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PERFORMANCE OF A
RECIPROCATING PACKED
EXTRACTION COLUMN

BY

THOMAS VARDAMAN KONKLE

A

THESIS

submitted to the faculty of the

UNIVERSITY OF MISSOURI AT ROLLA

in partial fulfillment of the requirements for the

Degree of

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Approved by

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ABSTRACT

The performance of a mechanically-aided liquid extraction column was investigated utilizing the methyl isobutyl ketone-acetic acid-water ternary extraction system. Mechanical power was added to the extraction system by reciprocating a wire-mesh packing of high void volume at various frequencies and displacements. The effects of total throughput on performance were studied in increments of 5,000 pounds per hour per square foot up to the onset of flooding, and the column performance was evaluated as a function of power addition to the extraction system.

In comparing the various types of mechanically-aided extraction columns, it was determined that the throughputs achieved with the wire-mesh packed column utilized in this work were considerably higher than any other reported throughputs for mechanically-aided extraction columns. Competitive performance was also achieved with the reciprocating packed extraction column at throughputs which were higher than those achieved with various other designs of mechanically-aided extraction columns.

PREFACE

Original design of the mechanically-aided extraction column utilized in this work was accomplished by Mr. J. J. Carr who also served as the major advisor of the work. Modifications of the original column design and accessories enabled further extension of the original work to the limits of performance achieved during this investigation. Sincere appreciation is extended to Mr. J. J. Carr for his professional and academic guidance during the course of this experimental investigation.

Additional acknowledgement is given to Mr. A. V. Kilpatrick, Professor Emeritus of Mechanical Engineering, to Mr. O. K. Lay, Assistant Professor of Chemical Engineering, and to Mr. G. Brown of Welder's Supply, Rolla, Missouri, for their assistance in the fabrication of the components used in the modification of the original extraction unit.

Special acknowledgement is given to Patricia Konkle, the author's wife, for her understanding and appreciation during the course of the work, and to her this work is dedicated.

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I. INTRODUCTION

Liquid extraction is an important mass-transfer operation used in the chemical industry. By making use of the preferential solubility of various solutes between two immiscible solvents, liquid extraction competes with other unit operations such as distillation, crystallization and absorption. Close boiling points and considerations such as heat degradation of components, justify the use of liquid extraction in many separation processes. The necessary separations may be much more difficult to achieve by other methods.

The keys to high liquid extraction rates are increased turbulence, large area for mass transfer between the contacted liquids, and maintenance of maximum point concentration differences over the length of the column. In conjunction with efficiency, consideration must be given to permissible operating throughputs of extraction columns. The simplest extraction equipment has been the spray column. The spray column, having no column internals, relied only on the liquid density difference for turbulence. The spray column was modified with various internals such as baffles, packing and plates to effect turbulence, and reduce back-mixing thus increasing efficiency, but was found to be relatively ineffective.

To overcome the problem of insufficient turbulence in extraction equipment, methods were devised for addition of mechanical power to the extraction system. In filling the extraction column with various internals for transmission of power to the extraction system, the maximum permissible throughput of the columns was decreased considerably. However, with addition of mechanical power to effect increased turbulence, the efficiencies were increased.

The purpose of this investigation was to determine the maximums in performance and the limits of throughput in a mechanically-aided liquid extraction column. Mechanical power was added to the system by reciprocating a wire mesh packing of high void content in the column. The methyl isobutyl ketone-acetic acid-water ternary system was utilized to evaluate the column performance. The column performance was investigated at the onset of flooding and at operating conditions below flooding. The performance of the column was correlated with the direction of acetic acid transfer, packing reciprocation frequency and displacement, effect of dispersed phase, and the total column throughput.

II. LITERATURE REVIEW

A review of the literature was made for the purpose of examining the performance of the various designs of mechanically-aided liquid extraction columns, and also to determine the methods of calculation of performance correlations. This review includes only extraction equipment utilizing the methyl isobutyl ketone-acetic acid-water system, thereby establishing a common basis for performance comparison.

Mechanically-Aided Liquid Extraction

The ideal extraction column is one which has a high permissible throughput and a high efficiency. The keys to improve mass transfer efficiency are increased turbulence and increased area for mass transfer and the maintenance of high local concentration differences over the column length⁽²¹⁾. With these objectives in view, the basic spray column has been modified with packing, sieve plates, and orifice plates to effect increased turbulence and minimize backmixing⁽¹⁾. Although increased turbulence and efficiency were effected by these modifications, the maximum column throughput was still limited because the source of turbulence was essentially the small difference in density

between the liquid phases. The obvious method for further increasing the column efficiency was a method of producing additional turbulence and transfer area⁽²²⁾. From these considerations, other means of adding mechanical power to extraction systems were developed.

Power Addition by Rotary Motion

In this review extraction columns using a rotating disc or impeller as an agitating element are classified as columns that add mechanical power to the extraction system by rotary motion. All extraction columns in this classification have in common the fundamental principle of being continuous-stagewise equipment.

Oldshue-Rushton Column. Performance data were taken for an extraction column by Oldshue and Rushton⁽¹⁴⁾. The column consisted of a six inch diameter vertical glass column, compartmented by horizontal plates, and baffled by vertical members. The system was agitated by mixing impellers on a vertical shaft. Acetic acid was extracted from water to methyl isobutyl ketone, and also from the ketone to the water. The performance of the column was determined using different size compartments, impellers, acid concentrations, and feed rates.

Throughputs at the onset of flooding as high as 4,400 pounds per hour per square foot were obtained based on the column cross-section area and the combined inlet streams. The best stage efficiency showed a minimum height equivalent to a theoretical stage of 3.7 inches for a throughput of 2,140 pounds per hour per square foot using eight compartments, each 3.0 inches high. This height equivalent to a theoretical stage corresponded to a stage efficiency of 81 per cent. The minimum height equivalent to a theoretical stage was observed with solute transfer from the water to the ketone phase. For a given throughput, the efficiency increased to a maximum with increasing impeller speed and decreased with further increase in speed.

Rotating-Disc Contactor. The rotating-disc contactor, designed by Reman and Olney⁽¹⁵⁾ consisted of a number of compartments formed by a series of stator rings, with a rotating disc centered in each compartment. Feed inlets were oriented vertically for the two and four inch columns, and tangentially for larger diameter columns up to 16 inches in diameter. An extra stator ring and wire mesh grid were fitted into the shell to separate the feed inlet zone from the settling compartment.

Contactors ranging in size from 2 inches to 16 inches were tested with two liquid systems, one being the methyl

isobutyl ketone-acetic acid-water system, to determine the effect of column diameter, throughput, and rotor speed on efficiency. The maximum throughput decreased with increasing rotor speed while the efficiency increased with increasing rotor speed. However, for some combinations of throughput and compartment height, the efficiency passed through a maximum with increasing rotor speeds. This phenomenon was ascribed to backmixing in the extraction zone.

Although the authors did not indicate the direction of solute transfer, throughputs as high as 1,030 gallons per hour per square foot were reported with the water phase dispersed, and a superficial phase velocity ratio of 1.0. Throughputs in excess of 1,030 gallons per hour per square foot resulted in flooding. A minimum height equivalent to a theoretical stage of 4.3 inches was obtained at flow rates of 980 and 700 gallons per hour per square foot.

In general the efficiency increased somewhat with increasing throughput. The main advantage was that little loss in efficiency occurred in scale up over the range of column sizes investigated.

Scheibel Column. The performance of a twelve inch diameter extraction column consisting of alternate agitated and packed sections was determined by Scheibel and Karr⁽¹⁷⁾

using the methyl isobutyl ketone-acetic acid-water system. The column consisted of three stages, with a rotating impeller mixing section and a wire mesh packed section considered to be one stage. The general trends observed by Scheibel and Karr were maximums in efficiency curves as a function of impeller speeds, and efficiency increase with increasing throughput until flooding was approached.

By considering a single stage as a combination mixing-packed section, stage efficiencies exceeding 100 per cent were realized. The minimum height equivalent to a theoretical stage was 9.2 inches with the water phase dispersed, for a maximum throughput of 595 gallons per hour per square foot. Flow rates in excess of 595 gallons per hour per square foot resulted in flooding in the column.

The design of an internally-baffled, rotary-agitated extraction column by Scheibel (16) was an extension of the original extractor design. The new design incorporated doughnut baffles above and below each turbine agitator. The purpose of the baffles was to control the flow pattern in the mixing section so that scale up could be effected with no increase in mixing compartment height.

The column used in this later investigation was an 11.5 inch diameter glass column, with turbine agitator and baffles. For low interfacial tension systems, provision was

made for incorporating alternate wire mesh packed sections to aid in coalescence between agitated sections. The work was with the methyl isobutyl ketone-acetic acid-water system with concentrations as high as 20 weight per cent acetic acid in the aqueous phase. During the tests the water phase was dispersed.

The minimum height equivalent to a theoretical stage observed in the 11.5 inch diameter column was 5.0 inches for a corresponding maximum total throughput of 458 gallons per hour per square foot. This was the maximum permissible column throughput before occurrence of flooding. When packed sections were utilized, the minimum height equivalent to a theoretical stage corresponding to the maximum throughput was reduced to 3.0 inches. Investigations were also made in a smaller, one inch, diameter column and results indicated a ratio of height equivalent to a theoretical stage to column diameter of about 1:4.

Power Addition by Reciprocating Motion

The method of adding mechanical power to an extraction system by reciprocating motion was originated by van Dijck⁽¹⁰⁾. It was proposed by van Dijck to add power by pulsing the liquid through stationary perforated plates, and also by

reciprocating the perforated plates through the liquid phases.

Pulsed Spray Column. The basic spray column was modified by Billerbeck, Farquhar, Reid, Bressie, and Hoffman⁽²⁾ in an effort to provide additional turbulence to increase column efficiency. The method of promoting turbulence was to pulse the liquid inventory in a spray column by means of a bellows connected to the bottom of the column. The pulsation system supplied an approximate sinusoidal pulse of 7/16 inch amplitude with a variable frequency of 0 to 500 cycles per minute. The extraction column was fabricated from a six foot section of glass pipe, 1.5 inches inside diameter. All the tests were carried out with solute transfer from the water phase to the ketone phase, with the ketone phase dispersed. The acetic acid concentration was about 14 weight per cent in the water feed, and the ketone feed was neutral.

The best performance of the pulsed spray column yielded an overall height of a transfer unit of 0.26 feet, for a corresponding throughput of 21,500 pounds per hour per square foot. This throughput of 21,500 pounds per square foot was the maximum throughput reported with pulsation before flooding occurred.

For comparison the column was operated as a spray column with no pulsation. The maximum throughputs before

flooding were approximately 21,800 pounds per hour per square foot with an overall height of a transfer unit of 1.36 feet, based on the ketone phase. Solvent ratios were approximately 1.1 for tests with pulsation and 1.16 for operation as a spray column.

For the most part, at constant flow rates, the overall height of a transfer unit decreased with increasing pulse frequency. At low throughputs the height of a transfer unit decreased to a minimum with increasing pulse frequency and then increased until flooding occurred.

Visual observations by the investigators indicated that increased pulse frequency decreased the drop size for a given flow rate. It was also observed that in tests with no pulsation, drop size decreased with increasing flow rate. On the basis of this work, the authors concluded that greater efficiency is generally obtained if mass transfer is into the dispersed phase in liquid extraction. Other investigators have indicated this, as well as recommending that the phase have the highest throughput be dispersed^(8,18).

Pulsed Sieve-Plate and Pulsed Packed Columns. Extraction efficiencies and performance were studied for the methyl isobutyl ketone-acetic acid-water system in both pulsed-packed and pulsed sieve-plate columns by Chantry, Von Berg, and Wiegant⁽⁷⁾. The two columns, interchangeable

with the auxiliary equipment, were constructed from a four foot section of 1.57 inch inside diameter glass tubing.

The packed column contained a 27 inch section with 1/4 inch procelain Raschig rings. After settling the packing had a dry void fraction of approximately 58 per cent. The sieve-plate column contained 11 plates, spaced three inches apart and held by a central rod. Two types of sieve-plates were used; one with twenty-four 5/64 inch holes, and the other with twenty-four 3/64 inch holes.

Frequencies from 300 cycles per minute were available at amplitudes ranging from zero to ten millimeters. In most tests, 20 weight per cent acetic acid concentration in water was extracted by neutral solvent. The ketone phase was the dispersed phase during operation.

For the pulsed packed column the maximum total throughput before flooding was 149 gallons per hour per square foot with a corresponding minimum height equivalent to a theoretical stage of 5.1 inches. The pulsed sieve-plate column was reported to have a maximum throughput before flooding of 267 gallons per hour per square foot with a corresponding height equivalent to a theoretical stage of 10.1 inches.

Controlled-Cycling Extraction Column. The relatively new concept of controlled-cycling was investigated in liquid extraction by Szabo, Lloyd, Cannon, and Speaker⁽²⁰⁾ using

the methyl isobutyl ketone-acetic acid-water system. With the application of controlled cycling only one phase flows at a time and for a controlled interval. The cycle used consisted of four parts. First, a light phase flow period through the column, then a coalescing period in the column. The third part was the heavy phase flow period through the column, followed by another coalescing period in the column.

Several combinations of column internals were tested, including: sieve plate, sieve plate-packed, and packed sections. A maximum throughput of 2,270 gallons per hour per square foot was found for the controlled-cycling sieve plate column with a height equivalent to a theoretical stage of 8.5 to 19 inches. Better values of heights equivalent to a theoretical stage were reported for other combinations, but at lower flow rates. Szabo, et al reported a height equivalent to a theoretical stage as low as 4.0 inches for a packed column with a corresponding total throughput of approximately 375 gallons per hour per square foot.

Reciprocating-Plate Column. An open type of reciprocating-plate extraction column was developed by Karr⁽¹³⁾. Plates having 5/8 inch diameter holes and 62.8 per cent free space were used. With this design, low heights equivalent to a theoretical stage were achieved at throughputs higher than those reported for other plate and disc-type columns.

Data were obtained in a three inch diameter column for the methyl isobutyl ketone-acetic acid-water system. Throughputs ranged from 547 to 8,837 gallons per hour per square foot, and the corresponding minimum height equivalent to a theoretical stage values were 4.3 and 7.5 inches. It was proposed by the author that scale up of the column would be independent of column diameter; however, information was lacking in confirmation of this conclusion.

It was observed by Karr that no minimums in height equivalent to a theoretical stage were experienced with increasing strokes per minute. Up to the flooding point the height equivalent to a theoretical stage was substantially independent of throughput, but the minimum height equivalent to a theoretical stage increased with increasing throughput. Also, it was observed that the strokes per minute at which flooding occurred decreased with increasing throughput.

The maximum throughput achieved with the column was 1,707 gallons per hour per square foot with a corresponding height equivalent to a theoretical stage of 7.75 inches, with solute transfer from the ketone to the water with the water phase dispersed. A maximum throughput of 1,837 gallons per hour per square foot was achieved with a corresponding height equivalent to a theoretical stage of 7.5 inches, with solute transfer from the water to the ketone with the water

phase dispersed. The author did not indicate the acetic acid concentrations used in the experimental work.

Reciprocating Wire-Mesh Packed Column. A new design of a packed column was developed by Carr⁽³⁾, employing reciprocation of wire mesh packing to add mechanical power to the extraction system. The column was fabricated from a three foot section of three inch inside diameter Pyrex pipe. Inside the column, uniformly wound around a central shaft and covering the inside cross sectional area, was a 23-3/4 inch section of wire mesh packing with approximately 95 volume per cent void space. During operation the packing could be reciprocated at frequencies of 0 to 520 cycles per minute and displacements of 3/16 and 1/8 inches.

The methyl isobutyl ketone-acetic acid-water system was investigated with solute transfer both from the water phase to the ketone phase and from the ketone phase to the water phase. For both directions of solute transfer, the ketone phase was dispersed. With transfer of solute from the water to the ketone, acetic acid concentrations of approximately 3.0 weight per cent in the aqueous feed and 0.5 weight per cent in the ketone feed were used. When transfer of solute was from the ketone to the water phase, feed acetic acid concentrations were approximately 1.5 weight per cent and zero weight per cent, respectively.

The effects of throughput, frequency, and displacement were investigated as parameters of performance. For transfer of acetic acid from the ketone to the water phase, a minimum overall height of a transfer unit of 0.41 feet corresponded to a maximum throughput of 6,000 pounds per hour per square foot, at a frequency of 520 cycles per minute and a displacement of $3/16$ inch. No minimums in the height of a transfer unit were experienced with increasing frequency for solute transfer in this direction.

For solute transfer from the water to the ketone phase, the minimum overall height of a transfer unit was 0.26 feet with a corresponding throughput of 2,000 pounds per hour per square foot, at a frequency of 360 cycles per minute and a displacement of $3/16$ inches. At the maximum reported throughput of 6,000 pounds per hour per square foot, a minimum overall height of a transfer unit of 0.38 feet was observed at a frequency of 200 cycles per minute and $3/16$ inch displacement. Minimums in the height of a transfer unit were experienced with the $3/16$ inch displacement. Minimums in the height of a transfer unit were not attained for the $1/8$ inch displacement. For the $1/8$ inch displacement at 520 cycles per minute and 6,000 pounds per hour per square foot, an overall height of a transfer unit of 0.32 feet was obtained.

The overall height of a transfer unit was based on the ketone phase for both directions of solute transfer. Maximum throughput was limited to 6,000 pounds per hour per square foot, thus preventing extension of the work to higher rates approaching flooding and maximum performance.

Method of Calculation

The calculations of the overall number of transfer units, overall height of a transfer unit, and the height equivalent to a theoretical stage are presented for both directions of solute transfer in the following section. The equations have been developed for dilute solutions and certain necessary assumptions were made to utilize these equations.

Assumptions. It was assumed that the operating lines were straight over the range of concentrations involved. In conjunction it was assumed that the equilibrium-distribution line was straight and passed through the origin. With the assumptions of straight operating and equilibrium lines, it is permissible to use the logarithmic averages of terminal concentration differences⁽²⁴⁾. The equations presented are applicable for differential contact equipment, and the analytical relationships between the height of a transfer

unit for differential equipment, and height equivalent to a theoretical stage for stagewise equipment are given. These equations were originally developed by Colburn and have been since modified for dilute solutions with the assumptions of straight operating and equilibrium lines.

The equations were developed for dilute solutions and may be used for countercurrent extraction without reflux. The calculation of the number of transfer units based on the raffinate and extract phases may be used for the raffinate-stripping and extract-enriching sections of a tower when the appropriate conditions apply. The criteria for dilute solutions are met by the range of acetic acid concentrations used.

Overall Number of Transfer Units. The following equation may be used to calculate the number of transfer units based on the raffinate phase⁽²⁵⁾:

$$N_{\text{toR}} = \frac{\ln \left[\frac{x_1 - y_2/m}{x_2 - y_2/m} \left(1 - \frac{R}{mE} \right) + \frac{R}{mE} \right]}{1 - R/mE} \quad (1)$$

where:

x = concentration of solute in raffinate phase, weight fraction

y = concentration of solute in extract phase, weight fraction

m = slope of equilibrium-distribution curve,
dimensionless

R = raffinate flow, pounds per hour per square foot

E = extract flow, pounds per hour per square foot

N_{toR} = overall number of transfer units, based on the
raffinate phase

subscripts:

1 = that end of a continuous column where raffinate
enters

2 = that end of a continuous column where extract
enters

The following equation may be used to determine the
overall number of transfer units based on the extract
phase: (26)

$$N_{toE} = \frac{\ln \left[\frac{y_2 - mx_1}{y_1 - mx_1} \left(1 - \frac{mE}{R} \right) + \frac{mE}{R} \right]}{1 - mE/R} \quad (2)$$

where:

N_{toE} = overall number of transfer units, based on the
extract phase

Overall Height of a Transfer Unit. The overall height
of a transfer unit based on the raffinate phase is defined
as (27):

$$H_{toR} = Z/N_{toR} \quad (3)$$

where:

Z = effective height of column, feet

H_{toR} = overall height of a transfer unit based on the raffinate phase, feet

The overall height of a transfer unit based on the extract phase was calculated by the following equation (28):

$$H_{toE} = Z/N_{toE} \quad (4)$$

where:

H_{toE} = overall height of a transfer unit based on the extract phase, feet

Height Equivalent to a Theoretical Stage. The height equivalent to a theoretical stage for stagewise equipment is analytically related to the overall height of a transfer unit by the following equation (23):

$$H.E.T.S. = \frac{H_{toR} \ln \epsilon}{1 - 1/\epsilon} = \frac{H_{toE} \ln \epsilon}{\epsilon - 1} \quad (5)$$

where:

ϵ = extraction factor, R/mE

H.E.T.S. = height equivalent to a theoretical stage, feet

III. EXPERIMENTAL

The experimental section of this thesis includes the purpose of investigation, plan of investigation, and a detailed description of the experimental procedure. A section discussing the experimental apparatus and materials used in the research project is also included. The experimental data obtained during the investigation and the calculated results for performance correlations, together with sample calculations and graphical presentation of the results complete the contents of the experimental section.

Purpose of Investigation

The purpose of this investigation was to determine the maximums in performance and the limits of throughput in a mechanically-aided liquid extraction column. Mechanical power was added to the system by reciprocating a wire mesh packing of high void content in the column. The methyl isobutyl ketone-acetic acid-water ternary system was utilized to evaluate the column performance. The column performance was investigated at the onset of flooding and at operating conditions below flooding. The performance of the column was correlated with the direction of acetic

acid transfer, packing reciprocation frequency and displacement, effect of dispersed phase, and the total column throughput.

Plan of Investigation

The plan of investigation was subdivided into three sections. First, a review of the literature was made to determine the factors influencing performance of mechanically-aided liquid extraction equipment, and the methods of calculations to evaluate performance. Second, modifications were made to the existing extraction column designed by Carr⁽⁴⁾, and new auxiliary equipment was provided. Finally, the performance of the mechanically-aided extraction column was evaluated as a function of operating parameters.

Literature Review. The published literature was reviewed for the purpose of determining the various types of mechanically-aided liquid extraction columns and their relative performance with the methyl isobutyl ketone-acetic acid-water system. The efficiencies of the various designs of mechanically-aided columns and the maximum throughputs of the different columns were determined. The effect of operating variables on the performance of the different types of

columns was also studied.

Extraction Column Modifications. The original extraction column, designed by Carr, was used for the experimental work with some minor modifications. New liquid distributors for both liquid phases were fabricated, as a provision for dispersing either phase during operation. The wire mesh packing was re-wound on the axial shaft, and was compressed from 23-3/4 inches to 23 inches total height in an effort to restrict as much as possible any effects of liquid channelling.

The extraction column auxiliary equipment was replaced or modified almost entirely. All process lines were re-installed with 3/8 inch stainless steel tubing, and 55 gallon stainless steel drums were used as feed and product storage tanks. Centrifugal pumps were installed as a means of supplying liquid feeds at higher flow rates than previously achieved with air pressure. A water still was installed for supply of distilled water together with a 55 gallon distilled water storage tank. A higher speed variable speed transmission was connected to the extraction column for operation at higher frequencies, and a new set of reciprocation cams was fabricated for reciprocation of the packing. A feed temperature control system was constructed to control feed temperature within the desired

range of 28 ± 1 degrees centigrade.

Extraction Column Performance. The performance of the extraction column was evaluated at the onset of flooding, and at conditions below flooding to establish the range of column operation. The measure of performance used in this investigation was the overall height of a transfer unit, based on the ketone phase. Corresponding heights equivalent to a theoretical stage values were also calculated. All tests were conducted with a superficial phase velocity ratio of one. The efficiency of column operation was determined both at flooding conditions and below flooding, or normal operating conditions. The performance was evaluated as a function of the direction of acetic acid transfer, packing reciprocation frequency, displacement of the packing, and the total combined inlet stream throughput.

Materials

All materials used in this experimental investigation and their specifications are listed in Appendix A.

Apparatus

The specifications of the equipment used in the experimental investigation are listed in Appendix A. Drawings of

the phase distributors and reciprocation cams, and a Bill of Materials may be found in Appendix B. Drawings and specifications of the other parts of the extraction column are found in the original work by Carr⁽⁵⁾.

Extraction Column. A complete view of the liquid extraction unit is presented in Figure 1, page 25. Close-up views of the top and bottom sections of the extraction column are presented in Figures 2 and 3, pages 26 and 27, respectively. At the time these photographs were taken, no mechanical power was being added to the system. In Figure 2 the interface is shown at the top of the column with the ketone phase dispersed. In Figure 3 the interface was maintained at the bottom of the column with the water phase dispersed.

Wire Mesh Packing. An overall view of the extraction column is presented in Figure 4, page 28, in which the wire mesh packing can be seen. The packing was wound around the shaft in four equal-height sections and stacked together to form a total packing height of 23 inches. The void volume of the wire mesh packing was determined to be 97.7 volume per cent.

Flow Rotameters. The rotameters used to measure the inlet water and ketone phase flow rates and the needle valves used to control flows are also shown in Figure 4.

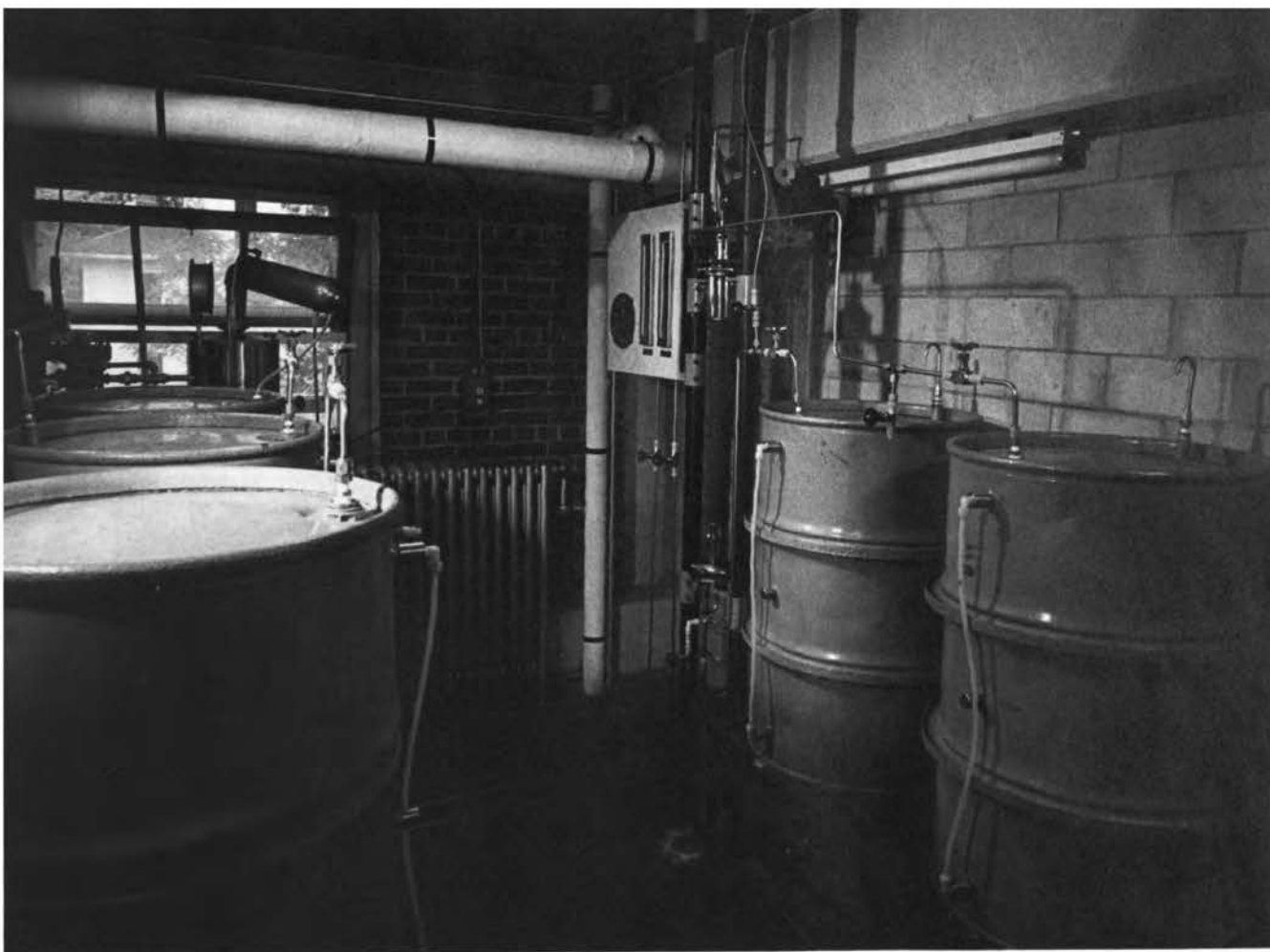


Figure 1. Overall View of the Experimental Extraction Unit and Accessories

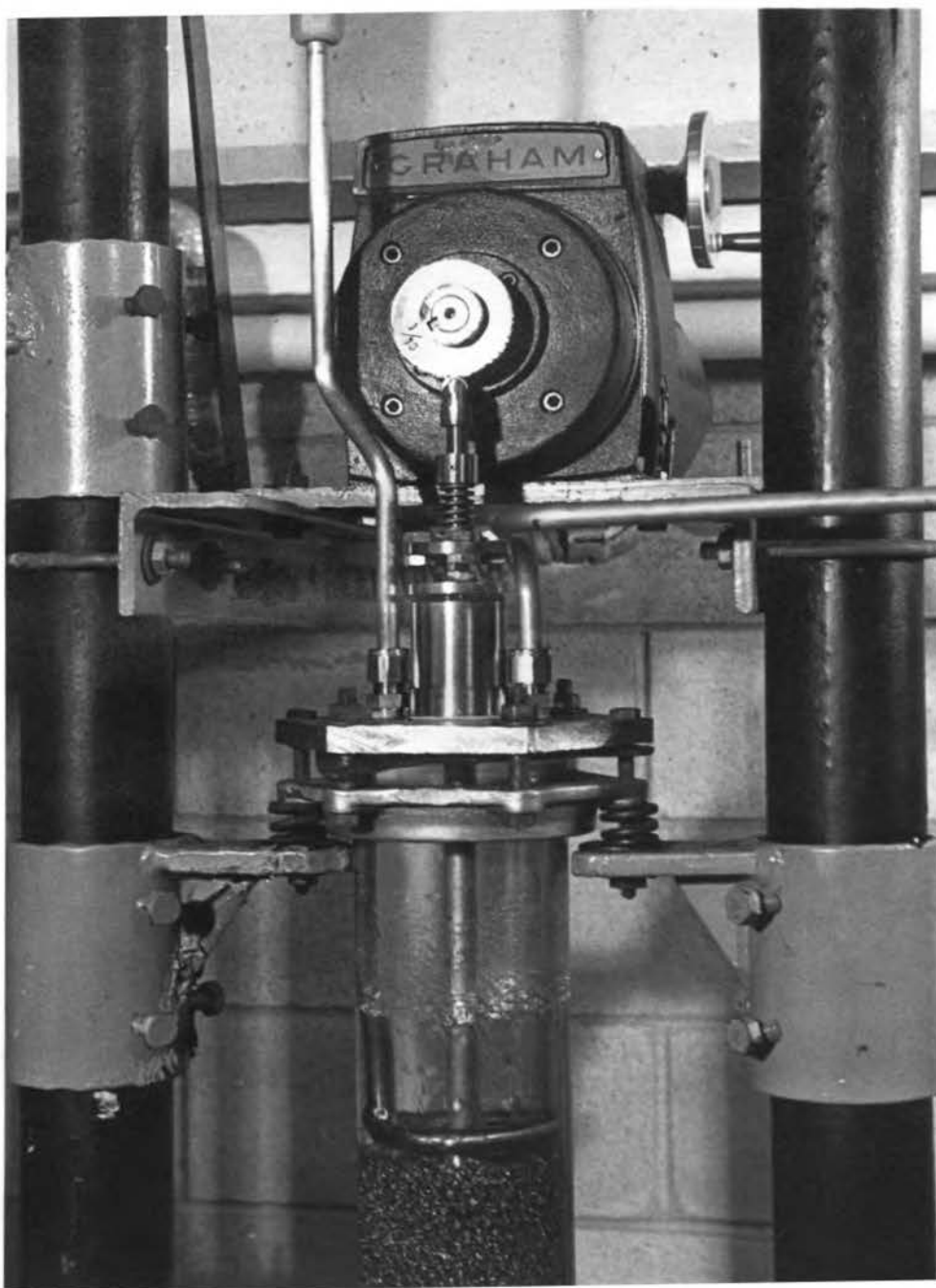


Figure 2. Detailed View of the Top of the Extraction Column

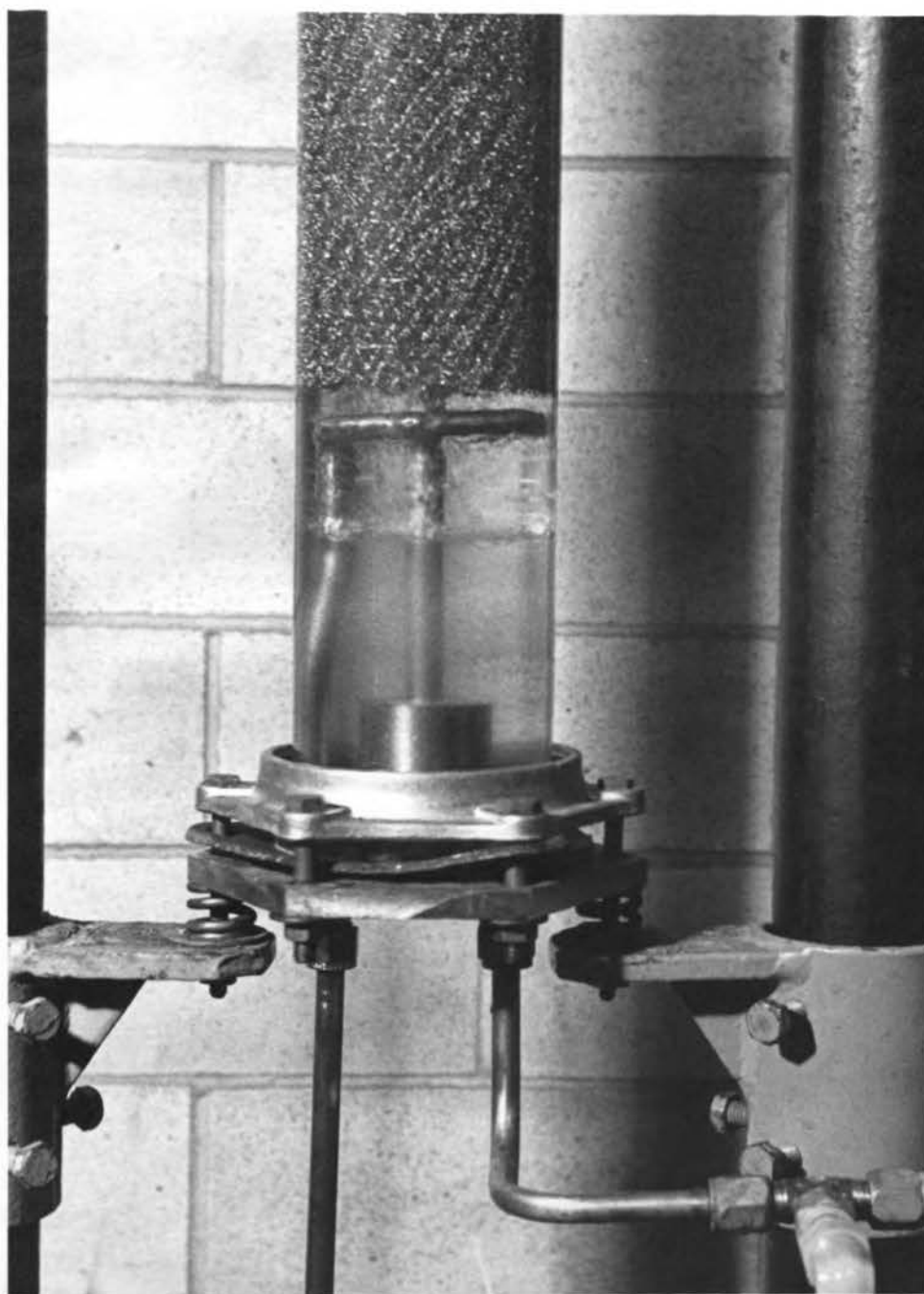


Figure 3. Detailed View of the Bottom of the Extraction Column

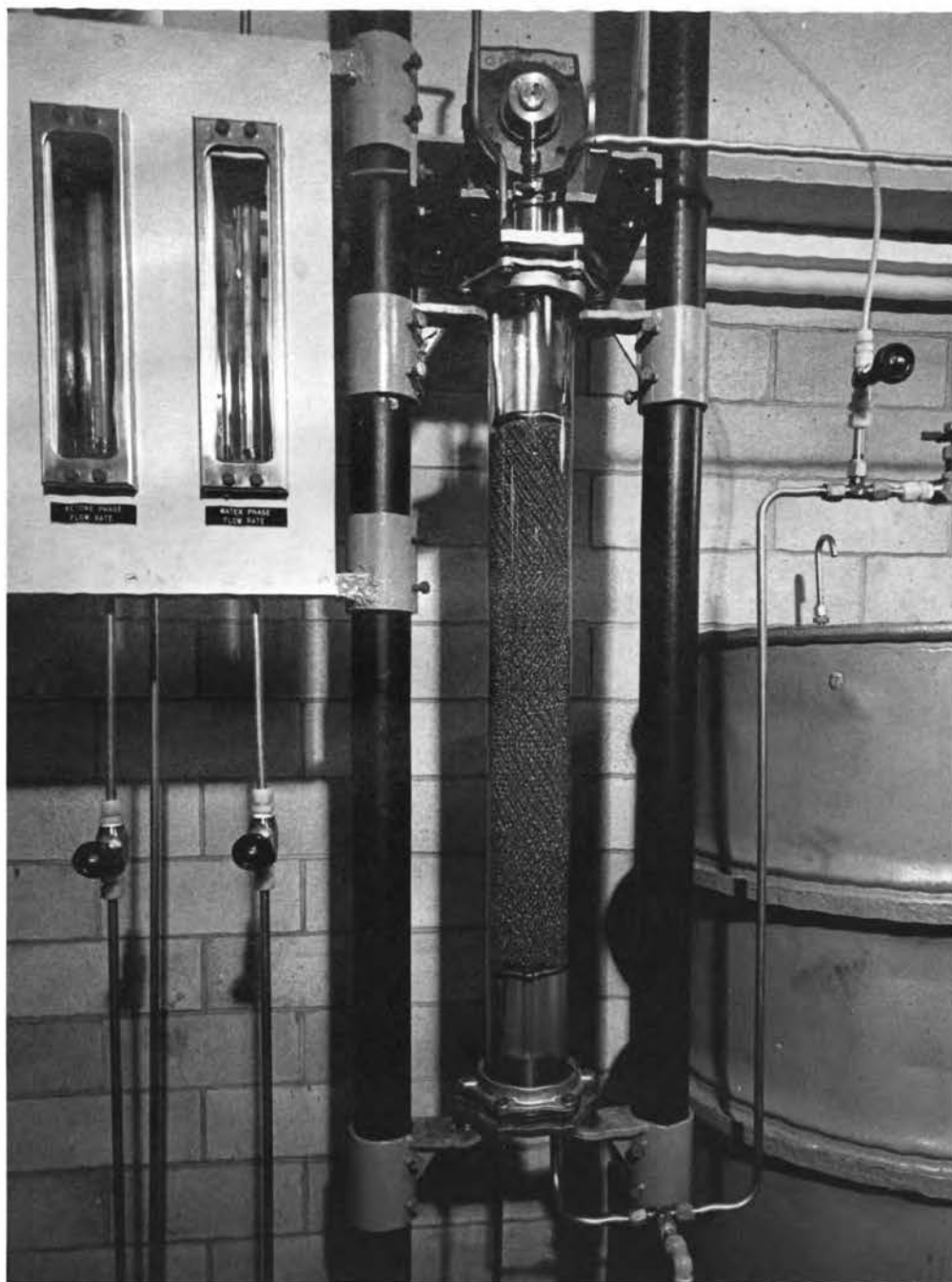


Figure 4. Overall View of the Experimental Liquid Extraction Column

During operation the rotameter float position could be maintained at $\pm 1/2$ scale division, maximum deviation. This deviation in float position corresponded to a maximum deviation of 124 pounds per hour per square foot per phase, giving a maximum possible error in combined inlet-stream flow rate of 248 pounds per hour per square foot.

Phase Distributors. A view of the ketone phase and water phase distributors can be seen in Figures 3 and 2 respectively. Both liquid phases were distributed over the packing cross-sectional area by these distributors and actual operation of the column with the interface at the top or bottom of the column determined which phase was dispersed. The liquid distributors were constructed from 3/8 inch stainless steel tubing, welded into a 2-1/4 inch circle. Thirty 0.0520 inch diameter holes were drilled in the face of the distributor circle to provide an even distribution of liquid over the packing cross-section.

Reciprocation Mechanism. A 0 to 1100 revolutions per minute variable speed transmission with eccentric circular cams was used to reciprocate the packing at different frequencies and displacements. The bearing arrangement for contact of the cam to the packing shaft was the original designed by Carr. The reciprocation cams were connected to the transmission drive shaft by a key and set screw. Three

eccentric cams were utilized, providing displacements of 0.1875, 0.1250, and 0.0938 inches. A close-up view of the reciprocation mechanism can be seen in Figure 2.

Storage Tanks. A view of the ketone feed, water feed, and distilled water storage tanks can be seen in Figure 5, page 31, left to right respectively. The ketone product and water product storage tanks are shown in Figure 1, to the right of the column. The storage tanks were 55 gallon stainless steel drums equipped with nylon sight gauges and nylon and stainless steel fittings.

Feed Temperature Control. The feed temperature control system consisted of double-pipe countercurrent heat exchangers in the feed tank recirculation lines and a constant temperature bath, complete with pump and heaters. If heat were to be removed from the process fluids, cooling water was passed through the bath cooling coil. If heat were to be added to the process fluids, the circulating bath water was heated by a bayonet immersion heater. In all tests, the feed temperatures were controlled at 28 ± 1 degrees Centigrade. A view of the feed temperature control system can be found in Figure 5.

Liquid Motive System. Each feed tank was equipped with a centrifugal pump. The pumps served the purpose of recirculating the process fluids in the feed tanks, supplying



Figure 5. Detailed View of Feed Storage Tanks and Accessories

feeds to the column, and transferring the liquid inventories from the product tanks back to the feed tanks.

Materials of Construction. Materials in contact with the liquid extraction system were: (1) Type 316 Stainless Steel, (2) Type 304 Stainless Steel, (3) Teflon, (4) Nylon, and (5) Pyrex glass.

Experimental Procedure

The calibration procedure for the ketone rotameter, water rotameter, and variable speed transmission are found in Appendix C. The equilibrium-distribution data used in the experimental work were determined by Carr⁽⁶⁾. Methods of experimental procedure for operation of the mechanically-aided extraction column are presented in the following section.

Direction of Solute Transfer. During experimental operation, acetic acid was alternately transferred between the ketone phase and the water phase. A complete cycle of operation consisted of first transferring the acetic acid from the ketone to the water, then increasing the total acetic acid concentration in the water, and transferring the acetic acid in the reverse direction. For both directions of solute transfer the feed solutions were mutually saturated.

For transfer of acetic acid from the ketone phase to the water phase, ketone of approximately 1.5 weight per cent acetic acid was contacted with water of almost zero per cent acid. As a result of this contact, the approximate concentrations of acetic acid in the ketone and water phases leaving the column were 0.5 and 1.0 weight per cent respectively.

After acetic acid had been transferred from the ketone to the water phase, the acetic acid concentration in the water was increased from approximately 1.0 weight per cent to about 3.0 weight per cent by addition of acid. This water of approximately 3.0 weight per cent acid was then contacted with the ketone product from the first contact, containing approximately 0.5 weight per cent acetic acid. As a result of this second contact the acid concentrations were about 2.0 weight per cent in the water and 1.5 weight per cent in the ketone. This second contact completed the second part of the cycle, and the water containing 2.0 weight per cent acid was discarded. Fresh distilled water was then saturated with ketone in preparation for repetition of the cycle.

Feed Preparation. The following procedure refers to the schematic diagram of the extraction unit, Figure 6, page 34. According to the direction of acetic acid transfer,

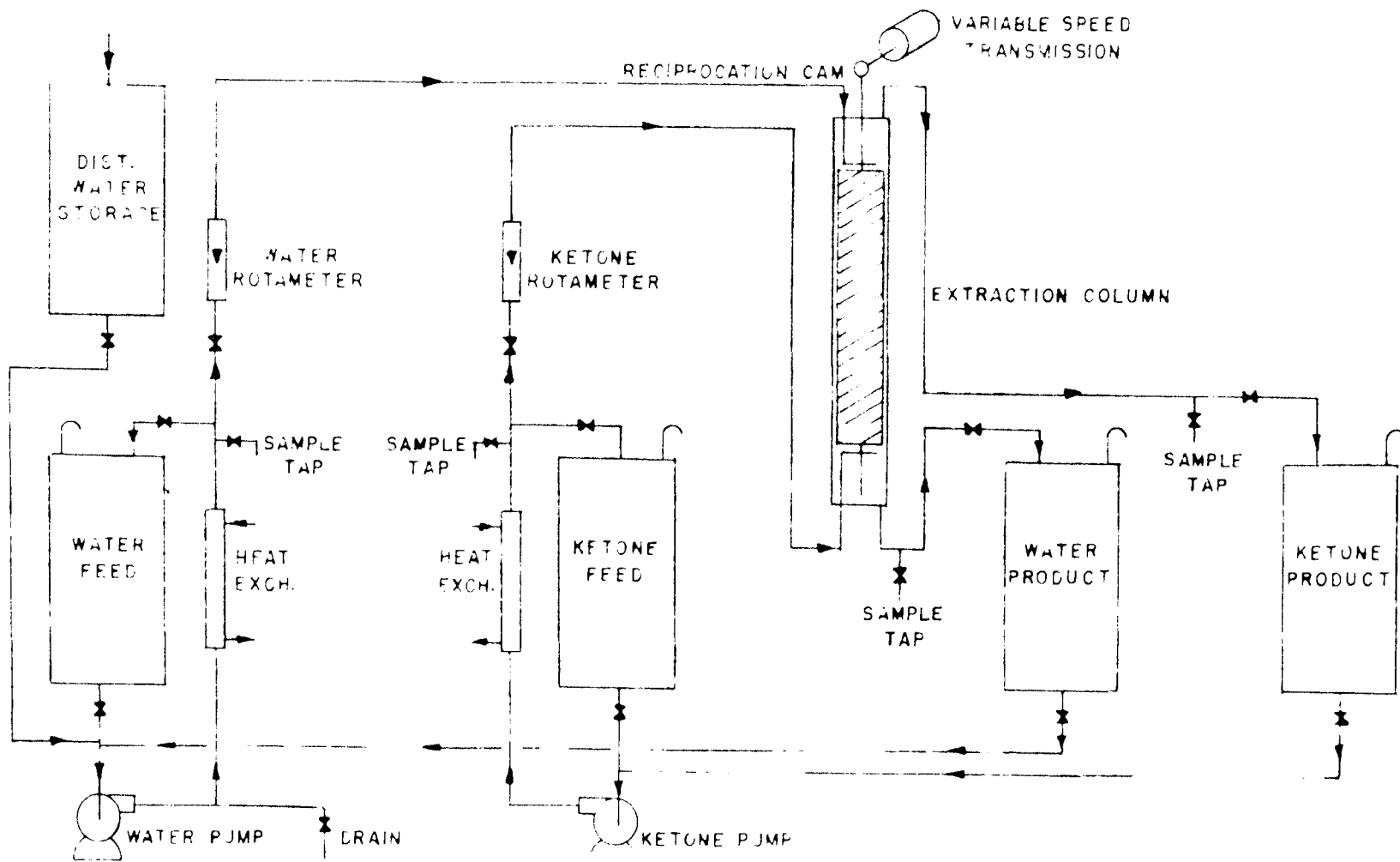


Figure 6. Schematic Drawing of the Experimental Liquid Extraction Unit

variations in feed preparation were employed.

For transfer of acetic acid from the ketone phase to the water phase, distilled water was pumped from the distilled water storage tank to the water feed tank with the water feed pump. An excess of ketone required for saturation of the water was added to the water feed tank, and the contents of the tank were circulated for approximately one-half hour to assure complete saturation of the water phase. The ketone product from the previous contact was pumped from the ketone product tank to the ketone feed tank with the ketone feed pump. Then the contents of the ketone feed tank were circulated for about one-half hour to assure a uniform concentration. The resulting concentration was approximately 1.5 weight per cent acetic acid in the ketone.

After the water had been contacted with the ketone containing 1.5 weight per cent acetic acid, provisions were then made for transfer of acetic acid from the water phase to the ketone phase. Both ketone and water products were pumped back to their respective feed tanks. A weighed amount of acetic acid necessary to increase the concentration of acetic acid to approximately 3.0 weight per cent was added to the water feed tank. The contents of both feed tanks were then circulated for approximately one-half hour to assure uniform concentrations. After circulation,

the acid concentrations were about 3.0 and 0.5 weight per cent in the water and ketone phases, respectively.

For both directions of transfer, the temperatures of the feeds were taken during circulation for uniform concentration. If either the water or ketone phase feed temperature were in excess of 29 degrees Centigrade, cool water was circulated through the heat exchangers until the feed temperatures were within the 27 to 29 degree Centigrade tolerance, and as close to 28 degrees Centigrade as possible. If either of the feed temperatures were below 27 degrees Centigrade the temperature of the constant temperature bath water was heated to approximately 40 degrees Centigrade. The warm water was circulated through the heat exchangers until the feed temperatures were approximately 28 degrees Centigrade.

After the feeds were circulated and at the correct temperature, the sample lines were purged and approximately 50 to 60 milliliter samples of both feed tanks were taken. The feed temperatures were then recorded and further preparations for start-up were made. Approximately eight to ten tests could be made from full feed tanks, comprising a single series of tests. One feed sample was taken for each test series.

Frequency and Displacement Adjustment. The reciprocation frequency was set by adjusting the transmission micrometer control to the proper setting, obtained from the variable speed transmission calibration plot. The selection of a particular eccentric circular cam determined the desired packing displacement. The cam was installed on the transmission drive shaft, aligned with the bearing, and then secured by the key and set screw. During a series of tests at a given displacement the frequency could be adjusted with the transmission running. The normal procedure was to select a displacement for a series of tests and to change the frequency or flow rates for the individual tests in the series.

Extraction Column Start-Up (Normal Operation). Both feed tank recirculation valves were closed, and the feed pumps were started. The ketone and water feed valves were opened simultaneously, allowing approximately one-third scale flow on both feed rotameters. The column was allowed to completely fill with the interface remaining somewhere in the middle of the column. Both the ketone and water phase outlet valves were opened slightly, and the variable speed transmission was started.

The interface was controlled at the top or bottom of the column by throttling the appropriate column outlet

valves. The interface was maintained at a constant position approximately plus or minus one-quarter of an inch. With the water phase dispersed, the interface was maintained at a position approximately three inches below the bottom of the packing, or three inches above the packing if the ketone phase was dispersed.

After the interface was at the desired level, the ketone and water phase flow rates were adjusted to predetermined settings to obtain the total desired flow and equal volumetric phase flow rates.

The minimum column volume exchanges required for equilibrium to be reached were determined by periodic sampling and analysis, for both directions of acid transfer at the largest power inputs and flow rates. The minimum volume exchanges necessary for equilibrium were determined prior to the beginning of the experimental tests. A plot of water phase rotameter position versus time required for equilibrium was constructed with parameters for direction of transfer. This plot was used to determine the time required for a test at a given flow rate and is shown in Figure 21, Appendix C.

If the acetic acid transfer were from the ketone phase to the water phase, a minimum time for exchange of six column volumes was allowed for equilibrium to be reached. For transfer of acetic acid from the water phase to the ketone

phase it was only necessary to allow time for five column volume exchanges for equilibrium to be reached.

Extraction Column Start-Up (Flooding Tests). The operation of the extraction column differed slightly from normal when flooding tests were being conducted. In general the start-up procedure for flooding tests was identical to normal except the flow rates of both phases could not be predetermined.

A plot of rotameter positions versus total flow rate was made giving corresponding ketone and water phase rotameter positions necessary for equal volumetric flow rates. This plot was used during flooding tests and is shown in Figure 22, Appendix C.

After initial normal start-up procedures had been accomplished and the interface was in position, both the water and ketone phase flow rates were increased simultaneously and incrementally according to Figure 22. The phase flow rates were increased until a total flow rate caused flooding to occur had been reached. Flooding is defined as the point at which the continuous phase flow rate is so high that the droplet flow of the dispersed phase is retarded and is entrained with the continuous phase, resulting in an unsteady-state operation. At this point the flow rates were decreased very slightly to a point at which the column was

operating in a stable manner with no build-up or entrainment of the dispersed phase in the continuous phase. This level of stable operation was termed the "onset of flooding", and the test was initiated by starting the timer.

Extraction Column Shutdown. After equilibrium conditions had been reached for steady-state operation, samples of the ketone and water phase outlet streams were taken. Before each sample was taken, the sample valve was opened and the sample lines were purged of any stagnant liquid. Approximately 50 to 60 milliliter samples of both outlet streams were taken and stored in glass-stoppered bottles.

The ketone and water phase inlet valves were closed; both outlet valves were closed, and the variable speed transmission was stopped. The feed pumps were shut off, and this completed the shutdown procedure.

Sample Analysis. For the water phase analysis, a ten milliliter sample was pipetted from the sample container, transferred to a 150 milliliter beaker and diluted with approximately 25 milliliters of carbon-dioxide free distilled water. Three drops of neutral phenolphthalein solution were added, and the solution was titrated with previously standardized, approximately 0.1 normal, sodium hydroxide solution. The contents of the beaker were stirred with a magnetic stirrer during titrations.

For the ketone phase titrations, the ten milliliter titration sample was diluted with approximately 75 milliliters of distilled carbon-dioxide free water to facilitate rapid transfer of acid from the ketone phase to the water phase. This aided the titration since neutralization of the acid took place in the aqueous phase only. Titration was then conducted in the same manner as the water phase titration, allowing time for the ketone phase to equilibrate with the water phase during titration.

Data and Results

The performance of the mechanically-aided extraction column is presented in the following section. The column performance was evaluated as a function of reciprocation frequency with parameters of packing displacement and combined inlet stream throughput. Performance was evaluated for transfer of acetic acid from the water to the ketone phase, and also for transfer of acid from the ketone to the water phase.

The performance was also observed at the onset of flooding. The effect of reciprocation frequency on flooding rates at various packing displacements was also investigated for both directions of solute transfer. The extraction

column performance was further subdivided into performance with the ketone dispersed, and performance with the water phase dispersed both at the onset of flooding and conditions below flooding.

The calculated results of performance for acetic acid transfer from the ketone phase to the water phase appear in Table I, page 43. The results for performance with acetic acid transfer from the water phase to the ketone phase are found in Table II, page 44. The data taken in the course of evaluation of column performance are found in Appendix C. The data and calibrations for the inlet stream rotameters and the variable speed transmission are also found in Appendix C.

The Effect of Combined Stream Throughput. The effect of combined stream throughput on performance with acetic acid transfer from the ketone phase to the water phase, with the ketone dispersed, appears in Figure 7, page 45, and with the water dispersed in Figure 8, page 46. The same effect for transfer from the water phase to the ketone phase, with the ketone and water dispersed, appears in Figures 9 and 10, pages 47 and 48 respectively.

The Effect of Reciprocation Frequency. The effect of reciprocation frequency on performance for transfer from the ketone to the water phase, with the ketone and water

TABLE I

Extraction Column Performance with Acetic Acid Transfer from the
K to the W Phase to the W to the K Phase

Test No.	Packing Displacement in.	Recirculation Frequency cycles/min.	Dispersed Phase	Combined Stream Throughput lb/hr-ft ²	Overall Height of a Transfer Unit ft.	Material Balance Deviation %
3.04F	.1875	1100	K	3,900	1,200	.62
13.10	.1875	1100	K	5,000	700	.55
3.03F	.1875	825	K	9,900	1,300	.66
13.01	.1875	825	K	5,000	700	.74
3.02F	.1875	550	K	11,800	1,600	.72
13.02	.1875	550	K	10,100	1,300	.91
13.03	.1875	550	K	5,000	700	.83
3.01F	.1875	275	K	14,500	1,900	.93
13.04	.1875	275	K	10,100	1,300	1.19
13.05	.1875	275	K	5,000	700	.94
1.06F	.1250	1100	K	14,300	1,900	.70
11.06	.1250	1100	K	10,100	1,300	.72
13.06	.1250	1100	K	5,000	700	.78
1.05F	.1250	825	K	14,500	1,900	.76
11.07	.1250	825	K	10,100	1,300	.81
13.07	.1250	825	K	5,000	700	.88
1.04F	.1250	550	K	15,100	2,000	.97
11.08	.1250	550	K	10,100	1,300	1.00
13.08	.1250	550	K	5,000	700	.93
1.03F	.1250	275	K	15,700	2,100	1.08
11.09	.1250	275	K	10,100	1,300	1.21
13.09	.1250	275	K	5,000	700	1.10
1.02F	.0938	1100	K	14,800	2,000	.93
3.06F	.0938	825	K	15,100	2,000	.85
20.10	.0938	825	K	10,100	1,300	.71
9.04F	.0000	0	K	18,200	2,400	1.26
22.06	.0000	0	K	15,100	2,000	1.08
22.05	.0000	0	K	10,100	1,300	1.10
22.04	.0000	0	K	5,000	700	.71
5.02F	.1875	1100	W	10,100	1,300	.95
20.07	.1875	1100	W	5,000	700	1.14
5.01F	.1875	825	W	11,700	1,600	1.10
5.05	.1875	825	W	10,100	1,300	.89
20.02	.1875	825	W	5,000	700	1.01
5.03F	.1875	550	W	15,400	2,100	.80
11.01	.1875	550	W	10,100	1,300	.67
13.11	.1875	550	W	5,000	700	.38
20.03	.1875	550	W	5,000	700	.42
5.04F	.1875	275	W	21,000	2,800	.71
11.02	.1875	275	W	15,100	2,000	.63
20.04	.1875	275	W	10,100	1,300	.39
13.12	.1875	275	W	5,000	700	.47
7.01F	.1250	1100	W	13,200	1,800	.87
18.01	.1250	1100	W	5,000	700	.96
7.02F	.1250	825	W	16,200	2,200	.77
11.03	.1250	825	W	10,100	1,300	.67
18.02	.1250	825	W	5,000	700	.50
7.03F	.1250	550	W	19,000	2,500	.80
11.04	.1250	550	W	15,100	2,000	.58
18.05	.1250	550	W	10,100	1,300	.43
18.03	.1250	550	W	5,000	700	.26
7.04F	.1250	275	W	20,600	2,700	.88
11.05	.1250	275	W	15,100	2,000	.66
20.01	.1250	275	W	10,100	1,300	.45
18.04	.1250	275	W	5,000	700	.40
9.01F	.0938	1100	W	19,300	2,600	.76
20.05	.0938	1100	W	10,100	1,300	.89
20.06	.0938	1100	W	5,000	700	.39
9.02F	.0938	825	W	19,700	2,600	.90
20.08	.0938	825	W	10,100	1,300	.26
20.09	.0938	825	W	5,000	700	.28
9.03F	.0000	0	W	16,500	2,200	1.13
22.03	.0000	0	W	15,100	2,000	.61
22.02	.0000	0	W	10,100	1,300	.60
22.01	.0000	0	W	5,000	700	.68

Notes: F indicates performance at the onset of flooding.
 K indicates ketone.
 W indicates water.

TABLE II
Extraction Rate Performance with Acetic Acid Transfer from the
Water Phase to the Aqueous Phase

Test No.	Parting Displacement in.	Reciprocation Frequency Cycles/min.	Dispersed Phase	Combined Stream Throughput lb/hr-Ft ² gal/hr-Ft ²		Overall Height of a Transfer Unit Ft.	Material Balance Deviation %
12.01F	.1875	1100	K	17,000	1,300	1.23	4.8
4.05F	.1875	825	K	13,800	1,900	.60	8.3
19.05	.1875	825	K	10,100	1,400	1.44	.7
19.07	.1875	825	K	5,000	700	.88	.2
4.06F	.1875	550	K	16,100	2,200	.60	9.4
19.06	.1875	550	K	10,100	1,400	.87	-.1
19.08	.1875	550	K	5,000	700	.82	-.5
12.02F	.1875	275	K	21,500	2,900	.92	.1
14.01	.1875	275	K	10,100	1,400	1.06	-.1
14.02	.1875	275	K	5,000	700	.83	2.0
4.04F	.1250	1100	K	15,500	2,100	.66	6.2
19.01	.1250	1100	K	10,100	1,400	1.03	.1
19.02	.1250	1100	K	5,000	700	.83	1.4
4.03F	.1250	825	K	16,900	2,300	.70	5.3
19.04	.1250	325	K	10,100	1,400	.93	.9
19.03	.1250	825	K	5,000	700	.85	.4
4.02F	.1250	550	K	19,700	2,700	.82	1.5
14.03	.1250	550	K	15,100	2,100	.96	-.2
14.04	.1250	550	K	10,100	1,400	.94	.2
14.05	.1250	550	K	5,000	700	.76	1.3
4.01F	.1250	275	K	20,800	2,800	1.27	-.6
14.06	.1250	275	K	15,100	2,100	1.24	1.0
14.07	.1250	275	K	10,100	1,400	1.22	.0
14.08	.1250	275	K	5,000	700	1.02	1.6
2.06F	.0938	1100	K	16,700	2,300	.63	4.5
12.03	.0938	1100	K	15,100	2,100	.83	-1.2
2.05F	.0938	825	K	19,300	2,600	.60	6.2
12.04	.0938	825	K	15,100	2,100	.88	.8
19.09	.0938	825	K	10,100	1,400	1.12	-.3
19.10	.0938	825	K	5,000	700	1.09	-.2
2.04F	.0938	550	K	21,600	2,900	1.16	1.6
12.05	.0938	550	K	15,100	2,100	1.07	.5
2.03F	.0938	275	K	22,600	3,100	1.15	2.2
12.06	.0938	275	K	15,100	2,100	1.28	.5
10.04F	.0000	0	K	19,700	2,700	1.30	.6
23.06	.0000	0	K	15,100	2,100	1.12	1.3
23.05	.0000	0	K	10,100	1,400	1.36	1.3
23.04	.0000	0	K	5,000	700	1.44	1.7
10.01F	.1875	825	W	12,500	1,700	1.16	.0
10.02F	.1875	550	W	15,400	2,100	.86	.8
8.01F	.1250	1100	W	15,000	2,000	1.20	-.7
8.02F	.1250	825	W	17,000	2,300	.97	.8
12.07	.1250	825	W	15,100	2,100	.90	.7
8.05	.1250	825	W	10,100	1,400	.75	.6
21.10	.1250	825	W	5,000	700	.58	.4
8.03F	.1250	550	W	18,600	2,500	.88	.3
12.08	.1250	550	W	15,100	2,100	.78	.5
16.02	.1250	550	W	10,100	1,400	.74	.4
14.10	.1250	550	W	5,000	700	.73	2.3
8.04F	.1250	275	W	21,200	2,900	1.13	.7
16.07	.1250	275	W	15,100	2,100	1.07	.3
16.06	.1250	275	W	10,100	1,400	1.03	-.2
14.09	.1250	275	W	5,000	700	.98	2.1
6.01F	.0938	1100	W	16,400	2,200	.67	-.2
19.11	.0938	1100	W	10,100	1,400	.45	-4.0
21.01	.0938	1100	W	5,000	700	.56	4.1
6.02F	.0938	825	W	18,200	2,500	.76	.7
21.02	.0938	825	W	15,100	2,100	.75	1.1
23.08	.0938	825	W	10,100	1,400	.88	.7
21.09	.0938	825	W	5,000	700	.78	2.1
6.03F	.0938	550	W	19,700	2,700	.98	.2
6.05	.0938	550	W	15,100	2,100	.99	-.1
21.04	.0938	550	W	10,100	1,400	.93	.9
21.05	.0938	550	W	5,000	700	.78	3.8
6.04F	.0938	275	W	20,700	2,800	1.13	.3
21.06	.0938	275	W	15,100	2,100	1.00	.8
21.07	.0938	275	W	10,100	1,400	.89	1.7
21.08	.0938	275	W	5,000	700	.89	3.4
10.03F	.0000	0	W	20,100	2,700	1.33	-.4
23.03	.0000	0	W	15,100	2,100	1.03	1.7
23.02	.0000	0	W	10,100	1,400	1.02	1.6
23.01	.0000	0	W	5,000	700	1.12	2.3

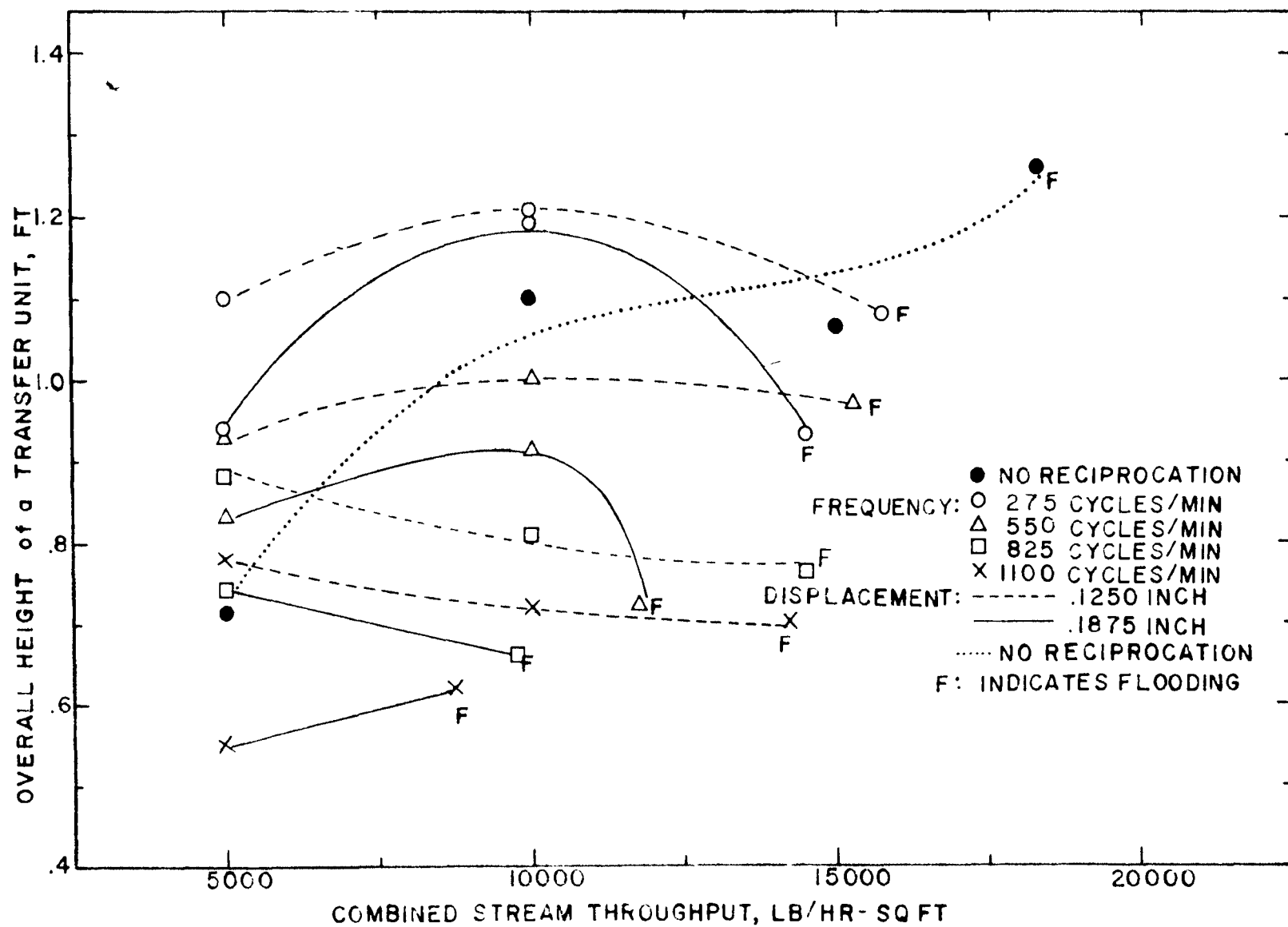


Figure 7. The Effect of Combined Stream Throughput on Performance with Transfer from the Ketone Phase to the Water Phase, Ketone Dispersed

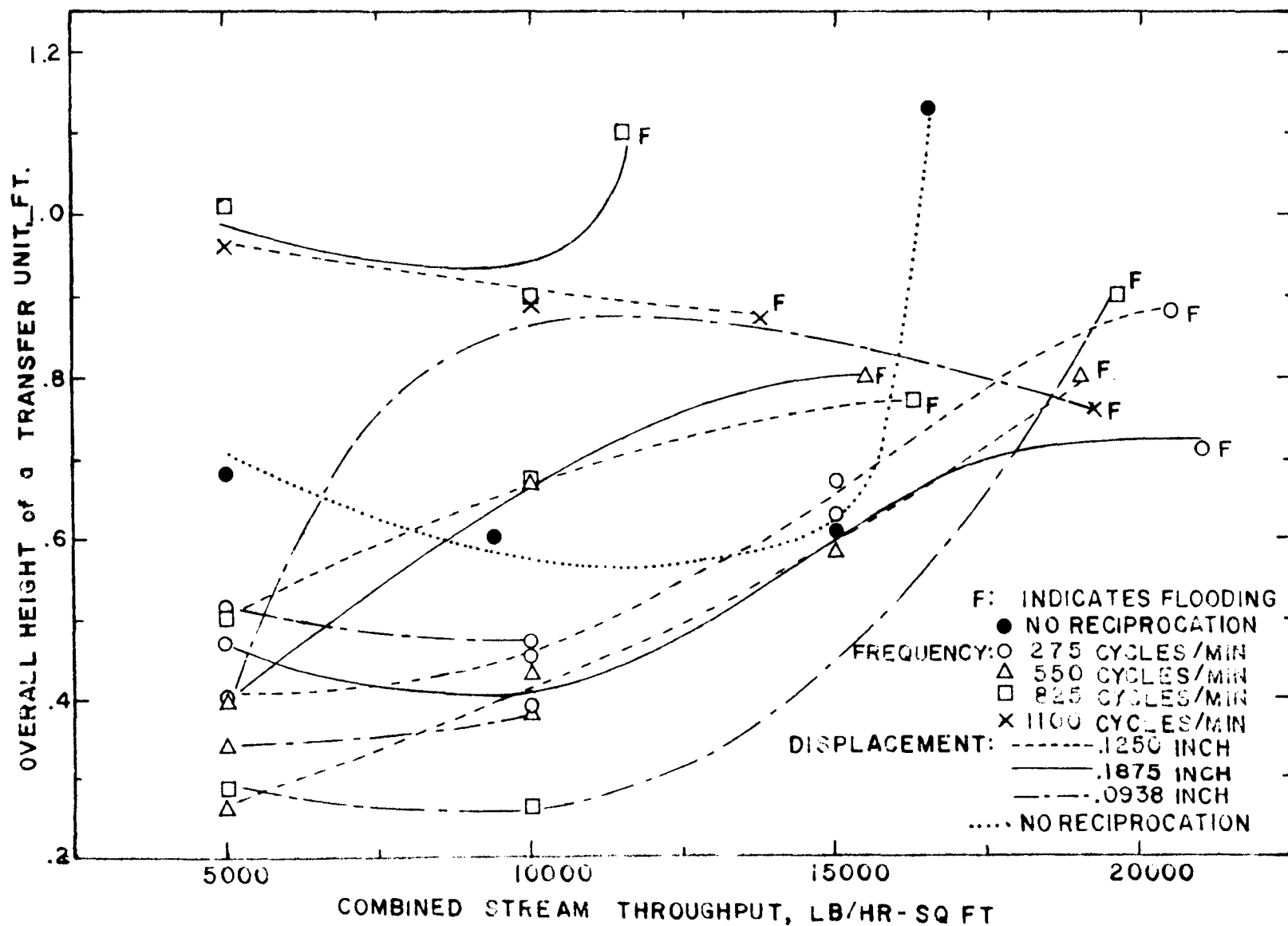


Figure 8. The Effect of Combined Stream Throughput on Performance with Transfer from the Ketone Phase to the Water Phase, Water Dispersed

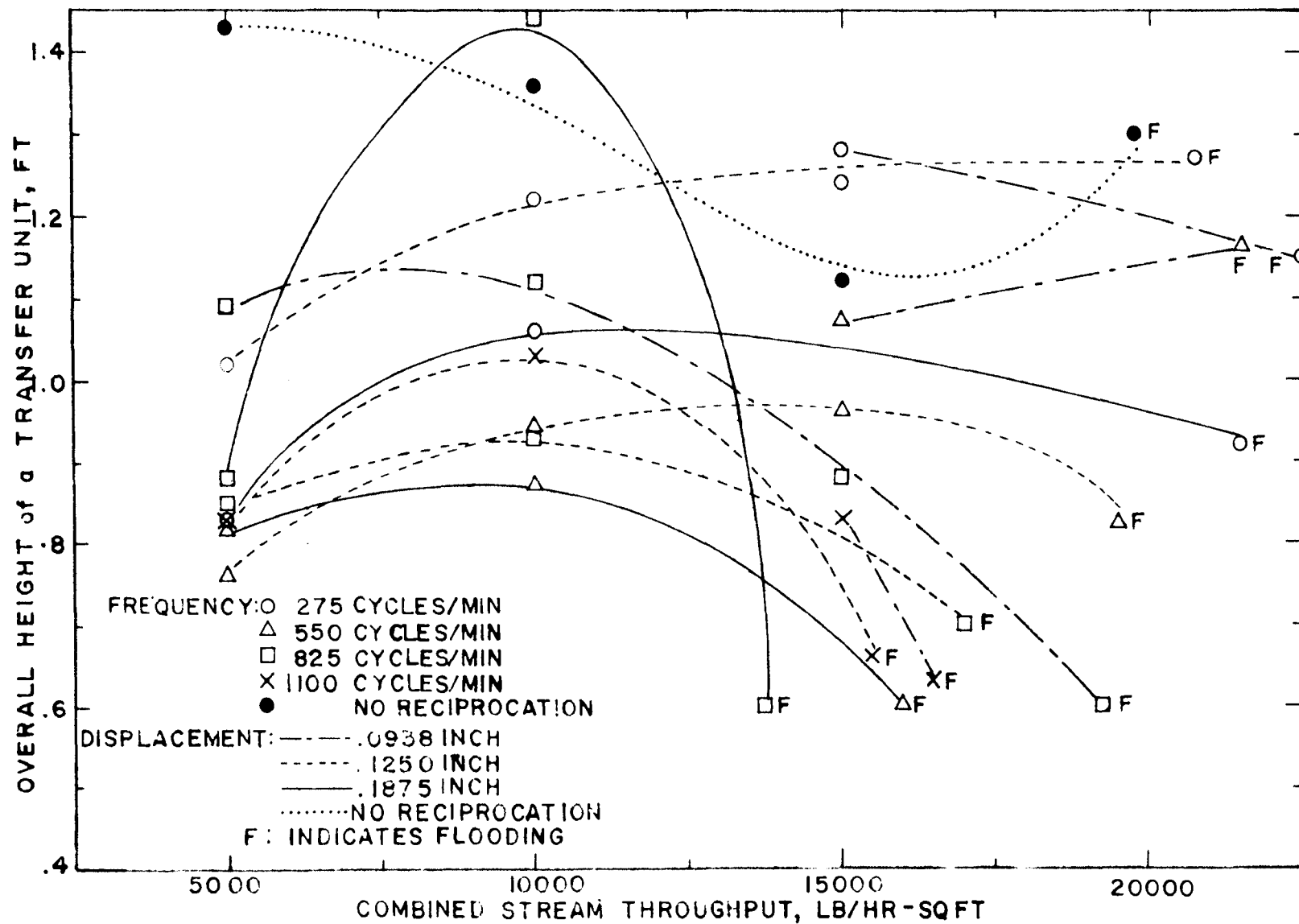


Figure 9. The Effect of Combined Stream Throughput on Performance with Transfer from the Water Phase to the Ketone Phase, Ketone Dispersed

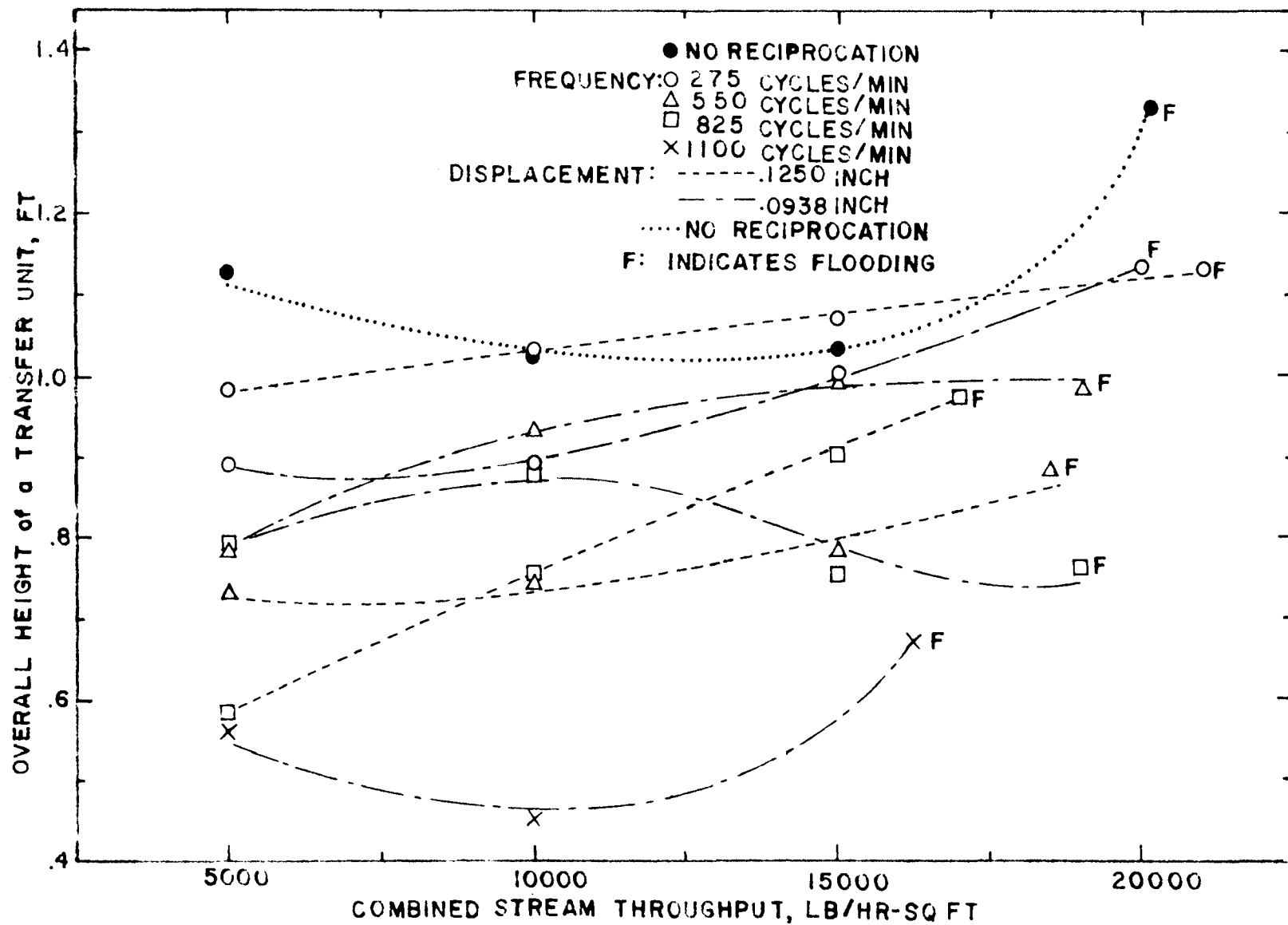


Figure 10. The Effect of Combined Stream Throughput on Performance with Transfer from the Water Phase to the Ketone Phase, Water Dispersed

dispersed, is found in Figures 11 and 12, pages 50 and 51, respectively. The effect of reciprocation frequency on performance for transfer from the water to the ketone phase is found in Figures 13 and 14, pages 52 and 53, respectively. Figure 13 shows the effect with the ketone dispersed, and Figure 14 with the water phase dispersed.

Performance at the Onset of Flooding. For solute transfer from the ketone phase to the water phase, the performance of the column was evaluated at the onset of flooding. The effect of packing reciprocation frequency on flooding rates is found in Figures 15 and 16, pages 54 and 55, with the ketone and water dispersed, respectively. For transfer from the water phase to the ketone phase, the effect of reciprocation frequency on flooding rates is found in Figures 17 and 18, pages 56 and 57, respectively. Figure 19 shows the effect with the ketone dispersed, and Figure 20 with the water dispersed.

Calculations. The calculation of extraction column performance from experimental data was accomplished with the aid of an IBM 1620 Model II computer. The computer program used for these calculations appears in Appendix C.

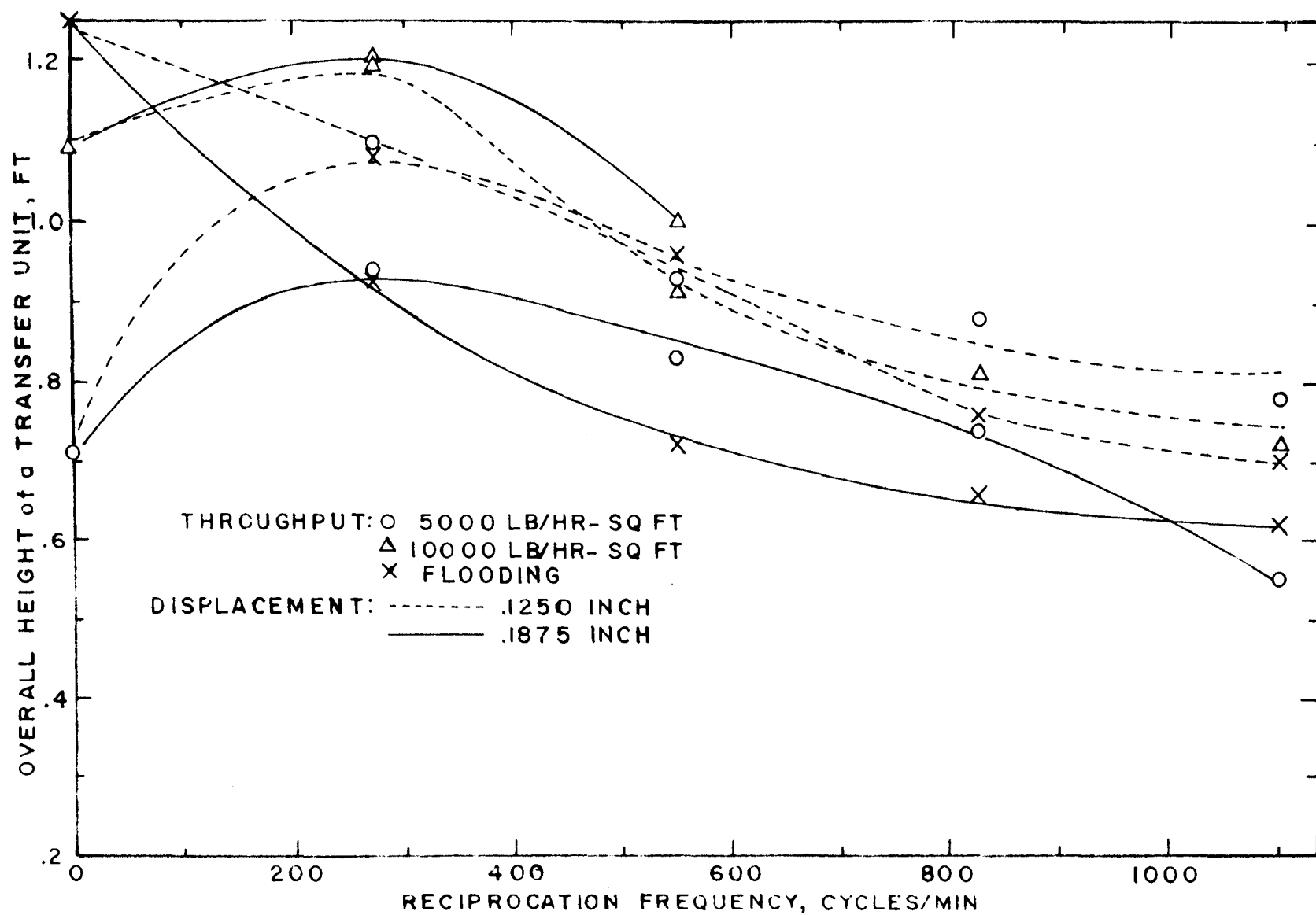


Figure 11. The Effect of Reciprocation Frequency on Performance with Transfer from the Ketone Phase to the Water Phase, Ketone Dispersed

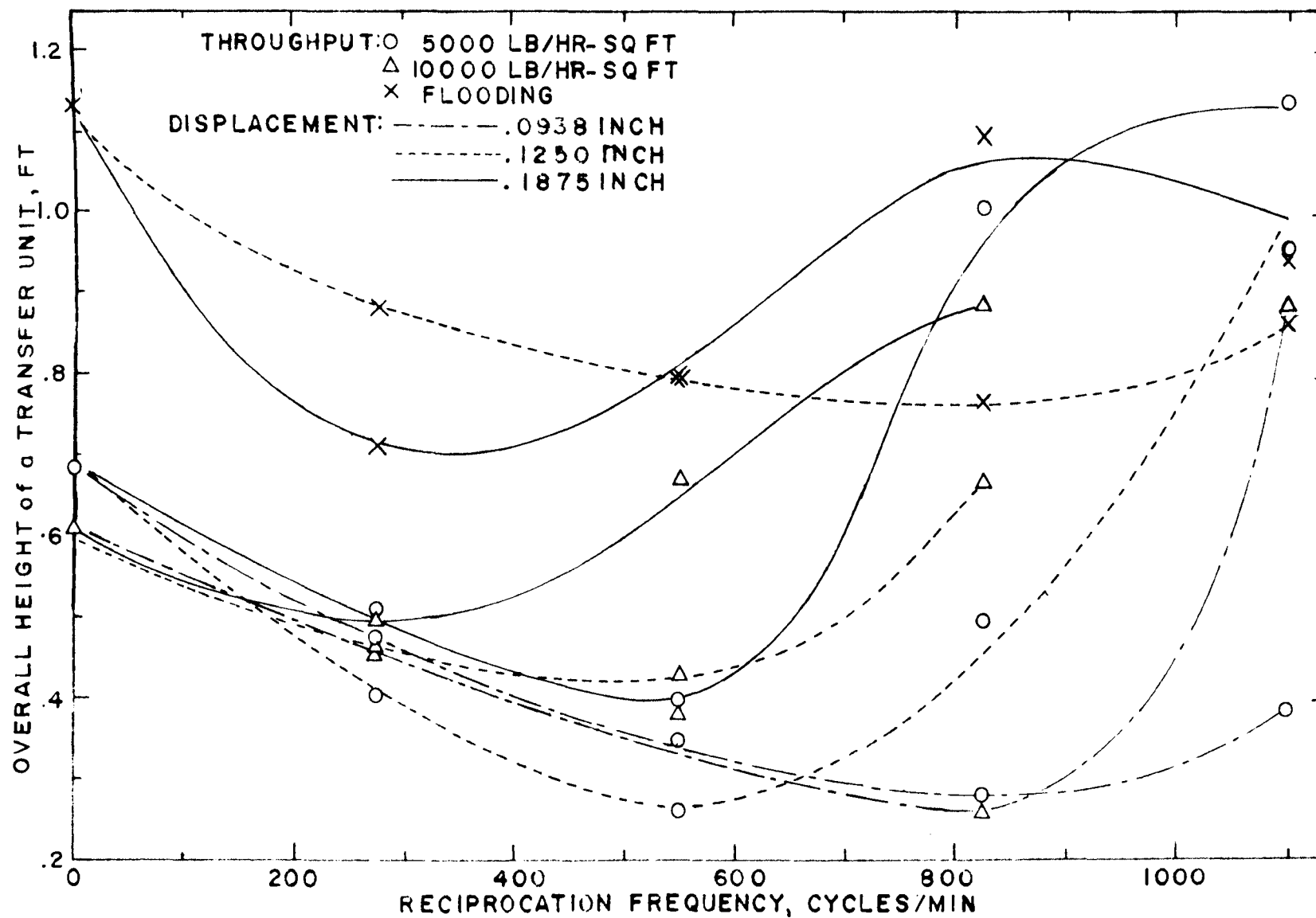


Figure 12. The Effect of Reciprocation Frequency on Performance with Transfer from the Ketone Phase to the Water Phase, Water Dispersed

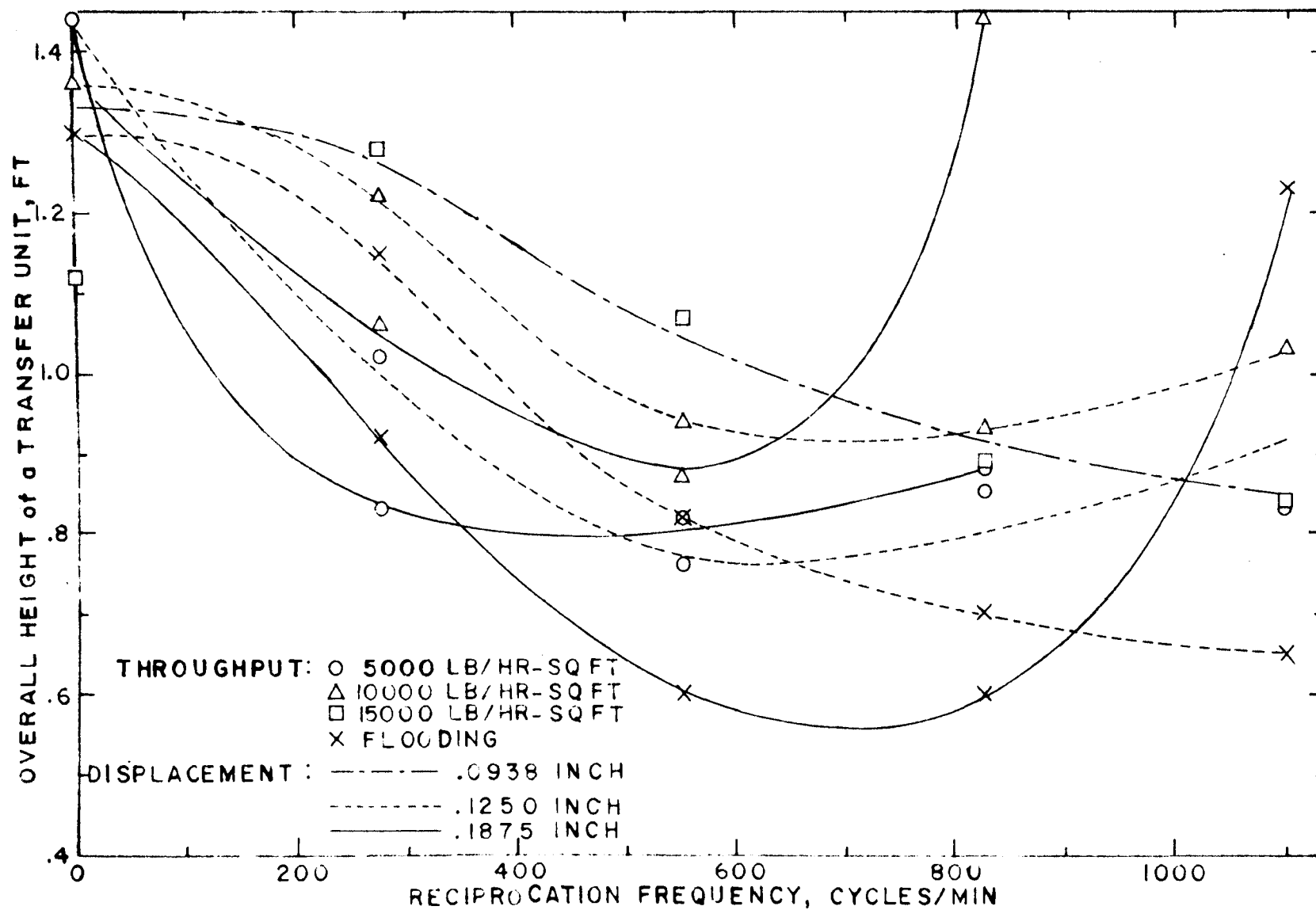


Figure 13. The Effect of Reciprocation Frequency on Performance with Transfer from the Water Phase to the Ketone Phase, Ketone Dispersed

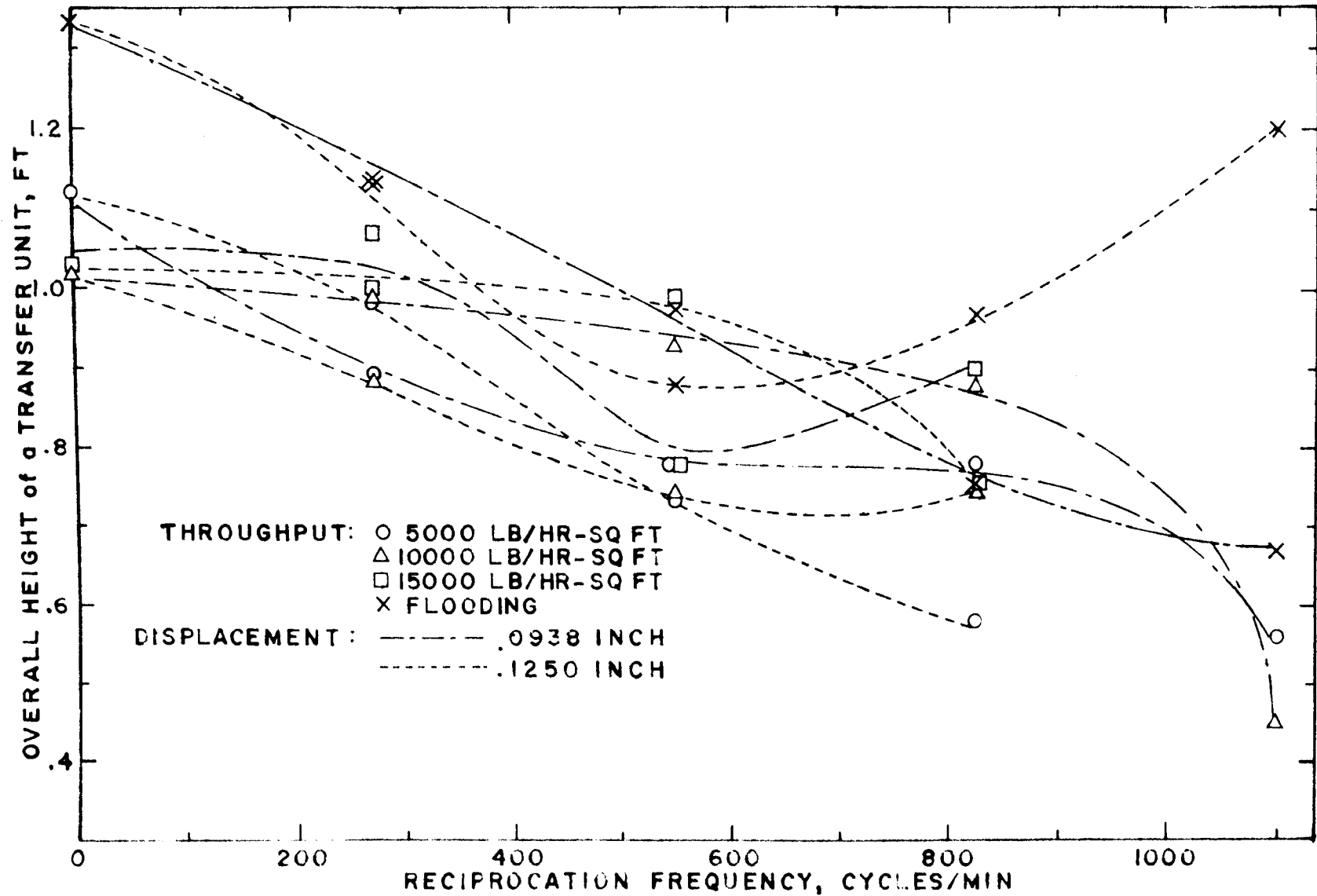


Figure 14 . The Effect of Reciprocation Frequency on Performance with Transfer from the Water Phase to the Ketone Phase, Water Dispersed

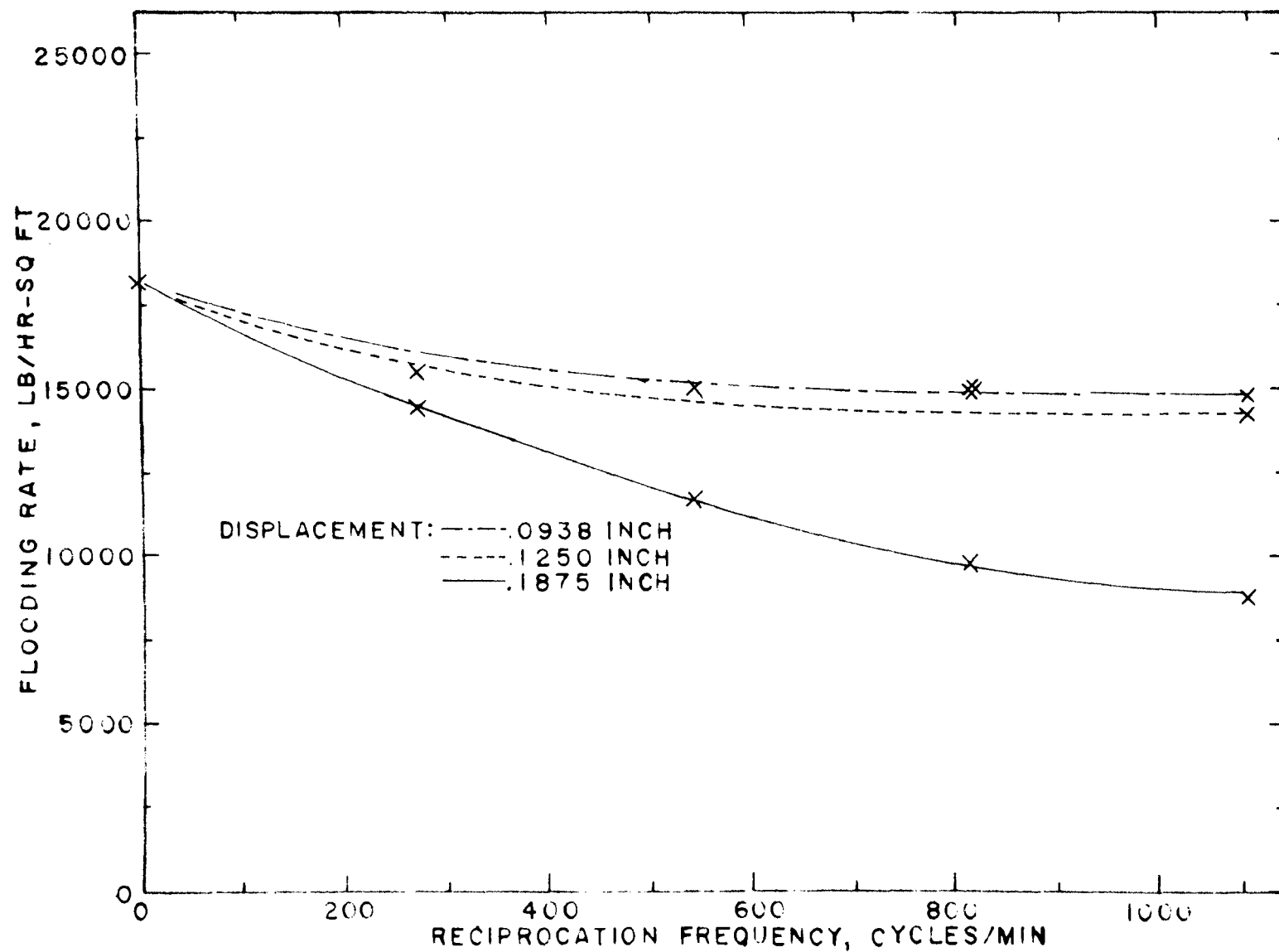


Figure 15. The Effect of Reciprocation Frequency on Flooding Rate with Transfer from the Ketone Phase to the Water Phase, Ketone Dispersed

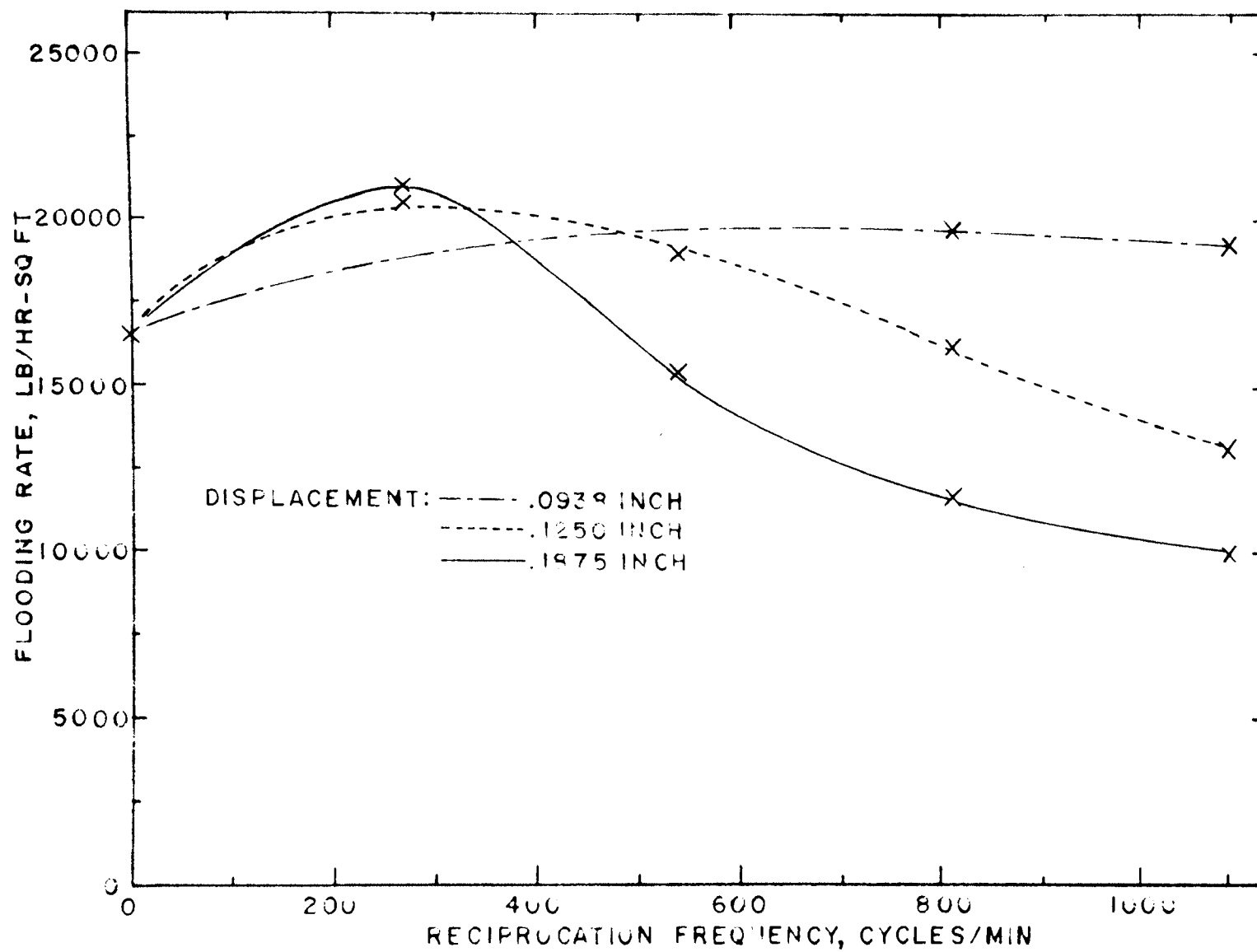


Figure 16. The Effect of Reciprocation Frequency on Flooding Rate with Transfer from the Ketone Phase to the Water Phase, Water Dispersed

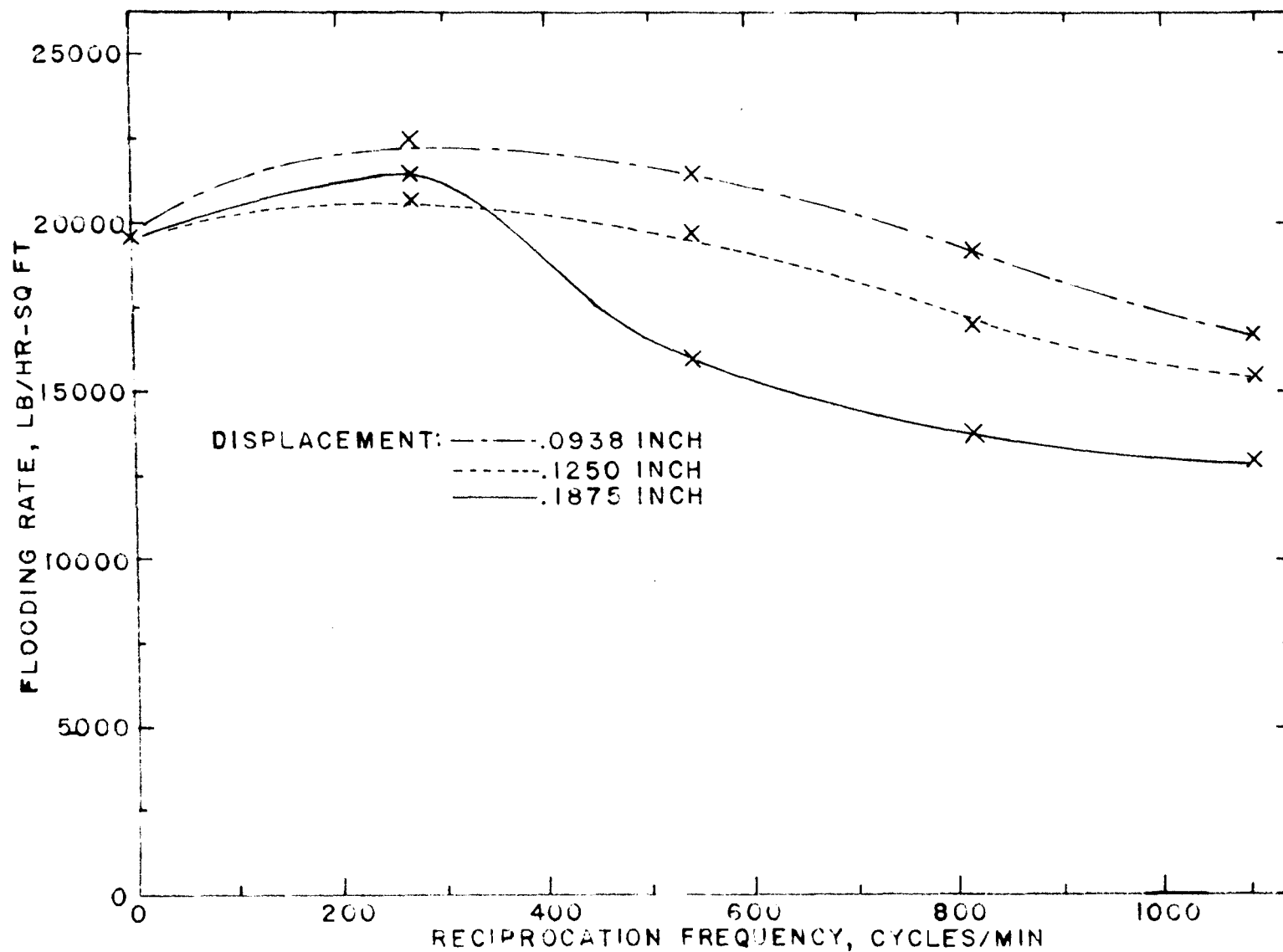


Figure 17. The Effect of Reciprocation Frequency on Flooding Rate with Transfer from the Water Phase to the Ketone Phase, Ketone Dispersed

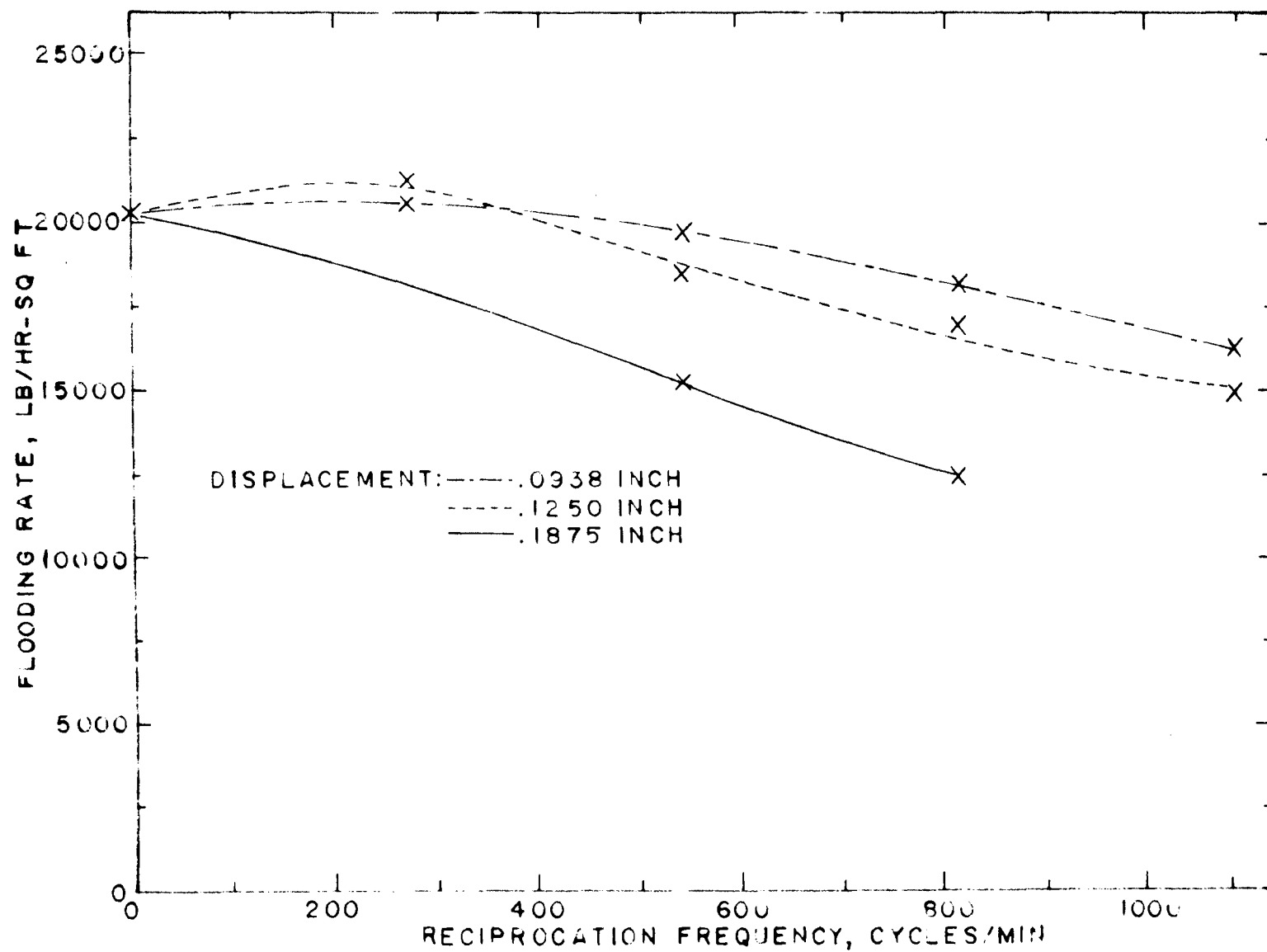


Figure 18. The Effect of Reciprocation Frequency on Flooding Rate with Transfer from the Water Phase to the Ketone Phase, Water Dispersed

IV. DISCUSSION

The discussion section of this thesis is comprised of an interpretation and a discussion of the experimental results. Recommendations for future experimental work, and the limitations of this work are also included. The discussion of the experimental results is presented in sections, treating the effect of each variable on performance as an independent topic. A comparison of the performance of various mechanically-aided extraction columns utilizing the methyl isobutyl ketone-acetic acid-water system is presented.

Effect of Combined Stream Throughput

The effect of combined inlet stream throughput on performance, segregated with respect to direction of transfer and dispersed phase, appears in Figures 7 through 10, pages 45 to 48 inclusive.

Transfer from Ketone to Water Phase, Ketone Dispersed.

The effect of throughput on the overall height of a transfer unit for transfer of acetic acid from the ketone to the water phase, with the ketone phase dispersed, is shown in Figure 7. In general, the effect of increasing throughput up to flooding had little effect on the overall height of a

transfer unit for all combinations of frequency and displacement chosen for power input to the system.

However, for tests with no power input to the system, the effect of increasing throughput was significant. For tests with no power input, the overall height of a transfer unit increased approximately 170 per cent between throughputs of 5,000 and 18,700 pounds per hour per square foot.

Definite trends of better performance with increased power input were exhibited. For a particular displacement, operation at higher frequencies yielded improved performance.

Transfer from Ketone to Water Phase, Water Dispersed.

Figure 8 shows the effect of throughput on performance, for transfer of acetic acid from the ketone phase to the water phase with the water phase dispersed. For most combinations of frequencies and displacements, the overall height of a transfer unit increased with increasing throughput. The selection of a larger displacement for a given frequency generally resulted in poorer performance. The best performance was achieved with selection of smaller displacements, 0.0938 and 0.1250 inches, frequencies between 275 and 825 cycles per minute, and occurred between throughputs of 5,000 and 10,000 pounds per hour per square foot.

The results indicated that performance was not enhanced with addition of more power to the system at flow rates

above 10,000 pounds per hour per square foot. However, performance was still considerably better for transfer from the ketone to water phase with the water dispersed as compared to performance with the ketone dispersed. Higher throughputs were achieved with the water dispersed, coupled with better overall performance over the entire range of throughputs investigated.

For transfer from the ketone to the water phase with the water dispersed, mass was transferred into the dispersed, or droplet phase. It has been generally observed for many continuous-dispersed systems, that mass transfer is generally better when transfer is into the drop or dispersed phase^(9,19).

Better performance with mass transfer into the dispersed phase can possibly be explained by considering the effects associated with transfer of mass out of the drop. It has been observed⁽¹¹⁾ that coalescence is more pronounced when mass is transferred out of the drop. More coalescence would result in the overall formation of very large drops. It is generally noted that mass transfer from relatively larger drops is slower than from smaller drops, resulting in poorer performance. This would be the case for transfer from the ketone to the water with the ketone dispersed, and one would conclude that performance would be better with

the water dispersed, as the experimental results indicated.

In conjunction with the effects of mass transfer out of the dispersed phase, consideration must also be given to the effect of drop size on the rate of mass transfer. With increased power input it would be expected that the droplet diameter would naturally be decreased. Increased power input to the system can be considered as being accomplished by increasing any one of the three variables affecting power, such as displacement, frequency, or throughput while holding the other two constant. In general an increase in displacement or frequency would result in a larger mechanical power increase in comparison to an increase in total throughput. This can be seen by the relative effects of increased displacement and frequency on performance as compared to the smaller effect of increased throughput on performance.

It was pointed out by Hughes and Gilliland⁽¹²⁾ that as the drop diameter is decreased, as would be the case with increased power addition, internal circulation within the drop is also decreased. With reduced internal circulation in the drop, molecular diffusion becomes the controlling factor for the rate of mass transfer with regard to very small drops. It follows that the performance of the extraction column would be significantly reduced for power inputs

which created drop sizes that would result in diffusion-controlled mass transfer. It was observed in many instances in the experimental work, that minimums in heights of a transfer unit were observed with increasing power input and further increases in power input, did not improve performance. It is suggested that such results support the premise of diffusion-controlled mass transfer for small drops, and would partially explain these minimums in the height of a transfer unit.

The physical property of interfacial tension would also play an important role in the size of droplet formation, and therefore effect the performance of the extraction column. The methyl isobutyl ketone-acetic acid-water system has been reported to be a ternary system of relatively low interfacial tension. Addition of solute to the system resulting in an increased acetic acid concentration would lower the interfacial tension of the system (29,30). The low interfacial tension, that is the low power requirement for creating additional surface area for mass transfer, results in a system where the liquid phases become easier to disperse with increasing acetic acid concentration. More effective dispersion with lowered interfacial tension by increased solute concentration would result in smaller drop size, which would in turn affect the performance.

Transfer from Water to Ketone Phase, Ketone Dispersed.

The effect of throughput on performance is shown in Figure 9, for transfer from the water to the ketone phase with the ketone phase dispersed. For all displacements and frequencies of 550 cycles per minute and above, the overall height of a transfer unit decreased with increased throughput. For a reciprocation frequency of 275 cycles per minute and most displacements, the overall height of a transfer unit increased slightly with increased throughput. With very few exceptions the performance with power input was better than without. Considering the total power input to be a combined function of frequency and throughput for a particular displacement, the performance was improved with increased power input to the system.

Transfer from Water to Ketone Phase, Water Dispersed.

The effect of throughput on performance with the water phase dispersed is shown in Figure 10, for transfer from the water to the ketone phase. The overall height of a transfer unit increased slightly with increasing throughputs up to flooding, for most combinations of frequency and displacement. Performance was better for operation with power addition as opposed to operation with no power addition to the system. The extraction column performance improved with increased power input. However, the performance generally decreased

somewhat for increasing throughput alone. For transfer from the water to the ketone, it may be noted that the performance for transfer out of the drop is not substantially poorer than into the drop, as was previously discussed.

Effect of Packing Reciprocation Frequency

The effect of packing reciprocation frequency, differentiated with respect to direction of transfer and dispersed phase, is shown in Figures 11 through 14, pages 50 to 53, inclusive.

Transfer from Ketone to Water Phase, Ketone Dispersed.

The effect of reciprocation frequency on the overall height of a transfer unit for acetic acid transfer from the ketone to the water phase is shown in Figure 11, with the ketone phase dispersed. At flow rates of 5,000 pounds per hour per square foot, the overall height of a transfer unit appears to pass through a maximum value with increasing reciprocation frequency for both the 0.1250 and 0.1875 inch displacements. In increasing the reciprocation frequency from 0 to 1100 cycles per minute, no net reduction in the overall height of a transfer unit was observed over values at zero frequency, or no power input. The possibility exists that the value of the height of a transfer unit at zero frequency is in error, but this was not confirmed by additional

experimental work.

For flow rates of 10,000 pounds per hour per square foot with 0.1875 inch displacement, the overall height of a transfer unit decreased slightly with increasing reciprocation frequency up to 550 cycles per minute. However, the performance was improved approximately 25 per cent with increasing frequency and smaller, 0.1250 inch, displacement for the same flow rate.

At flow rates greater than 10,000 pounds per hour per square foot and up to flooding, the performance improved significantly. It was generally concluded that the addition of more power to the system resulted in allowing higher throughputs with comparable performance to that at lower flow rates.

Transfer from Ketone to Water Phase, Water Dispersed.

The effect of reciprocation frequency on performance with solute transfer from the ketone phase to the water phase is shown in Figure 12, with the water phase dispersed. For all flow rates and displacements studied, the overall height of a transfer unit decreased to a minimum value and then increased with increasing reciprocation frequency. With the exception of the flooding data the effect of displacement and throughput were not apparent for flow rates of 5,000 and 10,000 pounds per hour per square foot, until frequencies

exceeding 550 cycles per minute were utilized. The results indicated that the choice of high frequencies coupled with smaller displacements resulted in best performance for throughputs up to 10,000 pounds per hour per square foot.

Generally the performance was improved with power input until reciprocation frequencies higher than 825 cycles per minute were used, indicating that further increase in power addition did not enhance average performance. The minimums observed in the overall height of a transfer unit with increasing reciprocation frequency can be explained by the creation of a drop size that would be diffusion-controlling with regard to mass transfer.

It was observed that performance was considerably better for transfer from the ketone phase to the water phase with the water phase dispersed, than with the ketone dispersed. This again indicated that performance was better with mass transfer into the dispersed phase, and could possibly be explained by the previous discussion of this topic.

Transfer from Water to Ketone Phase, Ketone Dispersed.

The effect of reciprocation frequency on performance with transfer from the water to the ketone phase is shown in Figure 13, with the ketone dispersed. For almost all flow rates and displacements studied, the overall height of a

transfer unit passed through a minimum value with increasing reciprocation frequency. The exceptions were flooding throughputs with 0.1250 inch displacement, and throughput of 15,000 pounds per hour per square foot with 0.0938 inch displacement. Generally minimums in heights of a transfer unit obtained with larger displacement occurred at lower frequency. Apparently with a combination of lower displacement for throughputs above 10,000 pounds per hour per square foot, insufficient power was added to the system to produce a diffusion-controlling size drop that would limit the rate of mass transfer. Therefore, it would be suspected that a further increase in reciprocation frequency would result in a minimum value of overall height of a transfer unit for flow rates above 10,000 pounds per hour per square foot with small displacements. The results indicated that best performance was at the highest throughputs, onset of flooding, with displacements of 0.1250 and 0.1875 inches. The results also indicated that after minimums in the height of a transfer unit had been attained, further increase in power addition did not improve performance.

Transfer from Water to Ketone Phase, Water Dispersed.

The effect of reciprocation frequency on performance with the water phase dispersed is shown in Figure 14, for transfer from the water to the ketone phase. In general the

overall height of a transfer unit decreased with increasing reciprocation frequency, and selection of larger displacements resulted in better performance for a given throughput.

Combinations of large throughputs, 15,000 pounds per hour per square foot and flooding, and small displacements, 0.0938 and 0.1250 inches, resulted in minimums in the height of a transfer unit with increasing reciprocation frequency. After these minimums had been reached, a further increase in power was futile in improving performance.

Performance was generally better for lower throughputs between 5,000 and 10,000 pounds per hour per square foot for both the 0.0938 and 0.1250 inch displacements. Power input to the system improved performance for the most part over performance with no power input by packing reciprocation.

Performance at the Onset of Flooding

Column performance was evaluated at the onset of flooding as a function of packing reciprocation frequency. The effect of reciprocation frequency on flooding rates, separated with respect to direction of transfer and dispersed phase is presented in Figures 15 through 18, pages 54 to 57 inclusive.

Transfer from Ketone to Water Phase, Ketone Dispersed.

The effect of reciprocation frequency on flooding rate for transfer of acetic acid from the ketone to the water phase is shown in Figure 15, with the ketone dispersed. The flooding rate decreased from a maximum of approximately 18,500 pounds per hour per square foot, at no power input, with increasing reciprocation frequency. With larger packing displacement the flooding rate decreased more rapidly with increasing reciprocation frequency. The flooding rate was lower, for a particular frequency with larger displacement and also decreased with increasing reciprocation frequency for a particular displacement, indicating the effect of increased power input decreasing total available throughputs.

Transfer from Ketone to Water Phase, Water Dispersed.

The effect of reciprocation frequency on flooding rate with transfer from the ketone phase to the water phase is shown in Figure 16, with the water phase dispersed. For increased reciprocation frequencies the flooding rates decreased to lower values with the larger two displacements studied, however, passing through a maximum value of approximately 22,000 pounds per hour per square foot at 275 cycles per minute. The flooding rate increased slightly with increasing frequency for the smallest displacement or lowest power

input studied. It could be generally concluded again that larger power input reduced available maximum throughput in the column. Flooding rates were somewhat higher with the water dispersed for the same direction of acetic acid transfer.

The higher flooding rates with the water dispersed could be explained by considering that larger drops would be entrained in the continuous phase more readily than smaller drops of dispersed phase. This would be the case for transfer of acid from the ketone to the water phase with the ketone phase dispersed, if the observation of increased coalescence with transfer out of the drop applies to performance at the onset of flooding.

Transfer from Water to Ketone Phase, Ketone Dispersed.

The effect of reciprocation frequency is shown in Figure 17 for transfer from the water phase to the ketone phase, with the ketone phase dispersed. The flooding rate increased from 20,000 pounds per hour per square foot, at no power input, through a maximum value at 275 cycles per minute and then decreased with further increase in reciprocation frequency to lower values than obtained at no power input. Larger displacements resulted in lower flooding rates for a particular packing reciprocation frequency. These results

again substantiate the effect of lower available column throughputs with increased power input.

Transfer from Water to Ketone Phase, Water Dispersed.

The effect of reciprocation frequency on flooding rates for transfer from the water phase to the ketone phase, with the water phase dispersed, is presented in Figure 18. Flooding rates decreased from approximately 20,000 pounds per hour per square foot at no power input to lower values with increased power input. It was generally observed during the study of flooding rates that larger power inputs, larger displacement for a particular frequency or increased frequency for a particular displacement, resulted in lower flooding rates as the experimental data again indicated.

Reproducibility of Experimental Data

In reproducing several experimental tests, a measure of the reproducibility of experimental data was obtained. During the course of the experimental investigation several tests were duplicated for the purpose of checking calculated results. It was found that calculated heights of a transfer unit were reproducible within the limits of ± 0.05 feet, for identical test conditions and operation below the onset of flooding.

Flooding rates were found to decrease as much as 2,000 pounds per hour per square foot maximum, for identical test conditions at a later time in the investigation. The reproduction of tests at the onset of flooding was conducted near the end of the experimental investigation. The decrease in flooding rates could possibly be attributed to a buildup of trace contaminants in the ternary liquid extraction system. The only source of known contamination of the system was the Molykote lubricant used on the process valve stems. It is known that addition of a solute or contaminant has a lowering effect on the interfacial tension of the system, which would in turn effect the flooding rates. However, no physical measurements were made to confirm this fact. At the present time little is positively known about the effect of interfacial tension and the magnitude of this effect. It is generally thought that interfacial tension plays an important role in resistance to mass transfer. Therefore, it would be suspected that a slight change in this physical property of the system might have a measurable effect on the performance of the system.

Comparison of Extraction Columns

A comparison of the performance of the extraction column described in this work with the performance of mechanically-aided extraction columns reported in the literature is presented in Table III, page 74. The performance comparison was made considering the maximum permissible column throughput before flooding, and the corresponding height equivalent to a theoretical stage as a relative basis for performance comparison. Consideration was also given to the best attainable value of height equivalent to a theoretical stage and the corresponding column throughput as a measure of relative performance. As some of the designs of mechanically-aided columns were continuous-stagewise equipment rather than differential contactors, the height equivalent to a theoretical stage was selected as a common basis for performance comparison. Heights equivalent to a theoretical stage corresponding to overall heights of a transfer unit were calculated with equation 5, page 19.

Maximum Total Throughput. A maximum throughput of 3,100 gallons per hour per square foot was attained with the reciprocating wire-mesh packed column used in this work. Excluding this work, maximum throughputs achieved before flooding ranged from 149 gallons per hour per square foot

TABLE III
Comparison of This Work with Reported Performance
of Mechanically-Aided Extraction Columns Utilizing
the Acetic Acid-Methyl Isobutyl Ketone-Water System

Type of Column	Column Diameter	Dispersed Phase	Performance at the Onset of Flooding		Performance at the Lowest H.E.T.S.		Reference
			Combined-Stream Throughput	H.E.T.S.	Combined-Stream Throughput	H.E.T.S.	
	in.		gal/hr-ft ²	in.	gal/hr-ft ²	in.	
Rotating Disc	8.0	Water	1,030	6.3	980	4.3	(15)
Turbine Agitator-Horizontal Baffles	11.5	Water	458	3.0	458	3.0	(16)
Alternate Agitated and Packed Sections	11.5	Water	595	9.2	595	9.2	(17)
Turbine Agitators in Baffled Compartments	6.0	Ketone	500	5.5	286	3.7	(14)
Pulsed-Packed	1.57	Ketone	149	5.1	149	5.1	(7)
Pulsed Sieve-Tray	1.57	Ketone	267	10.1	267	10.1	(7)
Sieve Plate Controlled-Cycling	2.0	Water	2,270	14.0	1,250	6.5	(20)
Reciprocating Plate	3.0	Water	1,837	7.5	547	4.3	(13)
Pulsed Spray	1.5	Ketone	920	3.1	920	3.1	(1)
Reciprocating Wire-Mesh Packing	3.0	Water	2,800	11.8	1,300	4.2	(This Work)
	3.0	Ketone	3,100	18.8	2,200	9.8	(This Work)

for the pulsed-packed column to 2,270 gallons per hour per square foot with the controlled-cycling extraction column. The total throughput of 3,100 gallons per hour per square foot achieved was 37 per cent higher than previous maximum reported throughputs for mechanically-aided extraction columns.

It may be observed that considerably higher throughputs were achieved with the extraction column utilized in this work than for other mechanically-aided columns. Greater throughputs can be attributed to the unique design for adding power by reciprocating a packing of high void content. A packing such as the 95 per cent void wire-mesh used would offer a relatively small resistance to fluid flow in comparison to columns packed with Raschig rings or columns filled with various internals which occupy a large portion of the column cross-sectional area.

Also, it can be seen that in reciprocating a packing vertically, no fluid energy would be expended radially as compared to turbine agitated or rotating disc columns. With an equal positive and negative directional displacement of the packing in the same plane as the fluid flow, it follows that there would be no net reduction in fluid energy as a result of packing movement such as changing the direction of flow by a rotating member in a column.

Relative Performance. The minimum height equivalent to a theoretical stage exhibited with the wire-mesh packed column was 4.2 inches at a total throughput of 1,300 gallons per hour per square foot. In observing Table III it can be seen that this performance is competitively equivalent with the best performances of other columns, and has the added benefit of considerably higher permissible throughputs.

Recommendation for Equipment Modification

The following recommendations are concerned with direct modifications of the extraction column proper, and of the column accessories.

Process Piping. It is recommended that all nylon fittings in the process piping be replaced with stainless steel equivalents to facilitate greater versatility in liquid extraction systems which could be studied.

Process Valves. Replacement of existing valves with valves with the packing below the valve stem threads would eliminate contamination of the system by any lubricants and would prevent galling experienced when lubricant was insufficient.

Water Supply. For the purpose of expediting experimental tests it is recommended that the distilled water unit

be replaced with a mixed-bed ion exchange column with a higher capacity for providing a pure water source.

Sampling. It is recommended that the existing column be modified or replaced with a column with provision for sampling of both liquid phases along the height of the column. This modification would allow studies of the effects of backmixing on extraction column performance.

Recommendations for Future Investigation

The following recommendations apply to the extension of this experimental investigation.

Extraction System. Evaluation of the extraction column performance utilizing other ternary liquid systems with different physical characteristics such as equilibrium distribution and interfacial tension would broaden the scope of possible applications for the wire-mesh packed column, and would provide informative data for further comparison with other equipment.

Effect of Backmixing. With provision for phase sampling along the column height it would be an interesting study to determine the effect of backmixing on performance as a function of power addition to the system.

Effect of Mass Transfer on Flooding Rate. On the basis of limited observation, the author suspects that in the absence of mass transfer the flooding rates decrease. It would prove informative to further investigate this phenomenon by experimental investigation.

Power Addition. While still employing the method of vertical reciprocation, it would be interesting as well as informative to operate the extraction column with different internals other than the wire-mesh packing. Possibly the packing could be arranged in alternate sections with sieve-plates or other means for producing turbulence to effect improved performance.

Photographic Study. With still photographs, the drop-let size could be determined for various magnitudes of power addition allowing determination of interfacial area existing for mass transfer. With motion photography the effects of coalescence and redispersion of the dispersed phase could be determined at least qualitatively. The associated effect of wetting or not wetting the packing with the dispersed phase might also be observed.

Limitations

The experimental investigation was conducted with the wire-mesh packed extraction column utilizing only the methyl isobutyl ketone-acetic acid-water system. Provision was made for dispersing either the ketone or the water phase. Acetic acid was transferred both from the ketone phase to the water phase and from the water phase to the ketone phase. The column performance was evaluated as a function of power input to the system and total throughput. The detailed experimental limitations are described in the following section.

Acetic Acid Concentrations. A maximum acetic acid concentration of approximately 3.0 weight per cent in the water phase feed was used for transfer of acetic acid from the water to ketone phase. The corresponding maximum acetic acid concentration in the ketone phase feed was approximately 0.5 weight per cent. For transfer of acetic acid from the ketone phase to the water phase, maximum acetic acid concentrations in the ketone and water feeds were approximately 1.5 and 0.0 weight per cent respectively.

Extraction Column Dimensions. The extraction column used was 3.0 inches in inside diameter, 3.0 feet in length, and was fabricated of Pyrex glass pipe.

Wire-Mesh Packing. The wire-mesh packing utilized had a void content of 95.2 per cent and was 23.0 inches in height.

Packing Displacement. Three packing displacements were used during the experimental investigation. Circular eccentric cams providing displacements of 0.0938, 0.1250, and 0.1875 inches were employed.

Reciprocation Frequency. Packing reciprocation frequencies from 0 to 1100 cycles per minute were studied, encompassing the full range of the mechanical transmission drive.

Combined Stream Throughput. The extraction column performance was evaluated at flow rates of 5,000, 10,000, and 15,000 pounds per hour per square foot and at throughputs at the onset of flooding. The maximum permissible throughputs were obtained at flooding, and were 21,000 and 22,600 pounds per hour per square foot for transfer from the ketone to the water phase and water to the ketone phase, respectively.

Solvent Ratio. All experimental tests were conducted with equal volumetric feed rates. The effect of solvent ratio on performance was not studied due to time limitations.

Temperature Control. The temperature range of 28 ± 1 degrees Centigrade was selected for liquid feeds and was maintained for all experimental tests.

V. CONCLUSIONS

The conclusions of this experimental work are presented in the following section and pertain to: (1) the overall performance of the extraction column with respect to the direction of acetic acid transfer, dispersed phase, and operating parameters, (2) the performance of the experimental extraction column at the onset of flooding, and (3) the comparative performance of the reciprocating wire-mesh packed column used in this work with other types of mechanically-aided extraction columns reported in the literature utilizing the methyl isobutyl ketone-acetic acid-water system.

1. In evaluating the extraction column performance, for transfer of acetic acid from the water to the ketone phase and from the ketone to the water phase, the effect of dispersing either the ketone phase or water phase was studied. Mechanical power was added to the system by reciprocating a wire-mesh packing of 95 per cent void volume. Packing displacements of 0.0938, 0.1250, and 0.1875 inches were utilized with variable frequency between 0 and 1100 cycles per minute. The selected reciprocation frequencies were 0, 275, 550, 825, and 1100 cycles per minute. Various combinations of packing displacements and reciprocation frequencies were

selected for power addition to the extraction system at throughputs in increments of 5,000 pounds per hour per square foot up to the onset of flooding. The overall performance of the extraction column was evaluated in terms of the overall height of a transfer unit based on the ketone phase. The conclusions pertaining to performance at normal operating conditions are:

a. For solute transfer from the ketone to the water phase, with the ketone phase dispersed, best performance was observed at a reciprocation frequency of 1100 cycles per minute, 0.1875 inch displacement, and a flow rate of 5,000 pounds per hour per square foot. The corresponding minimum overall height of a transfer unit was 0.55 feet. Generally, the overall height of a transfer unit decreased slightly with increasing reciprocation frequency for a particular displacement and throughput. Increasing throughput had little effect on performance for most combinations of frequency and displacement selected for power addition.

The minimum height of a transfer unit obtained with the water phase dispersed was exhibited at a frequency of 825 cycles per minute, 0.0938 inch displacement, and a throughput of 10,000 pounds per hour per square foot.

The minimum overall height of a transfer unit observed was 0.26 feet. Minimums in the overall height of a transfer unit were experienced with increasing reciprocation frequency. The overall height of a transfer unit passed through minimum values with increasing reciprocation frequency for the 0.0938 and 0.1250 inch displacements, at frequencies between 275 and 825 cycles per minute and for throughputs between 5,000 and 10,000 pounds per hour per square foot. Performance was not improved with increased power addition at flows above 10,000 pounds per hour per square foot.

b. For solute transfer from the water to the ketone, with the ketone phase dispersed, a minimum overall height of a transfer unit of 0.60 feet was realized. Corresponding parameters of operation were 0.0938 inch displacement, 825 cycles per minute, and a throughput of 19,300 pounds per hour per square foot. The overall height of a transfer unit passed through minimum values between frequencies of 275 and 825 cycles per minute for most displacements and throughputs, and further increase in frequency resulted in poorer performance.

For transfer with the water dispersed, a minimum overall height of a transfer unit of 0.45 feet occurred at a throughput of 10,000 pounds per hour per square

foot, 0.0938 inch displacement, and a frequency of 1100 cycles per minute. Generally the performance increased with increasing power addition to the extraction system.

2. Performance of the extraction column was investigated with maximum allowable throughputs at the onset of flooding for both directions of solute transfer. Performance at the onset of flooding was evaluated as a function of the operating parameters of frequency, displacement, and dispersed phase.

a. For transfer of acetic acid from the ketone to the water phase the flooding rate decreased with increased power addition, however, passing through a slight maximum at 275 cycles per minute with the water phase dispersed as compared to a continued decrease with the ketone dispersed. A maximum flooding rate of 18,200 pounds per hour per square foot was observed with the ketone dispersed, and 21,000 pounds per hour per square foot with the water phase dispersed.

b. Lower flooding rates were generally observed with increased power input to the system for transfer of acetic acid from the water phase to the ketone phase with increasing reciprocation frequency. However, slight maximums occurred at 275 cycles per minute with

the ketone phase dispersed. Maximum allowable throughputs of 22,600 and 21,200 pounds per hour per square foot were attained with the ketone and water phase dispersed respectively.

3. In comparing the various types of mechanically-aided extraction columns, consideration was given to the relative maximum allowable throughputs and the corresponding heights equivalent to a theoretical stage as a measure of performance. Also, emphasis was placed on the best attainable values of height equivalent to a theoretical stage for the different columns and the throughputs at which these values were achieved. The maximum throughput of 3,100 gallons per hour per square foot achieved with the wire-mesh packed column utilized in this work was 37 per cent higher than any other reported throughputs for mechanically-aided columns. The minimum height equivalent to a theoretical stage exhibited with the wire-mesh packed column was 4.2 inches at a total throughput of 1,300 gallons per hour per square foot. This performance was considered to be competitively equivalent to other extraction column performances with the corresponding throughput being considerably higher than most other types of columns at the same level of performance.

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VII. VITA

The author of this thesis, Thomas Vardaman Konkle, was born at Memphis, Tennessee on July 5, 1939. The author received his primary and secondary education in Memphis, Tennessee, and Benton, Kentucky, respectively. The author enrolled in Missouri School of Mines and Metallurgy in September 1957, and received the degree of Bachelor of Science in Chemical Engineering in July 1962. During the course of work toward a Bachelor of Science in Chemical Engineering, the author participated in a cooperative educational program with Union Carbide Nuclear Company, Paducah, Kentucky.

The author was married to Miss Patricia Jane Stites in April 1961. After graduation he was employed with General Aniline and Film Corporation in Calvert City, Kentucky. In September 1963 the author took an educational leave of absence from General Aniline and Film Corporation and returned to Missouri School of Mines and Metallurgy for the degree of Master of Science in Chemical Engineering. He was employed as a Graduate Teaching Assistant for the 1963-1964 term at Missouri School of Mines and Metallurgy while fulfilling the requirements for a Master's Degree in Chemical Engineering.

The author intends to return to industrial employment until such time when means become available for academic pursuit of the degree of Doctor of Philosophy in Chemical Engineering.

VIII. APPENDICES

The appendices of this thesis include: (1) Appendix A, materials and apparatus; (2) Appendix B, drawings and bill of materials; and (3) Appendix C, calibrations, experimental data, and computer program.

APPENDIX A

Materials used in the experimental investigation are listed in this appendix.

Methyl Isobutyl Ketone. (4-methyl-2-pentanone)

Distillation range, 114-117 °C; purity, 99% min.; acidity (CH₃COOH), 0.01%. Manufactured by Shell Chemical Company. Used as solvent in ternary liquid extraction system.

Acetic Acid. Glacial; Dichromate test, meets A.C.S. specifications; dilution test, to pass test; residue after evaporation, 0.0010%; chloride (Cl), 0.001%; sulphate (SO₄), 0.0001%; heavy metals (as Pb), 0.00005%; iron (Fe), 0.00002%; substances reducing KMnO₄, to pass 2 hour test; copper (Cu), 0.00001%; nickel (Ni), 0.00001%; substances reducing K₂Cr₂O₇, to pass 1/2 hour test; assay (CH₃COOH), minimum 99.7%; Lot No.'s D704271, D707111, D808131. Obtained from Allied Chemical, General Chemical Div., New York, N.Y. Used as the solute in the experimental liquid extraction system.

Distilled Water. Steam distilled with commercial water still from municipal city water, Rolla, Mo. Used as solvent in the liquid extraction system.

Potassium Biphtalate. Assay (KHC₈H₄O₄), 99.99%; insoluble matter, 0.002%; loss on drying at 105°C, 0.010%;

pH of 0.05 M solution at 25°C, 4.0; chlorine compounds (Cl), 0.003%; sulfur compounds (S), 0.001%; heavy metals (Pb), 0.0002%; Iron (Fe), 0.002%; Lot No. 91279, meets A.C.S. specifications. Obtained from J. T. Baker Chemical Company, Phillipsburg, N. J. Used as primary acidimetric standard.

Hydrochloric Acid. Reagent A.C.S. Meets A.C.S. specifications. Obtained from Allied Chemical, General Chemical Div., New York, N.Y. Used as secondary acidimetric standard.

Sodium Hydroxide. Pellets. Analytical reagent. Meets A.C.S. specifications. Obtained from Mallinckrodt Chemical Works, St. Louis, Mo. Used as standard base in acetic acid titrations.

The apparatus used in the experimental liquid extraction investigation is listed as follows:

Variable Speed Drive and Motor. Graham Model N29M, serial number 1h161F36 W, 0-1100 rpm speed range, output torque of 10-16 inch-pounds at max. and 1/10 max. speed respectively, horizontal output shaft, micrometer speed control. Input driving motor, 1/4 HP at 3450 rpm, single phase, 60 cycle, 115/230 v. Used for rotation of reciprocation cam.

Centrifugal Pump. Eastern Model D-11 centrifugal pump, single stage, type 100 SB/T, serial number C3K-1064, 316 stainless steel, 115 v, 60 cycle, single stuffing box with shredded Teflon packing. Used for water feed pump.

Centrifugal Pump. Eastern Model D-11 centrifugal pump, single stage, type 100/T, serial number C4A-1033, 316 stainless steel, 115v, 60 cycle, mechanical seal. Used for ketone feed pump.

Water Still. Barnstead water still, mfg's cat. no. SMO-58, serial number 49755, 5 gal/hr. capacity, manufactured by Barnstead Still and Sterilizer Co., Boston, Mass. Used for supplying distilled water for use as solvent.

Balance, Equal Arm. Eleven pound capacity, style 347. Serial number 48539, 0.01 lb. graduations, 0.005 lb. sensitivity over entire scale. Manufactured by the Exact Weight Scale Co., Columbus, Ohio. Used to weigh samples for rotameter calibrations and to weigh acetic acid for addition to the water phase.

Timer. One-tenth second graduation, push button start and stop, 9999.9 sec. capacity, 115v, 60 cycle, 5 watts. Manufactured by Precision Scientific Co., Chicago, Ill. Used to determine time elapsed for rotameter calibration and time of each extraction test.

Buret. 50 m. capacity, 0.1 m. subdivisions, \pm 0.05 ml tolerance, meets Federal Specifications DD-V-581 a. Obtained from Kimble Laboratory Div. of Owens-Illinois, Toledo 1, Ohio. Used for standardization and acetic acid titrations.

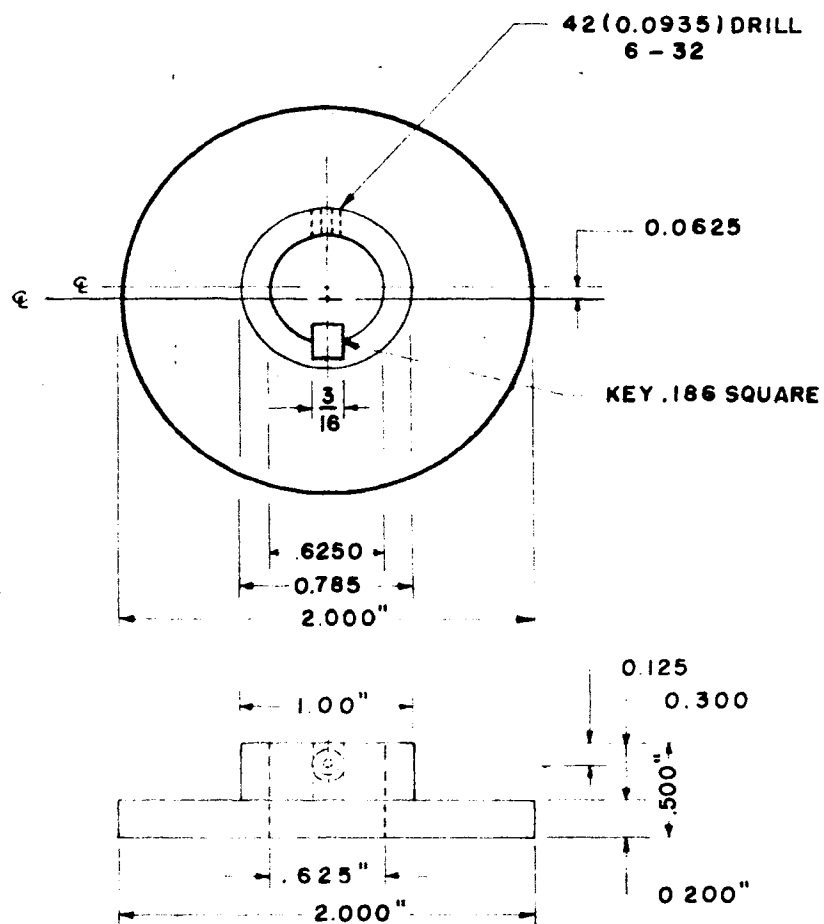
Rotameters. Type 316 stainless steel fittings, tube size 8-25-1, 250 mm. tube, serial number 1761, Type 1. Manufactured by Brooks Rotameter Co., Lansdale, Pa. Used to measure flow rate of water and ketone column inlet flow rates.

Constant Temperature Bath, Controller. Manufactured by Precision Scientific Co., Chicago, Ill. Cat. no. 66600, serial number M-3, 115v, 50/60 cycles, single phase, 450

watt immersion heater. Circulation pump model 13-061-1,
serial number 12422287M. Used for temperature control of
feed.

APPENDIX B

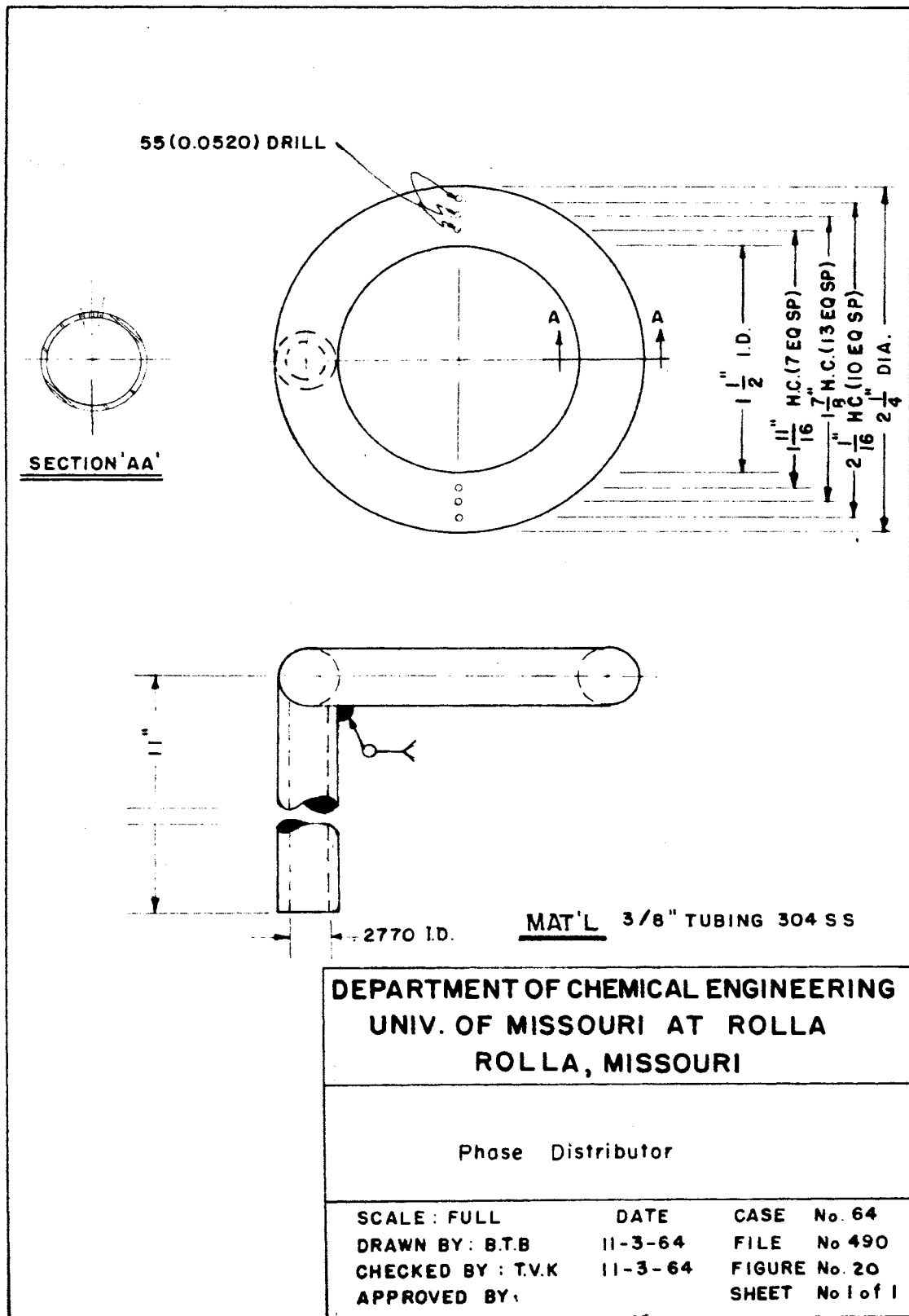
Drawings and materials used for modifications of the original extraction column and accessories are listed in this appendix with suppliers and units of the items used.



DEPARTMENT OF CHEMICAL ENGINEERING
UNIV. OF MISSOURI AT ROLLA
ROLLA, MISSOURI

1/8 Inch Reciprocation Cam

SCALE: FULL	DATE	CASE No. 64
DRAWN BY: B.T.B	11-3-64	FILE No. 490
CHECKED BY: T.V.K	11-3-64	FIGURE No. 19
APPROVED BY:		SHEET No. 1 of 1



List of Materials of Construction

<u>Description</u>	<u>Supplier</u>	<u>Unit</u>	<u>No. Units</u>
<u>Piping and Valves</u>			
Valve, 1/4 in. gate, 18-8 alloy stainless steel, Jenkins valve.	Donnley Pipe and Supply Co., St. Louis, Mo.	ea	2
Valve, 3/8 inch, needle, 316 stain- less steel, cat. no. Py272, Hoke valve.	Donnley Pipe and Supply Co., St. Louis, Mo.	ea	10
Tubing, Agaloy 316 stainless steel, 3/8 inch I.D., 0.035 in. wall thickness	Metal Goods Corp., St. Louis, Mo.	ft	200
Connector, 3/8 in. tube to 3/8 in. male P.T., Imperial cat. no. 268-N, nylon.	Clarkson Power Flow Engineering Co., Kansas City, Mo.		20
Coupling, female, 3/8 in. tube to 1/4 in. female P.T., Imperial cat. no. 266-N, nylon	Clarkson Power Flow Engineering Co., Kansas City, Mo.		3
Elbow, 3/8 in. tube to 1/4 in. male P.T., Imperial cat. no. 269-N, nylon	Clarkson Power Flow Engineering Co., Kansas City, Mo.		10
Sleeve, No. 840-FSS, stainless steel	Clarkson Power Flow Engineering Co., Kansas City, Mo.		75
Nut, No. 841-FS, steel	Clarkson Power Flow Engineering Co., Kansas City, Mo.		75

<u>Description</u>	<u>Supplier</u>	<u>Unit</u>	<u>No. Units</u>
<u>Piping and Valves</u>			
Coupling, pipe red. coupling, 1/2 in. to 1/4 in., NPT, 316 stainless steel	Geigher Pipe Supply Co. St. Louis, Mo.	ea	15
Cap, pipe cap, NPT, 1/2 in., 316 stainless steel	Geigher Pipe Supply Co. St. Louis, Mo.	ea	10
Bushing, NPT, 1/2 in. to 1/4 in., 316 stainless steel	Geigher Pipe Supply Co., St. Louis, Mo.	ea	10
<u>Storage Tanks</u>			
Drums, 55 gallon, 316 stainless steel, U.S. Pat. No. 2576767, I.C.C. Spec. 5-C, United States Steel	Nooter Corp. St. Louis, Mo.	ea	5

APPENDIX C

The calibrations, experimental data taken during the investigation, and computer program utilized for calculations are included in this appendix.

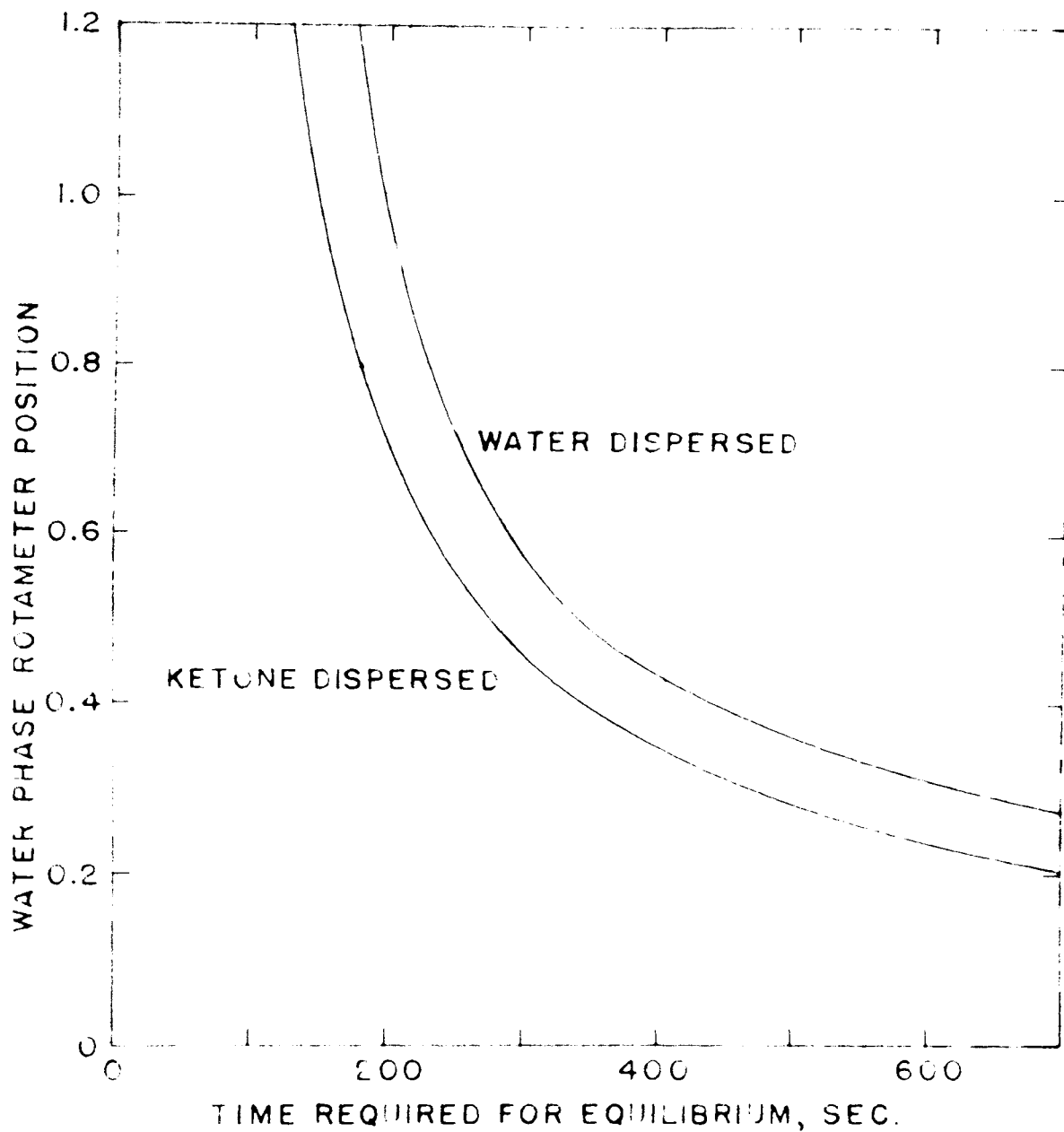


Figure 21. Extraction column Volume Exchanges Required for Equilibrium as a Function of Water Rotameter Position

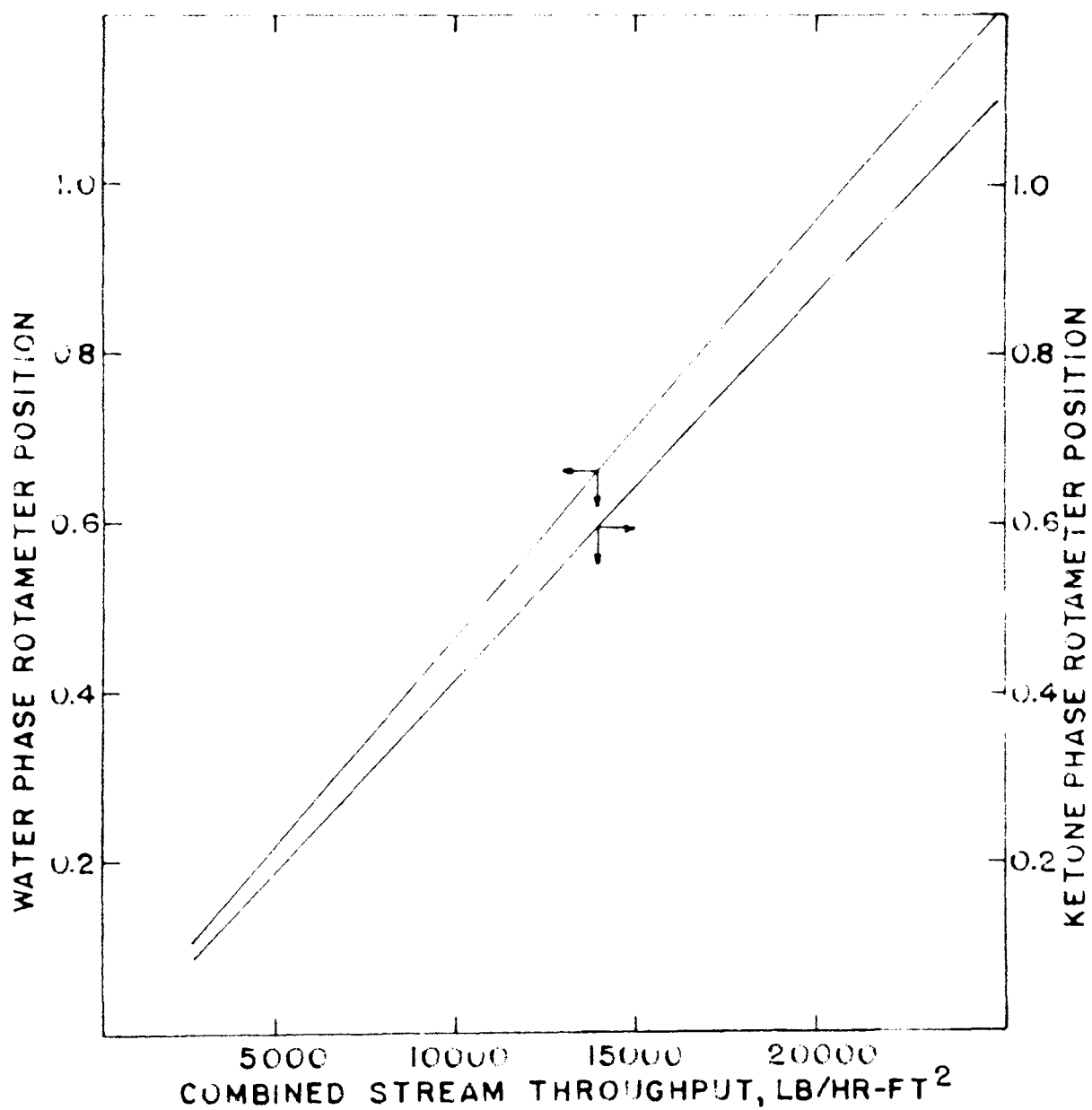


Figure 22. Combined Inlet Stream Throughput as a Function of Corresponding Rotameter Positions

The data, calibration curves, and methods of calibration of the inlet stream rotameters and variable speed transmission are included in this section.

Calibration of Rotameters. The calibration of the inlet stream rotameters was accomplished by weighing the quantity of liquid passing through the rotameters for a known time interval. Mutually saturated solutions of the ketone and water phases containing 0.5 weight per cent and 1.5 weight per cent acetic acid respectively, were used for the calibrations. The liquid temperatures were maintained at the normal operating temperature of 28 degrees centigrade during the calibrations. Both rotameters were calibrated in 0.1 scale intervals over the entire range of the rotameters. The liquids were weighed on an equal arm balance to the nearest 0.01 of a pound, and the time was measured with an electric timer, accurate to 0.1 seconds. The calibration data is presented in Tables IV and V, and the calibration curves are presented in Figures 23 and 24.

TABLE IV

Calibration of the Ketone Phase Rotameter with Methyl
Isobutyl Ketone Saturated with Water at 28 Degrees
Centigrade and 0.5 Weight Per Cent Acetic Acid

Scale Position	Weight of Ketone Phase Discharged	Time Interval	Flow Rate
	lb	min	lb/min
0.1	1.72	1.710	1.01
0.2	3.08	1.700	1.81
0.3	3.19	1.210	2.64
0.4	3.88	1.133	3.42
0.5	3.39	.800	4.24
0.6	4.22	.842	5.01
0.7	4.73	.805	5.88
0.8	4.81	.730	6.59
0.9	5.71	.797	7.16
1.0	5.48	.688	7.97
1.1	4.61	.638	8.79
1.2	6.71	.682	9.84

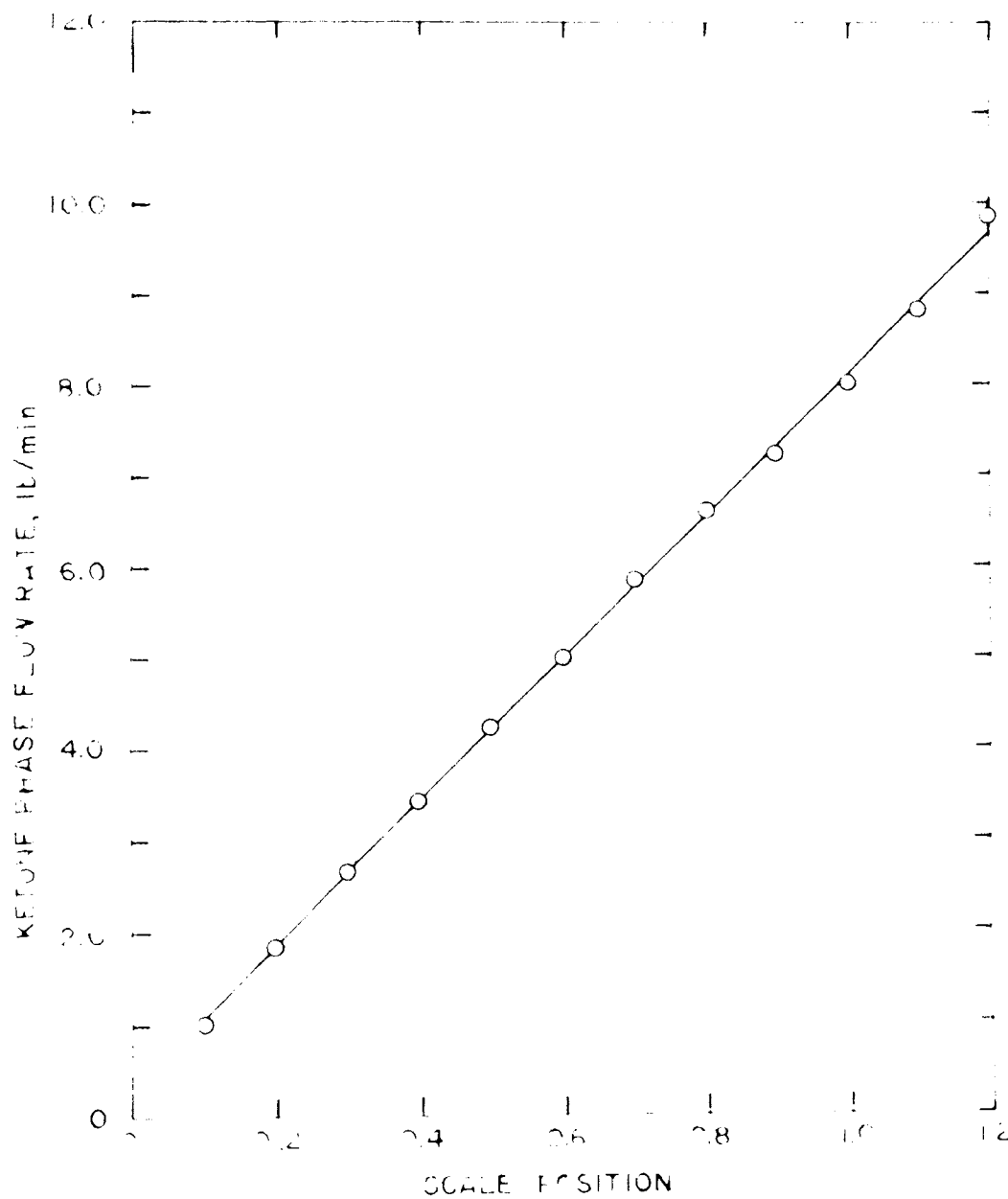


Figure 23 . Ketone Phase Rotameter Calibration Plot

TABLE V

Calibration of the Water Phase Rotameter with Water
Saturated with Methyl Isobutyl Ketone at 28 Degrees
Centigrade and 1.5 Weight Per Cent Acetic Acid

Scale Position	Weight of Water Phase Discharged	Time Interval	Flow Rate
	lb	min	lb/min
0.1	7.97	6.678	1.19
0.2	7.21	3.647	1.98
0.3	7.53	2.637	2.86
0.4	7.45	1.973	3.78
0.5	7.71	1.650	4.67
0.6	7.70	1.380	5.58
0.7	7.65	1.183	6.47
0.8	7.55	1.022	7.39
0.9	7.78	.947	8.22
1.0	7.62	.833	9.15
1.1	7.47	.748	9.99
1.2	7.45	.672	11.09

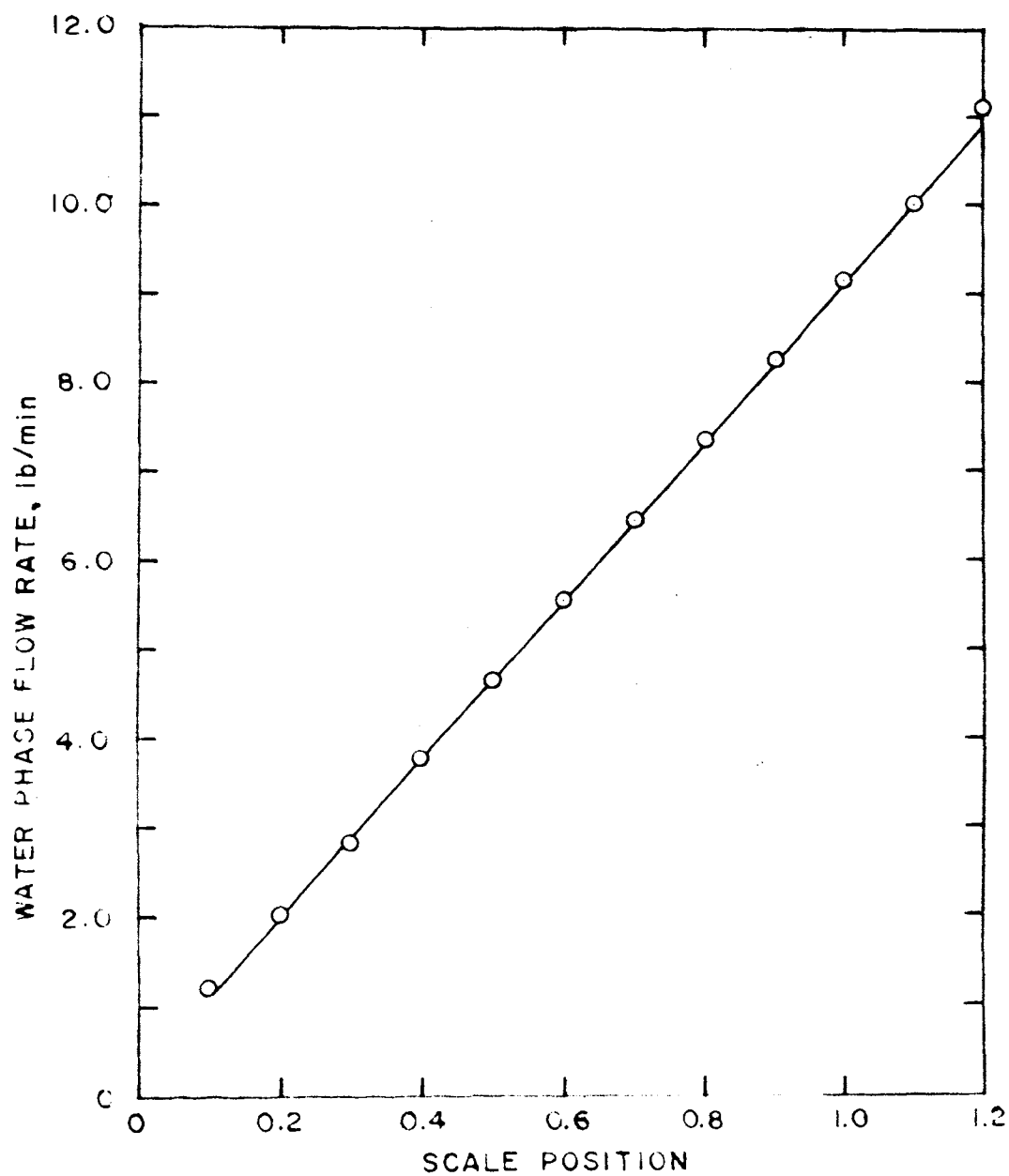


Figure 24. Water Phase Rotameter Calibration Plot

Calibration of the Variable Speed Transmission. The variable speed transmission was calibrated under simulated operating conditions by setting the micrometer speed control dial position and measuring revolutions per minute of the output shaft with a mechanical tachometer. The column was filled with water, simulating load conditions, and the 1/8 inch displacement cam was used to reciprocate the packing during the calibration. Measurements of shaft speed were made with a Hasler Type B Mechanical Tachometer, serial number 121762, obtained from the Mechanical Engineering Department. An average of three readings of shaft speed was used for each micrometer dial position. The micrometer dial position was varied in two division increments over the entire 32 division scale. The calibration data appears in Table VI, and the transmission calibration curve is presented in Figure 25.

TABLE VI

Calibration of the Graham VariableSpeed Transmission

Micrometer Dial Position	Output Shaft Speed
	rev/min
0.0	0.0
2.0	74.0
4.0	148.0
6.0	221.0
8.0	294.0
10.0	370.0
12.0	448.0
14.0	520.0
16.0	592.0
18.0	660.0
20.0	731.0
22.0	801.0
24.0	868.0
26.0	920.0
28.0	980.0
30.0	1040.0
32.0	1100.0

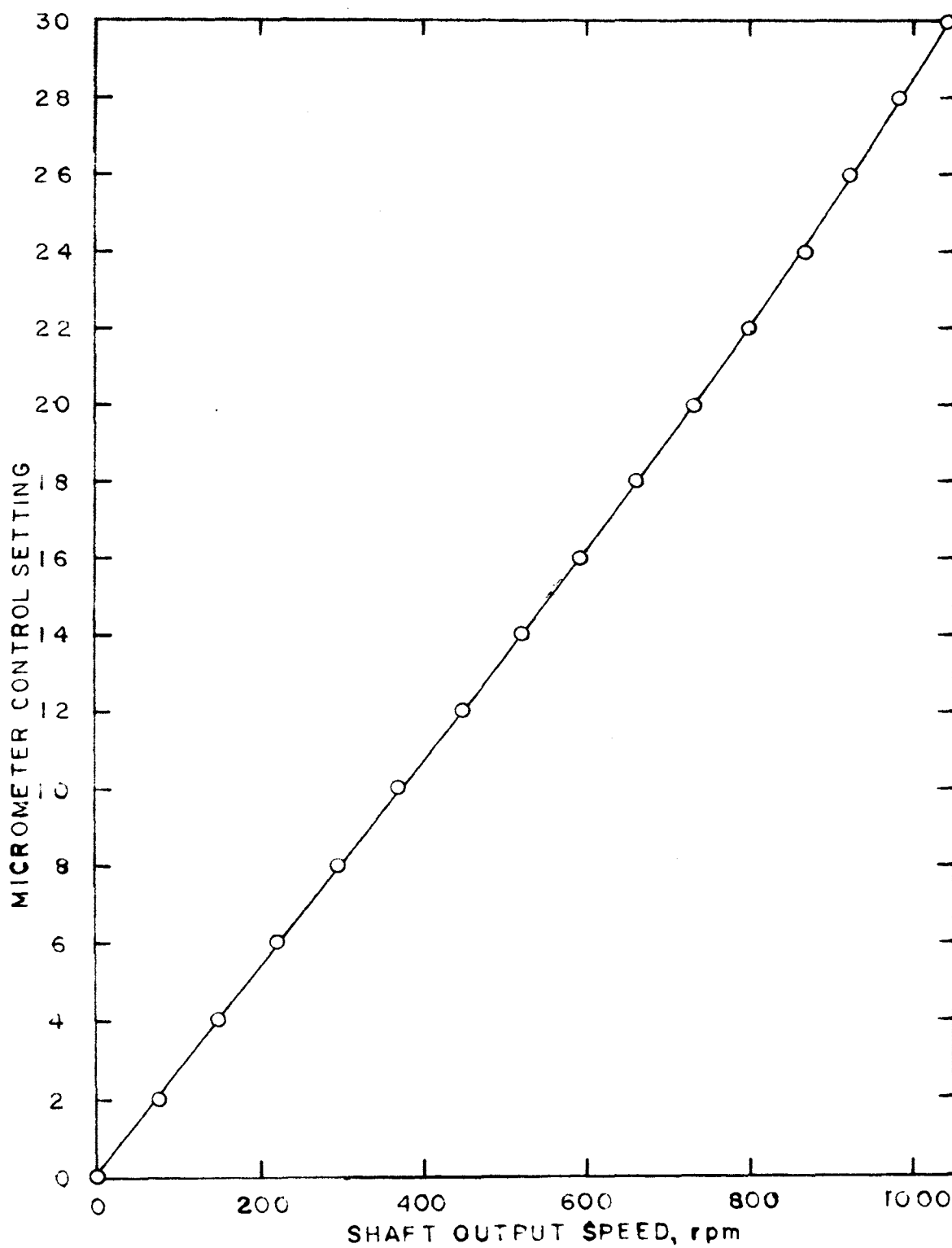


Figure 25. Variable Speed Transmission Calibration Plot

TABLE VII

Extraction Column Performance Data with Acetic Acid Transfer from the
Ketone Phase to the Water Phase

Test No.	Temperature OC	Water		Ketone		Concentration of Acetic Acid			
		Rotameter Position	Rotameter Position	Ketone Phase	Water Phase	Inlet Streams wt. %	Water Phase	Ketone Phase	Outlet Streams wt. %
3.04F	28.0	.42	.37	1.451	.007	.180	.701		
13.10	27.8	.23	.20	1.429	.023	.153	.965		
3.03F	28.0	.47	.42	1.451	.007	.197	.759		
13.01	27.8	.23	.20	1.429	.023	.242	.901		
3.02F	28.0	.57	.50	1.451	.007	.223	.883		
13.02	27.8	.48	.43	1.429	.023	.315	.864		
13.03	27.8	.23	.20	1.429	.023	.283	.846		
3.01F	28.0	.70	.63	1.451	.007	.322	.834		
13.04	27.8	.48	.43	1.429	.023	.283	.846		
13.05	27.8	.23	.20	1.429	.023	.332	.797		
1.06F	28.3	.69	.61	1.372	.007	.205	.757		
11.06	28.3	.48	.43	1.317	.010	.209	.873		
13.06	27.8	.23	.20	1.429	.023	.263	.876		
1.05F	28.3	.70	.63	1.372	.007	.233	.735		
11.07	28.3	.48	.43	1.317	.010	.246	.840		
13.07	27.8	.23	.20	1.429	.023	.306	.845		
1.04F	28.3	.73	.66	1.372	.007	.319	.726		
11.08	28.3	.48	.43	1.317	.010	.246	.840		
13.08	27.8	.23	.20	1.429	.023	.325	.802		
1.03F	28.3	.76	.68	1.372	.007	.360	.757		
11.09	28.3	.48	.43	1.317	.010	.393	.707		
13.09	27.8	.23	.20	1.429	.023	.394	.776		
1.02F	28.3	.72	.64	1.372	.007	.303	.849		
3.06F	28.0	.73	.65	1.451	.007	.283	.602		
20.10	27.8	.48	.43	1.622	.026	.255	1.117		
9.04F	28.3	.88	.80	1.450	.023	.459	.813		
22.06	28.2	.73	.66	1.549	.056	.430	.900		
22.05	28.2	.48	.43	1.549	.056	.438	.893		
22.04	28.2	.23	.20	1.549	.056	.268	.982		
5.02F	27.9	.48	.43	1.522	.013	.348	.876		
20.07	27.8	.23	.20	1.622	.026	.462	.906		
5.01F	27.9	.56	.50	1.522	.013	.410	.896		
5.05	27.9	.48	.43	1.522	.013	.319	.674		
20.02	27.7	.23	.20	1.622	.026	.406	.931		
5.03F	27.9	.74	.67	1.522	.013	.283	.853		
11.01	28.3	.48	.43	1.317	.010	.186	.883		
13.11	27.8	.23	.20	1.429	.023	.078	.916		
20.03	27.7	.23	.20	1.622	.026	.108	1.162		
5.04F	27.9	1.02	.93	1.522	.013	.238	.793		
11.02	28.3	.73	.66	1.317	.010	.168	.896		
20.04	27.7	.48	.43	1.622	.026	.090	1.171		
13.12	27.8	.23	.20	1.429	.023	.119	.978		
7.01F	27.3	.63	.57	1.524	.046	.332	.968		
18.01	27.6	.23	.20	1.533	.182	.442	1.037		
7.02F	27.3	.78	.71	1.524	.046	.283	1.015		
11.03	28.3	.48	.43	1.317	.010	.188	.836		
18.02	27.6	.23	.20	1.533	.183	.442	1.037		
7.03F	27.3	.92	.84	1.524	.046	.299	1.035		
11.04	28.3	.73	.66	1.317	.010	.147	.909		
18.05	27.6	.48	.43	1.533	.183	.199	1.238		
18.03	27.6	.23	.20	1.533	.183	.136	1.291		
7.04F	27.3	1.00	.91	1.524	.046	.334	.959		
11.05	28.3	.73	.66	1.317	.010	.184	.873		
20.01	27.7	.48	.43	1.622	.026	.118	1.173		
18.04	27.6	.23	.20	1.533	.183	.187	1.229		
9.01F	28.3	.94	.85	1.450	.033	.258	1.031		
20.05	27.7	.48	.43	1.622	.026	.349	1.057		
20.06	27.7	.23	.20	1.622	.026	.093	1.171		
9.02F	28.3	.96	.87	1.450	.033	.319	.925		
20.08	27.8	.48	.43	1.622	.026	.036	1.216		
20.09	27.8	.23	.20	1.622	.026	.045	1.213		
9.03F	28.3	.80	.72	1.450	.033	.414	.892		
22.03	28.2	.73	.66	1.549	.056	.212	1.090		
22.02	28.2	.48	.43	1.549	.056	.209	1.060		
22.01	28.2	.23	.20	1.549	.056	.251	.995		

TABLE VIII
Extraction Column Performance Data With Acetic Acid Transfer from the
Water Phase to the Ketone Phase

Test No.	Temperature °C	Water		Ketone		Concentration of Acetic Acid			
		Rotameter Position	Rotameter Position	Rotameter Position	Rotameter Position	Inlet Streams	Water Phase	Ketone Phase	Outlet Streams
						wt. %	wt. %	wt. %	wt. %
12.01F	28.2	.63	.56	.434		3.094		1.510	2.408
4.05F	28.2	.66	.60	.516		3.012		1.755	2.286
19.05	28.3	.48	.43	.381		3.571		1.612	2.616
19.07	28.3	.23	.20	.381		3.571		1.880	2.358
4.06F	28.2	.78	.70	.516		3.012		1.755	2.342
19.06	28.3	.48	.43	.381		3.571		1.888	2.358
19.08	28.3	.23	.20	.381		3.571		1.914	2.299
12.02F	28.2	1.05	.95	.434		3.094		1.637	2.134
14.01	28.2	.48	.43	.458		3.167		1.616	2.236
14.02	28.2	.23	.20	.458		3.167		1.719	2.210
4.04F	28.2	.75	.68	.516		3.012		1.727	2.246
19.01	28.3	.48	.43	.381		3.571		1.803	2.436
19.02	28.3	.23	.20	.381		3.571		1.910	2.377
4.03F	28.2	.82	.74	.516		3.012		1.710	2.233
19.04	28.3	.48	.43	.381		3.571		1.855	2.424
19.03	28.3	.23	.20	.381		3.571		1.896	2.351
4.02F	28.2	.96	.87	.516		3.012		1.653	2.151
14.03	28.2	.73	.66	.458		3.167		1.657	2.197
14.04	28.2	.48	.43	.458		3.167		1.670	2.204
14.05	28.2	.23	.20	.458		3.167		1.751	2.157
4.01F	28.2	1.01	.92	.516		3.012		1.483	2.215
14.06	28.2	.73	.66	.458		3.167		1.543	2.332
14.07	28.2	.48	.43	.458		3.167		1.551	2.291
14.08	28.2	.23	.20	.458		3.167		1.629	2.269
2.06F	28.2	.31	.73	.503		2.989		1.727	2.161
12.03	28.2	.73	.66	.434		3.094		1.675	2.055
2.05F	28.2	.94	.85	.503		2.989		1.739	2.204
12.04	28.2	.73	.66	.434		3.094		1.653	2.141
19.09	28.3	.48	.43	.381		3.571		1.758	2.456
19.10	28.3	.23	.20	.381		3.571		1.766	2.436
2.04F	28.2	1.05	.96	.503		2.989		1.506	2.233
12.05	28.2	.73	.66	.434		3.094		1.571	2.198
2.03F	28.2	1.10	1.00	.503		2.989		1.510	2.256
12.06	28.2	.73	.66	.434		3.094		1.489	2.266
10.04F	28.7	.96	.87	.499		3.061		1.489	2.289
23.06	27.7	.73	.66	.393		3.288		1.629	2.341
23.05	27.7	.48	.43	.393		3.288		1.530	2.426
23.04	27.7	.23	.20	.393		3.288		1.499	2.452
10.01F	28.7	.60	.53	.499		3.061		1.543	2.243
10.02F	28.7	.74	.67	.499		3.061		1.657	2.151
8.01F	27.4	.72	.66	.536		3.088		1.543	2.243
8.02F	27.4	.82	.75	.536		3.088		1.633	2.224
12.07	28.2	.73	.66	.434		3.094		1.702	2.091
8.05	27.4	.48	.43	.536		3.088		1.731	2.151
21.10	28.6	.23	.20	.399		3.393		1.965	2.126
8.03F	27.4	.90	.82	.536		3.088		1.670	2.184
12.08	28.2	.73	.66	.434		3.094		1.702	2.091
16.02	27.8	.48	.43	.471		2.989		1.670	2.039
14.10	28.2	.23	.20	.458		3.167		1.768	2.178
8.04F	27.4	1.03	.94	.536		3.088		1.571	2.276
16.07	27.8	.73	.66	.471		2.989		1.530	2.144
16.06	27.8	.48	.43	.471		2.989		1.547	2.118
14.09	28.2	.23	.20	.458		3.167		1.649	2.269
6.01F	27.4	.80	.71	.622		3.015		1.715	2.151
19.11	28.3	.48	.43	.381		3.571		2.006	2.247
21.01	28.6	.23	.20	.399		3.393		1.977	2.253
6.02F	27.4	.88	.80	.622		3.015		1.702	2.171
21.02	28.6	.73	.66	.399		3.393		1.867	2.247
23.08	27.7	.48	.43	.393		3.288		1.742	2.230
21.09	28.6	.23	.20	.399		3.393		1.850	2.286
6.03F	27.4	.96	.87	.622		3.015		1.616	2.223
6.05	27.4	.73	.66	.622		3.015		1.612	2.217
21.04	28.6	.48	.43	.399		3.393		1.772	2.326
21.05	28.6	.23	.20	.399		3.393		1.849	2.351
6.04F	27.4	1.00	.91	.622		3.015		1.563	2.276
21.06	28.6	.73	.66	.399		3.393		1.734	2.346
21.07	28.6	.48	.43	.399		3.393		1.792	2.339
21.08	28.6	.23	.20	.399		3.393		1.790	2.384
10.03F	28.7	.98	.88	.499		3.061		1.481	2.269
23.03	27.7	.73	.66	.393		3.288		1.669	2.322
23.02	27.7	.48	.43	.393		3.288		1.673	2.319
23.01	27.7	.23	.20	.393		3.288		1.624	2.371

COMPUTER PROGRAM

LIQUID-LIQUID EXTRACTION, SYSTEM, MIBK-ACETIC ACID-WATER
READ 110, NSETS
1 DO 100 J=1, NSETS
 READ 2, TEST, KTRAF, KTRDP, RPM, TEMP, DISPL, XNORM
 READ 3, VMKI, VMWI, VMKO, VMWO
 READ 3, RAFIN, EXTIN
 VOL=10.0
 WT=60.05
 IF (KTRAF) 5, 7, 5
 5 AKTD=0.8035
 AWAD=0.9956
 GO TO 10
 7 AKTD=0.8042
 AWAD=0.9976
10 WAKI=(VMKI*XNORM*WT)/(10.0*AKTD*VOL)
 WAKO=(VMKO*XNORM*WT)/(10.0*AKTD*VOL)
 WAWI=(VMWI*XNORM*WT)/(10.0*AWAD*VOL)
 WAWO=(VMWO*XNORM*WT)/(10.0*AWAD*VOL)
 FAKI=WAKI/100.
 FAKO=WAKO/100.
 FAWI=WAWI/100.
 FAWO=WAWO/100.
 IF (KTRAF) 12, 20, 12
12 RAFOT=(RAFIN)*(1.-FAKI)*(1.+FAKO)
 TOTAL=RAFIN+EXTIN
 EXTOT=TOTAL-RAFOT
 ACAIN=RAFIN*FAKI+EXTIN*FAWI

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ACAOT=RAFOT*FAKO+EXTOT*FAWO
DEV= ( (ACAOT-ACAIN)/ACAIN)*100.
AVGR= (RAFIN+RAFOT)/2.
AVGE=TOTAL-AVGR
SLOPE=1.57
A= (AVGR)/(SLOPE*AVGE)
B= (FAKI-FAWI/SLOPE)/(FAKO-FAWI/SLOPE)
ANTOD=LOGF(B*(1.-A) +A)/(1.-A)
D=23./12.
AHTOD=D/ANTOD
GO TO 29
20 RAFOT= (RAFIN)*(1.-FAWI)*(1.+FAWO)
TOTAL=RAFIN+EXTIN
EXTOT=TOTAL-RAFOT
ACAIN=RAFIN*FAWI+EXTIN*FAKI
ACAOT=RAFOT*FAWO+EXTOT*FAKO
DEV= ( (ACAOT-ACAIN)/ACAIN)*100.
AVGR= (RAFIN+RAFOT)/2.
AVGE=TOTAL-AVGR
SLOPE=0.637
A= (SLOPE*AVGE)/AVGR
B= (FAKI-FAWI*SLOPE)/(FAKO-FAWI*SLOPE)

ANTOD=LOGF(B*(1.-A) +A)/(1.-A)
D=23./12.
AHTOD=D/ANTOD
29 IF (KTRAF) 30,40,30
30 GPHF=RAFIN/6.69 + EXTIN/8.29
GO TO 41
40 GPHF=RAFIN/6.70 + EXTIN/8.31
41 IF (KTRAF) 42,43,42

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42  AHETS=(AHTOD*LOGF(1./A))/(1.-A)
    GO TO 50
43  AHETS=(AHTOD*LOGF(A))/(A-1.)
50  PUNCH 200, TEST
216 PUNCH 217,TEMP
    PUNCH 201,RPM,DISPL
    IF(KTDP)202,204,202
202 PUNCH 203
    GO TO 300
204 PUNCH 205
300 IF(KTRAF)206,208,206
206 PUNCH 207
    GO TO 400
208 PUNCH 209
400 PUNCH 210,TOTAL,RAFIN,EXTIN
    PUNCH 401,RAFOT,EXTOT,AVGR,AVGE
    PUNCH402,A ,GPHE
    PUNCH 211,WAWI,WAWO,WAKI,WAKO
    PUNCH 403, ACAIN,ACAOT
212 PUNCH 213,DEV
    PUNCH 215,ANTOD,AHTOD,AHETS
    2  FORMAT(E10.8,215,4E10.8)
    3  FORMAT (4E18.8)
110 FORMAT(I5)
200 FORMAT (7H3TEST =,F5.2)
217 FORMAT (14H TEMPERATURE = F4.1)
201 FORMAT (12H FREQUENCY =,F7.2,6X14HDISPLACEMENT =,F6.4 )
203 FORMAT (26H DISPERSED PHASE IS KETONE)
205 FORMAT (25H DISPERSED PHASE IS WATER)
207 FORMAT (20H RAFFINATE IS KETONE)
209 FORMAT (19H RAFFINATE IS WATER)

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210  FORMAT (13H THROUGHPUT =,F7.1,2X6HRAFF.=,F7.1,2X5HEXT.=,F7.1)
401  FORMAT(7H RAFOT=F7.1,2X6HEXTOT=F7.1,2X5HAVGR=F7.1,2X5HAVGE=F7.1)
402  FORMAT(20H EXTRACTION FACTOR =F6.3,6X6HGPHF =F7.1)
211  FORMAT(7H WAWI =F6.3,4X6HWAWO =F6.3,4X6HWAKI =F6.3,4X6HWAKO =F6.3)
403  FORMAT(17H ACETIC ACID IN =F9.3,4X17HACETIC ACID OUT =F9.3)
213  FORMAT (29H MATERIAL BALANCE DEVIATION =F12.2)
215  FORMAT (7H NTOD =F6.2,4X6HHTOD =F6.3,4X6HHETS =F6.3)
100  CONTINUE
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STOP
END
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