Application of azeotropic distillation to the separation of olefins and paraffins
by Padmanabh D Datar

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The two pairs were 2,,2,4 - trimethyl pentane, 2,4.,4 - trimethyl pentene-1, and normal heptane - 2,4,4 - trimethyl pentene-1.

The degree to which a compound (called entrainer) helped separation was measured by the increase in the relative volatility of the particular pair of compounds being separated over the value obtained by distilling the pure compounds without entrainer.

Compounds picked as entrainers were those which contained oxygen, nitrogen or halogen atoms and were for these reasons likely to give large deviations from Raoult's Law in solution. The compounds were picked to boil within about ± 20°C of the hydrocarbons being separated. Twenty-two compounds satisfying these requirements were tried as possible entrainers.

Major pieces of equipment used included four identical distillation columns packed with Fenske rings and fitted with cold finger condensing head, a gas chromatograph, and a refractometer.

The evaluation of an entrainer consisted of determining whether or not an azeotrope was formed, determining the azeotropic composition, and determining the relative volatility between the hydrocarbons using the entrainer. Relative volatilities were calculated from the Fenske equation after overhead and bottom samples from the distillation run had been analysed with the Chromatograph. Duplicate runs were made for each relative volatility determination and seemed to agree reasonably well.

For both the systems studied, isopropyl acetate appears to be the best entrainer. It increases the relative volatility from 1.041 to 1.130 and decreases the number of theoretical plates required from 156 to 48 in the case of isooctane - 2,4,4 - trimethyl pentene -1 system. In the case of normal heptane - 2,4,4 - trimethyl pentene -1 system, it increases the relative volatility from 1.0460 to 1.129 and decreased the number of theoretical plates required from 131 to 49.

APPLICATION OF AZEOTROPIC DISTILLATION TO THE SEPARATION
OF OLEFINS AND PARAFFINS

by

PADMANABH D. DATAR

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ABSTRACT

The purpose of this investigation was to study the possible application of azeotropic distillation to the separation of olefins and paraffins. An attempt was made to enhance the ease of separation of two pairs of compounds by using azeotropic distillation. The two pairs were 2,2,4 - trimethyl pentane, 2,4,4 - trimethyl pentene-1, and normal heptane - 2,4,4 - trimethyl pentene-1.

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INTRODUCTION

The purpose of this investigation was to study the possible application of azeotropic distillation to the separation of paraffins and olefins boiling close together.

Separation of paraffins and olefins is a very common problem in the petroleum and chemical industry. The separation studied in this investigation was not prompted by any specific problem but is a continuation of this kind of work done in the past at this university.

Three compounds selected for study are 2,2,4 - trimethyl pentane, normal-heptane and 2,4,4 - trimethyl pentene -1 having normal boiling points of 99.2°C, 98.4°C and 102°C respectively.

The most common method of separation of miscible liquids are fractional crystallization, solvent extraction and distillation. The distillation method is by far the most widely used for hydrocarbons, when their boiling points are different. When the boiling points are close a very tall column is required making the process uneconomical.

The purpose of azeotropic distillation is to reduce the number of theoretical plates required for a desired separation thus cutting down the equipment and operating cost to a considerable extent.

Along with its merit in reducing the height of distillation column required for a desired separation, azeotropic distillation introduces a new problem of separating the entrainer from the original compounds.

In general water washing and other physical separation methods can be suggested. Though this problem considerably affects the value of a particular compound as an entrainer, no attempt will be made here to resolve it. Our primary aim is to study the application of azeotropic distillation in improving the ease of separation.

THEORY

The distillation process can be essentially characterized by the following combination of operations; a liquid mixture is partially vaporized by the application of heat; the vapor leaving the liquid is condensed as it comes in contact with down coming liquid and evaporates a part of it which becomes richer in the more volatile compound. By continuation of this process it would be possible to split a given liquid mixture into any number of distillate fractions.

A distillation column is the means of achieving this. A single stage of distillation in which the vapor leaving this stage in equilibrium with the liquid, is called a theoretical stage. In a column this is achieved by bubble plates, sieve plates or some type of packing which brings the down coming liquid into intimate contact with the upcoming vapor, so as to reach equilibrium. The number of plates or height of packing required to give separation equivalent to a theoretical plate depends upon the design and material used. In distillation a number of such ideal stages are required and this number depends upon the magnitude and type of separation desired. In general, the greater the number of theoretical plates, the greater is the degree of separation.

In case of a binary mixture, it is possible to approach complete separation of the two components by using a series of theoretical plates. The two combinations under investigation (1) 2,2,4-trimethyl pentane - 2,4,4-trimethyl pentene-1, and (2) normal heptane - 2,4,4-trimethyl pentene -1. Since the boiling points, and therefore the vaporization tendencies of the three components under investigation, are

close, a large number of theoretical plates would be required.

The aim of azeotropic distillation is to reduce the number of theoretical plates to make the operation economical.

Azeotrope is the term applied to distillation which involves constant boiling mixtures (1). Liquid mixtures consisting of two or more components, which when partially vaporized give a vapor with exactly the same composition as that of remaining liquid, are known as azeotropes. Only binary azeotropes, those consisting of two components, were used in the investigation.

The formation of an azeotrope is a function of the non-ideality of the resulting mixture when two liquid components are mixed, and a function of the difference in boiling points between these two components. The deviation from ideality is attributed to the effects of hydrogen-bonding and internal pressure (1).

The actual formation of an azeotrope depends upon (a) the magnitude of the deviations from Raoult's Law and (b) the difference in boiling point between the two components. The smaller the deviation from Raoult's Law for a pair of substances, the smaller the difference between boiling points must be before an azeotrope will form (1).

When a system showing positive deviations from Raoult's Law forms an azeotrope, it will be a minimum boiling azeotrope (e.g. boiling point of an azeotrope is below the boiling points of the constituents). When the

deviations are negative the azeotrope, if formed, will be maximum

boiling. When the two liquids are completely miscible they give

homogeneous azeotropes and when they are partially miscible we get

heterogeneous azeotropes.

In the application of azeotropic distillation a third compound

called the entrainer is added to the mixture. The entrainer in most

of the cases is found to form an azeotrope with both the components

and so is called a non-selective entrainer. Moreover in some cases

it has been found that the entrainer forms azeotrope with one compound

but does not form one with the other. It is then called a selective

entrainer. In order to compare azeotropic distillation and straight

distillation, a measure of the degree of separation must be known.

The volatility of a substance is defined as the ratio of partial

pressure of vapor to the mole fraction in liquid (which is equilibrium

with this vapor). If a substance is in the pure state, its volatility

is identical with its vapor pressure (5). Therefore, volatility of a

component is a measure of a compound's tendency to vaporize. When the

degree of separation of two components is to be evaluated, the concept

of relative volatiliy is used. Relative volatility is defined as

ratio of volatility of one component to the volatility of the other

component with which it is compared (5). Generally volatility of the

more volatile component is compared with that of the less volatile

component which gives a value greater than one. It is evident from

the concept of the relative volatility that the greater the relative

the relative volatility, the greater is the ease of separation.

Thus $\qquad \alpha_{AB} = \dfrac{Y_A \ X_B}{X_A \ Y_B}$

Where, $\qquad \alpha_{AB}$ = relative volatility of A with respect to B.

$\qquad Y_A, Y_B$ = mole fraction of A and B in vapor

$\qquad X_A, X_B$ = mole fraction of A and B in liquid.

The above definition of relative volatility is applicable to a single

stage but the same can be extended to a distillation column and is

given as the Fenske equation (2).

$$\alpha_{AB}^{n+1} = \dfrac{Y_A \ X_B}{Y_B \ X_A}$$

where, $\qquad$ n = number of theoretical plates

$\qquad Y_A, \ Y_B$ = mole fractions of A and B in tops.

$\qquad X_A, \ X_B$ = mole fractions of A and B in bottoms.

By substituting values of Y_A, Y_B, X_A and X_B obtained by

quantitative analysis of top and bottoms, values of relative volatility

for a certain pair with and without entrainer can be calculated. A

comparison between the two values will tell whether or not addition of

an entrainer improves the separation. The criteria for comparison being

that the higher the relative volatility, the better is the separation.

RESEARCH OBJECTIVES

The objective of this investigation was to look into the possibility of separating close boiling olefins and paraffins by azeotropic distillation. A comparison of the relative volatility between paraffin and the olefin using azeotropic distillation to that using straight distillation was the basis for the evaluation of various compounds as azeotropic entrainers.

CHOICE OF ENTRAINERS

The formation of an azeotrope depends upon factors (a) the magni-
tude of the deviation from Raoult's Law in the mixture and (b) the
difference in boiling points between two pure components. The closer
the boiling points of the two components the smaller the deviation from
Raoult's Law must be for an azeotrope to form. If an azeotrope is
formed, positive deviation from Raoult's Law gives a minimum boiling
azeotrope. Negative deviations from Raoult's Law gives a maximum boil-
ing azeotrope. The deviation from Raoult's Law in a mixture can be
attributed to the effects of hydrogen bonds or internal pressure (1).

Berg (1) has classified liquids into five groups and has given a
systematic summary of the type of deviations from Raoult's Law that can
be expected when liquids from any two of these groups are mixed. Hydro-
carbons such as those used in this study fall into a group which, unless
an ideal solution is formed, always give positive deviations when mixed
with liquids from their own or any other group. Compounds containing
oxygen, nitrogen or halogen atoms such as alcohols, acids, amines,
esters, halogenated hydrocarbons etc. are likely to form solutions
with hydrocarbons which have large positive deviations from Raoult's
Law.

Thus compounds selected as entrainers are chosen from the types
just mentioned and boiled close to the hydrocarbons whose separation
was being attempted. Compounds boiling within about ± 20°C of the

hydrocarbons were selected. In some cases compounds fit the above

two criteria but were not used because of unavailability.

The compounds evaluated as entrainer are as follows:

Ethanol	n-Valer aldehyde
n-Propanol	Vinyl isobutyl ether
n-Butanol	1, 4 - dioxane
2-Butanol	Isopropanol anhydrous
Ethyl acetate	Dipropyl amine
n-Propyl acetate	Amyl amine
Isopropyl acetate	Triethyl amine
Isobutyl acetate	Methyl n-propyl ketone
Methyl vinyl acetate	Methyl isobutyl ketone

EQUIPMENT

The principal equipment used in this study is as follows:

1. Four batch rectification columns

2. One #A-90 C Aerograph gas chromatograph with thermal conductivity detector.

3. One Ohaus triple beam balance

4. One #1013 Valentine refractometer

5. One #AB-2 Christan Becker chainomatic balance

6. One # 620005 Keuffel and Esser compensating polar planimeter.

The four rectification columns used were identical. The columns are made from three concentric glass cylinders which were 48 inches long. The three concentric cylinders had diameters of 1.0, 1.75 and 2.5 inches and the inside cylinder was packed with 46" of 1/8" stainless steel helices (Fenske-rings). The rectification columns were equipped with cold finger condensing head. The cold finger condensing heads do not allow the setting of definite reflux ratio, but they are quite satisfactory as the columns were always operated at total reflux.

The still pots were one-liter round bottom glass flasks with thermowells and side arms for taking samples. A diagram of such column is shown in Figure 1 and a further description is given by Nelson (3).

Azeotropic compositions were determined by plotting weight percent vs. refractive index. A constant temperature bath was used to keep the temperature of the refractometer prism at 20 ± .3°C.

The Christan Becker balance was used in the preparation of known weight percent solutions of which were used to make the refractive index vs weight percent curves. The Ohaus triple beam balance was used for weighing the charges boiled up in columns.

The Aerograph chromatograph with a conductivity detector was used to analyze overhead and bottom samples from the rectification column when relative volatility was determined. The column used for separation was a 1/4" stainless steel tube, 7 feet in length containg 30-60 mesh, C-22 firebrick as the solid support and oxydipropinitrile as the stationary phase. This column gave sharp peaks for olefins and paraffins and there was no overlapping. This facilitated use of peak height percent rather than area percent, the former being easier and much more accurate in this case. In a few cases where the refractive indices of the entrainer and one of the hydrocarbons were too close, azeotropic compositions were determined using the chromatograph. In these cases, however, area percent was found to be more accurate. A Keuffel and Esser compensating polar-planimeter was used to measure peak area in the analysis.

EXPERIMENTAL PROCEDURE

The same experimental procedure was applied to each of the en-
trainers that was evaluated. Therefore, the general procedure will
be described with mention made of any variation while studying a
particular entrainer.

The first step in the evaluation of an entrainer was to charge a
50:50 (weight percent) mixture of the compound and the entrainer under
consideration. The boiling points of the entrainers at this eleva-
tion and 1 atmosphere pressure were determined separately. The 50:50
charge was boiled at constant rate for one hour and the temperature
of the overhead was noted. If the overhead temperature is less than
the boiling points of both the constituents of the charge an azeotrope
was formed. About one milliliter sample was taken from the top of the
column and its refractive index determined. Since the refractometer
was provided with a constant temperature bath the prism could be
maintained all the time at $20 \pm .3°C$. In most of the cases refractive
indices of the two compounds were quite different hence calibration
plots for composition versus refractive indices could be made by
weighing samples of known composition and determining their refractive
indices. From these plots the composition of the overheads were
determined. Next the charge in the stillpot was adjusted as per the
compositions of the overhead obtained from plots and then boiled for
another one hour at total reflux. Overhead and bottom samples were
then taken and their composition determined to see if they were the

same since the liquid and vapor composition for an azeotrope are
identical. If the top and bottom compositions were within 4 percent
of each other, the top composition was taken as the azeotropic com-
position. Care was taken to repeat the above process until the top
and bottom compositions were within 4 percent of each other. In
all the evaluations duplicate runs were made and an average was taken
which gives the final azeotropic composition.

Once azeotropic compositions were determined relative volatility
runs could be made. A charge was made up by first weighing the correct
amounts of material to give the azeotropic composition of more volatile
component with the entrainer. An amount of the less volatile component,
equal by weight to the more volatile, was added. Every time a charge
was made up enough material for two charges were weighed and then split
in two. Duplicate runs were made for every relative volatility deter-
mination and the two charges were in this way assured of being the
same in composition. The charges were then boiled and after removing
the first few traces from the top (about 1 milliliter) the charge was
allowed to boil at total reflux for three to four hours so as to attain
equilibrium. Since no sample was drawn during the run the equilibrium
is not disturbed. Small samples were taken from the top and bottom at
the end of the run. Since the samples were analyzed on a chromatograph
only a very small sample size was necessary. In many cases the en-
trainers were water soluble hence the samples were water washed before
inserting in chromatograph column.

For chromatographic analysis an oxydipropinitrile column was used because 1. It gives short retention time of about three to four minutes for the hydrocarbons. 2. It gives sharp and distinct paraffin and olefin peaks. 3. It gave long retention time for most of the entrainers thus there was never a problem of overlapping of peaks. A typical chromatogram is shown in Figure 2. Since the peak heights were easily measurable because of the sharpness of the peaks and the peaks were very narrow peak height percent vs. composition plots were obtained by getting peak height percent for ten samples of known composition. The plot is shown in Figure 5, and data used in Table VII. The column was operated at 81°C with a helium flow rate of 12.5 ml/min. and a chart speed of 1"/min. with the help of calibration plots, the composition of tops and bottoms was obtained.

After the tops and bottom's samples are analyzed relative volatilies were determined using the Fenske equation. A sample calculation for relative volatility is on page (21).

Fenske equation.

$$\alpha^{n + 1} = \frac{Y_A / Y_B}{X_A / X_B} .$$

n = number of theoretical plates.

The number of theoretical plates in each column was determined by running a test mixture comprising of toluene and methyl cyclohexane and McCabe-Thiel method.

DISCUSSION OF RESULTS

In this investigation twenty two chemical compounds are selected as possible entrainers. Each chemical compound selected for this purpose has a normal boiling point in the range of ± 20°C of the hydrocarbon under study. The compounds were expected to form an azeotrope with the hydrocarbons as they met the requirements regarding their chemical nature as in reference (1). Cost, stability to heat and availability in a pure state are also factors considered in the selection of these compounds. All the compounds are tabulated in Table I with their boiling point as determined at this elevation.

Out of the twenty-two compounds studied, fourteen formed azeotropes with at least one of the hydrocarbons. Out of the fourteen azeotropes three were formed only with 2,4,4-trimethyl pentene-1. The selective entrainers are methyl isobutyl ketone, methyl-n-propyl ketone and propyl acetate. They do not form an azeotrope either with normal heptane or isooctane. The remaining eleven compounds formed azeotropes with all three.

Azeotropic composition for each entrainer was determined with all three hydrocarbons and is listed in Table II, III and IV. Azeotropic compositions varied from 16 to 81%, 7 to 82% and 9 to 89% in case of 2,4,4-trimethyl pentene-1, 2,2,4-trimethyl pentane and normal heptane respectively. In all azeotropic composition determinations, duplicate runs were made. The values obtained by two runs are in reasonable

agreement with each other. On this basis an average value is used in making charges for relative volatility runs.

Homogeneous azeotropes are formed except in the case of methyl cellosolve. Therefore, a refractometer can be used for analysis. Calibration curves are obtained by weighing samples of known composition, finding out their refractive indices and plotting them on an x-y axis. In most of the cases the calibration plots are straight lines or smooth curves. No personal judgement is therefore required in using them. A constant temperature bath maintained the temperature of refractometer prism at $20 \pm .3°C$. Hence the use of a calibration curve is justified.

The four rectification columns used are calculated by the McCabe-Thiel method using toluene-methyl cyclo hexane test mixture. They are found to have 26, 28, 27, 28 plates. Instead of the usual graphic method of stepping off the stages, the number of theoretical plates was calculated by using a standard programme on a digital computer. It may be noted that an error of .003 in relative volatility is introduced by an error of one theoretical plate. The digital computer gives values up to three decimal places hence the chance of introducing an error in relative volatility due to error in theoretical plates is reduced considerably.

In determination of relative volatility the charge is made and then split in two parts to be charged to two columns. This avoided chances of having different compositions in the two columns.

Top and bottom samples are analyzed by a gas chromatograph. As the peaks obtained are tall, narrow and distinct, peak height percent instead of area percent is plotted against mole percent for calibration plots. Peak height measurement is easy and accurate with these types of curves. The calibration plots for the two systems are given in Figure 4 and a typical chromatogram is Figure 2.

The composition of tops and bottoms obtained by chromatographic analysis are used in Fenske's equation to calculate the relative volatility. These calculations were also performed on the digital computer which gives values up to four decimal places. The values of relative volatility obtained are listed in Table V and Table VI.

Tables V and VI show changes brought about in relative volatility by the use of an entrainer. All of the entrainers are found to increase the relative volatility.

In the case of 2,4,4-trimethyl pentene-1 - 2,2,4-trimethyl pentane system, the relative volatility without entrainer is 1.044 and 1.037 (as obtained from two runs). On the basis of the average value, the number of theoretical plates required for 95% separation is 150. This value is reduced to 55 when 1,4-dioxane is used as an entrainer.

For 2,4,4-trimethyl pentene-1 normal-heptane system the relative volatility is 1.048 and 1.043, which again differs only in the third decimal place. The number of theoretical plates are reduced from 131 to 49 by use of isopropyl acetate as an entrainer when 95% separation is desired.

None of the entrainers reduce the relative volatility below that obtained without entrainer suggesting that there is improvement in ease of separation. Isopropyl acetate increases the relative volatility the most for both the pairs studied but its commercial importance is dampened by the fact that the isooctane and normal heptane are present in only a small amount in the azeotrope, thus increasing the cost of the entrainer required.

In the chromatographic analysis, since most of the entrainers either are water soluble or have long retention times as compared to the hydrocarbons no problem of overlapping of peaks is incurred.

The maximum difference in relative volatilites obtained by two runs is .02 units which is well within the range of experimental error and for all practical purposes an average value of relative volatility is taken.

No effort was made to recover the entrainer. It was observed that most of the entrainers are water soluble whereas the hydrocarbons are not. This property could be used to recover the entrainers.

A study of the relative volatility results and the boiling points of the entrainers reveals that there exists no relationship between the boiling point of the entrainer and its effects on the relative volatility.

CONCLUSIONS

It is found possible to separate 2,2,4-trimethyl pentane
from 2,4,4-trimethyl pentene-1, and normal heptane from 2,4,4- tri-
methyl pentene-1 by azeotropic distillation using considerably fewer
theoretical plates than is required using straight distillation.
Economic factors depending upon the cost of entrainer and ease of
recovery would determine which entrainer would be best in a partic-
ular case and whether azeotropic distillation would be commercially
feasible.

<div align="center">SAMPLE CALCULATIONS</div>

Relative volatility calculations.

For isooctane - 2,4,4-trimethyl pentene-1 System

Run I

$$\alpha_{AB}^{n+1} = \frac{Y_A \cdot X_B}{X_A \cdot Y_B}$$

Y_A, Y_B = mole percent isooctane and 2,4,4-trimethyl pentene-1 in overhead

X_A, X_B = mole percent isooctane and 2,4,4-trimethyl pentene-1 in bottoms

n = number of theoretical plates

X_A = 74

Y_B = 26

X_A = 46.6

X_B = 53.4

$n + 1$ = 27.

$$\alpha_{AB}^{27} = \frac{74 \times 53.4}{26 \times 46.6}$$

$$\alpha = \frac{(74 \times 53.4)}{(26 \times 46.6)}^{\frac{1}{27}} = (3.255)^{.037}$$

$$= 1.0446$$

Calculation of the number of theoretical plates required to give 95%

separation of isooctane and 2,4,4-trimethyl pentene-1 using no entrainer.

$$(1.0446)^{n+1} = \frac{95 \times 95}{5 \times 5} = 361$$

$$n + 1 = \frac{\log 361}{\log 1.0446} = \frac{2.751}{.0189} = 150.2$$

$$n = 149.2 = 149 \text{ theoretical plates.}$$

using 1.4 - dioxane entrainer

$$(1.12)^{n+1} = 361$$

$$n + 1 = \frac{\log 361}{.0487} = 56.4$$

$$n = 55.4 \text{ theoretical plates.}$$

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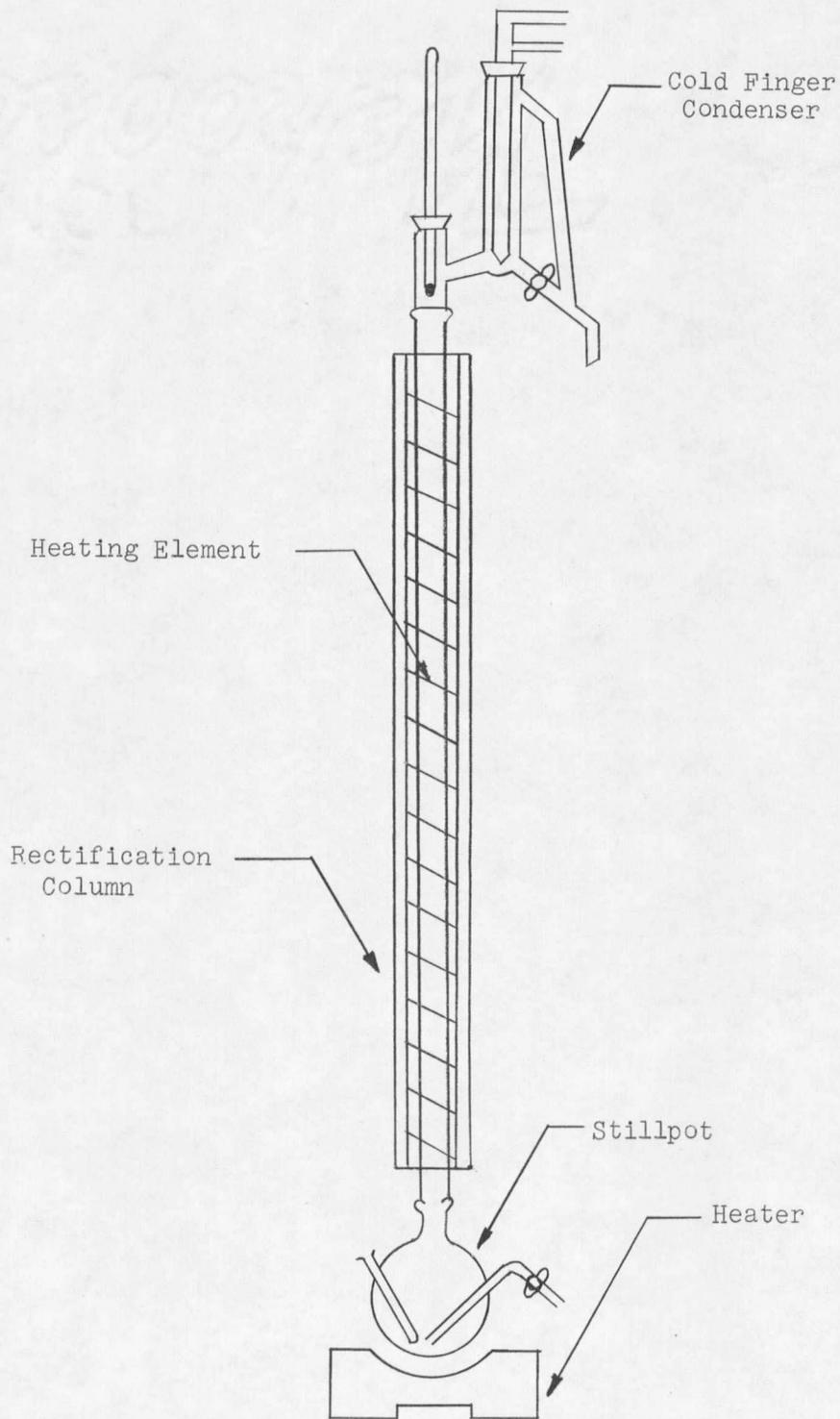5. Robinson, C., and Gilliland, E., Elements of Fractional Distillation, McGraw Hill Book Company, Inc. New York.

APPENDIX

FIGURE I: Rectification Apparatus