THE DESIGN AND CONSTRUCTION OF A FRACTIONATING COLUMN

A Thesis Presented to the Chemistry Department of the American University of Beirut by Kamal N. Saad in Partial Fulfilment of the Requirements for the Degree of Master of Arts in Chemistry.

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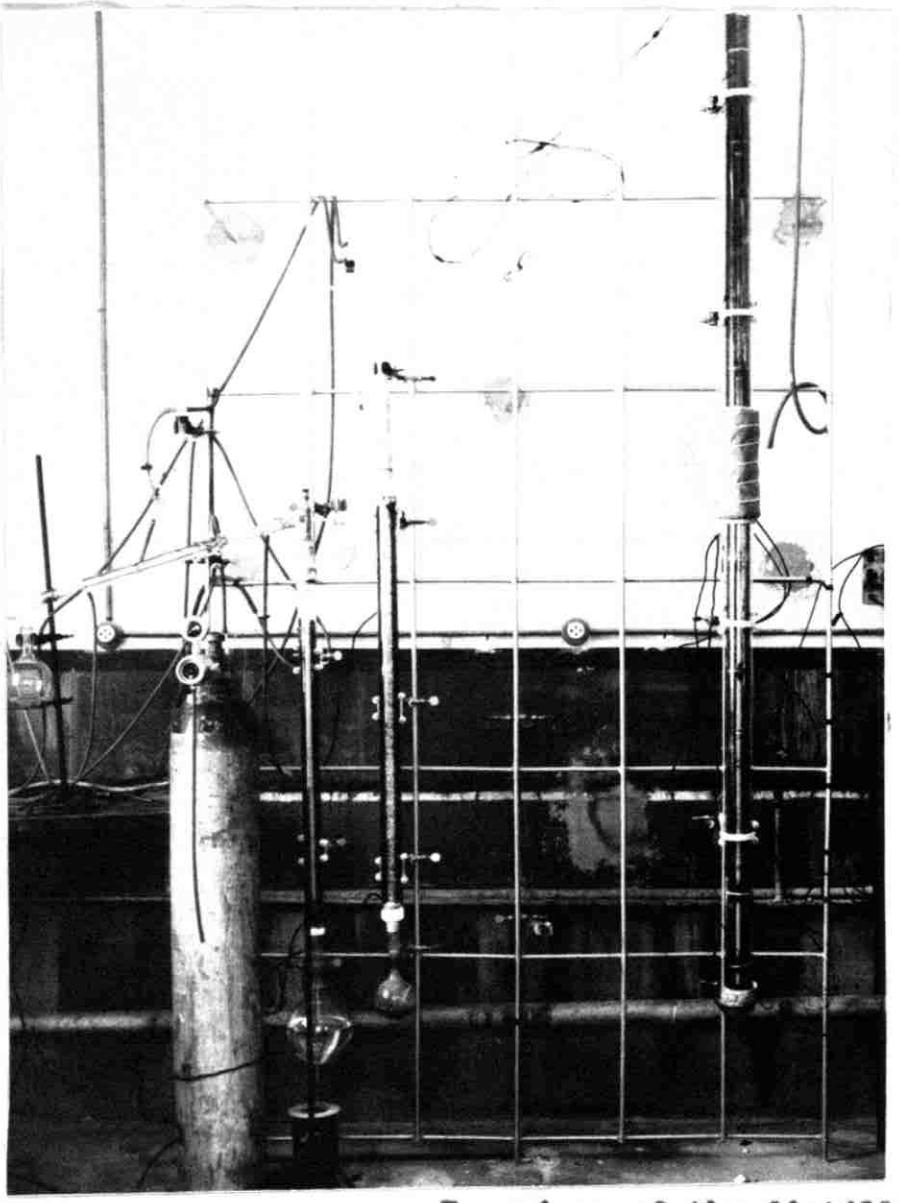

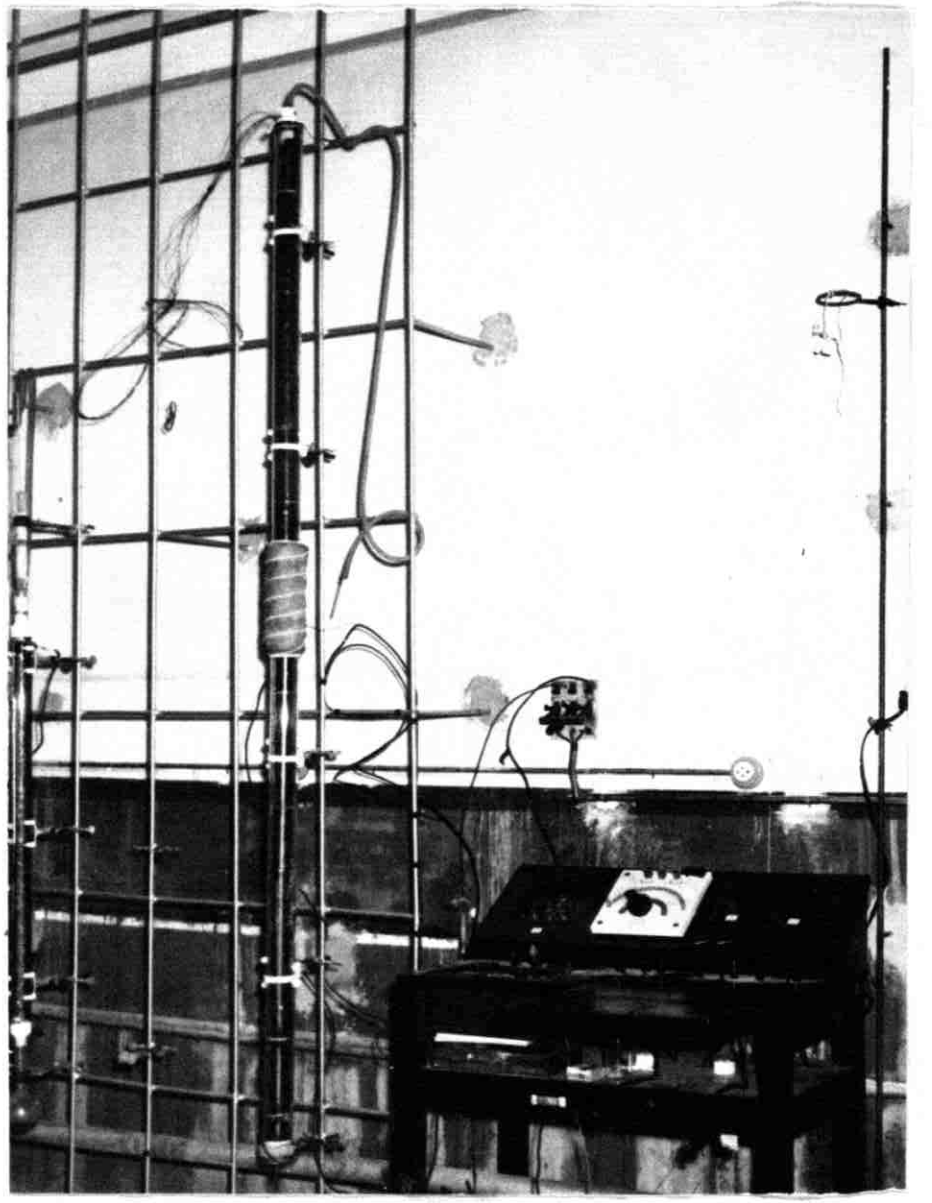

Two views of the distilling rack with the column under construction.

= Porta, (Natural Magic, Naples 1553 and 1589;
English Translation, London 1658.)

"Now I am come to the arts and I shall begin from distillation, an invention of later times, a wonderful thing to be praised beyond the power of man; not that which the vulgar and unskilful men use; but that which is done by skilful artists. This admirable art teacheth how to make spirits and sublime gross bodies, and how to condense and make spirits become gross bodies, and to draw forth of plants, minerals, stones and jewels, the strength of them that are involved and overwhelmed with great bulk, lying hid, as it were, in their chests; and to make them more pure and thin and more noble, as not being content with their common condition, and to lift them up as high as heaven. We can by chymical instruments, search out the vertues of plants and better than the ancients could do by tasting them. What therefore could be thought on that is greater ? Let one that loves learning and to search nature's secrets, enter upon this; for a dull fellow will never attain to this art of distilling ....

... Now we speak of oyls: these require the industry of a most ingenious artificer, for many of the most excellent essences of things do remain in the oyl so close, that without the greatest art, wit, cunning and pains, they cannot be brought to light; so that the whole art of distillation dependeth on this .....

... If you distill common oyl, it will hardly run. You must be very careful that the ashes and pot do not wax too hot,

for if the oyl within takes fire it will break the vessel and flie up, that it can hardly be quenched, and reach the very ceiling; so that it is best to operate upon oyls in arched rooms ...."

= From Egloff and Lowry(47).

INTRODUCTION

The "Art" of distillation has come a long way since the days of Porta and the alchemists. "Art, wit, cunning and pains" have all contrived to make of practical distillation a more or less precise science, although clarification upon certain theoretical points is still to be desired. However, distilling apparatus is now being built which is capable of operating continuously and automatically for days on end with a minimum of attention and which separates mixtures of liquids boiling as little apart as 2° C.

The fractionating column has now become an essential tool in any chemistry laboratory and is used for such basic operations as the analysis of samples and the purification of substances which makes it invaluable for research purposes.

The need for a precise fractionating column being an apparent one in this department it remained for us to design and construct one. It is to this purpose that this work is devoted.

The literature on the subject is wide and varied but disorganized. With a few exceptions, (9),(16),(42),(52),(59) there has been little effort on the part of the authors to present their material in systematic form.

There is confusion about the nomenclature and I have therefore included a list of definitions of terms arranged alphabetically at the end of the Introduction.

Free use has been made of standard texts and handbooks in outlining the theory of distillation. A special bibliography of those general references will be given at the end of the Thesis.

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DEFINITION OF TERMS

Adiabatic Operation

This refers to a condition of the system where there is no heat loss or where the heat loss is completely compensated for. This condition cannot be achieved in practice.

Channelling

Channelling of liquid refers to a condition where liquid overflow going down a packed column follows definite paths in the packing.

Distillate

This refers either to the overhead product taken off or to the rate of withdrawal of such product. The rate of withdrawal of overhead may be expressed in moles/unit time or, more usually, in c.c./unit time. Symbol - D.

Flooding of a Column

This is that state where liquid starts to move bodily up the column.

H.E.T.P.

This is an abbreviation for the height equivalent to a theoretical plate. (It is expressed in cms. or inches.)

Holdup

This is the amount of liquid and vapor in the column during operation. The amount of liquid needed to wet the walls and packing of a column is called the static hold up. This is expressed in c.c. of liquid.

Liquid Gradient

Liquid gradient across a plate is the difference in level between the liquid at the liquid inlet and the liquid at the liquid outlet or downpipe.

Overflow

This is the amount of liquid going down the column per unit time. It is expressed in moles/unit time or c.c./unit time. Symbol - D.

Plate Efficiency

This is expressed as a percentage of a theoretical plate, which percentage is calculated from the separating power of the plate as compared to that of a theoretical plate.

Pressure Drop

This is the difference in pressure between the stillpot and the stillhead. It is

usually expressed in mms of mercury.

Reflux

Reflux is the same as overflow

Reflux Ratio

This is defined as the ratio of the amount of overflow to the amount of distillate. Symbol - R

Still

This is the name usually given to the total assembly of the stillpot, fractionating section and stillhead.

Take-off

This is the same as distillate.

Theoretical Plate

A theoretical plate is one that fulfils the requirements that liquid leaving it to the plate below is in equilibrium with the vapor rising from it to the plate above.

Throughput

This is defined as the volume of liquid rising to the head per unit time. It may be expressed in moles/unit time or in c.c./unit time.

Vapor-Liquid Equilibrium Data

This correlates the composition of the vapor with the compositions of the liquid with which it is in equilibrium (at a constant pressure of 1 at m. usually).

Vapor Velocity

This is the velocity of the vapor rising through the column, usually expressed in ft/sec.

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Table of Contents

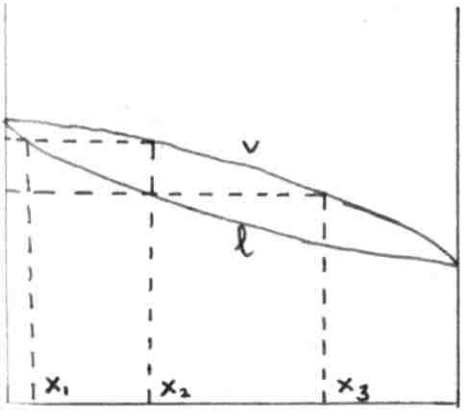
CHAPTER I -- An Introduction to the Theory of Fractionation, a Discussion of the Various Types of Columns, of the Mathematical Theory of Fractionation and of Methods of Testing Fractionating Columns.

I. Introduction

Distillation is the name given to those operations whereby a vapor phase of more than one component is obtained. Distillation is to be distinguished from evaporation which gives a vapor phase of only one component.

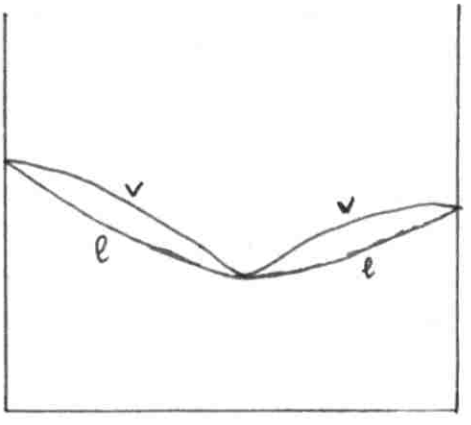
To be able to separate two substances by distillation, the vapor obtained by boiling a mixture of their liquids should be of a composition different from that of the boiling liquid. When this requirement is not fulfilled, we have what is known as an azeotropic (constant - boiling point) mixture.

Of the three types of completely miscible binary mixtures (figs. 1, 2, and 3), only the first are theoretically completely separable by distillation alone over the whole range of compositions.



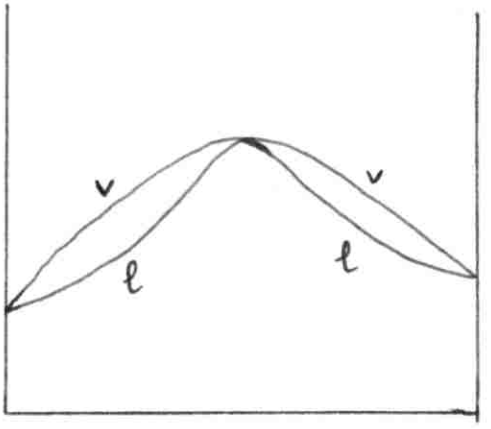
Composition

Fig. 1



Composition

Fig. 2



Composition

Fig. 3

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The second type mixtures form minimum-boiling-point azeotropes due to repulsion between the molecules while the third type mixtures form maximum-boiling-point- azeotropes due to abnormal molecular attraction. The last two types of mixtures can only be distilled within each range of compositions on either side of the constant-boiling mixture, i.e., the azeotropic composition cannot be crossed by distillation.

Let us now consider fig. 1. Boiling a liquid mixture of composition x_1, at T_1, gives us a vapor of composition x_2 at T_1. This vapor condenses to give liquid of composition x_2 at T_2, and so on . . . . The vapor gets progressively richer in the more volatile component while the liquid gets progressively richer in the less volatile component.

Practically, this may be accomplished by separate successive distillations and condensations. The operation may be made continuous by devising a special piece of apparatus. Such an apparatus is shown schematically in fig. 4.

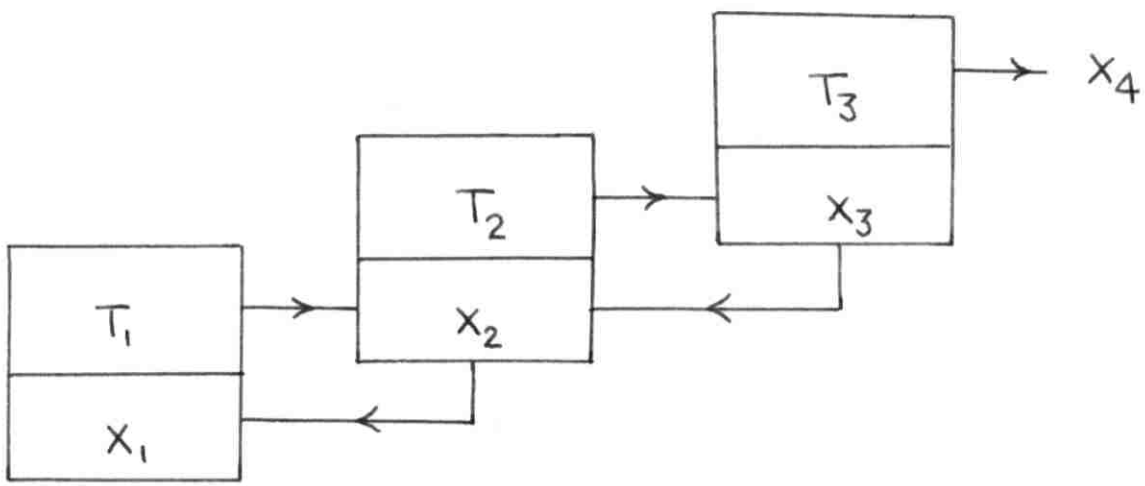

Fig. 4

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It is no more than a series of boilers. The vapor from the first boiler (composition x_2 at T_1) goes into the second boiler where it condenses and gives vapor of composition x_3 at T_2 which goes into the next boiler. Each boiler is equipped with a liquid downpipe which returns liquid, rich in the less volatile component to the boiler before it in the series.

If all the boilers are incorporated into one column, i.e., they are set one above the other, we get what is known as a fractionating column.

The essential parts of a fractionating still or column are three in number :-

(1) The Stillpot

This is the boiler of the still.

(2) The Column

This is where the separation of the liquids takes place. When the operation is continuous, i.e., when liquid of a constant composition is fed into the system at some point along the column, there are two sections to the column:

a) The fractionating or rectifying section, above the feed point.

b) The stripping section, below the feed point.

When the operation is a batch process, the whole of the column becomes a fractionating section.

(3) The Head

This is put at the top of the column and consists of a condenser (partial or total) and some device for the proportionation of reflux.

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In the column proper, we have a countercurrent flow of liquid and vapor and it is there that most of the fractionation takes place. The fractionating process is one of interdiffusion across the vapor-liquid interface. The aim of any designer of fractionating columns is to provide for adequate vapor-liquid contact.

II. Types of Columns

A. Plate Columns

Here the emphasis is laid on bubbling the vapors through a liquid head, the amount of contact being governed by the height of the layer of liquid, the size and number of bubbles, and their velocity.

There are two types of plate columns, both usually used in large scale operations, especially in the petroleum industry. They are impractical for laboratory use due to their large holdup.

The two types of plate column are both usually built out of metal. They are :-

(1) Sieve-Plate Columns

Sieve-plate columns consist of a series of horizontal, perforated plates spaced vertically above each other (fig. 5). A downpipe on each plate transfers liquid down the column.

General dimensions for plate and perforation spacings are given in Perry. (2).

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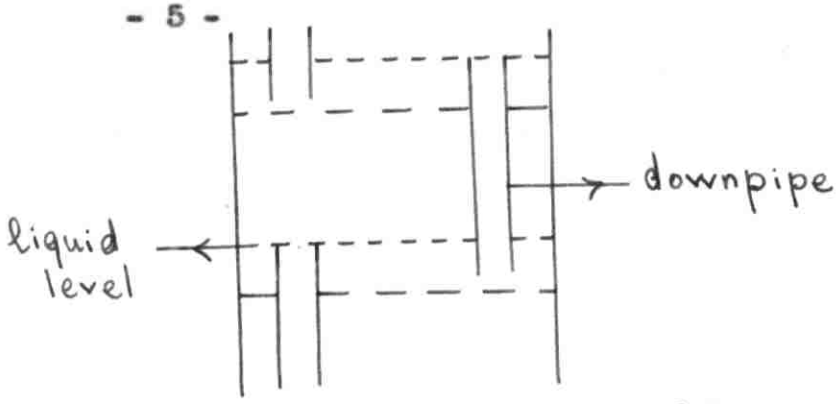


A Crossection of a Sieve-Plate Column

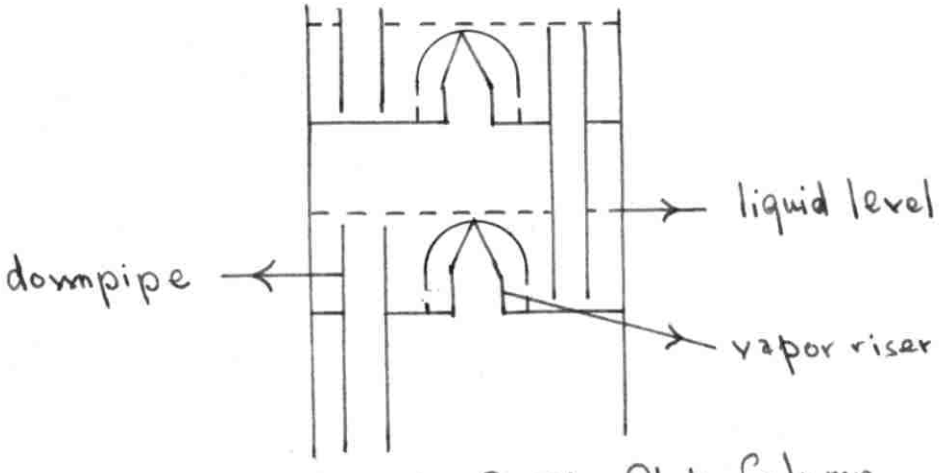
fig. 5

The liquid on the plates is held up by the pressure of the rising vapors and this necessitates that the plates be absolutely level. (1). Otherwise inadequate and nonuniform mixing resluts, decreasing efficiency of operation. This also limits operation to the higher ranges of vapor flow. (2).

For sieve-plate columns, the great danger is corrosion which enlarges the perforations and destroys the efficiency of the column.

(2) Bubble-Plate Columns

There are also a series of horizontal plates set vertically above each other (fig. 6).



A Crossection of a Bubble-Plate Column

fig. 6

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They are provided with va por risers topped by slotted inverted caps. The slots dip below the level of the liquid, t his level being controlled by the height of the downpipe above the plate .

Ge neral dimensions for ca p spacing, plate spacing, slot widths and downpipes are given in Perry (2)

(a) Design Consideration

The design of a Bubble-plate column is quite complicated. Three main factors have to be considered:-

(1) Pressure Drop

This has to be kept low so that the temperature gradient along the column should be a function of the composition only, (26).

The causes and means of reducing pressure drop are well discussed in the litera ture (13), (26), (49).

(2) Liquid Gradient

Liquid gradient across the plate also has to be low, (3), (26) so as to have uniform mixing of liquid and vapor.

(3) Ent rainment

This is defined as the mechanical ca rry-over of droplets of liquid by the vapor stream. Entrainment seriously impairs fractionating efficiency. A lot of importa nce has been accorded to it in the literature (2), (3), (26), (27), (30), (31), (32), (33).

Entrainment is high for close spacing (3) and may be reduced by using venetian-blind type baffles (26).

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B. Unpacked Columns

Unpacked columns, made of glass, are widely used in the laboratory but are only useful in separating mixtures with boiling points very far apart such as the distilling off of a low boiling solvent. In such a case, a column is to be preferred to an open vessel to eliminate entrainment.(10). They have a good efficiency at very low throughputs, (16).

C. Concentric-Tube Columns

These are made of two or more glass tubes that fit into each other, leaving an annulus of about 1.5 mm. The annular space must be very uniform and on no account may the tubes be allowed to touch. Contact between the tubes produces chanelling which decreases the efficiency. (11). They are thus very difficult to construct properly. Construction difficulties have been solved by various workers using several different methods.

With short columns of the order of 20 cms. no difficulty is experienced and a column with a 1.5 mm. annular space was quite efficient. (40).

Selker, Burk and Lankelma (41) built a column of four concentric tubes with 1 mm. annuli, the innermost one being closed. Spacing was made by sealing small glass tips every 37.5 cms. This gave an H.E.T.P. of 1.67 cms. at a throughput of 110 c.c. per hour.

Hall and Palkin (11) built an eight foot column using eight segments of tubing interlocked at top and bottom and provided each with a simple centering pronged collar at the top. This gave an H.E.T.P. of 2.7 cms. at a throughput

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of 117 c.c. per hour.

Na ra gen a nd Lewis (42), built their column by winding copper wire around the inner tube so that a close fit is just obtained between the two tubes. The column is then annealed, and the copper dissolved, using acid. This column had an H.E.T.P. of 0.49 cm. for a throughput of 107 c.c. per hour.

H.E.T.P. values for such columns are very low and they have very small liquid holdup. However, their throughput is very small. Moreover, (as is pointed out by Hall and Palkin (11)) it is difficult to obtain tubes of sufficient length, with uniform diameters to ma intain a uniform 1 to 2 mms. annular space a ll through.

Bailey (18) tried to duplicate the results of Selker, Burk and Lankelma (41) and fa iled. He suggests, however, that the fa ilure ma y have been due to his having used a different test mixture.

In addition to these difficulties of construction, there is the problem of providing for equal distribution of reflux. Failure to do so will impair efficiency of fractionation, the loss being serious with reflux ratios les than 100:1 (41).

D. Packed Columns

(1) Properties of Packing Materia l and Genera l Theory

Packed columns are by far the most widely used on the laboratory scale.

They consist of open tubes packed or filled with some inert materia l.

Zimmerman and Lavine (1) give a list of desira ble properties for packing materials.

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A packing material should:-

1. Be non-reactive.

2. Provide a large contact surface per unit of vol ume.

3. Have uniformly distributed interstices.

4. Have the free space desirable for a desirable throughput

It should also have low liquid holdup (4), (9), (24), (52) and some workers believe (34) that the efficiency is greater when the pa cking has sharp edges and cuts.

The great danger with pa cked columns is the channelling of liquid. Bragg (38) says tha t when there is continual mixing and separating of liquid and vapor, there is no possibilit; of cha nnelling. Penske and co-workers (4) claim that channelling may be reduced by varying t he free space in the packing by alt ernating different kinds of pa cking material. It is also recommended that:

1. The diameter of the column be greater than eight times that of the individual packing (57).

2. The column height be greater than fifteen times the column diameter (2).

Howe ver, Nelson(53) points out that channelling may be e xcessive if the diameter of the column is grea ter than 18 inche

(2) Types of Packing

Since Hempel (54) introduced laboratory packed columns in 1881, "almost anything that could be fitted into a tube has been tested as a packing" (20).

It would be superfluous to discuss all the various

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types of packings now generally in use. A good discussion of these is found in Carney. (60).

However, among the more efficient types of packing six types stand out. These are :-

1. Podbielniak Heligrid Packing (5) which has very low holdup and H.E.T.P.
2. Stedman Packing (38) also with low H.E.T.P. (of the order of 0.477 in.), low holdup and high throughputs.
3. Lecky and Ewell Spiral Screen Packing (39) which has H.E.T.P. values as low as 0.36 in. and low pressure drop.
4. McMahon Packing (58) usually made out of 100 mesh brass wire.
5. Silicon Carbide (34) has the same H.E.T.P. as wire packing but has a low throughput.
6. Fenske Single-turn Helices (60) are a very widely used type of packing.

   Although Fenske packing usually has higher H.E.T.P. values than 1, 2, and 3, it is the only one of the six that may be conveniently made out of glass. It consists of single turn helices and may be made "from almost any material" (20). It is probably the best possible packing for general laboratory work.

   It has been used effectively over a large range of temperature and pressure, (60). The operating characteristics of some of the different size Fenske Packings are given in Carney ((60)

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pp. 65 and 66).

Packed columns can be built to operate at the same capacity as plate columns but for large diameter columns, bubble-plate columns are the best. (4). They give a good throughput and have a low pressure drop. (36).

III. The Mathematical Theory of Distillation

Carney (61) discusses the many methods devised for the mathematical treatment of distillation. I shall only discuss here three of these methods :-

A. The first known mathematical theory of Distillation was proposed by Sorel. (62). He made four assumptions :-

(1) The moles of liquid overflow are equal to the moles of ascending vapor at total reflux at any plate.

(2) Operation of the column is continuous.

(3) Operation is adiabatic.

(4) There is no heat of mixing of the components of the mixture to be distilled.

Let us consider a section of a column between the n^{th} and the $(n+1)^{th}$ plates (fig. 7)

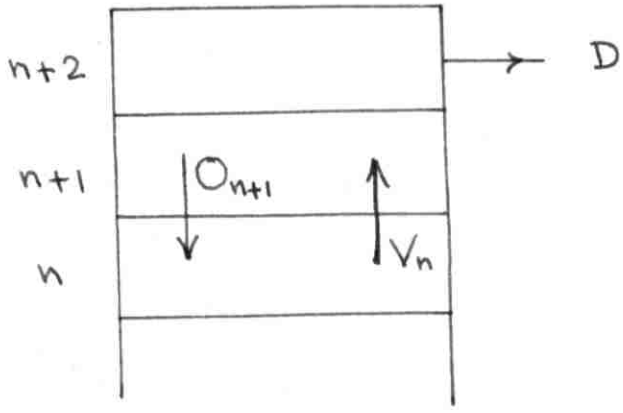

fig. 7

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A material balance gives:

$$V_n = O_{n+1} + D$$

Where:

D: moles of distillate/unit time

n: plate subscript

V: moles of ascending vapor/unit time

O: moles of liquid overflow/unit time

If x' and y' are the mole fractions of the more volatile component in the liquid and vapor respectively, a material balance of the more volatile component yields :-

$$y'_n V_n = x'_{n+1} O_{n+1} + Dx'_D$$

Therefore:

$$y'_n = \frac{O_{n+1}}{O_{n+1}+D} x'_{n+1} + \frac{D}{O_{n+1}+D} x'_D$$

But,

$$R = \text{reflux ratio} = \frac{O_{n+1}}{D}$$

Therefore:

$$y'_n = \frac{R}{R+1} x'_{n+1} + \frac{1}{R+1} x'_D$$

The solution of a problem using this equation necessitates stepwise and therefore tedious calculations. B. McCabe and Thiele (65) devised an ingenious and simple graphical solution of the problem.

The composition of the distillate and the reflux ratio, R, being known, they plotted Sorel's equation as an operating line on the vapor liquid equilibrium diagram of the particular mixture distilled. For the case of total

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reflux ($R=\infty$), the operating line is represented by

$$y' = x'_{n+1}$$

and lies on the x-y diagram diagonal. When operating at a finite reflux ratio, the operating line passes the points (X'_D, X'_D) and $(0, \frac{I}{R+1} X'_D)$.

The first point is obtained using the fact that $X'_D = y'_{n+1}$ (where the (n+1)st plate is the last plate in the column in this case) when a total condenser is used in the head. The second point is the y-intercept of the operating line. The number of plates in the column and stillpot was then obtained by drawing rectangular steps between the operating line and the equilibrium line, starting at the distillate composition, until the stillpot composition is passed. The number of such steps gives the number of plates in the column plus one for the stillpot.

C. Fenske (64) proposed a purely algebraic method for the determination of the number of theoretical plates in a column at total reflux. It is based on the definition of α, the relative volatility:-

$$\left(\frac{y'}{y''}\right)_0 = \alpha_0 \left(\frac{x'}{x''}\right)_0$$ for ideal systems, where y" and x" are the molefractions of the less volatile component in the vapor and liquid phases respectively and where the plates are numbered, starting with 0 for the stillpot.

Now, from Sorel's equation,

$$y'_n = x'_{n+1}$$ at total reflux ($R = \infty$)

And therefore:

$$y'_0 = x'_1$$

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Also,

$$y''_0 = x''_1$$

Thus,

$$\left(\frac{x'}{x''}\right)_1 = \alpha_0 \left(\frac{x'}{x''}\right)_0$$

And,

$$\left(\frac{y'}{y''}\right)_1 = \alpha_1 \alpha_0 \left(\frac{x'}{x''}\right)_0$$

Since

$$\left(\frac{y'}{y''}\right)_1 = \alpha_1 \left(\frac{x'}{x''}\right)_1$$

Assuming a total condenser, i.e. no rectification in the head, a nd following the same procedure, we get :-

$$\left(\frac{x'}{x''}\right)_D = \alpha_{average}^{n+1} \left(\frac{x'}{x''}\right)_0$$

where n is the number of theoretical plates in the column.

That the use of an average value of over the temperature range of distillation is justified, can be deduced by considering the following relationship for and bearing in mind Trouton's Law :-

$$\left(\frac{\partial \ln \alpha}{\partial T}\right)_P = \frac{\Delta H'_v - \Delta H''_v}{R T^2} \qquad (48)$$

where $\Delta H'_v$ and $\Delta H''_v$ are the heats of vaporization of the more volatile and less volatile components respectively.

When the differences in boiling points of the two liquids of the mixture is large, α is large. When the difference is small, α is small. The percentage error in using an average value of α is small (65) and is approximately the same in both cases.

Also, the larger α is, the easier it is to separate the two liquids by distillation. One method of making α larger is

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to lower the boiling point of the mixture. However, this does not necessarily mean that the effective separation is increased since, as Lloyd (59) points out, there may be a decrease in plate efficiency due to the increase in viscosity resulting from the lowering of the distilling temperature for any one pair of liquids.

Using the methods outlined above, the efficiency of any column may be determined in terms of its separation of known mixtures of liquids. Theory necessitates that operation of columns be continuous so that the results above may be applied since in batch operations the composition changes as product is taken off. However, the methods may be used to evaluate batch stills if the samples from the column head and stillpot are taken as simultaneously as possible. (2).

The efficiency of plate columns is expressed as an effective percentage of the number of actual plates, while the efficiency of packed and unpacked and concentric-tube columns is expressed in terms of the H.E.T.P.

IV. The Testing of Columns

Articles in the literature often evaluate columns only on the basis of the H.E.T.P. at total reflux and do not give sufficient importance to the nature of the test mixture. This has been resulting in false comparison of columns as in the case of Bailey. (18). Experiments run by Lloyd (59) show that, whereas a mixture of methyl cyclohexane and n-heptane gave a plate efficiency of 53 % the system benzene-ethylene chloride gave a plate efficiency of 43 % . According to Drickamer and Bradford (43) the plate efficiency varies inversely as the logarithm of the viscosity. Mixtures differ in ideality and properties, and different mixtures thus give different H.E.T.P. values with the

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same columns under the same operating conditions. For columns with a large number of plates, liquids with a small difference in boiling points have to be used.

In general, with columns built for specific fractionations it is advisable to use a test mixture with properties similar to the materials to be fractionated. However, this is not always possible in the case of laboratory fractionating columns. Methyl cyclohexane and n-heptane mixtures are especially suited for tests on columns for the fractionation of petroleum since both components are hydrocarbons (52).

Ward (52) mentions and discusses four different test mixtures. Willingham and Rossini (15) discuss some three more. To be able to use a test mixture, to determine the number of plates in a column, a knowledge of its vapor-liquid equilibrium data is necessary. An idea of the intricacy of the apparatus needed to determine such data with any reasonable degree of accuracy can be obtained from Robinson and Gilliland. (66).

An alternative way of testing columns is by measuring the amount of intermediate obtained between fractions. The sharper the transition from one fraction to the other, the more efficient the fractionation. This is the procedure used in analytical batch distillation.

Studying the shape of the curve of product composition against per cent distilled is better as a study of the effectiveness of separation (9), (67). An example of such a graph is given in fig. 8.

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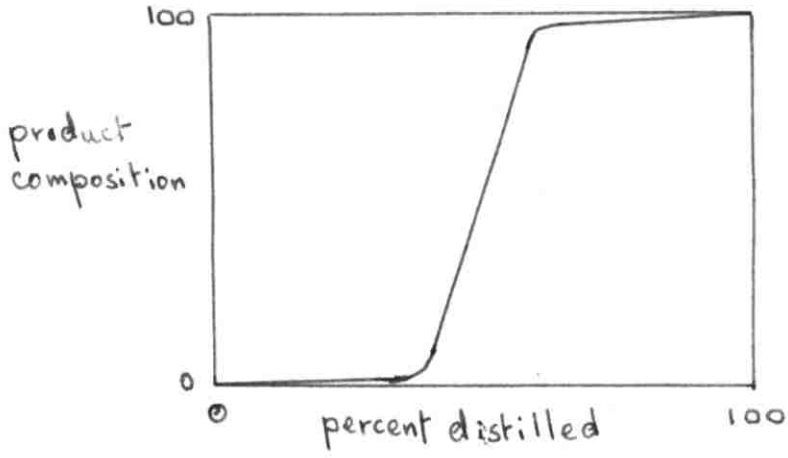

Fig. 8

The size of the intermediate fraction is determined by a variety of factors, discussed below :-

A. The separation fa ctor of the mixture, identified with α

For an ideal mixture, the size of the intermediate fraction is inversely proportional to the difference in boiling points of the components of the mixture. (9). This factor has been sufficiently discussed in a previous section.

B. Holdup

There is general agreement (4),(9),(23),(24) about the desirability of keeping holdup as low as possible. After allowing for increased scrubbing effectiveness (due to increased surface area per unit volume), minimizing holdup does more than increasing the height of a column. (9). The less the total holdup, the richer is the first fraction in the lighter component. (23). Bailey, (18) found that three factors were of importance in determining holdup in glass columns.

(1) The Surface Tension of the Liquid

Aqueous solutions have two to four times the holdup of organic mixtures (due to the former's greater surface tensions).

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(2) The Rate of Vaporization with Total Reflux

The decrease of rate of vaporization increased efficiency.

(3) The Temperature of the Jacket

Keeping the jacket within 1° C. of the temperature of the vapor greatly reduced holdup.

Holdup increases as the 1.6th power of the diameter of a column and is less for packings with a geometrical uniformity than for irregular packing. (9).

Bulky seals make for excessive holdup in glass columns and should be eliminated. (9).

C. Capacity

This is the maximum throughput of a column and is important, being related to the holdup. It increases as the second power of the diameter (i.e. with the volume). (9).

Throughput together with the holdup, reflux ratio and thermal insulation determine the optimum total time of distillation. (9). Operation beyond the maximum throughput floods the column and spoils fractionating efficiency.

The capacity decreases rapidly at low pressures. (8).

Podbielniak (9) states that the ratio of ten times the total holdup in the fractionating section to the column capacity should be small for maximum column efficiency.

D. The Reflux Ratio

A study of the McCabe and Thiele diagram reveals the fact that there is a certain minimum value of the reflux ratio below which a given separation cannot be effected. However, this cannot be applied to batch operations due to the fact that compositions at the head and stillpot and along the column are

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changing continuously. Thus the McCabe and Thiele diagram gives no idea of the amount of distillate obtainable from a certain batch. It is therefore desirable to use a distillation curve as a standard of separation. (67).

Thus, Rose (67) defines the standard separation as one which, when starting with 50 mole percent of each component gives an overhead product equivalent to 40 percent of the batch with 95 mole percent purity, assuming negligible holdup.

For such a separation, he points out, R has to be equal to $\frac{2.85}{\log_{10}\alpha}$ but in general may vary between $\frac{2.4}{\log_{10}\alpha}$ and $\frac{3.4}{\log_{10}\alpha}$.

From the McCabe and Thiele diagram, it may be deduced that increasing the reflux ratio increases the effectiveness of separation. However, the relationship is not as simple as may be imagined.

Rose and Long (19) state that the magnitude of the effect of increasing R depends on α , R and n (important in the order in which they are mentioned above). Thus, for a given α^n,

(1) With small α s, changes in R have little effect upon the sharpness of separation for all Rs unless n is made very large.

(2) With large α s, R changes have a marked effect for small Rs and less effect for high Rs.

If a constant reflux ratio is maintained throughout a batch distillation, the overhead purity decreases and it is therefore advisable to increase the reflux ratio as a fraction is removed or to use the higher reflux ratio throughout, which latter procedure gives a higher average purity of distillate. (59).

E. Scrubbing Efficiency

The scrubbing or fractionating efficiency of the column is

/20...

determined by two factors :-

(1) The H.E.T.P. of the Packing (or the percentage efficiency of a plate in plate columns)

This is usually determined at total reflux and varies inversely as the reflux ratio for finite values of the latter. It is also plausible to assume that H.E.T.P. varies inversely as the viscosity of the mixture being distilled for some mixtures in the way that plate efficiencies do (mentioned above). This would partly explain the fact, mentioned by Podbielniek (8), that the efficiency of packed columns diminishes rapidly at lower pressures.

For the same holdup per unit volume of a packing material, the less the H.E.T.P., the less the height of column required for a given separation and therefore the less the total holdup of the column, which thus decreases the size of the intermediate fraction.

(2) The Number of Theoretical Plates

Obviously for a given H.E.T.P. (or plate-efficiency), increasing the number of plates increases the scrubbing efficiency of the column. However, the relationship is not linear. The degree of separation of two components at high reflux ratios varies logarithmically with n (6), and for a given and R, increasing n has a greater effect for high Rs than when R is small. (19).

F. Thermal Insulation

Good thermal insulation of the column and other exposed parts lea ding to the condenser is essential from the point of view of control of reflux. It is also essential for efficient operation at low rates of distillation (16). Perfect insulation should give the same amount of reflux at the head as at the stillpot, i.e. there is then no condensation in the column proper.

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The subject of whether to have perfect insulation is controversial. Under certain operating conditions, non-adiabatic columns test more plates than nearly adiabatic columns, whereas under other conditions the 100 plate effectiveness of a column has been destroyed to 12 by supercooling or superheating it (5). No explanation of this has been given by distillation theory.

In a concentric tube column when a column jacket heating element was used a very good separation of diphenyl ether and methyl laureate was obtained. Otherwise, the liquid did not form a film but ran down in globules with very poor efficiency (40). On the other hand, in another concentric tube column, no difference was noticed between running a distillation of a mixture of methyl cyclohexane and n-heptane with the jacket at 1°C difference from the column and with its temperature about 20°C lower than that of the column (41).

It would, therefore, appear that among other factors, the type of mixture has an effect on this matter.

However, the principle of adiabaticity is generally agreed upon (4),(9),(11),(29).

Asbestos, Magnesite and glass wool have been used as thermal insulation. However, these do not afford visibility.

Thus it remains to use either a vacuum jacket or to compensate for heat loss by winding a dead air space jacket with resistance wire and controlling the amount of heat electrically.

Much work has been done by Podbielniak (9) on vacuum jackets. The best results were obtained by him with jackets fitted with silvered reflectors and pressures less than 10^{-5}mm.

However, a large-bore diameter Pyrex vacuum jacket cannot withstand a temperature differential of 135°C without expansion

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elbows and actually breaks at much lower temperature differentials (5). Vacuum jackets are impractical anyway for large units (17). Moreover, heat losses from visibility strips are considerable.

Blowing heated air through the jacket needs too much attention (17) and necessitates making the air jacket air-tight by sealing the glass which makes it liable to crack if for any reason the air stream is interrupted.

Dead air spaces, i.e. air at atmospheric pressure, between the outside wall of the distilling tube and the jacket, while not very effective an insulation, do not affect the thermal gradient of the distilling tube (13).

Below is a comparison of the thermal conductivities of various materials :-

	k (mean temp. 100°C. in cals./cm/cm^2/sec.)	
Glass Fibre	0.11×10^{-3}	.... (29)
Magnesite	0.16×10^{-3}	
Asbestos (fibre)	0.45×10^{-3}	
Asbestos (cellular)	0.16×10^{-3}	
Air	7.197×10^{-5}	

Although the specific heats are about the same, the heat capacity of the same volumes is much less for air than for other materials which means that the lagging as a whole will retain less heat but, on the other hand, it should respond quickly to heat changes.

G. Miscellaneous

Lloyd (59) has very amply discussed the weaknesses of batch distillation. He states that a batch still is only efficient as a rectifying section and not as a stripping section. He believes a batch still is not efficient as an instrument for giving

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overhead of high purity because the proportional amount of such distillate per batch is very small. However, for work on a laboratory scale, a batch still is best.

Polbielniak (9) believes that the best separations are obtained when the distillation is performed with the packing barely wet, whereas Nickels (51) holds that the best efficiency is obtained when the packing is wet just before the test is started by preflooding of the column.

Rose (23) believes that a larger first fraction is obtained by starting the operation at a low finite reflux ratio and returning some of the distillate directly to the still.

Columns operate better under intermittent withdrawal of distillate than under a continuous one, when high reflux ratios are used (of the order of 100:1). (25).

H. Conclusion

It can be seen that there is a lot of controversy and conflicting opinions as regards the operation of columns. This, I believe, is due to the use of different test mixtures. There is need for standardizing test procedures by choosing one mixture for each range of plates and specifying that the throughput be a definite fraction of the maximum, at a known reflux ratio.

This from the point of view of the correct comparison of columns and not with a view to finding whether a column is particularly suited for the fractionation of a certain type of mixture in which latter case the emphasis should of course be laid on the similarity of the properties of the test mixtures to those of the liquids to be fractionated.

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Chapter II

A Statement and Discussion of the Requirements and a Discussion of the Design and Construction of the Fractionating Column.

I Introduction

Some modifications of the apparatus may be introduced after testing the column. These, if any, will be described in Chapter III of this Thesis.

II The Requirements

A. Statement of the Requirements

The Column was built to serve three main purposes:

(1) The Column (with two smaller ones) was designed with a view to its being a useful tool for the instruction of students in fractionation and the theory of fractionating column operation.

(2) The column is primarily designed as a tool for the purification of compounds needed for research or other purposes.

(3) The Column will also serve an immediate aim: that of the purification of nitrogen heterocyclic compounds needed for research actually going on in the department. Most of these compounds boil in the range between 100° and 200° C.

B. Discussion of the Requirements

For instruction purposes, visibility of the fractionating section proper is desired and therefore a dead air space enclosed in a glass jacket wound with an electric heat element was used as insulation. This allows detection of channelling and flooding in the column. The use of electrical heat compensation is also desirable due to the fact that

/25...

it allows control of heat loss so that the latter may be made to stay constant over a wide range of operating temperatures.

When a column is built to serve as a tool of purification, it needs to be an efficient column of the order of 100 plates. Such columns are usually tall, have a low throughput, and, since operation is limited over the higher ranges of reflux ratios, they have a low take-off which means that it will take a long time to collect even 100 c.c. of distillate (several days, including the time it takes the column to come to equilibrium at total reflux). Thus we see that continuous operation over a long period of time is necessary, which makes it desirable to have automatic control of the heat jacket. This would make possible operation with a minimum of effort and attention. Automatic control of stillpot heat is not necessary when a constant-reflux-ratio head is used.

Finally, since few liquids are encountered which boil much above 200° C. without decomposing and since the boiling point may be reduced by operating the column under reduced pressure, the heating jacket does not need to supply heat above 200° C.

II The Design and Construction of the Column

A. The Stillpot

The stillpot was a Pyrex 200 c.c. round-bottom flask fitted with a Buchi RN 9 female ground glass joint, a thermocouple well and a sampling line which also serves to flush the column with nitrogen when operation in an inert atmosphere is required.

The source of heat to the stillpot was a Morey (14)

/2a

type hea ting mantle which was obtained commercially (Glas-Col Apparatus Co., Terre Haute, Ind., U.S.A.). This is a mantle with a heating element in it which envelops the stillpot and thus combines uniform heating over a wide surface area with fast response to the application of heat. It is thus superior to other bumping-reducing arrangements (12) which do not use direct application of heat, thus introducing an element of time lag.

A diagram of the stillpot is found in fig. 9.

B. The Fractionating Section

The fractionating section was a glass tube, 10.6 mm (±0.2 mm) in internal diameter and 13.5 mm (± 0.2 mm) in outside diameter and 254 cms long.

1/16 inch internal diameter Fenske single-turn helices were used as packing. These were obtained commercially (Scientific Glass Apparatus Co., Bloomfield, N.J., U.S.A.). Methods for making these helices are given in the literature (37),(55) but they need special equipment and laborious techniques.

The packing job is important. Plate values ranging from 20 to 95 may be obtained depending on the packing job (50).

The helices were supposed on a 2 cm high base of 1/8 inch internal diameter Fenske single-turn helices on the dropper ring seal at the bottom of the column.

Carney (44) states that helices should be added to the column "in such a way that no bunching takes place"(21). He recommends that a shallow box with a 1 cm diameter hole in it be placed above a funnel leading to the column. A small amount of helices are then poured into the box and a fire-

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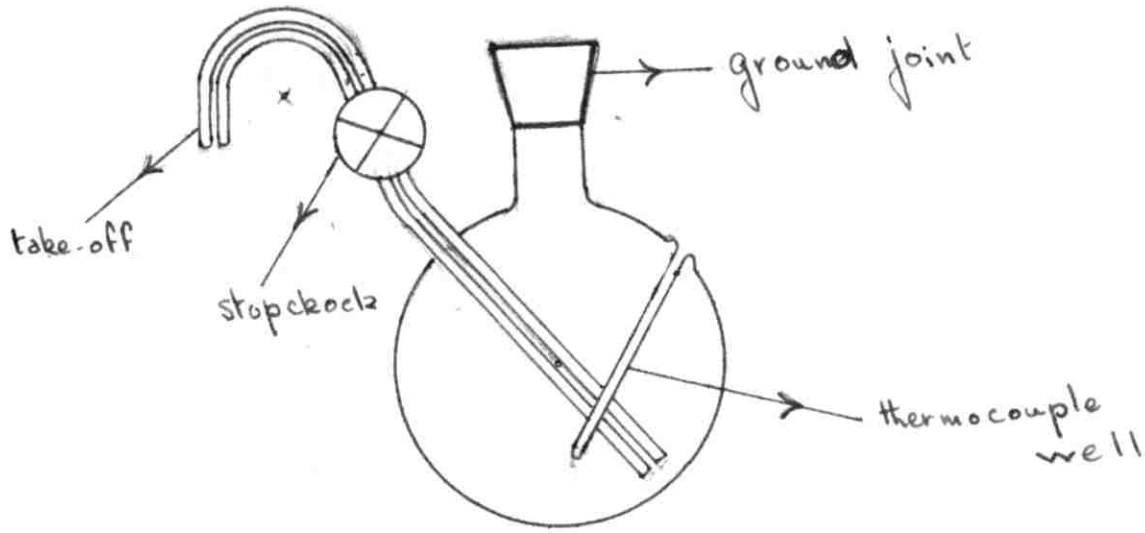
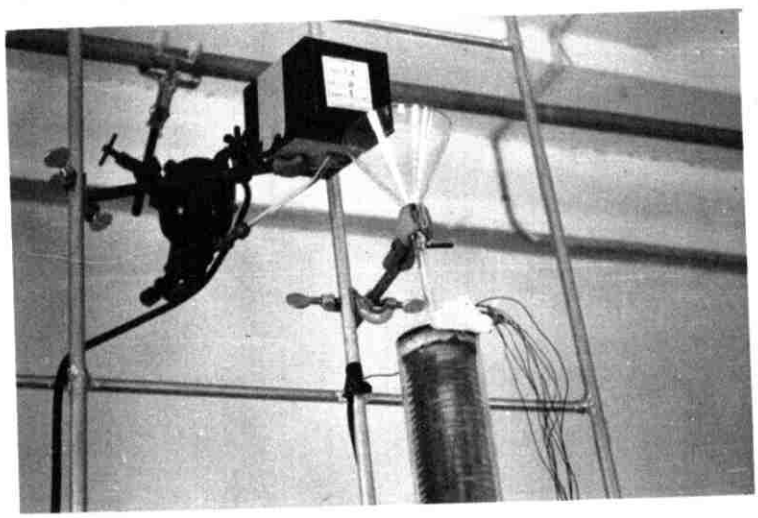


The Still pot

fig 9



A photograph of the packing setup

fig. 10

polished thin glasss rod is then used to separate the helices and push them down through the hole into the column. This method was tried and found to be extremely tedious and laborious. It took one hour to pack 2 c.c. of the column (i.e. a 1.5 cm length of the column) and a different method of packing was devised.

A small hole of about one and one half times the diameter of the helices was bored in a cardboard box about 10 cms high. The box was then clamped above the funnel in such a way that the hole came over the funnel and a part of the box protruded outside the funnel. The box was made to slant very slightly in the direction of the hole. Helices were poured into the box and the lid of the box, slit to accomodate the clamp, was adjusted on top of the box. An eccentric stirrer was then attached to a stirring motor and the stirring assembly was clamped so that the end of the stirrer came just under that end of the box outside the funnel. When the stirring motor was put into operation, the stirrer rotated in such a way as to agitate the box violently in a vertical plane. The result was that the helices were thrown about in the box, became separated from one another and fell through the hole one by one. The hole was too small for helices of more than a single turn to fall through in any appreciable quantities and in fact very few did. By constantly replenishing the helices and regulating the agitation of the box, it was possible to pack from 48 to 72 c.c. of helices per hour (60 to 90 cms of the column). A photograph of the packing setup is shown in Fig. 10.

The first half of the distilling tube was tamped on the outside with a glass rod at intervals of about 1 inch

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of packing each to insure uniform distribution of the packing. It was not possible to do this with the top section as it was packed after the two sections were sealed so that the top section was inside the jacket and there would have been danger of breaking the distilling tube.

The bottom section of the distilling tube was broken 111 cms from the bottom and had to be sealed. For a distance of 2 cms, including the seal, the packing was 1/8 inch Fenske single-turn glass helices in order not to construct vapor flow.

The glass tubes of the fractionating section were chosen from a large stock for straightness and uniformity but the best tubes that could be obtained were still very far from straight, and centering devices had to be used. Four asbestos-board circular disks were bored in the middle and slid onto each distilling section onto which they were firmly attached. They were then filed to give a sliding fit into the jacket. The three bottom disks of the bottom section and the three top disks of the top section were slit, each three on the same side to accomodate the electric wires inside the jacket. Centering the column is important so as to prevent superheating of any one side of the column.

Connections to both bottom and top of the distilling tube are made by means of ground joints. At the top of the distilling tube a Pyrex 24/25 ground glass joint is fitted while at the base of the distilling tube a Buchi RN 9 male ground glass joint is sealed.

A diagram of the fractionating section is shown in figure 11.

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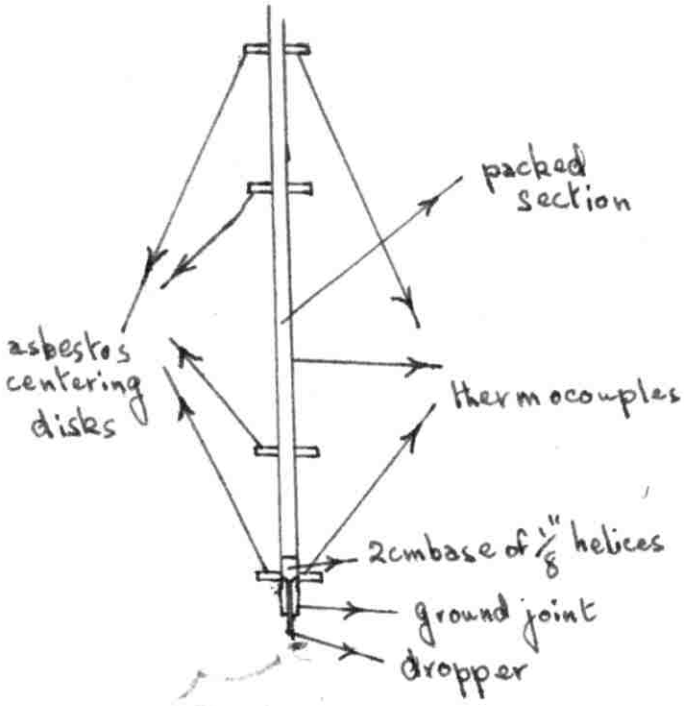

A crossection of the lower part of the fractionating section.

Fig. 11

C. The Head

The head consists of an upright total condenser 20 cms long. Below is a liquid trap fitted with two holes on opposite sides for vapor risers, and with a downpipe in the middle. The downpipe leads to a funnel attached to the downpipe by means of hooks in such a manner as to allow free sideways swinging of the funnel. Along the side of the funnel are attached two iron nails enclosed in glass. At the tip of the funnel, on the side opposite the nails, a very fine drip point is attached.

In the wall of the head, just below the tip of the funnel, a take-off tube leads to the take-off flask through a 6 cms long condenser. The take-off tube is attached to the take-offflask by means of a special spherical Pyrex 18/9 ground glass joint.

With the funnel in the vertical position liquid overflow goes back to the fractionating section. The funnel is made to swing sideways towards the take-off tube by means of an electromagnet placed as near to the wall of the head as

/30...

possible. In the take-off position, liquid flowing down the funnel drips off its tip into the take-off tube.

The electromagnet is activated by means of an electronic timer obtained commercially (G.C. Wilson and Co., Chatham, N.J. U.S.A.). The timer has an on-cycle variable between 0.2 to 316 seconds and an off-cycle variable between 0.1 to 75 seconds. Various combinations of off and on settings on the timer will regulate the reflux ratio.

This type of head is known as a liquid-dividing head and gives a constant reflux ratio independent of variations in the throughput.

The bottom of the head is a Pyrex 24/25 male ground glass joint. To the top of the head, a two-way Pyrex stop-cock is sealed. One arm of the stop-cock leads to nitrogen while the other arm leads to air or may be attached to a vacuum.

A diagram of the head is found in fig. 12.

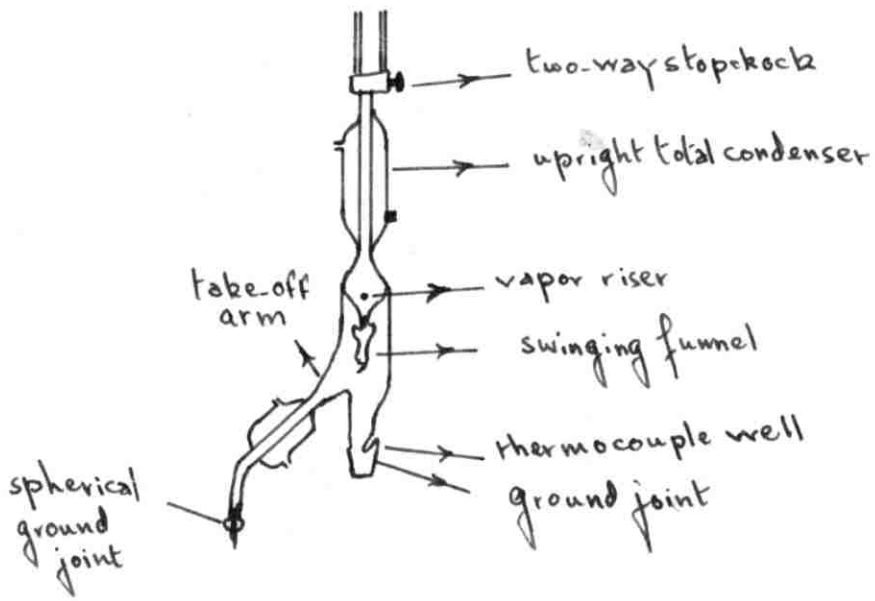

Fig. 12

D. The Heating Jacket

(1) Calculations for the Heating Jacket

Calculation of heat loss from the column was done according to the theory outlined by Highet and coworkers (29):

If heating is done by maintaining a main heating current continuously and switching an auxiliary current on and off and the ratio of the auxiliary heat input to the main heat input is made constant then :-

$$Q = \frac{2\pi k (T_1 - T_2) l}{\log_e r_3/r_2} \quad \text{(neglecting end losses)}$$

$$= k (T_1 - T_2)$$

where

Q : the rate of heat loss (cals./sec.)

k : conductivity of the lagging

T_1 : temperature of the column

T_2 : temperature of the outside of the lagging (assumed constant)

l : length of the column in cms.

r_3 : external radius of the lagging

r_2 : internal radius of the lagging

r_1 : outer radius of column itself

This heat loss is balanced by a constant heat input W and an auxiliary heat input w, the latter being on for a fraction α of the total heating time.

Therefore,

$$Q = W + \alpha w$$

Now, if :

$$\beta = w/W$$

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$$Q = W (1 + \alpha\beta)$$

As goes from 0 to 1, the temperature inside the jacket goes from $T_{1\ min.}$ to $T_{1\ max.}$

Thus,

$$T_{1\ max.} = T_2 + \frac{W}{k'} (1 + \beta)$$

$$T_{1\ min.} = T_2 + \frac{W}{k'}$$

At the midpoint of the temperature range, T_{1a}, $= \frac{1}{2}$ and :-

$$T_{1a} = T_2 + \frac{W}{k'} (1 + \tfrac{1}{2}\beta)$$

Therefore, the temperature range is :-

$$T = \frac{W}{k'}$$

$$= (T_{1\ min.} - T_2)$$

$$= \frac{\beta}{1 + \frac{1}{2}\beta} (T_{1a} - T_2)$$

Finally, of the heat supplied at the jacket winding, a part flows inward to the column and the other part flows outward, the ratio being approximately

$$\log {}^{r_3}/_{r_2} \Big/ \log {}^{r_2}/_{r_1} \qquad \ldots\ (29)$$

A sample calculation, based on the actual characteristics of the column will be given here :-

T_{1a} = 100° C. (mean operating temperature)

T_2 = 25° C.

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r_2 = 3 cm) neglecting the
) thermal resistivity
r_3 = 3.3 cm) of the glass.

l = 120 cms.

For air, $k(100^{\circ}C) = 7.197 \times 10^{-5}$ cals/cm/cm^2/sec.

$$Q = \frac{2\pi \times 7.197 \times 10^{-5} \times 75 \times 120}{\log_e \frac{3.3}{3}}$$

= 42.7 cals./sec.

= 179 watts.

Now, if $\beta = 1$ and $\alpha = \frac{1}{2}$

W = 2/3 Q

= 119 watts

$\Delta T = 50^{\circ}$ C.

Therefore with a main heat input of 119 watts and an auxiliary heat input of the same value the jacket will operate satisfactorily between 75° C. and 125° C.

(Actually due to the low value of $\log r_3/r_2 / \log r_2/r_1$ it was found necessary after the jacket was built to have a heat input of 265 watts to bring the dead airspace temperature up to 125° C.

(2) The Construction of the Jacket

The jacket consists of two sections of Pyrex glass, each 120 cms long and approximately 60 mms in outside diameter. Around each section, Chromel wire, B. & S. No. 24 was wound uniformly with 4 turns per inch leaving 1 cm on each end of a section unwound. The overall resistance of each section was 200 ohms. This was divided by a centre tap into two smaller sections

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of 100 ohms each which were then joined to the voltage in parallel giving a 50 ohm overall resistance to each section. Each section was then fitted into another Pyrex glass tube, 120 cms long and approximately 66 mms in inside diameter. A hole was bored in the middle of each of the outer tube, through which the centre tap lead from the heating jacket was passed. The annular space between the two tubes of the jacket was then sealed off with a paste of asbestos and water.

Chromel wire, no matter how firmly wound onto glass, will sag when it starts to heat up. To prevent such sagging, one side of the jacket was painted with Glyptal paint followed by a coat of sodium silicate.

When the film of paint hardened (it had to be baked for some 24 hours at about 80° C.) the jacket was taken up to 200° C. with no signs of sagging of the Chromel wire.

A diagram of the jacket is found in fig. 13.

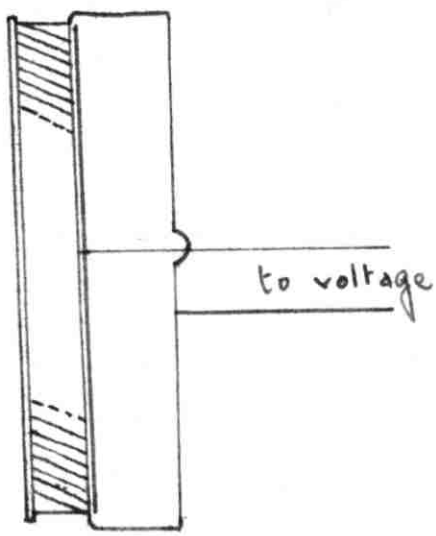

Fig. 13

(3) Temperature Measurement

Temperature was measured at six different points along the column and six opposite points in the dead air space 0.25 cms from the wall of the jacket, in addition to being measured at the head and the stillpot. In all cases copper-constantan thermocouples were used. These were all silver-soldered at the junctions. Temperatures were read from a calibration curve obtained in the following manner.

(a) A potentiometer which could be read within 0.4 millivolts, i.e. within 10° C., was made to allow reading of the temperature within 1° C. by a resistance of about 29 times that of the potentiometer and which could be thrown into the circuit by means of a triple-pole switch. This magnified the scale reading about 25 times. However the galvanometer of the potentiometer is not sensitive enough and this probably accounts for the lack of better sensitivity.

(b) The potentiometer and thermocouples were then calibrated together. This was done by measuring the boiling points of four liquids with the thermocouples. One bare thermocouple hot junction was put into the vapor stream from the boiling liquid and the scale reading of the potentiometer was noted. The temperature was determined by putting a calibrated thermometer in a mercury well

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placed in the vapor stream near the thermocouple and the mercury was stirred so that its temperature was uniform.

The ratios of the four scale readings for the four liquids to the tabulated copper-constantau thermocouple e.m.f. values corresponding to the four boiling points were calculated. An average of the ratios was taken.

(c) The average ratio (22.53) was then used to draw up the calibration curve by multiplying the tabulated values by 22.53 and plotting them against the temperature.

(d) A fifth liquid's boiling point was determined and was found to be on the curve.

The thermocouples are wired as shown in fig. 14 so that there are seven thermocouples to one cold junction in each of the top and bottom sections. Each cold junction is immersed in an ice bath in a Dewar flask.

The thermocouples of the fractionating sections are numbered from 1 to 6. Those against the distilling tube are numbered 1a, 2a, etc.... while those opposite them in the lagging are numbered 1b, 2b, etc.. They are placed at distances of 14, 70, 115, 140, 180, 230 cms respectively from the bottom of the column.

The copper leads from the junctions are connected to the knobs of a rotary multiple switch as shown in fig. 15 to allow quick reading of the temperature at various points along the column. The

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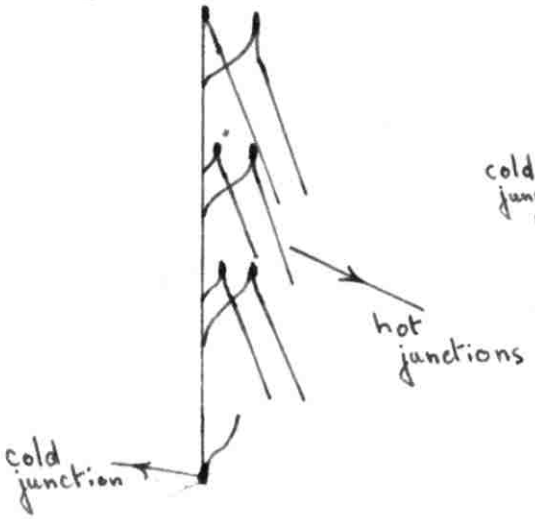

Fig. 14

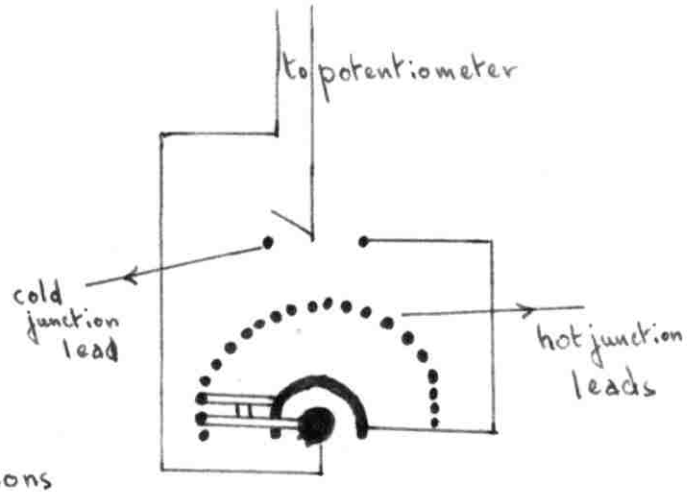

Fig. 15

switch is also provided with an arrangement which allows differential readings of the thermocouples 1a and 1b, 2a and 2b, etc.... so that the temperature differential across the lagging may be easily determined.

(4) <u>The Design and Construction of the Automatic Control Unit</u>

Highet and coworkers (29) used a method of control based on the current generated in a circuit of two thermocouples placed one at the wall of the distilling tube and the other 3/4 inch away in the lagging exactly opposite the first thermocouple, thus setting up a differential thermocouple. Theoretically the column may be made perfectly adiabatic by keeping the differential eaqual to zero. With their set-up they had a difference in temperature between the lagging and the column of 0.4° C. under the best operating conditions and 1° C. under the worst.

The arrangement, while a very ingenious and

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accurate one, necessitates the use of a galvanometer, a photocell, an amplifier, and a relay.

A simpler, while less accurate, method was devised, based on the differential expansion of nitrogen in the two arms of a U-tube placed with one arm against the column wall and the other arm as near to the heating jacket as possible. The U-tube contained mercury in the U and the excess expansion or contraction in the arm aginst the wall of the distilling tube (due to a difference in temperature between the two arms) made or broke a mercury to tungsten contact. The other lead was permanently in contact with the mercury.

When the column temperature is above that of the lagging, the mercury-tungsten contact is made and a circuit is closed. This activates a relay which shorts a resistance of 21 ohms (0.41 x 50 so as to give $\beta = 1$) in series with the jacket winding thereby increasing the heat input to the column. When the jacket side temperature rises, the mercury to tungsten contact is broken, the relay is deactivated and the auxiliary resistance again comes into the heating circuit, thus shutting off the auxiliary heat input.

Nitrogen was used in the arms and a 1000 ohm resistance was added in series to the 600 ohm resistance of the relay coil to reduce the current and thus prevent corrosion of the mercury in the U-tube.

One such control unit is placed in each section of the heating jacket. A diagram of the control unit

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is shown in fig. 16.

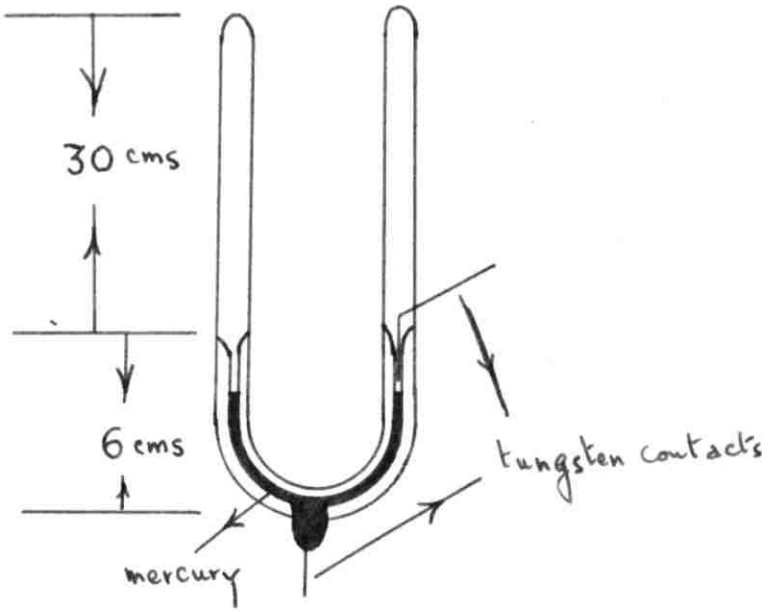


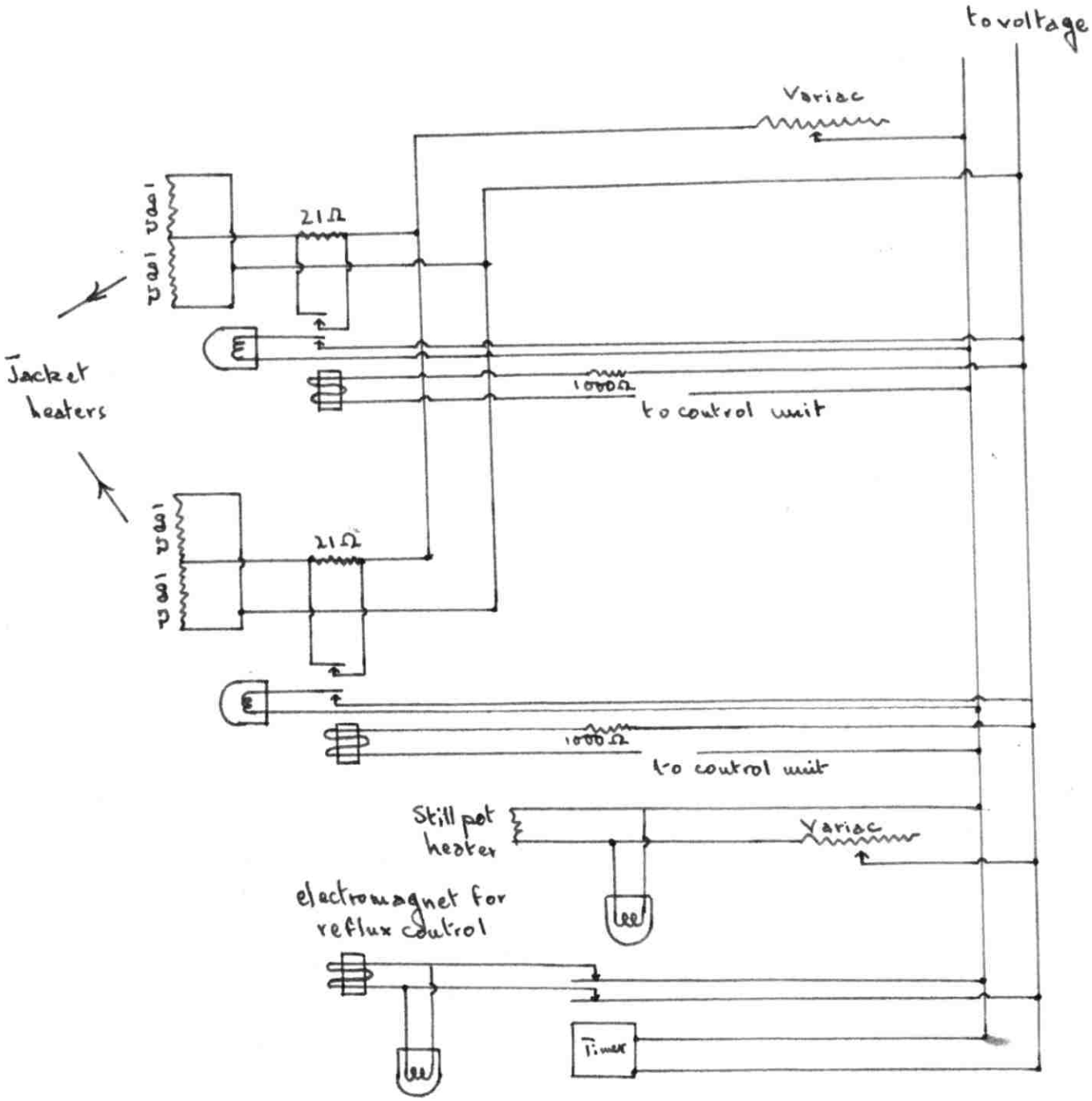
Fig. 16

The construction of the control unit presented certain difficulties. The chief problem was to seal off both arms while maintaining equal pressure in both arms. This was necessary in order to be able to control the level of the mercury below the tip of the tungsten. This was solved in the following manner :-

(a) After the U-tube was constructed and the tungsten contacts sealed into the glass, mercury was filtered and added to the tube until its level was 2 mms below the tip of the tungsten in the side arm to allow for thermal expansion of the mercury.

(b) Another U-tube was then made so that the distance between the arms was the same as the distance between the arms of the unit. This new U-tube had a side-arm attached to it and was about 8 cm long. The side arms

/40...



A diagram of the electrical circuit of the fractionating column.

Fig. 17

of this U-tube were then sealed to the side arms of the control unit. The side arms were then heated and drawn out so as to facilitate sealing them later on in the proceedings. The side arm was then attached by means of a rubber tube to a T-tube, the second and third arms of which were attached to nitrogen and to a vacuum pump respectively.

(c) The system was then repeatedly evacuated and then flushed with nitrogen at atmospheric pressure, after which the rubber tube on the side arm was clamped and a very hot and small flame was used to seal off the side arms at the constrictions.

Thus, the main input is controlled by means of a variac while the control unit controls the auxiliary heat input.

A diagram of the electric circuit of the column is given in fig. 17.

CHAPTER III -- Testing, Operation, and Modification of the Fractionating Column.

1. Testing and Operation of the Column.

A. The Available Free Space of the Column.

This is defined as that part of the distilling tube not occupied by solid packing materia l.

It was determined according to the method outlined in Carney (7). A section of tubing of the same diameter as the distilling tube of the fractionating column and of known volume was packed with 1/16 inch interna l diameter Fenske single-turn glass helices. The volume of alcohol ta ken up to fill this packed section was determined:

Volume of section of tubing= 8 c.c.

Volume of Alcohol added = 3.4 c.c.

Therefore,

$$\text{Available free space} = \frac{3.4}{8} \times 100 = 42.5\ \%$$

B. Testing of Control Units.

The control unit was tested after the column was a ssembled with t he top section unpa cked. This trial was made with wa ter as the boiling liquid.

The control unit of the first section kept the jacket between 2 a nd 6° C lower than the distilling tube temperature whereas the control unit of the top section kept the jacket between 4 and 7° C lower than the temperature of the distilling tube.

Later, the control unit of the top section stopped functioning, probably due to a crack in one of the arms of the U-tube. It was decided to connect the top jacket heater in parallel to the control unit of the lower section.

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After this was done, another trial was made with boiling water. It was found then tha t both ja ckets were functioning with the same te mperature differential (2 to 6° C) between the jackets and the distilling tube.

With a column of the order of 100 theoretical plates, only liquids with a small difference in boiling points need be fractiona ted since liquids with a la rge difference in boiling points ca n be separated by using columns with less theoretical pla tes. Therefore, the temperature gradient along the column from bottom to top will always be small, within 10° C at the most. Using the above arrangement then, we will get supercooling of the vapors at the bottom by 6° C at the most, whereas we will get less supercooling as we go up the column (due to the temperature gradient along the distilling tube) and may even get superheating.

The ratio of the gradients of the top and bottom section ma y be varied by insta lling a variable resistance instead of the constant resistance in series with t he top jacket hea ter winding so as to vary β for the top section.

A trial was made using toluene in the stillpot. Automatic control was used so that the jacket was between 2 and 6° C lower tha n the tube in the first section. Even at the maximum throughput and after boiling for 24 hours, no liquid reached the top of the column and only reached a point 140 cm. from the bottom of the column. It would seem therefore that the 4° C mean temperature differential along the 140 cm. we re enough to condense all the toluene vapors moving up the column. Tha t is probably due to the low heat of vaporization of toluene (98.549 cals. /gm at 25° C (56)). Calculation of the heat

lost due to the tempera ture differential of 4° C between the column a nd the jacket according to the method Hight and co-workers (29), yields:

Heat loss = 132 cals. /min.

The maxium throughput with the jacket 6° C below the column was of the order of 2.5 c.c /min. or 2.2 gms. /min.

We see then, that a heat loss of 132 cals. /min. is serious in the case of toluene (but not in the case of water). Thus the statement of Rose (16) that adiabaticity of operation is essential at low throughputs should be modified to read adiaticity is essential at low throughputs for liquids of low heats of vaporization.

When the heat input to the jackets was regula ted manually after shorting the control unit with a shunt and the temperature differential between the column wall a nd the jacket was adjuste d to zero, the toluene reached the top in 2 minutes. Moreover, the maximum throughput was increased to about 3.4 c.c /min.

In principle, the control unit may be made to opera te in such a way as to give temperature diffenentials of less than one degree by decreasing the distance between the mercury and the tip of the tungsten as much as possible. This would make the control unit suitable for columns built for specific fractiona tions, i.e. operation within a very limited range of temperatures.

The control unit will also operate satisfactorily for fractionations at high throughputs, but will only operate satisfactorily a t low throughputs when the liquids being fractionated have a high enough he at of vaporization as to render hea t loss due to lack of adiabaticity negligible.

Fina lly the control unit may be made to operate satisfactorily by making the heat loss due to a jacket temperature lower by 4° C from tha t of the column negligible. This can be done by decreasing the conductivity of the lagging material. If the jacket can be made air-tight by the application of paint or paste at the top middle and bottom of the jacket, the column may be evacuated and the conductivity cut down. Decreasing the conductivity by a factor of 40 would a llow control of reflux within an error of 5%.

C. Determination of the Holdup.

This was done according to the method of Tongberg and coworkers (46):

1.8960 gms. of stea ric acid were dissolved in 150 c.c. of benzene and placed in the still-pot. The jacket was then adjusted to the boiling point of the benzene and the trial was started. During the run the temperature difference between the column and the jacket was of the order of $\pm^{6}1^{\circ}$ C.

One half-hour after the liquid reached the top of the column, a sa mple was taken from the bottom and cooled in an ice bath to prevent evapora tion. 10 c.c of this sample were taken and eva pora ted and the stearic acid residue was weighed. The thrpughput was 124 c.c. /hour.

Wt. of stearic acid residue: 0.1510 gms.

Concentra tion of ste aric a cid is: 0.0151 gms. /c.c.

$$\frac{1.8960}{X} = 0.0151 \text{ gms./c.c.}$$

$$X = 125.4 \text{ c.c.}$$

where X is the number of c.c.s. left in the stillpot.

Therefore,

The Hold-up = 150 - 125.4 = 24.6 c.c.

Carney states that: that

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"A hold-up of less than 10% of the volume of any component in the starting charge will have little effect upon the shape of the distillation curve, and may therefore be considered negligible " (35) ." However, as this may not be the case with a 200 c.c. stillpot, it would be advisable to have a 500c.c. capacity stillpot for efficiency tests.

D. Determination of the Capacity.

The capacity was determined at total reflux with benzene as the boiling liquid, by counting drops at the bottom of the column as the heat to the stillpot is increased until the number of such drops starts decreasing. This denotes the beginning of flooding. This procedure gave a capacity of 204 c.c./hour. 100 drops of benzene off the drippoint of the column are equivalent to 1.6 c.c.

It is to be noted that as the reflux ratio becomes finite, the capacity will increase as the reflux ratio decreases, because there will be less over flow and the vapor velocity will increase.

E. Calibration of the Timer.

Each of the On and Off cycles of the timer were calibrated with a stopwatch. A graph of the control knob settings for each cycle against time in seconds is found with the control board.

It was found that if the On cycle is maintained at a minimum of 2.4 seconds, the reflux ratio could be determined within 4% from the ratio of the On and Off cycles.

The above calibration was done by pouring liquid at a very low rate through the top of the head, with the timer controling current to the electromagnet. Liquid was collected in two graduated cylinders placed one at the take-off and the

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other below the bottom of the head. The true reflux ratio was calculated from the ra tio of the volumes collected in each of the two graduated cylinders.

F. Modifications of the Head.

(1) A ground glass seal, operated with a magnet was put between the take of tube and the take off condenser to insure total reflux.

(2) A side arm was connected to the take off flasks which could be connected by means of a rubber tube to a side arm above the upright condenser. This was done in order to eliminate back pressure in the take-off flask and insure smooth flow of liquid into:it.

(3) It was found that there were too much condensation in the hea d below the liquid trap so tha t the highest reflux ratio that could be obtained was 2.5 : 1 . Chromel wire S.B. No. 32 was therefore wound uniformely around the head for a distance of 17 cm. from the ground point, i.e.to a point 2 cm. above the liquid trap. The wire was not wound directly onto the glass but onto a 1mm thick layer of asbestos and a layer of asbestos 0.5 mm thick was applied over the heating wire.

The overall resistance of the wire was approxima tely 67ohms and the current may be adjusted by mea ns of a 2 ampere/120 ohm slide resistance.

Two thermo couples were laid against the glass, one near the liquid trap and the other about 5cm. be low the first. The tempera ture of the head may then be brought up to the temperature of the fraction to be taken off before operation is started.

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The above procedure was found to give a maximum reflux ratio of 5:1 (with the funnel in the position of total reflux). This was due to the passage of vapors through the take-off tube. To prevent that, a liquid trap (a capillary U-tube) was sealed between the condenser and the ground glass seal.

It is advisable to keep the take-off arm sealed from the column during operation until equilibrium is attained at total reflux, while operating the timer at a low reflux ratio of the order of 10 : 1 so as to keep changing the liquid held up in the ta ke-off arm between the seal and the head.

Attainment of equilibrium conditions may be tested for by temperature measurment or by measurment of the refractive index of the liquid at the top of the column; the latter being by far the more accur ate procedure.

G. The Efficiency of the Packing.

It is felt tha t a safe estimate (based on article in the litera ture) for the number of plates in the column under total reflux should be of the ord r of 100 plates.

The number of plates cannot therefore be determined using mixtures of benzene and ca rbon tet rachloride or benzene and toluene because these are only suitable for the determination of a small number of plates (of the ord r of 30).

Due to dela ys in shipping, methylcyclolexane was not obtained in time to ca rry out a test with a mixture of n-heptane and me thylcyclolexane.

However work is going on in the department at present with a view to comparing the efficiency of the column in separating hydrocarbon mixtures and mixtures of nitrogen heterocyclic compounds. To that end, vapor-liquid equilibrium data for

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the nitrogen compounds is needed and work aimed at obtaining such data has already started.

Conclusion:-

There is need for further work to clear certain apparant controversies in distillation theory. In genera l most of the work done upon the subject has been carried out with a view to finding the most accura te methods for carrying out fractiona-t ions in the most efficient columns due to the significant material advanta ges to be derived from following such a procedure. Not much effort has been spent on the correlation of e xperiment al findings with distillation theory.

It is felt that there are four main problems which need to be a ttacked:-

(1) The correlation of the viscosity of liquids with the H.E.T.P. and the pressure drop at different throughputs.

(2) The correlation of the surface tension of liquids with holdup and H.E.T.P.

(3) The effect of superheating and supercooling of the insulating jacket upon the efficiency of fractionation and the correlation of the findings with distillation theory.

(4) The determination of the optimum pressure for ope-ration. Myles a nd co-workers (45) state that the optimum pressure is of the order of 200 mm of mercury which apparantly contradicts the findings of Lloyd (59). However, the difference ma y be due to the fact that Myles and coworkers (45) did not use the sa me test mixture a t 760 mm as at the lower pressures.

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For such work as is outlined above, the column needs certa in pieces of accessory equipment:-

(1) Appar atus for maintaining a const ant throughput. A good and simple method for doing so is found in the lit erature (44).

(2) Appa ra tus for ma intaining a consta nt pressure for operation under va ccum. An excellent manostat for the purpose is that of Williams (22).

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References Cited

(1) Zimmerman, O.T. and Lavine, I., " Chemical Engineering Laboratory Equipment," Section 5, (Industrial Reseatch Service , Dover, New Hampshire, 1943)
(2) Perry, J., " Chemical Engineers Handbook", pp. 1445-70 (McGraw-Hill Book Co., Inc., N.Y., and London 1941)
(3) Badger, W.L. and McCabe. W.L., "Elemnts of Chemical Engineering" , Chapter 9 (McGraw-Hill Book Co. Inc; N.Y. and London 1936)
(4) Fenske, M.R., Quiggle,D., and Tongberg,C.O., Ind.Eng. Chem. 24 : 408 (1932)
(5) Podbieluiak, W.J., Ind. Eng. Chem. Anal. Ed., 13: 639 (1941)
(6) Fenske, M.R., Tonberg, C.O., Quiggle, D., and Cryder, D.S., Ind. Eng. Chem., 28: 644 (1936)
(7) Carney, T.P. " Laboratory Fractiona l Distillation", Chapte r 16, (The McMillan Co., N.Y. 1949)
(8) Podbielnia k, W.J. Ind. Eng. Chem. Anal. Ed., 5 : 135 (1933)
(9) Podbielniak, W.J., ibid., 5: 119 (1933)
(10) Carney, T.P., " Laboratory Fractiona l Distillation," Cha pter 8, (The McMillan Co., N.Y., 1949)
(11) Hall,S.A. and Palkin,S., Ind. Eng. Chem. Anal. Ed. 14 : 807 (1942)
(12) Palkin, S. and Chadwick, T.C., Ind. Eng. Chem. Anal. Ed., 11 : 509 (1939)
(13) Podbiedniak, W.J., Ind. Eng. Chem. Anal. Ed., 3 : 181 (1931)
(14) Morey, G.H., Ind. Eng. Chem., Ana l. Ed., 10 : 531 (1938)
(15) Willingham, C.B. and Rossini, F.D., Jour. Resear., Natl. Bur. St. 37 : 15 (1946)
(16) Rose, Arthur, Ind. Eng. Chem., 28 : 1210 (1936)
(17) Othmer, D.F., Ind. Eng. Chem., 22 : 323 (1930)
(18) Bailey, A.J., Ind. Eng. Chem. Anal. Ed., 13 : 487 (1941)
(19) Rose, A, and Long, H.H, Ind. Eng. Chem., 33:684 (1941)
(20) Carney, T.P., "Laboratory Fractional Distillation," p.64 (The McMillan Co., N.Y.(1941)
(21) Carney, T.P., "Laboratory Fractional Distillation," P. 114 (The McMillian Co., N.Y., 1949)
(22) Williams, F.E., Ind. Eng. Chem., 39:779 (1941)
(23) Rose, A., Curtis Jhonson, R.L. and Williams, T.J. Ind. Eng. Chem. 42:2145 (1950)
(24) Daniels, F., Mathews, J.H. and Williams, J.W., "Experimental Physical Chemistry," (McGraw-Hill Book Co. Inc. N.Y., 1934)
(25) Oldroyd, D.M. and Goldblatt, L.A., Ind. Eng. Chem. Anal. Ed. 18:761 (1946)
(26) Chillas, R.B. and Weir, H.H., Ind. Eng. Chem., 22:206 (1930)
(27) Souders, M., Jr., and Brown, G.G., Ind. Eng. Chem., 26: 98 (1934)
(28) Souders, M., Jr. and Huntington, R.L., Corneil, H.G. and Ewest, F.L., Ind. Eng.Chem., 30:86 (1938)

(29) Highet, H.C., Godin, G.W., Philpotts, A.R. and Twigg, G.H., Jour. Soc. Chem. Ind., 69:249 (1950)
(30) Joachim and Beardsley, Am. Rept. Natl. Advisory Comm. Aeronaut., 13:489 (1927)
(31) Holbrook, G.E. and Baxter, E.M., Ind. Eng. Chem. 26:1063 (1934)
(32) Pyott, U.T., Jackson, C.A. and Huntington, R.L., Ind. Eng. Chem., 27:821 (1935)
(33) Sherwood, T.K. and Jenny, F.J., Ind. End. Chem., 27:265 (1935)
(34) Hall, J.H. and Bachman, C.B., Ind. Eng. Chem. Anal. Ed., 10:548 (1938)
(35) Carney T.P., "Laboratory Fractional Distillation", p.43 (The McMillan Co., N.Y., 1949)
(36) Fenske, M.R., Lawroski, S., and Tongberg, C.O., Ind. Eng. Chem.,30:297 (1938)
(37) Price, W.R. and McDermott,W.C., Ind. Eng. Chem.Anal. Ed., 11:289 (1939)
(38) Bragg, L.B., Ind. Eng. Chem. Anal. Ed., 11:283 (1939)
(39) Lecky, H.S. and Ewell, R.H., Ind. Eng. Chem. Anal. Ed., 12:544 (1946)
(40) Craig, L.C., Ind. Eng. Chem. Anal. Ed., 9:441 (1937)
(41) Selker, M.L., Burk, R.E. and Lankelma, H.P., Ind. End. Eng. Chem. Anal. Ed., 12:352 (1940)
(42) Naragon, E.A. and Lewis, C.J., Ind. Eng. Chem. Anal. Ed., 18:448 (1946)
(43) Drickamer, H.G. and Bradford, J.R., Trans. Am. Inst. Chem. Eng. 39:319 (1943)
(44) Carney, T.P., "Laboratory Fractional Distillation," Chapter (15) (The McMillan Co., N.Y., 1949)
(45) Myles, M., Feldman, J., Wender, I., and Orchin, M., Ind. Eng. Chem., 43:1452 (1951)
(46) Tongberg, C.O., Quiggle, D. and Fenske, M.R., Ind. Eng. Chem., 26:1213 (1934)
(47) Egloff, G. and Lowry, C.D. Jr., Ind. Eng. Eng. Chem. 21:920 (1929)
(48) Glasstone, "Thermodynamics for Chemists", pp.323-325 (D.van Norstrand Co., N.Y., 1950)
(49) Robinson, C.S., and Gilliland, E.R., "Elements of Fractional Distillation", Chapter 16 (McGraw-Hill Book Co., Inc., N.Y. and London, 1950)
(50) Hall, H.J. and Jonach, F.L., Proc. Am. Petroleum Inst., 26:48 (1946)
(51) Nickels, J.E., Thesis, Pa. State College (1936)
(52) Ward, C.C., Bureau of Mines, Tech. Paper No. 600 (1939)
(53) Nelson, W.L., "Petroleum Refinery Engineering", p.260, (McGraw-Hill Book Co. Inc., N.Y., 1941)
(54) Hempel, Z. Anal. Chem., 20:502 (1881)
(55) Wilson, C.D., Parker, G.T., and Laughlin, K.C., Jour. Am. Chem. Soc., 55:2795 (1933)
(56) J.Timmermans, "Physico-Chemical Constants of Pure Organic Compound", (Elsever Publishing Co., Inc., N.Y., Amesterdam, London, Brussels, 1950)

/52...

(57) Baker, T., Chilton, T.H. and Vernon, H.C., Trans. Am. Inst. Chem. Engrs. 31:302 (1935)
(58) Mc Mahon, H.O., Ind. Eng. Chem., 39:712 (1947)
(59) Lloyd, L.E., Pet. Ref., Fe bruary, 1950.
(60) Carney, T.P., "Laboratory Fractional Distilla tion, Chapter 9, (The McMillan Co., N.Y., 1949)
(6 1) Carney, T.P., "Labora tory Fractional Distillation", Chapter 2, (The McMillan Co., N.Y., 1949)
(62) Sorel, "La Rechification de l'alcool," Paris, 1893.
(63) McCabe, W.L. and Thiele, E.W., Ind. Eng. Chem., 17:605 (1925)
(64) Fenske, M.R., Ind. Eng. Chem., 24:482 (1932)
(65) Carney, T.P., "Laboratory Fractional Distillation", Chapter 1, (The McMillan Co., N.Y., 1949)
(66) Robinson, C.S., and Gilliland, E.R., "Elements of Fractional Distilla tion", Cha pter 1 (Mc Graw-Hill Book Co., Inc. N.Y., 1950)
(67) Rose, Arthur, Ind. Eng. Chem., 33:594 (1941)
(68) Rose, Arthur, Ind. Eng. Chem., 32:668 (1940)

Bibliography of General References.

Robinson, C.S., and Gilliland, E.R., "Elements of Fractional Distillation", (Mc Graw-Hill Book Co., Inc., N.Y., 1950)

Carney, T.P., "Laboratory Fractional Distillation", (The Mcmillan Co., N.Y., 1949)

Morton, A.A., "Laboratory Technique in Organic Chemistry", (Mc Graw-Hill Book Co. Inc., N.Y., 1938)

Badger, W.L., and McCabe, W.L., "Elements of Chemical Engineering", (Mc Graw-Hill Book Co., Inc., N.Y., 1936)

Zimmerman, O.T., and Lavine, I., "Chemical Engineering Laboratory Equipment", (Industrial Research Service; Dover, New Hampshire, 1943)

Perry, J., "Chemical Engineer's Handbook", (Mc Graw-Hill Book Co., Inc., N.Y., 1941)

EXPERIMENT 12

Fractional Distillation - Packed Columns

I. Theory

For any two liquids to be separated by distillation, the composition of the vapors obtained by boiling the mixture must be different from the composition of the boiled liquid.

Of the three classes of binary mixtures of completely miscible liquids - Figures 1, 2 and 3, only the first may be distilled continuously over the whole range of compositions. The other two classes give what are known as azeotrope (Constant boiling point mixtures) at a certain composition of the liquid and may not be distilled continuously over the whole range of composition but only for the range before and the range after the critical constant boiling point composition.

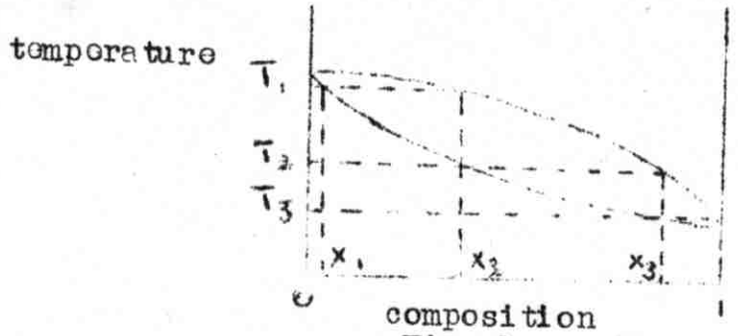

Fig. 1

Maximum boiling point curve

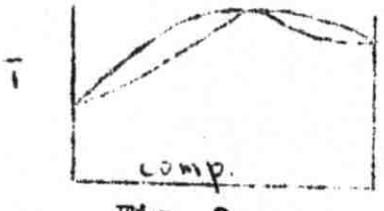

Fig. 2

Minimum boiling point curve

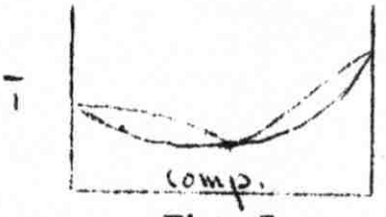

Fig. 3

Let us now consider the class represented by figure 1. Vapor of composition x_3 is in equilibrium with liquid of composition x_1 at the temperature T_1 and vapor of composition x_3 is in equilibrium with liquid of composition x_2 at the temperature T_2, and so on..... Now, if vapor from liquid x_1 is condensed it gives liquid of composition x_2 which in turn gives vapor of composition x_3, all the operations i.e. evaporations and condensations being equilibrium ones.

Fractionation is a series of such operations but, in practice, we can only approach equilibrium conditions

Since a "theoretical plate" is defined as one for which the vapor leaving it to the plate above is in equilibrium with the liquid going to the plate below, every step in the diagram in figure 1 represents one such plate.

A fractionating column provides for a series of evaporations and condensations all in one column and also uses the countercurrent principle for separation by returning part of the condensate, known as reflux, down the column so as to strip the vapors from some of their higher-boiling component.

II- Efficiency of Fractionating Columns

In Bubble-plate (1) or Sieveplate (2) columns, each step takes place on an actual plate. On each of these there is liquid, and every plate provides for vapor to bubble through the liquid on its way up the column.

With these types of columns, a measure of the efficiency is the ratio of the number of theoretical plates as determined experimentally, by the actual separation of a test mixture, to the actual number of plates in the column.

Packed columns used in the laboratory are tubes (made from some non corrosive material) packed with some inert material which increases the surface area for vapor liquid contact. The process here is differential in nature and the measure of the efficiency of a packing material is its H.E.T.P. or "Height equivalent to a theoretical plate".

Various methods (3) have been suggested for calculating this property of a packing material and therefore of its separatory effectiveness. We shall here consider only two of those:-

A.- The McCabe and Thiele Graphical Method (4)

This method is based upon the mathematical treatment of distillation by Sorel and Lewis, who derived the relationship:

$$(1.)\quad Yn = \frac{O_{n+1}}{O_{n+1}+D} X_{n+1} + \frac{D}{O_{n+1}+D} X_D$$

Where:

X_D = mole fraction of the more volatile component in the distillate.
D = Moles of distillate / unit time
n = plate subscript.
O = liquid over flow rate, moles / unit time.
X = mole fraction of the more volatile component in the liquid
Y = mole fraction of the more volatile component in the vapor.

Now, the reflux ratio is defined as

$$R = \frac{O}{D}$$

If the numerators and the denominators of both terms on the right hand side of equation (1) are both divided by D, we get:-

$$Yn = \frac{R}{R+1} Xn+1 + \frac{1}{R+1} X_D$$

which is the equation of a straight line (X_D being a constant) whose slope is: $\frac{R}{R+1}$, and whose Y - intercept is $\frac{1}{R+1} X_D$. It also crosses the x-y diagonal at the point (X_D, Yn) so that $X_D = Yn$.

When the column is operated at total reflux, i.e. R is infinite, the slope of the operating line is equal to one and it coincides with the diagonal of the X-Y diagram (Fig. 4).

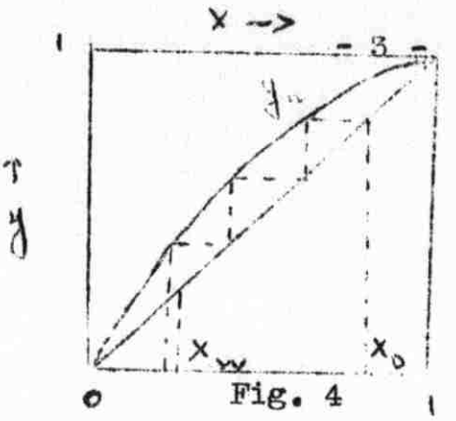

Fig. 4

N.B. for finite reflux, the operating line passes through $(0, \frac{1}{R+1} x_D)$ + (x_D, y_n).

When the operating line is drawn on the X-Y diagram, the drawing of rectangular steps, starting at X_D **until** X_W (the mole fraction of the more volatile component in the stillpot) is passed gives the number of theoretical plates in the **still** , the latter being equal to the number of such steps.

When the reflux ratio is finite, the operating line no longer coincides with the diagonal of the X-Y diagram but must be drawn through the two points (X_D, Yn) and $(0, \frac{1}{R+1} X_D)$. The number of theoretical plates is then determined by drawing rectangular steps in the same way as above between the new operating line and the X-Y curve.

The value thus obtained, since the bottom sample is obtained from the stillpot and not from the bottom of the column is one more theoretical plate than the column as the stillpot is considered one theoretical plate. Now, if N is the number of theoretical plates in the column,

H.E.T.P. = $\frac{\text{Height of packed section (cms or in.)}}{N}$

B.- The Fenske Algebraic Method for Total Reflux (5)

N.B. It should be pointed out that this method is only applicable to operation at total or infinite reflux.

Fenske uses the concept of the relative volatility, α , which is defined by:

$$\left(\frac{Y'}{Y''}\right)_W = \frac{(X')}{(X'')_W}$$

Where W is the stillpot subscript and a single prime stands for the more volatile and the double prime the less volatile component making up the binary mixture.

At total reflux, for the whole column,

$$\left(\frac{Y'}{Y''}\right)_{N+1} = \alpha^{N+1} \frac{(X')}{(X'')_W}$$

Where N is the number of theoretical plates in the column. It is apparant that:-

$$\left(\frac{Y'}{Y''}\right)_{N+1} = \frac{X'_D}{X''_D}$$

III.- Operation:-

A. Draw a graph of refractive index vs. composition (mole fraction of CCl_4) for the system CCl_4 - Benzene. To do this, it is advisable to make up three mixtures of approximate composition X_{CCl_4} = 0.25; 0.50; 0.75, fo CCl_4 and Benzene. For each mixture:

1. Weigh an empty bottle with a stopper.
2. Weigh the bottle after adding some CCl_4.
3. Weigh again after adding Benzene to the CCl_4.

The weighing should be accurate to 0.0002 gram.

B. Plot the X-Y diagram for the Benzene, Carbon tetrachloride mixture from Carney's Values (6) -

C. Put a charge of about 300 cc. of a mixture of CCl_4-Benzene of about 0.25 mole fraction CCl_4 in the stillpot. Turn on the water tap for the condenser such that the rate of flow of water is about 600 cc. per minute (measured with a graduated cylinder and stopwatch).

Close the take-off stopcock. Set the heat at about watts for about 40 minutes after which lower it to watts and keep it constant.

D. It will take about two and one half hours for the column to come to equilibrium (denoted by constant temperatures at still head and still-pot). When it does so, remove two samples, one from the head and one from the still pot of 1 cc. each, as simultaneously as possible and determine their composition by using the Abbe Refractometer. It may be necessary to use suction to obtain the stillpot sample.

Take readings every 5 or 10 minutes for the first hour and then every 20 minutes for the rest of the time until equilibrium is reached.

E. Fill a table of the form:-

Time Minutes	Heat input	Temperature °C.		Drops per minute		
		Stillpot	Head	Take-off	Head	Stillpot

F. Make a second run proceeding as before (it is not necessary to take readings) until the column attains equilibrium, after which the take-off stopcock is opened and takeoff started so as to maintain a finite reflux ratio, R. The value of R is determined by counting the drops dropping off the still head dropper per minute and dividing it by the number of drops coming out through the take-off tube per minute.

G. Calculate the H.E.T.P. under total reflux and under finite reflux. Under total reflux use both methods; for finite reflux the McCabe Thiele method will be used.

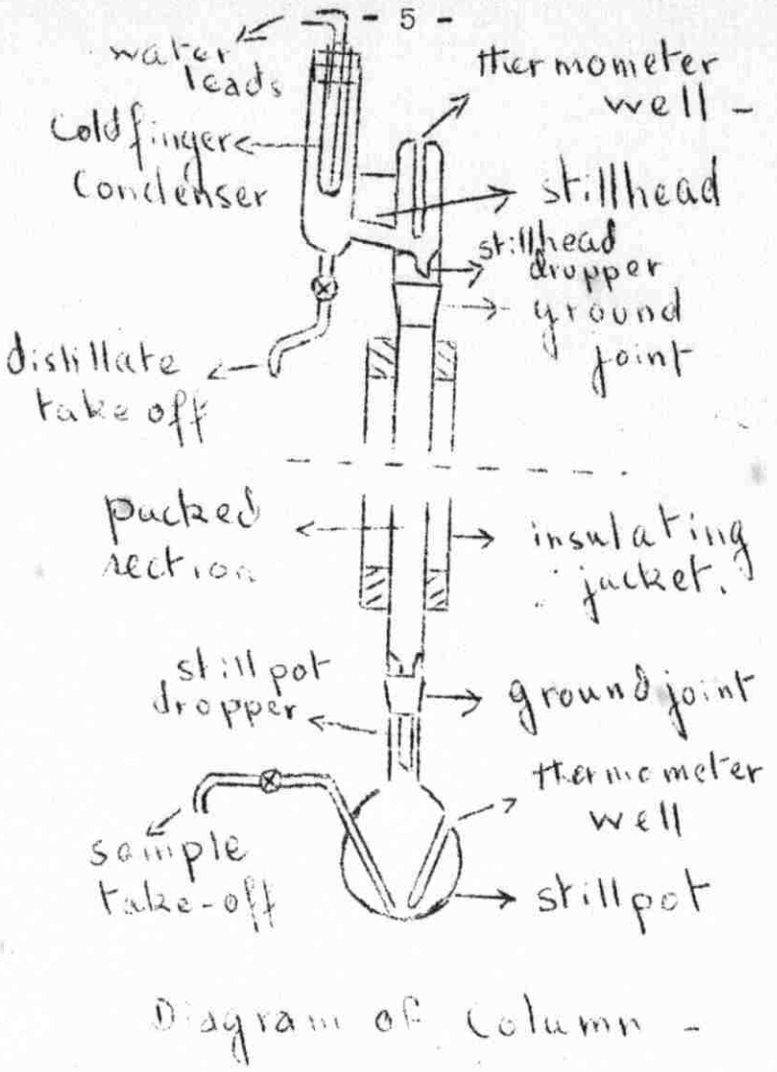

Diagram of Column -

Fig. 5

Referonces:-

1) Robinson and Gilliland, Elements of Fractional Distillation, pp. 403, 404 (McGraw Hill Book Company, N.Y. 1951)
2) Perry, J. Chemical Engineer's Handbook, p. 1445, (McGraw Hill Book Company, N.Y., 1941)
3) Carney, Laboratory Fractional Distillation, pp. 11-21, (McMillan Book Company, N.Y., 1949)
4) Same as (1), pp. 118-123.
5) Same as (4), pp. 174-176.
6) Same as (3), p. 150.