Copyright Warning & Restrictions

The copyright law of the United States (Title 17, United States Code) governs the making of photocopies or other reproductions of copyrighted material.

Under certain conditions specified in the law, libraries and archives are authorized to furnish a photocopy or other reproduction. One of these specified conditions is that the photocopy or reproduction is not to be "used for any purpose other than private study, scholarship, or research." If a, user makes a request for, or later uses, a photocopy or reproduction for purposes in excess of "fair use" that user may be liable for copyright infringement,

This institution reserves the right to refuse to accept a copying order if, in its judgment, fulfillment of the order would involve violation of copyright law.

Please Note: The author retains the copyright while the New Jersey Institute of Technology reserves the right to distribute this thesis or dissertation

Printing note: If you do not wish to print this page, then select "Pages from: first page # to: last page #" on the print dialog screen



The Van Houten library has removed some of the personal information and all signatures from the approval page and biographical sketches of theses and dissertations in order to protect the identity of NJIT graduates and faculty.

THE EFFECT OF ULTRASONIC VIBRATION

ON THE MASS TRANSFER IN A

PACKED COLUMN

ΒY

JOSEPH WALTER STANECKI

A THESIS

PRESENTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE

0F

MASTER OF SCIENCE IN CHEMICAL ENGINEERING

AT

NEWARK COLLEGE OF ENGINEERING

This thesis is to be used only with due regard to the rights of the author. Bibliographical references may be noted, but passages must not be copied without permission of the College and without credit being given in subsequent written or published work.

Newark, New Jersey

1972

APPROVAL OF THESIS THE EFFECT OF ULTRASONIC VIBRATION ON THE MASS TRANSFER IN A PACKED COLUMN

ΒY

JOSEPH WALTER STANECKI

FOR

DEPARTMENT OF CHEMICAL ENGINEERING NEWARK COLLEGE OF ENGINEERING

ΒY

FACULTY COMMITTEE

APPROVED:

NEWARK, NEW JERSEY

1972

TABLE OF CON	TENTS	
--------------	-------	--

Title	Page
Abstract	ii.
Preface	iv.
Acknowledgement	v.
List of Figures	vi.
List of Tables	vi.
Theory of Packed Columns	1.
Equipment	1.
Design	3.
Theory of Ultrasonics	5.
Ultrasonic Waves Sonic Generators Industrial Uses Past Experimentation Object of Research	5. 10. 12. 13.
Experimental Apparatus	14.
Experimental Procedure	20.
Determination of Operational Conditions	20.
Ultrasonic Operation	21.
Changing Systems	22.
System Variables	23.
Selection of Systems	23.
Determination of Experimental Data	23.
Limits of Ultrasonic Operation	24.
Description of Ultrasonic Operations	28.
Analysis of Packing	65.
Discussion and Evaluation of Results	66.
Author's Comments on Safety	77.
Recommendations	78.
Symbol Designations	79.
Footnotes	80.
References	84.

ABSTRACT

The object of this research was to investigate the possibility of using externally supplied ultrasonic vibration to surface of a packed distillation column operating at total reflux to improve the separation. The apparatus used for this experimentation consisted of a 2 inch ID Pyrex column having three pairs of ultrasonic transducers epoxied equidistance along the external surface of the column. Each pair of transducers was driven separately by a 50 watt amplifier/ power supply and a sine/square wave generator.

There were three types of binary mixtures covered during experimentation: minimum boiling azeotropes, maximum boiling azeotropes and full range mixtures. In all cases the use of ultrasonic vibrations increased the composition of the overhead when compared with the normal operation. The principles which govern this improvement in separation are not well defined but can be partially explained by a combination of the following:

- The velocity of the vapor and its direction are affected by the generation of high frequency sound waves and shock waves caused by caviation of the liquid.
- 2. The liquid loading as well as the path of the descending liquid are modified by the caviation of the liquid.

107657

ROBERT W. VAN HOUTEN LIBRARY NEWARK COLLEGE OF ENGINEERING 3. The arrangement of the packing is altered during ultrasonic operation which is evidenced by the presence of broken berl saddles in the areas of the transducers.

The above mentioned conditions are probably only a few of the many phenomena which occur in this quasi-steady state process. Only further experimentation can answer the questions which this experimentation has uncovered.

PREFACE

Because commercial distillation equipment is usually limited by reboiler and/or condenser duty or constant boiling mixtures which may occur, some other process must be found to increase the efficiency of separation without drastically increasing either initial cost or utilities.

The purpose of this thesis is to investigate the possibility of using ultrasonic vibration to improve the separation of a packed distillation column operated at total reflux. The author will make no attempt to explain the results from a strictly thermodynamic or thermophysical standpoint; however, a critical evaluation of each system as well as recommendations for further study will be presented.

ACKNOWLEDGEMENT

I would like to thank my advisor Dr. A. J. Perna, as well as Jerry Minter of the Components Corporation and Dr. R. H. Rose, for their guidance and assistance in procuring the equipment and laboratory space for my experimentation.

The funds for the experimentation were provided from a grant under Title IV of the National Defense Education Act.

I would also like to thank Barbara Lombardi and Karen Wagner for providing secretarial assistance and my employer, The Lummus Company, for giving me the opportunity to write portions of this text during working hours.

Especially, I would like to thank my parents who provided the incentive for me to complete my graduate studies even though they had been interrupted by a tour of duty with the United States Army, Republic of Vietnam.

List of Figures

<u>Title</u>		Page
Figure 1.	Simplifier Diagram of a Packed Column	2.
Figure 2.	Spectrum of Sounds That Can Be Produced in Gases	8,
Figure 3.	Spectrum for Liquids at Atmospheric Pressure	୍ୱ .
Figure 4.	Peak Sound Intensity Obtainable at the Focus of a Sound System as a Function of Frequency	11.
Figure 5.	Schematic Diagram of Experimental Apparatus	15.
Figure 6.	Separation Factor vs. Frequency for Benzene(A)- Ethanol(B) Mixture	69.
Figure 7.	Separation Factor vs. Frequency for Carbon Tetrachloride(A)-Ethanol(B) Mixture	70.
Figure 8.	Separation Factor vs. Frequency for Carbon Tetrachloride(A)-Ethylacetate(B) Mixture	71.
Figure 9.	Separation Factor vs. Frequency for Chloroform(A)-Acetone(B) Mixture	72.
Figure 10.	Separation Factor vs. Frequency for Chloroform(A)-Benzene(B) Mixture	73.
Figure 11.	Separation Factor vs. Frequency for Chloroform(A)-Ethanol(B) Mixture	74.
Figure 12.	Separation Factor vs. Frequency for Ethanol(A)-Water(B) Mixture	75.

List of Tables

<u>Title</u>		Page
Table l.	Key to Figure 5.	16.
Table 2.	Physical Properties of the Components	25.
Table 3.	Summary of 2D3 G-53 Specifications	18.
Table 4.	Steady State Conditions Experimental Data	26.
Table 5.	Benzene(A)-Ethanol(B) Mixture	32.
Table 6.	Benzene(A)-Ethanol(B) Mixture Experimental Data	33.
Table 7.	Benzene(A)-Ethanol(B) Mixture Separation Factors	35.
Table 8.	Carbon Tetrachloride(A)-Ethanol(B) Mixture	36.
Table 9.	Carbon Tetrachloride(A)-Ethanol(B) Mixture Experimental Data	37.
Table 10.	Carbon Tetrachloride(A)-Ethanol(B) Mixture Separation Factors	39.
Table 11.	Carbon Tetrachloride(A)-Ethylacetate(B) Mixture	41.
Table 12.	Carbon Tetrachloride(A)-Ethylacetate(B) Mixture Experimental Data	43.
Table 13.	Carbon Tetrachloride(A)-Ethylacetate(B) Mixture Separation Factors	44.
Table 14.	. Chloroform(A)-Acetone(B) Mixture	46.
Table 15.	. Chloroform(A)-Acetone(B) Mixture Experimental Data	47.
Table 16.	. Chloroform(A)-Acetone(B) Mixture Separation Factors	49.
Table 17	. Chloroform(A)-Benzene(B) Mixture	50.
Table 18	. Chloroform(A)-Benzene(B) Mixture Experimental Data	52.

Title		Page
Table 19.	Chloroform(A)-Benzene(B) Mixture Separation Factors	53.
Table 20.	Chloroform(A)-Ethanol(B) Mixture	55.
Table 21.	Chloroform(A)-Ethanol(B) Mixture Experimental Data	56.
Table 22.	Chloroform(A)-Ethanol(B) Mixture Separation Factors	58.
Table 23.	Ethanol(A)-Water(B) Mixture	60.
Table 24.	Ethanol(A)-Water(B) Mixture Experimental Data	61.
Table 25.	Ethanol(A)-Water(B) Mixture Separation Factor	63.
Table 26.	Conversion Factors	79.

THEORY OF PACKED COLUMNS

1. Equipment

Packed columns are used throughout the chemical process industries because they are an efficient and economical method of contacting liquids and vapors. A simplified diagram of a packed column is shown in Figure 1. In packed columns used for continuous countercurrent contacting operations, a vertical shell is filled with an inert material having a large surface to volume ratio.

The liquid phase L_0 enters the top of the column and is distributed over the upper surface of the packing by spray nozzles or weir distributors. The vapor phase V_0 enters the bottom of the column and rises through the voids of the packing where it contacts the descending liquid.

The packing can be made of any inert material, usually ceramic, metal or plastic. The packing used will differ depending on the service, but it should have the following general characteristics.⁽¹⁾

- a. A large wetted surface per unit volume to provide a large interfacial area for phase contacting.
- A large void volume to allow a tortuous path for the ascending vapor with minimum pressure drop.
- A porous surface to hold up the descending liquid for a longer resonant time.

1.



Figure 1. Simplified Diagram of a Packed Column

- A low bulk density so the weight of packing does not become prohibitive.
- e. Relatively low initial and operating costs.

2. Design

There are many companies involved in the production of packed columns for use in the chemical process industries. The leaders in the field are Norton (formerly U.S. Stoneware Inc.) and Koch; each has published extensive data on specific systems of interest. They have also developed experimental relationships which can be used to design a packed column for any service as a function of the desired loadings of liquid and vapor and the physical properties of the components.

Many theoretical approaches have been given to explain the mass transfer in a packed column. One of the first and probably the most significant article was authored by T. H. Chilton and A. P. Colburn in 1935.⁽²⁾ In the article they defined, by the use of graphical integration, the measure of the difficulty of separation called the number of transfer units. Also defined was the height of a transfer unit which is the necessary amount of contacting needed to accomplish the enrichment of one phase equal to the driving force in the same phase.

Recently there have been many simplications and modifications⁽³⁾⁻⁽¹²⁾ made to this technique, but it still serves today as the most rigorous method of design. With the advent of the computer this rigorous method is easily handled in a matter of seconds.

No attempt will be made in this thesis to treat the dynamics of a packed column from a theoretical approach; however, the effect of ultrasonic vibrations on a column of fixed design will be investigated.

THEORY OF ULTRASONICS

1. <u>Ul</u>trasonic Waves⁽¹³⁾

Ultrasonic waves are sound waves propagating in a media at a frequency above the audible range, roughly defined as 20-20,000 cycles/second. This energy is mechanically transmitted in the form of an elastic wave through a media, either fluid or solid at a velocity independent of the frequency and amplitude, but dependent on the physical properties of the media. Because they are inelastic, fluids cannot propagate shear waves; however, compressional waves are readily transmitted in both fluids and solids. This principle will be employed in the experimental section of this thesis.

A sonic wave is a series of compression and rearification zones which travel with a characteristic amplitude, frequency and velocity. The pressure produced by this unidirectional wave can be expressed as:

 $P = P_0 + P_1 \sin 2\pi f (t - x/c)$ [equation 1.] where P = total instantaneous pressure in the media $P_0 = \text{static pressure in the media}$ $P_1 = \text{magnitude of the pressure fluctuation}$ f = frequency of the wavet = time from referencex = distance along some directionc = velocity of propagation The power associated with this wave is called the sonic intensity and expressed in units of power per unit area.

I =
$$P_1^2/2\rho c$$
 [equation 2.]
where I = sound intensity
 $\rho = density$ of the media

Because the molecules in a fluid are free to undergo vibration, they will vibrate back and forth in the same direction as a propagating wave is traveling. The velocity of each particle is directly proportional to the sound pressure:

$$= \frac{P_1}{c} \quad \text{sin } 2\pi f(t-H_c) \qquad [equation 3.]$$

The specific acoustic impedance is defined as the ratio of sound pressure to particle velocity and at high frequencies in the ultrasonic range is only dependent on the media.

2. <u>Sonic Generators</u>⁽¹⁴⁾

Sonic generators fall into two broad classes:

- a. Fluid current interruption devices.
 - 1) Whistles and other gas current interrupting devices.
 - Valve devices, such as sirens and vibrating reed-type devices.

b. Piston devices.

- 1) Mechanical drive.
- 2) Electrical drive.
 - a) Electromagnetic
 - b) Magnetostriction
 - c) Piezoelectric

There are a variety of generators available depending on the frequency range and specific acoustic impedance required. Because it was desirable to operate in the 20 Kcps to 100 Kcps range and at a high specific acoustic impedance, a piezoelectric transducer was used as the sonic generator. 7.

Figure 2 shows the spectrum of sound in gases (vapors). Because gases cannot support tension, there is an upper intensity level which cannot exceed ambient pressure. At any appreciable distance from the generator the sound energy is diffused over a larger area and consequently there will be a decrease in intensity.

Figure 3 shows the spectrum of sound in liquid assuming the ambient pressure is atmospheric. For sound intensities above approximately 0.6 watts/cm² cavitation will occur. Cavitation is the formation and violent collapse of small bubbles or cavities in the liquid caused by localized changes in pressure. The negative pressure portion of the sound wave causes vaporization and the bubbles of vapor



Figure 2. Spectrum of Sounds that can be Produced



Figure 3. Spectrum for Liquids at Atmospheric

9.

act as points for further tearing apart of the liquid to form larger cavities. When the positive position of the wave is reached, these cavities collapse violently causing shock waves in both the liquid and vapor phases.

Piezoelectric transducers are able to produce intensities above the cavitation level. The maximum power output of a transducer is determined by the area of the transducer because the peak sound intensity is limited. Figure 4 gives the peak sound intensity obtainable as a function of the frequency of the sound wave. Even if the maximum intensity of the transducer lies in the cavitation region, this phenomenon may not occur because of the formation of bubbles which obstruct the path of the sound wave.

3. Industrial Uses

There have been many industrial applications of ultrasonic vibrations since the first time it was mentioned in the literature⁽¹⁵⁾; however, until the end of World War II little of the technology was put to practical use. During this period many technical publications appeared giving data on yields and power consumption which looked on the surface to be very promising.⁽¹⁶⁾ Major applications were made in ultrasonic dust precipitation plants, but the failure of these units to reach the theoretical optimum caused reservations on the part of many industrialists. Since then numerous studies have

10.



Figure 4. Peak Sound Intensity Obtainable at the

Focus of a Sound System as a Function

been made to improve the design of these systems. Although power consumption is still the major drawback, the units are able to handle very small particles and operate over a wide temperature range.

Until the late 1950's progress in the ultrasonic field was restricted to aerosols.⁽¹⁷⁾ At that time investigations were made to determine what effect pulsation and vibration had on the rate of diffusion processes. The first studies were made by a group of Russians,⁽¹⁸⁾ who studied what influence the pulsating motion of a liquid had on the rate of dissolution of a solid suspended in the liquid. Another group of Russians made subsequent studies which correlated the hydraulic resistance of a layer of zinc dust as a function of frequency. As interest grew they extended their work to study the rate of heat transfer in layers of free-flowing materials subjected to pulsation.⁽¹⁹⁾

4. Past Experimentation

The only application of ultrasonic energy to a packed column is covered in United States Patent 2,265,762 filed by Donald S. McKittrich and Robert E. Cornish of Shell Development Company, San Francisco. In the <u>Example</u> section of the patent they use an insulated column equipped with a spiral wire helix and an electric automobile horn attached to a right-angle extension of the column. The results show an increase of 55% in the number of theoretical plates in the column ρ ver what was observed without sound. The claims they made are quoted:⁽²⁰⁾

- 1. In a distillation process wherein ascending vapors contact liquid reflux in a reflux zone, the step of subjecting the overhead vapors in said zone to the influence of sonic vibration of frequencies between 50 cycles per second and 5 megacycles per second, thereby increasing efficiency of fractionation and reducing the necessary number of theoretical plates.
- The process of claim 1 wherein the sonic vibrations have such frequencies as to be in resonance with the natural frequencies of said reflux zone.

The patent discusses several other methods of sonic excitement including the use of several points of introduction of sonic vibrations, the method employed in the experimental section of this thesis.

There have been many other experiments performed with the aid of a sonic vibrations (21)-(26) but none to the author's knowledge use transducers affixed to the surface of a packed column to excite the packing as well as the two process phases.

5. Object of Research

The object of this research is to investigate the possibility of **us**ing ultrasonic vibration to improve the separation of a packed **co**lumn operating at total reflux.

EXPERIMENTAL APPARATUS

The experimental apparatus is shown schematically in Figure 2. A Pyrex column 2.0 inches ID and 40.0 inches long is packed to 36.0 inches with 0.25 inch ceramic berl saddles. The packing is supported by a perforated Teflon support plant having 55, 0.1875 inch holes on a 0.375 inch triangular pitch. Two Kimax heads are identical having 0.50 inch center nozzles and 0.75 inch side nozzles.

The overhead condenser, 18.0 inches long, provides approximately bubble-point reflux through the reflux return pipe. The gas sample outlet has a dual purpose; during normal operation the overhead temperature is monitored by a thermometer inserted in the nozzle through a flexible Neoprene coupling while during sampling operations a glass tube connected to a flash immersed in an ice bath is inserted through the coupling to condense an overhead vapor sample.

The necessary vapor is provided to the column by a reboiler consisting of a 2,000 ml two neck flash equipped with a hemispherical mantle and Powerstat. Tygon tubing and glass fittings connect the reboiler to the reboiler head. The reboiler temperature can be observed on a thermometer inserted in the oil well of the reboiler while the bottoms liquid can be removed through the liquid sample outlet.

14.





Table 1. Key to Figure 5.

Notation	Description
(1)	Atmospheric vent
(2)	Cooling water outlet
(3)	Cooling water inlet
(4)	Overhead reflux condenser
(5)	Reflux return pipe
(6)	Reflux head
(7)	Gas sample outlet (thermometer inserted)
(8)	Leads to top transducers
(9)	Leads to middle transducers
(10)	Leads to bottom transducers
(11)	Packing support plate
(12)	Reboiler head
(13)	Vapor return pipe
(14)	Liquid return pipe
(15)	Reboiler with thermometer well
(16)	Liquid sample outlet valve
(17)	Hemispherical heating mantle
(18)	Powerstat type 116
(19)	RCA WA-44C Sine/Square Wave Audio Generator
(20)	McIntosh No. 50-W-2 Amplifier
(21)	McIntosh NO. P-50-D Power Supply
(22)	Pyrex column
(23)	Ceramic packing
(24)	Vapor ice trap

Three pairs of Gulton 2D3-53G lead zirconate titanate transducers are epoxyed to the column 9, 18, and 27 inches from the packing support plate and oriented 180° apart. Each pair is wired in series with a McIntosh Model 50-W-2 Amplifier and Power Supply driven by an RCA Model 44 Sine/Square Audio Generator.

Specification	Quantity
Material	Lead zirconate titanate
Diameter	0.25 inch
Thickness	0.05 inch
Free dielectric constant	
К _З	720
к ₁	960
Loss tan	0.022
Normal density	7.6 gm/cm ³
Curie temperature	330°C
Coupling coefficients	
k ₃₃	0.60
, k _p	0.50
^k 31	0.29
k ₁₅	0.64
Piezoelectric charge coefficient	
d ₃₃	190*10 ⁻¹² meters/volt
d ₃₁	-84*10 ⁻¹² meters/volt
^d 15	300*10 ⁻¹² meters/volt
Piezoelectric voltage coefficient	
g ₃₃	30*10 ⁻³ volt-meter/Newton
g ³¹	-13*10 ⁻³ volt-meter/Newton
9 ₁₅	36*10 ⁻³ volt-meter/Newton

Table 3. Summary of 2D3 G-53 Specifications

Table 3. - continued

Specification

Elastic modulus

Y₃₃ Y₁₁ Y₅₅ Mechanical quality factor Coercive field Remanent polarization

<u>Quantity</u>

6.5*10¹⁰ Newtons/meters² 8.1*10¹⁰ Newtons/meters² 3.8*10¹⁰ Newtons/meters² 140

13 Kv/cm @ 60 cycles

 $26 \text{ microcoulombs/cm}^2$

EXPERIMENTAL PROCEDURE

1. Determination of Operating Conditions

Because the heats of vaporization for the components vary (refer to Table 2), it is necessary to obtain operating conditions for each system. Once these conditions are set they remain constant while the particular system is under consideration.

- a. The reboiler is charged with 1000 ml of a 50-50 volumetric mixture of the system and the Powerstat is set at 100.
- As vigorous boiling occurs, the overhead condenser is partially commissioned and the vent is opened to pressure relieve the column.
- c. As the condensing vapors become visible in the overhead condenser, the condenser is fully commissioned.
- d. The system is now observed for a period of 30 minutes and the powerstat setting and cooling water rate are adjusted to achieve stable operation and approximately bubble point reflux. The reflux conditions can be observed by sliding the thermometer in the gas sample outlet in and out observing the temperature of the ascending vapors and returning reflux. Because no overhead product is being taken the vapor and liquid will have the same

composition and the bubble point and dew point temperature will be identical.

- e. Once satisfactory conditions are reached the Powerstat setting, reboiler temperature, and overhead vapor temperature are recorded.
- f. A glass tube connected by a piece of flexible Tygon tubing to a flask immersed in an ice bath is inserted into the gas sample outlet to obtain a sample of the ascending vapor.
- g. The liquid sample outlet is opened and after draining the residual liquid a sample of the bottoms liquid is obtained.
- h. The unused sample of the condensed overhead vapor and bottoms liquid is combined with the residual liquid drained from the liquid sample outlet and introduced back into the column through the vent.

2. Ultrasonic Operation

The operating conditions for each system are established in Part 1 and will remain constant for the duration of experimentation made on each system.

 A frequency of 20 Kcps is set on the sine/square wave audio generator and the column is allowed approximately 15 minutes to come to steady-state. 21.

This steady state condition is determined by the fluctuation in the overhead vapor temperature.

- b. Repeat steps e. through h. of Part 1.
- c. Increment the frequency 5 Kcps and repeat steps
 b. and c. of Part 2 until samples are taken at
 100 Kcps.

3. Changing Systems

After the experimentation on a given system was completed the column was cleaned by refluxing 500 ml of acetone for 30 minutes at which time the reflux head was removed without disturbing the packing and the acetone was allowed to evaporate for at least 8 hours. The reboiler is then charged with 500 ml of the next system and again the system is operated under total reflux for 1 hour, then allowed to cool. The reboiler is drained and charged as in Part 1. a. and experimentation is begun.

SYSTEM VARIABLES

1. Selection of Systems

The seven systems used in this thesis were chosen because they consist of common chemicals whose normal boiling points are 100°C or less. They were paired to give the maximum difference in refractive index so this property could be used to measure their composition without the use of elaborate sampling techniques such as gas chromatography. A sample of only three or four drops is needed to determine the composition within the four place accuracy of the refractometer. This sample when compared to the 1,000 ml charge has a negligible effect on the liquid and vapor loadings in the column, thus eliminating the necessity of taking simultaneous overhead and bottoms samples. The pairings used were also chosen to give maximum and minimum boiling azeotropic binary mixtures as well as full range composition mixtures so the effect of ultrasonic operation would cover all possible combinations.

2. Determination of Experimental Data

Before any experimentation could begin it was necessary to determine the refractive indices of the various systems over the entire composition range. This was accomplished by determining the refractive indices of precisely measured samples of each system. It was found that the refractive index of these mixtures was, within experimental error, a linear function of the mole percent of one of the components. The mole percent was calculated from the given volume percent, the molecular weight and the specific gravity by assuming perfect mixing. Because only a comparative approach to the ultrasonic operations is under investigation here, this method of determining composition is sufficient.

3. Limits of Ultrasonic Operation

The power relationship used in the evaluations were developed for frequencies well below the resonant frequency, so an upper limit of 100 Kcps was set which is consistent with the response curve of the amplifier which is relatively flat between 20 Kcps and 100 Kcps.
Component	Normal Boiling Point, °C	Molecular Weight	Specific Gravity gm/cm3	Refractive Index
Acetone	56.5	58.08	0.7899	1.3543
Benzene	80.1	78.12	0.8787	1.4949
Carbon Tetrachloride	76.7	153.82	1.5940	1.4548
Chloroform	61.3	119.38	1.4832	1.4402
Ethano1	78.4	46.07	0.7893	1.3574
Ethylacetate	77.1	88.12	0.9003	1.3676
Water	100.0	18.06	0.9966	1.3314

Table 2. Physical Properties of the Components

Table 2. Physical Properties of the Components - Cont'd

		Heat of Vaporization	
Component	cal/gm mol	cal/gm_	<u>cal/cm3</u>
Acetone	7642	131.6	103.9
Benzene	8147	104.3	91.6
Carbon Tetrachloride	8272	53.8	85.7
Chloroform	7501	62.8	93.2
Ethanol	9674	210.0	165.7
Ethylacetate	8301	94.2	84.8
Water	7416	410.6	409.2

Table 4. Steady State Conditions Experimental Data

Mixture	To	Ro	<u>YA</u>	T _b	Rb	XA	<u>P</u>
Benzene (A) - Ethanol (B)	65.7	1.4206	45.96	70.6	1.4094	37.81	100
Carbon Tetrachloride (A) - Ethanol (B)	61.4	1.4076	51.54	65.1	1.3907	34.19	105
Carbon Tetrachloride (A) - Ethylacetate (B)	72.1	1.3980	34.86	74.1	1.4165	56.08	90
Chloroform (A) - Acetone (B)	60.3	1.4105	65.42	61.1	1.3939	46.09	85
Chloroform (A) - Benzene (B)	67.4	1.4535	75.69	77.5	1.4696	46.26	90
Chloroform (A) - Ethanol (B)	58.2	1.4123	66.31	61.6	1.3865	35.15	100
Ethanol (A) - Water (B)	76.2	1.3507	74.23	87.4	1.3335	8.07	85

DESCRIPTION OF ULTRASONIC OPERATIONS

1. <u>Benzene (A) - Ethanol (B) Mixture</u>

In normal distillation processes the pressure and temperature increase from the top to the bottom of the column. This would mean once the azeotropic composition had been reached the conditions in the column due to the pressure and temperature profiles would work against any improvement in separation.

As can be seen in this system, which is a minimum boiling azeotropic binary mixture, the overhead composition reached the azeotropic composition and remained relatively constant. To improve the separation beyond the atmospheric azeotropic composition, the column would have to be operated under vacuum which would mean greater initial and operating costs.

2. <u>Carbon Tetrachloride (A) - Ethanol (B) Mixture</u>

This is also a minimum boiling azeotropic binary mixture which is limited by the conditions mentioned in the previous section; however, other difficulties were encountered while dealing with this system.

Violent oscillation was encountered during the operation of this system. While driving the two top transducers above 65 Kcps slugs of liquid were noticed being carried by the vapor into the overhead condenser causing a momentary dry point followed by flooding in the upper section of the column. While driving the two middle transducers above 75 Kcps, there were short durations of no overhead vapor followed by no bottoms liquid which indicates reverse flow occuring in the mid-section of the column. When the two bottom transducers were driven above 90 Kcps, the reboiler started to pulsate, sending slugs of liquid into the reboiler head; this condition was alleviated by pinching down on the liquid return pipe (Tygon tubing).

The aforementioned upsets in the ultrasonic operation of the column caused the termination of experimentation on these systems at the respective frequencies.

3. Carbon Tetrachloride (A) - Ethylacetate (B) Mixture

This system is a minimum boiling azeotropic binary mixture which performed similar to the Benzene (A) - Ethanol (B) mixture. No difficulties were encountered during the experimentation on this system.

4. Chloroform (A) - Acetone (B) Mixture

This was the only maximum boiling azeotropic binary mixture investigated. Because the normal operating conditions of a distillation column (decreasing temperature from bottom to top) favors continued separation once the azeotropic composition has been reached, it was expected that the overhead composition would readily pass the atmospheric azeotropic composition. As can be seen in Figure 9., the azeotropic composition (Separation Factor = 1.000) was passed at a fairly low frequency and from the slope of the curve continued improvement could be expected as the frequency is increased.

5. Chloroform (A) - Benzene (B) Mixture

This is the only full range binary mixture used for experimentation. As in the previous systems, the separation factor increases with increasing frequency; however, unlike the previous systems a maximum is reached at which point there is a decrease in the separation factor. Because this condition was experienced only with this system, the components rather than the packing must begin to resonate at some characteristic frequency causing a decrease in the efficiency of contacting.

6. <u>Chloroform (A) - Ethanol (B) Mixture</u>

Because this system is a minimum boiling azeotropic binary mixture, it was expected the system would perform similarly to the previous systems, but this was not the case.

When the separation factor reached approximately 0.950 (95% of the azeotropic composition), a foaming mixture appeared on the top of the packing. As the frequency was increased, the foam totally filled the reflux head causing the investigation of this system to be terminated.

When the two bottom transducers were driven above 60 Kcps, there was no improvement in the overhead composition. This combined with the previous evidence of foam indicates the presence of foam in the center section of the packing.

7. Ethanol (A) - Water (B) Mixture

Because of previous experience with the ethanol - water mixture it was expected that difficulties would be encountered during operations. Because the 50-50 volumetric mixture is only 23.54 mole percent ethanol, a quasi-steam distillation effect governs this system during certain operations. As can be seen from Figure 12, when driving the two top transducers, the atmospheric azeotropic composition was easily passed and compositions in the 96%-plus range were experienced. Only small amounts of overhead vapor were noted indicating the reflux was vaporizing as soon as it came in contact with the upper surface of the packing. The bottoms liquid was 90%-plus water, reinforcing the theory that steam was supplying heat to the packing without contacting any descending liquid.

Volume Percent(A)	Mole Percent(A)	Refractive Index
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95	$\begin{array}{c} 0.00\\ 3.35\\ 6.81\\ 10.40\\ 14.12\\ 17.99\\ 21.99\\ 26.16\\ 30.49\\ 34.99\\ 39.68\\ 44.57\\ 49.67\\ 54.99\\ 60.55\\ 66.28\\ 72.41\\ 78.85\\ 85.55\\ 92.59\\ 100.00\\ \end{array}$	1.3574 1.3620 1.3668 1.3727 1.3768 1.3821 1.3876 1.3934 1.3993 1.4055 1.4120 1.4187 1.4257 1.4330 1.4407 1.4485 1.4570 1.4658 1.4750 1.4847

Table 5. Benzene(A)-Ethanol(B) Mixture

Mole	Percent(A)	
Liquid	Vapor	<u>Temperature,°C</u>
Ο	D	78 1
6	20	74.4
11	30	72.4
20	40	70.1
39	50	68.3
57	56	67.8
72	60	68.3
89	70	70.3
96	85	75.2
100	100	79.7

Minimum boiling point azeotropic binary mixture at 55.4 mole percent(A) and 67.9°C.

Mole Percent(A) = (727.27)*(Refractive Index) - 987.20

<u>Position</u>		Bottom				Midd1	e	
Frequency	To	<u>T</u> b	Ro	<u>R</u> b	<u>T</u> o	<u>T</u> b	<u><u>R</u>o</u>	<u>R</u> b
20 Kcps	65.7	70.6	1.4206	1.4094	65.7	70.6	1.4206	1.4094
25 Kcps	65.7	70.6	1.4206	1.4094	65.7	70.6	1.4226	1.4094
30 Kcps	65.7	70.6	1.4225	1.4094	65.7	70.6	1.4232	1.4094
35 Kcps	65 .7	70.6	1.4231	1.4094	65.8	70.6	1.4243	1.4094
40 Kcps	65.9	70.6	1.4243	1.4094	65.8	70.6	1.4254	1.4094
45 Kcps	65.9	70.6	1.4252	1.4094	65.8	70.6	1.4259	1.4093
50 Kcps	65.9	70.6	1.4258	1.4093	65.8	70.6	1.4265	1.4093
55 Kcps	65.9	70.6	1.4266	1.4093	66.0	70.6	1.4276	1.4093
60 Kcps	66.1	70.6	1.4275	1.4093	66.0	70.6	1.4286	1.4093
65 Kcps	66.1	70.6	1.4284	1.4093	66.0	70.6	1.4292	1.4094
70 Kcps	66.1	70.6	1.4291	1.4093	66.4	70.6	1.4299	1.4094
75 Kcps	66.4	70.6	1.4299	1.4094	66.5	70.6	1.4311	1.4094
80 Kcps	66.4	70.6	1.4309	1.4094	66.6	70.6	1.4319	1.4094
85 Kcps	66.6	70.6	1.4317	1.4094	66.6	70.6	1.4326	1.4093
90 Kcps	66.6	70.6	1.4323	1.4094	66 .7	70.6	1.4333	1.4093
95 Kcps	66.6	70.6	1.4330	1.4093	66.8	70.6	1.4335	1.4093
100 Kcps	66.8	70.6	1.4335	1.4093	66.8	70.6	1.4336	1.4093

Table 6. Benzene (A)-Ethanol (B) Mixture Experimental Data

Table 6. Benzene	(A)-Ethanol	(B) Mixture	Experimental	Data
Position		Тор		
Frequency	<u>T</u> o	<u>T</u> b	<u>R</u> o	<u>R</u> b
20 Kcps	65.7	70.6	1.4207	1.4094
25 Kcps	65.7	70.6	1.4228	1.4094
30 Kcps	65.8	70.6	1.4239	1.4094
35 Kcps	65.8	70.6	1.4251	1.4094
40 Kcps	65.9	70.6	1.4259	1.4093
45 Kcps	65.9	70.6	1.4264	1.4093
50 Kcps	66.0	70.6	1.4277	1.4093
55 Kcps	66.1	70.6	1.4286	1.4093
60 Kcps	66.3	70.6	1.4291	1.4093
65 Kcps	66.4	70.6	1.4298	1.4094
70 Kcps	66.5	70.6	1.4310	1.4094
75 Kcps	66.6	70.6	1.4320	1.4094
80 Kcps	66.6	70.6	1.4325	1.4093
85 Kcps	66.7	70.6	1.4334	1.4093
90 Kcps	66.8	70.6	1.4336	1.4093
95 Kcps	66.8	70.6	1.4337	1.4093
100 Kcps	66.8	70.6	1.4338	1.4093

ų

	1	able /.	Benzene(A)	-Ethanol (I	b) mixture	Separation	1 100001	-	
Position		Bottom	_		Middle			Тор	
Frequency	<u>Y</u> A	FA	X _A	Υ _A	FA	<u>X</u> A	<u>Y</u> A	FA	<u>×</u> A
20 Kcps	45.96	0.830	37.81	45.96	0.830	37.81	46.03	0.830	37.81
25 Kcps	45.96	0.830	37.81	47.41	0.856	37.81	47.56	0.858	37.81
30 Kcps	47.34	0.855	37.81	47.85	0.864	37.81	48.36	0.873	37.81
35 Kcps	47.78	0.862	37.81	48.65	0.878	37.81	49.23	0.889	37.81
40 Kcps	48.65	0.878	37.81	49.45	0.893	37.81	49.81	0.899	37.74
45 Kcps	49.31	0.890	37.81	49.81	0.899	37.74	50.18	0.906	37.74
50 Kcps	49.74	0.898	37.74	50.25	0.907	37.74	51.12	0.923	37.74
55 Kcps	50.32	0.908	37.74	51.05	0.921	37.74	51.78	0.935	37.74
60 Kcps	50.98	0.920	37.74	51.78	0.935	37.74	52.14	0.941	37 .8 1
65 Kcps	51.63	0.932	37.74	52.21	0.942	37.81	52.65	0.950	37.81
70 Kcps	52.14	0.941	37.74	52.72	0.952	37.81	53.52	0.966	37.81
75 Kcps	52.72	0.952	37.81	53.60	0.967	37.81	54.26	0.979	37.81
80 Kcps	53.45	0.965	37.81	54.18	0.978	37.81	54.61	0.986	37.74
85 Kcps	54.03	0.975	37.81	54.69	0.987	37.74	55.27	0.998	37.74
90 Kcps	54.47	0.983	37.81	55.20	0.996	37.74	55.41	1.000	37.74
95 Kcps	54.98	0.992	37.74	55.34	0.998	37.74	55.49	1.002	37.74
100 Kcps	55.34	0.998	37.74	55.41	1.000	37.74	55.56	1.003	37.74

Volum	e	Mole	Refractive
Percen	t(A)	Percent(A)	Index
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80		$\begin{array}{c} 0.00\\ 3.10\\ 6.32\\ 9.69\\ 13.20\\ 16.86\\ 20.66\\ 24.67\\ 28.85\\ 33.23\\ 37.81\\ 42.64\\ 47.71\\ 53.04\\ 58.66\\ 64.60\\ 70.87\end{array}$	1.3574 1.3604 1.3636 1.3639 1.3703 1.3738 1.3775 1.3814 1.3855 1.3898 1.3942 1.3989 1.4039 1.4091 1.4145 1.4203 1.4264
80		70.87	1.4264
85		77.51	1.4329
90		84.55	1.4398
95		92.04	1.4470
100		100.00	1.4548
Vanor	Mole Percent(A)	liquid T	emperature.°C

Table 8. Carbon Tetrachloride(A)-Ethanol(B) Mixture

Vapor	Liquid	Temperature,°C
0	0.0	77.9
25 35	0.4 11.4	72.8
45	17.6	68.0
55	33.6	65.0 Data at 745 mm Hg
60	60.0	63.8
67	72.8	64.3
100	100.0	75.9

Minimum boiling point azeotropic binary mixture at 61.3 mole percent(A) and 64.95°C.

Mole Percent(A) = (1026.69)*(Refractive Index) - 1393.63

	001 0011	1001 aoni or			INCOLC EXP	<u>er men eur</u>	Ducu	
Position		Тор				<u>Midd</u>]	e	
Frequency	<u>T</u> o	<u>т</u> ь	<u>R</u> o	<u>R</u> b	<u>T</u> 0	<u>T</u> b	<u>R</u> o	Rb
20 Kcps	61.4	65.1	1.4076	1.3907	61.4	65.1	1.4076	1.3907
25 Kcps	61.4	65.1	1.4080	1.3907	61.4	65.1	1.4082	1.3907
30 Kcps	61.4	65,1	1.4082	1.3907	61.4	65.1	1.4090	1.3907
35 Kcps	61.4	65.1	1.4088	1.3907	61.4	65.1	1.4099	1.3908
40 Kcps	61.4	65.1	1.4091	1.3907	61.4	65.1	1.4106	1.3908
45 Kcps	61.4	65.1	1.4098	1.3908	61.4	65.1	1.4114	1.3908
50 Kcps	61.4	65.1	1.4102	1.3908	61.5	65.1	1.4122	1.3908
55 Kcps	61.4	65.1	1.4109	1.3908	61.6	65.1	1.4129	1.3907
60 Kcps	61.4	65.1	1.4115	1.3908	61.5	65.1	1.4136	1.3907
65 Kcps	61.5	65.1	1.4121	1.3907	61.6	65.1	1.4143	1.3907
70 Kcps	61.5	65.1	1.4127	1.3907	61.6	65.1	1.4149	1.3906
75 Kcps	61.5	65.1	1.4134	1.3907	61.7	65.1	1.4154	1.3906
80 Kcps	61.6	65.1	1.4140	1.3906				
85 Kcps	61.6	65.1	1.4147	1.3906				
90 Kcps	61.7	65.1	1.4154	1.3906				
95 Kcps								
100 Kcps								

Table 9. Carbon Tetrachloride (A)-Ethanol (B) Mixture Experimental Data

Table 3. Carbon	Techacinoriae (MIXTURE EXPE	I Inclical Data
Position		Botto	<u>om</u>	
Frequency	<u> </u>	Т _b	<u>R</u> o	<u>R</u> b
20 Kcps	61.4	65.1	1.4076	1.3907
25 Kcps	61.4	65.1	1.4088	1.3907
30 Kcps	61.4	65.1	1.4101	1.3908
35 Kcps	61.4	65.1	1.4114	1.3908
40 Kcps	61.5	65.1	1.4124	1.3908
45 Kcps	61.5	65.1	1.4134	1.3908
50 Kcps	61.6	65.1	1.4142	1.3908
55 Kcps	61.6	65.1	1.4150	1.3909
60 Kcps	61.7	65.1	1.4154	1.3909
65 Kcps				
70 Kcps				
75 Kcps				
80 Kcps				
85 Kcps				
90 Kcps				
95 Kcps				
100 Kcps				

Table 9. Carbon Tetrachloride (A)-Ethanol (B) Mixture Experimental Data

				# (p)		
Table 10.	Carbon	Tetrachlor	ride (A)-Eth	nanol (B) Mi	ixture Sepai	ration Factors
Position	V	r	v	v	F	Y
Frequency	<u>Υ</u> Α	<u>+</u> A	<u>×</u> A	<u> </u>	- - A	<u>^A</u>
20 Kcps	51.54	0.841	34.19	51.54	0.841	34.19
25 Kcps	51.95	0.847	34.19	52.15	0.851	34.19
30 Kcps	52.15	0.851	34.19	52.98	0.864	34.19
35 Kcps	52.77	0.861	34.19	53.90	0.879	34.29
40 Kcps	53.08	0.866	34.19	54.62	0.891	34.29
45 Kcps	53.80	0.877	34.29	55.44	0.904	34.29
50 Kcps	54.21	0.884	34.29	56.26	0.918	34.29
55 Kcps	54.93	0.896	34.20	56.98	0.930	34.19
60 Kcps	55.54	0.906	34.29	57.70	0.941	34.19
65 Kcps	56.16	0.916	34.19	58.42	0.953	34.19
70 Kcps	56.77	0.926	34.19	59.03	0.963	34.09
75 Kcps	57.49	0.938	34.19	59.55	0.971	34.09
80 Kcps	58.11	0.948	34.09			
85 Kcps	58.83	0.960	34.09			
90 Kcps	59.55	0.971	34.09			
95 Kcps		~				
100 Kcps						

Table 10.	Carbon Tetrachloride	(A)-Ethanol (B) Mixture	Separation Factors
Position			
Frequency	<u>Y</u> A	FA	XA
20 Kcps	51.54	0.841	34.19
25 Kcps	52.77	0.861	34.19
30 Kcps	54.11	0.883	34.29
35 Kcps	55.44	0.904	34.29
40 Kcps	56.47	0.921	34.29
45 Kcps	57.49	0.938	34.29
50 Kcps	58.31	0.951	34.29
55 Kcps	59.14	0.965	34.39
60 Kcps	59.55	0,971	34.39
65 Kcps			
70 Kcps			
75 Kcps			
80 Kcps			
85 Kcps			
90 Kcps			
95 Kcps			
100 Kcps			

Volume Percent(A)	Mole Percent(A)	Refractive Index
0	0,00	1.3676
5	5.09	1.3720
10	10.18	1.3765
15	15.25	1.3809
20	20.31	1.3853
25	25.37	1.3897
30	30.41	1.3941
35	35.44	1.3981
40	40.46	1.4029
45	45.48	1.4073
50	50.49	1.4116
55	55.48	1.4160
60	60.46	1.4203
65	65.44	1.4247
70	70.41	1.4290
75	75.36	1.4333
80	80.31	1.4376
8 5	85.24	1.4419
90	90.17	1.4462
95	95.09	1.4505
100	100.00	1.4548

Table 11. Carbon Tetrachloride(A)-Ethylacetate(B) Mixture

Mole Per	cent(A)		
Vapor	Liquid	Temperature,°C	
0	0.0	76.5	
10	9.5	75.8	
20	17.9	75.2	
30	28.4	74.7 Data at 745 mm H	q
40	37.3	74.3	5
50	44.0	74.1	
60	58.0	74.1	
70	68.2	74.3	
85	83.9	74.9	
100	100.0	75.9	

Minimum boiling point azeotropic binary mixture at 43.0 mole percent(A) and 74.75°C.

Mole Percent(A) = (1146.79)*(Refractive Index) - 1568.35

Position		Bott	om			Mid	dle	
Frequency	<u>T</u> 0	<u>T</u> b	Ro	<u>R</u> b	<u>T</u> o	<u>T</u> b	<u>R</u> o	<u>R</u> b
20 Kcps	72.1	74.1	1.3980	1.4165	72.1	74.1	1.3980	1.4165
25 Kcps	72.1	74.1	1.3989	1.4165	72.1	74.1	1.3995	1.4165
30 Kcps	72.1	74.1	1.3995	1.4165	72.1	74.1	1.4004	1.4165
35 Kcps	72.1	74.1	1.4001	1,4165	72.1	74.1	1.4013	1.4165
40 Kcps	72.1	74.1	1.4006	1.4165	72.1	74.1	1.4020	1.4165
45 Kcps	72.1	74.1	1.4011	1.4165	72.1	74.1	1.4027	1.4165
50 Kcps	72,1	74.1	1.4017	1.4165	72.2	74.1	1,4032	1.4165
55 Kcps	72.2	74.1	1.4023	1.4165	72.3	74.1	1.4037	1.4166
60 Kcps	72.2	74.1	1.4026	1.4165	72.3	74.1	1.4042	1.4166
65 Kcps	72.2	74.1	1.4031	1.4165	72.3	74.1	1.4045	1.4166
70 Kcps	72.3	74.1	1.4035	1.4166	72.3	74.1	1.4047	1.4166
75 Kcps	72.3	74.1	1.4039	1.4166	72.3	74.1	1.4049	1.4166
80 Kcps	72.3	74.1	1.4041	1.4166	72.3	74.1	1.4051	1.4167
85 Kcps	72.3	74.1	1.4045	1.4166	72.4	74.1	1.4050	1.4167
90 Kcps	72.3	74.1	1.4048	1.4166	72.4	74.1	1.4050	1.4167
95 Kcps	72.3	74.1	1.4050	1.4167	72.4	74.1	1.4050	1.4167
100 Kcps	72,4	74.1	1.4051	1.4167	72.4	74.1	1.4051	1.4167

Table 12. Carbon Tetrachloride (A)-Ethylacetate (B) Mixture Experimental Data

Table 12. Carbon	Tetrachloride	(A)-Ethylacetate	(B) Mixture Experimental	Data
Position				
Frequency	<u>T</u> o	<u> </u>	<u>R</u> o	<u>R</u> b
20 Kcps	72.1	74.1	1.3983	1.4165
25 Kcps	72.1	74.1	1.4007	1.4165
30 Kcps	72.1	74.1	1.4018	1.4165
35 Kcps	72.2	74.1	1.4026	1.4165
40 Kcps	72.2	74.1	1.4031	1.4165
45 Kcps	72.3	74.1	1.4036	1.4165
50 Kcps	72.3	74.1	1.4038	1.4166
55 Kcps	72.3	74.1	1.4044	1.4166
60 Kcps	72.3	74.1	1.4048	1.4166
65 Kcps	72.3	74.1	1.4051	1.4166
70 Kcps	72.3	74.1	1.4050	1.4166
75 Kcps	72.4	74.1	1.4050	1.4166
80 Kcps	72.4	74.1	1.4051	1.4166
85 Kcps	72.4	74.1	1.4051	1.4167
90 Kcps	72.4	74.1	1.4050	1.4167
95 Kcps	72.4	74.1	1.4053	1.4167
100 Kcps	72.4	74.1	1.4055	1.4167

Position	E	Bottom]	Middle		
Frequency	<u>-</u> А	FA	<u>Х</u> А	<u>Y</u> A	FA	<u>×</u> A	
20 Kcps	34.86	0.811	56.08	34.86	0.811	56.08	
25 Kcps	35.89	0.835	56.08	36.59	0.851	56.08	
30 Kcps	36.58	0.851	56.08	37.61	0.875	56.08	
35 Kcps	37.27	0.867	56.08	38.65	0.899	56.08	
40 Kcps	37.84	0.880	56.08	39.45	0.917	56.08	
45 Kcps	38.42	0.893	56.08	40.25	0.936	56.08	
50 Kcps	39.11	0.909	56.08	40.83	0.949	56.08	
55 Kcps	39.79	0.925	56.08	41.40	0.963	56.19	
60 Kcps	40.14	0.933	56.08	41.97	0.976	56.19	
65 Kcps	40.71	0.947	56.08	42.32	0.984	56.19	
70 Kcps	41.17	0.957	56.19	42.55	0.989	56.19	
75 Kcps	41.63	0.968	56.19	42.78	0.995	56.19	
80 Kcps	41.86	0.973	56.19	43.00	1.000	56.31	
85 Kcps	42.32	0.984	56.19	42.89	0.997	56.31	
90 Kcps	42.66	0.992	56.19	42.89	0.997	56.31	
95 Kcps	42.89	0.997	56.31	42.89	0.997	56.31	
100 Kcps	43.00	1.000	56.31	43.00	1.000	56.31	

Table 13. Carbon Tetrachloride (A)-Ethylacetate (B) Mixture Separation Factor

Table 13.	Carbon Tetrachloride	(A)-Ethylacetate (E	s) Mixture Separatio	n ractor
Position		Top	<u>p</u>	
Frequency	<u>Y</u> A	<u> </u>	Α	X _A
20 Kcps	35.2	1 0.	.819	56.08
25 Kcps	37.9	4 0.	.882	56.08
30 Kcps	39.2	2 0.	.912	56.08
35 Kcps	40.1	4 0.	.933	56.08
40 Kcps	40.7	1 0.	.947	56.08
45 Kcps	. 41.2	.8 0.	.960	56.08
50 Kcps	41.5	1 0.	.965	56.19
55 Kcps	42.2	0 0	.981	56.19
60 Kcps	42.6	6 0	.992	56.19
65 Kcps	43.0	10 1	.000	56.19
70 Kcps	42.8	<u>9</u> 0	.997	56.19
75 Kcps	42.8	39 0	.997	56.19
80 Kcps	43.0	1 0	.000	56.19
85 Kcps	43.0	0 1	.000	56.31
90 Kcps	42.8	39 0	.997	56.31
95 Kcps	42.2	23 1	.005	56.31
100 Kcps	43.4	16 1	.001	56.31

12 Courter Taturchlamide (A) Ethylacetate (B) Mixture Separation Factor

Volume Percent(A)	Mole Percent(A)	Refractive Index
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95	$\begin{array}{c} 0.00\\ 4.61\\ 9.27\\ 13.96\\ 18.68\\ 23.45\\ 28.26\\ 33.11\\ 37.99\\ 42.92\\ 47.89\\ 52.90\\ 57.96\\ 63.03\\ 68.20\\ 73.39\\ 78.62\\ 83.89\\ 89.21\\ 94.53\\ 100.00\\ \end{array}$	1.3543 1.3583 1.3583 1.3623 1.3663 1.3704 1.3744 1.3786 1.3827 1.3869 1.3912 1.3954 1.3997 1.4041 1.4084 1.4129 1.4173 1.4218 1.4264 1.4309 1.4352 1.402

Table 14. Chloroform(A)-Acetone(B) Mixture

Mole	Percent(A)	
Liquid	Vapor	Temperature, °C
0.00	0.00	56.2
0.00	1 70	50,2
8.00	4.10	57.5
14.10	8.35	58.3
20,45	13.12	59.4
26.12	17.65	60.4
33.67	24.95	61.6
42.50	35.20	62.8
52.29	48.30	63.9
73.40	76.30	64.4
78.92	82.40	63.8
86.25	90.00	63.1
88.92	93.50	62.8
100.00	100.00	61.3

Maximum boiling point azeotropic binary mixture at 65.5 mole percent(A) and 64.5°C.

Mole Percent(A) = (1164.14)*(Refractive Index) - 1576.60

		<u>Table 15.</u>	Chlorofo	orm(A)-Aceto	one(B) Mix	ture Exper	imental Dat	ta
Position		Bottom				<u>Middle</u>		
Frequency	<u>T</u> o	<u>T</u> b	<u>R</u> o	<u>R</u> b	<u> </u>	Т _b	<u>R</u> o	R _b
20 Kcps	60.4	61.1	1.4104	1.3939	60.3	61.1	1.4105	1.3939
25 Kcps	60.4	61.1	1.4112	1.3938	60.3	61.1	1.4115	1.3938
30 Kcps	60.4	61.1	1.4120	1.3932	60.3	61.1	1.4125	1.3933
35 Kcps	60.5	61.1	1.4125	1.3930	60.4	61.1	1.4137	1.3929
40 Kcps	60.6	61.1	1.4129	1.3928	60.4	61.1	1.4148	1.3927
45 Kcps	60.6	61.1	1.4135	1.3927	60.4	61.1	1.4155	1.3925
50 Keps	60.6	61.1	1.4141	1.3926	60.4	61.1	1.4166	1.3923
55 Kcps	60.6	61.1	1.4144	1,3923	60.4	61.1	1.4169	1.3921
60 Kcps	60.6	61.1	1.4146	1.3923	60.5	61.1	1.4175	1.3920
65 Kcps	60.6	61.1	1.4148	1.3922	60.6	61.1	1.4180	1.3 9 19
70 Kcps	60.6	61.1	1.4150	1.3921	60.6	61.1	1.4186	1.3920
75 Kcps	60.6	61.1	1.4152	1.3920	60.6	61.1	1.4193	1.3920
80 Kcps	60.6	61.1	1.4155	1.3919	60.6	61.1	1.4200	1.3920
85 Kcps	60.6	61.1	1.4158	1.3921	60.6	61.1	1.4210	1.3920
90 Kcps	60.7	61.1	1.4164	1.3920	60.7	61.2	1.4219	1.3920
95 Kcps	60.6	61.1	1.4169	1.3920	60.6	61.2	1.4231	1.3920
100 Kcps	60.6	61.1	1.4173	1.3920	60.8	61.2	1.4241	1.3920

	TADIE 13. OIII	Drotoriii(A) - Acecone(B) mixture Exper	"Illental Data - Con
Position		Тор		
Frequency	<u> </u>	<u>т</u> ь	Ro	<u>R</u> b
20 Kcps	60.5	61.1	1.4105	1.3939
25 Kcps	60.5	61.1	1.4123	1.3937
30 Kcps	60.5	61.1	1.4140	1.3930
35 Kcps	60.6	61.1	1.4152	1.3927
40 Kcps	60.6	61.1	1.4163	1.3925
45 Kcps	60.8	61.1	1.4171	1.3923
50 Kcps	60.8	61.2	1.4180	1.3922
55 Kcps	60.8	61.1	1.4190	1.3920
60 Kcps	60.8	61.1	1.4200	1.3921
65 Kcps	60.9	61.1	1.4210	1.3921
70 Kcps	60.8	61.1	1.4219	1.3920
75 Kcps	60.9	61.2	1.4228	1.3920
80 Kcps	60.9	61.2	1.4236	1.3919
85 Kcps	60.9	61.2	1.4246	1.3919
90 Kcps	60.9	61.2	1.4256	1.3920
95 Kcps	60.9	61.2	1.4271	1.3918
100 Kcps	61.0	6.14	1.4283	1,3918

.

		Table]	6. Chlor	oform(A)-A	cetone(B)	Mixture Se	paration	Factors	
Position		Bottom			<u>Middle</u>			Тор	
Frequency	<u>Y</u> A	<u> </u>	XA	YA	FA	XA	<u>Υ</u> _Α	FA	XA
20 Kcps	63.30	0.997	46.09	65.42	0.999	46.09	65.42	0.999	46.09
25 Kcps	66.23	1.011	45.98	66,58	1.017	45.98	67.51	1.031	45.86
30 Kcps	67.17	1.025	45.28	67.75	1.034	45.40	69.49	1.061	45.05
35 Kcps	67.75	1.034	45.05	69.14	1.056	44.93	70.89	1.082	44.70
40 Kcps	68.21	1.041	44.81	70.43	1.075	44.70	72.17	1.102	44.46
45 Kcps	68.91	1.052	44.70	71.24	1.088	44.46	73.10	1.116	44.23
50 Kcps	69.61	1.063	44.58	72.52	1.107	44.23	74.15	1.132	44.12
55 Kcps	69.96	1.068	44.23	72.87	1.113	44.00	75.31	1.150	43.80
60 Kcps	70.19	1.072	44.23	73.57	1.123	43.88	76.48	1.168	44.00
65 Kcps	70.43	1.075	44.12	74.15	1.132	43.77	77.64	1.185	44.00
70 Kcps	70.66	1.079	44.00	74.85	1.143	43.88	78.69	1.201	43.88
75 Kcps	70.89	1.082	43.88	75.66	1.155	43.88	79.74	1.217	43.88
80 Kcps	71.24	1,088	43.77	76.48	1.168	43.88	80.67	1.232	43.77
85 Kcps	71.59	1.093	44.00	77.64	1.185	43.88	81.83	1.249	43.77
90 Kcps	72.29	1.104	43.88	78.69	1.201	43.88	83.00	1.267	43.88
95 Kcps	72.87	1.113	43.88	80.09	1.223	43.88	84.74	1.294	43.65
100 Köps	73.34	1.119	43.88	81.25	1.240	43.88	86.14	1.315	43.65

Volume Percent(A)	Mole Percent(A)	Refractive Index
0 5	0.00 5.52	1.4949 1.4919 1.4889
15 20 25	16.39 21.74 26.88	1.4859 1.4859 1.4830
23 30 35	32.26 37.43	1.4773 1.4744 1.4716
40 45 50	42.55 47.62 52.63	1.4689 1.4661
55 60 65 70	62.50 67.36 72.77	1.4607 1.4581 1.4551
70 75 80	77.56 81.63	1.4551 1.4525 1.4503
89 90 95 100	80.29 90.91 95.48 100.00	1.4473 1.4452 1.4427 1.4402

Table I/. Unioroiorn(A)-benzene(b) Mixu	able I/.	e I/. Chloroform(A)-Benzene(B) MIXTUR
---	----------	--------------------	------------	-----------

Mole	Percent(A) Vapor	Tomporating
LIQUIU	Vapoi	remperature, e
0	0	80.6
8	10	79.8
15	20	79.0
22	30	78.2
29	40	77.3
36	50	76.4
44	60	75.3
54	70	74.0
66	80	71.9
79	90	68.9
100	100	61.4

Mole Percent(A) = 2732.91 - (1828.15)*(Refractive Index)

				2011-0110(2)		Experiment	cui Ducu	
Position		Bottom				M	iddle_	
Frequency	To	Tb	Ro	Rb	To	Tb	Ro	<u>Rb</u>
20 Kcps	67.4	77.5	1.4535	1.4696	67.4	77.5	1.4535	1.4696
25 Kcps	67.1	77.5	1.4535	1.4696	67.3	77.5	1.4535	1.4695
30 Kcps	66.9	77.5	1.4535	1.4695	66.8	77.5	1.4532	1.4696
35 Kcps	66.7	77.5	1.4533	1.4695	66.1	77.5	1.4528	1.4695
40 Kcps	66.4	77.5	1.4531	1.4695	65.6	77.5	1.4523	1.4695
45 Kcps	65.7	77.5	1.4525	1.4695	65.1	77.5	1.4517	1.4696
50 Kcps	65.4	77.5	1.4520	1.4695	64.7	77.5	1.4510	1.4696
55 Kcps	65.0	77.5	1.4514	1.4695	64.0	77.5	1.4499	1.4696
60 Kcps	64.1	77.5	1.4507	1.4695	63.6	77.5	1.4487	1.4696
65 Kcps	63.7	77.5	1.4499	1.4696	63.1	77.5	1.4476	1.4697
70 Kcps	63.0	77.5	1.4492	1.4696	62.4	77.5	1.4460	1.4696
75 Kcps	62.8	77.5	1.4483	1.4696	61.8	77.5	1.4445	1.4696
80 Kcps	62.4	77.5	1.4476	1.4695	61.0	77.5	1.4435	1.4697
85 Kcps	61.2	77.5	1.4468	1.4695	60.7	77.5	1.4427	1.4696
90 Kcps	60.9	77.5	1.4459	1.4695	60 . 3	77.5	1.4420	1.4696
95 Kcps	60.7	77.5	1.4448	1.4696	60.1	77.5	1.4416	1.4695
100 Kcps	60.2	77.5	1.4443	1.4696	60.2	77.5	1.4420	1.4695

Table 18. Chloroform(A)-Benzene(B) Mixture Experimental Data

		То	<u>p</u>	
Frequency	To	Tb	Ro	<u>Rb</u>
20 Kcps	67.4	77.5	1.4533	1.4695
25 Kcps	67.0	77.5	1.4526	1.4695
30 Kcps	66.7	77.5	1.4519	1.4695
35 Kcps	65.3	77.5	1.4511	1.4696
40 Kcps	64.1	77.5	1.4501	1.4696
45 Kcps	63.6	77.5	1.4486	1.4696
50 Kcps	63.1	77.5	1.4470	1.4696
55 Kcps	62.6	77.5	1.4460	1.4697
60 Kcps	62.1	77.5	1.4443	1.4696
65 Kcps	61.7	77.5	1.4434	1.4697
70 Kcps	60.8	77.5	1.4427	1.4697
75 Kcps	60.2	77.5	1.4421	1.4697
80 Kcps	59.8	77.5	1.4418	1.4696
85 Kcps	59.7	77.5	1.4414	1.4697
90 Kcps	60.0	77.5	1.4416	1.4696
95 Kcps	60.3	77.5	1.4418	1.4696
100 Kcps	60.6	77.5	1.4423	1.4695

Table 18. Chloroform(A)-Benzene(B) Mixture Experimental Data - Cont'd

	Table 19. Ch	nloroform(A).	-Benzene(B)	Mixture Se	paration Fa	ct
		Bottom			<u>Middle</u>	
Frequency	YA	FA	XA	<u>YA</u>	FA	
20 Kcps	75.96	0.760	46.26	75.96	0.760	4
25 Kcps	75.96	0.760	46.26	75.96	0.760	46
30 Kcps	75.96	0.760	46.44	76.24	0.762	46
35 Kcps	76.06	0.761	46.44	76.97	0.770	46.
40 Kcps	76.43	0.764	46.44	77.89	0.779	46.
45 Kcps	77.52	0.775	46.44	78.98	0.790	46.
50 Kcps	78.44	0.784	46.44	80.26	0.803	46.2
55 Kcps	79.53	0.795	46.44	82.28	0.823	46.2
60 Kcps	80.81	0.808	46.44	84.47	0.845	46.2
65 Kcps	82.28	0.823	46.26	86.48	0.865	46.0
70 Kcps	83.56	0.836	46.26	89.41	0.894	46.2
75 Kcps	85.20	0.852	46.26	92.15	0.922	46.2
80 Kcps	86.48	0.865	46.44	93.98	0.940	46.0
85 Kcps	87.94	0.879	46.44	95.44	0.954	46.2
90 Kcps	89.59	0.896	46.44	96.72	0.967	46.2
95 Kcps	91.60	0.916	46.26	97.45	0.975	46.4
100 Kcps	92.51	0.925	46.26	97.08	0.971	46.4

		Top	
Frequency	YA	FA	XA
20 Kcps	76.06	0.761	46.44
25 Kcps	77.34	0.773	46.44
30 Kcps	78.62	0.786	46.44
35 Kcps	80.08	0.801	46.26
40 Kcps	81.91	0.819	46.26
45 Kcps	84.69	0.847	46.26
50 Kcps	87.58	0.876	46.26
55 Kcps	89.41	0.894	46.08
60 Kcps	92.51	0.925	46.26
65 Kcps	94.16	9.942	46.08
70 Kcps	95.44	0.954	46.08
75 Kcps	96.53	0.965	46.08
80 Kcps	97.08	0.971	46.26
85 Kcps	97.81	0.978	46.08
90 Kcps	97.45	0.975	46.26
95 Kcps	97.08	0.971	47.26
100 Kcps	96.72	0.967	46.44

Volume Percent(A)	Mole Percent(A)	Refractive Index
0 5 10 15 20 25 30 35 40 45 50 55 60 65 70 75 80 85 90 95 100	$\begin{array}{c} 0.00\\ 3.70\\ 7.51\\ 11.43\\ 15.45\\ 19.59\\ 23.85\\ 28.24\\ 32.77\\ 37.43\\ 42.23\\ 47.19\\ 52.30\\ 57.28\\ 63.04\\ 68.68\\ 74.52\\ 80.55\\ 86.81\\ 93.28\\ 100.00\\ \end{array}$	$\begin{array}{c} 1.3574\\ 1.3605\\ 1.3636\\ 1.3669\\ 1.3702\\ 1.3736\\ 1.3771\\ 1.3808\\ 1.3845\\ 1.3884\\ 1.3924\\ 1.3924\\ 1.3965\\ 1.4071\\ 1.4051\\ 1.4096\\ 1.4143\\ 1.4191\\ 1.4241\\ 1.4293\\ 1.4346\\ 1.4402\end{array}$
Mole Percent(B)	Temperature,°C	
0 7 15 30 40 50 60 70 80 90 100	60.95 59.1 59.6 61.4 63.3 65.7 68.4 71.0 73.6 75.8 77.9	

۵

Table 20. Chloroform(A)-Ethanol(B) Mixture

Minimum	boiling	point	azeotropic	binary	mixture	at	84	mole	percent(A)
and 59.3	3°C. Š	•	r ,	•					•	•

Mole Percent(A) = (1207.73)*(Refractive Index) - 1639.37

Position		B	ottom			Mi	ddle_	
Frequency	Тo	Tb	Ro	Rb	To	<u>Т</u> ь	<u>R</u> o	Rb
20 Kcps	58.2	61.6	1.4123	1.3865	58.2	61.6	1.4123	1.3865
25 Kcps	58.2	61.6	1.4133	1.3865	58.1	61.6	1.4134	1.3865
30 Kcps	58.2	61.6	1.4141	1.3865	58.0	61.6	1.4145	1.3865
35 Kcps	58.2	61.6	1.4146	1.3865	58.0	61.6	1.4154	1.3865
40 Kcps	58.1	61.6	1.4152	1.3865	58.0	61.6	1.4163	1.3865
45 Kcps	58.1	61.6	1.4156	1.3865	58.0	61.6	1.4171	1.3865
50 Kcps	58.2	61.6	1.4158	1.3865	58.0	61.6	1.4181	1.3866
55 Kcps	58.2	61.6	1.4163	1.3866	57.9	61.6	1.4190	1.3865
60 Kcps	58.2	61.6	1.4172	1.3866	57.8	61.6	1.4199	1.3865
65 Kcps	58.2	61.6	1.4173	1.3865	57.5	61.6	1.4206	1.3865
70 Kcps	58.2	61.6	1.4173	1.3865	57.6	61.6	1.4211	1.3866
75 Kcps	58.2	61.6	1.4174	1.3865	57.5	61.6	1.4215	1.3866
80 Kcps	58.2	61.6	1.4174	1.3865	57.3	61.6].4222	1.3865
85 Kcps	58.2	61.6	1.4175	1.3866	57.2	61.6	1.4231	1.3865
90 Kcps	58.2	61.6	1.4175	1.3865	57.1	61.6	1.4236	1.3866
95 Kcps	58.2	61.6	1.4175	1.3865				
100 Kcps	58.1	61.6	1.4176	1.3865				

Table 21. Chloroform(A)-Ethanol(B) Mixture Experimental Data

<u>Table 21. (</u>	<u>Chloroform(A)-Eth</u>	anol(B) Mixture	Experimental Data	- Cont'd
Position			Тор	
Frequency	To	<u>T</u> b	Ro	Rb
20 Kcps	58.2	61.6	1.4125	1.3865
25 Kcps	58.1	61.6	1.4149	1.3865
30 Kcps	58.1	61.6	1.4174	1.3865
35 Kcps	58.0	61.6	1.4183	1.3866
40 Kcps	57.8	61.6	1.4203	1.6866
45 Kcps	57.5	61.6	1.4216	1.3865
50 Kcps	57.4	61.6	1.4229	1.3865
55 Kcps	57.2	61.6	1.4229	1.3865
60 Kcps	57.1	61.6	1.4236	1.3865
65 Kcps				
70 Kcps	an an an			
75 Kcps				
80 Kcps				
85 Kcps				
90 Kcps	×			
95 Kcps				
100 Kcps				

	Bottom			Middle		
Position		<u> </u>	Хл	ΥA	FA	<u>×</u> A
Frequency	<u>Y</u> A	<u>r</u> A	<u>^</u> A	<u> </u>	0.789	35,15
20 Kcps	66.31	0.789	35.15	00.31	0.705	05.15
25 Kcps	67.51	0.803	35.15	67.64	0.805	35.15
30 Kcps	68.48	0.815	35.15	68.96	0.821	35.15
35 Kcps	69.08	0.822	35.15	70.05	0.834	35.15
40 Kcps	69.81	0.831	35.15	71.14	0.847	35.15
45 Kcps	70.29	0.837	35.15	72.10	0.858	35.15
50 Kcps	70.53	0.839	35.15	73.31	0.872	35.27
55 Kcps	71.14	0.847	35.27	74.40	0.885	35.15
60 Keps	72.22	0.859	35.27	75.49	0.898	35.15
65 Kcps	72.35	0.861	35.15	76.33	0.908	35.35
70 Kons	72.35	0.861	35.15	76.94	0.916	35.27
75 Kons	72.47	0.862	35.15	77.42	0.921	35.27
80 Kons	72.5]	0.863	35.15	78.26	0.931	35.15
85 Kons	72.59	0.864	35.27	79.35	0.944	35.15
00 Keps	72 59	0.864	35.15	79.95	0.951	35.27
DE Kons	72 59	0.864	35.15			
as vchz	16.00	0.004				
100 Kcps	72.71	0.865	35.15			

Table 22. Chloroform(A)-Ethanol(B) Mixture Separation Factors

Position		Тор	
Frequency	YA	FA	<u>X</u> A
20 Kcps	66.55	0.791	35.15
25 Kcps	69.48	0.827	35.15
30 Kcps	72.47	0.862	35.15
35 Kcps	73.55	0.875	35.27
40 Kcps	75.97	0.904	35.27
45 Kcps	77.54	0.923	35.15
50 Kcps	79.11	0.941	35.15
55 Kcps	79.11	0.941	35.15
60 Kcps	79.95	0.951	35.15
65 Kcps			~~ ~~
70 Kcps	_~		
75 Kcps	_~		
80 Kcps			
85 Kcps			
90 Kcps			
95 Kcps			
100 Kcps			

Volume Percent(A)	Mole Percent(A)	Refractive Index
0	0.00	1,3314
5	1.59	1,3318
10	3.31	1,3323
15	5.15	1.3327
20	7.15	1.3333
25	9.29	1.3338
30	11.66	1.3344
35	14.23	1.3351
40	17.03	1.3358
45	20.12	1.3366
50	23.54	1.3375
55	27.36	1.3385
60	31.61	1.3396
65	36.39	1.3409
70	41.82	1.3423
75	48.03	1.3439
80	55.18	1.3457
85	63.59	1.3479
90	73.48	1.3505
95	85.39	1.3536
100	100.00	1.3574

Table 23. Ethanol(A)-Water(B) Mixture

Mole P	Percent(A)	TABLES LUSI - OA
LIQUID	vapor	Temperature, 1
0.00	0.00	100.0
1.90	17.00	95.5
7.21	38.91	89.0
9.66	43.75	86.7
12.38	47.04	85.3
16.61	50.89	84.1
23.37	54.45	82.7
26.08	55.80	82.3
32.73	58.26	81.5
39.65	61.22	80.7
50.79	65.64	/9.8
51.98	65.99	/9./
57.32	68.41	/9.3
67.63	/3.85	/8./4
/4./2	/8.15	/8.41
89.43	89.43	/8.15

Mole Percent(A) = (3846.15)*(Refractive Index) - 5120.77
Position		B	ottom			M	iddle	
Frequency	To	<u>Т</u> р	Ro	Rb	Τo	Тb	Ro	Rb
20 Kcps	76.2	87.4	1.3507	1.3335	76.2	87.4	1.3507	1.3335
25 Kcps	76.3	87.4	1.3507	1.3335	76.2	87.4	1.3512	1.3335
30 Kcps	76.2	87.3	1.3508	1.3335	76.3	87.3	1.3515	1.3335
35 Kcps	76.1	87.3	1.3511	1.3335	76.2	87.4	1.3521	1.3335
40 Kcps	76.0	87.4	1.3516	1.3335	76.1	87.3	1.3526	1.3335
45 Kcps	75.8	87.4	1.3520	1.3335	75.8	87.4	1.3530	1.3336
50 Kcps	75.8	87.4	1.3521	1.3335	75.6	87.3	1.3532	1.3335
55 Kcps	75.8	87.3	1.3521	1.3335	75.6	87.3	1.3534	1.3335
60 Kcps	75.8	87.3	1.3522	1.3336	75.6	87.3	1.3535	1.3336
65 Kcps	75.7	87.3	1.3523	1.3335	75.4	87.4	1.3537	1.3336
70 Kcps	75.8	87.3	1.3523	1.3335	75.4	87.4	1.3538	1.3336
75 Kcps	75.8	87.3	1.3524	1.3335	75.5	87.4	1.3539	1.3335
80 Kcps	75.8	87.4	1.3525	1.3336	75.6	87.4	1.3540	1.3335
85 Kcps	75.8	87.3	1.3526	1.3336	75.6	87.4	1.3542	1.3336
90 Kcps	75.8	87.4	1.3527	1.3335	75.7	87.4	1.3542	1.3335
95 Kcps	75.9	87.4	1.3527	1.3335	75.8	87.4	1.3544	1.3336
100 Kcps	76.1	87.4	1.3528	1.3335	76.0	87.4	1.3545	1.3336

Table 24. Ethanol(A)-Water(B) Mixture Experimental Data

			Ton	
POSITION			<u>10p</u>	
Frequency	<u>T</u> o	<u>T</u> b	Ro	Rь
20 Kcps	76.2	87.5	1.3508	1.3336
25 Kcps	75.9	87.6	1.3511	1.3336
30 Kcps	75.3	87.5	1.3517	1.3337
35 Kcps	74.8	87.4	1.3534	1.3337
40 Kcps	74.1	87.6	1.3547	1.3336
45 Kcps	74.2	87.5	1.3564	1.3336
50 Kcps	74.4	87.5	1.3567	1.3336
55 Kcps	74.6	87.5	1.3567	1.3337
60 Kcps	75.0	87.4	1.3568	1.3337
65 Kcps	75.0	87.4	1.3569	1.3337
70 Kcps	75.2	87.3	1,3568	1.3336
75 Kcps	75.1	87.5	1.3568	1.3336
80 Kcps	75.3	87.4	1.3567	1.3336
85 Kcps	75.3	87.4	1.3568	1.3336
90 Kcps	75.2	87.4	1.3569	1.3337
95 Kcps	75.2	87.4	1.3569	1.3336
100 Kcps	75.3	87.4	1.3569	1.3337

Table 24. Ethanol(A)-Water(B) Mixture Experimental Data - Cont'd

	Table 25.	Ethanol(A)	-Water(B) Mis	ture Separat	ion Factors	
Position		Bottom			Middle	
Frequency	Υд	FA	<u>X</u> A	YA	<u> </u>	XA
20 Kcps	 74.23	0.950	8.07	74.23	0.950	8.07
25 Kops	74.23	0.950	8.07	76.15	0.975	8.07
20 Kops	74 61	0.955	8.07	77.30	0.989	8.07
25 Kops	75 76	0.970	8.07	79.61	1.019	8.07
35 KCps	77 69	0.994	8.07	81.53	1.044	8.07
40 Kcps	70 22	1 014	8.07	83.07	1.063	8.46
45 KCps	70 61	1 019	8.07	83.84	1.073	8.07
50 KCps	79.01	1.019	8 07	84.61	1.083	8.07
55 Kcps	79.01	1 02/	8 46	84,99	1.088	8.46
60 Kcps	/9.99	1.024	0.70 0.70	85 76	1.098	8.46
65 Kcps	80.38	1.029	0.07	86 15	1.103	8.46
70 Kcps	80.38	1.029	8.07	00.13	1 108	8.07
75 Kcps	80.76	1.034	8.07	86.53	1.100	0.07
80 Kcps	81.15	1.039	8.46	86.92	1.113	8.07
85 Kcps	81.53	1.044	8,46	87.69	1.122	8.46
90 Kcps	81.92	1.049	8.07	87.69	1.122	8.07
95 Kcps	81.92	1.049	8.07	88.46	1.132	8.46
100 Kcps	82.30	1.053	8.07	88,84	1.137	8.46

		Тор	
Emoquency	Y۵	FA	XA
Frequency	<u>.</u>	0.956	8.46
20 Kcps	74.00	0 972	8.46
25 Kcps	/5.94	0.572	8 84
30 Kcps	78.02	0.999	0.04
35 Kcps	84.55	1.082	8.84
40 Kcps	89.45	1.145	8.46
45 Kops	96.15	1.231	8.46
FO Kops	97.30	1.245	8.46
50 Keps	97.30	1.245	8.84
55 KCps	07.69	1.250	8.84
60 Kcps	97.09	1 255	8.84
65 Kcps	98.07	1.250	8.46
70 Kcps	97.69	1.250	9.16
75 Kcps	97.69	1.250	0.10
80 Kcps	97.30	1.245	8.46
85 Kcps	97.69	1.250	8.46
90 Kcps	98.07	1.255	8.84
95 Kons	98.07	1.255	8.46
100 Keps	98.07	1.255	8.84
too keps	20111		

ANALYSIS OF PACKING

The column was operated for 107 hours under normal or ultrasonic conditions. At the end of this period the packing was removed and examined for breakage. This examination revealed 4 berl saddles had been broken in the areas of the transducer (2 at the bottom, and 1 each at the middle and top). Because the packing was loaded with the column filled with water this breakage is a result of operation.

According to actual hand count there were 7,233 berl saddles loaded; this would mean a 0.0553% breakage due to operation. In most industrial applications ceramic packing is not used because of the superiority of plastic or metal packing; however, ceramic packing even when broken, offers good characteristics for separation. Packing reduced to dust can cause the pressure drop to increase substantially but there was no evidence of total destruction of any berl saddles because by actual count all the packing was accounted for.

Assuming approximately 3% breakage before the packing would be examined and/or replaced, an on stream time of 242 days of continuous operation can be expected. Of course the rate of packing deterioration may increase or decrease during operation and changing to a more flexible packing such as polypropylene should increase the on stream time; however, even the present operation can be used on an industrial "level without incurring prohibitive initial and operating costs.

DISCUSSION AND EVALUATION OF RESULTS

Six of the seven mixtures used for experimentation have azeotropic limitation so this composition at ambient pressure is defined as a separation factor of 1.000. For full range mixtures a separation factor of 1.000 corresponds to 100 mole percent in the overhead vapor.

Because the transducers are able to produce caviation in the liquid and sound waves in the vapor, it is assumed that these phenomena occur; however, observation of these phenomena are impossible when the column is lagged with asbestos tape. When the column was filled with water and before the ceramic packing was loaded, the two middle transducers were driven at 50 Kcps producing bubbles in the water which persisted long after residual dissolved air was liberated, indicating caviation was occuring near the wall. Combinations of three transducers were tried but no caviation was detected so only pairs of transducers were used during actual experimentation.

No measurements were made of actual power output of the amplifier which is rated at 50 watts because the determination of how much of this power was actually absorbed by the packing and the process streams was impossible. The use of more sophisticated techniques of sampling and power measurement could yield a correlation relating power consumption and separation which could be used to make an economic comparison of ultrasonic operation and other alternatives.

The energy relationships which may be used for transducers at frequencies well below resonance are:



$$f \checkmark ^{1/}(R \ast C_{o}):$$

for R \checkmark Z_{o}:
$$W = \frac{V^{2}}{R} = (F_{avg} g_{33} \frac{T}{A})^{2} R / (R + Z_{o})^{2} watts$$

for R \ll Z_{o}:
$$W = (2 f^{d} 33 F_{avg})^{2} R watts$$

for t < R *C_{o}:
$$E = 1/2(C_{o}V_{oc}) = 1/2 C_{o} \ast (F_{peak}g_{33} \frac{T}{A}) Joules$$

for short circuit:
$$E = (F_{avg} d_{33})^{2} R_{L}/t Joules$$

The results of the full range mixture are presented in Figure 9. and exhibit the same pattern as Figure 10., except there is a characteristic frequency at which increasing frequency has a negative effect on separation. This frequency must correspond to a pole (using an electrical analogy). The other systems probably have this same characteristic at some frequency outside the range used during this experimentation.

The minimum boiling azeotropic mixtures presented in the graphs (Figures 6., 7., 8., 11) show the overhead composition does not improve with increasing frequency once the azeotropic composition is reached. The slopes (dF_A/df) of the graphs can be related to the ease of separation below the azeotrope. For example, Figure 6. shows the slope remaining relatively constant for frequencies between 30-80 Kcps and independent of transducer position. Figures 7. and 11. show a fairly constant slope over the same frequency range but dependent on the transducer position. Figure 8. shows the most curvature over the entire frequency range indicating the slope is dependent on frequency and position. System 7., shown in Figure 12., is an exception to this pattern because the azeotropic temperature is less than 1°C from the normal boiling point of ethanol and small variations in pressure can affect this condition.

The maximum boiling azeotropic binary shown in Figure 10. has predictable results because decreasing temperature is favorable to continued separation once the azeotrope is passed. The slopes of



Figure 6; Separation Factor vs Frequency for C6H6C2H50H











Figure 9. Separation Factor vs Frequency for CHCl3-C3H60





74.



Figure 12. Separation Factor vs Frequency for C2H5OH-H2O

these curves indicate continued separation can be expected as frequency is increased.

In conclusion, the experimentation has proven that improved separation will result if a packed column is excited by externally supplied ultrasonic energy. The principles which govern this improvement in separation are not well defined but can be partially explained by a combination of the following.

- The velocity of the vapor and its direction are affected by the generation of high frequency sound waves and shock waves caused by caviation of the liquid.
- The liquid loading as well as the path of the descending liquid are modified by the caviation of the liquid.
- 3. The arrangement of the packing is altered during ultrasonic operation which is evidenced by the presence of broken berl saddles in the areas of the transducers.

The above mentioned conditions are probably only a few of the many phenomena which occur in this quasi-steady state process. Only further experimentation can answer the questions which this experimentation has uncovered.

AUTHOR'S COMMENTS ON SAFETY

<u>Extreme caution</u> must be taken when using high voltage amplifiers and flammable components. It must be noted that a transducer is a charged capacitor, and even though air, epoxy and glass are poor conductors, there is leakage which means a constant potential for arcing is present.

In order to achieve steady operation the system must be maintained at constant pressure, which in the case of the packed column meant using an atmospheric vent. Care must be taken to insure that flammable or toxic vapors do not accumulate causing a potential fire or health hazard.

Because the frequency is above the audible range, fatigue problems in the connections can be expected. All fittings should be epoxyed in place to decrease the possibility of leaking flammable or toxic streams.

If further experimentation is planned on this piece of equipment or similar apparatus, a protective enclosure as well as inert blanketing should be provided to insure the safety of the operator.

RECOMMENDATIONS

Further study in the application of ultrasonic energy sources to chemical processes could prove to be quite rewarding. This thesis has presented the "how" but still leaves unanswered the "why" of separational improvement by the use of ultrasonic vibrations. Because the liquid and vapor in a small packed column are hard to observe (impossible when the column is thermally insulated), it is not certain which phase is more affected by the excitement of the ultrasonic vibrations. It may prove very interesting to use a glass bubble cap column with organic compound having color to visually observe the actual separation in the areas of the transducers.

Externally supplied ultrasonic energy can also be adapted to other chemical processes such as extraction and adsorption with a resulting increase in efficiency. The only foreseeable limitation on the use of ultrasonic energy would be the prohibitive consumption of power because of the low efficiency of conversion from electric to mechanical energy in large units.

Table 26. Conversion Factors

Multiply	By	<u>To Obtain</u>
Meters	39.370	Inches
Meters	2.281	Feet
Meters ²	1550	Inches2
Meters ²	10.76	Feet ²
Centimeters ²	0.1550	Inches ²
Meters ³	61,020	Inches ³
Meters ³	35.31	Feet ³
Centimeters ³	0.06102	Inches ³
Feet ³	1728	Inches ³
Newtons	105	Dynes
Dynes	1.020*10-6	Kilogram
Kilogram	2.205	Pounds
Newtons	0.2248	Pounds
Grams	0.03527	Ounces
Dynes/cm ²	0.1	. Newtons/m ²
Dynes/cm ²	1.450*10-5	Pounds/in ²
Pounds/in ²	6895	Newtons/m ²
Grams/cm ³	1000	Kilogram/m ³
Pounds/in ³	27,680	Kilogram/m3
Pounds/ft ³	16.02	Kilogram/m ³
Pounds/in ³	27.68	Grams/cm ³

Symbol Designations

Symbol	Description
F _A	Separation Factor
R _b	Refractive Index of the Bottom Liquid
R _O	Refractive Index of the Overhead Vapor
Тb	Temperature of the Reboiler
To	Temperature of the Overhead Vapor
X _A	Bottom Liquid Composition
Y _A	Overhead Vapor Composition
٧	Voltage
Q	Electrical Charge
С	Capacitance
F	Force
T,W,L&D	Dimensions: Thickness, Width, Length and Diameter
dT,dL&dD	Small Changes in Dimensions
d33	Direct Charge Coefficient
d31	Transverse Charge Coefficient
d15	Shear Charge Coefficient
g33	Direct Voltage Coefficient
931	Transverse Voltage Coefficient
915	Shear Voltage Coefficient
Р	Direction of the Polar Axis
k33	Direct Electromechanical Coupling Coefficient
k ₃₁	Transverse Electromechanical Coupling Coefficient
k15	Shear Electromechanical Coupling Coefficient

Symbol	Designations	-	Cont'd

Symbol	Description
^k p	Planar Electromechanical Coupling Coefficient
K3	Relative Dielectric Constant Measured Along the Poling Axis
К	Relative Dielectric Constant Measured at Right Angles to the Poling Axis
Y _{ij}	Young's Modulus Measured at Constant Electric Field
Qm	Mechanical Q (Quality Factor)
Pr	Remanent Polarization
Ec	Coercive Field
Z _m	Impedance at Resonance
fr	Resonance Frequency
Ρ	Powerstat Reading
t	Time, Second

,

FOOTNOTES

- (1) Foust, Alan S. et al, <u>Principles of Unit Operations</u>, New York: John Wiley & Sons, Inc., 1960, pp. 267-270.
- (2) Chilton, T. H., and A. P. Colburn, <u>Industrial and Engineering</u> Chemistry, Vol. 27, No. 5, March 1935, pp. 255-260.
- (3) Molokanov, Yu. M., <u>Theoretical Foundations of Chemical</u> Engineering, Vol. 1, No. 3, May-June 1967, pp. 261-266.
- (4) Grinevich, A. T., <u>Chemical and Petroleum Engineering</u>, No. 2, February 1965, pp. 105-108.
- (5) Silvery, F. C. and G. J. Keller, <u>Chemical Engineering Progress</u>, Vol. 62, No. 1, January 1966, pp. 68-74.
- (6) Wall, K. J., Chemical and Process Engineering, Vol. 48, No. 7, July 1967, pp. 56-60.
- (7) Mykolnikov, I. A. and L. S. Oshurkova, <u>Coke and Chemistry</u>, No. 3, 1964, pp. 51-52.
- (8) Eckert, J. S. et al, <u>Chemical Engineering Progress</u>, Vol. 60, No. 10, October 1964, pp. 71-72.
- (9) Morton, F. et al, <u>Institute of Chemical Engineers Transactions</u>, Vol. 42, No. 1, 1964, pp. 35-43.
- (10) Molyneux, F., <u>Chemical and Process Engineering</u>, Vol. 41, No. 2, February 1960, pp. 43-47.
- (11) Blyakhman, L. I. and L. S. Davydov, <u>Journal of Applied Chemistry</u> (USSR), Vol. 41, No. 12, December 1968, pp. 2774-2777.
- (12) Norman, W. S. et al, <u>Industrial Chemist</u>, Vol. 37, No. 2, February 1961, pp. 55-59.
- (13) Samsel, R. W., <u>Chemical Engineering Progress Symposium Series</u>, Vol. 47, No. 1, May 1950, pp. 77-81.
- (14) Stokes, C. A. and J. E. Vivian, <u>Chemical Engineering Progress</u> <u>Symposium Series</u>, Vol. 47, No. 1, May 1950, pp. 11-21.
- (15) Koenig, W. Annals of Physics, Vol. 42, No. 353, 1891, p. 549.
- (16) Kanser, H. W., <u>Chemical Engineering</u>, Vol. 57, No. 5, March 1950, pp. 158-163.

FOOTNOTES - continued

- (17) Sun, Shiou Chuam, Mining Engineering, 1951, pp. 865-867.
- (18) Bretsznajder, S. et al, <u>International Chemical Engineering</u>, Vol. 3, No. 4, October 1963, pp. 496-502.
- (19) Lesniewicz, L. et al, <u>Chemical Stoichiometry</u>, Vol. 3, 1958, p. 259.
- (20) McKittrich, et al, U.S. Patent 2,265,762, December 9, 1941.
- (21) Yasunago, T. et al, <u>International Congress on Acoustics</u>, Vol. 5, August 21-28, 1968, pp. 61-64.
- (22) Lewis, M. F., <u>Journal of Acoustical Society of America</u>, Vol. 44, No. 3, September 1968, pp. 713-16
- (23) McCarthy, W. S., <u>Adhesives Age</u>, Vol. 11, No. 7, July 1968, pp. 21-24.
- (24) Tani, Y., <u>Electronic & Communication in Japan</u>, Vol. 48, No. 11, November 1965, pp. 192-198.
- (25) Brewer, R. G., <u>Applied Physics Letters</u>, Vol. 6, No. 8, April 15, 1965, pp. 165-166.
- (26) Neppiras, E. A., <u>Ultrasonics</u>, Vol. 3, January-March 1965, pp. 9-17.

REFERENCES

Perry, J. H. et al, <u>Chemical Engineers' Handbook</u>, New York: McGraw-Hill Book Company, 1963.

Washburn, E. W. ed. et al, <u>International Critical Tables of Numerical</u> Data, Physics, Chemistry and Technology, New York: McGraw-Hill Book Company, 1926.

Weast, R. C., ed. et al, <u>Handbook of Chemistry and Physics</u>, <u>51st</u> edition, Cleveland, Ohio: The Chemical Rubber Company, 1971.

<u>Glennite Piezoceramics Catalog</u> H-700, Gulton Industries, Inc., Fullerton, California, 1970.

Tower Packings, Bulletin TP-71, Norton Chemical Process Products Division, Akron, Ohio, 1971.

Design Information for Packed Towers, Bulletin DC-10R, Norton Chemical Process Products Division, Akron, Ohio, 1971. t