

FACTORS AFFECTING THE EXTRACTION EFFICIENCY  
OF AN EXPERIMENTAL, LIQUID-LIQUID,  
SIEVE-PLATE, PULSE EXTRACTOR

by

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## I. INTRODUCTION

Liquid-liquid extraction is the term applied to any unit operation in which one or more materials dissolved in one liquid phase are transferred to a second liquid phase. The extracting solvent must be insoluble, or soluble to a limited extent only, in the carrier solvent in which the solute is dissolved. This operation has been applied successfully in many important industrial and domestic processes. In the past, the liquid-liquid extraction operation has been effected in towers employing countercurrent flow of the liquids. These conventional towers have been equipped with bubble-cap trays, sieve plates, any of several arrangements of baffles, one or more spray heads, or have been filled with various types of packing, to increase the interfacial area between the two liquid phases. More recently, mixer-settler combinations and centrifugal extractors have been employed.

The columns employing countercurrent operation suffer from the disadvantage that the only energy available for the maintenance of the large interfacial area required for efficient extraction

is the density difference between the two liquid phases. Thus, the available energy, no matter how efficiently it is utilized by skillful design of packing or baffles, is too small to overcome drop coalescence resulting in a decrease in the area of contact. The many mixer-settler types of extractors, while offering a partial solution, are inherently batch extractors in operation. Recently, several new types of extractors have been developed that combine the desirable countercurrent features of the towers and the good mixing of the mixer-settler combinations. These devices usually take the form of a number of mixing sections, mounted one above the other to form a column, separated by packed or baffled settling sections. Agitation is accomplished by turbine mixers. An alternative design is to use an unpacked column with the fluids being forced through perforated baffles by mixers.

Another recent design for the improvement of liquid-liquid extraction is a column utilizing countercurrent flow, mechanical agitation, and baffled settling zones. The column is fitted with sieve plates that may be moved up and down relative to the liquid, producing the agitation, while the

liquids flow countercurrently through the plates. Settling takes place during the pause between the strokes. The operation and construction of this column can be simplified by using stationary plates, and agitating by imparting a reciprocating motion to the liquids relative to the plates.

The purpose of this investigation was to determine the optimum column operating characteristics, and to study the effect of pulse amplitude, solvent-to-feed ratio, and feed concentration on the overall mass transfer coefficient for the extraction of acetone from toluene using water as the extracting solvent in an experimental, sieve-plate, liquid-liquid pulse extractor.

## II. LITERATURE REVIEW

Liquid-liquid extraction is becoming increasingly important as a mass transfer operation and considerable development work is being accomplished to increase the efficiency of liquid-liquid extractors. It has been found that the pulsation of the liquid contents in a sieve-plate, countercurrent extraction column greatly increases the rate of mass transfer between the two liquid phases. The development of these types of extractors will be discussed in this review after a discussion of the general concepts and definitions pertaining to extraction, and a review of some of the existing extraction equipment.

### Concepts of Extraction

The following section presents some of the general concepts pertaining to extraction and its uses.

Liquid-liquid Extraction. Liquid-liquid extraction is one of a series of separation techniques that depends upon the tendency of a substance, when distributed between two phases, to occur in different concentrations in the two phases at equilibrium<sup>(24)</sup>.

Some of these methods of separation are distillation, evaporation, and adsorption. Liquid-liquid extraction<sup>(6)</sup> is a diffusional process involving the transfer of one or more components from one liquid phase to another.

Liquid-liquid extraction may be accomplished by any one of the following methods, depending upon the nature of the solution to be treated<sup>(24)</sup>: (1) the addition of a third liquid immiscible, or nearly immiscible, with one of the components of the original solution; (2) the cooling or heating of the original solution to effect the formation of two liquid phases in which the concentration of solute in each phase will differ; (3) the addition of a third component which will cause the original solution to separate into two phases; and (4) the addition of two immiscible liquids each of which dissolves a separate component of the original solution.

Uses of Liquid-liquid Extraction. According to Elgin<sup>(6)</sup>, extraction may be preferable to other separation operations when the following conditions are present: (1) the boiling points of the components of the liquid mixture are within a very

few degrees of each other; (2) one or more of the liquids are sensitive to heat; (3) the concentration of solute in the original solution is very low and the solute is the least volatile; and (4) when the solvent used has a lower heat of vaporization than the original solution. Brown<sup>(3)</sup> adds a fifth condition such that the two liquids are non-volatile, or nearly so.

Treybal<sup>(24)</sup> points out that before deciding to use any extraction operation, the following points must be considered. First, the addition of more components has several disadvantages; namely, a large amount of capital is invested in inventories, the selection of materials of construction to insure corrosion resistance is made complicated, plant equipment must necessarily be made larger since storage and recovery facilities must be available, and the danger of contamination of the final product is increased. Second, the extraction process must be followed by a solvent recovery process, usually distillation. This solvent recovery quite often represents a major portion of the total cost of the extraction process.

Some of the industrial applications of extraction<sup>(6)</sup> are in the refining of lubricating



oils, purification of petroleum naphthas, refining of vegetable oils to separate drying from non-drying fractions, concentration and recovery of dilute acetic acid, recovery and concentration of penicillin, and the purification and decolorization of crude wood rosins.

### Definitions

The following section defines specific terms that are often used regarding liquid-liquid extraction.

Feed. The original solution to be extracted is called the feed<sup>(30)</sup>, and consists of the solute which is dissolved in what is termed the carrier solvent. The solute is not necessarily the desired product of the extraction operation.

Solvent. The solvent is the liquid with which the feed is contacted<sup>(30)</sup> in order to bring about the separation of the components of the feed.

Extract Phase. The contacting of the feed solution with the solvent results in the formation of two liquid phases<sup>(6)</sup>. The solvent-rich layer containing the preferentially dissolved solute is

termed the extract phase. The extract is the term given to the solvent-free solute.

Raffinate Phase. The layer that is made up of the carrier solvent, some still dissolved solute, and possibly some solvent, is called the raffinate phase<sup>(6)</sup>. The raffinate is the term given to the solvent-free solution of carrier solvent and solute.

Extraction Stage. Brown<sup>(3)</sup> states that the physical operation of extraction consists of two stages: first, the mixing together of the two phases, allowing time for equilibrium to be attained; and, second, the separation of the two resulting phases. Each combination of these two stages, in the order mentioned, constitutes one extraction stage.

Ideal Stage. An ideal stage<sup>(6)</sup> is one in which the two phases have attained equilibrium. Equilibrium is the condition resulting from the distribution of the solute between the two phases as required by the distribution law<sup>(25)</sup>. An ideal stage represents the limiting amount of extraction for any stage.

### Extraction Operations

The following paragraphs contain a review of extraction operations.

Types of Contact. There are two principal methods of contacting the feed solution with the solvent. The first, stagewise contact<sup>(20)</sup>, consists of alternately mixing and separating the two liquid phases. A more rigorous definition of a stagewise contactor would be any extractor in which the interfacial area between the two phases varies with position in the equipment or with time. The second, continuous contact<sup>(20)</sup>, involves the continuous flow of the two phases past each other. Continuous contact may also be defined as any extraction process in which the interfacial area between the two phases does not vary substantially with position in the column or with time during the operation of the column.

Classification of Extraction Operations. There are two primary methods of classifying extraction. The first is described by Treybal<sup>(27)</sup> and is based on the different solvent systems. The second, described by Elgin<sup>(5)</sup>, is based on equipment arrangement.

### Rate of Mass Transfer

The following section describes briefly the concept of mass transfer and its application to this investigation.

Mass Transfer Concept. The two-film theory<sup>(26)</sup>, although inadequate to completely describe extraction behavior, has been widely accepted as the closest available approximation. In this theory the interface between the two phases is believed to be formed of two compressed, laminar films of the type encountered in heat transfer. It is assumed that all the resistance to transfer of the solute takes place in the films. Experimental results show that there is a measurable resistance<sup>(21)</sup> to the transfer of solute from phase to phase. In effect, this means that a finite time is required for a system to approach equilibrium. The reciprocal of the equilibrium-time function is called the overall mass transfer coefficient, and is a measure of the rate at which extraction takes place.

Overall Mass Transfer Coefficient. There are many ways of expressing the overall mass transfer coefficient, and for this investigation a modification of the form used by Elgin<sup>(7)</sup> was selected. The

interfacial area between the two phases could not be determined, so this term was included in the overall mass transfer coefficient.

Elgin's form of the equation for the overall mass transfer coefficient is given as:

$$K_R = \frac{N}{(C_{RL} - C_R^*) A}$$

where:

- $K_R$  = overall mass transfer coefficient,  
lb mol/hr-sq ft-lb mol/cu ft
- $N$  = amount of solute transferred,  
lb mol/hr
- $C_{RL}$  = concentration of solute in main body  
of raffinate phase, lb mol/cu ft
- $C_R^*$  = concentration of solute in raffinate  
which would be in equilibrium with  
the solute concentration of the  
main body of the extract phase,  
lb mol/cu ft
- $A$  = total interfacial area, sq ft.

Because the total interfacial area is unknown, the following relation is used:

$$A = aV$$

where:

A = total interfacial area, sq ft

a = effective interfacial area of columns  
per unit volume, sq ft/cu ft

V = effective volume of column, cu ft.

A further modification was used in that the concentration difference expressed in pound mols per cubic foot was replaced by the log-mean average<sup>(32)</sup> of the concentration difference expressed in mol fraction of the solute-free raffinate.

The final form of the equation used for calculating the overall mass transfer coefficient is given below:

$$K_{ra} = \frac{N}{(1 - Y)_m V}$$

where:

$K_{ra}$  = overall mass transfer coefficient,  
lb mol/hr-mol fraction -cu ft

$N$  = amount of solute transferred,  
lb mol/hr

$(1 - Y)_m$  = log mean average of solute-free  
raffinate, mol fraction

$V$  = effective volume of column, cu ft.

### Extraction Equipment

The following section includes a discussion of some design features of various types of commercial extraction equipment, the use of external power in extraction equipment, and pulse columns.

Liquid-liquid Extractor. A liquid-liquid extractor<sup>(7)</sup> may be defined as any type of equipment used to effect liquid extraction. There are many different commercially used types of liquid-liquid

extractors on the market. This condition is due to several factors<sup>(15)</sup>: lack of reliable performance data on existing equipment, individual development by the different industries, modifications of distillation and absorption apparatus to extraction, and the need for specialized design of extractors for specific purposes.

Classifications of Liquid-liquid Extractors.

There are three general methods presented for the classification of liquid-liquid extractors. Elgin<sup>(7)</sup> uses the two broad headings of mixer-settler extractors and continuous, countercurrent columns, with further subdivision for specific designs. Treyabl<sup>(28)</sup> modifies Elgin's classification by outlining in more detail the continuous, countercurrent types of extractors. Morello and Poffenberger<sup>(11)</sup> classify extractors as to the method of separating the two liquid phases. The gravity separation class is subdivided into the methods of producing the interfacial area, then into specific types of equipment. The second method of phase separation, centrifugal force, is a class not further subdivided.



The Use of External Power in Extraction Equipment.

Studies have been made on extraction equipment<sup>(28)</sup> to determine the economic feasibility of using external power in its operation. External power may be defined<sup>(15)</sup> as any power supplied to the extractor in addition to that power necessary to cause flow of the liquid phases.

This additional power, if used efficiently, can be made to increase the interfacial area of contact between the two liquid phases, thus increasing the extraction efficiency. In an economic study this advantage must be balanced against the additional mechanical complexity of the equipment, and consequently the increased possibility of mechanical failure, and the additional cost of operation.

Centrifugal Extractor. The only successful extractor of this type is the Podbielniak centrifugal extractor<sup>(7,29)</sup>. This device consists of a rotor made up of a 300-foot length of steel sheet wound spirally inside a steel casing. The heavy phase is pumped through one end of the rotor shaft to the center of the spiral while the light phase is pumped through the other end of the rotor shaft to the periphery of

the spiral. High rotative speeds cause the heavy phase to flow countercurrently to the light phase, to the periphery of the spiral, from where it is led back to the shaft and out of the machine. In the same fashion the light phase flows to the center of the spiral and is removed from the shaft. Some designs consist of perforating the steel sheet causing the heavy phase to short-circuit through the light phase which, because the interfacial area is increased, increases the efficiency of the unit.

Podbielniak extractors have very large capacities (up to 2400 gallons per hour of combined liquids) relative to their size and hold-up (nine gallons in a 2400 gallon per hour machine). Such extractors usually have about five theoretical stages.

Film Type Extractor. Film extractors are of three types: the wetted wall, the horizontal tube, and the Gordon and Zeigler<sup>(8)</sup> extractor.

The wetted wall column<sup>(7)</sup> consists of one phase flowing down the inside wall of a column and the other phase flowing countercurrently up through the middle. Such a column has the limitation of low flow rates,

but is advantageous where a known interfacial area is required for fundamental studies.

A larger range of flow rates is possible in the horizontal tube extractor<sup>(21)</sup> in which the interfacial area can also be accurately measured. The heavy phase flows along the bottom of the tube countercurrently to the light phase which flows through the top.

The film extractor of Gordon and Zeigler<sup>(8)</sup> consists of passages, rectangular in cross section, inclined at an angle to induce layer formation within the passage. This type of design is a commercial one, and allows liquids to be treated that would tend to emulsify if dispersed.

Spray Tower Extractor. The spray tower extractor<sup>(29)</sup> is the simplest and most economical continuous, countercurrent extractor. It consists of a cylindrical shell through which the two phases pass countercurrently. One phase, either light or heavy, is continuous throughout the column, and the other phase is dispersed into droplets by nozzles, and travels by gravity through the continuous phase.

This type of tower is used widely in laboratory studies because of its cheapness and simplicity, but is little used industrially because of certain disadvantages. These disadvantages are primarily the coalescence of the dispersed phase before it has traveled the length of the extractor, and the vertical recirculation of the continuous phase. The reader is referred to Blanding and Elgin<sup>(2)</sup> for a discussion of spray tower designs in which these defects have been eliminated or alleviated.

Packed Extraction Column. The packed extraction column<sup>(29)</sup> is similar to the spray column except that the shell is filled with packing to alter the motion of the phases. The packing serves several purposes<sup>(30)</sup>:

- (1) it increases the turbulence of the continuous phase,
- (2) by hindering the movement of the drops of dispersed phase it continually distorts their shape resulting in exposure of fresh surface, and
- (3) it prevents the vertical recirculation of the continuous phase.

The packing material should be such that it is preferentially wetted by the continuous phase in order for the dispersed phase

to pass through the column in droplets rather than rivulets.

Perforated Plate Column. The perforated- or sieve-plate column<sup>(29)</sup> operates by having one of the liquid phases repeatedly dispersed and recombined at intervals, by perforated plates spaced throughout the column. The continuous phase exists as a layer above or below each perforated plate, and is directed through the column by pipes or baffles which are long enough to cause a liquid seal in the continuous phase at the next plate.

Baffle Extractor. Baffle extractors<sup>(29)</sup> are continuously and countercurrently operated, and use horizontal baffles to effect contact between the two liquid phases. They were developed solely for use in liquid-liquid extraction and are of three basic types. These are the disk and doughnut, side-to-side, and center-to-side types. Either the light or the heavy phase may be continuous with the other phase being caused to flow through the continuous phase by the baffles.

Baffle towers<sup>(29)</sup> are capable of handling a wide variety of liquids and flow rates, and are free of the

tendency<sup>(31)</sup> toward vertical recirculation of the continuous phase, which is present in the spray tower, and clogging in the presence of suspended solids which may make packed towers undesirable.

Mixer and Settler Extractor. Mixer and settler type extractors<sup>(28)</sup> consist of a mixing chamber followed by a settling chamber for each stage, and are usually dependable units. They are, however, quite bulky, the separate components taking up a great deal of floor space.

To compromise between the high efficiency and inherent dependability of the mixer-settler extractors and the small size of the continuous extraction towers, many combinations of the two types have been proposed. Kiersted<sup>(12)</sup> suggested mounting a series of settling chambers one above the other, the liquids being removed, mixed, and returned to the proper settling chamber. McConnell<sup>(14)</sup> developed the idea of mounting the mixing sections in the tower between the settling sections, the mixing being accomplished by turbine mixers. Variations of this arrangement include Othmer's<sup>(16)</sup> baffles and Scheible's<sup>(19)</sup> fine, wire mesh packing.

Pulse Columns. In 1934, Van Dijck<sup>(33)</sup> proposed an extractor that consisted of a vertical tower equipped with movable, perforated plates. The plates were raised and lowered through the main body of the fluid to produce agitation. Because the bulky mechanism for imparting motion to the plates was located inside the column, and was thus subject to corrosion, Van Dijck proposed an alternate design. The operation of the redesigned column was similar to the old one except that the plates remained stationary and a reciprocating motion was imparted to the fluids in the column by a piston.

At any time during the operation of the column both phases were present in the sections between the plates. During the rest portion of the reciprocating cycle the light phase collected just beneath the plates and the heavy phase collected just above the plates. The hole size of the plates was just small enough to prevent flow through the column due to differences in density of the two phases. A cyclic pulse was applied to the light phase, and on the upstroke the light phase was pulsed through the holes in the plates and dispersed as droplets into the layer of heavy phase.

The light phase coalesced beneath the plates and on the downstroke the heavy phase was dispersed into the light phase just beneath the plates. Fresh surface was created with each pulse, and mass transfer rates were therefore higher than for a similar spray or packed column.

### Pulse Column Operations

This section includes a discussion of some of the various pulse columns and pulse column techniques that have been undergoing investigation during the past ten years.

Iowa State College Extractor. The pulse column studied at the Iowa State College<sup>(4)</sup> consisted of a pyrex column one inch in diameter and 20 inches high. The column contained ten perforated plates with a hole diameter of 0.040 inch and a plate free area of nine per cent. A diaphragm pump with the check valves removed was used to impart a pulsing motion to the liquid with frequencies ranging from 35 to 72 cycles per minute. For this column, values of the HTU (height of a transfer unit) as low as 0.48 foot were reported.



North Carolina State College Extractor. The column studied at North Carolina State College<sup>(17)</sup> consisted of a precision bore glass tube 1.5 inches in diameter and eight inches long. The column contained four plates with a two-inch plate spacing, a hole size of 0.059-inch diameter, and a plate free area of 20 per cent. The pulsing motion was applied by a constant volume feed pump with modified check valves at a pulse frequency of 24 strokes per minute. For this column, values of the HTU as low as 0.14 foot were reported.

Oak Ridge Pulse Column. A pulse column was investigated at the Oak Ridge National Laboratories<sup>(22)</sup> for the separation of rare earth isotopes and radioisotopes. The column was made of three-foot sections of 1/2-inch pyrex tubing, flanged together with stainless steel flanges and polyethylene gaskets. One-inch diameter separating sections, six inches long, were welded onto the flanges by bell reducers at each end of the column. About 34 plates per glass section were used, each one containing thirty-two 0.039-inch holes corresponding to a plate free area of about 22 per cent.

Griffity, Jasny, and Tupper<sup>(9)</sup>, in an investigation of pulse columns at Oak Ridge, studied the extraction of nickel and copper using water and methyl isobutyl ketone as solvents. The aqueous phase was maintained continuous, and the volumetric ratio of the dispersed to the continuous flow rate was 1.57. Using a plate spacing of one inch the flooding velocity was 955 gallons per hour-square foot of column cross section. For this system and column, values of the HTU varied from 1.1 inches at low flow rates to a maximum of 34 inches at high flow rates. In every case, however, the value was lower than the corresponding value for a spray column.

Pulsed Spray Column. Billerbeck and others<sup>(1)</sup> operated a pulsed spray column to study the effect of pulse frequency at constant flow rates and the effect of flow rates at constant pulse frequency on the extraction efficiency. The column consisted of a six-foot section of 1.5-inch glass pipe. The hole size of the plates at the stream inlets was 1/16-inch and the pulse was furnished by a motor-driven cam and brass bellows. The frequencies used were between

200 and 500 cycles per minute, and the pulse amplitude was 7/16-inch.

Concatenated Pulse Column. Jealous and Lieberman<sup>(11)</sup> have investigated concatenated pulse columns designed to provide maximum contact height without requiring excessive structural head room. A concatenated column consists of two or more contacting sections mounted in parallel, and connected in series for countercurrent flow with check valves in the connecting lines. Laboratory tests were made on two-inch diameter pyrex glass pipe columns, six feet long, and containing sieve plates with 0.125-inch holes, a plate free area of 22 per cent, and a plate spacing of two inches. The pulsing mechanism consisted of a variable-speed, variable-stroke, positive displacement piston pump with the pump head check valves removed. Only one pulser was required for the combined unit, and the power used was less than that for a tall column of the same diameter and equivalent length. The frequencies used in this investigation ranged from 30 to 70 cycles per minute, and the pulse amplitude was varied from 0.5 to 1.2 inches. The combined flow rates were varied from

300 to 1000 gallons per minute. Results of the tests indicated that the flow capacities of the concatenated column were 10 to 40 per cent greater than for a tall column of the same diameter and equivalent length, and that the extraction efficiencies for the two arrangements were about equal.

Pulse Column Techniques. Early work with pulse columns was carried out with normal types of random packing. Thornton<sup>(23)</sup>, however, has shown that randomly packed towers are not ideally suited for such purposes, as the pulsations cause the packing to orientate. It has been found that the best packing to use is fixed or regular packing such as corrugated screens or sieve plates.

Pulsing has been accomplished by means of valveless piston pumps, and also by an electronic oscillator unit and transducer operating a diaphragm on the column base. A new method of air pulsation has been developed which eliminates bellows, diaphragms, and other moving parts in contact with the liquid. The pulser is located above the level of the liquid in the column, so that the pulse is transmitted by a pocket of air or inert gas through a dip-tube which passes down

the entire length of the column, either internally or externally.

Power Requirements for Pulse Columns. Jealous and Johnson<sup>(10)</sup> present a detailed mathematical analysis of power requirements for pulse generators. The theory is augmented by experimentally determined empirical corrections. Several types of pulse generators are described, and experimental details for power analysis are given. The power was found to be a function of static head, inertia, and friction of the fluid in the column.

Redistribution Plate. The development of a redistribution plate, which improves extraction performance in a large diameter pulse column, is described by Woodfield and Sege<sup>(24)</sup> as overcoming the adverse effects of channeling. The redistributor consists of a solid horizontal plate fitting snugly into the column, and provided with louvers designed to swirl the column contents as they are pulsed. In a particular investigation, redistributors reduced the solute content in the raffinate stream from 6.00 to 0.01 per cent of the amount of solute

in the feed stream for the solvent extraction of uranyl nitrate with tributyl phosphate.

### III. EXPERIMENTAL

The experimental section contains the purpose of investigation, a list of the materials and apparatus used, the method of procedure followed in the investigation, the data and results obtained during the experimentation, and the sample calculations.

#### Purpose of Investigation

The purpose of this investigation was to determine the optimum operating characteristics of a two-inch diameter, liquid-liquid, sieve-plate, pulse extraction column, and using the system acetone-toluene-water, to determine the effect of pulse amplitude, solvent-to-feed ratio, and feed concentration on the overall mass transfer coefficient for the extraction of acetone from toluene using distilled water as the extracting solvent.

### Plan of Experimentation

The experimental plan followed in this investigation consisted of a review of the literature, modification of some of the existing equipment, determination of the column operating characteristics, operation of the column for extraction of acetone from toluene using distilled water as the extracting solvent, and the presentation of the data and results obtained.

Literature Review. A review of the literature was made which consisted of a study of general definitions and concepts pertaining to extraction, a review of the theory of mass transfer rates and the overall mass transfer coefficient, and a study of liquid-liquid extraction equipment including pulse columns and pulse column techniques.

Modifications to Equipment. The existing 55-gallon drums, which had been used as storage tanks for the feed, solvent, extract, and raffinate, had been painted to withstand the effects of corrosion due to the chemical action of toluene and acetone. This coating, however, was slowly being removed by chemical action with toluene and acetone. For this reason all four steel drums were replaced with 50-gallon, war surplus, aluminum drums. Coils were installed in the



feed and solvent tanks through which steam or tap water could be passed separately or simultaneously. A network of copper tubing and valves was installed so that liquids could be pumped from the extract and raffinate tanks to either the feed or solvent tanks, or to tanks located in the next room. Liquids could also be pumped from the next room into the feed and solvent tanks.

Column Operating Characteristics. Before it was possible to operate the column for extraction studies it was necessary to establish the conditions under which stable operation could be attained. Stable operation was the condition obtained at which the flow rates were adjusted so as to maintain constant interface levels in both the top and bottom disengaging sections. These conditions were determined for pulse amplitudes of one, two, and three inches, and at a pulse frequency of 5.3 cycles per minute.

Extraction Rate Studies. A total of forty-five different tests were made in order to determine the extraction efficiency of the column in terms of the overall mass transfer coefficient. The following variables were investigated: (1) feed concentrations

of five, ten, and fifteen weight per cent acetone in toluene; (2) pulse amplitudes of one, two, and three inches; and (3) solvent-to-feed ratios of 0.50, 0.75, 1.00, 1.25, and 1.50. Mass transfer was accomplished in the extraction of acetone from toluene using distilled water as the extracting solvent.

Presentation of Data and Results. The column operating characteristics were plotted in terms of the light and heavy phase flow rates for each of the three pulse amplitudes.

The mass transfer coefficients obtained from the calculation of operational data were plotted against the operational variables of pulse amplitude, solvent-to-feed ratio, and feed concentration. Two series of curves were plotted for this purpose.

The first series of curves consisted of three sets, each set being for one particular pulse amplitude. In each of these sets of curves the overall mass transfer coefficient was plotted on the ordinate against the solvent-to-feed ratio on the abscissa, for different feed concentrations. The second series of curves consisted of five sets, each set being for one particular solvent-to-feed ratio. In each of

these sets the overall mass transfer coefficient was plotted on the ordinate against the feed concentration on the abscissa for different values of pulse amplitude.

### Materials

The following section presents a list of materials used in this investigation, with specifications and source.

Acetone. Grade NF, lot No 90777. Manufactured and distributed by J. T. Baker Chemical Company, Phillipsburg, New Jersey. Used as the solute in the feed solution.

Toluene. Purified, lot No 90129. Manufactured and distributed by J. T. Baker Chemical Company, Phillipsburg, New Jersey. Used as the carrier solvent in the feed solution.

Water, Distilled. Obtained from and distilled in the Unit Operations Laboratory of the Chemical Engineering Department at the Virginia Polytechnic Institute, Blacksburg, Virginia. Used as the extracting solvent.

Apparatus

The apparatus used in this investigation are described in the following paragraphs.

Balance, Analytical. Two hundred-gram capacity, 0.0001-gram increments, sensitivity 0.05 mg at full load, model No 220-0, serial No M16011. Manufactured by Voland and Sons, Inc., New Rochelle, New York. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used to weigh solutions in the determination of specific gravity data.

Balance, Westphal. Specific gravity, chainomatic, range 0.0000 to 2.1000, increments of 0.0001 with vernier. Manufactured by Christian Becker, Inc., New York, New York. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in the determination of specific gravity data.

Column, Pulse. The pulse column used in this investigation was the same as that constructed by Knapp<sup>(13)</sup>.

Constant Temperature Bath. Range to 210 °F, serial No J-4, catalog No 66600, 115 v, 4 amp, 50/60 cy, 450 w, single phase. Manufactured by

Precision Scientific Company, Chicago, Illinois.  
Obtained from Fisher Scientific Company, Silver Spring,  
Maryland. Used to control temperature of solutions  
being measured for refractive index.

Cylinders, Graduated. Three used, capacity  
1000 ml, graduated in units of 10 ml, catalog  
No 8-552. Obtained from Fisher Scientific Company,  
Silver Spring, Maryland. Used for collection of  
samples in the measuring of liquid flow rates.

Gages, Pressure. Two used, range 30 in. mercury,  
vacuum, to 300 lb per sq in., gage, increments of  
five pounds per square inch. Obtained from Noland  
Company, Roanoke, Virginia. Used to measure the line  
pressure in the feed and solvent flow lines.

Glassware, Miscellaneous. Standard laboratory  
glassware such as flasks, beakers, and sample bottles.  
Obtained from Fisher Scientific Company, Silver Spring,  
Maryland. Used in the preparation of solutions for  
the determination of specific gravity data and the  
collection of samples from column operation.

Manometers, U-tube. Two used, 16-inch scale,  
0.1-inch graduations. Manufactured by Meriam  
Instrument Company, Cleveland, Ohio. Used to

measure the differential pressure across the orifices in the solvent and feed lines.

Motor, Electric. Model No 5KH45AB1647H, 1725 rpm, 115 v, 5.6 amp, single phase, 60 cy, ac, 1/3 hp. Manufactured by General Electric Corporation, Schenectady, New York. Used to drive the feed pump.

Motor, Electric. Serial No CR19483, style 220-402, type RA, frame H56, 1/4 hp, 115 v, 4 amp, single phase, 60 cy, ac, 1725 rpm. Manufactured by Master Motor Company, Dayton, Ohio. Used to drive the solvent pump.

Plates, Perforated. Eight used, 5-inch diameter, made from No 14 B&S gage aluminum sheet with perforations of No 8B, staggered 1/16-inch holes, with a plate free area of 23 per cent. Obtained from Charles Mundt and Sons, Jersey City, New Jersey. Used as baffle dispersion plates in the pulse column.

Pulsing Mechanism. The mechanism used to generate pulsation in the pulse column was the same as that constructed by Hayford<sup>(34)</sup>.

Pumps, Gear. Two used, model No FFH28, 3/4-inch inlet and outlet ports. Manufactured by Viking Pump

Company, Cedar Falls, Iowa. Used to pump the feed and solvent to the pulse column.

Scales, Beam. Three hundred-pound capacity, 1/4 lb graduations, model No 5732, serial No 1554093. Manufactured by Howe Scale Company, Rutland, Vermont. Used in the preparation of the feed.

Still. Serial No 18434, capacity 10 gallons per hour. Manufactured by Barnstead Still and Sterilizer Company, Inc., Boston, Massachusetts. Used in the preparation of the distilled water to be used as the extracting solvent.

Tanks, Storage. Four used, aluminum 52-8, 50-gallon capacity. Obtained from U. S. Government Surplus. Used to contain the feed, solvent, extract, and raffinate solutions.

Timer, Electric. Direct reading, increments of 0.1 second, 115 v, 60 cy, ac, catalog No 14-653. Manufactured by Precision Scientific Company, Chicago, Illinois. Obtained from Fisher Scientific Company, Silver Spring, Maryland. Used in the calibration of the solvent and feed orifices.

### Procedure

The method of procedure followed in the performance of this investigation consisted of the determination of refractive index data for the system acetone-toluene-water, the determination of specific gravity data for the system, calibration of the feed and solvent orifices, operation of the column, determination of the column operating characteristics, and the determination of the effect of pulse amplitude, feed concentration, and the solvent-to-feed ratio on the overall mass transfer coefficient.

Refractive Index Data. The refractive index data that were used were determined by Rood<sup>(18)</sup>.

Determination of Specific Gravity Data. The specific gravity of distilled water saturated with toluene and containing varying weight per cent of acetone, and toluene saturated with distilled water and containing varying weight per cent of acetone was determined at  $25 \pm 1.0$  °C by means of a Westphal balance.

Sufficient water and toluene to complete the specific gravity determinations were placed in a flask and shaken vigorously for five minutes to



completely saturate each component with the other. The two saturated phases were then separated by a separatory funnel. About 20 grams of the water-saturated toluene were placed in a tared test tube mounted on a cork stand and accurately weighed. Sufficient acetone was placed in the test tube to produce a solution of approximately one weight per cent, and the test tube was closed with a stopper of known weight. The solution, test tube, and stopper were then accurately weighed. The weight of acetone in the solution was determined by difference.

The test tube was placed in a constant temperature bath at  $25 \pm 1.0$  °C for about 15 minutes, and the specific gravity was determined with a Westphal balance.

This same procedure was followed for additional solutions of acetone in water-saturated toluene and toluene-saturated water until the specific gravity of solutions containing up to 12 per cent acetone was measured. Increments of one weight per cent were used.

Calibration of Solvent and Feed Orifices. The solvent and feed orifices were calibrated for flow expressed in liters per second. The time for one

liter of liquid to flow through the orifices was measured with an electric timer, for quantities of flow corresponding to pressure differentials indicated by the manometers from 0.2 to 5.0 inches of mercury.

Operational Procedure. In preparing the column for operation it was necessary to prepare the feed and solvent, and to adjust the equipment to the desired pulse frequency and pulse amplitude.

Preparation of Feed. The initial feed was prepared at the desired concentration by manual mixing of known weights of acetone and toluene directly in the feed tank. Subsequent feeds were prepared by using the raffinate from an extraction test, determining its acetone concentration by means of its refractive index, and raising or lowering it to the desired acetone concentration by adding acetone or toluene, respectively.

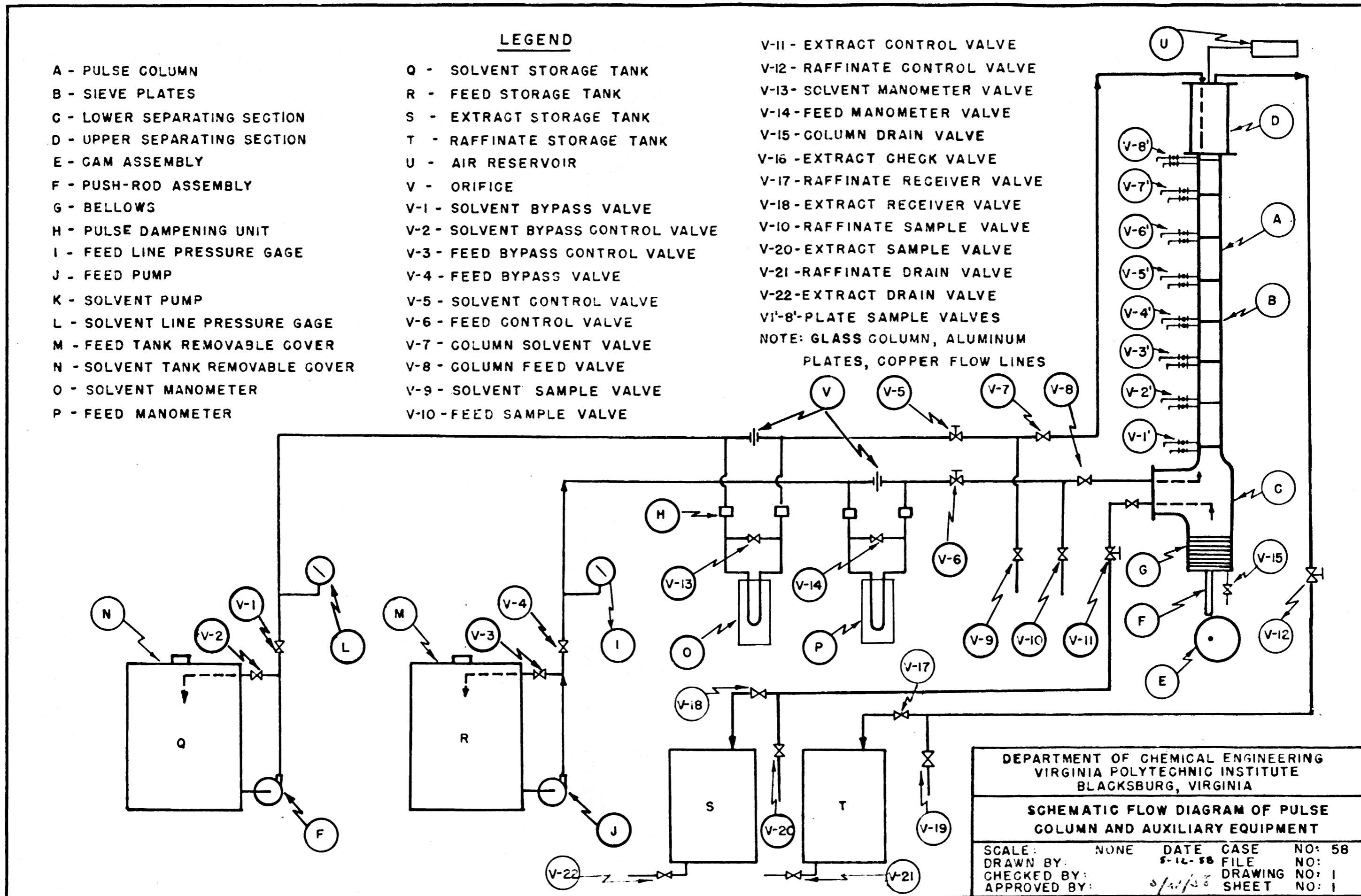
Pulse Frequency. The pulse frequency of the column was a function of the rotational speed of the motor-driven cam. The speed of the cam could be varied by changing the pulley

diameter ratio for the belt drive between the motor and the reduction-gear box driving the cam. For this investigation a two-inch pulley was used on the electric motor shaft, and a 7-1/2-inch pulley was used on the input shaft to the gear box. This arrangement resulted in a pulse frequency of 5.3 cycles per minute.

Pulse Amplitude. The pulse amplitude used in this investigation was that distance traversed by the liquid during one stroke in the two-inch diameter section of the column, and was a function of the distance of the lower end of the push rod to the circumference of the cam.

All references to equipment and valves in the discussions that follow refer to Drawing 1, page 42. To adjust the pulse amplitude it was necessary to partially fill the column with liquid. All valves were closed except valves V-1, V-2, V-5, V-7, V-12, and V-13. Pump K was put into operation and valve V-2 was gradually closed until the pressure in the solvent line as indicated by gage L was about 30 pounds per square inch, gage. The solvent

Drawing 1. Schematic Flow Diagram of Pulse  
Column and Auxiliary Equipment



water filled the column to the midpoint of the tee at which time the pump was stopped, and valve V-2 completely opened and valve V-7 closed. Valves V-3, V-4, V-6, V-8, and V-14 were then opened and pump J put into operation. Valve V-3 was closed until the line pressure as indicated by gage I was about 30 pounds per square inch, gage. The toluene feed was admitted to the column until the level of the toluene was between the top two sieve plates. At this time pump J was stopped, valve V-3 opened, and valve V-8 closed. The pulse motor was then started.

Since the pulse amplitude was dependent on the distance the push rod moved, it was necessary to vary this distance in order to vary the pulse amplitude. Lowering the push rod by turning the adjusting nut counterclockwise caused the cam to contact the push rod sooner than it did in the previous position, thus increasing the displacement of the push rod and the magnitude of the pulse amplitude. The pulse amplitude could be increased by turning the adjusting nut clockwise causing the push rod to move away from

the cam. After adjusting the pulse amplitude the pulse motor was stopped.

Column Operation. With the column partially filled with solvent and feed (see Pulse Amplitude, page 41), column operation was continued.

Valve V-8 was opened, pump J put into operation, and valve V-3 adjusted until the pressure in the feed line was about 30 pounds per square inch, gage. When the feed had begun to enter the top disengaging section, solvent was admitted by opening valve V-7, putting pump K into operation, and adjusting valve V-2 until the pressure in the solvent line was about 30 pounds per square inch, gage. When the interface between the light and heavy phases in the top disengaging section had raised to two inches below the raffinate outlet, the pulse motor was started, causing the heavy phase to move down the column. When the heavy phase began entering the bottom disengaging section, valve V-11 was opened slightly allowing the heavy phase to flow out of the column to maintain the position of the interface constant.

During the operation of the column two distinct effects were noted which were referred to as blowing and dumping. Blowing was the term applied to the condition of operation in which the light phase continued to flow upward through the column during the pause between pulses. Dumping was the term applied to the condition of operation in which the heavy phase continued to flow downward through the column during the pause between pulses. Calm operation was the term used when neither blowing nor dumping was taking place.

For operation of the column while blowing was occurring, the only way to keep the level of the interface in the top disengaging section constant was to control the heavy phase input by adjusting valve V-5, and to keep it constant by the proper valve setting. The interface in the bottom disengaging section could be kept constant, for a constant light phase flow rate, by controlling the heavy phase flow rate out of the column using valve V-11. If the light phase flow rate were changed, a new position of valve V-11 would be required to maintain the interface at the same level.

For operation of the column while dumping was occurring, the position of the interface in the top disengaging section, for a constant heavy phase flow rate, was kept constant by adjusting valve V-11 to control the heavy phase leaving the bottom disengaging section. The position of the interface in the bottom disengaging section, for a particular heavy phase flow rate, was held constant by controlling the light phase flow rate into the column by using valve V-6.

For operation of the column when neither dumping nor blowing was taking place, the position of the interface in the top disengaging section was held constant by controlling the heavy phase input rate with the proper setting of valve V-5. It was necessary to adjust valves V-6 and V-11 in order to both prevent dumping or blowing, and to keep the position of the interface in the bottom disengaging section constant.

#### Column Operating Characteristics at Steady State.

Physical steady state, for this investigation, was defined as the condition at which the positions of the light and heavy phase interfaces in the top and



bottom disengaging sections remained constant with time. It has been noted in the preceding section that the position of these interfaces was a function of the light and heavy phase flow rates. For this reason it was decided to plot the two flow rates against each other at values for which the positions of the two interfaces could be maintained constant, in order to graphically illustrate the operating characteristics.

To determine the operating characteristics, the feed (approximately ten per cent acetone in toluene) and solvent were prepared, the pulse amplitude was adjusted to one inch, and the column was put into operation as described in the preceding sections. The heavy phase flow rate was set at  $2.26 \times 10^{-3}$  liters per second, and the light phase flow rate was varied until the position of the two interfaces remained constant with time. When the position of the two interfaces became constant and remained constant for a period of one-half an hour, the manometer readings were noted and the column operation was temporarily stopped to measure the liquid flow rates. Two 2-1/2-gallon buckets were placed under sample

valves V-9 and V-10 and then, with two persons operating, valves V-9 and V-10 were opened, and valves V-7, V-8, and V-11 were closed as quickly as possible. The manometer readings, which had increased because of the removal of column resistance to flow, were then readjusted by means of valves V-5 and V-7 to the previously noted values. One thousand-milliliter graduated cylinders were placed in the buckets under the sample valves, and the time for a quantity of 1000 milliliters of solution to flow was measured by an electric timer and recorded. Before the column operation was interrupted to measure the flow rates, the operating condition with respect to the occurrence of dumping, blowing, or the absence of dumping and blowing (calm operation) was noted and recorded.

When one point had thus been determined, the heavy phase flow rate was increased by adjusting control valve V-5 to  $3.58 \times 10^{-3}$  liters per second, the light phase flow rate was varied until the positions of the two interfaces remained constant, and the above procedure was followed in measuring the flow rates. This procedure was continued until,

at very high light phase flow rates, the positions of the two interfaces could no longer be held constant. The column operating characteristics were then determined for pulse amplitudes of two and three inches.

Operation of the Column for Extraction. It was desired to determine the effect of three variables on the overall mass transfer coefficient. The variables and their magnitudes were pulse amplitude, one, two, and three inches; feed concentration, six, ten, and fifteen weight per cent acetone in toluene; and solvent-to-feed ratio, 0.50, 0.75, 1.00, 1.25, and 1.50. To accomplish this, forty-five tests were made, and with duplicates, there were ninety tests in all.

To establish the correct flow rates to use with the chosen solvent-to-feed ratios in order that physical steady state might be attained, the column characteristic curves in Figure 5 , page 62, were redrawn with the points omitted. Lines of constant solvent-to-feed ratio were drawn on these curves as shown in Figure 6, page 63. The intersection of these five, constant solvent-to-feed lines with the three

column characteristic curves of one-, two-, and three-inch pulse amplitudes gave fifteen points as the basis for the flow rates used.

The column was put into operation as described in Operational Procedure, page 40, with a feed concentration of ten weight per cent acetone in toluene and a pulse amplitude of one inch. The correct flow rates for a solvent-to-feed ratio of 0.50 were set by adjusting the control valves V-5 and V-7 until the corresponding pressure differentials as taken from the orifice calibration curves in Figures 3 and 4, pages 58 and 60, were indicated by the mercury manometers.

Preliminary tests were made to determine the time for the column to approach approximate steady state equilibrium conditions. This time was found to be about ten or fifteen minutes. Therefore, the column was assumed to be operating at steady state equilibrium conditions after fifteen minutes of operation, at which time the temperature of the solvent and feed in the recirculating lines in the tanks was measured and recorded, and samples of the feed, raffinate, and extract were collected. The column

was allowed to operate at the same conditions for ten minutes, at which time the feed and solvent temperatures were measured and duplicate samples of the feed, raffinate, and extract were collected.

The above procedure was followed for solvent-to-feed ratios of 0.50, 0.75, 1.00, 1.25, and 1.50.

The above procedures were followed in collecting operational feed, raffinate, and extract samples, with duplicates, at the chosen solvent-to-feed ratios for pulse amplitudes of one, two, and three inches. This procedure was repeated using feed concentrations of approximately six and fifteen weight per cent acetone in toluene.

Column Shut-down. As soon as a series of tests was completed the column operation was stopped and the column drained. The pumps J and K were stopped, valves V-2, V-3, V-13, V-14, and V-19 were opened and valves V-5, V-6, V-7, and V-8 were closed. Valve V-11 was opened completely, the heavy phase being drained into the extract drum, and the pulsations continued until the light and heavy phase separated to form two layers in the column. When the toluene-water interface had dropped to a point one-half an inch above the

extract exit line, valve V-18 was closed and the remainder of the light and heavy phase in the column was drained into a 2-1/2-gallon bucket by opening valve V-15.

### Data and Results

The data and results obtained from this investigation are presented in the following sections.

Specific Gravity Data. The data obtained from the specific gravity of solutions, as determined by the Westphal balance are presented in Table I, page 54, and graphically presented in Figures 1 and 2, pages 55 and 56.

Orifice Calibration Data. The calibration data for the orifices in the feed and solvent lines are presented in Tables II and III, pages 57 and 59. These data are shown in Figures 3 and 4, pages 58 and 60, respectively.

Column Operating Characteristics. The data obtained during operation of the column at pulse amplitudes of one, two, and three inches, and at a pulse frequency of 5.3 cycles per minute, for the determination of the column operating characteristics

are presented in Table IV, page 61. These data are shown graphically in Figure 5, page 62.

Operational Data and Sample Analyses. The operational data and analyses of samples taken during the course of operation of the column for extraction are presented in Tables V, VI, and VII, pages 64, 65, and 66, for feed concentrations of approximately six, ten, and fifteen weight per cent acetone in toluene, respectively.

Results Obtained. The results obtained from the calculation of the overall mass transfer coefficient are presented in Table VIII, page 67. Figures 7, 8, and 9, pages 68, 69, and 70, show the overall mass transfer coefficient plotted against the solvent-to-feed ratio for the three feed concentrations in three sets of curves, each set being for one particular pulse amplitude. Figure 10, page 71, shows the overall mass transfer coefficient plotted against the feed concentration for the three pulse amplitudes in five sets, each set being for one particular solvent-to-feed ratio.

TABLE I

Specific Gravity Data for the System  
Acetone-Toluene-Water at 25 °C

Toluene Phase		Water Phase	
Acetone Concentration	Specific Gravity	Acetone Concentration	Specific Gravity
wt per cent		wt per cent	
0.93	0.8634	1.05	0.9933
2.04	0.8631	2.00	0.9920
3.07	0.8614	2.90	0.9900
4.04	0.8610	3.95	0.9890
5.00	0.8604	4.84	0.9872
6.10	0.8591	5.83	0.9863
7.13	0.8586	6.91	0.9844
8.87	0.8572	8.06	0.9831
9.83	0.8560	9.20	0.9822
11.42	0.8549		



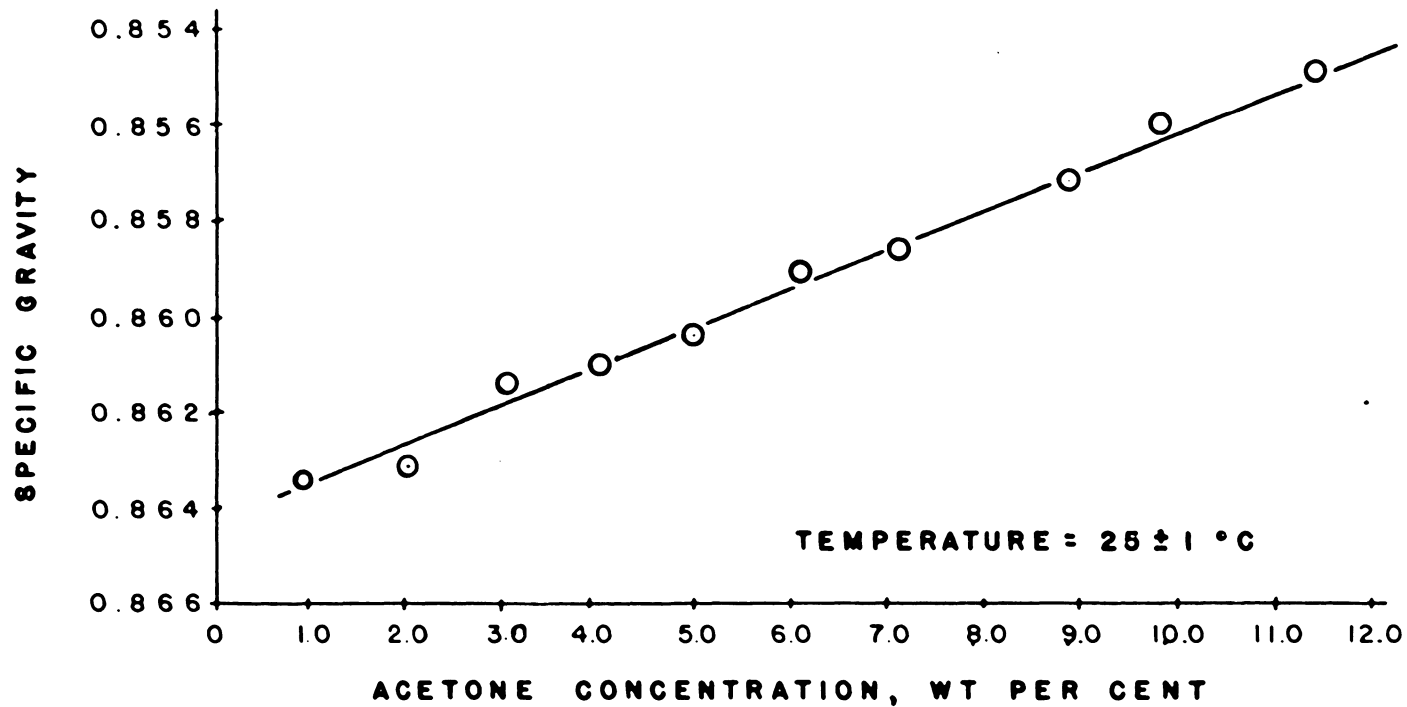


FIGURE I. RELATIONSHIP OF ACETONE  
CONCENTRATION TO THE SPECIFIC  
GRAVITY OF THE LIGHT PHASE

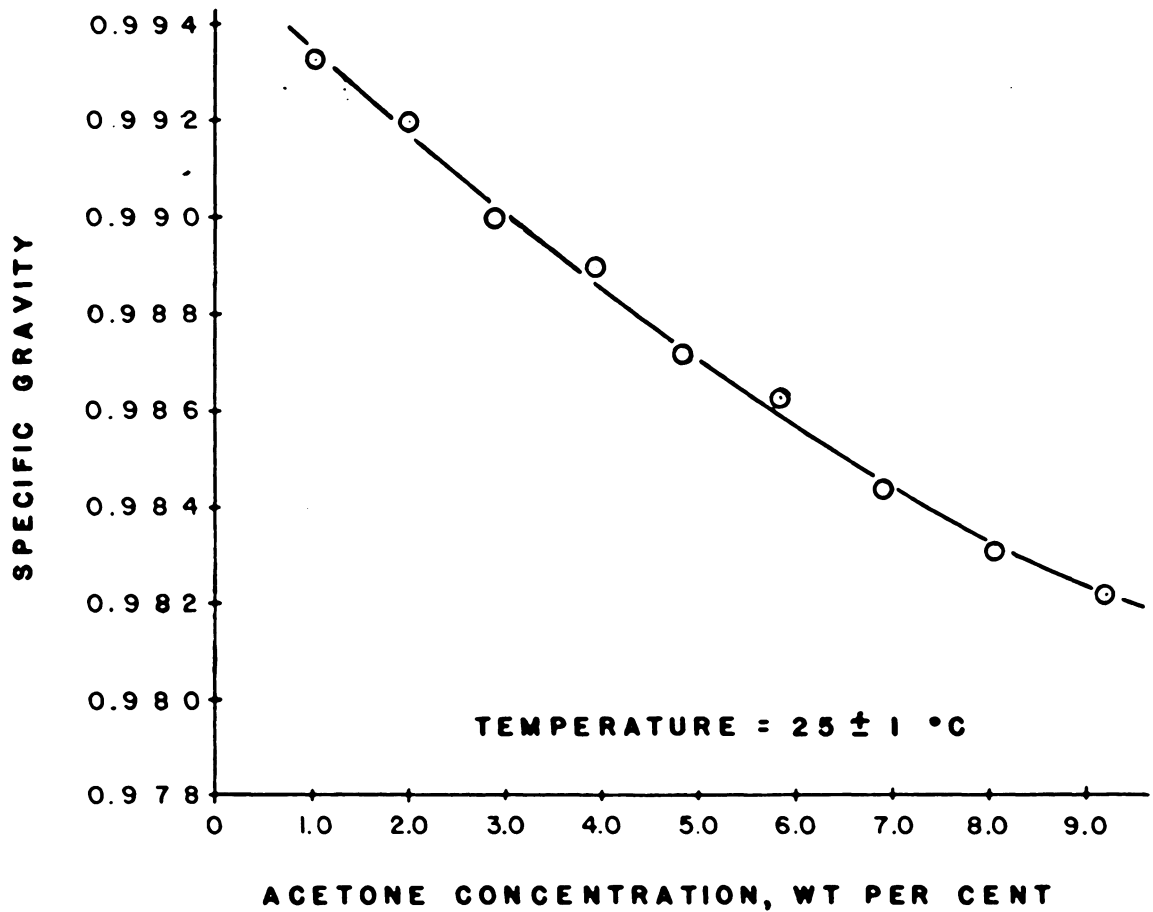


FIGURE 2. RELATIONSHIP OF ACETONE CONCENTRATION TO THE SPECIFIC GRAVITY OF THE HEAVY PHASE

TABLE II

Calibration Data for the Feed Orifice

Pressure Differential	Feed Flow Rate	
	in. of mercury	sec/l
0.2	186.5	5.36
0.4	132.0	7.58
0.6	103.0	9.26
0.8	90.4	11.08
1.0	82.4	12.13
1.5	68.8	14.56
2.0	60.8	16.47
2.5	54.5	18.36
3.0	44.6	20.81
4.0	43.6	22.90
5.0	39.0	25.65

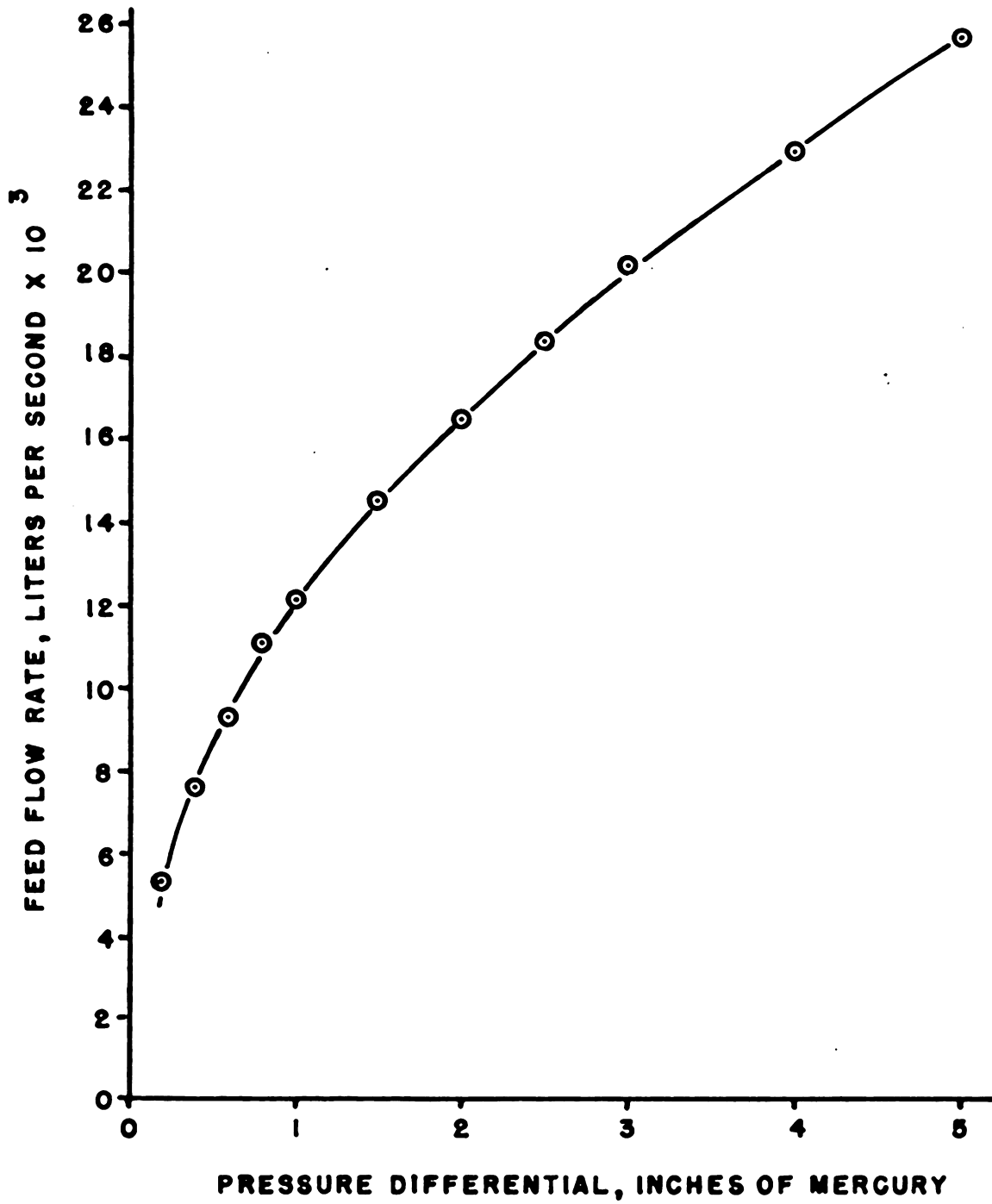


FIG. 3. CALIBRATION CURVE FOR THE FEED ORIFICE

TABLE III

Calibration Data for the Solvent Orifice

Pressure Differential in. of mercury	Solvent Flow Rate	
	sec/l	l/sec x 10 <sup>3</sup>
0.2	299.0	3.34
0.4	216.8	4.61
0.6	173.6	5.76
0.8	145.0	6.96
1.0	133.2	7.50
1.5	109.8	9.11
2.0	95.6	10.46
2.5	87.2	11.48
3.0	78.4	12.76
4.0	67.6	14.80
5.0	61.5	16.27
6.0	56.5	17.71
7.0	52.0	19.22
8.0	48.7	20.50
9.0	45.8	21.87
10.0	43.4	23.15

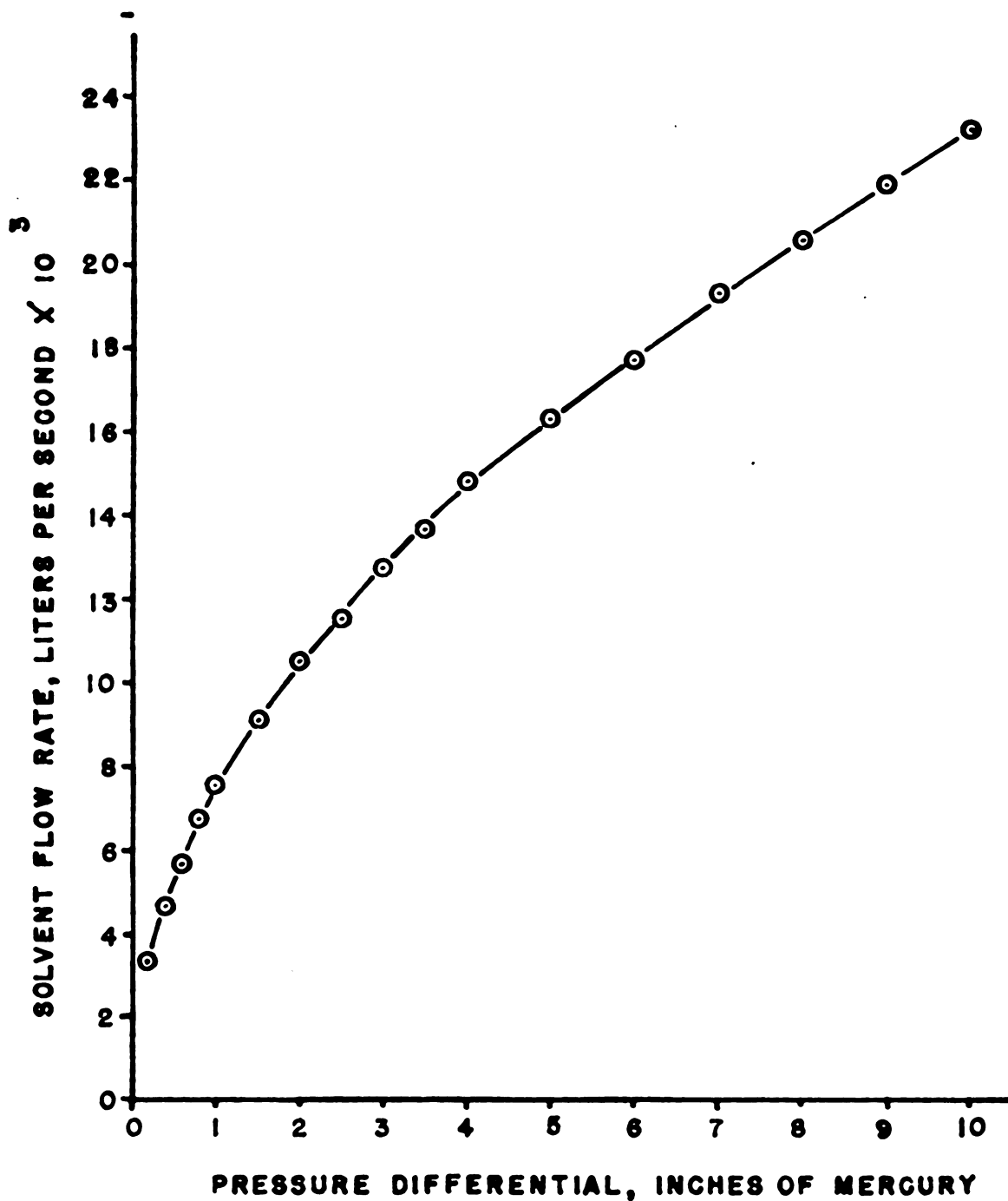


FIG. 4. CALIBRATION CURVE FOR THE SOLVENT ORIFICE

TABLE IV  
Operating Characteristics of the Pulse Column for  
Various Pulse Amplitudes and Liquid Flow Rates<sup>a</sup>

Pulse Amplitude  in.	Flow Rates		Observations
	Feed	Solvent	
	1/sec x 10 <sup>3</sup>		
1	2.85	19.80	Dumping
	3.06	16.60	Dumping
	3.44	12.30	Dumping
	4.11	7.72	Slight Dumping
	5.02	5.12	Slight Dumping
	6.12	4.13	Blowing
	7.19	4.20	Blowing
	9.45	3.58	Blowing
	13.00	2.26	Blowing
2	7.10	21.08	Dumping
	8.12	13.82	Dumping
	8.91	9.86	Dumping
	9.22	8.38	Calm Operation
	10.50	8.00	Blowing
	11.00	8.35	Blowing
	11.80	8.75	Blowing
	15.45	8.01	Blowing
	18.10	7.33	Blowing
	24.30	6.05	Blowing
3	11.40	21.40	Dumping
	12.40	16.40	Dumping
	12.53	14.18	Calm Operation
	13.50	14.00	Calm Operation
	13.64	13.34	Blowing
	15.01	12.90	Blowing
	16.00	12.50	Blowing
	18.50	12.80	Blowing
	19.95	12.82	Blowing
	24.50	11.50	Blowing
	25.70	10.90	Blowing

a. Operating Conditions:

Pulse Frequency = 5.3 cyc/min

Hole Size = 1/16 in.

Plate-Free Area = 23 per cent

Number of Plates = 8

Plate Spacing = 6 in.

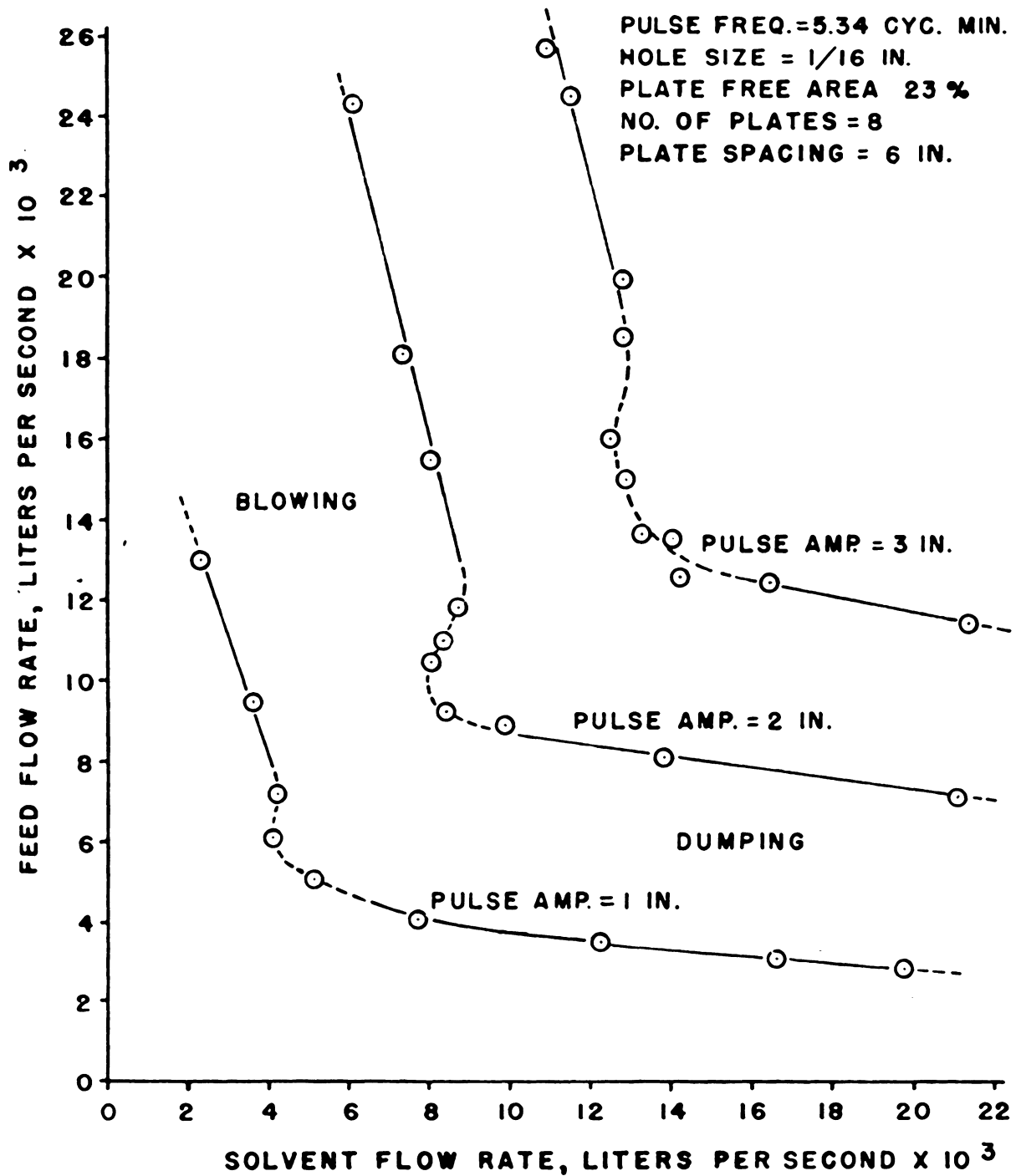


FIG. 5. OPERATING CHARACTERISTICS OF A PULSE COLUMN FOR VARIOUS PULSE AMPLITUDES AND LIQUID FLOW RATES



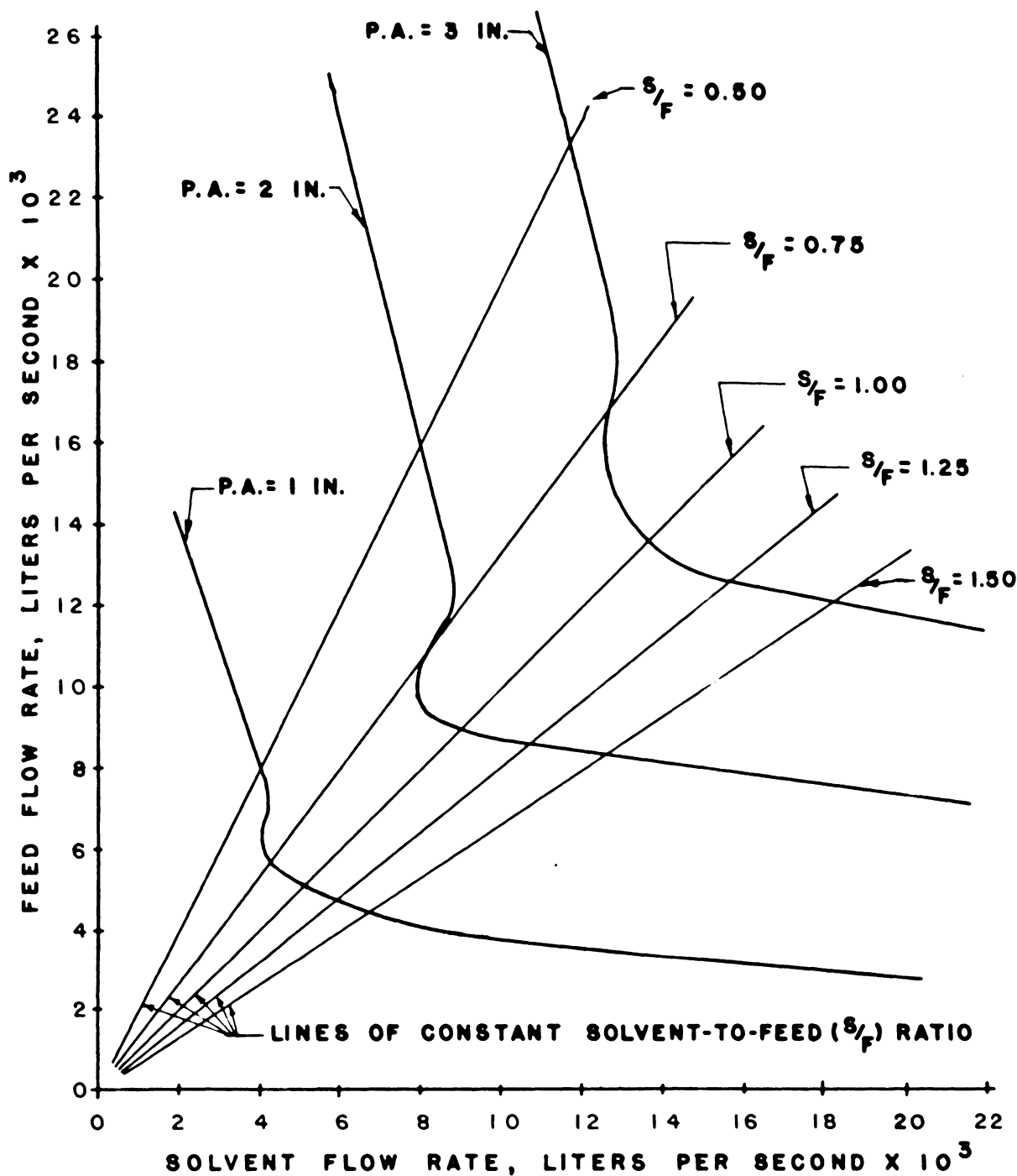


FIGURE 6. DETERMINATION OF OPERATING FLOW RATES FOR THE SELECTED SOLVENT-TO-FEED RATIOS

TABLE V

Operational Data and Sample Analyses from the  
Operation of a Two-inch Diameter Pulse  
Extraction Column Using a Feed of  
Approximately Six Weight Per  
Cent Acetone in Toluene

Test No	Phase <sup>b</sup> Sample	Pulse Amplitude in.	Volumetric Solvent-to-feed Ratio	Scale <sup>c</sup> Reading	Refractive Index <sup>d</sup>		Acetone Concentration wt per cent	Average Acetone Concentration wt per cent
					$n_D^{30}$			
1	F R E	1	0.50	18.43	1.48215	6.41	6.40	
				30.50	1.48666	3.33	3.38	
				20.87	1.33546	4.71	4.77	
1-a	F R E	1	0.50	18.40	1.48214	6.42		
				30.12	1.49651	3.43		
				21.08	1.33554	4.83		
2	F R E	1	0.75	-	-	-	6.40	
				31.98	1.48720	2.97	2.89	
				19.96	1.33511	4.25	4.23	
2-a	F R E	1	0.75	-	-	-		
				32.60	1.48743	2.81		
				19.86	1.33508	4.21		
3	F R E	1	1.00	18.44	1.48216	6.40	6.40	
				33.22	1.48766	2.66	2.59	
				19.40	1.33490	3.98	3.73	
3-a	F R E	1	1.00	-	-	-		
				33.79	1.48787	2.52		
				18.90	1.33454	3.49		
4	F R E	1	1.25	-	-	-	6.40	
				35.52	1.48851	2.08	2.05	
				17.54	1.33418	3.00	2.92	
4-a	F R E	1	1.25	-	-	-		
				35.77	1.48860	2.02		
				17.27	1.33407	2.85		
5	F R E	1	1.50	-	-	-	6.40	
				35.85	1.48863	1.99	2.00	
				17.14	1.33402	2.79	2.88	
5-a	F R E	1	1.50	18.45	1.48217	6.39		
				35.76	1.48860	2.02		
				17.46	1.33414	2.96		
6	F R E	2	0.50	18.50	1.48218	6.38	6.38	
				29.86	1.48642	3.49	3.51	
				21.36	1.33565	4.97	5.04	
6-a	F R E	2	0.50	-	-	-		
				29.73	1.48637	3.53		
				21.60	1.33574	5.10		
7	F R E	2	0.75	18.51	1.48218	6.38	6.38	
				32.00	1.48721	2.97	2.94	
				19.98	1.33512	4.26	4.22	
7-a	F R E	2	0.75	-	-	-		
				32.21	1.48729	2.91		
				19.85	1.33507	4.19		
8	F R E	2	1.00	18.51	1.48218	6.38	6.38	
				34.14	1.48800	2.42	2.47	
				18.35	1.33449	3.43	3.40	
8-a	F R E	2	1.00	-	-	-		
				33.77	1.48780	2.52		
				18.27	1.33446	3.88		
9	F R E	2	1.25	-	-	-	6.38	
				35.49	1.48850	2.08	2.09	
				16.91	1.33393	2.66	2.72	
9-a	F R E	2	1.25	-	-	-		
				35.44	1.48848	2.10		
				17.12	1.33401	2.78		
10	F R E	2	1.50	-	-	-	6.38	
				36.15	1.48875	1.92	1.90	
				16.40	1.33374	2.40	2.40	
10-a	F R E	2	1.50	18.52	1.48219	6.37		
				36.30	1.48880	1.88		
				16.40	1.33374	2.40		
11	F R E	3	0.50	19.80	1.48267	6.07	6.07	
				29.76	1.48638	3.52	3.47	
				21.05	1.33553	4.82	4.78	
11-a	F R E	3	0.50	-	-	-		
				30.14	1.48652	3.42		
				20.92	1.33548	4.74		
12	F R E	3	0.75	-	-	-	6.07	
				32.64	1.48745	2.79	2.83	
				18.85	1.33468	3.68	3.68	
12-a	F R E	3	0.75	-	-	-		
				32.36	1.48734	2.87		
				18.85	1.33468	3.68		
13	F R E	3	1.00	-	-	-	6.07	
				34.37	1.48809	2.36	2.37	
				17.28	1.33408	2.88	2.83	
13-a	F R E	3	1.00	19.80	1.48267	6.07		
				34.32	1.48807	2.38		
				17.37	1.33411	2.90		
14	F R E	3	1.25	-	-	-	6.07	
				35.63	1.48856	2.05	1.98	
				16.76	1.33388	2.60	2.60	
14-a	F R E	3	1.25	19.80	1.48267	6.07		
				36.10	1.48873	1.92		
				16.76	1.33388	2.60		
15	F R E	3	1.50	-	-	-	6.07	
				36.98	1.48905	1.68	1.72	
				16.14	1.33363	2.23	2.29	
15-a	F R E	3	1.50	19.80	1.48267	6.07		
				36.90	1.48902	1.73		
				16.30	1.33370	2.34		

<sup>a</sup> Feed and solvent streams entering the column were  $77 \pm 2$  °F for all data.

<sup>b</sup> F refers to feed, R refers to raffinate, and E refers to extract.

<sup>c</sup> All scale readings refer to Bausch and Lomb dipping refractometer prisms.

<sup>d</sup> Refractive indices of all extract samples were measured using the A prism.  
 Refractive indices of all feed and raffinate samples were measured using the E prism.

Operational Data<sup>a</sup> and Sample Analyses from the  
Operation of a Two-inch Diameter Pulse  
Extraction Column Using a Feed of  
Approximately Ten Weight Per  
Cent Acetone in Toluene

Test No	Phase <sup>b</sup> Sample	Pulse Amplitude in.	Volumetric Solvent-to-feed Ratio	Scale <sup>c</sup> Reading	Refractive Index <sup>d</sup> <sub><math>n_D^{30}</math></sub>	Acetone Concentration wt per cent	Average Acetone Concentration wt per cent
16	F R E	1	0.50	2.14	1.47599	10.47	10.51
				20.75	1.48302	5.83	5.74
				27.60	1.33804	8.39	8.36
16-a	F R E	1	0.50	1.83	1.47588	10.55	
				21.47	1.48329	5.65	
				27.50	1.33800	8.32	
17	F R E	1	0.75	1.64	1.47580	10.59	10.56
				24.65	1.48448	4.82	4.78
				25.03	1.33706	6.84	6.76
17-a	F R E	1	0.75	1.85	1.47588	10.53	
				24.93	1.48458	4.74	
				24.80	1.33697	6.68	
18	F R E	1	1.00	1.89	1.47589	10.54	10.54
				26.54	1.48518	4.34	4.01
				25.11	1.33709	6.88	6.55
18-a	F R E	1	1.00	1.88	1.47589	10.54	
				29.12	1.48614	3.69	
				24.02	1.33668	6.22	
19	F R E	1	1.25	1.74	1.47584	10.57	10.55
				29.74	1.48637	3.53	3.48
				21.46	1.33569	5.03	4.93
19-a	F R E	1	1.25	1.87	1.47589	10.53	
				30.16	1.48653	3.42	
				21.04	1.33553	4.83	
20	F R E	1	1.50	1.80	1.47586	10.57	10.53
				36.34	1.48882	1.86	2.01
				19.84	1.33507	4.19	4.21
20-a	F R E	1	1.50	2.09	1.47597	10.49	
				35.15	1.48838	2.17	
				19.92	1.33510	4.23	
21	F R E	2	0.50	1.10	1.47560	10.73	10.76
				20.57	1.48296	5.88	5.90
				29.98	1.33895	9.75	9.61
21-a	F R E	2	0.50	1.03	1.47551	10.79	
				20.40	1.49289	5.92	
				29.51	1.33877	9.47	
22	F R E	2	0.75	1.88	1.47589	10.53	10.53
				23.66	1.48411	5.08	4.96
				24.80	1.33697	6.68	6.66
22-a	F R E	2	0.75	1.86	1.47589	10.53	
				24.58	1.48445	4.84	
				24.70	1.33694	6.65	
23	F R E	2	1.00	1.14	1.47561	10.72	10.72
				28.28	1.48583	3.90	3.85
				24.46	1.33684	6.49	6.55
23-a	F R E	2	1.00	1.26	1.47566	10.73	
				28.66	1.48598	3.80	
				24.62	1.33691	6.60	
24	F R E	2	1.25	1.91	1.47591	10.53	10.53
				29.82	1.48640	3.52	3.33
				21.57	1.33573	5.08	4.90
24-a	F R E	2	1.25	1.90	1.47590	10.52	
				31.30	1.48695	3.14	
				20.87	1.33546	4.71	
25	F R E	2	1.50	1.25	1.47562	10.72	10.71
				34.33	1.48807	2.38	2.68
				20.21	1.33521	4.39	4.68
25-a	F R E	2	1.50	1.24	1.47565	10.70	
				31.98	1.48720	3.97	
				21.38	1.33566	4.98	
26	F R E	3	0.50	1.23	1.47565	10.70	10.73
				19.95	1.48272	6.04	6.08
				27.35	1.33827	8.28	8.48
26-a	F R E	3	0.50	1.04	1.47558	10.76	
				19.60	1.48259	6.12	
				28.18	1.33827	8.72	
27	F R E	3	0.75	1.76	1.47585	10.56	10.59
				25.12	1.48465	4.70	4.67
				25.12	1.33713	6.95	6.92
27-a	F R E	3	0.75	1.53	1.47576	10.62	
				25.30	1.48472	4.64	
				25.10	1.33709	6.90	
28	F R E	3	1.00	1.10	1.47560	10.74	10.74
				26.28	1.48509	4.39	4.28
				23.12	1.33633	5.90	5.79
28-a	F R E	3	1.00	1.07	1.47559	10.74	
				27.15	1.48542	4.18	
				22.72	1.33617	5.68	
29	F R E	3	1.25	1.58	1.47578	10.61	10.61
				30.67	1.48672	3.28	3.30
				20.94	1.33549	4.75	4.86
29-a	F R E	3	1.25	1.60	1.47579	10.61	
				30.50	1.48665	3.33	
				21.33	1.33564	4.97	
30	F R E	3	1.50	1.06	1.47588	10.74	10.75
				31.65	1.48708	3.05	3.13
				20.02	1.33514	4.30	4.34
30-a	F R E	3	1.50	1.00	1.47556	10.76	
				31.02	1.48685	3.21	
				20.20	1.33521	4.38	

<sup>a</sup> Feed and solvent streams entering the column were  $77 \pm 2$  OF for all data.

<sup>b</sup> F refers to feed, R refers to raffinate, and E refers to extract.

<sup>c</sup> All scale readings refer to Bausch and Lomb dipping refractometer prisms.

<sup>d</sup> Refractive indices of all extract samples were measured using the A prism.  
Refractive indices of all feed and raffinate samples were measured using the E prism.

TABLE VII

Operational Data<sup>a</sup> and Sample Analyses from the  
Operation of a Two-inch Diameter Pulse  
Extraction Column Using a Feed of  
Approximately 15 Weight Per  
Cent Acetone in Toluene

Test No	Phase <sup>b</sup> Sample	Pulse Amplitude	Volumetric Solvent-to- feed Ratio	Scale <sup>c</sup> Reading	Refractive Index <sup>d</sup>	Acetone Concentration	Average Acetone Concentration
31	F R E	1	0.50	85.16	1.46875	15.27	15.28
				11.35	1.47948	8.16	8.28
				33.52	1.34030	11.72	12.02
31-a	F R E	1	0.50	84.87	1.46870	15.29	
				10.38	1.47911	8.40	
				34.63	1.34072	12.32	
32	F R E	1	0.75	84.97	1.46874	15.27	15.27
				15.55	1.48016	7.12	6.95
				32.10	1.33976	10.92	10.81
32-a	F R E	1	0.75	85.03	1.46876	15.27	
				16.87	1.48516	6.79	
				31.70	1.33961	10.70	
33	F R E	1	1.00	85.13	1.46879	15.24	15.25
				18.83	1.48231	6.30	6.25
				29.64	1.33882	9.55	9.42
33-a	F R E	1	1.00	85.00	1.46875	15.26	
				19.23	1.48246	6.21	
				29.20	1.33866	9.30	
34	F R E	1	1.25	85.03	1.46876	15.27	15.27
				26.67	1.48523	4.30	4.41
				23.59	1.33651	6.14	6.15
34-a	F R E	1	1.25	85.03	1.46876	15.27	
				25.80	1.48491	4.52	
				23.64	1.33653	6.16	
35	F R E	1	1.50	85.03	1.46876	15.27	15.27
				26.03	1.48499	4.47	4.52
				21.70	1.33578	5.14	5.33
35-a	F R E	1	1.50	85.00	1.46875	15.26	
				25.54	1.48481	4.58	
				29.38	1.33604	5.52	
36	F R E	2	0.50	84.65	1.46863	15.34	15.24
				10.33	1.47910	8.40	8.48
				35.10	1.34090	12.58	12.68
36-a	F R E	2	0.50	85.20	1.46882	15.22	
				9.72	1.47887	8.57	
				35.48	1.34104	12.78	
37	F R E	2	0.75	-	-	-	15.24
				16.54	1.48413	6.88	6.77
				31.56	1.33955	10.61	10.53
37-a	F R E	2	0.75	-	-	-	
				17.36	1.48175	6.67	
				31.23	1.33943	11.45	
38	F R E	2	1.00	85.58	1.46895	15.14	15.24
				20.05	1.48276	6.02	5.86
				28.41	1.33836	8.85	8.74
38-a	F R E	2	1.00	-	-	-	
				21.25	1.48321	5.71	
				28.02	1.33821	8.64	
39	F R E	2	1.25	-	-	-	15.24
				24.52	1.48443	4.85	4.80
				25.70	1.33732	7.25	7.01
39-a	F R E	2	1.25	-	-	-	
				24.93	1.48458	4.75	
				24.90	1.33701	6.77	
40	F R E	2	1.50	85.11	1.46879	15.24	15.24
				26.11	1.48503	4.44	4.28
				24.30	1.33678	6.38	6.23
40-a	F R E	2	1.50	-	-	-	
				27.40	1.48551	4.12	
				23.80	1.33659	6.08	
41	F R E	3	0.50	87.43	1.46959	14.70	14.70
				10.66	1.47922	8.33	8.38
				34.10	1.34052	12.02	12.21
41-a	F R E	3	0.50	-	-	-	
				10.23	1.47906	8.43	
				34.80	1.34018	12.40	
42	F R E	3	0.75	-	-	-	14.70
				18.35	1.48212	6.43	6.35
				31.06	1.33936	10.33	10.20
42-a	F R E	3	0.75	-	-	-	
				19.00	1.48237	6.27	
				30.55	1.33917	10.07	
43	F R E	3	1.00	87.55	1.46963	14.69	14.70
				19.93	1.48271	6.03	5.66
				28.17	1.33826	8.70	8.52
43-a	F R E	3	1.00	-	-	-	
				22.79	1.48374	5.30	
				27.55	1.33802	8.35	
44	F R E	3	1.25	-	-	-	14.70
				24.76	1.48452	4.79	4.75
				25.80	1.33735	7.30	7.31
44-a	F R E	3	1.25	-	-	-	
				25.02	1.48462	4.72	
				25.85	1.33737	7.32	
45	F R E	3	1.50	87.60	1.46964	14.68	14.70
				28.95	1.48608	3.72	3.73
				23.12	1.33633	5.90	6.00
45-a	F R E	3	1.50	-	-	-	
				28.88	1.48606	3.74	
				23.35	1.33642	6.02	

<sup>a</sup> Feed and solvent streams entering the column were  $77 \pm 2$  °F for all data.

<sup>b</sup> F refers to feed, R refers to raffinate, and E refers to extract.

<sup>c</sup> All scale readings refer to Bausch and Lomb dipping refractometer prisms.

<sup>d</sup> Refractive indices of all extract samples were measured using the A prism. Refractive indices of all feed samples were measured using the D prism. Refractive indices of all raffinate samples were measured using the E prism.

TABLE VIII

Calculated Results for the Determination of the Overall  
Mass Transfer Coefficient for the Extraction of  
Acetone from Toluene Using Water as the  
Extracting Solvent in a Two-inch  
Diameter Pulse Extractor

(See table in pocket, back cover)

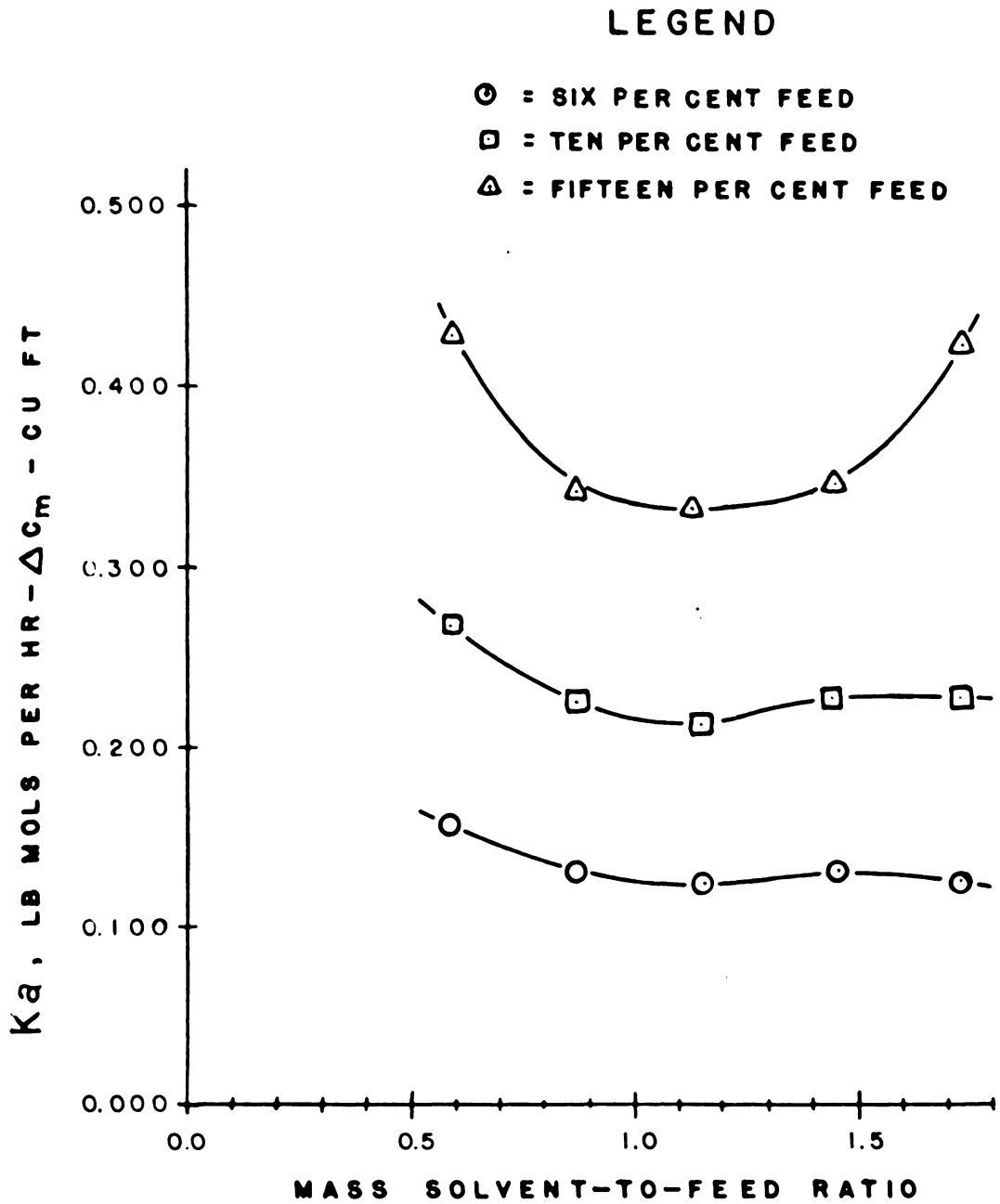


FIGURE 7. VARIATION OF THE OVERALL MASS TRANSFER COEFFICIENT WITH SOLVENT-TO-FEED RATIO USING A ONE-INCH PULSE AMPLITUDE IN A TWO-INCH DIAMETER PULSE COLUMN

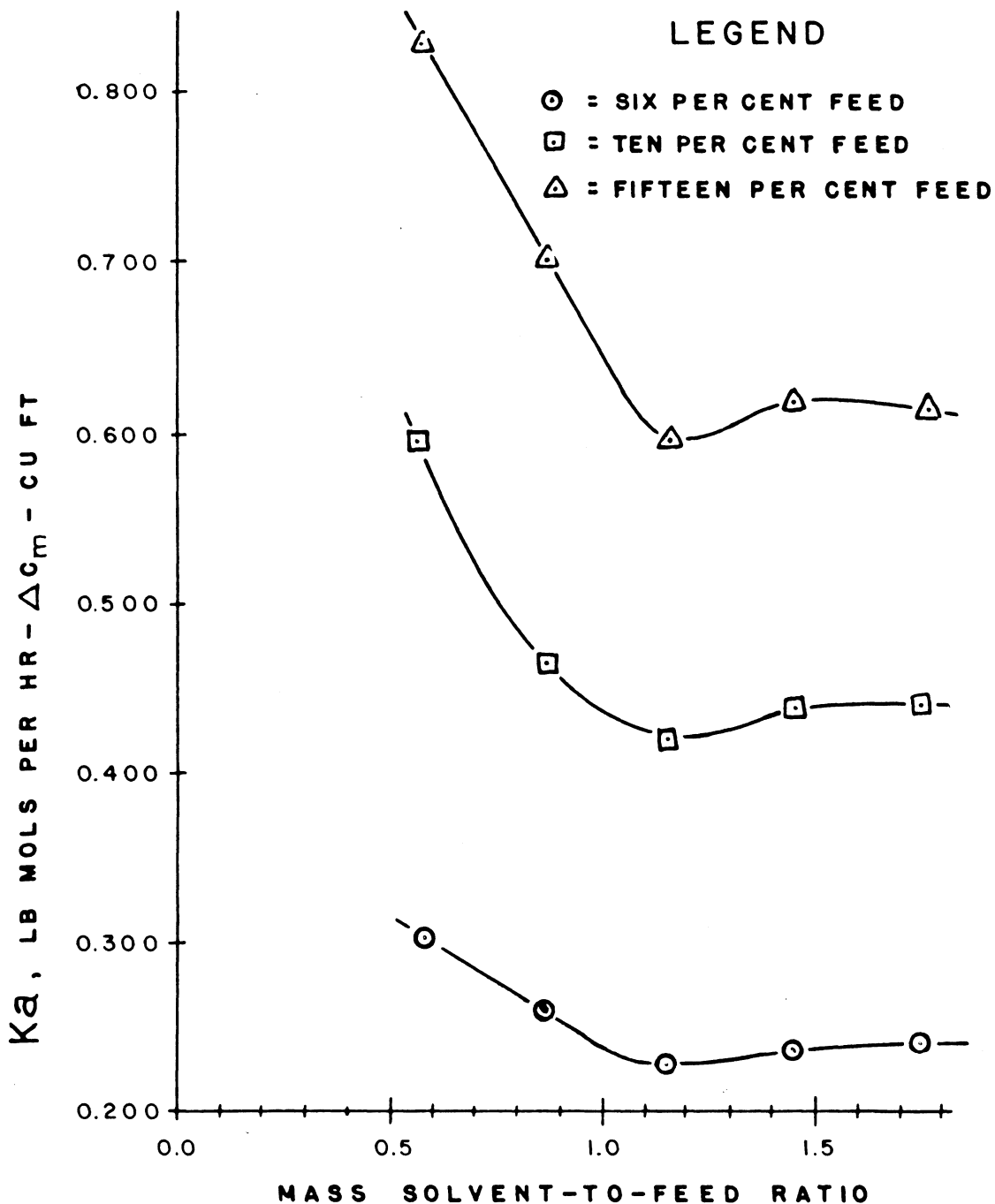


FIGURE 8. VARIATION OF THE OVERALL MASS TRANSFER COEFFICIENT WITH SOLVENT-TO-FEED RATIO USING A TWO-INCH PULSE AMPLITUDE IN A TWO-INCH DIAMETER PULSE COLUMN

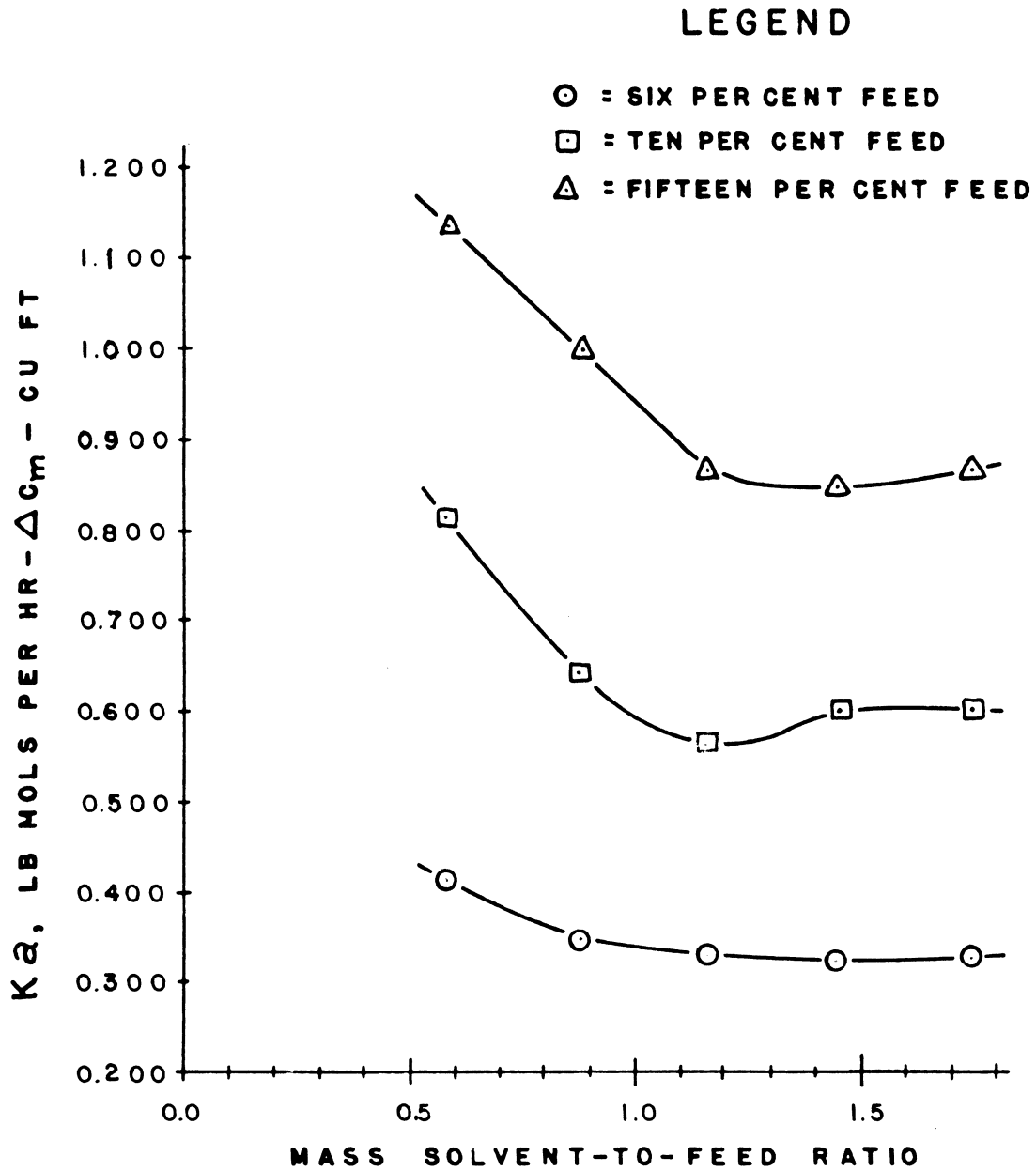
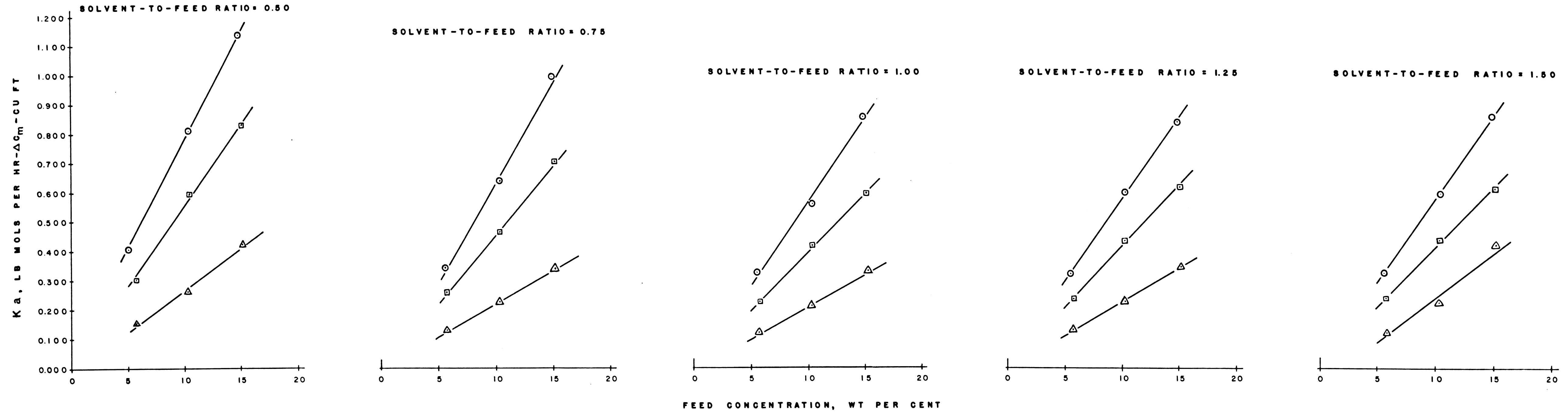


FIGURE 9. VARIATION OF THE OVERALL MASS TRANSFER COEFFICIENT WITH SOLVENT-TO-FEED RATIO USING A THREE INCH PULSE AMPLITUDE IN A TWO-INCH DIAMETER PULSE COLUMN





**LEGEND**  
 Δ = 1-INCH PULSE AMPLITUDE  
 □ = 2-INCH PULSE AMPLITUDE  
 ○ = 3-INCH PULSE AMPLITUDE

FIGURE 10. VARIATION OF THE OVERALL MASS TRANSFER COEFFICIENT WITH FEED CONCENTRATION IN A TWO-INCH DIAMETER PULSE EXTRACTION COLUMN

Figure 10. Variation of the Overall Mass Transfer Coefficient with Feed Concentration in a Two-inch Diameter Pulse Extraction Column

Sample Calculations

The calculations used to determine the results in this investigation are illustrated in the following sections.

Conversion of Volumetric to Mass Flow Rate. The following equation was used to convert volumetric flow rates to mass flow rates. Data are for Test 1, at a pulse amplitude of one inch, a feed concentration of approximately six weight per cent acetone in toluene, and a solvent-to-feed ratio of 0.50, and are taken from Figure 6, page 63, and Figure 1, page 55.

$$L = k(F_v)(S)$$

where:

- L = mass flow rate, lb mol/hr-sq ft
  - $F_v$  = volumetric flow rate, liter/sec
  - S = specific gravity
  - k = constant =  $3.636 \times 10^5$  lb-sec/hr-sq ft-liter
- $$L = (3.636 \times 10^5)(0.00801)(0.8590)$$
- $$L = 2502 \text{ lb mol/hr-sq ft.}$$

Amount of Acetone Transferred. The amount of acetone transferred in the extraction of acetone from toluene using distilled water as the extracting solvent was calculated by the following equation. Data are from Test 1, Table VIII, page 67.

$$Q = \frac{(L_{LP})(A)(y_F - y_R)}{(M_A)(1 - y_R)}$$

where:

- Q = amount of acetone transferred, lb mol/hr
- $L_{LP}$  = feed flow rate, lb mol/hr-sq ft
- A = cross sectional area of column, sq ft
- $y_F$  = acetone concentration in feed, wt fraction
- $y_R$  = acetone concentration in raffinate, wt fraction
- $M_A$  = molecular weight of acetone, lb/lb mol

$$Q = \frac{(2502)(0.218)(0.0640 - 0.0338)}{(58.05)(1 - 0.0338)}$$

$$Q = 0.0283 \text{ lb mol/hr.}$$

Overall Log Mean Average Concentration

Difference. The following equation was used to calculate the overall log mean concentration difference of the non-diffusing component, toluene. Data are from Test 1, Table VIII, page 67.

$$(1 - y)_m = \frac{(1 - y^*) - (1 - y)}{\ln \frac{1 - y^*}{1 - y}}$$

where:

- $(1 - y)_m$  = overall, log mean average concentration difference of toluene, mol fraction
- $(1 - y^*)$  = toluene concentration in raffinate at equilibrium with extract, mol fraction
- $(1 - y)$  = toluene concentration in raffinate, mol fraction

$$(1 - y)_m = \frac{(0.9359) - (0.9473)}{\ln \frac{0.9359}{0.9473}}$$

$$(1 - y)_m = 0.9500, \text{ mol fraction.}$$

Overall Mass Transfer Coefficient. The following equation was used to calculate the overall mass transfer coefficient from the amount of acetone transferred, the overall, log mean average toluene concentration difference, and the effective volume of the column. The effective volume of the column was taken as the volume between the interfaces in the top and bottom disengaging sections. Data are from Test 1, Table VIII, page 67.

$$K a = \frac{(Q)}{(1 - y)_m (V)}$$

where:

$K a$  = overall mass transfer coefficient,  
lb mol/hr- $\Delta C_m$ -V

$A$  = amount of acetone transferred,  
lb mol/hr

$(1 - y)_m$  = overall log mean average  
concentration difference of  
toluene, mol fraction

$V$  = effective volume of column  
= 0.195 cu ft

$$K a = \frac{(0.0293)}{(0.9500)(0.195)}$$

$K a$  = 0.158 lb mol/hr- $\Delta C_m$ -cu ft.

#### IV. DISCUSSION

The following sections contain a discussion of the results obtained during this investigation, recommendations for future work, and the limitations imposed upon the investigation.

##### Discussion of Results

The following sections contain a discussion of the results which includes a discussion of sample analysis, column operating characteristics, operation of the column for extraction of acetone from toluene using water, and the results obtained.

Analysis of Samples. All samples of the feed, raffinate, and extract that were taken during column operation were placed in 40-milliliter sample bottles and sealed with screw-type bakelite caps lined with aluminum foil. A test was made to determine the rate of acetone evaporation from a sample bottle sealed in this manner. For a particular feed sample it was found that the acetone concentration was reduced from 9.25 to 9.00 weight per cent acetone in toluene over a period of sixty-two hours. For a total drop in

concentration of 0.25 per cent, the drop per hour was 0.004 per cent. All samples were analyzed within six hours of column operation, and thus the error introduced by acetone evaporation from the sample bottles was only about 0.024 per cent.

All samples were analyzed by means of the Bausch and Lomb dipping refractometer at a temperature of  $30 \pm 0.1$  °C. The total range of the dipping refractometer was from 1.32000 to 1.54000 units of refractive index, and was divided into six overlapping ranges, each one requiring the use of a separate prism.

The samples were placed in a constant temperature bath to raise the temperature of the samples to the required value of  $30 \pm 0.1$  °C. The samples were then poured into a steel cup having a transparent bottom into which the prism, attached to the refractometer, was immersed. To minimize loss of acetone during the measurement of the refractive index the steel cup was tightly screwed onto the prism base.

Measurements made with the dipping refractometer could be repeated with an accuracy of  $\pm 0.0002$  unit of refractive index. This range corresponds to

$\pm 0.015$  weight per cent acetone in the toluene phase, and  $\pm 0.025$  weight per cent acetone in the water phase.

Column Operating Characteristics. Liquid flow rates which can be used in a pulse extraction column are dependent upon column dimensions and pulsing conditions. The pulsing conditions have a definite effect on the flow rates that can be used in that the magnitude of the flow rates is directly proportional to the pulse amplitude and to the pulse frequency. As the pulse amplitude is increased, the displacement of the liquid in the column increases and a larger volume of liquid is pulsed through the plates, increasing the flow rates. It is easily seen that a higher pulse frequency also increases the flow rates.

The use of various flow rates of the light and heavy phases produced certain operating characteristics which were referred to as dumping, blowing, calm operation (the absence of dumping and blowing), and a condition at which either or both of the interfaces in top and bottom disengaging sections could not be held constant. The condition at which either or both of the two interfaces continually increased because of an increasing volume of heavy phase was referred



to as heavy-phase flooding, while the condition at which either or both of the two interfaces continually decreased because of an increasing volume of light phase was referred to as light-phase flooding. Dumping was the term used to describe column operation under conditions at which the heavy phase continually passed through the plates down through the column during the pause between pulses. Blowing was the term used to describe the column operation under conditions at which the light phase continually passed up through the column during the pause between pulses.

The operating characteristics were determined using a system consisting of ten weight per cent acetone in toluene as the light phase, and distilled water as the heavy phase, at different light- and heavy-phase flow rates, pulse amplitudes of one, two, and three inches, and at a pulse frequency of 5.3 cycles per minute.

In making these tests it was found that as the heavy-phase flow rate was increased, the light-phase flow rate had to be decreased, and vice versa, in order to maintain constant interface levels in the

disengaging sections. This relationship is shown by the curves of Figure 5, page 62. Eventually, in both cases flow rate were reached beyond which flooding could not be prevented. This condition is represented by points at the extremities of the curves of Figure 5, page 62.

Dumping and blowing occurred at all flow rates represented by points on the curves except at a volumetric solvent-to-feed ratio of 1.00. At this point the volumetric flow rates were equal and consisted only of the displaced volume resulting from the pulse stroke at the particular pulse amplitude used. The magnitude of the total flow rate was the product of the pulse frequency and the pulse volume. When one of the flow rates was increased such that the volumetric solvent-to-feed ratio was no longer 1.00, the incremental liquid flow passed through the column as dumping or blowing, depending on which flow rate was increased. The total flow rate of the dumping or blowing phase, in this case, was the product of the pulse volume and pulse frequency plus the additional flow due to dumping or blowing. The total flow rate

of the other phase was still the product of the pulse frequency and the pulse volume.

The rates of dumping and blowing, although they could not be measured, could be varied by controlling either the light phase input using valve V-6 or the heavy phase output using valve V-11. A change in the light phase input rate or the heavy phase output rate caused the pressure of the light phase under the plates to change, allowing more or less of the respective phase to dump or blow through the column.

Significance of Operating Characteristic Curves.

Points on each of the characteristic curves, Figure 5, page 62, represent conditions at which the interface levels in both the top and bottom disengaging sections could be maintained constant. When operating at a point on any one curve, a change in either flow rate will produce a flooding condition. For operation on the curve under conditions of dumping (volumetric solvent-to-feed ratio greater than 1.00), an increase in the solvent flow rate will cause heavy-phase flooding in the top disengaging section by admitting more solvent into the top section than is being

permitted to dump down through the column, and a decrease in the solvent flow rate will cause light-phase flooding in the top disengaging section. An increase in the feed flow rate during dumping, while operating at a condition represented by a point on the curve, will cause heavy-phase flooding in the top disengaging section by permitting less dumping, and will cause light-phase flooding in the bottom disengaging section. A decrease in the feed flow rate during dumping, while operating at a condition represented by a point on the curve, will cause light-phase flooding in the top disengaging section by permitting more heavy phase to dump than is being admitted to the section, and will cause heavy-phase flooding in the bottom disengaging section as a result of more heavy phase entering the bottom section than can be drawn off by the heavy-phase outlet line. For operation on the curve under conditions of blowing (volumetric solvent-to-feed ratio less than 1.00), an increase in the solvent flow rate will cause heavy-phase flooding in the top disengaging section, while a decrease in the solvent flow rate will cause light-phase flooding. An increase in the feed flow rate

during blowing, while operating on the curve, produces no change in the levels of the interfaces since the increased flow is forced up the column as an increase in the blowing rate. A decrease in the feed flow rate during blowing, while operating on the curve, only reduces the amount of light phase moving up the column as blowing, and has no effect on the positions of the two interfaces.

As the solvent-to-feed ratio was changed from 1.00 for conditions of calm operation to less than 1.00 for conditions of blowing, certain changes in the flow pattern of the light phase were noted that seem to explain the shape (humps) in the column characteristic curves. As the rate of blowing increased, a cellular formation appeared in the layer of light phase liquid beneath the plates and in the top disengaging section that consisted of droplets of light phase coated with heavy phase. These droplets were about 1/4-inch in diameter, and a tiny drop of heavy phase could be seen clinging to the underside of each larger droplet. When this cellular formation first appeared, light-phase flooding occurred in the two disengaging sections

between the sieve plates. It is believed that the heavy phase coating the light phase droplets caused a heavy phase hold-up within the column which resulted in the light phase flooding. By increasing the heavy-phase flow rate the condition of light-phase flooding was eliminated. This increase in the heavy-phase flow rate resulted in the shifting of the characteristic curves toward the right at conditions of blowing.

The column operating characteristics determined in this investigation are valid only for the column studied. Any change in the physical characteristics of the column which would tend to affect the flow rates, or the flow pattern, will undoubtedly cause a change in the operating characteristic curves. The sieve plates used in this investigation consisted of 1/16-inch thick aluminum disks punched with round holes, 1/16-inch in diameter, spaced on triangular centers, and having a plate-free area of twenty-three per cent. In the event that an investigation be made to determine the effect of column diameter, or the hole size, shape, or plate-free area of the sieve

plates on extraction rates, new column operating characteristic curves would have to be determined.

Operation of the Column for Extraction. All column operations were carried out at  $25 \pm 1$  °C. It was generally necessary to increase the temperature of the feed and solvent before any tests were made. This was accomplished by mixing steam and cold water in the coils within the tanks so that hot water circulation was obtained. Once the required temperature was reached, the recycling of the solvent and feed provided any additional heat necessary to maintain the temperature at  $25 \pm 1$  °C. Cold water was circulated through the coils if the temperature exceeded that desired.

Since it was planned to determine the effect of the solvent-to-feed ratio on the extraction rate, accurate solvent and feed flow rates were required to give the desired solvent-to-feed ratio for conditions at which there was no flooding. This was accomplished by first redrawing the column characteristic curves of Figure 5, page 62, on rectangular coordinates on which the desired, constant, solvent-to-feed ratio lines had been drawn.

These curves are shown in Figure 6, page 63. The intersection of the constant solvent-to-feed ratio lines with the column characteristic curves gave points the coordinates of which were the required solvent and feed flow rates for the particular pulse amplitude and solvent-to-feed ratio used.

When the pulse column was being operated at the flow rates taken from Figure 6, page 63, a physical, steady state operation was attained in that there was no light- or heavy-phase flooding. This physical steady state condition was necessary in order to be able to approach steady state conditions between the two phases in the column. Preliminary extraction tests were made to determine the length of time required for the phases to reach steady state conditions. It was found that thirty minutes were required for the two phases to come to steady state after the initial filling of the column and column start-up. Steady state was defined as the condition at which the acetone concentration in the extract and raffinate phases was constant with time to within 0.3 weight per cent. Duplicate samples were taken



in each extraction test, the second set of samples being taken from ten to fifteen minutes after the first.

Results Obtained. The results obtained from the calculation of the overall mass transfer coefficients are presented in Table VIII, page 67. These results are graphically presented in two series of curves. The first series of curves, shown in Figures 7, 8, and 9, pages 68, 69, and 70, for pulse amplitudes of one, two, and three inches, respectively, show the effect of the mass solvent-to-feed ratio on the overall mass transfer coefficient for different feed concentrations. The second series of curves, shown in Figure 10, page 71, consisting of five sets of curves for volumetric solvent-to-feed ratios of 0.50, 0.75, 1.00, 1.25, and 1.50, respectively, show the effect of feed concentration on the overall mass transfer coefficient for different pulse amplitudes.

All nine curves shown in Figures 7, 8, and 9, pages 68, 69, and 70, show that the lowest mass transfer rates occurred at a volumetric solvent-to-feed ratio of 1.0 at which there was no blowing or dumping. The highest rates occurred at a volumetric solvent-to-feed ratio of 0.50 at which there was a high degree of

blowing. Mass transfer rates were slightly higher than the minimum when the column was operated at a volumetric solvent-to-feed ratio of 1.50 at which there was a high degree of dumping.

When the column was being operated at a volumetric solvent-to-feed ratio less than 1.00, corresponding to a mass solvent-to-feed ratio of about 1.15, the condition of blowing existed and the light phase continued to flow up the column during the pause between pulses. As the light phase flowed up the column it was repeatedly dispersed by the sieve plates into the heavy phase on the plates, and coalesced beneath the plates. Thus, not only was extraction taking place during the pulse stroke when the light and heavy phases were alternately dispersed into each other, but also during the pause between pulses as a result of blowing. At a volumetric solvent-to-feed ratio of 1.00, extraction took place only during the pulse stroke, since there was no dumping or blowing taking place. The mass transfer that took place as a result of blowing was in excess of that resulting from the pulse stroke alone, and caused the overall

mass transfer coefficient to be higher for the condition of blowing than for calm operation.

At volumetric solvent-to-feed ratios greater than 1.00 dumping occurred, and extraction took place during the pause between pulses as the heavy phase was dispersed down into the light phase by the sieve plates. This mass transfer that took place as a result of dumping was also in excess of that occurring in the absence of either dumping or blowing, and caused the overall mass transfer coefficient to be higher for the condition of dumping than for calm operation.

The reason for the overall mass transfer coefficients being so much higher for conditions of blowing than for conditions of dumping is believed to be a result of the fact that blowing occurred under pressure due to pumping, whereas dumping occurred due only to gravity and the head produced by the liquid in the column. The high pressure of the light phase caused a finer dispersion during blowing than was experienced during dumping, resulting in more surface area through which mass transfer could take place.

In all the curves of Figures 7, 8, and 9, pages 68, 69, and 70, the overall mass transfer coefficient increased as the feed concentration increased. The fact that the slope of the curves in the blowing region increased as the feed concentration increased indicates that higher driving forces increase the effect of blowing on the mass transfer rate. Since the driving force appears in the denominator of the equation for the overall mass transfer coefficient, the effect of increasing it by increasing the feed concentration should tend to lower the magnitude of the overall mass transfer coefficient. However, the overall mass transfer coefficient increased with an increase in the feed concentration, so it is evident that an increase in the driving force causes a proportionately greater increase in the rate of mass transfer.

The curves in Figure 10, page 71, show that the variation of the overall mass transfer coefficient with feed concentration is nearly a straight-line relationship. All but one of the curves, when extended to the left, passed approximately through zero, which gives some basis for the assumption of a

straight-line relationship. The curves show that an increase in pulse amplitude increased not only the mass transfer rate, but also the effect of feed concentration on the mass transfer rate. An increase in the feed concentration produced a greater increase in the extraction rate for a high pulse amplitude than for a lower one.

The amount of agitation between the two phases within the column is directly affected by the pulse amplitude, being greater for the higher amplitudes. The increased pulse amplitudes, by increasing the agitation between the two phases, increased the rate of extraction.

There are certain characteristics of the column itself that may have an effect on the rate of mass transfer. The shape of the holes in the plates, when varied, may produce dispersions of various shaped droplets which would tend to have different effects upon the extraction rate. The hole size would also affect the size of the droplets in the dispersion and consequently the interfacial area available for mass transfer. The distance between plates affects the time that one phase is dispersed in the other,

and would thus have a direct relationship with the amount of extraction taking place. In order to obtain a more complete evaluation of the pulse extraction column these characteristics should be investigated.

### Recommendations

As a result of this investigation the following recommendations are made for further studies on pulse extraction.

Column Operating Characteristics. The column operating characteristics determined in this investigation are valid only for the physical characteristics of the column used. It is recommended that operating characteristics be determined using columns having diameters of three and four inches, sieve plates having 1/32- and 1/8-inch diameter holes, sieve plates having square and rectangular shaped holes, plate spacings of four and sixteen inches, and combinations of four, eight, and sixteen plates with the suggested plate spacings, in order that extraction tests might be made using these variations in the column.

Extraction Tests. It is recommended that further tests be made to determine the effects of pulse frequency and other physical characteristics of the column on the overall mass transfer coefficient. These tests can be made after the above recommended operating characteristics are determined, using the suggested, column physical characteristics as variables.

Liquid-level Controller. In order to facilitate the determination of the column operating characteristics and the actual operation of the column itself, it is recommended that some form of liquid-level controller be installed on the column to automatically control the positions of the light and heavy phase interfaces in the top and bottom disengaging sections.

### Limitations

The limitations imposed upon this investigation are presented in the following sections.

System. The ternary system, acetone-toluene-water, was used in the extraction of acetone from toluene using distilled water in all phases of this investigation.

Temperature. The column was operated during extraction studies at a temperature of  $25 \pm 1$  °C. The equilibrium data for the system used was determined by Rood<sup>(18)</sup> at a temperature of  $25 \pm 0.2$  °C. All refractive index determinations were made at a temperature of  $30 \pm 0.1$  °C.

Pulse Column. A two-inch diameter glass column, consisting of seven 6-inch sections of glass pipe, each separated by two 1-inch thick plate spacers, and having a section of four-inch glass pipe at the top of the column and a 4 x 2 x 4-inch glass tee at the bottom of the column for use as disengaging sections, was employed in this investigation.

Sieve-plate Geometry. Eight perforated plates having 1/16-inch diameter holes on 1/8-inch



triangular centers, a plate-free area of twenty-three per cent, and a plate spacing of eight inches were used in this investigation.

Pulse Frequency. The pulse frequency used in this investigation was maintained constant at 5.3 cycles per minute.

Pulse Amplitude. The column was operated at pulse amplitudes of one, two, and three inches.

Feed Concentration. The feed concentrations used in this investigation were approximately six, ten, and fifteen weight per cent acetone in toluene.

Solvent-to-feed Ratio. The volumetric solvent-to-feed ratios that were used in this investigation were 0.50, 0.75, 1.00, 1.25, and 1.50.

## V. CONCLUSIONS

An investigation using the system acetone-toluene-water to determine the column operating characteristics of a pulse column, and the effect of solvent-to-feed ratio, pulse amplitude, and feed concentration on the overall mass transfer coefficient for the column was performed on a two-inch diameter glass column containing eight sieve plates, spaced eight inches apart, with 1/16-inch diameter holes on triangular centers, and having a plate-free area of twenty-three per cent. The temperature at which the column was operated was  $25 \pm 1$  °C. A constant pulse frequency of 5.3 cycles per minute was maintained throughout the investigation. The following variables were studied as to their effect of the overall mass transfer coefficient: feed concentrations of six, ten, and fifteen weight per cent acetone in toluene; pulse amplitudes of one, two, and three inches; and solvent-to-feed ratios of 0.50, 0.75, 1.00, 1.25, and 1.50. The results obtained from this investigation led to the following conclusions.

1. The column operating characteristics consist of curves, whose shape is independent of the pulse amplitude, that form the loci of the light- and heavy-phase flow rates at which the column can be operated without light- or heavy-phase flooding.

2. Conditions of blowing and dumping, corresponding to volumetric solvent-to-feed ratios less than 1.00 and greater than 1.00, respectively, resulted in the highest rates of mass transfer, the rates being higher for blowing than for dumping.

3. An increase in the feed concentration increases the overall mass transfer coefficient.

4. An increase in the pulse amplitude increases the overall mass transfer coefficient.

## VI. SUMMARY

It was the purpose of this investigation, using the ternary system acetone-toluene-water in the extraction of acetone from toluene using distilled water, to determine the operating characteristics of a pulse extraction column, and to determine the effects of solvent-to-feed ratio, feed concentration, and pulse amplitude on the overall mass transfer coefficient. The equipment used was operated at a temperature of  $25 \pm 1$  °C, and consisted of a two-inch diameter glass column with eight sieve plates having 1/16-inch diameter holes on triangular centers, and a plate-free area of twenty-three per cent. The plate spacing used was eight inches. Pulsation of the liquids in the column was accomplished by means of a brass bellows actuated by a push rod driven by a motor-driven cam.

The determination of the column operating characteristics resulted in curves defining the operating flow rates at which the column could be operated without light- or heavy-phase flooding. The shape of the curves was a function of the feed and

solvent flow rates, but was independent of the pulse amplitude.

Using flow rates taken from the column operating characteristic curves, a total of forty-five tests were made to determine the effect of pulse amplitude, feed concentration, and the solvent-to-feed ratio on the overall mass transfer coefficient. It was found that the overall mass transfer coefficient was the highest at a volumetric solvent-to-feed ratio of 0.50 for conditions of blowing, reached a minimum at a volumetric solvent-to-feed ratio of 1.00 at which there was no blowing or dumping, and increased slightly at a volumetric solvent-to-feed ratio of 1.50 at which dumping occurred. The overall mass transfer coefficient increased with increased feed concentration in a straight-line relationship. The overall mass transfer coefficient was also increased by an increase in the pulse amplitude.

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Addenda

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ABSTRACT

FACTORS AFFECTING THE EXTRACTION EFFICIENCY  
OF AN EXPERIMENTAL, LIQUID-LIQUID,  
SIEVE-PLATE, PULSE EXTRACTOR

by

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Thesis submitted to the Graduate Faculty of the  
Virginia Polytechnic Institute  
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## ABSTRACT

It was the purpose of this investigation, using the ternary system acetone-toluene-water in the extraction of acetone from toluene using distilled water, to determine the operating characteristics of a pulse extraction column, and to determine the effects of solvent-to-feed ratio, feed concentration, and pulse amplitude on the overall mass transfer coefficient. The equipment used was operated at a temperature of  $25 \pm 1$  °C, and consisted of a two-inch diameter glass column with eight sieve plates having 1/16-inch diameter holes on triangular centers, and a plate-free area of twenty-three per cent. The plate spacing used was eight inches. Pulsation of the liquids in the column was accomplished by means of a brass bellows actuated by a push rod driven by a motor-driven cam.

The determination of the column operating characteristics resulted in curves defining the operating flow rates at which the column could be operated without light- or heavy-phase flooding. The shape of the curves was a function of the feed and

solvent flow rates, but was independent of the pulse amplitude.

Using flow rates taken from the column operating characteristic curves, a total of forty-five tests were made to determine the effect of pulse amplitude, feed concentration, and the solvent-to-feed ratio on the overall mass transfer coefficient. It was found that the overall mass transfer coefficient was the highest at a volumetric solvent-to-feed ratio of 0.50 for conditions of blowing, reached a minimum at a volumetric solvent-to-feed ratio of 1.00 at which there was no blowing or dumping, and increased slightly at a volumetric solvent-to-feed ratio of 1.50 at which dumping occurred. The overall mass transfer coefficient increased with increased feed concentration in a straight-line relationship. The overall mass transfer coefficient was also increased by an increase in the pulse amplitude.

TABLE VIII

## Calculated Results for the Determination of the Overall

## Mass Transfer Coefficient for the Extraction of

## Acetone from Toluene Using Water as the

## Extracting Solvent in a Two-inch

## Diameter Pulse Extractor

Test No.	Acetone Concentration in Feed	Pulse Amplitude	Volumetric Solvent-to-feed Ratio	Entering Flow Rates		Mass Solvent-to-feed Ratio	Acetone Concentration in Raffinate	Acetone Concentration in Raffinate	Toluene Concentration in Raffinate	Acetone Concentration in Extract	Acetone Concentration in Raffinate at Equilibrium with Extract <sup>a</sup>	Acetone Concentration in Raffinate with Extract	Toluene Concentration in Raffinate with Extract	Log-mean Average Concentration Difference of Toluene in Raffinate	Amount of Acetone Transferred	Overall Mass Transfer Coefficient
	wt per cent	in.		lb/hr-sq ft	lb/hr-sq ft		wt fraction	mol fraction	mol fraction	wt fraction	wt fraction	mol fraction	mol fraction	mol fraction	mol fraction	lb mols/hr
1	6.40	1	0.50	2502	1477	0.590	0.0338	0.0526	0.9473	0.0477	0.0414	0.0641	0.9359	0.9500	0.0293	0.158
2	6.40	1	0.75	1782	1551	0.870	0.0289	0.0451	0.9549	0.0423	0.0366	0.0568	0.9432	0.9512	0.0242	0.130
3	6.40	1	1.00	1595	1804	1.131	0.0259	0.0405	0.9595	0.0373	0.0320	0.0498	0.9502	0.9588	0.0234	0.125
4	6.40	1	1.25	1471	2130	1.448	0.0205	0.0321	0.9679	0.0292	0.0252	0.0394	0.9606	0.9733	0.0246	0.130
5	6.40	1	1.50	1328	2383	1.729	0.0199	0.0312	0.9688	0.0279	0.0241	0.0377	0.9623	0.9701	0.0233	0.123
6	6.38	2	0.50	4972	2883	0.580	0.0351	0.0546	0.9454	0.0504	0.0439	0.0679	0.9321	0.9433	0.0555	0.302
7	6.38	2	0.75	3627	3138	0.865	0.0294	0.0458	0.9542	0.0422	0.0364	0.0566	0.9434	0.9558	0.0483	0.259
8	6.38	2	1.00	2818	3246	1.152	0.0247	0.0386	0.9614	0.0340	0.0293	0.0457	0.9543	0.9595	0.0424	0.227
9	6.38	2	1.25	2724	3936	1.445	0.0209	0.0328	0.9672	0.0272	0.0234	0.0366	0.9634	0.9744	0.0449	0.236
10	6.38	2	1.50	2630	4586	1.744	0.0190	0.0298	0.9702	0.0240	0.0207	0.0325	0.9675	0.9643	0.0451	0.240
11	6.07	3	0.50	7380	4288	0.581	0.0347	0.0540	0.9450	0.0478	0.0415	0.0643	0.9357	0.9490	0.0756	0.409
12	6.07	3	0.75	5166	4473	0.866	0.0283	0.0442	0.9558	0.0368	0.0317	0.0494	0.9506	0.9630	0.0647	0.345
13	6.07	3	1.00	4258	4911	1.153	0.0237	0.0371	0.9629	0.0289	0.0249	0.0390	0.9610	0.9500	0.0606	0.327
14	6.07	3	1.25	3913	5633	1.440	0.0198	0.0311	0.9689	0.0260	0.0225	0.0353	0.9647	0.9767	0.0613	0.322
15	6.07	3	1.50	3726	6463	1.735	0.0172	0.0270	0.9730	0.0229	0.0199	0.0312	0.9688	0.9767	0.0619	0.325
16	10.51	1	0.50	2490	1455	0.585	0.0574	0.0880	0.9120	0.0836	0.0747	0.1135	0.8865	0.9110	0.0474	0.267
17	10.53	1	0.75	1778	1550	0.872	0.0496	0.0766	0.9234	0.0666	0.0586	0.0901	0.9099	0.9000	0.0391	0.223
18	10.54	1	1.00	1559	1800	1.152	0.0401	0.0622	0.9378	0.0655	0.0568	0.0873	0.9127	0.9650	0.0398	0.212
19	10.53	1	1.25	1468	2114	1.439	0.0333	0.0518	0.9482	0.0490	0.0427	0.0661	0.9339	0.9530	0.0420	0.226
20	10.57	1	1.50	1374	2380	1.730	0.0201	0.0315	0.9685	0.0421	0.0364	0.0551	0.9449	1.0260	0.0452	0.226
21	10.76	2	0.50	5050	2865	0.568	0.0590	0.0904	0.9096	0.0961	0.0874	0.1318	0.8682	0.8800	0.1022	0.596
22	10.56	2	0.75	3620	3138	0.867	0.0478	0.0738	0.9262	0.0676	0.0595	0.0912	0.9088	0.9150	0.0830	0.465
23	10.72	2	1.00	2805	3240	1.154	0.0385	0.0598	0.9402	0.0655	0.0575	0.0881	0.9119	0.9430	0.0771	0.420
24	10.55	2	1.25	2715	3945	1.451	0.0348	0.0542	0.9458	0.0493	0.0429	0.0664	0.9336	0.8710	0.0741	0.436
25	10.71	2	1.50	2620	4575	1.745	0.0268	0.0419	0.9581	0.0468	0.0407	0.0629	0.9371	0.9550	0.0817	0.439
26	10.74	3	0.50	7360	4215	0.574	0.0608	0.0932	0.9068	0.0848	0.0759	0.1152	0.8848	0.8700	0.1373	0.810
27	10.59	3	0.75	5140	4475	0.872	0.0467	0.0722	0.9278	0.0692	0.0610	0.0932	0.9068	0.9550	0.1192	0.641
28	10.74	3	1.00	4240	4890	1.152	0.0428	0.0662	0.9338	0.0579	0.0508	0.0781	0.9212	0.9830	0.1076	0.561
29	10.61	3	1.25	3900	5650	1.448	0.0330	0.0514	0.9486	0.0486	0.0424	0.0656	0.9344	0.9450	0.1106	0.600
30	10.75	3	1.50	3720	6450	1.736	0.0313	0.0488	0.9612	0.0434	0.0376	0.0583	0.9417	0.9450	0.1103	0.599
31	15.28	1	0.50	2487	1470	0.591	0.0828	0.1253	0.8747	0.1202	0.1130	0.1682	0.8318	0.8563	0.0713	0.427
32	15.27	1	0.75	1774	1543	0.870	0.0695	0.1060	0.8940	0.1081	0.1000	0.1499	0.8501	0.8762	0.0583	0.341
33	15.25	1	1.00	1587	1796	1.132	0.0625	0.0957	0.9043	0.0942	0.0854	0.1291	0.8707	0.8813	0.0572	0.333
34	15.27	1	1.25	1465	2124	1.450	0.0441	0.0682	0.9318	0.0615	0.0540	0.0831	0.9169	0.9312	0.0625	0.344
35	15.26	1	1.50	1371	2378	1.734	0.0452	0.0699	0.9301	0.0533	0.0466	0.0720	0.9280	0.9130	0.0751	0.422
36	15.24	2	0.50	4937	2865	0.580	0.0848	0.1293	0.8707	0.1268	0.1200	0.1779	0.8221	0.8467	0.1370	0.830
37	15.24	2	0.75	3602	3122	0.867	0.0677	0.1033	0.8967	0.1053	0.0970	0.1456	0.8544	0.8776	0.1212	0.708
38	15.24	2	1.00	2801	3233	1.154	0.0586	0.0899	0.9101	0.0874	0.0784	0.1190	0.8810	0.9037	0.1048	0.595
39	15.24	2	1.25	2708	3924	1.449	0.0480	0.0741	0.9259	0.0701	0.0619	0.0948	0.9052	0.9241	0.1115	0.619
40	15.24	2	1.50	2597	4572	1.760	0.0428	0.0662	0.9338	0.0623	0.0547	0.0841	0.9159	0.9323	0.1116	0.614
41	14.70	3	0.50	7337	4286	0.584	0.0838	0.1268	0.8732	0.1221	0.1149	0.1708	0.8292	0.8560	0.1900	1.138
42	14.70	3	0.75	5136	4450	0.866	0.0635	0.0972	0.9028	0.1020	0.0936	0.1408	0.8592	0.8844	0.1719	0.997
43	14.70	3	1.00	4233	4886	1.154	0.0566	0.0869	0.9131	0.0850	0.0763	0.1159	0.8841	0.9062	0.1523	0.862
44	14.70	3	1.25	3891	5610	1.442	0.0475	0.0734	0.9266	0.0731	0.0648	0.0991	0.9009	0.9278	0.1526	0.843
45	14.70	3	1.50	3708	6444	1.738	0.0373	0.0580	0.9420	0.0600	0.0527	0.0811	0.9189	0.9429	0.1587	0.863

<sup>a</sup> Equilibrium data were obtained from Rood (18).