL FLOW - 545 GM./MIN.
FREQ. - 500 RPM.

HEXONE DISCONTINUOUS
1/2" PULSE HEIGHT
FLOWS ON SOLUTE FREE BASIS

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SPRAY TOWER STUDIES

DATE 8-6-'54	DRAWN BY D.C.O'B.	FILE NO. EPS-K209	PAGE 17
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TABLE I

PRELIMINARY EXTRACTION RUNS (Aqueous Discontinuous)

Run No.	Frequency RPM.	Q_a Solute Free Aqueous Flow gm./min.	Q_o Solute Free Organic Flow gm./min.	Q_o/Q_a	Per Cent Deviation Material Balance	HTU _{oo} Inches	HTU _{oa} Inches
1	30	83.0	160	1.93	+0.8	45.9	33.9
2	30	166	160	0.96	-1.9	25.9	36.2
3	30	332	160	0.48	-0.2	---pinch---	
4	30	83.0	320	3.86	-6.9	91.8	31.1
6	30	165	317	1.92	-0.3	45.4	32.9
7	30	330	317	0.96	-1.1	22.6	31.6
8	30	74.2	63.2	0.85	+0.8	27.2	63.2
9	30	152	62.7	0.41	+0.8	---pinch---	

TABLE II

STRIPPING RUNS (Aqueous Discontinuous)

Run No.	Frequency RPM.	Q_a Solute Free Aqueous Flow gm./min.	Q_o Solute Free Organic Flow gm./min.	Q_o/Q_a	Per Cent Deviation Material Balance	HTU _{oo} Inches	HTU _{oa} Inches
5	45	472	345	0.73	-4.5	9.7	20.4
10	75	730	535	0.72	+7.8	9.6	17.5
16	75	652	545	0.84	+5.3	9.4	15.3
22	60	627	574	0.92	+3.2	12.6	19.2
28	90	717	669	0.93	+4.8	12.4	18.3
34	105	626	567	0.90	+4.5	9.9	17.9
40	120	406	364	0.90	+6.8	9.2	13.5
46	120	518	470	0.91	+4.3	11.5	16.4
52	200	215	180	0.84	+7.9	15.5	24.2
58	105	629	556	0.88	+2.4	12.0	18.7
59	105	642	546	0.85	+9.1	10.7	16.3
66	120	518	461	0.89	+8.5	9.5	13.7

V. DISCUSSION OF RESULTS

A. Precision and Accuracy of Results

The calculations for this investigation were based on the assumption that the operating line was straight. Such an assumption was in error. In several of the preliminary extraction runs, inlet flow rates were measured with rotameters at the same time that outlet flow rates were determined by the "graduate and stopwatch" method. The latter method was used in nearly all of the runs. A comparison of the two measurements showed that the amount of water removed from the bottom of the column was less than the amount of water entering at the top. A similar comparison indicated more organic solvent leaving the column than had entered. From these observations, it was evident that some water was transferred to the organic phase.

These facts were consistent with a phase diagram used by Belaga and Bigelow (3) for the acetic acid, water, hexone system. Over the range of concentrations in the column the solubility of water in hexone practically doubled with the increase of acetic acid content in the hexone, whereas the solubility of hexone in water changed very little. Thus the use of mutually saturated feeds did not prevent the extraction of water into the hexone. The transfer observed was roughly 5% of the entering water. Measurements of solute free flow rates therefore were biased as evidenced by a consistently positive deviation in material balances (See Appendix, E. Summary of Data and Calculated Values). Moreover, the HTU values were also biased due to the change in flow rates along the column and the effect of the change on "K", the overall mass transfer coefficient, and "a", the transfer area per unit volume of column.

Another major source of error was the simplifying choice of a log mean driving force for the calculation of HTU. The equilibrium line is slightly curved (See Figure 20, Appendix). By graphical integration of a plot of concentration versus the reciprocal of the

driving force, it was found that the HTU values calculated with the assumption were too high and thus conservative. The magnitude of the error increased as the HTU became smaller. The error was 11% for HTU values of 13 inches and 6% for HTU values of 24 inches (See Appendix, D. Sample Calculations).

HTU values were also conservative from another standpoint. About 6 inches of the total 85 inches chosen as the column extraction length were not representative of true countercurrent flow. This 6 inches was the distance between the continuous phase inlet and the interface. No estimate was made of the portion of extraction which took place in this section. The interface was kept midway between the continuous phase inlet and the discontinuous phase outlet.

Accuracy of the equilibrium curve also constituted a source of error. The data used (See Appendix, B. Chemical System and Analytical Methods) were for a temperature range of 82 - 90 °F. and were obtained from Belaga and Bigelow (2). According to these investigators, the data were substantially in agreement with Sherwood, Evans and Longcor (Ind. Eng. Chem., 31, 1144 (1939)) but not with Othmer, White, and Trueger (Ind. Eng. Chem., 33, 1240, (1941)). Room temperatures were recorded in this study as an indication of tower temperatures and varied from 75 to 95 °F. A comparison of equilibrium data by Fleming and Johnson (5) taken over a range of 69 - 75 °F. with those of Belaga and Bigelow indicates that the error due to change in equilibrium with temperature is probably slight. Since the equilibrium curve was fixed, the lower HTU values had more per cent error due to smaller driving forces.

Normalities were reported to 4 significant figures with a precision of 0.5%. Concentrations of samples taken from the same outlet stream as an indication of steady state operation usually checked within 1 - 2%. In several cases the first sample disagreed with the second by as much as 10%. The majority of these samples were of the aqueous phase taken from aqueous discontinuous runs at low throughput. Material balances for these runs usually agreed more closely when concentrations of the second samples were used.

At steady state operating conditions, checks on volume flow measurements were found to vary by as much as 3%. The combined error of the graduate and stopwatch was estimated to be 0.5%. The remaining error was attributed to fluctuations in inlet flows, interface level variations, slugging in the outlet lines and imprecisions in the measuring techniques. Hydrometers were calibrated to 1 part in the third decimal place. Errors in specific gravity measurements due to temperature variations were estimated to be somewhat larger than reading errors of the hydrometers, but were less than 1%. Pulse height was measured to the nearest 1/32 inch and error in the frequency measurement was negligible.

Holdup data probably were no more accurate than 10%. Measuring techniques probably contributed most to the scatter of the data. Quick closing valves were not used, thus making it difficult to shut off flows quickly and simultaneously. Some error was undoubtedly due to actual operating fluctuations.

Flooding data similarly do not have a high degree of accuracy, primarily because the degree of coalescence was somewhat arbitrary and flooding was a transient phenomenon. Error in flooding determination also was partly due to the use of discreet values of throughput rather than a continuous spectrum. Quantitative application of the flooding data to other columns is not advisable since the effect of the inlet constrictions at the distributor nozzles was not known.

B. Factors Affecting HTU

The following relationships were used in expressing extraction efficiencies based on the organic phase: (Similar equations were used for calculations based on the aqueous phase.)

$$NTU_{oo} = \int_{Y_1}^{Y_2} \frac{dY}{(Y^* - Y)} = \int_0^Z \frac{Ka S dZ}{Q_o}$$

$$HTU_{oo} = Z / NTU_{oo}$$

where HTU_{oo} = Height of a Transfer Unit, overall, based on the organic phase
 Z = Effective height for extraction
 NTU_{oo} = Number of Transfer Units, overall, based on the organic phase
 K = Overall mass transfer coefficient
 a = Area for mass transfer per unit column volume
 S = Column cross-sectional area
 Q_o = Organic flow rate
 Y = Concentration of solute in the organic phase

Analysis of the effect of a variable on extraction efficiency thus requires an examination of the effect of that variable on "K", "a", and the validity of the assumption that the operation truly can be represented by a differential equation.

The validity of the assumption of differential operation is reduced by stage-wise operation. Moreover, representation by differentials would not present a true picture if the column operated with back-mixing, a phenomenon which effectively reduces the number of countercurrent stages available for extraction. Back-mixing is essentially a localized reversal of countercurrent flow to cocurrent flow which tends to mix higher concentrations of a phase with lower concentrations of that phase. Since back-mixing would therefore tend to lower the driving force for mass transfer throughout the column, it would consequently tend to increase the HTU .

True spray column operation may be considered as differential, whereas studies by Gavin and Thies (6) show that a sieve plate pulse column essentially operates as a stage-wise device. Idealized operation of the columns is based on the assumption that little or no back-mixing occurs in a non-pulsed spray column or in a sieve plate pulse column, because the plates allow phase separation at each stage. Back-mixing would be much more probable in an orifice plate pulse column than in a sieve plate pulse column due to the lower resistance to flow of the orifice.

As the frequency of pulsing is increased, the acceleration of the fluids is also increased, tending to increase the shear forces on the discontinuous phase at the walls and the orifice plates. It would seem logical, therefore, that average drop size would decrease with increased frequency. Visual observations showed that drop size did vary inversely with frequency (See Figures 8, 9). Stoke's Law predicts that small drops move more slowly through the continuous phase than large drops because they have more surface resistance per unit mass. A decrease in drop size of the discontinuous phase at constant throughput would mean an increase in the discontinuous phase holdup. The results are in accord with this reasoning and show an increase in holdup with pulsing, particularly at higher frequencies (See Figures 5-11). The combination of smaller drops and increased holdup, with the related possibility that small, slow-moving drops would be carried cocurrently with the continuous phase, suggests that back-mixing would increase with frequency.

As the flooding range was approached, agglomeration and coalescence of the drops occurred as a result of increased holdup. Although coalescence would tend to increase the average drop size, it would also tend to reduce back-mixing to some extent. Current theories suggest that coalescence followed by regeneration of surface area tends to increase transfer and lower the HTU (2, 12). At very low frequencies coalescence occurred due to a longer settling time afforded the larger drops on the reverse pulse stroke (See Figures 8, 9).

The following is a list of factors influencing HTU which are affected by pulsing:

Effect of Increased Pulsing Below the Flooding Range

<u>Factor</u>	<u>Change in Factor</u>	<u>Independent Change in "a"</u>	<u>Independent Change in "K"</u>	<u>Independent Change in HTU</u>
drop size	decrease	increase	----	decrease
terminal drop velocity	decrease	----	decrease	increase
holdup	increase	increase	----	decrease
back-mixing	increase	----	----	increase
turbulence and drop oscillation	increase	----	increase	decrease
coalescence and regeneration of surface area	decrease	----	decrease	increase

Several investigators have found "Ka" for studies on spray and packed columns to vary almost linearly with the discontinuous phase flow rate up to the flooding point (10). The effect of the continuous phase flow rate on "Ka" is small but not negligible (10). Assuming that the distributor produced drops of approximately the same size over the range of throughputs investigated, any change in drop size with increased throughput would be due primarily to increased coalescence. Lurie and Shaver (7) found that the average drop size in their orifice plate column increased with an increase in the dispersed phase flow rate. Photographs for this investigation (See Figures 13 and 14) were consistent with their observations. Since the pictures and the data showed a considerable increase in holdup with throughput, it was concluded that increased throughput affected "Ka" primarily by increasing "a", regardless of the increase in drop size. An increase in drop size, however, would tend to increase terminal velocity by Stoke's Law (10) and thus to raise "K". Moreover, increased turbulence and drop oscillation would also tend to raise "K". Since the drop size did change appreciably with throughput, especially when the aqueous phase was dispersed, it is believed that back-mixing decreased with increasing throughput.

The following is a list of factors influencing HTU which are affected by throughput:

Effect of Increased Throughput Below the Flooding Range

<u>Factor</u>	<u>Change in Factor</u>	<u>Independent Change in "a"</u>	<u>Independent Change* in "K"</u>	<u>Independent Change in HTU</u>
flow rate for calculation basis	increase	----	----	increase
drop size	increase	decrease	----	increase
terminal drop velocity	increase	----	increase	decrease
holdup	increase	increase	----	decrease
turbulence and drop oscillation	increase	----	increase	decrease
back-mixing	decrease	----	----	decrease
coalescence and regeneration	increase	----	increase	decrease

* Dependent only on change in factor, independent of the other effects of throughput.

04/1
40

Choice of a particular phase to be discontinuous is dependent upon at least three factors. Normally the phase receiving solute is dispersed in order to reduce coalescence (5, 10). In order to increase surface area, the phase which preferentially wets the packing (or orifice material) should be dispersed (10). For the same reason, the phase with the larger volume flow rate should be dispersed. For the chemical system and the flow ratio used in this investigation, these factors all favor dispersion of the organic phase in extraction runs.

C. Examination of HTU Results

Both HTU_{oo} and HTU_{oa} were calculated for this investigation. Although the HTU_{oa} was nearly always slightly higher than the HTU_{oo} , the following discussion uses HTU in reference to both since their curves showed the same characteristics.

1) Aqueous Phase Dispersed - Transfer into Organic Phase

As may be seen in Figure 5 (75 gm./min. solute free aqueous flow), the HTU changed only slightly over the range of 0-45 RPM. The change, if any, was probably due to a decrease in transfer area by coalescence (See Figures 8 and 9), since the column flooded easier at lower frequencies than at intermediate frequencies (See Part D, Flooding Range Curve). Holdup also changed very little over this range. The HTU decreased slowly and steadily from 45-105 RPM; the holdup increased roughly in proportion. The decrease of the HTU over this range probably could be attributed to a controlling effect of increased transfer area and turbulence. After 105 RPM, the HTU slowly increased. Although holdup increased at a faster rate than at lower frequencies, apparently the increase in transfer area was overbalanced by increased back-mixing.

The curves in Figure 6 (160 gm./min. solute free aqueous flow) exhibit much the same properties as those in Figure 5. The minimum HTU obtained at these conditions was somewhat lower than for the smallest throughput, however, and relatively little increase was noted in the HTU after 120 RPM. Holdup again increased faster at

higher frequencies than at lower frequencies. Apparently, for this throughput, back-mixing in the orifice plate column was still a controlling factor at high holdups.

For the orifice plate pulse column, change in throughput below the flooding range produced little change in the HTU at low frequencies. Preliminary Runs 1 and 6 shown in Table I indicated this fact as did the curves in Figures 5-7, 10 and 11. At higher frequencies, however, an increase in throughput decreased the HTU as was discussed in Part B. Since holdup increased with throughput in all cases, it was concluded that the decrease in HTU observed for all curves as a function of throughput was due primarily to increased transfer area, although increased turbulence, increased drop oscillation and decreased back-mixing definitely were influencing factors.

Referring to Figure 7 (240 gm./min. solute free aqueous flow), data were not taken at 0 and 180 RPM because the column flooded at those frequencies. Over the frequency range of 45-165 RPM., the HTU decreased steadily and more rapidly than was observed at lower throughputs. Holdup again increased over this range, more noticeably at higher frequencies, and apparently back-mixing was not a controlling factor when throughputs were also high.

The curves in Figures 10 and 11 (320 and 400 gm./min. solute free aqueous flow) exhibit much the same properties as the curves in Figure 7, except that the HTU values were respectively lower and the holdup values were higher. As was mentioned earlier, these differences were attributable to increased transfer area, increased turbulence and drop oscillation, and decreased back-mixing as a result of increased throughput.

2) Organic Phase Dispersed - Transfer into Organic Phase

The HTU (organic discontinuous) curve on Figure 7 exhibits a minimum over a frequency range of 45-165 RPM., but the overall change in HTU was relatively slight. Although holdup increased with frequency, apparently the factors affecting HTU were compensating. At this throughput, the HTU was lower for organic discontinuous than for

aqueous discontinuous. This observation may be explained in part by the fact that the discontinuous phase holdup, and thus transfer area, was higher when the organic phase was dispersed (See Figure 12).

Examination of Figures 5-7, 10 and 11 at 120 RPM shows that the organic discontinuous holdup was always higher, and yet at low throughputs the organic discontinuous HTU was higher or equal to the aqueous discontinuous HTU. It was therefore concluded, as in the case of aqueous discontinuous, that back-mixing was an important factor at high holdups and low throughputs.

D. Flooding Range Curve (Figure 15)

The most important point for discussion is the fact that the flooding range curve for the orifice plate pulse column exhibits a maximum. Apparently at low frequencies, pulsing tends to increase the maximum throughput by pumping material from one cell to the next. Visual observations of this pumping action have been noted in other investigations (2, 3, 12), especially for sieve plate pulse columns. For idealized operation of a sieve plate pulse column, the maximum throughput would be equal to twice the pulse displacement times the frequency, if no back-mixing occurred.

As was previously discussed, however, holdup and thus back-mixing did increase with increased frequency. Moreover, it was mentioned that coalescence also was more probable at higher holdups. All these factors would tend to decrease the maximum throughput. It is reasonable, therefore, to expect that at some frequency, holdup and back-mixing would overbalance the effect of pumping action and decrease the maximum throughput with increased frequency thereafter.

Buchanan, et. al. (3) found that flooding data for a sieve plate pulse column could be correlated with frequency when flow was expressed as the sum of the flows of both phases. Although the same phase was always dispersed, the correlation held for several phase ratios. Other investigators (5, 10) have found that flooding is dependent upon both flow rates, one varying inversely with the other at maximum throughput. Since the phase ratio was approximately

constant for this investigation, it is easily seen that the flooding data could have been correlated as well with total flow as with the flow of either phase.

An interesting point for discussion is the fact that data from organic discontinuous studies as well as aqueous discontinuous studies were used in obtaining the flooding range curve. No detailed analysis was made of this observation, but it appears that for this system, flooding was probably more dependent upon total flow than upon flow of either discontinuous phase.

E. Discussion of Overall Operation

For the conditions of this investigation, the orifice plate pulse column produced minimum HTU values very near to 1 foot. These values were obtained at high frequencies and high flow rates, very close to flooding conditions. For the same chemical system, the same phase dispersed, and the same direction of transfer, at a similar flow ratio, but much lower flow rates, Belaga and Bigelow (2) obtained HTU values as low as 3 inches in a sieve plate pulse column. In general, they found extraction to be best at low frequencies and low pulse heights. Their column had the same internal diameter as the orifice plate pulse column, and the plates had very nearly the same free area, but their plate spacing was 1 inch and pulse heights ranged from 1/8 to 2 inches. Later work on the same sieve plate pulse column by Baker and Lang (1) showed that HTU values were lowest when the column was operated almost at flooding. In their investigation, however, frequency was kept constant at 40 CPM. and no flooding data as a function of frequency were obtained for comparison with results of the current investigation.

Quantitative comparison of the two types of extraction devices therefore requires some speculation. Certainly there is no indication that the design of the orifice plate pulse column used in this investigation was optimum from the standpoint of either throughput or extraction. Since Gavin and Thies (6) found that extraction in Belaga's column varied inversely with the distance between plates, over a range

of plate spacing of $1/4$ to 2 inches, probably reducing the plate spacing on the orifice plate pulse column would also improve its extraction characteristics. Moreover, the current investigation provided little knowledge of the effect on extraction of orifice size and pulse amplitude, undoubtedly two very important variables.

The most interesting comparison was made by Buchanan, et. al. (3) for another chemical system, using larger equipment than was used for this investigation. In their investigation an orifice plate pulse column achieved the same extraction as was obtained from a sieve plate pulse column operating at the same flow rates. At comparable frequencies they also found that the orifice plate pulse column permitted a higher throughput.

Based on development up to this time, it is felt that the orifice plate pulse column has less favorable extraction characteristics than the sieve plate pulse column, but permits a higher maximum throughput. Therefore, just as the spray tower is frequently chosen in preference to the packed tower because of its higher throughput, although its extraction may not be as favorable (10), situations probably exist where the orifice plate pulse column would be more desirable from economic considerations than the sieve plate pulse column. Certainly the possible use of orifice plate pulse columns in slurry extraction is a potentially important application.

A comparison of pulsed and nonpulsed spray tower operation (See Figure 17) with that of the orifice plate pulse column also requires considerable speculation. As was expected, the three organic discontinuous nonpulsed spray tower runs showed a fairly large decrease in HTU with increased throughput. At 240 gm./min. solute free aqueous flow, the HTU value obtained for spray tower operation was 14 inches, a value equal to the lowest HTU achieved by the orifice plate column at the same throughput over the frequency range studied.

At the same throughput, pulsing the spray tower at 200 RPM. resulted in no change in HTU although the holdup increased slightly.

However, when the spray tower was pulsed at 300 RPM., again at the same throughput, the HTU decreased to 11 inches, the lowest obtained throughout the investigation. It is suspected that the increased dispersion and holdup observed for this latter run were not independent of the type of distribution plates employed and the resulting constrictions at the inlets (See Figure 18). The results, however, were in direct contrast with the observations of Lurie and Shaver (7). Although these results are not conclusive, it would appear that pulsing a spray tower may increase extraction efficiency. The spray tower is a limiting design of an orifice plate column; i.e., with the orifice diameter equal to the tower diameter. Under these conditions throughput would be expected to be a maximum, thus increasing the potential of the pulsed apparatus as an extraction device.

VI. CONCLUSIONS

On the basis of the results of this investigation it was concluded that:

- 1) Pulsing tended to increase holdup and improve extraction efficiency, particularly at high frequencies and at high throughputs.
- 2) Pulsing tended to create smaller drops and increase back-mixing, particularly at low throughputs.
- 3) An increase in throughput tended to increase holdup and improve extraction efficiency, particularly at high frequencies.
- 4) An increase in throughput tended to increase drop size and decrease back-mixing.
- 5) Dispersion of the organic phase in preference to the aqueous phase, when solute transfer is into the organic phase, resulted in more holdup and increased transfer area. These factors increased the extraction efficiency at high flow rates and low frequencies.
- 6) Pulsing in an orifice plate pulse column tended to increase maximum throughput by pumping action and to decrease maximum throughput by increasing holdup. Maximum throughput was highest in the range of intermediate frequencies.

VII. RECOMMENDATIONS

In view of the effects of pulsing in an orifice plate pulse column and the possibility that the potential of the spray tower as an extraction device may be increased by pulsing, it is recommended that:

- 1) A more detailed study be made on an orifice plate pulse column to determine the effect of frequency on average drop size, back-mixing and coalescence, perhaps with the aid of motion pictures and dyes.
- 2) Studies be made to determine the effect of pulse height, cam shape and orifice size, shape and spacing on HTU, factors influencing HTU, and flooding.
- 3) Studies be made to determine the effect of pulsing on spray tower operation and extraction efficiency, with particular emphasis on the mechanism of dispersion and the effect of varied inlet design.

VIII. APPENDIX

A. Description of Apparatus

Figure 1 is a flow diagram of the system and Figures 2-4 are pictures of the experimental equipment. The principal unit, an orifice plate pulse column, was constructed from sections of standard double tough Pyrex pipe, 1-1/2 inches inside diameter. Inlet and outlet connections were made with standard Pyrex pipe tees. The sections were joined with standard metal flanges in the following order: starting from the bottom, 1 tee (7"), 1 straight (4"), 1 tee (7"), 18 straight (72"), 1 tee (7"), 1 straight (4"), 1 tee (7"), and 1 straight (4"). For spray tower operation a single piece of straight 1-1/2 inch inside diameter pipe was substituted for the 18 straight sections.

Teflon gaskets, 1/16" thick, brought the overall height of the column to 114". The internal diameter of the gaskets was 1-1/2" except for the orifice gaskets (19 total, located from the bottom through the top of the 72" section) which were 3/4". Orifice gaskets, therefore, restricted the area for flow at the joints to 25% of the column cross-sectional area.

Flanged aluminum plugs tapped internally and externally were used to seal off the tees. No internal fittings were used on the outlet tees, located at the extreme top and bottom of the column. The inlet tees were fitted with 3/8" brass elbows, 90°, to direct flow vertically along the column (see Figure 18). Two brass plates, each containing 25 drilled holes 1/16" in diameter, were soldered to the elbows and acted as distributors.

The pulsing unit (See Figure 3) was driven by a 1/6 HP Electro Machine Co. AC motor with a variable speed transmission. The drive frequency range was roughly 0-400 RPM. and was obtained with the aid of suitable pulleys. A 1/2" thick brass circle 2-3/4" in diameter with a drive shaft displacement of approximately 1/8" served as the cam. These dimensions produced a pulse pattern which was very close to a sine wave (See Figure 19). The cam follower was

ORGANIC DISTRIBUTION PLATE
(SAME AS AQUEOUS)

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DETAIL OF DISTRIBUTION PLATE

DATE 8-13-'54	DRAWN BY D. G. O'B.	FILE NO. EPS-K209	FIG. 18
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O = SHAFT CENTER

C = CAM CENTER

d = SHAFT DISPLACEMENT FROM CAM CENTER ---- 1/8"

r = CAM RADIUS ----- 1 3/8"

h = LENGTH FROM SHAFT TO BEARING POINT

θ = ANGLE OF REVOLUTION

a = PISTON DISPLACEMENT

2a = PULSE HEIGHT

$$r^2 = h^2 + d^2 + 2hd \cos \theta$$

— ACTUAL PULSE
○ SINE

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
ENGINEERING PRACTICE SCHOOL
CARBIDE AND CARBON CHEMICALS COMPANY
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COMPARISON OF PULSE SHAPE
WITH SINE WAVE

DATE 7-30-54 DRAWN BY D.C.O'B. FILE NO. EPS-K209 FIG. 19

a small brass wheel attached to a 1/2" spring-loaded shaft. Brass bellows were fitted to this shaft and were flanged to the bottom of the column. The bellows had the following characteristics: Fulton Sylphon Reference Number 1098, 2-7/16" outside diameter x 1-15/16" inside diameter, 18 active corrugations, one-ply brass, 135 lb./sq.in. maximum.

Feed and storage tanks were standard bilged 55 gallon single piece stainless steel drums. Three of these drums had 3/8" pipe sections welded through the two top bung caps. One of the sections extended several inches into the drum; the other extended to approximately 2" from the bottom. The shorter pieces of pipe were connected with 1/4" Saran tubing and 1/4" pipe through a reducing valve to a 40 psig. air supply. The longer pieces were fitted to 1/4" Saran tubing and could be interchangeably connected to two rotameters.

The inlet side of each rotameter was constructed with a tee and valve arrangement to allow for feed stream sample removal. Special Fisher-Porter 3A-60-A rotameters were used and were fitted with standard Hoke 1/4" needle valves on the outlet side. The tubing was then connected directly to the respective inlet tees.

The organic outlet was simply an overflow into 1/4" Saran tubing. The aqueous outlet was fitted with a 3/8" Hoke needle valve and consisted of an adjustable, non-syphoning gravity flow liquid leg also made from 1/4" Saran tubing and fittings. Flows were such that both overflows would operate without being completely filled.

B. Chemical System and Analytical Methods

The chemical system employed was acetic acid, water and hexone (methyl-isobutyl ketone). Factors which made use of this system desirable were: practical regeneration, slight mutual solubility of the two phases, low interfacial tension of hexone, large density difference, and relative ease of analysis. Moreover, use of the system by other investigators allowed comparison of data. Equilibrium data were obtained from Belaga and Bigelow (2) and are shown in Table III.

TABLE III
EQUILIBRIUM DATA
(Acetic Acid, Water, Hexone System)

<u>A (Aqueous Phase)</u>	<u>O (Organic Phase)</u>
(weight per cent acetic acid)	
2.95	1.79
6.58	4.38
8.98	6.14
12.11	8.77
1.51	0.90
3.47	2.22
6.61	4.47
9.40	6.73
11.96	8.82
14.55	11.09
16.52	12.92

First four points at 32.5°C, remainder at 28-29°C.

Source: Belaga, M.W., and Bigelow, J.E., S.M. Thesis, "Effect of Pulse Column Operating Variables on HTU", Report No. KT-133, Oak Ridge, M.I.T. Practice School, Carbide and Carbon Chemicals Co., K-25 (1952).

The acid was 99.5% reagent grade acetic acid produced by J.T. Baker Chemical Co. The water was untreated plant water and the hexone was technical grade methyl-isobutyl ketone purchased from Ansco Solvents and Chemicals Co.

Samples of both phases, ranging in acid concentration from 0.03 N to 2.3 N, were all titrated with 0.1 N NaOH to a phenolphthalein end point. Aliquots were chosen so that roughly 20 ml. or more of base were required for neutralization. Generous amounts of water and ethyl alcohol were added to the hexone aliquots to produce a single phase.

C. Details of Procedure

The first step in preparing for an extraction run was to make up the aqueous feed. Acid rich water from a previous stripping run was pumped to the feed tank with a small centrifugal pump. In general, it was found that 15 lb. of glacial acetic acid would bring the feed up to 2.3 N, the starting concentration. After the contents of the drum was thoroughly stirred, a sample was taken and titrated. Addition and titration was continued until the desired concentration was obtained. Specific gravities were determined with a hydrometer.

A drum of hexone usually lasted for five runs. At the start of a five run series, 200 ml. samples were withdrawn from both inlet drums. These samples were assumed to be representative of the entire drum and few checks were taken during a series. The checks always confirmed the assumption of homogeneity.

In beginning an extraction run, the pulsing unit was usually first to be started. Sometimes the pulse height was checked by manually turning the cam to the extreme positions, marking off the change in interface level on a piece of tape and measuring the height with a ruler. Although the cam surface did wear slightly, occasional checks showed that the pulse height was constant at 1/2". The frequency was then set at the desired level and was determined by counting revolutions with a stopwatch. Checks made during a run rarely showed any change from the original setting.

After adjusting the air pressure on the displacement pump feed tanks to 8 psig., flow of the continuous phase was started and was adjusted to the predetermined rotameter setting. The interface level was set roughly midway between the continuous phase inlet and the discontinuous phase outlet. Flow of the discontinuous phase was then started and again fixed by the rotameter setting. The interface level was readjusted by raising or lowering the height of the aqueous overflow.

After roughly six or seven changes of the continuous phase, the column was assumed to be at steady-state. Flows of both phases were checked by timing the collection of outlet streams in a 1000 ml. graduate with a stopwatch. Hydrometer measurements were made before the two phases were returned to their respective drums. Sometimes part of the material collected for flow rate determination was used as a sample. If not, 200 ml. samples were still taken at the same time, but directly from the outlet lines. The same procedure was repeated 30 minutes later as a check on steady-state.

The procedure for ending a run varied depending upon which phase was discontinuous. If the aqueous was discontinuous, the aqueous outlet and inlet valves were shut as quickly as possible. The organic flow was allowed to continue but the pulser was stopped. After the phases were separated, the aqueous outlet line was opened and the interface level was allowed to drop to the operating level. The aqueous overflow was collected and was taken as the holdup.

If the organic phase was discontinuous, the interface level was dropped and both inlet flows were shut off as quickly as possible. This operation stopped the organic outflow. The aqueous outlet line was then shut. After stopping the pulser and allowing the phases to separate, aqueous phase was fed to the column until the interface had climbed to the normal operating level. The organic overflow was collected and was taken as the holdup.

At the end of an extraction run series the aqueous raffinate was discarded. The acid-rich organic tank was connected to the air

line and the organic rotameter. The aqueous rotameter was connected directly to the plant water line, and the column was then ready to strip. Operating procedure was the same for extraction except that normally only one run was taken per drum.

As noted before, rotameters were used primarily for setting flow rates but not for measuring them. For flooding studies, however, rotameters were used for flow rate measurement due to the transient nature of the total discontinuous phase holdup. They were calibrated by using flow data from the most recent runs. The plots of rotameter setting versus solute-free flow rate based on outlet flow rate proved to be fairly good straight lines. Rotameter settings for flooding studies were chosen at intervals of approximately 40 gm./min. solute-free aqueous flow at a Q_o/Q_a of 1.33.

In flooding studies, the operating procedure was much the same as in other runs. At a given frequency, rotameter settings were changed from low to high values. The lowest setting at which flooding occurred was called the flooding point.

Aqueous discontinuous flooding was defined as the throughput at which not all of the aqueous phase could pass through the first orifice, but was partially forced out the organic outlet. Evidence of this type of flooding was seen as a secondary interface which formed at the aqueous inlet and tended to rise to the top of the column. The heavier phase actually consisted of an emulsified mixture of hexone and water. Such a formation frequently did not take place immediately, and consequently 15 minutes was allowed at each setting.

Organic discontinuous flooding was more arbitrary in definition. It was defined as the throughput at which the organic coalesced to form large drops to such an extent that it nearly filled a cell and became the continuous phase in that cell.

D. Sample Calculations

Run No. 65 was chosen as a basis for the sample calculation.

1. Column Characteristics:

Effective height - 85"
Plate spacing - 4"
Orifice diameter - 3/4"

2. Operating Conditions:

Discontinuous phase organic
Transfer aqueous to organic
Volume in effective extraction
 height 2350 ml.
Holdup 690 ml.
Room Temperature 81° F.

3. Flow Rates:

<u>Aqueous</u>	<u>ml. collected</u>	<u>Time (sec.)</u>
Sample 1	992	189.6
Sample 2	990	189.0
<u>Organic</u>		
Sample 1	1002	112.6
Sample 2	996	111.0

	<u>Specific Gravity*</u>	<u>Normality</u>
Inlet Aqueous	1.011	2.307
Inlet Organic	0.799	0.0364
Outlet Aqueous: Sample 1	0.998	0.569
Sample 2	0.998	0.566
Outlet Organic: Sample 1	0.826	1.272
Sample 2	0.826	1.247

* No temperature correction was made for the hydrometer.

058
4 3 57

5. Concentrations of the four streams were calculated using averaged normalities as follows:

$$N \frac{\text{gm. equiv. acid}}{\text{liter sample}} \times \frac{60.05 \text{ gm. acid}}{\text{gm. equiv. acid}} \times \frac{1 \text{ liter sample}}{1000 \text{ ml. sample}} \times \frac{1}{\text{spec. grav.} \times \text{dens. H}_2\text{O}} \frac{\text{ml. sample}}{\text{gm. sample}} = \frac{\text{gm. acid}}{\text{gm. sample}}$$

$$\text{Inlet Aqueous: } A_1 = \frac{(2.307)(60.05)}{(1000)(1.011)} = 0.1370 \frac{\text{gm. acid}}{\text{gm. sample}}$$

$$\text{Inlet Organic: } O_2 = \frac{(0.0364)(60.05)}{(1000)(0.799)} = 0.0027 \frac{\text{gm. acid}}{\text{gm. sample}}$$

$$\text{Outlet Aqueous: } A_2 = \frac{\left(\frac{0.569 + 0.566}{2}\right)(60.05)}{(1000)(0.998)} = 0.0342 \frac{\text{gm. acid}}{\text{gm. sample}}$$

$$\text{Outlet Organic: } O_1 = \frac{\left(\frac{1.272 + 1.247}{2}\right)(60.05)}{(1000)(0.826)} = 0.0916 \frac{\text{gm. acid}}{\text{gm. sample}}$$

6. These concentrations were converted to gm. acid/gm. acid-free solvent, and the corresponding equilibrium values were obtained from a plot of X^* vs. Y^* for this system (See Figure 20):

				Equilibrium Values
Inlet Aqueous	$X_1 = \frac{A_1}{1 - A_1} = \frac{0.1370}{0.8630} = 0.1587$		$Y_1^* = 0.1130$	
Inlet Organic	$Y_2 = \frac{O_2}{1 - O_2} = \frac{0.0027}{0.9973} = 0.0028$		$X_2^* = 0.0042$	
Outlet Aqueous	$X_2 = \frac{A_2}{1 - A_2} = \frac{0.0342}{0.9658} = 0.0354$		$Y_2^* = 0.0229$	
Outlet Organic	$Y_1 = \frac{O_1}{1 - O_1} = \frac{0.0916}{0.9084} = 0.1008$		$X_1^* = 0.1438$

7. Solute-free flow rates were calculated as follows;

$$Q = \frac{\text{ml. sample collected}}{\text{sec.}} \times \frac{60 \text{ sec.}}{\text{min.}} \times (\text{sp.g.} \times \text{dens. H}_2\text{O}) \frac{\text{gm. sample}}{\text{ml. sample}} \times \frac{\text{gm. solute-free solvent}}{\text{gm. sample}}$$

$$= \frac{\text{gm. solute-free solvent}}{\text{min.}}$$

Outlet Aqueous:

$$\text{Sample 1: } Q_a = \frac{992}{189.6} \times 60 \times 0.998 \times 0.966 = 303 \frac{\text{gm. solute-free solvent}}{\text{min.}}$$

$$\text{Sample 2: } Q_a = \frac{990}{189} \times 60 \times 0.998 \times 0.966 = 303 \frac{\text{gm. solute-free solvent}}{\text{min.}}$$

$$\text{Average } Q_a = 303 \frac{\text{gm. solute-free solvent}}{\text{min.}}$$

Outlet Organic:

$$\text{Sample 1: } Q_o = \frac{1002}{112.6} \times 60 \times 0.826 \times 0.908 = 400 \frac{\text{gm. solute-free solvent}}{\text{min.}}$$

$$\text{Sample 2: } Q_o = \frac{996}{111.0} \times 60 \times 0.826 \times 0.908 = 404 \frac{\text{gm. solute-free solvent}}{\text{min.}}$$

$$\text{Average } Q_o = 402 \frac{\text{gm. solute-free solvent}}{\text{min.}}$$

8. Ratio of solute-free organic flow to solute free aqueous flow:

$$Q_o/Q_a = \frac{402}{303} = 1.33$$

9. Material balance on acid:

$$\text{Acid in: Inlet Aqueous: } (Q_a \times X_1) = 303 \times 0.1587 = 48.1 \frac{\text{gm.}}{\text{min.}}$$

$$\text{Inlet Organic: } (Q_o \times Y_2) = 402 \times 0.0028 = 1.1 \text{ "}$$

$$\text{Total acid in } 49.2 \frac{\text{gm.}}{\text{min.}}$$

$$\text{Acid out: Outlet Aqueous: } (Q_a \times X_2) = 303 \times 0.0354 = 10.7 \frac{\text{gm.}}{\text{min.}}$$

$$\text{Outlet Organic: } (Q_o \times Y_1) = 402 \times 0.1008 = 40.5 \text{ "}$$

$$\text{Total acid out } 51.2 \frac{\text{gm.}}{\text{min.}}$$

Per cent deviation based on acid in:

$$\left(\frac{51.2 - 49.2}{49.2} \right) 100 = +4.1\%$$

06 /
-60

10. Evaluation of HTU_{oo} and HTU_{oa} .

The following assumptions were made:

$$NTU_{oo} = \int_{Y_1}^{Y_2} \frac{dY}{Y^* - Y} \approx \frac{Y_2 - Y_1}{(Y^* - Y)_{\log \text{ mean}}}$$

$$NTU_{oa} = \int_{X_1}^{X_2} \frac{dX}{X - X^*} \approx \frac{X_1 - X_2}{(X - X^*)_{\log \text{ mean}}}$$

Calculations were made as follows:

$$\underline{NTU_{oo}}$$

$$Y_2 - Y_1 = 0.1008 - 0.0028 = 0.0980$$

$$\begin{aligned} (Y^* - Y)_{\log \text{ mean}} &= \frac{(Y^* - Y)_2 - (Y^* - Y)_1}{\ln \frac{(Y^* - Y)_2}{(Y^* - Y)_1}} \\ &= \frac{(0.1130 - 0.1008) - (0.0229 - 0.0028)}{\ln \frac{(0.1130 - 0.1008)}{(0.0229 - 0.0028)}} \\ &= 0.0159 \end{aligned}$$

$$NTU_{oo} = \frac{0.0980}{0.0159} = 6.16$$

$$HTU_{oo} = \frac{\text{Effective column height}}{NTU_{oo}} = \frac{85''}{6.16} = 13.8''$$

$$\underline{NTU_{oa}}$$

$$X_1 - X_2 = 0.1587 - 0.0354 = 0.1233$$

$$\begin{aligned} (X - X^*)_{\log \text{ mean}} &= \frac{(X - X^*)_1 - (X - X^*)_2}{\ln \frac{(X - X^*)_1}{(X - X^*)_2}} \\ &= \frac{(0.1587 - 0.1438) - (0.0354 - 0.0042)}{\ln \frac{(0.1587 - 0.1438)}{(0.0354 - 0.0042)}} \\ &= 0.0220 \end{aligned}$$

RUN 65

NTU₁₀₀ = 6.91

NTU₁₀₀ = 6.33

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DETERMINATION OF NTU'S

DATE
8-13-'54

DRAWN BY
D.C.O'B.

FILE NO.
EPS-K209

FIG.
21

$$NTU_{oa} = \frac{0.1233}{0.0220} = 5.60$$

$$HTU_{oa} = \frac{\text{Effective column height}}{NTU_{oa}} = \frac{85''}{5.60} = 15.2''$$

11. More accurate determinations of NTU_{oo} and NTU_{oa} were made by graphical integrations of plots of Y vs. $1/(Y^* - Y)$ and X vs. $1/(X - X^*)$ (See Figure 21).

The following values were obtained:

$$NTU_{oo} = 6.91 \qquad HTU_{oo} = \frac{85''}{6.91} = 12.3''$$

$$NTU_{oa} = 6.33 \qquad HTU_{oa} = \frac{85''}{6.33} = 13.4''$$

12. The errors introduced by the assumption of log-mean driving force were:

$$NTU_{oo} : \left(\frac{6.91 - 6.16}{6.91} \right) \times 100 = 10.8\%$$

$$NTU_{oa} : \left(\frac{6.33 - 5.60}{6.33} \right) \times 100 = 11.5\%$$

13. Similar calculations for run No. 41 showed:

$$NTU_{oo} (\text{log mean}) = 3.56 \qquad HTU_{oo} = 23.9''$$

$$NTU_{oa} (\text{log mean}) = 3.18 \qquad HTU_{oa} = 26.7''$$

$$NTU_{oo} (\text{graphical integration}) = 3.79$$

$$NTU_{oa} (\text{graphical integration}) = 3.38$$

The errors introduced by the assumption of log-mean driving force were:

$$NTU_{oo} : \left(\frac{3.79 - 3.56}{3.79} \right) \times 100 = 6.1\%$$

$$NTU_{oa} : \left(\frac{3.38 - 3.18}{3.38} \right) \times 100 = 5.9\%$$

It is seen from these two runs that the error tends to decrease with increasing HTU .

14. Discontinuous organic holdup in terms of volume per cent was (for run No. 65):

$$\left(\frac{690}{2350}\right) \times 100 = 29.4\% \text{ holdup}$$

E. Summary of Data and Calculated Values

Table IV is a summary of data and calculated values.

Table IV
SUMMARY OF DATA AND CALCULATED VALUES

ORIFICE PLATE COLUMN																
Run No.	Discontinuous Phase	Phase Receiving Solute	Pulse Frequency (RPM.)	Q _a * Solute Free Aqueous Flow (gm./min.)	Q _o * Solute Free Organic Flow (gm./min.)	Q _o /Q _a	% Holdup (volume)	X ₁ gm. acid gm. water inlet	Y ₂ gm. acid gm. hexone inlet	X ₂ gm. acid gm. water outlet	Y ₁ gm. acid gm. hexone outlet	% Deviation Material Balance (inlet basis)	(Y*-X) _{1m}	(X-X*) _{1m}	HTU _{oo}	HTU _{oa}
1	Aqueous	Organic	30	83.0	160	1.93	-	0.1566	0	0.0359	0.0634	+0.8	0.0342	0.0481	45.9	33.9
2	Aqueous	Organic	30	166	160	0.96	-	0.1566	0	0.0638	0.0932	-1.9	0.0284	0.0395	25.9	36.2
3	Aqueous	Organic	30	332	160	0.48	-	0.1566	0	0.1016	0.1135	-0.2	-----pinch-----			
4	Aqueous	Organic	30	83.0	320	3.86	-	0.1566	0	0.0187	0.0327	-6.9	0.0353	0.0505	91.8	31.1
5	Aqueous	Aqueous	45	472	345	0.73	-	0	0.0583	0.0404	0.0003	-4.5	0.0066	0.0097	9.7	20.4
6	Aqueous	Organic	30	165	317	1.92	-	0.1640	0.0066	0.0437	0.0691	-0.3	0.0334	0.0466	45.4	32.9
7	Aqueous	Organic	30	330	317	0.96	-	0.1640	0.0066	0.0701	0.1025	-1.1	0.0255	0.0349	22.6	31.6
8	Aqueous	Organic	30	74.2	63.2	0.85	-	0.1640	0.0066	0.0846	0.1011	+0.8	0.0302	0.0412	27.2	44.3
9	Aqueous	Organic	30	152	62.7	0.41	-	0.1640	0.0066	0.1185	0.1201	+0.8	-----pinch-----			
10	Aqueous	Aqueous	75	739	535	0.72	-	0	0.0833	0.0647	0.0006	+7.8	0.0094	0.0133	9.6	17.5
11	Aqueous	Organic	45	78.1	113	1.45	-	0.1539	0.0020	0.0483	0.0787	+4.1	0.0298	0.0420	33.1	33.9
12	Aqueous	Organic	45	167	220	1.32	-	0.1539	0.0020	0.0536	0.0839	+5.0	0.0289	0.0406	30.0	34.4
13	Aqueous	Organic	45	252	328	1.30	-	0.1539	0.0020	0.0542	0.0856	+5.8	0.0282	0.0395	28.7	33.7
14	Aqueous	Organic	45	335	433	1.29	7.0	0.1539	0.0020	0.0538	0.0859	+5.1	0.0279	0.0392	28.3	33.3
15	Aqueous	Organic	45	413	548	1.33	-	0.1539	0.0020	0.0509	0.0870	+6.2	0.0265	0.0370	26.5	30.6
16	Aqueous	Aqueous	75	652	545	0.84	-	0	0.0799	0.0696	0.0008	+5.3	0.0088	0.0125	9.4	15.3
17	Aqueous	Organic	60	76.2	98.4	1.29	2.0	0.1643	0.0025	0.0538	0.0921	+3.9	0.0291	0.0400	27.6	30.8
18	Aqueous	Organic	60	164	204	1.24	4.0	0.1643	0.0025	0.0562	0.0964	+5.5	0.0272	0.0378	24.6	29.7
19	Aqueous	Organic	60	245	303	1.24	5.7	0.1643	0.0025	0.0566	0.0955	+4.4	0.0280	0.0387	25.6	30.6
20	Aqueous	Organic	60	325	413	1.27	6.6	0.1643	0.0025	0.0545	0.0940	+3.9	0.0282	0.0390	26.2	30.1
21	Aqueous	Organic	60	404	528	1.31	9.4	0.1643	0.0025	0.0522	0.0931	+3.8	0.0280	0.0386	26.2	29.3
22	Aqueous	Aqueous	60	627	574	0.92	-	0	0.0695	0.0636	0.0022	+3.2	0.0099	0.0144	12.6	19.2
23	Aqueous	Organic	75	77.0	105	1.36	2.4	0.1609	0.0023	0.0490	0.0944	+8.7	0.0248	0.0345	22.9	26.2
24	Aqueous	Organic	75	158	209	1.32	4.1	0.1609	0.0023	0.0486	0.0942	+5.8	0.0248	0.0344	23.0	26.1
25	Aqueous	Organic	75	243	304	1.25	5.6	0.1609	0.0023	0.0523	0.0960	+5.3	0.0248	0.0345	22.5	27.0
26	Aqueous	Organic	75	318	416	1.31	8.0	0.1609	0.0023	0.0502	0.0932	+5.0	0.0258	0.0359	24.1	27.6
27	Aqueous	Organic	75	396	544	1.37	9.4	0.1609	0.0023	0.0490	0.0915	+6.6	0.0264	0.0368	25.1	28.0
28	Aqueous	Aqueous	90	717	669	0.93	-	0	0.0806	0.0761	0.0028	+4.8	0.0114	0.0164	12.4	18.3
29	Aqueous	Organic	90	75.3	103	1.37	2.8	0.1574	0.0028	0.0452	0.0900	+4.7	0.0243	0.0338	23.7	25.6
30	Aqueous	Organic	90	161	212	1.32	4.8	0.1574	0.0028	0.0444	0.0949	+5.0	0.0213	0.0297	19.7	22.4
31	Aqueous	Organic	90	247	316	1.28	6.0	0.1574	0.0028	0.0472	0.0958	+4.0	0.0216	0.0300	19.8	23.2
32	Aqueous	Organic	90	320	426	1.33	8.9	0.1574	0.0028	0.0445	0.0943	+5.4	0.0217	0.0302	20.1	22.7
33	Aqueous	Organic	90	400	546	1.36	9.9	0.1574	0.0028	0.0435	0.0925	+5.3	0.0223	0.0311	21.2	23.2
34	Aqueous	Aqueous	105	626	567	0.90	-	0	0.0862	0.0794	0.0024	+4.5	0.0099	0.0167	9.9	17.9
35	Aqueous	Organic	105	75.0	115	1.53	4.0	0.1626	0.0030	0.0405	0.0900	+7.2	0.0246	0.0342	24.0	23.8
36	Aqueous	Organic	105	161	226	1.40	6.2	0.1626	0.0030	0.0412	0.0973	+6.3	0.0214	0.0295	19.3	20.6
37	Aqueous	Organic	105	248	329	1.33	7.2	0.1626	0.0030	0.0428	0.0994	+4.8	0.0206	0.0286	18.2	20.3
38	Aqueous	Organic	105	322	443	1.38	9.5	0.1626	0.0030	0.0422	0.0982	+5.8	0.0211	0.0293	18.8	20.7
39	Aqueous	Organic	105	402	555	1.38	10.6	0.1626	0.0030	0.0417	0.0965	+4.9	0.0220	0.0303	20.0	21.3
40	Aqueous	Aqueous	120	406	364	0.90	-	0	0.0923	0.0871	0.0013	+6.8	0.0099	0.0138	9.2	13.5

* Flows for Runs 1-7 based on rotameter calibrations.

Table IV (Cont'd.)
SUMMARY OF DATA AND CALCULATED VALUES

Run No.	Discontinuous Phase	Phase Receiving Solute	Pulse Frequency (RPM.)	Q _a Solute Free Aqueous Flow (gm./min.)	Q _o Solute Free Organic Flow (gm./min.)	Q _o /Q _a	% Holdup (volume)	X ₁ gm. acid gm. water inlet	Y ₂ gm. acid gm. hexone inlet	X ₂ gm. acid gm. water outlet	Y ₁ gm. acid gm. hexone outlet	% Deviation Material Balance (inlet basis)	(Y*-Y) _{lm}	(X-X*) _{lm}	HTU _{oo}	HTU _{oa}
															(in.)	(in.)
41	Aqueous	Organic	120	79.1	106	1.34	3.7	0.1627	0.0026	0.0494	0.0942	+5.3	0.0257	0.0356	23.9	26.7
42	Aqueous	Organic	120	163	210	1.29	6.2	0.1627	0.0026	0.0447	0.1010	+5.6	0.0205	0.0284	17.7	20.5
43	Aqueous	Organic	120	240	320	1.33	7.4	0.1627	0.0026	0.0429	0.1014	+7.3	0.0197	0.0273	16.9	19.4
44	Aqueous	Organic	120	311	420	1.35	8.8	0.1627	0.0026	0.0391	0.1001	+4.8	0.0193	0.0267	16.8	18.4
45	Aqueous	Organic	120	393	542	1.38	-	0.1627	0.0026	0.0394	0.0990	+5.8	0.0201	0.0277	17.7	19.1
46	Aqueous	Aqueous	120	518	470	0.91	-	0	0.0936	0.0864	0.0024	+4.3	0.0123	0.0167	11.5	16.4
47	Aqueous	Organic	150	77.9	104	1.34	5.3	0.1612	0.0034	0.0537	0.0919	+6.2	0.0273	0.0379	26.2	29.9
48	Aqueous	Organic	150	157	211	1.34	11.4	0.1612	0.0034	0.0420	0.0995	+6.2	0.0195	0.0270	17.2	19.3
49	Aqueous	Organic	150	240	318	1.32	14.5	0.1612	0.0034	0.0400	0.1033	+6.5	0.0167	0.0230	14.2	16.1
50	Aqueous	Organic	150	309	416	1.35	15.0	0.1612	0.0035	0.0388	0.1034	+7.4	0.0165	0.0223	13.9	15.5
51	Aqueous	Organic	150	387	536	1.38	(27.6)	0.1612	0.0035	0.0335	0.1018	+5.1	0.0157	0.0216	13.6	14.4
52	Aqueous	Aqueous	200	215	180	0.84	-	0	0.0912	0.0787	0.0044	+7.9	0.0158	0.0224	15.5	24.2
53	-----	-----	---	---	---	----	----	-----	-----	-----	-----	----	-----	-----	-----	----
54	Aqueous	Organic	180	80.0	110	1.38	10.4	0.1629	0.0082	0.0551	0.0903	+2.9	0.0270	0.0373	28.0	29.4
55	Aqueous	Organic	180	160	222	1.39	19.9	0.1629	0.0082	0.0432	0.0958	+1.1	0.0203	0.0279	19.7	19.8
56	Aqueous	Organic	165	249	331	1.33	12.1	0.1629	0.0082	0.0418	0.1024	+2.3	0.0164	0.0226	14.8	15.8
57	Aqueous	Organic	165	306	437	1.43	25.5	0.1629	0.0082	0.0380	0.1019	+5.0	0.0155	0.0213	14.1	14.5
58	Aqueous	Aqueous	105	629	556	0.88	-	0	0.0962	0.0850	0.0024	+2.4	0.0132	0.0187	12.0	18.7
59	Aqueous	Aqueous	105	642	546	0.85	-	0	0.0922	0.0840	0.0018	+9.1	0.0114	0.0161	10.7	16.3
60	Aqueous	Organic	0	78.0	106	1.36	1.6	0.1576	0.0028	0.0565	0.0840	+5.6	0.0311	0.0434	32.6	36.5
61	Aqueous	Organic	0	164	221	1.35	3.8	0.1576	0.0028	0.0552	0.0850	+5.3	0.0301	0.0421	31.1	35.0
62	Organic	Organic	120	76.0	105	1.38	7.9	0.1587	0.0028	0.0530	0.0879	+6.4	0.0284	0.0396	28.3	31.8
63	Organic	Organic	120	159	205	1.29	13.7	0.1587	0.0028	0.0447	0.0988	+5.8	0.0197	0.0273	17.4	20.3
64	Organic	Organic	120	238	302	1.27	22.3	0.1587	0.0028	0.0412	0.1030	+6.0	0.0160	0.0224	13.5	16.2
65	Organic	Organic	120	303	402	1.33	29.4	0.1587	0.0028	0.0354	0.1008	+4.1	0.0159	0.0220	13.8	15.2
66	Aqueous	Aqueous	120	518	461	0.89	-	0	0.0944	0.0897	0.0015	+8.5	0.0104	0.0145	9.5	13.7
67	Organic	Organic	45	236	314	1.33	12.0	0.1613	0.0024	0.0394	0.1008	+5.1	0.0185	0.0256	16.0	17.8
68	Organic	Organic	75	233	325	1.39	13.9	0.1613	0.0024	0.0342	0.1035	+8.3	0.0155	0.0213	13.0	14.2
69	Organic	Organic	105	235	329	1.40	17.0	0.1613	0.0024	0.0348	0.1010	+7.0	0.0170	0.0235	14.6	15.8
70	Organic	Organic	150	255	327	1.28	30.1	0.1613	0.0024	0.0346	0.1019	+0.5	0.0165	0.0228	14.1	15.3
71	Organic	Organic	0	71.9	112	1.56	3.7	0.1651	0.0035	0.0405	0.0916	+6.5	0.0248	0.0351	23.9	23.9
72	Organic	Organic	0	110	162	1.47	5.4	0.1651	0.0035	0.0399	0.0971	+6.9	0.0219	0.0301	19.9	20.4
73	Organic	Organic	0	150	211	1.41	7.8	0.1651	0.0035	0.0392	0.0994	+5.5	0.0205	0.0282	18.2	19.0
74	Organic	Organic	0	185	270	1.46	11.9	0.1651	0.0035	0.0361	0.1000	+7.3	0.0192	0.0263	16.9	17.3
----- SPRAY COLUMN																
75	Organic	Organic	0	77.0	104	1.35	1.8	0.1641	0.0028	0.0626	0.0860	+6.2	0.0350	0.0484	35.2	39.9
76	Organic	Organic	0	150	211	1.41	5.9	0.1641	0.0028	0.0407	0.0994	+7.5	0.0207	0.0287	17.9	19.5
77	Organic	Organic	0	226	318	1.41	9.1	0.1641	0.0028	0.0343	0.1042	+7.6	0.0161	0.0223	13.3	14.4
78	Organic	Organic	200	225	322	1.43	10.8	0.1641	0.0028	0.0330	0.1036	+7.9	0.0161	0.0222	13.4	14.2
79	Organic	Organic	300	224	316	1.41	14.9	0.1641	0.0028	0.0305	0.1061	+6.9	0.0140	0.0192	11.4	12.0

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F. Table of Nomenclature

$A_{1,2}$	- Acid concentration in the aqueous stream, gm. acid/gm. sample.
a	- Area for mass transfer per unit column volume, sq.in./cu.in.
HETS	- Height equivalent to a theoretical stage, inches.
$HTU_{oo, oa}$	- Height of a transfer unit (Z/NTU), inches.
K	- Overall mass transfer coefficient, gm. acid transferred/ (min.)(sq.in.)(unit conc. driving force)
$NTU_{oo, oa}$	- Number of transfer units.
$O_{1, 2}$	- Acid concentration in the organic stream, gm. acid/gm. sample.
$Q_{a, o}$	- Mass flow rate, gm. solute-free solvent/min.
S	- Column cross sectional area, sq.in.
$X_{1, 2}$	- Acid concentration in aqueous stream, gm. acid/gm. solute-free solvent.
$X_{1, 2}^*$	- Equilibrium acid conc., based on $Y_{1, 2}$, gm. acid/gm. solute-free solvent.
$Y_{1, 2}$	- Acid concentration in organic stream, gm. acid/gm. solute-free solvent.
$Y_{1, 2}^*$	- Equilibrium acid conc., based on $X_{1, 2}$, gm. acid/gm. solute-free solvent.
Z	- Equivalent transfer height of column, inches.

Subscripts:

- 1 refers to top of column, aqueous inlet and organic outlet.
- 2 refers to bottom of column, aqueous outlet and organic inlet.
- a refers to aqueous phase
- o refers to organic phase
- oa refers to overall based on aqueous phase
- oo refers to overall based on organic phase

G. Location of Original Data and Calculations

The original data are found in part on pages 61 - 96, Data Book No. 2487. The data book, the remainder of the data and the calculations may be found at the M.I.T. Engineering Practice School, Oak Ridge, Tennessee.

H. Literature Citations

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